MEASUREMENT BY X-RAY DIFFRACTION OF MACRO-STRESSES
AND MICRO-STRESSES IN TITANIUM AND A TITANIUM ALLOY

by

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SUMMARY

Methods have been developed for the measurement of macro- and micro-stresses in commercially pure titanium (IMI.130) and macro-stresses in 6%Al-4%V titanium alloy (IMI.318A). Macro-stresses were measured by the X-ray line shift technique, using both the film and diffractometer methods. Micro-stresses were measured by the X-ray line broadening technique, using a diffractometer, and 'line' profiles were analysed by the integral breadth method. The accuracy of macro- and micro-stress measurement is discussed.

Good agreement was obtained in the measurement of macro-stresses in IMI.130 and IMI.318A deformed unidirectionally in four point loading, with X-ray diffraction methods, strain gauges and bending theory. The residual macro-stresses only occur in titanium after it has been plastically deformed and are intimately connected with the X-ray limit of proportionality. The effect of deformation in unidirectional and uniaxial tension on the applied and residual macro-stresses in IMI.130 is discussed in the light of current theories and a hypothesis is put forward regarding the existence and measurement of residual lattice strains.

The effects of grinding, machining or shot peening on the residual macro- and micro-stresses in IMI.130 and macro-stresses in IMI.318A are discussed. It is concluded that the deformation processes give rise to both residual macro- and micro-stresses. The various deformation processes produce different patterns of macro- and micro-stress distribution, thus showing the importance of measuring both types of stresses.

Comparison is made between the modes of deformation in shot peened, machined surfaces and in plastically deformed specimens in uniaxial tension. Recommendations for future work are stated.

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List of Symbols

<u>Symbol</u>	<u>Definition</u>
d	Interplanar spacing
d_0	Interplanar spacing, stress-free
d_ψ	Interplanar spacing at angle ψ
$d_\perp$	Interplanar spacing at $\psi = 0^\circ$.
ψ	Angle between specimen and diffracting plane normals
ϕ	Angle indicating direction of stress, ($= 90^\circ - \theta$) Appendix 2 Fig. 1.
n	An integer <i>representing diffraction order</i>
λ	Wave length of radiation
θ	Angle of diffraction
θ_ψ	Angle of diffraction when $\psi = \psi^\circ$.
$\theta_\perp$	Angle of diffraction when $\psi = 0^\circ$.
η	Angle between incident beam and diffracting plane normal Appendix 3 Fig. 1.
$K\alpha_1, K\alpha_2$	Characteristic emission lines of incident radiation
ϵ	Unit strain, micro-strain
E	Young's modulus
ν	Poisson's ratio
x, y, z	Co-ordinate axes
$\epsilon_1, \epsilon_2, \epsilon_3$	Principal strains
$\epsilon_x, \epsilon_y, \epsilon_z$	Strains in directions noted
ϵ_n	Normal strain
ϵ_ψ	Strain in direction ψ
$\alpha_1, \alpha_2, \alpha_3$	Direction cosines of ψ direction vector Fig. 1. Appendix 2
$\sigma_1, \sigma_2, \sigma_3$	Principal stresses
$\sigma_x, \sigma_y, \sigma_z$	Stresses in directions noted
β	Integral breadth
β_{PF}	Broadening due to crystallite size
t	Mean particle dimension
K	Constant which varies with crystallite shape
β_S	Broadening due to micro-strain
β^*	$\frac{\beta \cos \theta}{\lambda}$
d^*	$\frac{2 \sin \theta}{\lambda}$
β_T^*	Total broadening (crystallite size, micro-strain and instrumental)
β_S^*	Broadening due to crystallite size and micro-strain
μ	Linear absorption co-efficient.

SymbolDefinition β_I^*

Instrumental broadening

 R_C

Rockwell hardness

a

/Y Intercept

b

Slope of line

c

Y Intercept lay in the range

d

Slope of line lay in the range

e

Co-efficient of correlation

f

T distribution parameter

Fig. 29 - Fig. 30

1. INTRODUCTION

Residual stresses result from non-uniform plastic deformation, brought about by heat treatment, deformation or machining. The strains produced by such operations, and retained after all external forces are released, are known as residual strains and the accompanying stresses are known as residual stresses.

Heat treatment, bending or rolling produce stresses which vary through the section of a component or structure whilst operations such as turning, milling, grinding, shot blasting, shot peening, plating, or etching produce effects which are generally confined to the surface regions.

When significant residual stresses are present in a material, these can influence many properties such as distortion during machining, work-hardening, corrosion resistance or fatigue strength.

The fundamental principles upon which stress determination by the X-ray diffraction technique is based are set forth in most text books (Cullity (1), Barrett (2), Taylor (3), Barrett & Massalski (4)). Various methods of applying X-ray diffraction techniques to the measurement of surface stresses have been published, from early papers of Frommer & Lloyd (5), Thomas (6), Hawkes (7), Moore (8) to the more recent methods employing geiger-counters for stress measurement of case hardening steels. An evaluation of these later techniques has been published by Beu (9). The basis of macro-stress measurement by X-ray diffraction is, that when a crystalline piece of metal is deformed elastically in such a manner that the strain is uniform over relatively large distances, the lattice plane spacings in the constituent grains change from their stress-free value to some new value corresponding to the magnitude of the applied stress, this new spacing being essentially constant from one grain to another for any particular set of planes. This uniform macro-strain causes a shift of the diffraction line to a new position. On the other hand, if the metal is deformed plastically the lattice planes usually become distorted in such a way that the spacing in any particular set of planes varies from one grain to another or from one part of a grain to another. This non-uniform micro-strain causes a broadening of the corresponding diffraction lines. Further broadening of the lines is produced by a reduction in crystallite size, by faulting on certain planes, and by the instrumental effect associated with the method of recording the profile. In a plastically deformed material,

both kinds of strain are usually superimposed, and diffraction lines are both shifted and broadened.

There are four review articles (Greenough (10), Vasileb Smirnor (11), Macherauch (12), Denton (13) which have summarised the state of affairs in this field up to 1952, up to 1961 and up to 1966 respectively. Recently Kirk (188) has reviewed the theoretical considerations, experimental features and practical applications of X-ray diffractometer techniques to the accurate determination of residual macro-stresses. Lewis and Northwood (14), in their review article, have discussed the effect of micro-strains on the physical and chemical properties of various materials. Evans and Millans (26) have shown the effect of micro-strain and particle size on the fatigue properties of steels at various hardness levels. Evans and Buerneke (198) have investigated the influence of micro-strains and particle size resulting from hardening and cold working of SAE 1045 steel and further correlated these measured parameters to fatigue properties.

Some published information had indicated that attempts to measure macro-stresses in titanium by the X-ray back reflection method had not been successful for the following two reasons:-

- i) insignificantly small line displacement
- ii) lack of requisite basic data on titanium necessary for the calculations involved.

Earlier work carried out in these laboratories indicated that with Cu K α radiation the quality of films obtained was poor, firstly because the wave length for Cu K α ($\lambda = 1.54\text{\AA}$) is shorter than the K absorption edge of titanium ($\lambda = 2.54\text{\AA}$) and this probably caused the reflections to be weak. The fluorescent radiation emitted by titanium was also considered to be a contributory factor to the poor film quality.

The surface treatment of titanium prior to stress measurement is very important because titanium has a strong affinity for oxygen, and even thin oxide films could affect the stress analysis. X-rays of the wave length used for diffraction only penetrate surfaces to a depth of the order of a few tenths of thousandths of an inch and this requires that special attention should be paid to surface preparation.

Weaver and Muller (15), Voigt (16), Ogilvie (17), Lihl (18) and Koisten and Marburger (19) have stressed the importance of surface preparation in the stress analysis of steels.

Earlier work carried out in these laboratories (20) had indicated that electropolishing of titanium was not a satisfactory surface treatment, because it resulted in changes in the diffraction pattern. The effect of surface preparation of titanium on the quality of films produced by the X-ray back reflection technique was therefore considered to be a very important factor in stress analysis of titanium.

It is assumed in the classical theory of elasticity that the material being examined is elastic, homogeneous and isotropic. Hawkes (7) and Moore (8) successfully applied the assumption of isotropy to materials not necessarily isotropic, in fact, having F C C and B C C crystal structures. At room temperature, titanium consists of α (CPH structure), and high strength titanium alloys in current use have a duplex structure consisting of α phase (CPH structure) in a matrix of β phase (B C C structure). In view of the fact that the α phase (CPH structure) is a fairly anisotropic material, it was considered doubtful whether the classical theory of homogeneous elasticity could be applied successfully to titanium and its alloys.

In the initial stages therefore investigation was confined to establishing the technique for measuring surface residual stresses by the multi-exposure X-ray back reflection technique. The material investigated was commercially pure titanium (IMI.130) using different unfiltered K α radiations with a suitable calibration powder. The effect of the surface preparation procedure on the quality of X-ray films was fully investigated. The surface strains induced by bending were calculated using elementary bending theory and compared with those evaluated by the multi-exposure X-ray back reflection technique. Later a method was developed to measure both the micro-strains and macro-strains using the diffractometer.

The main aims of the present investigation were:-

- (1) to see if X-ray diffraction methods could be applied to measure residual stresses in titanium;
- (2) to develop techniques for surface preparation prior to measuring stresses by X-ray diffraction;
- (3) to develop multi-exposure X-ray back reflection technique to measure stresses in commercially pure titanium (IMI.130) and to compare these with the applied stresses measured by strain gauges and also calculated by bending theory;

- (4) to develop a suitable jig to measure stresses by the diffractometer. The development of the jig involved the application of either tensile or compressive stresses to the upper surface of strips;
- (5) to develop methods for measuring micro-stresses in IMI.130;
- (6) to investigate the effect of surface treatments i.e. grinding, shot peening, vapour blasting, various modes of machining, bending and uniaxial tension on both the surface and subsurface macro and micro-stresses for IMI.130;
- (7) to develop multi-exposure X-ray back reflection and diffractometer techniques to measure macro-stresses in the Ti 6%Al-4%V (IMI.318A) alloy and to compare these with the applied stresses measured by strain gauges and calculated by the bending theory;
- (8) to calculate the X-ray elastic constants for both the IMI.130 and IMI.318A;
- (9) to explain the origin of residual lattice strains in IMI.130 deformed plastically in uniaxial tension.

2. LITERATURE SURVEY

2.1. Technological Importance of Residual Stresses.

2.1.1. Residual stresses are present in virtually every structure or machined part as a result of the processes used to make them. Residual stresses from non-uniform plastic deformation are brought about by heat treatment, deformation, machining. Normally residual stresses are an intrinsic feature of most of the machining and fabrication operations. The magnitude and sign of the residual stress system depends largely upon the processing conditions and the mechanical properties of the material at the time of processing.

2.1.2. The influence of residual stresses on the performance of a component in service is of great importance. This influence may be beneficial or detrimental. The full exploitation of available residual stresses produced by proper design and process control depends upon the reliability of the method of measurement and an adequate understanding of the origin and effects of these stresses. High surface residual tensile stresses can be deleterious as they may cause stress corrosion and fatigue fractures. In the past, techniques have been developed and used to measure the macroscopic surface residual stresses for example by strain gauges, diffraction techniques, and sectioning procedures. The influence of surface residual stresses arising from surface treatments such as grinding, shot peening, carburising and from various cutting and forming operations on the apparent behaviour of material properties has been investigated and applied with success. Much of this work has been reported in the standard references. Although the theory and practice of micro-stress measurement by X-rays is well established, it has been rarely applied. This is surprising when it is considered how widely X-rays are used for measurement of macro-stresses.

2.1.3. The existence of a system of micro-stress in a body is reflected in many of its properties, but in spite of this fact it is very difficult to obtain a detailed picture of the magnitude and distribution of the micro-stresses. This is due to the fact that the sensitive properties are generally those which can only be measured on a bulk specimen whilst the stresses exist on a localised scale hence the result is some sort of root-mean-square value of the stress. The general micro-stress level is thus available but nothing of a very specific nature. Typical properties which are measured are:-

- (1) intensity of the measured magnetisation for ferro magnetic substances
or
- (2) the shape of the X-ray diffraction line.

2.1.4. The micro-stresses play a very important role in the performance of an engineering structure, particularly in those basic considerations dealing with the mechanisms of fracture. The existence of micro-stresses is a fundamental feature of the problem of crystalline solids and the question of crystallinity and its consequences cannot be divorced from any precise treatment of the behaviour of solids. The initiation of fracture is a very localised phenomenon and is apparently influenced by the local stress situation. Micro-stresses also effect the mechanical and chemical properties of the material.

2.1.5. In view of the technological importance, several attempts have been made to examine the X-ray diffraction line breadth in relation to the fatigue phenomenon with varying results. Gough and Wood (21) working on steel, considered that the line breadth only increased markedly if the applied stress was of such a magnitude as to cause eventual failure. Barrett (22) did not consider that there was any such correlation of line breadth with safe and unsafe limits. This conclusion was also reached by Terminassov (23) who made many quantitative measurements of line breadth during the fatigue life of steel specimens tested at various stress levels. He found that the line breadth always increased during the early cycles, and then remained approximately constant for all stress levels, and that there was no significant difference between the effects of safe and unsafe stresses.

2.1.6. Niemann and Stephenson (24) have studied the line breadth in relation to the damping capacity shown by specimens of alpha brass after cold working. They concluded that there was no correlation between the two properties.

2.1.7. Hawkes (25) studied the effect of micro-stresses on the stress corrosion properties of the aluminium alloy DTD.5044 and concluded that micro-stresses arising from cold work did not affect the stress corrosion properties of this alloy. Evans and Millans (26) have studied the effect of micro-strains and particle size on the fatigue properties of steel shot peened to give various hardness levels. They found that r.m.s. strain increased and particle size decreased with the hardness.

2.1.8. Evans and Bueneke (198) have investigated the influence of micro-strains resulting from hardening and cold working of SAE 1045 steel. They have shown that micro-strains caused by either hardening or shot peening varied markedly over micro-regions. In general, increasing the hardness

or peening intensity increased the r.m.s. strain and decreased the particle size. They have further related these measured parameters to fatigue properties.

- 2.1.9. Lewis and Northwood (14) in a review article have shown that micro-stresses can greatly affect the physical and chemical properties of the material i.e. corrosion resistance, reactivity, mechanical properties, hardness, etc. For example, they have shown that micro-strains can affect the electrode potential and hence the corrosion rate of copper wire, the rate of dissolution of lithium fluoride powder, and the crushing strength of sintered compacts. They have further shown that micro-strains can produce a change in the structure of the material, which can affect its subsequent ease of fabrication.
- 2.1.10. Titanium has a unique combination of high strength, low density and excellent corrosion resistance and its applications vary widely from aerospace industry, metal finishing, to the general engineering industry. In spite of the commercial importance of titanium the true effect of macro- and/or micro-strains on the physical, mechanical and chemical properties of titanium has not been fully evaluated. Recently Braski and Royster (27) have shown the influence of glass bead peening, sand and oxide blasting and vibratory tumbling treatment on the surface residual stress patterns of 6%Al-4%V-titanium alloy and 8%Al-1%Mo-1%V titanium alloy.
- 2.1.11. The aim of this investigation was to establish the technique for measuring both the macro and micro-strains in titanium, and to correlate the influence of macro as well as micro-strains on the physical, mechanical and chemical properties as far as possible.
- 2.1.12. A table follows of the technological properties of titanium affected by micro-strains:-

Physical Properties	Chemical Properties
Hardness, Ductility, Fatigue Wear, Damping Capacity, Fracture-toughness, Fracture mode	Etching, Corrosion, Chemical Milling

2.1. Transformations in Titanium and Titanium Alloys.

2.2.1. The available titanium alloys are of three types: alpha, alpha-beta and beta. These designations refer to the predominant phases present in the micro-structure.

The heat treatment potentialities of most titanium base alloys are based on the fact that titanium possesses an allotropic transformation at 882°C . Below this temperature titanium has a close packed hexagonal crystal structure (alpha) - whilst above this temperature it has a body centred cubic form (beta). The formation of the higher temperature phase, beta, by heating, followed by rapid cooling to below the transformation temperature to retain some of the beta, and its subsequent transformation to the lower temperature phase, alpha, constitutes the basis of the mechanism of titanium alloy transformation hardening.

2.2.2. The behaviour of titanium alloys in passing through the beta - alpha change has certain features in common with that of steel passing through the gamma - alpha change i.e. martensitic and pearlitic types. The terms involved in the micro-structures are defined as follows:-

Alpha: hexagonal close packed crystal structure.

Alpha prime: alpha which has formed from beta by a shear mechanism (martensitic reaction) which gives rise to an acicular micro-structure. Alpha prime has the same crystal structure as alpha. It is super-saturated alpha.

Beta: body centred cubic crystal structure.

Beta prime: has a quenched beta micro-structure but has higher hardness than the equilibrium beta. It is unstable beta which has partially transformed giving submicroscopic alpha with consequent hardening.

Unstable beta: non-equilibrium beta which, at the temperature under consideration, has composition lying outside the equilibrium range of beta stability. Being unstable it has a tendency to transform to other phases. Properly controlled this transformation can achieve useful strengthening while retaining adequate ductility.

2.2.3. The heat treatment of titanium alloys has to be approached with caution because of certain metallurgical problems which are peculiar to

titanium. For example, titanium alloys exhibit "omega" embrittlement. Ageing at 350 - 400°C beta transforms to a transition phase "omega" when the retained unstable beta decomposes to alpha:-

2.2.4. The formation of omega in the beta matrix is accompanied by maximum hardness and severe embrittlement. Ductility can be restored by over-ageing, when transformation to alpha occurs.

2.2.5. If alloys are cooled slowly from the beta range, alpha separates from beta by a nucleation and growth mechanism; the alpha forms both at the grain boundary and within the grains. The residual beta may be retained or may change to a martensitic-like form of alpha.

2.2.6. In practice, the heat treatment of alpha-beta alloys is seldom performed apart from annealing to relieve stresses and to form small rounded islands of beta. Heating above the beta transformation temperature often results in loss in ductility and an undesirable alpha + beta Widmanstätten structure. Good properties are obtained by forging the material in the temperature range in which fine alpha separates from beta.

2.2.7. Alloying elements are important since they affect the alpha - beta change point and often favour one or other of the two structures, or are neutral. Aluminium, for example, stabilises the alpha structure, by raising the temperature of alpha-beta transformation (constitutional diagram Fig.34). Unlike steel, the martensite formed is not hard and is of little practical value because the solute is substitutional instead of interstitial (as carbon in steels) and far less distortion is produced. Aluminium has two outstanding advantages, firstly it is cheap and readily available, and secondly its atomic weight is appreciably less than that of titanium and hence aluminium additions will decrease the overall density and thus increase the strength to weight ratio. Jaffee et al (187) have reported that the addition of aluminium to titanium produces a rapid increase in tensile strength, especially if the amount of aluminium is greater than 4% wt whilst ductility reaches a minimum value of 15%.

2.2.8. Vanadium, on the other hand, lowers the transformation temperature making beta stable at lower temperatures (Constitutional diagram, Fig.36). The relative amounts of alpha and beta stabilisers in the alloy and the heat treatment, determine whether the micro-structure is predominantly single phase alpha, a mixture of alpha and beta phases, or single phase beta over its useful temperature range.

2.3. Deformation Mechanism in Titanium.

2.3.1. Titanium deforms at room temperature both by slip and by twinning.

Slip normally occurs in metals on the plane of greatest atomic density and widest interplanar spacing. In hexagonal metals having an axial ratio greater than that for ideal close-packing (i.e. Zn 1.856, Cd 1.886), this plane is the basal plane. The ideal value of axial ratio is 1.633. In titanium which has an axial ratio (1.587) appreciably smaller than the ideal value, the basal plane is neither so closely packed nor so widely spaced as in zinc and cadmium and therefore other slip systems operate as well. The greater part of the slip observed occurs on the $\{10\bar{1}0\}$ plane in the $\{11\bar{2}0\}$ direction. Slip on the $\{10\bar{1}1\}$ planes also occurs in the $\{11\bar{2}0\}$ direction but this is less important. Rosi, Dube and Alexander (140) observed that in coarse grained specimens of commercial titanium slip occurred only when all three $\{10\bar{1}0\}$ systems were operative.

2.3.2. Kink bands have been observed (141, 143) in titanium during basal or $\{10\bar{1}0\}$ slip with the bend plane $\{11\bar{2}0\}$ at right angles to the operative slip direction. These bands have the same crystallographic geometry as those observed in aluminium and a number of other hexagonal metals.

2.3.3. Rosi and co-workers have found that the twinning occurs in titanium as a result of room temperature deformation, the twin planes being $\{10\bar{1}2\}$, $\{11\bar{2}1\}$, $\{11\bar{2}2\}$, $\{11\bar{2}3\}$ and $\{11\bar{2}4\}$. Anderson and Jillson (144) have confirmed the findings of Rosi and his co-workers. They have established a relationship for the deformation mode in terms of the initial orientation in the following manner:- for orientations in which the angle between the basal plane and the tension axis is about 40° or less, prismatic slip occurs; for angles between 40° and 60° , basal slip occurs but is replaced by $\{11\bar{2}1\}$ twinning when restraints such as grips or grain boundaries are present; for compression $\{11\bar{2}1\}$ twinning gives way to $\{11\bar{2}2\}$. Above 60° , $\{11\bar{2}1\}$ and $\{10\bar{1}2\}$ twinning occurs and at 75° twinning on $\{10\bar{1}2\}$ is apparently the only mechanism. Rosi and his co-workers have shown how slip can be considered in terms of corrugated slip planes.

2.3.4. Churchman (134) has studied the formation and removal of twins in grains of α titanium deformed by bending. On bending a crystal 2 mm thick to a radius of 110 mm, $\{10\bar{1}2\}$ twinning was observed to form on the compressive side of the neutral axis. Further bending caused these twins to increase both in length and breadth and to assume the characteristic lenticular form. On bending the specimen in the opposite sense so that

it again became flat, the twins almost disappeared. Traces of the twins remained, however, forming nuclei, which were capable of growing again immediately the crystal was rebent in the original sense. Churchman found that if this last bending process is continued, the twins eventually meet and form a continuously twinned area, after which unbending the specimen leaves an increased number of twin traces, including those already present before severe bending. Twin boundary migration during growth and absorption occurred on each face of the twins but not necessarily at the same rate on each side, and not always continuously. This type of behaviour strongly supports the view that a twin boundary consists of an ordered array of dislocations and that in titanium, unlike iron, the stacking faults so introduced into the lattice by the movements of twinning dislocations are of low energy. The fact that a greater stress is required to initiate a twin than to increase its size after formation is in accordance with the theory of mechanical twinning of Cottrell and Bilby (145).

2.3.5. Partridge (155) has studied the mode of deformation in close packed hexagonal metals including titanium after tensile deforming, using the transmission electron microscopic technique. At low strain of $\frac{1}{2}\%$ he found that deformation varied greatly in different grains. The examination indicated that the dislocations mostly lay in their slip planes and appeared as long screws in basal plane foils. Small loops and jogs were frequently associated with these screws. Similar observations have been observed in single crystals (199). After 10% strain, the interaction between slip and twinning was reported, and after 15% strain, After 15% strain, the dislocation density was much greater with a large number of dipoles and severely jogged dislocations. After 15% strain, the first evidence of sub-structure formation was apparent as long low angle boundaries parallel to the traces of the predominant prism slip planes. After 25% strain, well developed polygonised structure was observed. Partridge also found two types of twins i.e. $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$; $\{10\bar{1}2\}$ being wide and lenticular whilst $\{11\bar{2}1\}$ formed narrow intersecting lamellae. Some grains contained extensive twinning after 10% strain and it became difficult to identify twins from the transmission electron microscopy. Therefore Partridge developed the use of stereographic projection for identifying twins from electron diffraction patterns. Partridge also found stacking faults within the twins. Partridge and Peel (200) whilst studying the effect of cyclic strain on commercially pure titanium have shown that in cyclic strain, fatigue cracks nucleate at the twin boundaries.

2.3.6. Cass (201) observed $\leq C + a \rangle$ slip in deformed polycrystalline titanium. Partridge has argued that twin formation and perhaps some interaction between slip and twin could give rise to $\langle C + a \rangle$ slip and if so then $\langle C + a \rangle$ could be associated with twins. Cass, however, did not observe this during his experimental observations. Generally the problem of relative significance of slip and twinning is not clear. Reed-Hill et al (202) have shown that $\{0\bar{1}2\}$ twins can be attributed to the ductility of both titanium and zirconium. They also observed that the macroscopic geometry of deformation of a tensile specimen, with basal planes approximately parallel to the rolling direction, differs markedly depending on the extent of twin formation. This could be attributed to the onset of $\langle C + a \rangle$ slip, to twin formation or to new slip directions in the twinned material.

2.3.7. Keeler and Geisler (203) have studied the preferred orientation in rolled and annealed titanium by the Geiger counter spectrometer X-ray diffraction technique and concluded that there were five annealing textures depending upon the annealing temperature. Dillamore and Roberts (204) in their review article on "preferred orientation in wrought and annealed metals" have discussed the deformation texture, theories of texture development and the theories of the formation of recrystallisation texture in close packed hexagonal metals including titanium.

2.3.8. Industrial Deformation Processes

- 2.3.8.1. The plastic flow characteristics of the materials when bent in a bending jig, and when shot peened, turned, ground, etc., are different. On either side of a bent beam, the outer fibres are stressed predominantly in the one direction, namely, parallel to the neutral axis. If the beam is bent plastically and then unloaded, the spring back of the elastically strained interior throws the side which was in tension during bending into compression, whereas in shot peened materials the flow occurs in many directions at once.
- 2.3.8.2. Bending, uniaxial tension, etc. produce stresses which vary through the section of a component, whilst shot peening, grinding, turning, milling, etc. produce effects which are confined to the surface regions. In this investigation, an attempt has been made to study the effects of these two modes of deformation. The two processes, i.e. shot peening and grinding are briefly reviewed in the following sub-paragraphs:

2.3.9. Shot Peening.

2.3.9.1. Shot peening is the process of work hardening a metal surface by bombardment with high-velocity hardened steel balls or shot, thus leaving the surface covered with overlapping shallow indentations. The plastic deformation and work hardening of the shot peened surfaces leaves the surface layers in a state of residual compressive stress. The depth of penetration of this stress field depends on the intensity of the peening operation. Below the compressive state in the metal surface, a balancing residual tensile stress field is present.

2.3.9.2. The residual stresses induced depend upon the control of the following variables:-

- i) Material and shape of the part being peened.
- ii) Material and size of the shot.
- iii) The velocity and angle of impingement of the shot.
- iv) The interference of the shot in the shot stream with rebounding shot.
- v) Quantity of shot flowing.
- vi) Time of peening.

2.3.9.4. The details of the equipment and operation have been summarised elsewhere (160, 161, 162, 163). Almen developed a convenient test which is widely accepted as a guide for specifying and controlling peening intensity (160, 161, 162, 163). Horger and Neifert (164) suggested that the impact pressure of a single shot, calculated from Herz theory, might serve as an index of shot peening intensity. They recognised, however, the problem involved in determining the actual shot velocity and that coverage is an important factor not recognised by this index. The more important factors to be considered in selecting shot peening conditions have been studied by several investigators (160, 161, 162, 165, 166, 167, 168, 169, 170) but the large number of variables involved make it advisable to check each new peening operation experimentally. Several investigators have measured the residual stresses induced by shot peening (171, 172, 173, 174).

2.3.9.5. Evans and Millan (26) have studied the effect of micro-strain and crystallite size on the fatigue properties of steel at various hardness levels. Changes in these quantities were produced by shot peening. They concluded that at 50 R_c the increase in fatigue limit was mainly due to high induced compressive macro residual stresses while at low hardness levels the fatigue limit of steel was enhanced by large changes in micro-strain and particle size.

2.3.10. Grinding.

2.3.10.1. Residual stresses introduced by grinding operations may vary widely in magnitude and sign. Generally it has been shown that the following variables affect the nature and magnitude of the stresses produced in grinding high strength steels:-

- i) Wheel speed.
- ii) Grinding wheel.
- iii) Depth per pass i.e. down feed.
- iv) Grinding fluid.
- v) Wheel dressing.

2.3.10.2. During grinding extreme heat can be generated at the work piece in contact with the grinding wheel. The heated region tries to expand but is restrained by the surrounding cold metal and thus becomes compressively stressed, resulting in plastic deformation. As the momentary contact between the grinding wheel and the work piece terminates, the previously heated region is suddenly cooled by heat dissipation to the surrounding material, and contraction occurs producing tensile stresses depending upon the grinding conditions applied. The main problem is, therefore, to avoid burning of the extreme surface. For example in steels, severe heating can be sufficient to form a skin of austenite which subsequently transforms to martensite due to the steep temperature gradients and rapid cooling rates involved. The untempered martensite formed will be compressively stressed but is inherently brittle and subject to cracking. Field and Kahles (175) have summarised the surface integrity of machined and ground high strength steels.

2.3.10.3. Following the service failure of a major component in BS.S28 steel, the author (176) investigated by the X-ray back reflection technique the macro stress distribution (surface and subsurface) in specimens showing

- (a) overtempering burns,
- (b) rehardening and overtempering burns, and
- (c) no apparent surface defects.

The stress distribution/depth curve for the defect free area, showed a compressive stress of 32 tonf/in^2 at the surface. The material was affected to a depth of about 0.0015 in with a moderate subsurface peak tensile stress. The overtempered ground region exhibited a surface tensile stress of 8 tonf/in^2 with a peak of 23 tonf/in^2 at approximately 0.001 in below the surface. The material was affected to a depth of 0.003 in . The severely burnt region of rehardening and overtempering showed a compressive

stress of 19 tonf/in² at the surface with a steep stress gradient giving a maximum tensile stress of 29 tonf/in² subsurface at a depth of 0.0015 in. The total depth of affected material was over 0.010 in. Several other investigators (177, 178, 179, 182) have investigated the origin and measurement of residual grinding stresses.

2.3.10.4. Grinding of titanium and its alloys can present certain problems. It has been shown (180) that titanium alloys can be ground with precautions and at lower than normal speeds, to give low residual stresses.

2.4. Measurement of Macro-Strain and Micro-Strain by X-Ray Diffraction.

2.4.1. Theoretical Background

2.4.1.1. Mechanical deformation produces lattice strains which may be considered to be due to a combination of:

(a) Macro- or long-range strains. A strain is produced which is uniform over relatively large distances. The interplanar spacings change from their stress-free values to new values corresponding to the stress and the appropriate elastic constants.

(b) Micro-strains. Plastic deformation occurs giving rise to non-uniform variations in the interplanar spacings. These are referred to as micro-strains, and can be considered as random displacements from the original lattice spacings.

2.4.1.2. The Bragg angle for the reflection of X-rays from a set of crystal planes is sensitive to any factors that influence the interplanar spacing of the reflecting planes. Since stresses within the elastic range can alter the interplanar spacing, d , enough to change the angle θ in the Bragg law by a measurable amount, the magnitude of the strains that alter the normal spacing of the planes can be deduced from the observed change in θ angles.

2.4.1.3. The X-ray determination of stress depends upon the measurement of the elastic strains in a chosen direction in the atomic lattice in the region concerned. These strains are then converted into equivalent macroscopic stresses by applying the theory derived from the standard elastic theory, making allowance when necessary for the elastic anisotropy in the material, (Greenough 1952 (10)). The lattice stresses thus determined are correlated with the stresses calculated from the theory of isotropic elasticity.

2.4.1.4. To measure lattice strains, a collimated X-ray beam of a suitable wave length is used. The high Bragg angle lines from a suitable set of planes (hkl) of the specimen are recorded by the back reflection film cameras, or with a diffractometer with proportional or scintillation counters. The basis of this method is that if a crystal is strained, the lattice plane spacing d changes by Δd so that in a Bragg reflection where:

$$n\lambda = 2d\sin\theta$$

where λ = X-ray wavelength, θ = Bragg angle, n = order of reflection, the strain is given by:-

$$\frac{\Delta d}{d} = -\cot\theta \Delta\theta$$

2.4.1.5. Where the strains are uniform, the change in the position of the reflected ring is determined by the change $\Delta \theta$ in θ . When the strains are present as micro-strains, each individual crystallite may give a different $\Delta \theta$, and the ring, instead of being sharply reflected will become broadened and diffused. This line-broadening is used to measure the micro-strains.

2.4.1.6. There is, however, one particular feature inherent in the X-ray technique, that is, the lattice strain deduced from the change in the position of the peak of the X-ray line represents a value in a given direction for only those grains in the polycrystalline aggregate which are so orientated as to contribute to the particular X-ray reflection examined. In this sense, the X-ray technique is inherently selective, and by the use of different wavelength radiations, the lattice strains for various families of planes in the lattice may be determined. Also, in the elastically anisotropic materials, the conversion of strains into the equivalent stresses implies that the residual stress system is essentially uniform in all the grains irradiated in a single phase metal or constant through one phase in a two phase material. It is therefore necessary to be cautious about drawing conclusions from these results as to the state of the specimen as a whole. This assumption is, however, not valid if the volume of the specimen irradiated by the X-ray beam has suffered appreciable prior plastic deformation. In the latter case, a complex system of heterogeneous internal stresses is generated by the process of plastic flow. In consequence, whilst the residual lattice strains may be measured without ambiguity, it is not possible to relate these directly to an equivalent stress system owing to uncertainty as to the causes of the strain induced by plastic flow. Although several possible explanations have been advanced over the last 20 to 30 years, no clearcut concept has yet emerged as to the predominant operative processes.

2.4.1.7. The X-ray diffraction technique is a non-destructive method for evaluating surface stresses in bodies of complicated shapes. During the last 30-40 years various papers on the subject have been published. In 1929 Aksenov (28) discussed the theory of X-ray measurement of stresses. In 1934/36 Barrett (29 - 30) gave a comprehensive review of theory and practice. A description of the practical application to the problem was by Thomas (6) who worked on steels and by Frommer and Lloyd (5) working on aluminium.

- 2.4.1.8. Sachs and Weerts (31) described a method for determining the sum of principal stresses in the plane of a surface by X-ray analysis. They used the single exposure technique; in this method, two photographs are taken, one in the unstressed condition and the other in the stressed condition. Norton and Rosenthal (32) and many others have described the details of the two-exposure technique. The diffracted ray forms a circular ring in the film whose diameter is a function of the film to specimen distance. This dimension is conveniently measured by coating the specimen with the powder of a pure metal of known diffraction angle, thereby obtaining a reference circle on the film.
- 2.4.1.9. Newton and Vacher (33) expressed the opinion that the two-exposure method is preferable to the single exposure method for the following reasons:-
- (i) the change in observed data between normal and inclined exposure is greater than the change to be detected in the single exposure comparison.
 - (ii) no assumption of equality in determining the stress factor between a stressed and unstressed specimen is necessary.
- 2.4.1.10. Recently Norton (34) has outlined the practical advantages of the single exposure technique over the two-exposure technique for many routine applications.
- 2.4.1.11. The two-exposure back reflection technique employed by various investigator suffered from the disadvantage that only two experimental points were used to determine the slope of the line. These points were subject to considerable scatter due to inherent material inhomogeneities, inaccurate alignment of the camera, etc. In order to obtain a better estimate of the parameters, Hawkes (7) developed a multi-exposure technique, with graphical solutions and applied it successfully to the stress analysis of aluminium alloys and martensitic steels. Later Moore (8) applied this technique to determine the surface stresses in austenitic steels.
- 2.4.1.12. The basis of the analytical procedure for stress measurement by the multi-exposure X-ray back reflection technique with graphical solutions is recapitulated in Appendix 2.
- 2.4.1.13. Although the back reflection technique has been used for many years, an interest was initiated by the discovery that with a diffractometer technique it is possible to measure stresses in high hardness steels.

The accuracy with which the shift in the Bragg angle θ can be measured from the X-ray photographs, and hence the degree of accuracy to which the stress can be determined, depends upon the intensity and sharpness of the diffraction line. However, severely cold worked, quenched and tempered steels give broad and diffused lines.

2.4.1.14. Recent developments in the measurement of residual stresses have mostly been in the use of diffractometers to determine the position of peak intensity in a broad diffraction line. In this technique, the diffraction pattern is detected by an electronic counting device in step-by-step samples (35) instead of from a film (19, 36). The approximate peak position is found from the counts, and then at three points straddling the peak, the X-ray intensities are found with high precision. The intensity values so recorded are corrected for the Lorentz and polarisation factors and for absorption effects (37), they are then fed into a formula to yield the axis position of a parabola that passes through the three given points (38). A reproducibility of the peak position to 0.02° or better has been reported using this technique (35). During the past 5-6 years, numerous papers have been published (27, 189-197) concerning the wider use of diffractometers for measuring residual stresses in different materials.

2.4.1.15. The methods hitherto discussed measured the surface stress system at a particular point only; recently Bainbridge (40) has described a new technique for measuring elastic strain in the interior of crystalline materials by means of X-ray diffraction. Working on single crystals of LiF, with Mo K α radiation, Bainbridge (40) has demonstrated that the new technique can permit complete evaluation of a tri-axial state of stress to a certain depth in the interior of the crystalline material.

2.4.2. Effect of Beam Penetration

- 2.4.2.1. In stress analysis by X-rays, choice of radiation and surface preparation are very important factors particularly where a steep gradient is involved. A method for determining the penetration X-radiation is given in Appendix 1.
- 2.4.2.2. The penetration of the X-ray beam beneath the metal surface is very shallow. In specimens that have steep stress gradients as produced by grinding and honing, X-rays provide only a measure of the average stress over the depth of penetration rather than the true value. The shallow penetration of X-rays requires that proper attention should be given to the preparation of the surface. Hauk (149) using an arbitrary surface penetration measured the stresses in a bent beam with Co K α and Cr K α radiations. His stress measurements with Cr K α radiation were about 35% lower than with the Co K α radiation. He interpreted his results in terms of the difference in penetrating power of the two radiations in that the Cr K α radiation has a longer wave length than that of Co K α radiation and hence the depth of penetration is about half that with Co K α radiation. The Fe K α radiation with penetrating power intermediate between Cr K α and Co K α radiations gave intermediate values of stress.
- 2.4.2.3. Glocker and Hasenmeier (53) examined plastically deformed mild steel specimens, while the deforming load was being applied and showed that the stresses determined using Co K α and Cr K α radiations were different. They attributed the difference to the penetrative power of the radiations and also concluded that surface layers had a lower stress than the interior. Weaver and Muller (15) have shown that machined surfaces may contain stresses that differ by 60,000 lbf/in² (26.8 tonf/in²) from the stresses in the underlying layers unless the disturbed layer is removed by chemical or electrolytic means. Voigt (16) observed that even gently abrading a hard steel surface with fine emery by hand induced a compressive stress of more than 50,000 lbf/in² (22.3 tonf/in²). Letner (63) has shown that grinding destroys the stress pattern present and imposes a new one characteristic of the grinding technique used. Koisten and Marburger (19) have shown that ordinary metallographic polishing induces stress. Ogilvie (17) found that it was possible to remove surface layers by acid etching while Lihl (18) reported that compressive stresses were induced during etching. The findings of Ogilvie were however confirmed by Garrod and Hawkes (121) in that stress measurement, by the multi-exposure X-ray back reflection technique, on an annealed steel specimen thinned progressively by electropolishing, gave no evidence of residual stresses.

2.4.2.4. Hawkes (7) has recommended that polishing aluminium alloy with 0000 emery + a 2-minutes mixed acid, or an electrolytic etch, or even simple mechanical preparation, has little effect on the X-ray stress measurement.

2.4.2.5. At the very surface of a specimen subjected to a multi-axial residual stresses, the normal stress component is zero. The assumption usually made is that the stress component perpendicular to the surface does not influence the X-ray stress determination because of the small depth of effective penetration of X-rays into the metallic specimens. That is indeed the case with many problems. In other cases, where large stress gradients exist near the surface of the specimens, the third stress component may have to be taken into consideration. Standard methods have been developed to evaluate this influence. (151 - 153).

2.4.2.6. The surface treatment of titanium prior to stress measurement was therefore considered important because titanium has a strong affinity for oxygen and a thin oxide film could affect the stress analysis. In view of the fact that the beam penetration into the metal surface is very shallow, choice of suitable radiation with an appropriate wave length is considered important in the stress analysis of titanium.

2.4.3. Depth of Stressed Layer

- 2.4.3.1. The possibility that macroscopic stresses in the surface layers of plastically deformed specimens balanced by opposite stresses in the interior were responsible for the observed residual lattice strains has been examined by many investigators. The changes in residual lattice strains at normal incidence to the X-ray beam as the specimens were etched away progressively have been observed.
- 2.4.3.2. Bollenrath, Hauk and Oswald (57) using the X-ray diffraction technique have shown that the residual lattice strains in a plastically extended mild steel decreased as the specimen became thinner. Smith and Wood (75) performed an analogous experiment and showed that the observed residual lattice strains remained approximately constant at surfaces exposed by etching. Greenough reported similar experiments on wires and these also showed an approximately constant strain in the surface exposed by etching. Davidenkov and Timofeeva (89) made mechanical strain measurements on a plastically deformed aluminium sheet as the surface layers were etched. Moller and Neerfield (12) performed a similar experiment on plastically extended mild steel bars. Both concluded that there was no appreciable surface layer containing macroscopic stresses differing from these in the interior.
- 2.4.3.3. Garrod and Hawkes (121) using X-ray diffraction techniques have shown that for steel the strain decreased as successive surface layers were removed and tended to approach constancy after a few thousandths of an inch had been removed from each face of the specimen. However, in iron the stresses were lower than those in steel, but were still significant for 310 reflection even after .012 in had been etched away. Similar results were obtained by Karashima, Kojima and Fujiwara (122) for electrolytic iron. Donochie and Norton (87) found that for Armco iron the strain did not alter with depth.
- 2.4.3.4. In conclusion it could be inferred that surface macroscopic stresses might play some part in producing residual lattice strains and apparently different mechanisms would seem to be operating for the commercially pure materials (armco iron, electrolytic iron) and the heterogeneous materials (steels).
- 2.4.3.5. When stressed surface layers are removed successively, the measured stresses below the surface must be corrected by an amount proportional to the relaxation created by the removed layers. This means that all the determinations except the initial value at the surface must be corrected in order to obtain the true stress existing before the layers were removed. These corrections have been determined from the theory of elasticity by Moore and Evans (123).

2.5. Measurement of Lattice Strain in Materials Loaded Elastically.

2.5.1. In the past much work has been done to establish the function connecting the surface lattice strain and the applied macroscopic stresses (10). Using X-ray measurement many authors (58-61, 64, 65) found a strictly linear relationship between both quantities up to the yield point of the investigated materials. Others (53) have shown deviation from Hooke's law prior to macroscopic yielding of the tensile specimen. Most X-ray diffraction work which has been performed on elastically strained aggregates, has been concerned with the measurement of the X-ray diffraction line peak as the stress applied to the aggregate has changed. The investigation of the lattice strains in elastically strained aggregates necessitates the examination of specimens under stress. Normally a case of simple uniaxial tension has been investigated, the tensile stress being applied either directly or by using a bent strip, but experiments using torsional stresses have also been reported. Practical interest lies in the application of X-ray measurements to locked up body stresses in fabricated metal components. Theoretical interest lies in the light they shed on the elastic behaviour of an aggregate of crystals, each of which is anisotropic.

2.5.2. Since the crystallites contributing to a given point on the Debye-Scherrer ring may have any orientation, it is evident that in a stressed aggregate the crystals will exhibit different amounts of strain. In addition, whereas the theoretical strain calculated for a crystal of a particular orientation applies to the average grain of that orientation surrounded by average neighbours, the strain in any individual crystal will be influenced by its particular neighbour, and differ somewhat from the theoretical value. For both these reasons, of which the first may be more important, it is expected that X-ray diffraction lines will broaden as the stress applied to an aggregate is increased. There may, of course, be additional causes of line broadening.

2.5.3. No experimental work has been carried out to correlate the possible line broadening with the theoretical treatment of the elastically strained aggregates. During the course of a quantitative investigation of the line broadening caused by stresses applied to the aggregates, Weaver and Pfarr (41) made some observations on elastically stressed specimens. They concluded that any line broadening which did occur was too small to be detected by their measurement. Smith and Wood (42), during their experiments on copper, made qualitative observations on the line broadening and calculated that it was small when their specimens were elastically deformed. Generally,

the evidence available indicates that there is probably a certain amount of line broadening in the elastically strained aggregates but that it is small. It does not seem likely that present experimental methods of determining line broadening will be able to give results of sufficient accuracy to be compared with any theoretical values. Stacking faults also cause peak shifts as well as broadening of peaks in materials of relatively low stacking fault energy.

- 2.5.4. Young's modulus and Poisson's ratio, which enter into the stress analysis, are the fundamental properties of the materials being examined. The bulk values of these constants are readily available for most polycrystalline materials, but the use of bulk values in the computation of stresses by X-ray measurement is open to criticism.
- 2.5.5. The elastic constants that are measured mechanically do not necessarily apply accurately to X-ray determination of stress. Each grain is anisotropic and the stress is measured along a certain crystallographic direction. Therefore the grains that reflect have only certain orientations with respect to the axes of the stress and the effective values of Young's modulus E and Poisson's ratio ν will differ from those of overall average orientations, as being measured in a mechanical test. The effective values of E and ν are also influenced by grain boundary interactions in a polycrystalline mass. Obviously the X-ray determination of E and ν is desirable and should preferably be determined for the particular condition under which these are to be used because these constants depend upon indices of the reflecting plane, X-ray wave length used, and probably on grain size and other micro-structural variables.
- 2.5.6. Greenough (10) has shown that it is possible to correlate E and ν with some values calculated from a knowledge of single crystal elastic constants, but the fundamental difficulty which must be overcome in the calculations is to allow for the interaction of one crystal on its neighbour during deformation. It is not possible for both the stress and strain to be continuous across the boundary of contact between two anisotropic crystals of different orientation and some assumptions have to be made in the calculations as to which components do maintain continuity.
- 2.5.7. So far, it appears that only the two simplest possible assumptions (43 - 44) have been applied to the case of lattice strains, although more complicated assumptions have been made by Bruggeman (45) and by Boas & Schmid (46) for the case of macroscopic relations. Voigt (43) performed the calculations for the case of mechanically measured strains making the

assumption that the strains in each grain of the aggregate were the same, whilst Reuss assumed that the stresses in each crystallite in the aggregate were the same and equal to that applied to the aggregate. In both cases it was assumed that the aggregate as a whole obeyed the usual elasticity laws for isotropic bodies. In the theoretical treatment of random aggregates three assumptions are made implicitly:-

- i) that crystals in the aggregate are small compared with the volume examined,
- ii) that crystals have a random orientation in the aggregate,
- iii) that the crystals in which strains are examined are constrained by their neighbours at all surfaces.

The theoretical treatment can be applied to a crystal of any symmetry. Neerfield (47) and Moller (48), however, have shown that the experimental value for the macroscopic elastic constants of the polycrystalline aggregates do not agree with the results calculated from either theory, but they do agree remarkably well with the average of the two theoretical values. Chung and Buessem (49) have shown that the Voigt-Reuss-Hill (VRH) approximation is a useful scheme by which anisotropic single crystal elastic constants can be converted into isotropic polycrystalline elastic moduli. Chung and Buessem have studied the VRH approximation for highly anisotropic cubic crystals and the crystals of lower symmetry, e.g. hexagonal, tetragonal and trigonal symmetries, etc.

2.5.8. Hanstock and Lloyd (50) have obtained good agreements between values obtained mechanically and by X-ray measurements on the 420 reflections from Duralumin. This is not surprising because aluminium is one of the least anisotropic of metals. Donachie and Norton (51) working on 420, 511 and 333 reflections from 2024 aluminium alloy have confirmed the findings of Hanstock and Lloyd. Gisen (52) observed that stresses in polycrystal iron computed from the 211 reflections, using Cr K α radiation, were more in agreement with those determined mechanically than when using the 314 reflections, from Co K α radiation, even though the 310 reflections occurred at a higher Bragg angle and gave more precise spacing values. This finding was substantiated by Glocker and Hasenmeier (53), and later by Thomas (54) who found the 310 reflections gave stress values up to 30% too high. Donachie and Norton (51) working on 211 and 310 reflections in Armco iron have confirmed the findings of these authors.

2.5.9. Maloof (55), Letner and Maloof (56) have stated that for annealed SAE 8742 and annealed tool steel, the X-ray gave value for E 30-40%

higher than the engineering value. Ogilvie (17) has mentioned that X-ray and engineering values for E & ν seem to agree when using $Co K\alpha$ radiation and 310 reflections of iron. It would therefore seem that stresses calculated using the 310 reflections are representative of a sample of annealed steel, whilst those calculated using 211 reflections are not representative. On the other hand Ogilvie, Blount and Elias, in their discussion of Christian and Rowland's (36) work, agree that the X-ray values of stress constants are 20-30% higher than the engineering values whilst Christian, Rowland and Beu (9) found that the X-ray and engineering values of the stress constants agree within experimental error. The problem due to anisotropic response is recognised but no solution is offered.

2.5.10. Titanium, unlike steel or aluminium, can exhibit considerable difference in Young's modulus and Poisson's ratio depending upon heat treatment, processing variables, alloying additions. Further, there is lack of basic information concerning the effect of preferred orientation upon the elastic properties. The inherent anisotropy of the single crystal properties and the preferred orientation developed in the final wrought product are the two complementary factors. It is possible to derive theoretical X-ray elastic moduli from a knowledge of the anisotropic behaviour of single crystals but in general the agreement between these and the experimental values is not good (12).

2.5.11. The elastic constants have been determined for single crystals by Flowers et al (205) and Fishers and Ranken (206). The compliances measured by Bradfield have been reported by Schoening and Witt (207). Using the compliances determined by Flowers et al and Hooke's law, Zarkades and Larson (208) have shown that when the stress is applied parallel to the basal plane, the Young's modulus = 14.5×10^6 lbf/in² when the stress is applied perpendicular to the basal plane the Young's modulus = 21.0×10^6 lbf/in². Further, it has been shown that Young's modulus varies with other testing orientations. Similarly large variations in Poisson's ratio values in single crystals are reported. Zarkades and Larson have concluded that considerable improvement in elastic moduli may be achieved through texture control resulting in the production of improved material for critical applications.

2.5.12. The basic stress equation (Appendix 2) has been rewritten in different form to show how the elastic moduli can be determined experimentally by X-radiation in titanium without taking into account the elastic constants (Page 123).

2.6. Measurements of Lattice Strains in Materials Loaded Plastically.

2.6.1. Introduction

2.6.1.1. Plastic deformation is caused by a glide in the crystal.

Part of the crystal slides as a whole relative to the remainder on a specific crystallographic plane. The distance it moves is an integral multiple of the inter-atomic spacing in the direction of the glide. Since in the final position each atom has been replaced by another (except for a few at the sides of the crystal) and all atoms are identical, it would be impossible to detect this deformation by X-ray diffraction methods if it were not accompanied by other phenomena. Glide actually takes place by dislocations travelling through the crystals, and if these remain in the crystal, as many of them probably do, they cause local regions of lattice distortion and hence a broadening of the diffraction line. It is possible that the presence of many regions of disorder in the lattice will also cause a general average expansion of the lattice. Such an effect would also produce a residual lattice strain but one which is of approximately the same value in all directions.

2.6.1.2. Weaver and Pfarr (41) in their studies on grain size and re-crystallisation of rolled steel sheet by X-rays observed that, when a test piece from the sheet was held in tension, a marked contraction of the lattice occurred, but if this sample was stretched beyond the yield point and then unloaded the lattice spacing increased to a much higher figure than the original stress free value. These observations have been confirmed by Bollenrath, Hauk and Oswald (57). Later Smith and Wood (58-60) carried out work of a similar character and published the stress strain curves for aluminium, copper, iron and mild steel. Similar curves have been described for steel by Greenough (61). Hauk (62) has published details for aluminium and aluminium-copper magnesium alloy although not in the form of lattice stress strain curves. All these authors agree that lattice strain ceases to be proportional to the applied stress just above the macroscopic yield stress of the metal. However, Glocker and Hasenmeier (53) observed for the 211 reflection for mild steel that the lattice strain ceases to be proportional to the applied stress for the stress above about three quarters of the macroscopic yield stress. This is directly contrary to the findings of Wood (64). Finch (65) observed that the lattice strain for 310, 211, 220 reflections for mild steel were always proportional to the applied stress up to the yield stress and that the residual lattice strains never developed until macroscopic plastic deformation had occurred. The majority of the experimental evidence is in favour of the view that a non-proportional region for

lattice strain does commence just above the macroscopic limit of proportionality.

2.6.1.3. While it is established that residual lattice strains are left in the individual grains at normal temperatures, considerable speculation exists as to the origin and distribution of the strains. Numerous attempts have been made to explain the origin of residual lattice strains after uniaxial deformation of poly-crystals. Two main trains of thought have been pursued to account for the experimental facts; one leads to residual macro-stresses and the other to residual micro-stresses. Both are based on the common assumption that under tensile loading soft and hard regions occur and that after removing the external load the hard regions remain under tensile stress giving rise to compression of the soft regions. The following suggestions have been proposed by various authors:-

- i) The surface crystallites of a polycrystalline material should have a lower yield stress than the crystallites in the interior (surface effect) (53, 56, 57).
- ii) After passing the yield point, an inhomogeneity of the work hardened state arises between a small surface layer and the interior of the polycrystals, increasing with increasing amount of deformation (hardening effect) (67, 68).
- iii) An intergranular stress system is established in plastically deformed aggregates due to yield point and work hardening anisotropy. The selective nature of the X-ray method reveals the mean lattice strain of a specially orientated group of crystallites only (orientation effect) (10, 69, 70, 71, 72).
- iv) In the case of two phase alloys, intergranular stresses develop due to difference in yield strength of the two phases. After plastic deformation each phase takes residual stresses of opposite sign (interphase effect) (73, 74).
- v) A system of orientated micro-stresses, localised within regions of coherent scattering, appear after uniaxial plastic deformation. According to this view, all centres of coherent scattering are soft regions. The hard regions, in homogeneous materials, are represented by grain boundaries, sub-boundaries, or highly distorted regions, and in heterogeneous materials by a sufficient quantity of hard phase (coherent area effect) (75, 76, 77).
- vi) In plastically deformed polycrystals, containing more than one phase, differences in Young's modulus could cause residual stresses. Furthermore, due to inhomogeneity of strain near the phase boundaries and inhomogeneity of work hardening near the interphase boundary areas, residual stresses arise (heterogeneity effect) (78, 79).

2.6.1.4. Summarising, in the case of homogeneous materials the lattice strains are probably due to the hardening effect and the surface effect and, accordingly, measured stresses would be interpreted as macro-stresses. However, for the case of heterogeneous materials, hardening effect, surface effect, heterogeneity effect, together with coherent area effect, cause the residual lattice strain distributions. Which of these factors contributes most to the measured lattice strain depends upon the type of material and on the amount of heterogeneity. Nevertheless the super-imposition of micro- and macro-stresses occurs most frequently.

Three main types of experiment have been applied:-

- i) Surface residual strains have been determined as a function of the plastic deformation of different materials
- ii) Residual strains have been determined using different X-ray wave lengths
- iii) Changes in residual strain distribution have been observed after progressive thinning of plastically deformed specimens.

2.6.1.5. Greenough (70), by using different radiations, showed that f.c.c. metals exhibited residual lattice strain due to intergranular micro-stresses. He confirmed that, with respect to the magnitude and sign, these residual lattice strains depended upon the orientation of the measured grains of the polycrystalline samples. Greenough put forward his hypothesis on the concept originally introduced by Heyn (80) to explain creep recovery. Proceeding from this hypothesis of Heyn (80) and Masing (81) and using Taylor's (82) theory for polycrystals, Greenough explained his results qualitatively and quantitatively as due to the yield point anisotropy of the crystallites of the polycrystals. Measurements of Kappler and Reimer (69) and Reimer (74) of polycrystalline nickel by X-ray diffraction and magnetic methods agreed well with Greenough's theory. Furthermore, Reimer published measurements on pure copper which were also in agreement with the extended theoretical treatment of Greenough. Bateman (84), however, was able to show that in the case of aluminium alloys considerable deviation from the theoretically predicted residual lattice strains existed after plastic deformation.

2.6.1.6. A systematic study of the formation of residual lattice strains after different amounts of plastic deformation of polycrystalline aluminium and copper by Macherauch and co-workers (68, 85, 86) led them to the conclusion that the hypothesis of Heyn (80) and Masing (81), and consequently

the theory of Greenough, was principally not valid with respect to these f.c.c. metals. By X-ray measurement, these authors found the existence of a mainly macroscopic residual stress state due to inhomogeneity in work hardening over the cross section of the investigated f.c.c. polycrystals. Moreover they showed that the greater part of the experimental data of the other authors on these metals was consistent with this view. Greenough (66) himself carrying out residual strain measurements on plastically deformed iron and steel interpreted his results on these materials as affected by a superimposition of residual macro- and micro-stresses due to the yield point anisotropy and due to a yield point difference between the surface parts and the interior parts of the specimen.

2.6.1.7. Donachie and Norton (87) have considered and rejected a similar hypothesis. Wood and Dewsnap (76) did not think that Heyn intergranular stresses are likely to make a significant contribution to observed lattice strains because Heyn stress in any case is dependent on its neighbour. They state that since on the whole all arrangements of neighbours are possible, the various numbers of the group taken together will exhibit the whole range of stresses from tension to compression with a zero mean stress. Greenough (88) agreed that residual lattice strains in crystals of one orientation would vary as the orientation of the neighbouring crystals varied, but pointed out that the average strain taken over many grains of the particular orientation would not be zero, but would depend on the difference of the yield tension of the grain with the given orientation and the average yield tension of the aggregate.

2.6.1.8. Mechanical methods of residual stress determination have been applied to a metal bar subjected to uniaxial plastic deformation and indicate that there are no residual macro-stresses after unloading (87, 89). On the other hand, the X-ray measurements (35, 75, 90) show the line shift indicative of macro-stresses which are opposite in sign to the stress causing the initial yield. Since the stresses are constant through the cross section they can only be micro-stresses which appear to the applied X-ray technique as macro-stresses. The X-ray observations may be tentatively accounted for by assuming a distribution of micro-stresses such that sub-grain boundaries are in tension and sub-grain interiors are in compression. Because of this anomaly Hyler and Jackson (91) maintain that the X-ray method is unreliable when used to determine residual stresses produced by plastic flow. Cullity (92) argues that X-ray measurements of macro-stresses due to grinding (19) and shot peening (93) (both of which cause severe plastic deformation) show excellent

agreement with mechanical determinations. Tiara and Yoshioka (209) have observed an anomaly in stress measurement by the X-ray diffraction and mechanical methods in 0.39% carbon steel after $3\frac{1}{2}\%$ plastic extension. These findings have been confirmed by Ricklefs and Evans (190) who have also observed that differences in stress level increased with an increase in the hardness level of the steel. They argue that the anomalous stress evidently required unidirectional plastic flow and further conclude that it is real and represents a situation of local balance in micro regions.

2.6.1.9. From the literature survey it can be concluded that the existence of residual lattice strains in metals deformed by unidirectional plastic flow is a real phenomenon but no clearcut explanation has yet emerged to account for their origin. In this investigation, the presence of micro-strains and macro-strains resulting from various working processes, including a specimen pulled in uniaxial tension, are discussed in terms of deformation characteristics of titanium.

2.6.2. X-ray Line Broadening Studies of Plastically Deformed Metals

2.6.2.1. Since Van Arkel (94) first reported that cold work produced a broadening of the X-ray diffraction peaks, both qualitative and quantitative studies of the effect of cold work on X-ray diffraction lines have been pursued. Experimental evidence has been obtained from both filings and solid metals. The majority of the experimental evidence is in favour of the view that broadening of diffraction lines is usually due to both the crystallite size and micro-strain but that the micro-strain usually give rise to a major part of the broadening. Metal studies have included copper (95), nickel (96), brass (97), aluminium (98), iron (99), steel (100) (all with cubic structures), cobalt (101), zirconium (102) and magnesium (103) (hexagonal close packed structures). Wood and Rachinger (104) showed that filings give much more line broadening than tensile specimens pulled to fracture. Probably in filings the residual stresses due to working may have any orientation and magnitude, thus giving rise to a large strain broadening. An alternative explanation is that working by rolling or by wire drawing affects the extreme surface layers more than the interior and these extreme surface layers contribute to the major portion of the diffracted X-ray intensity. The present position regarding the interpretation of line broadening observations may be summarised as follows:-

- i) In all worked metals variations of strain from point to point of the irradiated area causes a broadening. In the case of a metal worked by heterogeneously directed stresses as in filings or in ground surfaces, strain broadening predominates.
- ii) In all cold worked metals some fragmentation occurs giving crystallites of a size small enough to cause line broadening.
- iii) Stacking faults could be introduced producing local changes in structure.
- iv) Instrumental effect associated with the method of recording line profile must be taken into consideration.
- v) To examine the line shapes and to interpret them in terms of dislocation or some other fundamental theory of plastic deformation Hall (105) has interpreted his observations on aluminium and tungsten filings in terms of the dislocation concept.

2.6.3. Measurement of Peak Broadening

2.6.3.1. The methods of X-ray line broadening analysis can be divided into three main groups depending upon the quantity that is used to describe the line broadening:-

- i) Integral Breadth Method (106). In the earlier work the half peak breadth (the width at half maximum height) was extensively employed, but this is a very arbitrary quantity and its use is only justified if peaks of all the samples investigated remain similar. These days it is rarely used. A better method for determining the diffraction peak is the integral breadth (β) which is defined by Van Laue (107) as the area under the peak divided by the peak height.
- ii) The Variance Method. Wilson (108) and his co-workers have used the variance $W(S)$ of the line profile, i.e. the second moment of area above the centroid. The separation of the crystallite size and strain is considerably simpler since the variance is the sum of the crystallite size variance and the strain variance.
- iii) Warren-Averbach Fourier Method. Warren and Averbach (109) have worked out an interpretation employing the precise shape of a line, not just the integral width. They start with an analysis of the line shapes by Stokes' (150) method to obtain the true broadening, uninfluenced by the instrumental broadening and the $K\alpha$ doublet broadening. A difficulty that enters into the determination of the coefficients (A_n) in this analysis and in related ones, is the problem of determining the true intensity of the background between the diffraction lines, since the tails of the line spread far out and tend to overlap and obscure the true background intensity. Williamson and Smallman regard the uncertainties as a serious barrier to the determination of r.m.s. strains from observed profiles and prefer to make assumptions regarding the line shape that provide upper and lower limits for the particle size and r.m.s. strain.

2.6.3.2. Since stacking faults interrupt the coherent pattern of a diffracting crystal, these produce broadening that is similar to particle size broadening in that it is independent of the order of reflections for the particular reflections that are affected; but fault broadening differs from particle size broadening in that some reflections remain unaltered by faults and others broaden asymmetrically. Since stacking faults are to be expected in many metals and alloys, Warren and Warekois (110) developed a method for evaluating these.

- 2.6.3.3. In a further refinement of the method Wagner (97) developed an analysis to differentiate between the effects of deformation faults from those of twin faults. Small and Westmacott (111) have shown that an increasing faulting probability occurs with increasing solute content in several binary copper based alloys.
- 2.6.3.4. A Fourier analysis of strain, particle size and two kinds of stacking faults, has been worked out for hexagonal crystals by Housker and Averbach (112). These causes of broadening are difficult to separate when they are all present, but it is found that hexagonal cobalt could be produced by transformation and annealing so that only stacking fault broadening was present and an analysis could be carried out, provided certain assumptions were made.
- 2.6.3.5. Fourier analysis for body centred cubic structures cannot be as complete as for face centred cubic structures because all lines are made up of components that are shifted opposite ways by deformation faults on (211) planes, so there is no net displacement of line by deformation faults. The small magnitude of the line shapes expected from twin faults may preclude a reliable measurement of these, but r.m.s. strains and effective particle size can be determined from the broadening (113).
- 2.6.3.6. The Warren-Averbach Fourier method (109) requires multiple order reflections for the separation of micro-strain and crystallite size terms. With Cu K α or Co K α radiation, no multiple order reflections could be obtained; hence this method of line analysis was not used.
- 2.6.3.7. The variance method has the advantage of not requiring multiple orders of reflection to separate the micro-strain and crystallite size terms, and of enabling the correct background to be determined analytically. To be accurate and reliable, however, it is necessary to have intense peaks associated with a low background intensity. Many of the peaks obtained with alpha titanium were broad and had a high background intensity.
- 2.6.3.8. Often the profiles have long tails and the peaks overlap. In this case, the Fourier and Variance methods become unreliable since they are very dependent upon the tails. It has recently been shown (156) that these methods of X-ray profile analysis give comparable results with regard to micro-strain and crystallite size. Therefore, taking into consideration the intensity, overlapping reflections and absence of multiple order reflections, it was decided to use the integral breadth method to study titanium in this work. This method is simple to use and has the added advantage of not requiring the use of elaborate computational facilities.

2.6.4. Effect of Lattice Deformation and Lattice Distortion on X-ray Line Profile

2.6.4.1. Various attempts have been made to draw conclusions about the strain and/or dislocations in imperfect and in cold worked crystals from the measurements of integrated intensities of reflections. The fundamental difficulty is that of measuring the tails of the lines, especially when these extend out so far that they overlap with the tails of the neighbouring lines; differentiation between primary and secondary extinction and finally the true changes in integrated intensities in cold work may be only a few percent for most reflections.

2.6.4.2. Smith and Wood (58) noted a marked drop in the intensity of the 310 reflections in addition to the spacing changes occurring at the yield point of mild steel. Smith and Wood showed that the drop in X-ray intensity on passing the yield point is of the order of 50%. Similar results have been reported by Hengstenberg and Mark (114) for rolled Mo, Ta, W and by Brindly (115) and Ridley (116) for filings of Cu, Ni and Rh. Since the intensities decreased with increasing $\sin \theta$ it was interpreted that the effect of cold working the metal was to leave the atoms in a state of frozen heat motion, i.e. at small permanent random displacement from the mean position. Williamson and Hall (117) using a curved-crystal focussing monochromator of the Guinier type has revealed that the diffraction lines from cold worked metals have long "tails" which extend far out into the background. These tails are not of a great intensity at any particular θ value, but since these tails extend over many degrees, the integrated intensity represented by them may be quite large. Neglect of such tails could therefore introduce a considerable error into the intensity determinations made by the earlier workers. The error would increase with increasing θ values. The findings of Williamson and Hall were substantiated by Warren and Averbach (118) using filings of alpha brass and a Geiger counter diffractometer to measure the intensities of the diffraction lines and any changes occurring in background scatter. This investigation revealed that cold work actually increased the integrated line intensities owing to the marked reduction in extinction. Wagner and Kochendorfer (119) using a Geiger counter measured the intensity for the whole range of θ for single crystal specimens of zinc, and after various plastic extensions up to 32% and for polycrystalline specimens of aluminium and silver up to reductions in thickness of up to 99% by rolling, concluded that the level of the background intensity rose slowly with increasing θ value, whilst Warren and Averbach (118) found that it was approximately constant. By taking intensity measurements from a specimen

at 350°C, they showed that the changes produced by cold work differed from those produced by increased thermal vibration. They observed that cold work caused the broad lines to have very long tails and produced no detectable change in the background intensity level, whilst thermal vibration caused rather less line broadening, very much shorter tails, but a marked increase in the background intensity. Hall (117) also employed the Geiger counter technique and confirmed the findings of Wagner and Kochendorfer. He found that the intensity of background scatter from filings of both the pure and commercial aluminium increased at all θ values by about 10%. He also found that the total integrated intensity of the pattern was the same both for the specimen of filings and for the annealed material. Hall also observed that the secondary extinction was of importance in his specimen of annealed aluminium, whereas normally the primary extinction is considered to be the important factor.

2.6.4.3. Huany (216) has shown that the random static displacement of atoms in a crystal influence the diffraction of X-rays, in that Laue-Bragg intensity is reduced by an exponential factor, and further the reflections are shifted indicating expansion or contraction of the average lattice. Schoening and Witt (207) whilst studying the lattice distortion ^{by} of oxygen in titanium have shown that it is possible to predict the intensity reduction due to lattice strains. They measured the intensities of (hoo) and (ool) reflections with a single crystal diffractometer and observed that variation of the intensities with oxygen concentration could be attributed to:

- (a) additional scattering from oxygen atoms
- (b) change in Debye-Waller factor and
- (c) an exponential factor originating from distortion around the oxygen atoms.

They concluded that it was possible to estimate the defect concentration from X-ray measurements of lattice expansion and intensity reduction.

2.6.5. Effect of Stacking faults

2.6.5.1. Relatively few studies regarding the effect of stacking faults have been made in cold worked hexagonal close packed structures. Measurements by Edwards and Lipson (124) on hexagonal cobalt showed a broadening of certain lines. The original interpretation of these results by Wilson (125) was partially successful because only growth faults were considered. New measurements on hexagonal cobalt have been made by Anantharaman and Christian (126) and interpreted in terms of both deformation and growth faults. It has been shown (124 - 125) that stress free close packed metal, e.g. cobalt, possesses random stacking faults parallel to planes of closest packing and as a result certain reflections could become broadened as if the crystals were broken down into independently scattering crystallites. Barrett (127 - 128) has shown that stacking faults can be introduced into a crystal during plastic deformation in addition to dislocations and may significantly add to the line broadening which results from plastic deformation and sub-grain formation.

2.6.5.2. Recent experiments have indicated that stacking faults can introduce errors into stress measurements for materials of relatively low stacking fault energy. Stacking faults of deformation type, intrinsic faults, which result from a displacement at one atomic layer cause peak shifts and symmetrical broadening of peaks (97); whereas those of double deformation type, extrinsic faults, cause peak shifts and asymmetrical broadening of peaks (129 - 130). Stacking fault probabilities have been determined in silicon bronze (Cu - 6.6 atomic % Si - 1.2 atomic % Mn) and it was found (131 - 132) that stacking fault probabilities increased with increasing applied stress. The resulting shifts in peaks yielded computed stresses that were 25% or more too low unless the shifts resulting from stacking faults were removed (133). Errors in stresses computed from X-ray data were negligible, however, in similar experiments on brass (Cu - 30% Zn) which has a higher stacking fault energy.

2.6.5.3. Theoretical analysis (154) indicates that the presence of stacking faults in close packed hexagonal metals does not lead to line shift, but only to a modified line broadening pattern. However, Churchman (134) while studying the twinning mechanism in deformed grains of α -titanium by bending has observed that in titanium the stacking fault energy is low.

- 2.6.5.4. Partridge (155) using the transmission electron microscope technique observed stacking faults within a $\{10\bar{1}2\}$ type twin in titanium in the basal and non basal planes after tensile deformation. The origin of the stacking faults is not clear but these may account for the faulting detected by Lele and Anantharaman (210) in titanium, using X-rays. Basal plane faults have been observed within transformation twins in quenched pure titanium (211, 212). The stacking fault energy (basal plane) has been estimated to be ~ 300 ergs/cm² (213, 214). Lele and Anantharaman suggested that greater fault broadening of the $\{10\bar{1}2\}$ profile as compared to $\{1011\}$ profile indicated the absence of growth stacking faults in deformed titanium. Their findings were in marked disagreement with the earlier findings of Spreadborough and Christian (215) who obtained a much higher value of deformation stacking fault density. The latter performed their analysis on the 1124 and 1231 reflections. The difference is attributed firstly to the high Bragg angle reflections used by Spreadborough and Christian and secondly the probability of stacking faults on the prismatic or pyramidal planes besides basal planes in the case of close packed hexagonal metals with less than ideal axial ratio.
- 2.6.5.5. Warren (218) has discussed the work of Wilson (125) on growth faults and Christian (219) on deformation faults both on the 002 planes in H.C.P. structure. He has concluded that there was no peak displacement or peak asymmetry produced by these faults, whilst reflections with $H-K = 3N$ remain sharp compared to those with $H-K = 3N - 1$ which become broadened. Growth and deformation faults have different effects on the peak intensity depending on L being even or odd.
- 2.6.5.6. Hirsch (225) has studied the arrangement and movement of dislocations in a number of metals with variation in stacking fault energy. In face-centred cubic metals the type of arrangement of dislocations is related to the ability of the screw-dislocations to cross-slip, and this is determined by the stacking fault energy. In metals of low stacking fault energy, such as stainless steel and α -brass, the dislocations are piled-up against grain boundaries for very low deformations, but for larger strains, networks are formed on the slip planes by interaction with dislocations on other systems. No thermally activated cross-slip is observed in these metals. In Cu, Au and Ni, for which stacking fault energy has an intermediate value, the dislocations are arranged in poorly developed substructures

after deformation at room temperature. In Al, which has a high stacking fault energy, the dislocations are arranged in almost perfect sub-boundaries. Cross-slip has been observed in Cu, Au, Ni and Al.

2.7. Measurements of Stresses in Two Phase Alloys.

2.7.1. Residual lattice strains may occur in two phase alloys more readily than in single phase alloys after uniaxial plastic deformation because of the likelihood of the presence of different elastic constants as well as a variation in the direction and magnitude of the glide. The variation in the coefficient of thermal expansion with direction in each grain can give rise to residual lattice strains in cooling from high temperature and these stresses can be even greater in a two phase system with different coefficients for each phase.

2.7.2. Smith and Wood (75) have observed these stresses when taking measurements on the 310 reflections of mild steel using Co K α radiation. They have been further investigated, using the X-ray diffraction technique, by Wilson (135) on 1.26% carbon steel measuring separately the ferrite and cementite constituents after 90% compression; by Reimer (136) on iron and steel, and by Hawk (137) on steel and copper aluminium alloys.

2.7.3. Boas and Honeycombe (138) showed that electrolytically polished specimens of tin, cadmium and zinc (all non-cubic) when thermally cycled between 30° and 50°C were subjected to plastic deformation after only a small number of cycles. In cadmium and zinc the deformation was possible as slip, but in tin, distortion in the region of the grain boundary was possible. Lead (cubic) gave no sign of deformation for the same treatment. Later the work was extended to include a two phase alloy, tin-rich tin-antimony alloy. Gurland (39) published his results on tungsten carbide; Newton and Vaucher (139) obtained the value of stresses in single phase alpha brass, two phase alpha/beta brass and 1.05% carbon steel. Christenson and Rowland (36) have published the details of stress measurements in both the austenitic and martensitic phases of case hardened steel. French and MacDonald (189) have published the details of grinding stresses in WC in a WC + 10% Co composite.

2.7.4. Phase transformations will induce further residual lattice strains owing to the changes in volume that occur. Martensitic transformations, precipitation reactions, reaction of gases which on precipitation from solutions become concentrated at various types of lattice defects, are all typical examples of lattice strains.

3. EXPERIMENTAL PROCEDURE

3.1. Materials.

3.1.1. The following two grades of titanium were used in this investigation.

(a) IMI.130

(b) IMI.318A

Both materials were in the form of cross rolled sheet, or obtained from wrought bar.

(a) By precise control of the interstitial elements, carbon, oxygen and nitrogen, it is possible to produce commercially pure titanium to specific ultimate tensile strength requirements within the overall range 25/45 tonf/in². Three grades are produced, i.e. soft, medium and hard. The soft grade is utilised where exceptional cold formability is required and the hard grade for higher strength requirements. The medium grade is widely used for general construction work in both the aircraft and chemical fields.

IMI.130 is medium grade commercially pure titanium with the following typical mechanical properties:-

0.1% P.S. tonf/in ²	T.S. tonf/in ²	Elongation % on 2 in	Fatigue limit % of T.S.	Bend Radius 180° bend 15 S.W.G. or thinner	Density g/cm ³
22 min.	30-40	20 min.	50	2t	4.51

The micro-structure of this material is shown in Photo 1 and the mechanical properties obtained from test blanks cut both in the longitudinal and transverse directions are recorded in Table 1.

(b) IMI.318A is an alpha-beta alloy. At room temperature it has a duplex crystal structure consisting of alpha (close packed hexagonal) and beta (body centred cubic). At higher temperatures this progressively transforms to an all beta structure; the transformation being complete at 930° ± 20°C. Depending on the heat treatment, the following properties are obtained from IMI.318A:-

0.1% P.S. tonf/in ²	T.S. tonf/in ²	Elongation % on 2 in	Fatigue limit % of T.S.	Bend Radius 180° bend 10 S.W.G. or thinner	Density g/cm ³
57 min.	62 min.	8 min.	55-60	5t max.	4.42

The structure of 6% Al, 4% V Ti alloy quenched from 840°C (in the beta field) consists of a retained beta matrix with islands of primary alpha. Upon ageing at 480°C the alloy becomes stronger, presumably by the beta - omega - alpha hardening reaction. However, the same alloy when quenched from 954 - 980°C is apparently largely alpha prime with some residual alpha in the transformed beta matrix. Upon ageing at 480°C the alloy develops higher strength. The fact that little or no beta is retained during 980°C quench suggests that the reaction: alpha prime → alpha + beta may be responsible for the hardening.

IMI.318A material is annealed between 800° and 900°C.

The chemical composition of IMI.318A as supplied by the manufacturer was as follows:-

Batch	Chemical composition %					
	Al	V	C	Fe	N ppm	O ₂ ppm
A 6309	6.08	4.08	0.02	0.06	105	1100

3.2. Treatment of Specimen.

3.2.1. For determining the macro-strains, flat strips (8.0 in x 0.72 in) were cut in the transverse direction from the sheet materials in IMI.130 and IMI.318A and all faces were polished with emery papers. For determining the bulk Young's modulus values that enter into the stress factor calculations, suitable blank lengths were cut in the longitudinal and transverse directions for the two materials.

3.2.2. For determining the micro-strains, commercially pure titanium specimens were treated using the following processes:-

3.2.3. Grinding

A test blank (3.0 in x 0.75 in x 0.50 in) was ground. In the preliminary stages 0.015 in material was removed by grinding. For test grinding, the blank was sectioned into five test coupons, 0.006 in material was removed, with various down feed rates, whilst keeping the following factors constant:-

- (a) wheel grade
- (b) wheel speed
- (c) grinding fluid

Further details are given in Appendix 6 (Section 1).

The ground specimens were examined for surface abnormalities using the stereoscan electron microscope and for residual macro-strains and micro-strains by X-rays.

3.2.4. Shot Peening

The test blanks (3.0 in x 0.75 in x 0.50 in) were shot peened to three peening intensities 0.008A2, 0.014A2 and 0.020A2. Almen strips were also shot peened to peening intensity 0.014A2. The other details are given in Appendix 6 (Section 4).

3.2.5. Milling and Turning

Two test coupon (0.75 in x 0.75 in x 0.50 in) were milled and turned using the variables listed in Appendix 6 (Section 2 and 3).

3.2.6. Vapour Blasting

A sheet specimen (1.50 in x 1.50 in x 0.036 in) was vapour blasted using the conditions given in Appendix 6 (Section 6).

3.2.7. Uniaxial Pulling

A sheet specimen (6.0 in x 1.62 in x 0.036 in) was pulled in uniaxial tension using the conditions given in Appendix 6 (Section 7). The magnitude of micro-strains was determined in the region of maximum plastic tensile strain.

3.3. Heat Treatment.

The strips and blanks were stress relieved as follows:-

Commercially pure titanium IMI.130 540°C $\pm$ 5°C, $\frac{1}{2}$ hr, air cool

Titanium alloy IMI.318A 600°C $\pm$ 5°C, 1 hr, air cool

In order to preclude the ingress of air and so prevent surface contamination, the strips and blanks were clamped in a specially designed jig. The ends of the jig were sealed with fireclay cement and dried before charging in the furnace.

3.4. Surface Preparation.

The strips for use in X-ray stress analysis were chemically milled as follows:-

- (i) Degrease with acetone
- (ii) Wash with distilled water
- (iii) Pickle with the following solution
 - (a) HNO_3 36°B 250 ml + 67 ml
- 0 ml
 - (b) HF 40% 40 ml + 3 ml
 - (c) H_2O distilled, to 1 litre
Temperature of the bath 50-60°C

- (iv) Wash and dry
- (v) Pickle in the above solution for 30 seconds at room temperature
- (vi) Wash and dry

3.5. Manufacture of Specimens.

The laboratory design S₁ and S₂ tensile test pieces (Fig.1) were cut from the stress relieved blanks in commercially pure titanium IMI.130 and 6% Al-4% V Ti alloy IMI.318A respectively.

Shot peening control strips to Almen design were manufactured from the IMI.130.

3.6. Testing of Tensile Test Pieces.

The tests were carried out on a Denison testing machine in axially aligned grips. Load/extension curves were plotted from which 0.1%, 0.2%, 0.5% proof stress values and Young's modulus were obtained.

3.7. Micro Hardness Testing.

Subsurface micro-hardness tests were made with ^aG.K.N. Micro-hardness tester on metallographically prepared specimens, cut from both the Almen strip and the test blanks in IMI.130 and blanks only in IMI.318A.

3.8. Stereoscan Electron Microscopy.

In order to detect any surface abnormalities due to grinding of commercially pure titanium IMI.130, the test coupons ground with different down feed rates were examined using the Cambridge Stereoscanning Electron Microscope.

3.9. Transmission Electron Microscopy.

3.9.1. Transmission electron microscopy was carried out using an E.M.I. EM6G microscope to study the deformation characteristics of commercially pure titanium IMI.130. The following deformation processes were studied:-

(a) Uniaxially Pulled Specimen

Initially tensometer test pieces (Fig.1) manufactured from the sheet material were polished on one face using a gamma alumina pad impregnated with 10% oxalic acid. The specimens were then uniaxially deformed giving 4.5%, 10% and 15% strain in a Hounsfield tensometer. An unstrained test piece was also examined.

(b) Shot peened, ground and milled surfaces

The shot peened specimens were taken from the blank peened to 0.014A2 intensity whilst the ground test coupon G3 with down feed rate of .0005 in per pass was studied. The details of the working process are included in Appendix 6.

3.9.2. Preparation of Thin Foils

On chemical milling the commercially pure titanium, preferential attack occurs along the grain boundaries and the twin boundaries with possible hydrogen pick up. Further, the titanium has a strong affinity for oxygen and hence tends to form an oxide film. In order to avoid these problems and to produce a thin foil, the following procedure as recommended by Partridge (155) was used:-

Perchloric acid	30 ml
Methanol	295 ml
n-butanol	175 ml
Angle of inclination of jet to the specimen surface	30°
Cathode	Steel
Temperature of bath	-20°C
Current density	0.2 to 0.75 A/cm ²

3.9.2.1. The uniaxially pulled tensometer specimens were lacquered except for the area to be thinned. The electrolyte was agitated and jets of electrolyte impinged at each face of the specimen until perforation of the sheet occurred.

3.9.2.1. For studying the surface behaviour of shot peened, ground and milled surfaces, the specimens were sectioned and mechanically polished to a thickness of ~ 0.018 in. The specimens were lacquered as before but the electrolyte was allowed to impinge only on the mechanically polished face and electropolishing was continued from one side until a thin foil was obtained.

3.10. Optical Microscopy

The optical microscopy was carried out using a Vickers projection microscope. The grain size and directionality was examined in specimens sectioned in the longitudinal direction from each of the materials. The specimens were prepared by polishing on a gamma alumina pad impregnated with 10% oxalic acid.

To study the basic mechanism of plastic deformation in titanium in unidirectional deformation such as in uniaxial tension, the tensometer

test pieces (Fig.1) manufactured from sheet material were polished on one face and examined microscopically before and after straining for indications of surface markings. In comparison, the shot peened specimens, where plastic flow occurs in many directions at once, were also examined.

4. MEASUREMENT OF MACRO-STRAIN IN TITANIUM AND A TITANIUM ALLOY BY X-RAY DIFFRACTION

4.1. Introduction.

4.1.1. The basic theory of the X-ray diffraction technique for measuring macro-strains has been covered in the literature survey. The following two methods are used to measure the changes involved:-

- 1) X-ray back reflection.
- 2) Diffractometer.

4.1.2. The basis of the analytical procedure for macro-strain measurement by these two methods are given in Appendix 2. When the macro-strains are measured with a diffractometer, the position of the diffracted beam is measured in terms of angular position 2θ , rather than interplanar spacings as in the back reflection technique. The X-ray back reflection and the diffractometer techniques have been developed for measuring macro-strain in titanium and titanium alloy. The development of the two techniques is described in detail in the following paragraphs:-

4.2. X-Ray Back Reflection Technique.

4.2.1. Choice of Radiation

The diffraction equipment used was Raymax X-ray crystallographic unit. Unfiltered Cu K α , Co K α and Cr K α radiations were used, the appropriate radiation being obtained by inserting a suitable target into the X-ray unit. Theoretical calculations using the $\frac{c}{a}$ values reported in literature for titanium (3), gave the following Bragg angle for a number of high angle peaks of suitable intensity:-

Radiation	Crystallographic plane	Bragg angle	Tube current m.A	Voltage KV
Cu	006	80° 41'	10	50
	302	74° 13'		
Co	114	77° 14'	5	35
Cr	004	77° 54'	9	35

4.2.1.2. For evaluating stresses in commercially pure titanium Co K α radiation was used, whilst the effect of beam penetration was investigated using the three radiations, i.e. Co K α , Cu K α and Cr K α .

4.2.1.3. The X-ray unit was operated with tube current and voltage as shown in the above table.

4.2.1.3. For the purpose of separately determining stresses in the alpha as well as beta phases in 6% Al-4% V Ti alloy IMI.318A, Co K α radiation was selected. Co K α radiation gave high angle peaks of sufficient intensity from the (114) planes of the alpha phase and the (222) planes of the beta phase with Bragg angles of 77° 14' and 72° 35' respectively.

4.2.2. Choice of Calibration Powder

4.2.2.1. For accurate stress measurements, using X-ray back reflection technique, it is necessary to calibrate the film to specimen distance. This is achieved by painting the metallic surface under examination with a very thin coating of a pure, strain free metallic powder. The suitable powder gives its own back reflection lines which can then be used for calibration.

4.2.2.2. Silver calibration powder was selected for measuring stresses in commercially pure titanium IMI.130. Theoretical calculations gave the following high angle reflections of sufficient intensity with the three radiations used:-

Radiation	Crystallographic plane (calibrating powder)	Bragg angle
Cu	511	78° 24'
	333	
Co	331	72° 35'
	420	78° 13'
Cr	222	76° 38'

4.2.2.3. Iron calibration powder was used for determining stresses in 6% Al-4% V Ti alloy IMI.318A giving a Bragg angle of 80° 42' from the (310) plane with Co K α radiation.

4.2.3. Procedure for Determining Stresses

4.2.3.1. A flat-plate back reflection camera was used to record the X-ray diffraction lines. The back reflection films were rectangular in shape and the radiation was collimated with vertical slits, perpendicular to the length of the film. A 0.030 in slit was used for the normal and 30° exposures and a 0.020 in slit for the 40° incidence to compensate for the spreading of the irradiated area upon rotation. In order to bring more planes into the reflecting position during exposure, each specimen was oscillated through 14°. This arrangement was proposed by Bragg and Lipson (147) and Frohnmeyer (148) and by this method, the

scattering of the interference points on the film is avoided. The alternative arrangement proposed by Leiber and Macherauch (86) allows for a shift of the X-ray tube parallel to a loaded specimen thus giving more crystallites in the reflecting position.

4.2.3.2. The appropriate calibration powder was smeared on the metallic surface. For each stress measurement six back reflection films were taken as follows:-

- (i) Two photographs with incident beam normal to the surface of the specimen.
- (ii) Two photographs with incident beam inclined at 30° to the surface normal i.e. one on either side of the normal.
- (iii) Two photographs with incident beam inclined at 40° to the surface normal i.e. one on either side of the normal.

4.2.3.3. The films were measured with a vernier scale by visual means. Since the lines were not diffused recourse was not made to a micro-photometer. The stress values were calculated using the method outlined in Appendix 3. The stress calculation sheets along with the graphs corresponding to a tensile stress of $14.9 \pm 0.7 \text{ tonf/in}^2$ and compressive stress of $14.8 \pm 0.8 \text{ tonf/in}^2$ are also attached to the Appendix 3.

4.2.4. Comparison of X-ray Stress Measurements with those from Strain Gauges and Bending Theory

4.2.4.1. In order to confirm the reliability of the method over a wider range of stresses, the stress value, determined by the X-ray back reflection technique, using the simple isotropic theory, were compared with the theoretical values from elementary bending theory and those determined with strain gauges. The strain gauges were cemented onto both faces of two strips of each of the materials IMI.130 and IMI.318A. Initial surface stress values were obtained for the straight strips by the X-ray back reflection technique. Each strip was then constrained into a circular arc in the 5° , 10° , 15° , 20° , etc. bending jigs (Fig.2) and the stresses measured by the X-ray method on both the tension and compression faces. The tensile strain on the outer layers of each strip was measured with strain gauges and converted to stress using the bulk Young's modulus values obtained from simple tensile tests. After each stress measurement in the bending jigs, the radius of curvature of the strip was determined using a travelling microscope and the theoretical stress was calculated by elementary bending theory.

4.2.5. Choice of Area for Stress Evaluation with Regard to the Radius of Curvature.

4.2.5.1. Normally all the X-ray stress measurements were taken at the mid-position of the various strips and in between the strain gauges. From theoretical considerations, when the strip is constrained to a circular arc in a four point loading jig (Fig.2), the stress should be uniformly distributed along the arc of the circle. In order to confirm this hypothesis and also to minimise any possible error involved in measuring the stress by X-rays on the same spot when the strip was transferred from one jig to another, X-ray stress measurements were taken at the mid-position of the strip and then at a distance of 0.125 in on either side of the mid-position of the strip. The stress measurements were taken when the strip was constrained in a 5° bending jig and the following results were obtained.

Material	Position on the strip	Stress tonf/in ²
IMI.130	Mid-position	5.7 ± 0.5
	0.125 in on the R.H.S. of mid-position	5.3 ± 1.0
	0.125 in on the L.H.S. of mid-position	5.7 ± 0.7

4.2.5.2. The results agreed remarkably well within the experimental error and this eliminated the necessity of taking X-ray stress measurements at the same spot.

4.2.6. Effect of Beam Penetration

4.2.6.1. An earlier investigation had indicated that different stress results were obtained with Cr K α , Co K α and Cu K α radiations. The magnitude of the surface stresses as measured on the tension face of a strip in commercially pure titanium IMI.130 when constrained in a 5° bending jig (Fig.2) increased linearly with decrease in wave length of the X-rays. However, the surface stress pattern on the compression face was of "hook" distribution type giving a subsurface maximum stress.

4.2.6.2. In order to find out the influence of beam penetration on the X-ray stress analysis the strip was constrained at various stress levels, to form the arc of a circle in different bending jigs both in the elastic as well as the plastic range. The X-ray stress measurements were taken on both the tension and compression faces, using unfiltered Cr K α , Co K α and Cu K α radiations. The theoretical stress was derived using the elementary bending theory. The depth of penetration was calculated using the method outlined in Appendix 1.

4.3. Diffractometry.

4.3.1. Instrumentation

A general view of the diffractometer used for measuring macro-strains is shown in Photo 21. A Phillips 1010 X-ray generator and goniometer with Panax electronic equipment was used. Filtered Cu K α radiation operating at 36 KV and 20 mA was used. Further details of the diffractometer ancillaries are given on Pages 68, 69 Chapter 5. A fixed count step scanning technique was employed and the time taken to record the fixed count was printed out on a Teletype machine.

4.3.2. Diffractometer Alignment

4.3.2.1. The diffractometer was aligned such that the divergence slits, goniometer axes and receiving slit fell on a straight line through 0 degree 2 θ . For determining the accuracy of alignment, the high angle reflections from stress free silver powder and a silicon disc, whose peak positions are similar to those given by the Ti, were chart recorded at $\psi = 0,45$ degrees and the 2 θ values obtained, were compared with the theoretical values.

4.3.2.2. When the specimen is rotated to the $\psi = 45$ degrees position, a radial alignment of the counter track is necessary. For example, the usual diffractometer arrangement is shown in Fig. 23a in which the sample surface always makes equal angles with the primary and diffracted beams ($\psi = 0$ degree). When the sample is rotated to $\psi = 45$ degrees, the focussing conditions are destroyed Fig. 23b. Thus the diffracted rays converge at point B instead of point A on the goniometer circle such that on arrival at A not only are they dispersed but their mean position is also changed. For positive values of specimen inclination angle, ψ , the receiving slit and counter must be moved forward along the radius of the goniometer circle to the point B to maintain the Bragg-Brentano focussing conditions. The distance AB which the counter tube receiving slit must be moved forward was calculated using the following equation suggested by Koistinen and Marburger (19).

$$AB = R - R \frac{\cos (\psi + \phi)}{\cos (\psi - \phi)}$$

where :-

R = Source to sample distance or radius of the goniometer circle.

$$\phi = 90 - \theta$$

4.3.2.3. Although true focussing conditions can never be obtained, results from the strain free silicon disc and silver powder showed that this formula gave a satisfactory approximation.

In order to improve the diffraction intensity, all the soller baffles in the receiving slit and the divergent slit systems were removed.

4.3.3. Instrumental Correction for Peak Shifts due to Counter Movement

4.3.3.1. In order to correct for any peak shift contribution resulting from the radial alignment and rotation of specimen through $\psi = 45$ degrees, high angle reflections from stress free silicon disc and silver powder with peak positions close to those of titanium were chart recorded. The difference in peak position relative to $\psi = 0^\circ$, $\psi = 45^\circ$ was calculated. Subsequently the peak position of silver powder was checked at regular intervals by the fixed count step scanning technique.

4.3.4. Procedure for Stress Measurements

4.3.4.1. In the two-exposure method the elastic strains associated with a set of crystallographic planes are related to the stress by the formula derived in Appendix 2.

$$\sigma_\phi = K (2\theta_\perp - 2\theta_\psi)$$

where:-

σ_ϕ = residual surface stress.

K = Stress factor determined experimentally.

$2\theta_\perp$ = Position of selected peak with specimen in normal position.

$2\theta_\psi$ = Position of selected peak with specimen at angle ψ relative to normal position; $\psi = 45$ degrees was used in this study.

4.3.4.2. For accurate stress measurement, the following requirements are essential:-

- (i) the diffraction peak must be of sufficient intensity so that its position can be accurately measured.
- (ii) the peak should occur at a higher Bragg angle.
- (iii) the peak position must be identical at zero stress for different values of ψ .

4.3.4.3. Thus the 213 reflection at $2\theta = 139.4^\circ$ with Cu K α radiation was found to be suitable for IMI.130 and IMI.318A. The third requirement was satisfied by correct diffractometer alignment.

4.3.5. Determination of Diffraction Peak Position

4.3.5.1. The determination of the diffraction peak position can be subdivided into 3 parts as follows:-

- (i) Recording the diffraction peak.
- (ii) Correcting it for 2θ - dependent intensity factors such as Lorentz, polarisation and absorption factors.
- (iii) Locating and measuring the peak positions.

4.3.5.2. A fixed count step scanning technique was used to record the peak. The intensity was measured as reciprocal intensity by recording the time for 10,000 counts by automatically step scanning at 0.05 degree 2θ intervals. From these data points, five positions of 2θ were selected for a more accurate determination of the inverse intensities at each position by accumulating 100,000 counts, by step scanning manually at 0.1 degree 2θ intervals. The time taken for 10,000 or 100,000 counts was printed out on a teletype machine.

4.3.5.3. The measured inverse intensities were corrected for factors sensitive to 2θ by multiplying the inverse intensities by Lorentz, polarisation and absorption factors. The Lorentz-polarisation factors were used to correct the data obtained at 0 degree ψ and the combined Lorentz-polarisation and absorption factors were used at an angle of $\psi = 45^\circ$.

4.3.5.4. The peak positions were determined by the three point parabola method of Koistinen and Marburger (19). The three point parabola was fitted to the data, and angle 2θ corresponding to the maximum intensity was calculated using the following equation:-

$$2\theta = 2\theta_1 + \frac{C}{2} \left(\frac{3a + b}{a + b} \right)$$

where:-

$2\theta_1$ = First data angle.

$2\theta_1, 2\theta_2, 2\theta_3$ = Consecutive 2θ positions at which inverse intensity is determined.

C = $2\theta_2 - 2\theta_1$ or $2\theta_3 - 2\theta_2$

a = $t_1 - t_2$ (corrected times)

b = $t_3 - t_2$ (corrected times)

t_1, t_2, t_3 = time required to accumulate given number of counts (100,000) at $2\theta_1, 2\theta_2$ and $2\theta_3$ positions.

4.3.6. Stress Factor Calibration

4.3.6.1. The accuracy of stress measurement ultimately depends upon the stress constant K in equation ²¹ on Page 122.

4.3.6.2. In order to determine the stress factor experimentally, a special bending jig (Photo 21) was designed to stress the specimen in a four point loading device. This device allowed either tensile or compressive stresses to be applied to the upper surface of a rectangular beam specimen. Specimens 6 in x .070 in x .036 in were deformed by four point loading so that uniform bending moments were applied between the inner supports, and the component of the applied stress in the irradiated volume of metal applied stress in the irradiated volume of metal was constant. The strain gauges were cemented onto both faces of the strips. Each strip was constrained into a circular arc in the 5° , 10° , $12\frac{1}{2}^\circ$, 15° , 20° , 25° , 30° , 35° , 40° , 45° , 50° and 55° bending jigs (Fig. 2) as appropriate and the applied strain was noted from the strain gauges. The strain was also calculated using bending theory. The applied strain was converted into stress using the bulk elastic constants determined from tensile specimens of each material. In order to eliminate any possible errors arising from the time and temperature dependence of strain gauges, the strain was noted in each fixed bending jig (Fig. 2) and the same strain was applied when the strip was transferred to the variable jig (Photo 21). The process was repeated for all the jigs. This procedure had the further advantage that the strain measured by the back reflection technique using a fixed jig, diffractometry using the variable jig, strain gauges and bending theory could be directly compared. For calibration purposes the elastic limit of the material was not exceeded.

4.3.6.3. In order to determine the effects of unidirectional plastic deformation on stress measurement, the specimens were further strained in the plastic region. The observed values of $\Delta 2\theta = 2\theta_1 - 2\theta_{45}$ were corrected for instrumental effects; $\Delta 2\theta$ was plotted against the applied stresses and the slope of the line gave the stress factor for each material.

4.3.7. Determination of X-ray Elastic Constants.

4.3.7.1. The stress factor K (equation 21 Appendix 2 Page 122) contains the quantities E and ν . The values of E and ν , however, vary with the crystallographic direction of a particular reflection and the wave length of radiation used. It is reported in literature (Page 135) that there is an appreciable difference (up to 40%) between the values of E and ν determined mechanically and those measured by the X-ray diffraction technique. Therefore the bulk

values determined mechanically are not the correct ones to apply to diffraction measurement.

4.3.7.2. The theory of the method for determining X-ray elastic moduli is included in Appendix 2. The strain was applied by means of four point loading. The applied strain computed from the bending theory and also from strain gauges was converted into stress using the bulk elastic constants.

4.3.8. Calibration of Diffractometer for Study of Curved Surfaces

4.3.8.1. The Phillips diffractometer is designed only for a flat specimen. In order to achieve proper focussing it is essential that the following conditions are satisfied for all diffraction angles:-

- (i) the line source, specimen surface, and receiving slit axis are all parallel;
- (ii) the specimen surface coincides with the diffractometer axis;
- (iii) the line source and receiving slit both be on the diffractometer circle.

4.3.8.2. When determining the stress factor, the rectangular strip is placed in the bending fixture (Photo 21) and increments of strains are applied. The convex or concave sides of the strip which is exposed to the X-ray beam is then no longer tangent to the focussing circle and hence the proper focussing conditions are destroyed. Whilst designing the bending fixture provision was made to move the whole frame up or down so that the specimen surface always coincided with the diffractometer axis.

4.3.8.3. The effect of roughness of specimen surface on the X-ray stress analysis has been investigated (217) whilst the effect of curved surfaces on the determination of the stress factor using the diffractometer has not been investigated so far. The problem of stress determination using the X-ray back reflection technique is not so serious because collimated X-ray beam with vertical slits perpendicular to the length of the film are used. Normally a 0.030 in slit is used for the normal and 30° exposures and a 0.020 in slit for the 40° incidence to compensate for the spreading of the irradiated area upon rotation. In the diffractometer the X-ray beam is wider. In order to investigate the effect of curved surfaces on the stress factor calibration, experiments were designed to stress relieve the strips of commercially pure titanium IMI.130 in various bending jigs. (Fig. 2) made in steel. The annealed strips were examined by the X-ray back reflection technique and radial streaking was observed. The strips were not stress free. Further details are included in Appendix 7.

4.4. Measurement of Residual Lattice Stress in Plastically Deformed Commercially Pure Titanium IMI.130.

4.4.1. Generally polycrystalline materials deformed plastically in uniaxial tensile show a line shift indicating residual lattice strains which are not shown by mechanical methods. However, when residual stresses are induced by distortion accompanying hardening, shot peening or machining, good agreement has been obtained between measurement of these stresses by X-rays and mechanical methods. Several attempts have been made to explain the origin of the residual lattice strains existing in specimen deformed in a tensile test but no clear cut explanation has yet emerged. Further changes have been observed on progressively thinning the deformed specimen. So far, the investigation has been confined to either face centred cubic or body centred cubic material but close packed hexagonal material has received little attention.

4.4.2. Because of the lack of agreement between X-ray and mechanical methods of stress measurement in a uniaxial tensile test, a tensile test piece (Fig.1) was manufactured from IMI.130 sheet material in the as received condition. The specimen was subjected to uniaxial loading at a constant rate in a 50 ton Denison testing machine. The specimen was extended to 10% plastic strain. The parallel portion of the tensile test piece was cut off for subsequent strain analysis. The macro-stresses were measured by the X-ray back reflection technique in the longitudinal direction i.e. direction of pulling and transverse direction on the maximum strained portion of specimen. Occasionally, stresses were also measured using the diffractometer. The variation of strain was further investigated as successive layers were removed by chemical polishing using the following solution:-

HNO ₃ (36°B)	250 ml	+67 ml - 0 ml
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HF (40%)	40 ml	+ 3 ml
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H₂O distilled to one litre

Temperature of bath - cold.

4.4.3. Micro-strains were also measured using the integral breadth method outlined in Chapter 5.

4.4.4. For comparison, the effect of other deformation processes such as shot peening and grinding, which produce significant alterations in the surface layers of metals resulting in residual lattice strains were also investigated. The shot peened specimens were taken from the blanks peened to 0.014A2 and 0.020A2 intensities whilst the ground test coupon G2 with down feed rate of .001 in per pass was studied. The details of the work processes are included in Appendix 6. Both the macro-stresses and micro-stresses were measured on the treated surfaces as well as below the surfaces. The surface layers were removed by chemical milling with the solution mentioned in the preceding paragraph. During chemical milling the untreated surfaces were stopped off with a lacquer.

4.4.5. The X-ray technique was then applied to measure macro-stresses in IMI.130 deformed by shot peening, vapour blasting, grinding, milling and turning. The details of these treated surfaces are given in Appendix 6.

5. MEASUREMENT OF MICRO-STRAIN IN TITANIUM BY X-RAY DIFFRACTION

5.1. Introduction.

5.1.1. Preliminary investigation showed that in the X-ray back reflection films taken from the IMI.130 strips plastically deformed in bending, before and after releasing the strips from the bending jigs, the $K\alpha$ doublet was clearly distinguished. On the other hand, films taken from the shot peened and machined surfaces in IMI.130 indicated that the diffraction lines were broadened and diffused, and the $K\alpha$ doublet was not distinguishable. Even after long exposures it was difficult to determine the stress in shot peened or machined surfaces by the film technique. It was therefore postulated that the plastic flow in commercially pure titanium IMI.130 could be explained in terms of two different modes of plastic deformation. Probably micro-stresses, crystallite sizes and stacking faults, played a major part in the plastically deformed shot peened surfaces. Machining, grinding or other working processes can produce significant alterations in the surface layers of metals such as plastic deformation, high temperature gradients, rehardening and/or over-tempering in steels, and residual stresses. The residual micro-stresses may influence stress corrosion and mechanical properties including fatigue and distortion of the component. In order to study the influence of various working processes, experiments were initially designed to study the effect of shot peening, grinding, milling, turning and vapour blasting on the microstress measurements in commercially pure titanium. One specimen was also pulled in uniaxial tension. The details of these working processes are included in Appendix 6. After the initial experimentation, the effect of shot peening and grinding was further studied. The effect of various working processes on the line intensity of various profiles was also studied.

5.2. Diffractometer Equipment and Technique.

5.2.1. The X-ray diffraction line profiles were chart recorded using a standard Phillips diffractometer. This consisted of a P.W.1010 stabilised X-ray generator, a P.W.1050 goniometer and proportional counter, and a P.W.1051 scalar and recording panel. The slowest scanning speed of $\frac{1}{8}^{\circ}$ per minute in deviation angle, 2θ , was used. Ni-filtered Cu $K\alpha$ radiation was used. The following divergence, scatter and receiving slits were used to ensure that at least half the specimen area was irradiated for any 2θ .

<u>Angular Range</u>	<u>Divergence Slit</u>	<u>Receiving Slit</u>	<u>Scatter Slit</u>
20 - 40°	1°	0.1 mm	1°
40 - 80°	2°	0.1 mm	2°
80 - 150°	4°	0.2 mm	4°

5.3. Analysis of Peak Profile.

5.3.1. A brief review of X-ray line broadening studies of plastically deformed metals is given in the literature survey. Taking into consideration the intensity, overlapping reflections and absence of multiple order of reflections, it was decided to use the integral breadth to study titanium. This method is simple to use and has the added advantage of not requiring the use of elaborate computational facilities.

5.3.2. Integral breadth method

5.3.2.1. The line broadening produced by plastic deformation is due mainly to two factors:-

- (a) reduction in crystallite size due to the production of new domains and faulting
- (b) increase in micro-strains.

5.3.2.2. The integral breadth due to crystallite size and micro-strains can be derived from Bragg's law. The Scherrer (157) formula relating broadening to mean particle dimensions is as follows:-

$$\beta_{PF} = \frac{K\lambda}{t \cos \theta}$$

β_{PF} = broadening due to small particle size

t = mean particle dimension

K = constant which varies with particle shape but is close to unity.

5.3.2.3. The profile due to crystallite size effects approximates to a Cauchy distribution ($y = (1 + a^2 x^2)^{-1}$).

5.3.2.4. When the broadening is due to micro-strains, Stokes and Wilson (158) have shown the following relationship between broadening β_S and strain ϵ :-

$$\beta_S = 2 \epsilon \tan \theta$$

5.3.2.5. The profile due to micro-strains approximates to a Gaussian distribution ($y = \exp(-a^2 x^2)$).

5.3.2.6. There is no simple relation to separate the effect of β_{PF} and β_S in the measured broadening. Hall and Williamson (117) assumed that these two effects are additive. They used the following expression:-

$$\beta_T = \beta_{PF} + \beta_S$$

where β_T = broadening due to cold work.

This method of analysis assumed that both quantities were represented by a Cauchy distribution of intensity. In the above equation

$$\beta_T = \frac{K\lambda}{t \cos \theta} + 2\epsilon \tan \theta$$

or

$$\frac{\beta_T \cos \theta}{\lambda} = \frac{1}{t} + 2\epsilon \frac{\sin \theta}{\lambda}$$

or

$$\beta_T^* = \frac{1}{t} + \epsilon d^*$$

where:

$$\beta_T^* = \frac{\beta \cos \theta}{\lambda}$$

$$d^* = \frac{2 \sin \theta}{\lambda}$$

Thus a plot of β^* versus d^* will give a straight line relationship, the slope of such plots being a function of strain and the intercept on the β^* axis being a function of the crystallite size.

5.3.2.7. For a Cauchy strain distribution, it can be shown that the mean squared strain is infinite, therefore a Cauchy strain distribution is physically unrealistic. The Hall-Williamson method would therefore be suitable, where crystallite size is the major cause of broadening.

Wagner and Aqua (159) have used the following relationship:-

$$(\beta_T)^2 = (\beta_{PF})^2 + (\beta_S)^2$$

or

$$(\beta_T^*)^2 = \frac{1}{t^2} + \epsilon^2 (d^*)^2$$

5.3.2.8. This method assumes that both micro-strains and crystallite sizes give rise to a Gaussian distribution of intensity. Therefore this method would be more suitable where micro-strains are the major cause of broadening.

5.3.3. Method of applying the Integral Breadth Method

5.3.3.1. Since the radiation used was not monochromatic but consisted of a $K\alpha_1$ and $K\alpha_2$ doublet, it was necessary to separate the $K\alpha_2$ component. This was achieved by using the Rachingers separation method (Appendix 5). It

was assumed that the intensity of $K\alpha_1$ peak is twice that of $K\alpha_2$ peak.

Using Rachingers method the $K\alpha_2$ peak was resolved out of the $\alpha_1 \alpha_2$ doublet leaving the $K\alpha_1$ peak for subsequent analysis.

5.3.3.2. Next $\beta(2\theta)$ was calculated. The total area under the curve was calculated manually by counting the area under the peak and then dividing by the peak height. The area under the curve was also calculated using the Allbrit Compensating Planimeter.

5.3.3.3. The broadening β_S due to the presence of micro-strains and crystallite sizes is obtained by using the following empirical equation developed by Wagner and Aqua (159) :-

$$\beta_S^* = \beta_T^* - \frac{(\beta_I^*)^2}{\beta_T^*}$$

Where:

$$\beta^* = \frac{\cos \theta}{\lambda}$$

$$\beta_S^* = \text{broadening due to crystallite size, and microstrain}$$

$$\beta_T^* = \text{total broadening (crystallite size, micro-strain and instrumental.)}$$

$$\beta_I^* = \text{instrumental broadening.}$$

Commercially pure titanium IMI.130 annealed under vacuum at 900°C - 1 hr vacuum cool was used to correct for instrumental broadening.

5.3.3.4. The values for micro-strain ϵ and crystallite size, D_{PF} are obtained using the following equation:-

$$(\beta_S^*)^2 = \frac{1}{(D_{PF})^2} + \frac{16 \epsilon^2 \sin^2 \theta}{\lambda^2}$$

Plots of $(\beta_S^*)^2$ versus $(d^*)^2$ are made. The intercept at $\frac{4 \sin^2 \theta}{\lambda^2}$

yields directly $\frac{1}{(D_{PF})^2}$ and the slope is equal to $4\epsilon^2$, where $2\epsilon = \frac{\Delta d}{d}$.

5.3.5. Application of Integral Breadth Method

5.3.5.1. The integral breadths of line profiles of the specimen in commercially pure titanium subjected to various working processes were calculated. These results were analysed by the Hall-Williamson method (117) and the Wagner-Aqua

method (159). Two examples of the variations (β^*) versus (d^*) and $(\beta^*)^2$ versus $(d^*)^2$ are shown in Figs. 29, 30 respectively. The best straight line was drawn by the least square method using the computational facilities. From the results, slopes of the lines, intercepts on the (β^*) and $(\beta^*)^2$ were obtained.

5.4. Effect of Preferred Orientation on Intensity Distribution and Instrumental Effect

5.4.1. Generally two methods are used to allow for the instrumental effects:-

- (i) Theoretical analysis of the individual factor contributing to peak width and shifts such as slit width, beam divergence etc.
- (ii) Measurement of the broadening of profiles of various reflections from a well annealed sample of the material under investigation.

5.4.2. The second method is widely used since the instrumental correction is obtained from the annealed and strain free material sample. However, preferred orientation can be a major source of error in determining the intensity of various reflections. For example, Klug and Alexander (227) have shown the effect of preferred orientation on intensity distribution Birks (228) has concluded that the reduction of crystallite size by prolonged grinding is the most effective method of removing preferred orientation in powder materials.

5.4.3. The experimental evidence showed that intensity distribution of the various reflections obtained from shot peened, ground surfaces or sheet material varied in comparison with those recorded in the ASTM Card Index and this factor lead to some difficulty in correcting the observed broadening for instrumental effects. A sample of titanium powder was considered unsuitable because of lattice distortion resulting from oxygen adsorption. The details are given in Appendix 8.

5.5. Effect of Annealing Temperature on Shot Peened Materials

5.5.1. The effect of annealing temperatures on the shot peened block specimens, peened to an intensity of 0.014A2, was studied in the temperature range 300°C and 500°C. The block specimens were sealed in an evacuated silica tube to prevent oxidation of the specimen surface during heat treatment. The tubing containing the small block specimen was flattened at one end to reduce end distortion during heat treatment. The heat treatment was carried out in electric muffle furnaces and the specimen in the silica tube were allowed to cool in the furnace.

6. RESULTS

6.1. Static Mechanical Properties

6.1.2. The results of static mechanical properties obtained from IMI.130 and IMI.318A are shown in tables 1 and 2 respectively.

6.2. Surface Properties

6.2.1. The surface treatment of titanium prior to stress measurement is an important factor because titanium has a strong affinity for oxygen, and the oxide film could affect the stress analysis. Preliminary work indicated that electropolishing was not satisfactory (20) because it resulted in changes in the diffraction pattern. For surface preparation of the annealed strips, prior to stress analysis, in both materials, IMI.130 and IMI.318A, chemical milling using the following solution is used to give a stress free surface:-

HNO ₃ 36°B	250 ml + 67 ml - 0 ml
HF 40%	40 ml ± 3 ml
H ₂ O (distilled)	balance to make up the solution to one litre
Temp. of bath	50° - 60°C

6.2.2. The same solution at room temperature is used to remove layers for subsurface measurement. The total removal is uniform even after repeated polishing. The area used is large enough to minimise a possible stress concentration effect.

6.3. Micro-Hardness Testing of Shot Peened Surfaces

6.3.1. Depth/micro-hardness distribution measurements were made and used to indicate the presence of cold worked material due to shot peening. The results in Figs.4 and 5 indicate the apparent influence of initial hardness of IMI.130 on subsequent strain hardening due to shot peening to an intensity of 0.014A2; an unrestrained Almen strip with a core hardness of 172 HV 10 strain hardened to a greater degree on the surface and to a greater depth below the surface (Fig.4) compared to the rigid block with core hardness of 127 HV 10. The results in Fig.6 obtained with three different shot peening intensities confirm the effect of initial hardness on strain hardening in IMI.130. However, with a duplex phase alpha-beta titanium alloy IMI.318A (core hardness 313 HV 10) strain hardening effects shown in Fig.7 were difficult to detect. This could possibly be attributed to the morphology of alpha, beta phases in alloy.

6.4. Measurement by X-ray Back Reflection Technique of Applied and Residual Macro Stresses in IMI.130

6.4.1. The results of applied surface macro-stresses, measured by X-ray diffraction, strain gauges and calculated by the bending theory on bending a strip TT2 are shown in Table 3 and illustrated graphically in Figs.8 and 9. The strip was deformed elastically and plastically in specially designed jigs (Fig.3 Photo 20). The X-ray diffraction data were obtained using Co K α radiation and Ag calibration powder. The results show that with the application of external loads in the elastic range, the X-ray stress values are in good agreement with those measured by strain gauges and calculated by the bending theory. The curves (Fig.9) also show that the onset of plastic deformation commences at a threshold value of 13-15 tonf/in² approximately in both tension and compression in that the curves flatten out above this stress value. The residual stresses measured on the compression face of the unconstrained strip condition, indicate that the residual stresses do not develop before the onset of plastic deformation. The increase in the magnitude of residual stress is $\sim$ linear with the increase in plastic strain. Results in Figs.8 and 9 show that in the elastic range there is good agreement between stress values obtained by the three methods.

6.4.2. Fig.10 shows the relationship between applied surface stress and strain on bending a second strip TT3, using Cu K α , Co K α and Cr K α radiations and Ag calibration powder. The results obtained with three radiations are shown in Tables 4 - 6 and illustrated individually for each radiation in Figs.11-13 respectively. The slopes of the curves obtained with the three radiations are marginally different in that the slopes of the curves obtained with Cu K α and Co K α radiations are greater than those with Cr K α radiation. Further the slopes of the line, with either radiation are smaller in magnitude on the compression face than those on the tension face. The curves also show that with the three radiations used, the onset of plastic deformation commences at $\sim$ 13-15 tonf/in² in tension and compression.

6.4.3. Figs.14-18 show the effect of beam penetration using different X-ray wave lengths on applied surface stresses and strain on bending strip TT3. Similar curves are obtained on both tension and compression faces of the strip in the elastic as well as plastic deformation range.

With smaller amount of plastic strain (Figs.16, 17 bending jigs 20° , 25°), the difference in stress values obtained with three radiations is not appreciable but with a larger amount of plastic deformation (Fig.18), the difference obtained is discernible. The results in Fig.18 show that the stress values obtained with Cr K α radiation are virtually half ^{than} those obtained with Cu K α radiation.

6.5. Measurement by X-ray Back Reflection Technique of Applied and Residual Macro-stresses in IMI.318A

6.5.1. The results of applied surface stresses measured by strain gauges and calculated by bending theory on bending strips BB2 and CC3 are shown in Figs.19, 21. The strip CC3 was deformed both elastically and plastically in specially designed bending jigs (Fig.2). The results show that there is good agreement between the two method of stress determination.

6.5.2. Fig.20 shows the relationship between the applied surface macro-stress measured by X-ray diffraction and strain (for both alpha and beta phases) on bending strip BB2 in bending jigs. Co K α radiation and Fe calibration powder was used. The results show that the curves are similar on the compression and tension faces of the strip. The relationship is linear between the measured applied stress and strain in the elastic range. The measured stress in alpha phase are lower in magnitude than those measured in beta phase, the difference increasing with increase in stress level.

6.5.3. Fig.22 shows the relationship between the surface applied macro-stresses and strain (for both alpha and beta phases) on bending strip CC3 in specially designed jigs. The residual macro-stresses measured (for both alpha and beta phases) on the compression and tension faces after releasing the strip from various bending jigs are also shown. It is to be seen that the applied macro-stresses are similar in both phases. Further, the onset of plastic deformation occurs at a similar stress level in both the alpha and beta phases. The results also show that the residual macro-stresses on the tension as well as the compression faces are closely associated with the onset of plastic deformation. The residual macro-stresses measured on the compression face give a higher magnitude of tensile stresses compared to the residual compressive macro-stresses measured on the tension face. Further the level of

residual macro-stresses is higher in the beta phase compared to the alpha phase irrespective of stress measurement on the tension or compression faces of the strip.

6.6. Development of Jig to Measure Macro-stresses using a Diffractometer

6.6.1. Photo 21 shows the variable bending jig developed to measure macro-stresses in both IMI.130 and IMI.318A. This device allows either tensile or compressive stresses to be applied to the upper surface of the strip. In order to achieve accuracy and standardisation for measuring macro-stresses the following major problems were resolved:-

- (a) radial alignment of the diffractometer
- (b) parafofocussing conditions with specimen in the normal, and at 45° to the normal, positions
- (c) location of the peak position using the 3 point parabola method
- (d) instrumental correction factor.

6.6.2. Fig.23 shows the focussing geometry for a flat specimen in the normal, and at angle ψ to the normal, positions. In the normal position (Fig.23a) the diffracting crystal planes are virtually parallel to the specimen surface. When the specimen is rotated through angle ψ , the focus of the beam is now closer to the sample surface (Fig.23b). It is therefore necessary to move forward the detector and receiving slits to get the optimum focus positions. For example Figure 24a shows the diffractometer traces of the standard silicon disc. It is to be seen that with the sample position being rotated from $\psi = 0^\circ$ to $\psi = 45^\circ$, considerable shift in the angular position occurs. Further there is a change in the background level as well as the intensity of the diffracting planes. Fig.24b shows the effect of radial alignment with the sample at $\psi = 45^\circ$ to the normal position. It is to be noted that the distance of the counter tube has an important bearing on the parafofocussing conditions. The peak positions are sharper and the $K\alpha_1$ $K\alpha_2$ doublet well resolved when distance the counter tube is correct. Further there is change in the background level as well as the intensity of the diffracting planes with the change in counter head tube distance.

6.6.3. The diffractometer traces obtained subsequently from annealed IMI.130 and stress free silver powder are shown in Fig.25. The results show that instrumental correction is necessary for the correct evaluation of stress analysis data.

6.6.4. During the experimental stages it was observed that even the tightening up of the nuts and bolts after moving the counter tube to and fro affected the peak positions. After the standardisation of the technique, stress free silver powder was analysed to check for any peak contribution from specimen rotation through $\Psi = 45^\circ$. The peak position of 422 reflection from silver powder occurs closely to the specimen peak position. Measurements were made at positions of 422 reflection with the specimen in the normal, and at 45° to the normal positions respectively. The peak of the 422 reflection in the normal position occurred within $0.005^\circ 2\theta$. The deviation obtained was $-0.254^\circ 2\theta$ and this instrumental correction factor was checked periodically. This value has been used throughout to correct the experimentally observed peak positions.

6.7. Stress Factor Calibration

6.7.1. The results of calibration to determine the stress factor for IMI.130 and IMI.318A are shown in Tables 9 and 10, and Figure 26. The stress determined from strain gauges and calculated by bending theory is plotted against peak $\Delta 2\theta$, measured by X-ray diffraction. The data in Fig.26a were obtained by the combined use of the variable jig (Photo 21) and the bending jigs (Fig.2). The data in Fig.26b were obtained using the variable jig only. The slopes of the lines give a stress factor of 34 and 30 tonf/in² for IMI.130 and IMI.318A respectively. It is to be seen that the slopes of the lines for IMI.130 shown in Figs.26a and 26b are identical. This compares with the stress factor of 31.25 tonf/in² (70 Ksi) determined by Esquivel (230) for the material similar to IMI.318A. He, however, used a 'u' bend device for bending the strips, and this may not give unidirectional deformation as in four point loading.

6.8. Determination of X-ray Elastic Constants

6.8.1. The results for determining the X-ray elastic constants by the two exposure technique for IMI.130 and IMI.318A are shown in Figs.27 and 28 respectively. The theory of the method is given in Appendix 2. The data in Tables 9 and 10 were used to plot the change in interplanar spacing against stress shown in Figs.27 and 28 for both the materials.

The slopes of the lines $\psi = 0^\circ$, $\psi = 45^\circ$ are calculated. The values of X-ray elastic constants thus determined are given below:

<u>Material</u>	<u>Young's Modulus</u> <u>lbf/in² x 10⁶</u>	<u>Poisson's ratio</u>
IMI.130	16.45	.297
IMI.318A	14.63	.375

6.8.2. It is to be noted that the value of d_{11} is obtained without assuming that the specimen is stress free.

6.9. Measurement of Micro-stresses in IMI.130 using a Diffractometer

6.9.1. The theory of micro-strain measurement is given in Chapter 5. The results obtained by both the Hall and Williamson method, and the Wagner and Aqua method are shown in Tables 11, 12, 14 and 15, and shown graphically in Figs. 32, 33, 35, 37 and 38. Figs. 29 and 30 show typical examples of determining micro-strains by the two methods used. For the Hall and Williamson method (β^*) against (d^*) as shown in Fig. 29 is plotted whilst for the Wagner and Aqua method $(\beta^*)^2$ against $(d^*)^2$ as shown in Fig. 30 is plotted. The best straight lines are drawn by the least square method using the computational facilities. The slopes of the lines and intercepts on the (β^*) and $(\beta^*)^2$ are obtained. The data obtained are further processed to get the micro-strains values and crystallite sizes. Bulk values of Young's modulus determined from the tensile testing is used to convert micro-strain to micro-stresses.

6.10. Effect of Plastic Deformation in Uniaxial Tension on Residual Macro- and Micro-stresses, in IMI.130

6.10.1. Fig. 31 shows the results of macro-stress measurements by X-ray back reflection technique using Co K γ radiation in both the longitudinal and transverse directions. The tensometer test pieces S_1 Fig. 1 were deformed plastically in uniaxial tension. The macro-stresses are virtually constant in the transverse direction but vary in the longitudinal direction. In the latter case macro-stresses increased with increase in amount of plastic strain and further these were of a higher magnitude than in the former case.

6.10.2. Fig. 32 shows the variation of macro- and micro-stresses in a tensile test piece (S_3 Fig. 1) deformed by 10% plastic strain in uniaxial tension. The macro-stresses are compressive at the surface and, after decreasing with depth, become virtually constant. The micro-stresses, however, initially increase with depth and then remain substantially constant.

6.11. Effect of Surface Treatments on the Residual Macro- and Micro-stresses in IMI.130 and Macro-stresses in IMI.318A.

6.11.1. The results of macro- and micro-stress measurements both on the surface and subsurface for IMI.130 subjected to a variety of deformation processes are given in Tables 12-14 and shown in Figs.33-38.

6.11.2. Fig.33 shows the influence on the surface macro- and micro-stresses of varying the down feed rate used in grinding. Increasing the down feed rate initially causes the surface compressive macro-stress to increase, and then to decrease, still higher rates lead to a further increase. The macro-stresses measured in the grinding direction as well as orthogonal to the grinding direction give similar results. The micro-stresses also show a variation in magnitude with down feed rate. The correlation between the compressive macro-stresses and micro-stresses is to be noted; thus the micro-stress increases as the compressive macro-stress increases, and decreases as the macro-stress decreases. It is believed that the shapes of the curves are valid within the limits of accuracy.

6.11.3. Figs.34 and 35 show the variation of macro-stress and micro-stress respectively, with depth below surface, in the ground and shot peened materials. In ground specimen the stresses were measured orthogonal to the grinding direction. The similarity between macro- and micro-stresses is again to be noted. Fig.36 shows the macro-stress profile in the grinding direction. The macro-stress subsurface profiles in the grinding direction (Fig.36) and orthogonal to the grinding direction (Fig.35) are similar.

6.11.4. The initial hardness of the specimens ground and shot peened to an intensity 0.014A2 was 128 HV 10 compared with that of the specimen shot peened to an intensity 0.020A2 which was 190 HV 10. The specimens ground and shot peened to intensity 0.014A2 are seen to develop considerably different surface compressive macro-stresses without any difference in micro-stresses; similarly, the two peening intensities give differences in the surface macro-stresses, but not in micro-stresses.

6.11.5. The influence of initial hardness may also be seen in the variation of both macro- and micro-stress subsurface profiles in the shot peened specimen. In the higher hardness material, both the macro-stress and the micro-stress is affected to a far greater depth than with the lower hardness material.

6.11.6. Although the macro-stresses in ground specimen change below the surface from compressive to tensile (Figs. 34 and 36) in the grinding direction as well as orthogonal to the grinding direction, the magnitude in the latter case is insignificant.

6.11.7. Fig. 37 shows the influence on the surface stresses of varying the shot peening intensities in both IMI.130 and IMI.318A. Increasing the peening intensity causes the macro-stress to increase and tends to reach a saturation value. The micro-stresses in IMI.130 also show a similar variation in magnitude with peening intensities. The similarity between the compressive macro-stresses and micro-stresses in IMI.130 is again to be noted.

6.11.8. The results of macro- and micro-stresses and crystallite sizes obtained from vapour blasting, turning and milling IMI.130 are shown in Table 14. It is to be seen that in face end milled specimen both the macro- and micro-stresses are of higher magnitude than in the turned specimen. The level of micro-stresses in milled specimen is approaching the limit of proportionality of titanium determined in a tensile test.

6.11.9. Fig. 38 shows the variation of micro-stresses and crystallite sizes with annealing temperature of specimens shot peened to an intensity of 0.014A2 in IMI.130. The rate of micro-stress relief increases with increase in annealing temperature, whilst crystallite sizes increase from an initial value of 279 to 602 Å⁰. The results are typical of cold worked and annealed materials.

6.11.10. Fig. 39 shows the effect of shot peening in IMI.318A on the macro-stress distribution with depth below surface. The surface compressive stresses initially increase and then decrease with depth below the surface. The blank shot peened to an intensity of 0.014A2 exhibited higher subsurface compressive stress than the blank peened to an intensity of 0.018A2. The correlation between the subsurface micro-hardness (Fig. 6) results and macro-stresses is to be noted.

6.12. Stereoscan Microscopy

6.12.1. Photos 9-16 show the stereoscan micrographs of the ground surfaces of IMI.130, with different down feed rate. Apparently the surface damage has increased with increase in down feed rate (Photos 9-14) but in comparison the surface damage is considerably more in the case of specimen ground with down feed rate of 0.003 in/pass (Photos 15, 16).

6.13. Transmission Electron Microscopy

6.13.1. Photos 17 and 18 show the electron micrographs obtained from the thin foils of shot peened surface in IMI.130. The micrographs show the high dislocation density in the damaged subsurface region. The straight and diffuse bands probably correspond to the mechanical twins observed in Photo 6. Photo micrograph 18 shows that there is evidence of the initial stages of substructure formation due to peening.

6.13.2. Photo 19 shows the electromicrograph obtained from thin foil of ground surface in IMI.130 with down feed of 0.0005 in/pass. The dislocations are arranged in poorly developed substructure boundaries. The difference in shot peened surface (Photo 7-8) and ground surface (Photo 10) is to be noted.

6.14. Optical Microscopy

6.14.1. Photos 4 and 5 exhibit the fine grain structure of annealed strips in IMI.130 and IMI.318A respectively.

6.14.2. Photo 6 taken at right angle to the surface shows the density of twins in the damaged region of shot peened IMI.130 to an intensity of 0.014A2. The twinning density decreases with depth below the surface, thus indicating the relationship between twinning and work hardening due to shot peening in IMI.130.

6.14.3. Photos 7 and 8 show the surface damage due to grinding with down feed of 0.003 in/pass in IMI.130. The deformed layer is associated with twinning and further micro-crack initiation at the twin interfaces is to be seen. The photos were taken orthogonal to the ground surface.

7. DISCUSSION

This work has been concerned with the development of methods for the measurement of macro- and micro-stresses in titanium produced by various deformation processes used in the production of aircraft components. The results have shown that these production deformation processes give rise to residual macro- and micro-stresses and that these stresses can be accurately measured by X-ray diffraction methods. The different deformation processes produce different patterns of stress distribution. The residual macro- and micro-stresses occur simultaneously in the surface regions and vary in a similar way in the subsurface regions, thus demonstrating their association.

When the residual stresses are induced by industrial methods such as shot peening, machining or grinding, good agreement is obtained between the X-ray diffraction and mechanical methods of measurement (19,93). When the residual stresses are produced by deforming the specimen in unidirectional or uniaxial tension, there is lack of agreement between these two methods of measurements. Uniform bending in four point bending is included because the surfaces are deformed predominantly in one direction. The presence of residual stresses in titanium specimens deformed in uniaxial tension must be examined in the light of current theories and the origin of residual lattice strains explained.

The results are discussed in the following main sections :-

- 7.1. Experimental methods.
- 7.2. Factors affecting the accuracy of macro- and micro-stress measurements.
- 7.3. Effect of deformation in unidirectional and uniaxial tension on the applied and residual stresses in IMI 130 and IMI 318A.
- 7.4. Proposed hypothesis to explain the origin of residual lattice strains in IMI.130, plastically deformed in unidirectional and uniaxial tension.
- 7.5. Effect of surface treatments on the residual macro- and micro-stresses in IMI 130 and macro-stresses only in IMI 318A.
- 7.6. Industrial importance of measuring residual stresses.

7.1. Experimental Methods.

The experimental methods are discussed in the following subsections:-

7.1.1. Surface preparation.

7.1.2. Film and diffractometer techniques for measuring macro-stresses.

7.1.3. Micro-stress measurements in IMI. 130 using a diffractometer

7.1.1. Surface Preparation.

7.1.1.1. The surface preparation of materials depends upon the purpose of the stress investigation. Often stresses are measured on ground, shot peened, milled, machined, heat treated or other surfaces which require no surface preparation prior to stress analysis. In such cases the stress gradient may be large and any material removal from the surface may alter the surface characteristics and hence obscure the true stress existing on the surface. On the other hand stress factor calibration procedures and sub-surface stress measurements require a method of preparing the surfaces which does not introduce stresses. Surface preparation techniques are generally required:-

- (i) to ensure that the surface layer examined is representative.
- (ii) to present a smooth surface to the incident beam.
- (iii) to remove layers for subsurface measurements with depth below the surface.

7.1.1.2. Stress measurement by X-ray method, involve diffraction from a surface layer only a few microns thick. Electropolishing has been considered by various workers to provide a fast stress free removal of metal surfaces, as compared to chemical milling which is a slow process of surface removal. Electropolishing of titanium has been shown to be unsatisfactory (20) because it results in changes in the diffraction pattern. Nevertheless removal of oxide film from titanium prior to stress analysis is an important factor.

7.1.1.3. For fast removal of material for applied stress measurements on both IMI.130 and IMI.318A a HNO_3/HF solution at temperature of $50-60^\circ\text{C}$ has been shown to be satisfactory to give a stress free surface. In order to prevent any stress relief resulting from layer removal for sub-surface measurements, the same solution is used at room temperature. Although the process is slow, nevertheless it ensures that material removal is uniform even after repeated polishing over a large area thus avoiding any stress concentration effect.

7.1.1.4. The experimental evidence showed that for measuring macro-stresses in titanium, by the film method, careful selection of material in the cross rolled condition is essential to minimise preferred orientation. However, this consideration is not necessary for measuring stresses with a diffractometer. Thus suitable selection of materials, coupled with careful surface preparation procedure, has made it possible to measure macro-stresses in IMI.130 and IMI.318A by the back reflection technique.

7.1.2. Film and Diffractometer Techniques for Measuring Macro-Stresses.

7.1.2.1. For measuring macro-stresses in materials which yield sharp X-ray diffraction lines with excellent peak to background ratio, the measurement by the film and diffractometer methods are equally good. For example, the macro-stresses measured in both IMI.130 and IMI.318A strips (Figs. 9, 20, 22 and 26) by the two methods give comparable results. The strips were deformed by four point loading in the elastic as well as plastic deformation ranges.

7.1.2.2. The diffractometers, however, have a distinct advantage over the film method for measuring macro-stresses, with greater accuracy from broadened and diffused diffraction lines. For example, it was impossible to measure macro-stresses by the film method in both materials IMI.130 or IMI. 318A which exhibited directionality, or when the specimen was subjected to various deformation modes, i.e. shot peening, machining or grinding. In such cases, line broadening or line shift can only be measured using diffractometers.

7.1.2.3. There is considerable saving of time for measuring macro-stresses in similar types of specimens, using the diffractometer compared to the film method. For example, macro-stresses in identical specimens can be measured in 1-2 hours using the diffractometer compared to 5-6 hours needed in the film method for exposing, developing and analysing 6 films for each stress measurement.

7.1.2.4. The data using the diffractometer is printed out on teletype machine, which can be fed into the computer programming for subsequent stress analysis compared to the film method where all the films have to be analysed manually and analysis by computer is not possible.

7.1.2.5. The conventional diffractometer, however, suffers from the distinct disadvantage that only small and flat specimens can be examined. For stress factor calibration for both IMI.130 and IMI.318A (Fig. 26), a special bending jig had to be designed for parafofocussing.

Similarly, for handling complicated shapes and different sizes, other special bending fixtures may be necessary. With the film method, there is no such limitation in that wide range of specimen sizes with convex or concave surfaces can be investigated.

7.1.2.6. With the conventional diffractometer there is a further limitation on the angular range 2θ over which macro-stresses can be measured. For accuracy in macro-stress measurement, reflections occurring at high Bragg angle (well above $130^\circ 2\theta$) are required to be analysed. The maximum usable angular range is up to $160^\circ 2\theta$ compared to the film method where there is no such limitation and reflections occurring above $160^\circ 2\theta$ can be investigated. For example for measuring macro-stresses in IMI.130 and IMI.318A, 213 reflections occurring at $139.4^\circ 2\theta$ angular position with Cu K α radiation could only be used with conventional diffractometers. On the other hand, with the film method, the 006 reflection occurring at $161.4^\circ 2\theta$ angular position were used for stress analysis in IMI.130.

7.1.2.7. With the film method, radiation can be changed with considerable ease compared to the diffractometer where considerable time is necessary for alignment purposes. For example, the effect of beam penetration, on surface stresses using different X-ray wave lengths (Figs. 14-18) was investigated with considerable ease using the film method. It is easier to detect other effects such as preferred orientation, grain size changes or radial streaking occurring probably due to oxygen adsorption in the lattice during heat treatment (details Appendix 7).

7.1.3. Micro-stress Measurements in IMI.130 using a Diffractometer.

7.1.3.1. The experimental evidence obtained by the X-ray back reflection technique shows that the K α doublet was resolvable in bent strips of IMI.130 and IMI.318A, both in the elastic and plastic deformation ranges. On the other hand a K α doublet could not be observed from shot peened or machined IMI.130/IMI.318A.

7.1.3.2. The results in Tables 12-15 and Figs. 33-35 and 38 show that shot peening, grinding, machining and vapour blasting of IMI.130 produced a high magnitude of micro-stresses with corresponding smaller crystallite size, thus resulting in broadening of the diffraction line profiles with long 'tails' consequently liable to be lost in the background.

7.1.3.3. The back reflection films obtained from rolled and annealed sheet material in IMI.130 and IMI.318A exhibiting directionality, showed that diffraction lines could not be observed. It was believed that directionality affected the intensity distribution of the line profiles used for determining macro-stresses. Further experimental evidence showed that

preferred orientation produced by various deformation modes affected the intensity distribution of line profiles. The details of the effect of preferred orientation on the intensity distribution are given in Appendix 8. From these diversified results it is believed that different deformation modes occur in the plastic deformation of IMI.130 and careful interpretation of data is necessary whilst measuring residual stresses in titanium.

7.1.3.4. Titanium has ^astrong affinity for oxygen making it difficult to obtain oxygen free titanium metal powders. During annealing treatment in vacuum, oxygen is adsorbed in the lattice producing distortion. It is therefore difficult to obtain stress free titanium powder necessary for the evaluation of instrumental correction for micro-stress analysis. These observations are in agreement with the experimental evidence of Huany (216), and Schoening and Witt (217) who have shown the influence of oxygen on the intensity of the diffraction lines and on lattice distortion.

7.1.3.5. Recourse was therefore made to use block titanium for obtaining instrumental broadening correction. Further, the use of block material was considered desirable to interpret the results of micro-stress analysis for practical applications.

7.1.3.6. For determining instrumental broadening IMI.130 cross rolled sheet material was used. Generally, H.C.P. metals give preferred orientation and it is difficult to remove preferred orientation texture by annealing or cross-rolling. The sheet material was, though, cross-rolled to give an equiaxed grain structure shown in Photo 4, but some preferred orientation still existed. Further experimental evidence showed that the intensity distribution of the various reflections varied in comparison with those recorded in the ASTM card index. This is to be expected since data recorded in the ASTM card index is obtained from powder material with complete random orientation compared to the block materials. This factor led to difficulty in correcting the observed broadening for instrumental effects. However, the variation of β^* and β^{*2} values obtained was within $\pm 5\%$ of the mean value. This is within the limits of accuracy in measuring micro-stresses and as such did not introduce serious errors.

7.1.3.7. The integral breadth method was used to measure residual micro-stresses in IMI.130 after taking into consideration intensity, overlapping reflections and the absence of multiple order of reflections. The results were analysed both by the Wagner and Aqua method (159) and the Hall and Williamson method (117) (see page 70). The former method was

used for the micro-stress results shown in Figs. 33-35 and 38 but results obtained by both the methods are shown in tables 12-15. The results obtained by the Wagner and Aqua method, for ground IMI.130 with down feed of 0.003 in/pass (Fig. 33 table 12) give micro-stress values of 25.4 tonf/in² compared with the 18.5 tonf/in² obtained by the Hall and Williamson method. The photomicrographs (Photos 7 and 8) show that grinding caused micro-cracking on the surface in spite of the presence of compressive macro-stresses. The experimental observations show that grinding might have resulted in micro-stress values well above the limit of proportionality (~ 23.0 tonf/in², table 1) determined by tensile testing, thus causing micro-cracking. Therefore the Wagner and Aqua method has been considered to give micro-stress values in better agreement with the other experimental evidence than the values given by the Hall and Williamson method. In order to obtain consistency in results, the best straight lines were drawn by the least square method using the computer.

7.2. Factors Affecting the Accuracy of Macro- and Micro-Stress Measurements.

7.2.1. Accuracy of Macro Stress Measurement.

7.2.1.1. The accuracy of macro-stress measurements using the diffractometer depends upon the following factors:-

- (i) Specimen preparation and surface roughness.
- (ii) Diffractometer alignment,
- (iii) Location of diffraction peaks,
- (iv) Stress factor,
- (v) Elastic Constants.

7.2.1.2. The effect of surface preparation prior to stress measurement has been discussed in the beginning of the discussion. The actual stress measurement depends to some extent on the degree of surface roughness. Taira and Arima (217) have shown that the results can be in error by 1 - 2%.

7.2.1.3. Diffractometer Alignment

The diffractometer alignment is carried out to ensure that the divergence slit, goniometer axis and the receiving slits are in line when $2\theta = 0$. The diffractometer alignment carried out, using silicon disc, for the 311 and 533 reflections at $56.1^\circ 2\theta$ and $136.9^\circ 2\theta$ respectively gave an accuracy of $\pm 0.005^\circ 2\theta$ of the true angular position.

Counter Track Alignment

The specimen is required to be positioned accurately so that it contains the line of the goniometer axis. Direct displacement from

this axis for a flat sample and direct displacement due to curvature of the sample will both introduce errors. Both sources of errors were carefully controlled whilst measuring macro-stresses in IMI.130 and 318A. The effect of curved surfaces could not be fully evaluated.

The counter track alignment is critical and serious errors can be introduced into stress measurements. For obtaining the best focal position, the receiving slits and counter ^{are} moved forward along the track. The effect of counter track movement for silicon disc for 533 reflection at $\Psi = 45^\circ$ is shown in figure 24(4). The distance AB by which the counter and receiving slits must be moved forward is given by the following relationship:-

$$AB = R - R \frac{\cos(\Psi + \Phi)}{\cos(\Psi - \Phi)} \quad \text{Details section 4.3.}$$

For IMI.130 and IMI.318A using 213 reflection and $\text{Cu K}\alpha$, the receiving slits and counter must be moved by 93mm from $R = 173$ mm (radius of goniometer circle). If the movement is less than the calculated distance, asymmetrical peaks similar to that shown in Fig. 24(c) for silicon disc, can occur with drop in intensity. On the other hand if the movement is slightly greater than the calculated distance, symmetrical peaks but with reduced intensity result. Therefore it is absolutely necessary that the receiving slits and counter are moved according to the calculated distance when the specimen is rotated through Ψ .

7.2.1.4. Location of Diffraction Peaks.

On the "two-exposure" technique, the 'three point parabola' method (19) of peak location is used. Kelly and Short (193) have pointed out that there will be statistical errors in the calculated diffraction angles due to random counting errors in the measurement of X-ray diffraction intensities. They have derived an equation giving the standard deviation in the residual stresses due to random counting errors, Kirk (188) has shown that the total error of precision for the quenched and tempered steel is $\sim 1 \text{ ton/in}^2$, this value is reduced to $\frac{1}{2} \text{ ton/in}^2$ for the annealed steel with a relatively sharp peak.

Additional errors can, however, result due to instability of X-ray source, and by variations in the efficiency of the monitoring system. Increase in precision cannot be achieved simply by timing very large number of counts as these additional errors may increase with time.

Further caution is needed in the peak location strategy using the three point parabola method. This may work very well when the peaks are symmetrical, such as quite broadened peaks occurring due to shot peening or in high strength materials (for example BS S 99, or maraging steels).

The technique is also suitable for annealed materials where the peaks are sharp. However, in between these conditions, there may be areas where the lines are not quite resolved and the peaks may be asymmetrical. An attempt to fit the parabola to asymmetrical peaks may shift the curve where the three points are taken. Kirk (188) has recommended that errors in peak location to the symmetrical data may be reduced by using two three point estimates. The first estimate gives the approximate peak position; this position is then used as the centre for the second estimate.

7.2.1.5. Stress Factor Calibration.

The results of stress factor calibration for IMI.130 and IMI.138 using the diffractometer are shown in Fig. 26. The stress factor can be determined experimentally or calculated theoretically and the accuracy of stress depends upon this factor. In order to preclude the influence of composition, structure or preferred orientation, it is desirable that calibration specimens should be in a similar metallurgical condition and be of the same chemical composition as that of the component.

For determining stress factor experimentally, the calibration specimen can be stressed as follows:-

- (i) By the four point loading device in fixed bending jig as shown in Fig. 2 or in a variable jig shown in photo 21.
- (ii) By direct tension devices: for applying stresses to reasonable size specimen, the tension devices have to be relatively massive and may not allow calibration to be extended into the compression region.
- (iii) By tapered cantilever beam suggested by French and MacDonald (190) which on bending exhibits uniform longitudinal stresses along the beam. This does not offer any particular advantage over the four point bending device and has the marked disadvantage of requiring the machining of a relatively complicated specimen. Therefore for stress factor calibration, the four point loading devices shown in Fig. 2 and photo 21 have been used.

The results of stress factor determined experimentally in the elastic range is generally applied to measure residual macro - stresses in finished components subjected to plastic flow resulting, for example, from machining or shot peening operations. The deformation characteristic of the material when bent in four point loading device, or pulled in uniaxial tension are different from those of machined or shot peened surfaces. On either side of a bent beam, the outerfibres are stressed predominantly in one direction, namely, parallel to the neutral axis. If the beam is

deformed plastically and then unloaded, the springback of the elastically strained interior throws the side which was in tension during bending into compression. However, the plastic flow in shot peened materials occurs in many directions at once and in addition the plastic flow varies with machining operations. Further plastic deformation cannot be entirely eliminated in structural parts undergoing service loadings, and thus it is important to know the extent to which plastic deformation affects residual stress measurements in these parts.

It is generally accepted that X-ray diffraction techniques measure only elastic macro-stresses.

Further the strength of the materials in the work hardened regions is considerably increased compared to the strength of the material. Therefore, the measured residual macro-stresses on the surface of work hardened surface would reflect the surface condition, rather than the actual material. For example, in shot peened IMI.130, results in Fig. 37 show that residual macro-stresses measured on the surface are 28 - 32 ton/in², compared to the limit of proportionality of 23.0 ton/in² determined in a tensile test Table 1. Further the results in Figs. 9 to 13 show that the X-ray limit of proportionality is only 13 - 15 tonf/in². Thus it is believed that higher residual macro-stresses measured in shot peened material must be associated with higher tensile strength and yield strength of the surface layers. It must be remembered that X-ray diffraction techniques measure surface stresses up to only few microns below the surface depending upon the radiation used. Apparently, the use of stress factor determined experimentally using unidirectional or uniaxial tension, in the elastic range, may lead to errors in the evaluation of stresses in plastically deformed materials. Thus, for correct evaluation of results in plastically deformed materials, it is desirable to determine stress factors using specimen having different types of plastic strain.

The component of stress σ_ϕ is related to the angular position of the diffracted beam by the following equation:-

$$\sigma_\phi = (2\theta_\perp - 2\theta_\psi) \frac{\cot \theta}{2} \cdot \frac{E}{1 + \nu} \cdot \frac{1}{\sin^2 \psi} \text{ where } K = \frac{\cot \theta}{2} \cdot \frac{E}{1 + \nu} \cdot \frac{1}{\sin^2 \psi}$$

The constant K is the stress factor. It is generally assumed that $\cot \theta$ term is constant but this assumption may induce serious errors in each stress calculations where the original peak has shifted due to deformation on straining of the specimen. This approximation may induce little error when the stress level is low, up to 20 tonf/in² but, at higher stress level range the errors introduced may be considerable. For example

when measuring macro-stresses in IMI.130 using 213 reflection at 139.4° 2θ angular position, the maximum peak shift of 0.5° Table 9 will induce a smaller error compared to IMI.318A a high strength material. In this case for a maximum peak shift of 1.6° 2θ (Table 10) the error will increase three times. This is, however, the case for specimen deformed in four point bending devices but is shot peened material where a peak shift of 1.77° has been observed, the error will further increase.

The errors can possibly be reduced by making the change in peak location as large as possible for a given stress level. This can be done by making the angle of rotation ψ as large as possible thereby reducing the term $\frac{1}{\sin^2 \psi}$ or by selecting 2θ so large as to reduce the $\cot \theta$ term. There are, however, practical limitations in that 160.0° 2θ and $\psi = 60^\circ$ are considered to be the maximum usable variables using the conventional diffractometers. The alternative method may be to convert the peak 2θ to d and use the current expression that stress is proportional to Δd .

For measuring macro-stresses by the X-ray back reflection technique, the following expression is used:-

$$\text{Cosec } \theta = \frac{\sigma (1 + \nu) \sin^2 \psi}{E \sin \theta_{\perp}} + \frac{1}{\sin \theta_{\perp}}$$

In this expression the stress factor $K = \frac{E \sin \theta_{\perp}}{\sin^2 \psi}$ calculated theoretically and similar errors involving the Bragg angle, as discussed in the preceeding paragraphs using diffractometer may arise.

In the film method, if $K \alpha$ doublet is resolvable, thus serious errors do not occur. For example, if the vernier readings are in error by 1 mm or 2 mm the errors in final stress value will be within the scatter of points on the $\text{Cosec } \theta / \sin^2 \psi$ graph. For measuring macro-stresses both in the elastic and the plastic deformation ranges, the $K \alpha$ doublet was resolvable in both the materials IMI.130 and IMI.318A, thus no serious errors occurred in the macro-stress results. The errors would, however, increase when the diffraction lines become broadened and diffused. In such cases the $K \alpha$ doublet is not resolvable and scatter in macro-stress results increases with increase in the level of residual macro-stresses. The following limits for high strength materials such as BS.99 or maraging steels are not uncommon:-

<u>Surface Residual Macro-Stress</u>	<u>Limits</u>
10.0 tonf/in ²	± 1.0 tonf/in ²
10.0 to 30.0 tonf/in ²	$\pm 2 - 3.0$ tonf/in ²
30.0 to 50.0 tonf/in ²	$\pm 4.0 - 5.0$ tonf/in ²
From 50.0 tonf/in ²	$\pm 7 - 8$ tonf/in ²
upwards.	

Further errors can arise due to film shrinkage resulting from temperature changes. However, with the use of calibration powder, all the films are standardised and allowance is made for the film shrinkage.

7.2.1.6. Elastic Constants.

The accuracy of stress measurements ultimately depends upon the stress factors used in stress analysis. For measuring stresses by the X-ray back reflection techniques, in both IMI.130 and IMI.318A, the stress factor is calculated theoretically. For measuring stresses using a diffractometer the stress factor for both materials is determined experimentally. The Young's modulus and Poisson's ratio which enter into the stress analysis (Theory given in Appendix 2) are the fundamental properties of the material and are sensitive to preferred orientation, composition and structure. Materials show considerable elastic anisotropy with respect to crystallographic directions. The measured strain, which corresponds to one particular direction, may not be accurately related to stress by mechanically measured values of bulk elastic constants. The alpha phase (H.C.P. structure) in titanium/titanium alloys is anisotropic and as such the value of Young's modulus in the range $14-17 \times 10^6$ lbf/in² in the longitudinal as well as transverse directions for both IMI.130 and IMI.318A are ~~typical~~. The X-ray values of Young's modulus determined by the two exposure methods for only 213 reflections in IMI.130 and IMI.318A are comparable to those determined mechanically in tensile testing (Tables 1 and 2). Therefore, bulk values of elastic constants have been used in stress analysis of IMI.130 and IMI.318A without introducing any serious errors in the stress results.

7.2.2. Accuracy of Micro-Stress Measurement.

It is not possible to quote the limit of accuracy of micro-stress values since it is difficult to check and compare with any other standard method. Using the integral breadth method for micro-stress measurement, Lindley (222), Northwood (223) and Pearce (224) have quoted the reproducibility of results within $\pm 5\%$.

In this investigation, using integral breadth method, the peak area was checked by manual counting and the Planimeter and agreement of area was within $\pm 1\%$. For improved accuracy and consistency in results, the best straight lines were drawn by the least square method using computational facilities. This method, however, does not allow, due weighting to more important points on the graph, nevertheless, the method is reliable and consistent.

The errors in the measurement of integral breadth increase with higher Bragg angle diffraction peaks. Further, with increase in plastic deformation the peak intensity drops and the broadened line profiles tend to merge with

the background, thus making it difficult to select the correct background level. This results in overestimation of the peak area and consequently overestimation of peak height and vice versa. The errors thus cancel out (224) and estimated errors are within 4-6% for plastically deformed specimens.

The intensity distribution of various reflection varies with the mode of plastic deformation and with the block material used. This factor leads to difficulty in correcting the observed broadening for the instrumental effects. The variation of β^* and $(\beta^*)^2$ values obtained was within $\pm 5\%$ of the mean value. This is within the limits of accuracy in measuring micro-stresses and as such no serious errors have occurred in quoting the actual level of micro-stress values.

As pointed out by Warren (154), the presence of stacking faults in H.C.P. metals results in the modification of line broadening. The effect of stacking faults in titanium has not been investigated and is required to be investigated

7.3. Effect of Deformation in Unidirectional and Uniaxial Tension on the Applied and Residual Stresses in IMI.130 and IMI.318A.

7.3.1. The experimental results in Figs. 9 - 13, 20, 22 show that in the elastic range, the rate of increase in the lattice strain closely follows the total applied strain. With the onset of plastic deformation, the rate of increase in lattice strains, however, decreases. The results obtained are similar on both the tension and compression faces of the strips for both the materials using Co K α radiation. Further, for IMI.130 the results in Figs. 10 - 13 show that macro-stresses are similar on both compression and tension faces of the strip using three radiations ~~Co~~ K α , Co K α and Cu K α for 004, 114 and 006 reflections respectively. The important observation is that the residual or applied surface macro-stress values were obtained in the elastic as well as plastic deformation regions whilst K α_1 , α_2 doublet remained resolved. This implied that there can be no doubt that the stresses measured were not due to any systematic errors in measuring the diffraction lines. Further the similarity of results on the compression and tension faces indicate that errors are also not due to the bending of the test strips.

7.3.2. The results in Figs. 9 and 22 also show that residual stresses in both materials do not develop until the X-ray limit of proportionality is exceeded. In IMI.130, there is a difference between the X-ray and tensile limits of proportionality, the former being two thirds of the latter. In IMI.318A there is also a difference between the two limits of proportionality, the X-ray limit of proportionality being four fifths of the tensile limit of

proportionality. Further plastic deformation occurs at a similar stress level in the alpha and the beta phase. In the plastic deformation range, the increase in lattice strain per unit stress is less than that in the elastic range for both the materials; further increase is greater than in the elastic range.

7.3.3. The observations regarding the limits of proportionality and development of residual lattice strains are in partial agreement with the experimental evidence of Glocker and Hasenmaier (53) who have reported that for 211 reflections in mild steel strain ceases to be proportional to the applied stress at about three-fourths of the tensile limit of proportionality. These findings are, however, directly contrary to the experimental results of Smith and Wood (58-60, 64), Finch (65) and Greenough (61) who have observed that a non-proportional limit commences at the tensile limit of proportionality.

7.3.4. The observed difference between the X-ray and tensile limits of proportionality may be due to the selective nature of the X-rays. The macro-stresses are measured in a given direction for only those grains in the polycrystalline aggregate, which are so orientated as to contribute to the particular X-ray reflection examined. Further, the grains that reflect, have only certain orientation with respect to the stress axes and the effective value of elastic constants can differ from those of the overall orientation, as measured in a mechanical test. Titanium is considerably more anisotropic than steel or aluminium, and the elastic constants may be altered by heat treatment and other processing variables. In comparison, in the mechanical tests, elongation is measured over a larger volume of material and local surface yielding response may not be picked up by the extensometers used.

7.3.5. Further, the macro-stresses in IMI.130 were measured by the X-ray back reflection technique using three radiations with different wave lengths for the following reasons:-

- (i) to investigate the effect of beam penetration on stress results in the elastic and plastic deformation ranges;
- (ii) to investigate the surface, i.e. yield point anisotropy of the surface and sub-surface crystallites;
- (iii) surface hardening effect.

7.3.6. The experimental results shown in Figs. 14-18 for IMI.130 regarding the effect of beam penetration are in partial agreement with the observations of Hauk (149). He observed that in a bent beam of steel, the stresses measured with Cr K α radiation were less by 35% than those measured

with Co K α radiation. Hawk interpreted his results in terms of the difference in penetrating power of the two radiations. On the other hand Glocker and Hasenmeier (53) investigating the stresses in plastically deformed steel specimen whilst load was still being applied, attributed the difference in stresses to the following two factors:-

- (i) difference in penetrative power of the radiation.
- (ii) surface effect in that surface ~~crystallites~~ may have a lower yield stress than the crystallites in the interior.

7.3.7. The results for IMI.130 shown in Figs. 16, 17 reveal that with a smaller amount of plastic deformation, the difference in surface macro-stresses obtained with three radiations Cu K α , Co K α and Cr K α using 006, 114, 004 reflections is not appreciable but with a larger amount of plastic deformation (Fig. 18), the difference in macro-stresses obtained from 3 reflections is discernible. From the results it may be inferred that large stress gradients develop in heavily deformed titanium. The depth of penetration of the 3 radiations used for stress analysis of titanium is given below:-

Radiation K α	Wave Length λ Å	Depth of Penetration in titanium - 0.001 mm
Cu	1.54051	3.8
Co	1.78892	2.5
Cr	2.28962	1.4

7.3.8. The beam penetration of Cr K α is in the extreme surface layers, and as such, the stresses measured are only representative of the surface condition. On the other hand beam penetration ^{of} Cu K α radiation is greater than that of Cr K α radiation and therefore the stresses measured are the weighted average of surface and sub-surface stresses. The surface macro-stresses measured with Cr K α radiation are approximately 50% less than those measured with Cu K α radiation (Fig. 18) whilst results obtained with Co K α radiation are in between these values commensurate with the difference in depth of beam penetration. These observations support the evidence obtained by Glocker and Hasenmeier (53) in that crystallites in the surface layers may have a lower yield stress than those in the interior, hence low stress values measured with Cr K α radiation than Cu K α or Co K α radiations.

7.3.9. In X-ray stress analysis it is assumed that the effective beam penetration is so small that the stress component in direction normal to the surface is zero and consequently it does not affect the stress determination. This assumption may be valid if the stress gradient below the surface is small. However, if the stress gradient is large, as can be

expected with heavy plastic deformation in uniaxial or unidirectional, shot peened, ground, vapour blasted or machined surfaces it may be necessary to take into account the effect of beam penetration and apply the necessary correction.

7.3.10. The macro-stresses measured with Co K α radiation and Ag calibration powder has shown good correlation with the stresses measured by strain gauges and calculated by the bending theory (Figs. 8, 9, 19-22). It was for this reason that Co K α radiation was used for the stress analysis of IMI.130 using the X-ray back reflection technique. Further Co K α radiation gave a high Bragg angle peak of sufficient intensity for measuring stresses with greater accuracy in both IMI.130 and IMI.318A using 114 reflection. This is important to make direct comparison between the materials without errors arising from preferred orientation.

7.3.11. The experimental results shown in Figs. 9 and 22 indicate that residual macro-stresses measured in both IMI.130 and IMI.318A are intimately connected with the onset of plastic deformation. In both materials the residual macro-stresses have increased with increase in plastic strain. Further the shape of the curves for residual macro-stresses are similar on the tension and compression faces but the level of residual macro-stresses in tension measured in the unconstrained condition on the compression face of the strip are of higher magnitude than compression stresses measured on the opposite face (Fig. 22). Also, for any level of plastic strain, the residual stresses measured in beta phase are of higher magnitude than the alpha phase. The onset of plastic deformation has occurred at a similar stress in both the alpha and beta phases.

7.4. Proposed Hypothesis to Explain the Origin of Residual Lattice Strains in IMI.130 Plastically Deformed in Uni-directional and Uniaxial Tension.

7.4.1. In this section, current theories regarding the origin of residual lattice strains and specific observations regarding the effects of plastic deformation in uniaxial tension, are discussed in light of experimental results obtained from IMI.130. A hypothesis is proposed to explain the origin of residual lattice strains in plastically deformed IMI.130.

7.4.2. Generally, polycrystalline materials deformed plastically in uniaxial tension show a line shift indicating the existence of residual lattice strains but these residual lattice strains are not detected by the mechanical methods. However, when residual stresses are induced by shot peening, grinding or machining, good agreement has been obtained (19,93)

between X-ray diffraction and mechanical methods. The residual lattice strains do exist after uniaxial or unidirectional plastic deformation but considerable speculation exists as to the origin and distribution of these strains.

7.4.3. In order to explain the origin of residual stresses in IMI.130, the following experimental evidence has been obtained.

- (i) surface residual macro-stresses have been measured after deforming tensile test pieces for different plastic strains in uniaxial tension (Fig. 31). The residual macro-stresses have been measured both in the longitudinal and transverse directions of pulling the test pieces;
- (ii) Changes in residual macro-and micro-stresses have been observed after progressive thinning of plastically deformed specimen (Fig. 32);
- (iii) Applied stresses have been measured using $\text{Cu K}\alpha$, $\text{Co K}\alpha$ and $\text{Cr K}\alpha$.

7.4.4. The experimental results in Figs. 9 and 22 show that the origin of residual macro-stresses is intimately connected with the limit of proportionality between the lattice and applied stress in the region above yield stress. The residual macro-stresses have not been observed in both materials IMI.130 and IMI.318A until the X-ray limit of proportionality has been exceeded. It is possible that the proportionality between lattice strain and applied stress ceases as soon as some dislocation movement is caused by applied stress and this may be the region into which a specimen may be required to be strained before residual stresses are observed.

7.4.5. Probable Cause of Residual Lattice Strains

7.4.5.1. The Deformation of the Metal Lattice.

Elastic deformation which completely disappears on removal of the externally applied stress to the crystal is caused by the change in inter-planar spacing. It occurs in a manner similar to that in which the external dimensions change. Since the atoms have left their equilibrium positions, there are resulting forces acting on them and these forces balance the external stress applied to the lattice. Since this type of deformation causes changes in the interplanar spacing and is measured by X-ray diffraction, therefore the residual strains should be elastic in character. Residual lattice strains caused by stresses should vary in sign if these are measured in various directions relative to the crystal.

Plastic deformation is caused by glide in the crystal. Part of the crystal glides as a whole relative to the remainder across a plane

of specific crystallographic orientation. The distance it moves is an integral multiple of the interatomic spacing in the direction of glide. Since in the final position each atom has been replaced by another, and all atoms are identical, it would be impossible to detect this deformation by X-ray diffraction methods if it were not accompanied by other phenomena. After heavy plastic deformation (154, 190) each grain in the aggregate is composed of particles with misorientations between the neighbours of a few degrees, and with a non-uniform distribution of dislocations. Within particles local twist and curvature result in strains which are uniform only over distances of the order of $100A^0$ and which increase in magnitude as the boundaries approach. The boundaries between the particles are not considered to be narrow discontinuities in the lattice but wider volumes in which concentrated dislocations pile up associated with intersecting slip bands or walls of screw dislocations form the highly distorted regions. In commercially pure titanium, the uniaxial deformation is by slip and twinning and certain twins (155) contain bands of high dislocation density near the twin boundaries. The dislocations are ^{associated} with severely jogged dislocations and occasional stacking faults. The dislocation density increases with increase in plastic deformation. After $\sim 25\%$ plastic strain a well developed polygonized structure is developed.

The presence of many regions of disorder could cause general expansion of the lattice and such an effect would also produce residual lattice strains.

7.4.5.2. General Expansion of the Lattice due to Plastic Deformation.

The experimental results in Fig. 26 show that, in the elastic range, the interplanar spacing contract linearly with increase in applied stress but, at the X-ray limit of proportionality, the interplanar spacing undergo an abrupt expansion. It is known (155) that in titanium, the dislocation density increases with increase in plastic strain and eventually at $\sim 25\%$ plastic strain, well developed polygonized structure is developed. The accumulation of dislocations resulting from plastic deformation would result in the displacement of atoms from their equilibrium positions. Further, assuming that polygonization due to plastic straining in uni-directional or uniaxial tension is analogous to heating a cold worked structure where lattice expansion would be expected, it is likely that plastic deformation beyond the limit of proportionality would cause general expansion of the lattice.

Weaver and Pfarr (41) also observed marked contraction of the lattice when a test piece from rolled sheet was held in tension but when the test piece was stretched beyond yield point the lattice expanded. These observations have been confirmed by Bollenrath et al (57), Smith and Wood (58-60) and Hauk (149). Although the plastic deformation may produce expansion of the lattice but this may not be the sole cause for the existence of residual lattice strains, but strains may also be caused by some form of locked up stress system in the lattice.

7.4.5.3. The Form of the Locked Up Stresses

7.4.5.3.1. For the locked up stress system several possible explanations have been offered. Since the metal aggregate as a whole is in equilibrium and is under no externally applied stress, these postulated residual lattice strains must be opposed by a residual stress of opposite sign in some part of the aggregate which is not contributing to the diffracted X-ray beam. These may be called Part A and part B.

7.4.5.3.2. Assuming that different parts of the aggregate have different yield stresses, i.e. the part A yields under a lower applied stress than part B. After the application of tensile stresses to deform the whole aggregate, the elastic strain in A will be less than in B. When the applied stress is removed, B will tend to contract further than A but will be prevented from so doing by the restraining influence of A. The final equilibrium state will be one in which A is in compression and B in tension to balance the forces. There is general agreement with this argument but considerable controversy exists as to the exact nature of the parts A and B. Two views are held; one leads to residual micro-stresses and the other to residual macro-stresses and it is believed that the superimposition of residual macro- and micro-stresses occurs frequently. Numerous theories to account for the contributory causes have been put forward from time to time i.e. (a) surface effects, (b) hardening effect, (c) orientation effect, (d) coherent area effect and (e) heterogeneity effect in both homogenous and heterogeneous materials but no clear cut explanation has yet emerged.

7.4.5.3.3. (a) Surface Effects.

The theory of surface effects (53, 56, 57) is based upon the fact that surface crystallites have a lower yield stress than the crystallites in the interior because these are less constrained. This should lead to surface macro-stress system which should disappear as the specimen is thinned by chemical milling. The experimental results in Fig. 32 show that this may not be the case. On the other hand, surface stresses

measured with Cr K α radiation which has a longer wave length and smaller depth of beam penetration, give lower surface macro-stresses compared to the stresses measured with Cu K α radiation which has a shorter wave length and deeper beam penetration (Figs. 10 - 13). Therefore, surface effects may be partially responsible for residual lattice strains but some other explanation may be sought to account for the residual lattice strains.

7.4.5.3.4. (b) Surface Hardening

The theory of surface hardening (67, 68) implies that in the plastic range the surface layers will harden less rapidly than the interior and the magnitude of stress should increase with increase in plastic strain. The experimental results in Fig. 31 show that the surface stresses in the longitudinal direction increase with increase in plastic strain. Further the macro-stresses measured with Cr K α , Co K α and Cu K α radiations (Figs. 10-13) indicate that in the subsurface regions about a few microns below the surface, the macro-stresses are of a higher magnitude. Therefore it is possible that in IMI.130, the theory of surface hardening may account for residual lattice strain. However, results in Fig. 32 show that the compressive stresses, decrease with depth below surface and then virtually stay constant. The presence of an approximately constant stress after removal of a few thousandths of an inch suggests that some process in addition to the hardening effective may be operative.

7.4.5.3.5. (c) Orientation Effect.

In this theory it is assumed that an intergranular stress system is established in plastically deformed aggregates due to yield point and work hardening anisotropy. The selective nature of the X-rays reveals the mean lattice strains of a specially orientated group of crystallites only (10-69 - 72).

A weakness of this hypothesis is that no experimental evidence concerning the measurement of micro-stresses has been put forward and the analysis is only on a qualitative basis. The experimental results in Fig. 32 show that the residual macro- and micro- stresses occur in association of each other in IMI.130, after plastic deformation in uni-axial tension, thus the importance of these stresses is emphasised. Further in this hypothesis it is assumed that the development of a compressive residual stress in the surface may be due to the difference of the yield stress of grains with a free surface and of those which are in the interior of a polycrystal. If this assumption is true then residual stress should

be measured immediately after passing the yield point and should not increase with increasing deformation but rather remain constant. The experimental results in Fig. 31, however, show that stress increases with increase in plastic deformation. Further the intergranular stress system in any case is dependent on its neighbours and statistically all arrangements are possible, and various groups taken together will exhibit the whole range of stresses from tension to compression with zero mean stress (26). This hypothesis has been considered and rejected by other investigators (68, 67, 87).

The experimental evidence obtained in Figs. 31, 32 may partially support this hypothesis in that it is likely that such an effect may be occurring but in addition other factors may be operating.

7.4.5.3.6. (d) Coherent Area Effect.

Regardless of the sign of the residual stress which is detected by X-rays from coherently diffracting regions and as such from the bulk of the material, a balancing stress must be present in some other noncoherently diffracting region. According to the coherent area theory (75 - 77) a system of orientated micro-stresses, localised within regions of coherent scattering, appears after uniaxial plastic deformation. All centres of coherent scattering are considered to be soft regions and hard regions are represented by grain boundaries, sub-boundaries or highly distorted regions. Further, the weak regions comprise most of the material with the result that X-rays measure stresses only in these regions.

The 'coherent area' theory may possibly explain the existence of residual lattice strains and their detection by X-rays to a certain extent, but it may not be considered to be the predominant operative mechanism. It is likely that residual lattice strains develop in IMI.130 after plastic extension develop due to a variety of causes and no single mechanism can account for these strains.

7.4.6. None of the theories, for example, taken individually, can explain the presence of residual lattice strain so intimately connected with the onset of plastic deformation commencing at the X-ray limit of proportionality in IMI.130 (Figs. 9, 22) or lattice contraction and expansion (Fig. 26). The existence of stress gradient in the surface regions of heavily deformed IMI.130 strip and measured using three radiations (Fig. 18) is also not taken into account. Further, the increase in macro-stresses in the longitudinal direction with increase in

plastic strain (Fig. 31) nor the association and level of macro- and micro-stresses in the surface and sub-surface regions is considered.

7.4.6.1. In the light of experimental results obtained for IMI. 130 (Figs. 9, 22, 18, 31, 32) and inter-related specific observations in plastically deformed commercially pure titanium using electric microscope (155), a hypothesis is proposed for the presence of residual lattice strains in plastically deformed IMI.130 in unidirectional or uniaxial tension.

7.4.7. Proposed Hypothesis.

7.4.7.1. It is believed that the residual lattice strains are elastic in nature. The residual lattice strains in IMI.130 do not occur unless the X-ray limit of proportionality is exceeded and their existence may be due to the presence of weak and strong regions. The origin of residual lattice strains may be explained by a combination of the various theories discussed above. The hard regions in IMI.130 occur at the twin interfaces, low angle and developed sub-structure boundaries and other regions associated with dipoles and severely jogged dislocations observed by Partridge. The crack initiation at the twin interfaces in ground IMI. 130 is shown in Photos 7 and 8 and it is expected that in IMI 130 deformed in unidirectional or uniaxial tension, a heavy density of dislocations forming highly distorted regions would occur at the twin interfaces, low angle or sub-structure boundaries. The weak regions comprise most of the material with centres of coherent scattering. There will then be gradual transition towards highly distorted region, where the profiles with long 'tails' tend to merge with the background. It must be remembered that X-rays reflect selectively from grains in the correct Bragg position. Also, near perfect crystals diffract a higher proportion of the diffracted energy near the profiles maxima than do less perfect crystals; the latter give profiles with long tails which tend to become lost in the background. Thus X-rays give a weighted average in favour of the most perfect grains, which will tend to be in a state of macro-stress. The micro-stresses will occur simultaneously and are measurable by the line broadening techniques rather than the line shift techniques used for measuring the macro-stresses. Further, where compressive residual macro-stresses are shown by the X-rays, tensile stresses must also be present somewhere in the specimen to balance the forces. Thus the location of the balancing tensile stresses may vary, but must be associated with the plastically deformed distorted regions, and may diffract with lower intensity.

7.4.7.2. It is concluded that all experimental data concerning the residual lattice strains shown in Figs. 9, 22, 18 and 31 may be explained by the yield point anisotropy of the crystallites of the polycrystals, and due to yield point difference, between the surface parts and the interior parts of the specimen. The results in Fig. 26 agree with the assumption of an overall contraction of the lattice in the elastic region and expansion of the lattice when the X-ray limit of proportionality is exceeded, and may partially account for the origin of residual lattice strains. Further, in the plastically deformed IMI.130, the superimposition of macro- and micro-stresses occurs, thus giving rise to soft and hard regions. The stresses in the soft regions are balanced by the stresses in hard regions and X-ray measurements give a weighted average rather than the absolute value in favour of the weak regions, which are in compression and comprise most of the material [Fig 32].

7.5. Effect of Surface Treatments on the Residual Macro- and Micro-Stresses in IMI.130 and Macro-Stresses only in IMI 318A.

7.5.1. The surface roughness and surface integrity are two main aspects of surface quality arising from machining operations. Machining introduces a wide variety of surface layer alterations which include: overheating of the surface, cracks, tears and laps, residual stresses or distortion. These alterations result due to:-

- (i) high temperatures or temperature gradients produced in the machining operation.
- (ii) plastic deformation.
- (iii) chemical reaction and absorption of products of chemical reaction into the work surface.

7.5.2. The undesirable surface alterations resulting from abusive machining and grinding all have a pronounced effect on the potential strength of components. The residual macro-stresses thus introduced into the surfaces, may be tensile or compressive, high or low, and shallow or deep. The residual stress at the surface may be small and completely different than the subsurface maximum residual macro-stress. It is therefore necessary to know the residual stress distribution below the surface. There are two significant features indicated by the stress distribution curves, firstly the integrated area under the curve and secondly the maximum subsurface stress, which may be either tensile or compressive. The integrated area under a curve is indicative of the total stress in the surface layers that tends to produce distortion

whilst maximum value of sub-surface stress may influence the dynamic properties of the component. The depth of stressed layer is of considerable importance since it determines the degree of penetration of the surface disturbance and gives an indication of how much material is required to be removed from the surface to eliminate any surface effects.

7.5.3. After machining, the components are further processed for two reasons:

- (i) to improve the static and dynamic properties;
- (ii) to protect against corrosion.

In order to improve the dynamic properties of the machined surface, compressive surface and subsurface layers are imparted by shot peening, tumbling, burnishing, vapour blasting or ballising, whilst components are painted, plated or electroplated to inhibit corrosive attack.

7.5.4. It has been shown (180) that titanium alloys can be ground with precautions and at lower than normal speeds, to give low residual stresses. Moderately light down feed rates must be used and frequent wheel dressing carried out to obviate wheel loading. The loading problem is due to the low thermal conductivity of titanium leading to high surface temperatures, coupled with the readiness with which the surface galls. Further, due to strong affinity of titanium for oxygen, the metal surface is prone to atmospheric contamination during grinding if extremely high temperatures are generated. Absorption of oxygen at the surface can result in the formation of a thin oxygen rich layer of stabilised alpha titanium, which is hard and brittle. The micro-structure of good and abusively ground Hylite 51 titanium alloy, (4Al-4Sn-4Mo-0.5 Si, 90 Tonf/in²) are shown in Photos 1 and 2. For comparison, the micro-structure of turned Hylite 51 is shown in Photo 3. In the abusively ground specimen considerable flow (Photo 2) in the grinding direction has occurred; further, an extremely brittle skin layer associated with micro-cracks is present.

7.5.5. Surface/Subsurface examination of IMI.130

7.5.5.1. The surface damage due to grinding of IMI.130 with variations in down feed/pass are shown in Photos 9-14. The grinding variables used are given in Appendix 6. The metallographic examination (Photos 7-8) shows that titanium ground with a down feed 0.003 in/pass has flowed in the direction of grinding. Further, the deformation is associated with twinning and micro-cracks at the twin interfaces. The twin interfaces are probably associated with dipoles and severely jogged

dislocations as observed by Partridge (155). Apparently the plastic relaxation was not sufficient and therefore cracks appeared at twin interfaces associated with high dislocation density. Electron micrographs in Photo 19 shows the dislocation configuration in ground titanium with a down feed 0.0005 in/pass.

7.5.5.2. Similarly, the metallographic evidence obtained from shot peened IMI.130 is shown in Photo 6. The deformation is associated with twinning and the twinning density has decreased with depth below the surface. The electron micrographs (Photos 17 and 18) show the high density of dislocations and initial stages of sub-structure formation due to shot peening.

7.5.5.3. The evidence obtained from electro-micrographs is, however, inconclusive and more experimental work is needed to study the behaviour of these surface parameters. Nevertheless, the high dislocation density observed even with a moderate down feed 0.0005 in/pass suggests that titanium should be ground with caution.

7.5.6. Surface/Subsurface Residual Macro- and Micro-Stresses.

7.5.6.1. The results of residual macro- and micro-stresses produced by grinding and shot peening of IMI.130 on the surface and the distribution with depth below the surface are shown in Tables 12-15 and Figs. 33-37. The results show that the residual macro-stresses always occur in association with the residual micro-stresses, hence the importance of measuring both is emphasised. Further, the macro-and micro-stresses occur simultaneously in the surface regions and vary in a similar way in the subsurface regions, thus demonstrating their association.

7.5.6.2. Grinding.

The variation of surface macro- and micro-stresses in ground IMI.130 (Fig. 33) as a function of down feed rate per pass, may be associated with the severity of grinding and associated thermal effects, i.e. surface temperatures, temperature gradients coupled with low thermal conductivity of titanium.

Initially, the material ground with a down feed 0.0005 in/pass was compressively stressed due to plastic deformation and work hardening of the surface fibres, thus producing compressive macro-stresses and micro-stresses of lower magnitude. With change in down feed to 0.001 in/pass, the surface layers were further compressed resulting in increase of both surface compressive macro-stresses and micro-stresses due to the increased level of plastic deformation and work hardening of the surface layers. With further

increase in down feed to 0.002 in/pass, the increase in plastic deformation and work hardening was probably associated with high surface temperatures and may have resulted in surface stress relief; thus the magnitude of compressive macro-stresses and micro-stresses decreased. Close correlation between the compressive macro-stresses and micro-stresses is to be noted, thus the micro-stresses increase as the compressive macro-stress increases, and decrease as the compressive macro-stress decreases. Further, increase in down feed to 0.003 in/pass resulted in abusive grinding due to overheating and still further increase in plastic deformation and work hardening. The stereoscan micro photographs (Photos 15 and 16) show that the surface is considerably damaged. The metallographic examination (Photos 7 and 8) shows that cracking has occurred at the twin interfaces and the material has flowed in the direction of grinding. During grinding the material may have been heavily compressed resulting in high compressive macro-stresses and micro-stresses compared to those resulting from a down feed of 0.002 in/pass. The compression of the surface layers probably had the overriding effect and surface temperatures might not have been high enough to cause sufficient surface stress relief. Further the grinding might have resulted in fairly higher level of micro-stresses which may have been well in excess of the yield strength of the material, thus causing the occurrence of micro-cracks at areas of heavy dislocation density at the twin interfaces.

It is believed generally that surface compressive macro-stresses are beneficial and prevent crack initiation and inhibit crack propagation. Nevertheless micro-cracking has occurred (Photos 7 and 8) in spite of the presence of beneficial compressive macro-stresses. It therefore implies that high levels of micro-stresses play a vital role in the surface integrity of ground titanium. Further, the similarity between the residual macro-stresses measured in the grinding direction and orthogonal to the grinding direction is to be noted.

The actual surface stress pattern, however, is not indicative of the overall stress pattern within the surface layers, since the depth of penetration and the level and sign of the induced residual stresses may vary with the severity of the grinding condition. The residual sub-surface macro- and micro-stresses distribution with down feed of 0.001 in/pass are shown in Figs. 34-36. The correlation between macro-stresses and micro-stresses is again to be seen.

7.5.6.3. Machining (Turning and Face End Milling).

Generally these machining processes develop compressive macro-stresses in the major working direction of the cutting zone. The magnitude of the stress is a function of the cutting forces involved and the mechanical properties of the material being machined. Machining variables, which may affect the stress distribution, are cutting tool sharpness, cutting speed, feed rate, depth of cut, tool geometry and cutting fluid.

The surface residual stresses produced by face end milling and turning of IMI.130 are shown below:-

Process	Stress tonf/in ²	
	Macro-stress	Micro-stress
Face end milling	-15.3	22.0
Turning	-11.6	14.2

These machining processes have developed surface compressive macro-stresses and micro-stresses. The association between the two types of stresses is again to be seen. Face end milling has produced a higher magnitude of macro-stresses and micro-stresses compared with turning. Although surface macro-stresses resulting from milling are compressive, the level of micro-stresses is virtually approaching the yield strength of IMI.130. It is possible that a situation similar to that brought about by grinding with a down feed of 0.003 in/pass may arise, and therefore it is important that the optimum machining conditions should be used for face end milling of titanium.

7.5.6.4. Shot Peening.

The surface residual stresses induced by shot peening IMI.130 are shown in Figs. 34, 35 and 37. The correlation between compressive macro-stresses and micro-stresses is again to be noted. As nearly all fatigue and stress corrosion failures emanate from a focal point on the surface, such as a crack or tear, the compressive residual stresses induced by shot peening greatly improves the fatigue life and resistance to stress corrosion cracking, because cracks will not readily propagate into a compressed layer. The effectiveness of the process depends upon the uniform distribution of the compressive stresses coupled with optimum peening intensity. The beneficial effects of shot peening can be negated by overpeening (226). Compensating tensile stresses always exist below the compressively stressed layer. If the part is thin and the peened surface thick, the centre may be stressed in tension beyond its yield point, and this may affect the static (27) and dynamic properties. Overpeening may

also cause sufficient cold work to induce surface softening. Both surface compressive macro- and micro-stresses in IMI 130 have increased up to a peening intensity of 0.014A2 but tended to decrease slightly with a peening intensity of 0.020A2, thus reaching a saturation value. Similarly, for IMI. 318A, the surface macro-stresses have increased with peening intensity up to 0.014A2 and tended to level off above this intensity and show a slight decrease at an intensity of 0.018A2. The results of macro-stress distribution with depth below the surface (Fig. 39) show that for both peening intensities, the macro-stresses are of lower magnitude at the surface and increases with depth below the surface reaching a maximum value at a depth of 0.003 in below the surface. The macro-stresses further decrease with depth below the surface. The difference in the stress distribution below the surface between the two peening intensities is to be noted. Wedden and Liard (220) have shown that the fatigue performance of shot peened IMI.318A under reverse direct stress conditions diminished with the combination of increasing shot size and peening intensity, thus demonstrating close relationship between fatigue properties and peening intensity.

7.5.6.5. Vapour blasting the sheet specimen 0.036 in thick of IMI.130 has given the following stress values.

Micro-stresses	26.3 tonf/in ²
Macro-stresses	-22.0 tonf/in ²

The surface micro-stresses are of higher magnitude than those obtained with shot peened block specimens. The residual stresses have produced considerable distortion in the vapour blasted specimen. However, the depth of cold work may be shallow, and any removal of the vapour blasted texture may completely destroy the beneficial effects of the process.

7.5.6.6. The important difference between the residual micro-stresses produced by the machining operations and those induced by cold working the surface of IMI.130 is to be noted. In the case of ground IMI. 130 with a down feed 0.003 in/pass, the micro-stresses of 25.4 tonf/in² magnitude are considered to be responsible for crack initiation at areas of high dislocation density whereas no cracking has been observed for shot peened IMI.130 with the similar magnitude of micro-stresses. It may be that with the ground specimen the micro-stresses reached the fracture stress of IMI.130 due to grinding and stress relief occurred due to cracking and thermal effects, thus leaving a lower magnitude of residual micro-stresses on the surface.

7.5.6.7. The results in Figs. 33-35 and 37 show that the micro-stresses increase as the compressive macro-stresses increase, and decrease as the compressive macro-stresses decrease. This is true for the macro- and micro-stress induced by shot peening or produced by machining both in the surface and sub-surface regions. The results in Fig. 32, however, show that for IMI.130 deformed by 10% plastic strain in uniaxial tension, the reverse is true, in that micro-stresses increase as the compressive macro-stress decreases, thus demonstrating the essential difference between the two types of deformation. This may probably account for the difference concerning the agreement between the X-Ray diffraction and mechanical methods in shot peened and machined surfaces whilst disagreement between the method of measurement for specimen⁵ deformed plastically in uniaxial tension referred to in paragraph 7.1. Although the evidence may be inconclusive and further work is needed to confirm the results, nevertheless the essential difference is to be noted.

7.6. Industrial Importance of Measuring Residual Stresses.

7.6.1. In conclusion, the special role of micro-stresses arising from surface treatments such as grinding, machining or shot peening must be emphasised.

7.6.2. Compressive macro-stresses have been generally recognised as beneficial to the fatigue and stress-corrosion properties of the materials. Compressive surface macro-stresses may be produced by machining or grinding operations; however, a more predictable compressive stress distribution, extending to appreciable depth, may be induced by cold working the surface by shotpeening, surface rolling or ballising (the process of forcing an oversized hardened ball through the hole). However, a compressive stress system accompanied by an exposed balancing tensile stress is undesirable. It may be necessary to remove some material by chemical etching to examine for surface flaws. Also, where the surface compressive layer is shallow and stress gradient is steep, the removal of a thin layer, even for chemical inspection procedure, may be harmful. It is well known that the influence of macro-stresses arising from various industrial working processes in materials other than titanium, has been applied with success. On the other hand, the theory and practice of micro-stress measurement is well established but it has been rarely applied to industrial problems. Recently, some investigators (14, 26, 198) have attempted to show the effect of micro-stresses on the physical, chemical and fatigue properties of certain materials other than titanium.

7.6.3. This investigation has shown that macro- and micro-stress measurement in titanium are equally important. For example, in grinding, the role of micro-stress is really significant in the understanding of micro-cracking of IMI.130 (Photos 7 & 8), even in the presence of compressive macro-stresses. The grinding of titanium is further complicated by the temperature effects due to low thermal conductivity, tendency to gall easily, wheel loading and strong affinity for oxygen absorption. The experimental evidence shows (220) that tests conducted on IMI.318A to evaluate optimum grinding parameters, based on the response of the surface etch inspection technique, were not satisfactory, in that etch responsive grinding was extremely damaging. Further, non-etch responsive surfaces, which would normally have been considered satisfactory, also produced lower fatigue strength. Attempts at mitigation by shot peening proved unsatisfactory and substantial recovery in fatigue properties was obtained only by removal of the affected surface. On this basis the grinding of critically fatigue loaded components is prohibited at Westland Helicopters Ltd. The tests conducted were only empirical with no fundamental basis. The initiation of fracture is a very localised phenomenon and may be influenced by the local micro-stress situation. On the basis of evidence obtained from IMI.130 (Fig. 33) it is postulated that the reduction in fatigue properties due to grinding may be interlinked with the level of macro- and micro-stress distribution. Grinding is a useful machining operation and its use cannot be precluded for the normal production of critically stressed vital parts. It is therefore necessary that further tests should be carried out to inter-relate the effect of macro-and micro-stresses on the grinding parameters and premature failures. The same sort of consideration may apply to other machining operations such as face end milling.

7.6.4. Basically, the aircraft components are shot peened to impart a beneficial compressive stress system and to inhibit crack initiation and propagation. Fatigue test results obtained from IMI.318A (220) indicated that under reverse direct stress conditions, fatigue performance diminished with the combination of increasing shot size and peening intensity. The stress distribution curves obtained from shot peened IMI.318A to two peening intensities (Fig. 39), show that peening to an intensity 0.018A2 resulted in a lower level of sub-surface macro-stresses than that peened to an intensity 0.014A2. These results may possibly account for the discrepancy in results obtained by Wedden and Liard (220). Further the role of micro-stresses has also to be taken into account. For example, results obtained from IMI.130

(Fig. 35) show that micro-stress level in both surface (23.4 tonf/in^2) and sub-surface regions (25.7 tonf/in^2) can exceed the limit of proportionality of 23.0 tonf/in^2 obtained from tensile testing. Thus, it is important to understand the combined effect of macro- and micro-stresses on the shot peening parameters and to correlate the results with stress corrosion and fatigue properties.

7.6.5. The existence of micro-stresses is a fundamental feature of the material's property, and therefore the effect of macro- and micro-stresses should be taken into account when evaluating the effect of industrial working process on the static or dynamic properties.

7.6.6. The conventional diffractometer technique is limited in that only a specific type of specimen is readily examined and this difficulty has been largely overcome by the development of a transportable diffractometer specially designed for residual stress measurement (229). The back reflection film method, however, seems to be more appropriate as an industrial tool but for measuring residual macro-stresses in titanium or titanium alloy, it is essential to use material without significant directionality such as occurs in the cross rolled sheet. However, the broadened reflections, which are an essential indication of the presence of micro-strains/crystallite sizes, are not readily analysed by the film method.

8. CONCLUSIONS

1. Methods have been developed for the measurement of macro- and micro-stresses in IMI.130 and macro-stresses in IMI.318A.
2. Good agreement was obtained for macro-stress values in IMI.130 and IMI.318A deformed unidirectionally in four point loading using the following methods:-
 - (i) multi-exposure X-ray back reflection technique (film method),
 - (ii) diffractometer,
 - (iii) strain gauges,
 - (iv) beam bending theory,
3. Good agreement was obtained for macro-stress values on the tension and compression faces of the strip in IMI.130, using the film method and three different radiations, Cu K α , Co K α and Cr K α .
4. Good agreement was obtained for macro-stress values on the tension and compression faces of the strips in IMI.130 and IMI.318A, using the film method with Co K α radiation^{the} and diffractometer with Cu K α radiation.
5. For measuring macro-stresses in IMI.130 and IMI.318A by the film method, it was essential to use material without significant directionality such as occurs in the cross rolled sheet. Macro-stresses in IMI.130 and IMI.318A, by the diffractometer, were measured irrespective of the condition of the material.
6. Applied and residual macro stresses were measured by the film method in the alpha- and beta phases of IMI.318A deformed unidirectionally in four point loading. The onset of plastic deformation occurred at a similar stress level in both the alpha and beta phases. The residual macro-stresses measured in the beta phase were of higher magnitude than in the alpha-phase.
7. Micro-stresses were measured in IMI.130 by the X-ray line broadening technique using a diffractometer.
8. Macro- and micro-stresses were measured in IMI.130 subjected to industrial working processes, i.e. grinding, turning, face end milling, vapour blasting, shot peening and deformed in uniaxial tension, and gave different stress distributions.
9. Macro-stresses were measured in shot peened IMI.318A to peening intensities 0.014A2 and 0.018A2. The two peening intensities gave similar stress distribution but different level of sub-surface macro-stresses.
10. The values of stress factor determined experimentally, using a diffractometer, were 34 and 30 tonf/in² for IMI.130 and IMI.318A respectively.

11. The X-ray values of Young's modulus and Poisson's ratio determined for 213 reflection of alpha phase in IMI.130 and IMI.318A, using a diffractometer, were as follows:-

<u>Material</u>	<u>Young's Modulus</u> <u>Lbf/in² x 10⁶</u>	<u>Poisson's Ratio</u>
IMI.130	16.45	.297
IMI.318A	14.63	.375

The X-ray values of Young's modulus were similar to the bulk values determined by tensile testing.

12. These results are discussed in the light of existing knowledge and the significance of measuring both macro-and micro-stresses is emphasised.

The results in this investigation have shown that various deformation processes give rise to macro- and micro-stresses in titanium and that these can be accurately measured by the X-ray diffraction methods. In order to obtain a better understanding about the deformation characteristics of titanium and titanium alloys, it is proposed that the following programme of work be carried out.

- 1) Develop methods for measuring micro-stresses in duplex phase titanium alloys and in particular for IMI.318A.
- 2) Study the effect of shot peening, grinding, turning, milling and vapour blasting on the macro- and micro-strain measurements in duplex phase titanium alloys and in particular for IMI.318A.
- 3) The dislocation density is related to work hardening and it may be desirable to examine the dislocation density on the surface of commercially pure titanium subjected to the following two modes of deformation, which are known to produce different amounts of micro-strain:-
 - (a) uniaxial plastic deformation or bending,
 - (b) shot peening, turning, grinding, milling and vapour blasting.

The dislocation density can be derived by the Williamson and Smallman (183) method. Also the dislocation density could be deduced using the electron microscope.

- 4) Study the effect of plastic deformation on the stress factor. Previous investigators (12, 66, 75, 87, 92, 121, 184) have been cautious of possible errors when measuring stresses in material subjected to uniaxial plastic deformation. Since plastic deformation cannot be entirely eliminated in structural members subjected to service loads, it is important to know the extent to which plastic deformation affects residual stress measurements in these parts. Residual stress measurements, however, involve the calculation of stress factors, which could possibly vary with alloy composition, heat treatment, plastic deformation, etc. It would, therefore, be desirable to study the effect of plastic deformation uniaxial or otherwise on the stress factor calculations in commercially pure titanium as well as duplex phase titanium alloys and in particular IMI.318A.

- 5) Study the effect of heat treatment and phase transformation on the macro- and micro-stresses in IMI.318A. Bartlo (185) has shown that variations in micro-structure tend to alter the tensile and fatigue properties of 6%Al-4%V Ti alloy. He used forged and hot-rolled bar stock and employed three different cooling rates, i.e. furnace cooling, air cooling and water quenching, to produce the structural variations after heat treating specimens from 840-1065°C. He concluded that the best combinations of tensile and fatigue properties was obtained in material with a fine grained alpha-beta structure or in material with a micro-structure consisting of a mixture of fine primary alpha and martensitic alpha. Wedden (186) has also investigated the influence of micro-structure on the tensile and fatigue properties of 6%Al-4%V Ti alloy. He utilised rolled and forged stock of varying sized sections having accompanying micro-structural variations. Wedden concluded that a micro-structure having between 60-75% fibrous fine grained primary alpha in a beta matrix, or beta transformation products irresolvable at x 750, produced the optimum fatigue properties of the material examined.
- 6) Develop a technique for investigating the effect of micro-strains on the electro-chemical potential of titanium as part of a stress corrosion study.
- 7) Develop methods for determining the elastic constants for alpha titanium to understand the effect of elastic constants on the macro- and micro-stresses, and also the effect of preferred orientation on the elastic properties of the material.
- 8) Develop methods to investigate the effect of stacking fault energy on both the line shift and line broadening for reflections occurring above 130° 2θ angular range for IMI.130.
- 9) Study the causes and effects of radial streaking resulting from oxidation of IMI.130 and IMI.318A.
- 10) Study the effect of various deformation processes on the intensity distribution of X-ray line profiles in IMI.130.
- 11) Study the effect of macro- and micro-stresses on the static and dynamic properties such as fatigue, stress corrosion, for duplex phase titanium alloys and in particular IMI.318A.
- 12) Confirm the comparison obtained between shot peened/machined surfaces and plastically deformed specimens in uniaxial tension for IMI.130.

APPENDIX 1

Depth of penetration of X-rays

It is assumed that the amount of radiation diffracted from a depth x is proportional to the amount of incident radiation at that depth. Also both the incident and reflected radiations are absorbed exponentially.

If 50% of the reflected radiation comes from a depth $\leq \bar{x}$ then

$$\begin{aligned}\int_0^{\bar{x}} e^{-2\mu x} dx &= \frac{1}{2} \int_0^{\infty} e^{-2\mu x} dx \\ \therefore \left[-\frac{1}{2\mu} e^{-2\mu x} + \frac{1}{2\mu} \right] &= \frac{1}{2} \left[0 + \frac{1}{2\mu} \right] \\ \therefore e^{-2\mu \bar{x}} &= \frac{1}{2} \\ \therefore \bar{x} &= \frac{0.693}{2\mu}\end{aligned}$$

where μ is the linear absorption coefficient.

APPENDIX 2

BASIC PRINCIPLES OF STRESS MEASUREMENT BY X-RAY DIFFRACTION METHOD

The basic principles for determining stresses by X-rays are based upon measuring strain, which is then converted into stress by equations developed in the classical theory of elasticity. The X-ray method detects elastic strain only, as the method is fundamentally a measure of the inter atomic spacings; which are altered by elastic stresses.

The X-ray technique is strictly valid for measurement of stress in a material which is elastic, homogeneous and isotropic. Polycrystalline metals satisfy these requirements to a good approximation. It is further assumed that the penetration of the X-ray beam into the sample surface is sufficiently limited and the stress normal to a free surface is zero.

It may be shown theoretically that in any homogeneously stressed body, it is possible to set up special axes (Fig.1 below), such that an infinitesimal cube orientated with its edges parallel to these axes, will have no shear stresses on its cube faces. The stresses are thus all tensile, and are called the principal stresses ($\sigma_1, \sigma_2, \sigma_3$) and the corresponding strains the principal strains ($\epsilon_1, \epsilon_2, \epsilon_3$). They are related as follows:-

$$\epsilon_1 = \frac{1}{E} \sigma_1 - \nu (\sigma_2 + \sigma_3)$$

$$\epsilon_2 = \frac{1}{E} \sigma_2 - \nu (\sigma_3 + \sigma_1)$$

$$\epsilon_3 = \frac{1}{E} \sigma_3 - \nu (\sigma_1 + \sigma_2)$$

Appendix 2. Fig. 1.

In the case of surfaces stresses, the principal stresses $\sigma_1 + \sigma_2$ are in the surface plane, thus stress σ_3 , which is normal to the free surface, is zero, then

$$\epsilon_3 = \frac{1}{E} [0 - \nu(\sigma_1 + \sigma_2)]$$

or $\epsilon_3 = -\frac{\nu}{E} [\sigma_1 + \sigma_2]$ (2)

When the material is stressed, the spacing of the atomic planes lying parallel to the surface will be altered from d_0 (unstressed value) to $d_\perp$ such that

$$\epsilon_\perp = \frac{d_\perp - d_0}{d_0}$$

Writing $\epsilon_\perp = \epsilon_3$, the measurement of $d_\perp$ and d_0 will give the sum of the principal stresses:-

$$\sigma_1 + \sigma_2 = -\frac{E}{\nu} \left(\frac{d_\perp - d_0}{d_0} \right)$$

The sum of the principal stresses is usually of little value to the engineer, further, it is often impossible to obtain the same material in the unstressed condition.

Two exposure method

By the two exposure method surface stress in any known direction can be determined. One measurement of the interplanar spacing is made with the X-ray beam normal to the surface of the specimen where stress is desired ($d_\perp$), and a second determination is made with the X-ray beam inclined at a known angle to the surface, and lying in the vertical plane fixed by the surface direction of interest (d_ψ).

The interplanar spacings $d_\perp$ and d_ψ are related to the stress to be measured in the following manner.

Suppose it is required to measure the stress in the direction OP (Fig.1). Whatever the stress system, three mutual perpendicular tensile stresses on planes which carry no shear stress can be found. These are principal directions, and stresses acting in these directions $\sigma_1, \sigma_2, \sigma_3$ are called the principal stresses, a_1, a_2, a_3 are the direction Cosines of the ψ direction relative to these axes.

The strain ϵ_n , in any chosen direction with direction Cosines a_1, a_2, a_3 , to the principal strains ϵ_1, ϵ_2 and ϵ_3

$$\epsilon_n = a_1^2 \epsilon_1 + a_2^2 \epsilon_2 + a_3^2 \epsilon_3 \quad (5)$$

from the theory of the strain quadric () and the stress

$$\sigma_n = a_1^2 \sigma_1 + a_2^2 \sigma_2 + a_3^2 \sigma_3 \quad (6)$$

For stresses lying wholly in the surface $\sigma_3 = 0$ (Fig.2 below) and the stress is a function of ϕ only:-

Appendix 2 Fig. 2

writing $a_1 = \cos \phi$ $a_2 = \sin \phi$ $\sigma_n = \sigma_\phi$

$$\sigma_\phi = \sigma_1 \cos^2 \phi + \sigma_2 \sin^2 \phi \quad (7)$$

In terms of angular co-ordinates in three dimensions, these direction Cosines are written:- (Fig.1)

$$\begin{aligned} a_1 &= \sin \psi \cos \phi \\ a_2 &= \sin \psi \sin \phi \\ a_3 &= \cos \psi = \sqrt{1 - \sin^2 \psi} \end{aligned} \quad (8)$$

Substituting equation (8) in equation (6), the strain is expressed in the direction ψ, ϕ as:-

$$\epsilon_{\psi\phi} = \epsilon_1 (\sin \psi \cos \phi)^2 + \epsilon_2 (\sin \psi \sin \phi)^2 + \epsilon_3 \cos^2 \psi \quad (9)$$

Since stress normal to a free surface is zero, i.e. $\sigma_3 = 0$, and taking the values of strain in terms of stress as expressed in equation (1), then equation (9) may be rewritten:-

$$\epsilon_\psi = \frac{1}{E} [(\sigma_1 - \nu \sigma_2) \cos^2 \phi \sin^2 \psi + (\sigma_2 - \nu \sigma_1) \sin^2 \phi \sin^2 \psi] + \epsilon_3 \cos^2 \psi \quad (10)$$

writing $\cos^2 \psi = 1 - \sin^2 \psi$, and rewriting equation 10, we have

$$\epsilon_{\psi} - \epsilon_3 = \frac{1}{E} [(\sigma_1 - \nu \sigma_2) \cos^2 \phi + (\sigma_2 - \nu \sigma_1) \sin^2 \phi] \sin^2 \psi - \epsilon_3 \sin^2 \psi \quad (11)$$

Also from equation (2) we have

$$\epsilon_3 = -\frac{\nu}{E} [\sigma_1 + \sigma_2]$$

Multiplying both sides of the above equation by $\sin^2 \psi$ we have

$$\epsilon_3 \sin^2 \psi = -\sin^2 \psi \frac{\nu}{E} (\sigma_1 + \sigma_2)$$

∴ equation (11) can be rewritten as:-

$$\epsilon_{\psi} - \epsilon_3 = \frac{\sin^2 \psi}{E} [(\sigma_1 - \nu \sigma_2) \cos^2 \phi + (\sigma_2 - \nu \sigma_1) \sin^2 \phi + \nu (\sigma_1 + \sigma_2)]$$

$$\text{or } \epsilon_{\psi} - \epsilon_3 = \frac{\sin^2 \psi (1 + \nu)}{E} (\sigma_1 \cos^2 \phi + \sigma_2 \sin^2 \phi) \quad (12)$$

Substituting $\sigma_{\phi} = \sigma_1 \cos^2 \phi + \sigma_2 \sin^2 \phi$ we have

$$\epsilon_{\psi} - \epsilon_3 = \frac{\sin^2 \psi (1 + \nu)}{E} \sigma_{\phi}$$

$$\text{or } \sigma_{\phi} = (\epsilon_{\psi} - \epsilon_3) \frac{E}{1 + \nu} \frac{1}{\sin^2 \psi} \quad (13)$$

If d_0 is the interplanar spacing in the unstressed condition, $d_{\perp}$ the interplanar spacing between the planes parallel to the surface (measured by normal X-ray shot), d_{ψ} the interplanar spacing in the direction defined by the angles ψ and ϕ then the expression $\epsilon_{\psi} - \epsilon_3$ can be written in terms of interplanar spacing

$$\epsilon_{\psi} - \epsilon_3 = \frac{d_{\psi} - d_0}{d_0} - \frac{d_{\perp} - d_0}{d_0} = \frac{d_{\psi} - d_{\perp}}{d_0} \quad (14)$$

The accuracy of the above expression is governed largely by the precision with which the numerators are obtained. The same accuracy is not needed for denominator; hence $d_{\perp}$ can be substituted for d_0 in the denominator. This valid substitution therefore eliminates the determination of d_0 in the unstressed condition, the equation (14) may be written as:-

$$\epsilon_{\psi} - \epsilon_3 = \frac{d_{\psi} - d_{\perp}}{d_{\perp}} \quad (15)$$

Equation (13) may be written as:-

$$\sigma_{\phi} = \frac{d_{\psi} - d_{\perp}}{d_{\perp}} \cdot \frac{E}{1 + \nu} \cdot \frac{1}{\sin^2 \psi} \quad (16)$$

Multi-exposure X-ray back reflection technique

Again $n\lambda = 2d \sin \theta$ (Bragg's Law)

$$\therefore \frac{d_{\psi} - d_{\perp}}{d_{\perp}} = \frac{\sin \theta_{\perp} - \sin \theta_{\psi}}{\sin \theta_{\psi}} \quad (17)$$

Therefore equation (16) may be expressed as:-

$$\sigma_{\phi} = \frac{\sin \theta_{\perp} - \sin \theta_{\psi}}{\sin \theta_{\psi}} \cdot \frac{E}{(1 + \nu) \sin^2 \psi}$$

$$\text{or } \operatorname{Cosec} \theta_{\psi} = \frac{\sigma_{\phi} (1 + \nu) \sin^2 \psi}{E \sin \theta_{\perp}} + \frac{1}{\sin \theta_{\perp}} \quad (18)$$

i.e. $\operatorname{Cosec} \theta_{\psi}$ is a linear function of $\sin^2 \psi$.

Thus, if back reflection photographs are taken with the specimen surface direction, ϕ , at various inclinations to the incident beam, a number of values of $\operatorname{Cosec} \theta_{\psi}$ and corresponding values of $\sin^2 \psi$, are obtained. A $\operatorname{Cosec} \theta_{\psi} / \sin^2 \psi$ graph may be constructed and from the resulting straight line, the value of the surface stress component σ_{ϕ} may be evaluated.

In order to obtain accurate values of $\operatorname{Cosec} \theta_{\psi}$ and $\sin^2 \psi$ from a back reflection film, it is necessary to calibrate the specimen to film distance. This is achieved by covering the metallic surface under examination with a very thin coating of a suitable powder. This furnishes on the film its own back reflection lines, which are then used for calibration, and also as a fiduciary mark from which to measure the unsymmetrical line shift encountered during oblique exposures.

Stress measurement with diffractometer equipment

When the stress determination by the X-ray technique is conducted with a diffractometer, the position of the diffracted beam is measured in terms of angular position 2θ , therefore equation (16) should be expressed in terms of 2θ rather than the interplanar spacing.

$$\text{Again } n\lambda = 2d \sin \theta$$

Differentiating we get

$$\begin{aligned} \frac{\Delta d}{d} &= -\cot \theta \Delta \theta \\ \text{or } \frac{\Delta d}{d} &= -\cot \theta \frac{\Delta 2\theta}{2} \end{aligned} \quad (19)$$

∴ combining equation (16) and (19) we get:-

$$\sigma_{\phi} = (2\theta_{\perp} - 2\theta_{\psi}) \frac{\cot \theta}{2} \cdot \frac{E}{1+\nu} \cdot \frac{1}{\sin^2 \psi} \quad (20)$$

$$\text{Let } K = \frac{E \cot \theta}{2(1+\nu) \sin^2 \psi}$$

$$\text{Then } \sigma_{\phi} = K (2\theta_{\perp} - 2\theta_{\psi}) \quad (21)$$

The constant K is referred to as the stress factor.

Method for determining X-Ray Elastic Moduli
(Young's modulus E & Poisson's ratio ν)

From equation (13),

$$\sigma_{\psi} = (\epsilon_{\psi} - \epsilon_3) \frac{E}{1+\nu} \frac{1}{\sin^2 \psi}$$

rewriting this,

$$\epsilon_{\psi} - \epsilon_3 = \frac{(1+\nu) \sigma_{\phi} \sin^2 \psi}{E}$$

$$\therefore \epsilon_3 = -\frac{\nu}{E} (\sigma_1 + \sigma_2) \quad (\text{equation (2)})$$

$$\therefore \epsilon_{\psi} = \frac{1}{E} \left[(1+\nu) \sigma_{\phi} \sin^2 \psi - \nu (\sigma_1 + \sigma_2) \right] \quad (22)$$

For simple tension $\sigma_2 = \sigma_3 = 0$ and σ_1 is +ve.

Also ϵ_{ψ} is equivalent to the difference in the interplanar spacing of the atomic planes in the stressed and unstressed conditions which lie perpendicular to ψ , thus

$$\epsilon_{\psi} = \frac{d_{\psi} - d_u}{d_u} \quad (23)$$

Substituting the value of equation (23) in equation (22) we have:-

$$\frac{d_{\psi} - d_u}{d_u} = \frac{1}{E} \left[\frac{1 + \nu}{E} \sigma_1 \sin^2 \psi - \nu (\sigma_1) \right]$$

$$\begin{aligned} \text{or } d_{\psi} &= d_u + \frac{d_u}{E} \left[(1 + \nu) \sigma_1 \sin^2 \psi - \nu (\sigma_1) \right] \\ &= d_u + \frac{d_u}{E} \sigma_1 \left[(1 + \nu) \sin^2 \psi - \nu \right] \end{aligned} \quad (24)$$

The relationship between d_{ψ} and σ_1 is linear.

In order to determine the X-ray elastic modulus, a uniaxial test has to be carried out in the elastic range. The stress σ_1 can be applied directly to a tensile test piece or indirectly to a rectangular specimen by means of four point loading. In the former case, stress can be measured by the strain gauges attached to the tensile test piece but in the latter case, stress can be measured either by strain gauges or by calculation from bending theory.

In practice two linear plots will be obtained for sample inclination $\psi = 0$ and $\psi = \psi$. The intersection of the two plots would give the value of d_u . The values of E and ν will be obtained from the measured values of the slopes. Since the slope, m_{ψ} , for any value of ψ , is given by

$$m_{\psi} = \frac{d_u}{E} \left[(1 + \nu) \sin^2 \psi - \nu \right] \quad (25)$$

$$\therefore m_0 = - \frac{d_u}{E} (\nu) \quad \text{or} \quad d_u = - \frac{m_0 E}{\nu} \quad (26)$$

$$\text{and } m_{45} = \frac{d_u}{E} \left[(1 + \nu) \frac{1}{2} - \nu \right] = \frac{d_u}{E} \left[\frac{1 + \nu - 2\nu}{2} \right] = \frac{d_u}{2E} [1 - \nu] \quad (27)$$

Substituting equation (26) in (27) we have

$$m_{45} = \frac{m_0 E}{2\nu \times E} [1 - \nu] = - \frac{m_0}{2\nu} [1 - \nu]$$

$$\text{or } \frac{1 - \nu}{\nu} = - \frac{2m_{45}}{m_0}$$

$$\text{or } \frac{1}{\nu} = 1 - \frac{2m_{45}}{m_0} = \frac{m_0 - 2m_{45}}{m_0} \quad (28)$$

$$\text{or } \nu = \frac{m_0}{m_0 - 2m_{45}} \quad (28)$$

Substituting the value of v in equation (26) we have

$$d_u = -\frac{m_o E}{v} \quad \text{or} \quad E = -d_u \frac{m_o}{m_o - 2m_{45}} \quad (29)$$

APPENDIX 3

Geometry of diffraction with back reflection cameras

The type of back reflection photographs obtained for stress measurements showing the geometry of the back reflection technique with the specimen in an inclined position is shown in the figure below:-

Appendix 3 Fig.1

Evidently two different values of $\sin^2 \psi$ and the corresponding values of $\text{Cosec } \theta$ are obtainable from each photograph.

Diffraction lines A and H are the $K\alpha_1$ lines of Cobalt radiation diffracted from (114) planes of alpha titanium, B and G are the corresponding $K\alpha_2$ component. Similarly C and F are the $K\alpha_1$ lines diffracted from (420) planes of silver calibration powder. SN_1 and SN_2 are the normals to the reflecting planes contributing to the diffraction lines A and H. The position of the line peak is noted visually by Vernier scale.

The diameter of the calibration powder lines is assigned a convenient constant value of 7 centimeters. The diameter of the calibration powder diffraction lines is determined and the numerical factor (i.e. standardising factor) by which this diameter is to be reduced to 7 cm is calculated. The distances AC and FH are then multiplied by the standardizing factor and resulting corrected values are recorded as Δ_1 and Δ_2 . The relationship between Δ and $\text{Cosec } \theta$, and Δ and $\text{Sin}^2 \psi$ is obtained from the constructed graphs of Δ against these functions (see Appendix 4). The stress calculation sheets and graphs corresponding to a tensile stress of $14.8 \pm 0.8 \text{ tonf/in}^2$ and a compressive stress of $14.9 \pm 0.7 \text{ tonf/in}^2$ are appended to this appendix, as two illustrated examples.

X-RAY STRESS CALCULATION SHEET

Film No.	8675/1	Film No.	8675/4
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9.55		A		8.84
10.08		C		9.39
21.27		F		20.59
21.77		H		21.10

F	21.27	F	20.59
C	10.08	C	9.39
2R ₁	11.19	2R ₁	11.20
	7.00		7.00
S.F.		S.F.	

C	10.08	H	21.77	C	9.39	H	21.10
A	9.55	F	21.27	A	8.84	F	20.59
Δ ₁	.53	Δ ₂	.50	Δ ₁	.55	Δ ₂	.51
	.332		.313		.344		.319

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8675/1	η	.332	.049	1.02535
8675/1	η	.313	.048	1.02514
8675/4	η	.344	.049	1.02550
8675/4	η	.319	.049	1.02520

X-RAY STRESS CALCULATION SHEET

Film No.	8675/2	Film No.	8675/5
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8.35		A		8.39
9.05		C		8.93
20.23		F		20.18
20.76		H		20.84

F	20.23	F	20.18'
C	9.05	C	8.93
2R ₁	11.18	2R ₁	11.25
	7.00		7.00
S.F.		S.F.	

C	9.05	H	20.76	C	8.93	H	20.84
A	8.35	F	20.23	A	8.39	F	20.18
Δ 1	.70	Δ 2	.53	Δ 1	.54	Δ 2	.66
	.438		.332		.336		.411

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8675/2	$30 + \eta$	.438	.467	1.02659
8675/2	$30 - \eta$	.332	.088	1.02535
8675/5	$30 - \eta$	.336	.088	1.02540
8675/5	$30 + \eta$	.411	.465	1.02628

X-RAY STRESS CALCULATION SHEET

Film No.	8675/3	Film No.	8675/6
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8.78		A		7.80
9.54		C		8.35
20.74		F		19.60
21.31		H		20.35

F	20.74	F	19.60
C	9.54	C	8.35
2R ₁	11.20	2R ₁	11.25
	7.00		7.00
S.F.		S.F.	

C	9.54	H	21.31	C	8.35	H	20.35
A	8.78	F	20.74	A	7.80	F	19.60
Δ_1	.76	Δ_2	.57	Δ_1	.55	Δ_2	.75
	.475		.356		.342		.467

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8675/3	$40 + \eta$	.475	.641	1.02702
8675/3	$40 - \eta$	.356	.208	1.02563
8675/6	$40 - \eta$	.342	.209	1.02548
8675/6	$40 + \eta$	.467	.641	1.02693

Appendix 3. Fig.2 . Relationship of $\text{Cossec } \theta$ and $\text{Sin}^2 \psi$ corresponding to a tensile stress of $14.8 \pm 0.8 \text{ ton/in}^2$

X-RAY STRESS CALCULATION SHEET

Film No.	8676/1	Film No.	8676/4
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10.13		A		11.60
10.80		C		12.29
22.03		F		23.50
22.72		H		24.18

F	22.03	F	23.50
C	10.80	C	12.29
2R ₁	11.23	2R ₁	11.21
	7.00		7.00
S.F.		S.F.	

HC	10.80	H	22.72	C	12.29	H	24.18
A	10.13	F	22.03	A	11.60	F	23.50
Δ_1	.67	Δ_2	.69	Δ_1	.69	Δ_2	.68
	.418		.430		.431		.425

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8676/1	η	.418	.051	1.02636
8676/1	η	.430	.051	1.02650
8676/4	η	.431	.051	1.02652
8676/4	η	.425	.051	1.02644

X-RAY STRESS CALCULATION SHEET

Film No.	8676/2	Film No.	8676/5
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7.00		A		9.50
7.50		C		10.20
18.70		F		21.42
19.36		H		21.96

F	18.70	F	21.42
C	7.50	C	10.20
2R ₁	11.20	2R ₁	11.22
	7.00		7.00
S.F.		S.F.	

C	7.50	H	19.36	C	10.20	H	21.96
A	7.00	F	18.70	A	9.50	F	21.42
Δ_1	.50	Δ_2	.66	Δ_1	.68	Δ_2	.54
	.313		.413		.424		.337

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8676/2	$30 + \eta$	.313	.460	1.02514
8676/2	$30 - \eta$	.413	.086	1.02630
8676/5	$30 - \eta$	.424	.085	1.02643
8676/5	$30 + \eta$	.337	.461	1.02540

X-RAY STRESS CALCULATION SHEET

Film No.	8676/3	Film No.	8776/6
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9.06		A		11.18
9.53		C		11.82
20.72		F	/	23.05
21.33		H		23.49

F	20.72	F	23.05
C	9.53	C	11.82
2R ₁	11.19	2R ₁	11.23
	7.00		7.00
S.F.		S.F.	

C	9.53	H	21.33	C	11.82	H	23.49
A	9.06	F	20.72	A	11.18	F	23.05
Δ_1	.47	Δ_2	.61	Δ_1	.64	Δ_2	.44
	.294		.382		.399		.274

Film No.	ψ	Δ	$\sin^2 \psi$	$\operatorname{Cosec} \theta$
8673/3	$40 + \eta$	.294	.632	1.02492
8673/3	$40 - \eta$	.382	.207	1.02594
8673/6	$40 - \eta$	.399	.206	1.02614
8673/6	$40 + \eta$	.274	.631	1.02468

Appendix 3. Fig.3 Relationship of $\text{Cosec } \theta$ and $\text{Sin}^2 \psi$ corresponding to a compressive stress of $14.9 \pm 0.7 \text{ tonf/in}^2$

APPENDIX 4

Compilation of tables and preparation of graphs

The geometry of the back reflection technique is shown in appendix 3 Fig.1 SN_1 and SN_2 are the normals to the reflecting planes contributing to lines A and H. The diffracted beam for line H makes an angle $2\eta = 180 - 2\theta$ with the incident beam where θ is the Bragg angle.

If the inclination of specimen normal to the incident beam is ψ then the inclination of the reflecting plane for line H is $\psi - \eta$, and similarly $\psi + \eta$ for line A.

When the $K\alpha_1$ ring for calibration powder silver (CF) has been reduced to 7 cms, then it is possible to determine the corrected film to specimen distance from lattice constant of silver and an application of Bragg Law for the particular radiation used.

The distance OS (appendix 3 Fig.1) is calculated, OF is corrected to 3.5 cms and values are assigned to FH (Δ). Hence by giving a range of values to Δ , series of values of 2η may be determined by applying the tangent ratio to the triangle O S H. Since $2\eta = \pi - 2\theta$, $\text{Cosec } \theta$ is plotted against Δ .

Similarly other curves are obtained for $\sin^2 \psi$ against Δ when

- (i) $\psi = \eta$
- (ii) $\psi = 30^\circ - \eta$
- (iii) $\psi = 30^\circ + \eta$
- (iv) $\psi = 40^\circ - \eta$
- (v) $\psi = 40^\circ + \eta$

An example to illustrate is given below

Alpha Titanium

Radiation :- Co $K\alpha$ $\lambda K\alpha_1 = 1.78892\text{\AA}$

Calibrating powder :- Ag $a_o = 4.08621\text{\AA}$

For Ag (hkl 420)

$$\sin \theta = \frac{\lambda \sqrt{h^2 + k^2 + l^2}}{2a_o}$$

$$= \frac{1.78892}{8.17242} \sqrt{20}$$

$$\theta = 78^\circ 13'$$

In the $\triangle OSF$ (appendix 3 Fig.1)

$$\text{Angle } OSF = \pi - 2 = 23^{\circ} 34'$$

$$\text{In } \triangle FOS \text{ specimen to film distance } OS = \frac{3.5}{\tan 23^{\circ} 34'} = 8.024 \text{ cm}$$

The accompanying table has been compiled for 114 reflections of titanium using Co $K\alpha$ radiation and 420 reflections of silver calibrating powder.

Table showing the values of Cosec θ versus Δ and $\sin^2 \psi$ versus Δ

η	$\theta = \frac{\pi}{2} - \eta$	Cosec θ	2η	OH = $\frac{8.024}{\tan 2\eta}$	$\Delta =$ OH-3.5 cm.	$\sin^2 \psi_1$		ψ_2		ψ_3		ψ_4		ψ_5	
						$\psi_1 = \eta$	$\psi_1 = \eta$	$\psi_2 = 30 + \eta$	$\sin^2 \psi_2$	$\psi_3 = 30 + \eta$	$\sin^2 \psi_3$	$\psi_4 = 40 + \eta$	$\sin^2 \psi_4$	$\psi_5 = 40 + \eta$	$\sin^2 \psi_5$
11° 48'	78° 12'	1.02159	2336	3.506	0.006	0.0418	41° 48'	0.4443	0.0976	18° 12'	0.0976	51° 48'	0.6175	28° 12'	0.2233
12°	78°	1.02171	24	3.572	0.072	0.0432	42°	0.4477	0.0955	18°	0.0955	52°	0.6209	28°	0.2204
12° 12'	77° 48'	1.02311	2427	3.640	0.140	0.0446	42° 12'	0.4512	0.0934	17° 48'	0.0934	52° 12'	0.6243	27° 48'	0.2175
12° 24'	77° 36'	1.02388	2448	3.708	0.208	0.0461	42° 24'	0.4947	0.0914	17° 36'	0.0914	52° 24'	0.6277	27° 36'	0.2146
12° 36'	77° 24'	1.02468	2512	3.776	0.276	0.0476	42° 36'	0.4582	0.0894	17° 24'	0.0894	52° 36'	0.6311	27° 24'	0.2117
12° 48'	77° 12'	1.02548	2536	3.844	0.344	0.0491	42° 48'	0.4616	0.0874	17° 12'	0.0874	52° 48'	0.6345	27° 12'	0.2089
13°	77°	1.02630	26	3.914	0.414	0.0506	43°	0.4651	0.0855	17°	0.0855	53°	0.6378	27°	0.2061
13° 12'	76° 48'	1.02714	2624	3.983	0.483	0.0521	43° 12'	0.4686	0.0835	16° 48'	0.0835	53° 12'	0.6412	26° 48'	0.2033
13° 24'	76° 36'	1.02799	2648	4.053	0.553	0.0537	43° 24'	0.4721	0.0816	16° 36'	0.0816	53° 24'	0.6445	26° 36'	0.2005
13° 36'	76° 24'	1.02885	2712	4.124	0.624	0.0553	43° 36'	0.4756	0.0797	16° 24'	0.0797	53° 36'	0.6478	26° 24'	0.1977
13° 48'	76° 12'	1.02972	2736	4.195	0.695	0.0569	43° 48'	0.4790	0.0778	16° 12'	0.0778	53° 48'	0.6512	26° 12'	0.1949
14°	76°	1.03061	28	4.266	0.766	0.0585	44°	0.4826	0.0760	16°	0.0760	54°	0.6545	26°	0.1922
14° 12'	75° 48'	1.03152	2824	4.339	0.839	0.0602	44° 12'	0.4860	0.0741	15° 48'	0.0741	54° 12'	0.6578	25° 48'	0.1894
14° 24'	75° 36'	1.03244	2848	4.411	0.911	0.0618	44° 24'	0.4895	0.0723	15° 36'	0.0723	54° 24'	0.6611	25° 36'	0.1867
14° 36'	75° 24'	1.03337	2912	4.484	0.984	0.0635	44° 36'	0.4930	0.0705	15° 24'	0.0705	54° 36'	0.6644	25° 24'	0.1840
14° 48'	75° 12'	1.03432	2936	4.558	1.058	0.0652	44° 48'	0.4965	0.0687	15° 12'	0.0687	54° 48'	0.6677	25° 12'	0.1813
15°	75°	1.03528	30	4.633	1.133	0.0670	45°	0.5000	0.0670	15°	0.0670	55°	0.6710	25°	0.1786
15° 12'	74° 48'	1.03625	3024	4.708	1.208	0.0687	45° 12'	0.5035	0.0652	14° 48'	0.0652	55° 12'	0.6783	24° 48'	0.1759

APPENDIX 5

Rachinger's Method for the separation of $K\alpha_1$ α_2 doublet

This method depends on the assumption of a 2:1 ratio of the maximum peak intensities of α_1 α_2 peaks. To separate the α_1 and α_2 components of the peak, it is first necessary to calculate $\Delta\theta$ separation of the peaks due to the α_1 and α_2 radiations. This is obtained from the following equation:-

$$\Delta\theta = \sin^{-1} \left[\frac{\lambda\alpha_2}{2d_{hkl}} \right] - \sin^{-1} \left[\frac{\lambda\alpha_1}{2d_{hkl}} \right]$$

This angular separation has to be converted into distance along the chart record. If d is the doublet separation, the first step is to divide the profile into strips by ordinates whose separation is d , starting at the termination of the $K\alpha_1$ peak marked by position "a" in Fig.1. From a to a- d the α_1 peak is the same as the combined trace. By halving the ordinates between a and a- d and translating them through a distance d in the 2θ direction, the α_2 curve is obtained for the a- d and a-2 d . The α_1 curve in this range is then obtained by subtracting the α_2 curve from the combined α_1 - α_2 curve. The procedure is then repeated, the α_2 curve in the range a-2 d to a-3 d being obtained by halving the α_1 ordinates from a- d to a-2 d and displacing this reduced curve by a distance " d ". This procedure is repeated if necessary until the curve is completely resolved.

Appen.5. Fig.1 Rachinger's Method for the separation of $\alpha_1 \alpha_2$ doublet

APPENDIX 6

1. Grinding

Preliminary Grinding

A test blank (30 in x 0.75 in x 0.50 in) in commercially pure titanium was ground using a 921 Elliott machine at the University of Surrey. The grinding details were as follows:-

Wheel grade:	38A46H8VBE (Norton)
Wheel speed:	2140 surface feet/minute
Depth of metal removal:	0.015 in.
Down feed rate:	0.013 in @ 0.001 in/pass
:	0.002 in @ 0.0005 in/pass
Grinding Fluid:	Soluble oil
Cross feed:	Approx. 0.010 in after each table traverse (direction of feed towards and away from the operator)
Grinding direction:	Along the length of the specimen

Note. The grinding wheel was diamond dressed after every cut up to 0.013 in and subsequently after every 0.001 in of metal removed.

Test Grinding

The blank was sectioned into 5 test coupons. Keeping all other variables constant, the down feed rate was varied and .006 in material was removed as follows:-

<u>Test Coupon</u> <u>Identity</u>	<u>Down feed rate</u>	<u>No. of Passes</u>
G1	0.0005 in per pass	12
G2	0.001 in per pass	6
G3	0.002 in per pass	3
G4	Preserved	
G5	0.003 in per pass	2
* G6	0.0005 in per pass	3

*Ground without preliminary grinding.

2) Milling

A test coupon (0.75 in x 0.75 in x 0.50 in) was milled using the following variables:-

Cutter speed:	107.8 R.P.M.
Cutter diameter:	0.625 in Clarkson End Mill
Cutter feed rate:	0.422 in per minute
Depth of cut:	0.025 in
Cutting Fluid:	Soluble oil

3. Turning

A test coupon (0.75 in x 0.75 in x 0.50 in) was turned using the following machining variables:-

Tool material: High Speed Steel
Tool nose radius: 0.020 in
Feed IPR:
Depth of cut: 0.078 in
Speed: 150 R.P.M.
Cutting Fluid: Soluble oil

4. Vapour Blasting

The "vapour blast" process is essentially a form of liquid honing and consists of a high velocity water jet carrying, in suspension, certain abrasive material such as alumina and silicon carbide. This process is capable of removing metal from the sprayed surface and leaves a shallow but significant cold worked layer. The honing effect of a "vapour blast" treatment leaves a degree of surface finish superior to that typical of shot peening. The sheet material in DTD.5023 was vapour blasted using the following conditions:-

Diameter of nozzle: 12 mm
Grit size: CS 60
Time: 30 seconds
Specimen to nozzle distance: 8 in approx.

5. Shot Peening

Material	Shot Peening Conditions	Shot peening intensity			
		0.008A2	0.014A2	0.014A2 Lower hardness	0.020A2
IMI.130	Shot diameter - inches	0.023	0.023	0.031	0.023
	Pressure lbf/in ²	6	38	15	25
	Nozzle/work piece distance - inches	6	2	14	3
	Nozzle diameter - inches	0.375	0.375	0.375	0.375
	Angle of impingement	Normal to surface of test piece	Normal to surface of test piece	Normal to surface of test piece	Normal to surface of test piece
	Exposure time	40 sec.	1 min. 40 sec.	30 sec.	40 sec.

Material	Shot Peening Conditions	Shot peening intensity			
		0.008A2	0.014A2	0.014A2 Lower hardness	0.018A2
IMI.318A	Shot diameter - inches	0.023	0.023		0.023
	Pressure lbf/in ²	18	38		50
	Nozzle/work piece distance - inches	2	2		2
	Nozzle diameter - inches	0.375	0.375		0.375
	Angle of impingement	Normal to surface of test piece	Normal to surface of test piece		Normal to surface of test piece
	Exposure time				
	Exposure time	1 min. 20 sec.	1 min. 40 sec.		2 min. 40 sec.

6. Plastic Strain in Uniaxial Tension

Type of Specimen (Fig.1)	Plastic Strain %	Machine	Section	Area
S	4½	Hounsfield	0.124 in x 0.027 in	0.00335 in ²
S	10	"	0.123 in x 0.0235 in	0.00289 in ²
S	15	"	0.125 in x 0.0235 in	0.00294 in ²
S	10	Denison	0.500 in x 0.0295 in	0.0148 in ²

Appendix 7

Effect of oxidation and strain on radial streaking in IMI.130

During the course of the investigation, it was considered desirable to find out the effect of curved surfaces on the stress factor calibration using a diffractometer (For details see Page 65). Experiments were designed to stress relieve strips of IMI.130 in various bending jigs (Fig.2) manufactured from steel. Initially a silica tube shown in Photo 22 containing a bending jig was evacuated to give a vacuum of 10^{-4} mm Hg and heat treated at $850^{\circ} \pm 5^{\circ}\text{C}$ for 1 hour. The strip was cooled in the silica tube but the vacuum was not good enough and the strip was oxidized. The strips in various jigs were also deformed and retained the curvature of the bending jigs.

In order to heat treat the strips under identical conditions, to preclude any errors arising due to heat treatment, recourse was made to heat treat the strips in various bending jigs together. The heat treatment was carried out under continuous vacuum of 10^{-5} mm Hg or better at Nemo Heat Treatment Ltd., Stockport, Cheshire, as follows:-

<u>Cycle details</u>	<u>Vacuum mm Hg</u>
Cold before putting on heat	8×10^{-5}
At temperature 850°C	3×10^{-4}
Cooled to 400°C	3×10^{-5}
Cooled from 400°C to room temperature	- Specimen quenched in argon gas

The strips were plastically deformed and retained the curvature of ^{the} bending jigs. The strips were also oxidized and showed light brown - light blue colours. The argon gas used was of commercial grade and probably contained impurities such as O_2 , N_2 or H. The strips were examined by the X-ray back reflection technique and radial streaking, as shown in Appendix 7 Photo 1, was observed. The radial streaking increased in intensity with increase in the bending jig angle θ , tending to merge with the background, thus demonstrating the association of radial streaking with strain.

The observed streaking is similar in appearance to asterism observed in Laue patterns of deformed metals using white radiation, although the explanation is different.

The existence of radial streaking was confirmed by heat treating small and flat samples from IMI.130 strip. The heat treatment was carried

out in an electric muffle furnaces in air in the temperature range 260° - 750°C . The X-ray back reflection films taken from the heat treated flat samples are shown in Appendix 7 Photo 2. The strip in the 'as received' was also examined. It is to be seen that radial streaking has increased with increase in temperature. The rate of oxidation of titanium increased with increase in temperature and showed light brown - light blue - blue - dark brown colours; thus the association of radial streaking with rate of oxidation is demonstrated. With rise in annealing temperature, ^acorresponding change in grain size is also to be expected.

The experimental evidence thus shows that radial streaking may be a function of oxidation of titanium with variation in temperature and strain at a given temperature. The radial streaking probably occurs due to the following causes:-

- (i) scattering from oxygen atoms
- (ii) distortion resulting from oxygen atoms as a function of strain
- (iii) change in grain size with increase in temperature.

On the other hand when the shot peened block specimen or smaller samples from IMI.130 strip were sealed in an evacuated silica tube (vacuum 10^{-5} mm Hg), the specimens did not oxidize and were cleaner. When the vacuum was better than 10^{-5} mm Hg, the surface layers evaporated and titanium was deposited around the silica tube. This effect was equivalent to etching off the surface layers and might alter the surface properties.

The evidence is, however, inconclusive and further experimental work is needed to relate the effect of oxidation on radial streaking; nevertheless the following two pertinent facts are to be noted.

- (i) During commercial heat treatment of titanium under vacuum, it is important to bear in mind that the components in the surface layers may be more strained after than before annealing. The depth of contaminated surface layers may increase with time and temperature and it may be necessary to machine off the contaminated surface layers.
- (ii) During the heat treatment cycle, if the vacuum is higher than the optimum value necessary, then etching off the surface layers may result, thus altering the surface characteristics. Therefore extreme caution may be necessary in the interpretation of data obtained from machined, ground or shot peened surfaces involving heat treatment. It may be desirable to determine the optimum vacuum conditions during the heat treatment cycle for consistent and accurate results.

Fig. 1 Appendix 7 Effect of bending jig angle on radial streaking for curved surface of IMI 130.

As received

$\frac{1}{2}$ hour 260°C

$\frac{1}{2}$ hour 450°C

$\frac{1}{2}$ hour 550°C

$\frac{1}{2}$ hour 650°C

$\frac{1}{2}$ hour 750°C

Fig. 2 Appendix 7 Effect of temperature on radial streaking for flat strips of IMI 130.

Appendix 8

Effect of preferred orientation and deformation modes on 'line' intensity distribution in IMI.130

The experimental evidence showed that it was not possible to obtain stress free titanium powder to determine the instrumental correction necessary for micro-stress analysis. During annealing treatment in vacuum, oxygen was adsorbed in the lattice and produced distortion. Recourse was, therefore, made to obtain instrumental correction using block titanium (sheet or bar material).

The sheet material used had been cross rolled to minimise preferred orientation and to give an equiaxed grain structure. The experimental evidence revealed that some preferred orientation existed. The intensity distribution of the various reflections of annealed IMI.130 varied from the data recorded in the ASTM Card Index. Further, it was found that the intensity distribution of the sheet material exhibiting stronger preferred orientation, was considerably affected, in that the diffraction lines could not be observed with the film method. A similar effect was observed with bar material, as machined, ground or shot peened.

In order to investigate the effect of various deformation modes on the intensity distribution, preliminary experiments were carried out to obtain data from the cross rolled sheet material in the as received condition as follows:-

- i) Longitudinal direction
- ii) Transverse direction
- iii) 45° to the longitudinal axis (RHS)
- iv) 45° to the longitudinal axis (LHS)

Appendix 8 Fig.1.

The intensity of various reflections in the angular range $30^\circ 2\theta - 145^\circ 2\theta$ was chart recorded using a standard Phillips diffractometer. The fastest scanning speed of 1° per minute in deviation angle 2θ was used. Ni filtered Cu $K\alpha$ radiation with the following slits were used:-

<u>Angular range</u>	<u>Divergence slits</u>	<u>Receiving slits</u>	<u>Scatter slits</u>
$30^\circ - 50^\circ$	1°	0.1 mm	1°
$50^\circ - 90^\circ$	2°	0.1 mm	2°
$90^\circ - 145^\circ$	4°	0.2 mm	4°

The intensity values % were obtained by subtracting the background. In order to reduce the effects of the slits used, prismatic and basal planes were selected from each of the 3 angular ranges mentioned above. The ratio of the % intensity of prismatic/basal planes was calculated and the average value was obtained from these three results. Similarly, the ratio of % intensity of the same prismatic/basal planes was calculated from the % intensity data recorded in the ASTM Card Index for titanium powder with random orientation. The results are shown in Table 1. It is probable that, for values greater than 1.08, the prismatic mode of deformation may be operative, whilst for values less than 1.08, the basal mode may be predominant. On the basis of this proposed hypotheses, extreme caution is needed for the interpretation of the data and further experimental data is needed for basic understanding of the causes and effects of the modes of deformation on intensity distribution and slip system. The results for sheet material show that, even though the material was cross rolled, nevertheless some preformed orientation existed, and ^{the} basal mode was predominant.

The effect of surface treatments i.e. shot peening (peening intensities: 0.014A2 and 0.020A2) and grinding (down feed: 0.001 in/pass) on the intensity distribution was investigated for IMI.130. The details of the surface treatments are given in Appendix 6. The initial hardness of the specimen, ground and shot peened to an intensity 0.014A2, was 128 HV 10, compared to that of the specimen peened to an intensity 0.020A2, which was 190 HV 10. The intensity distribution with depth below surface was also examined, and successive layers were removed by chemically milling with HF/HNO₃ solution.

The ratio of the % intensity of prismatic/basal planes was calculated, and the results are shown in Appendix 8 Tables 1, 2 and Fig.2. It is to be seen that similar values were obtained on the surface for shot

peened specimens with an intensity of 0.014A2 or 0.020A2, and ^{the} basal mode was predominant. There was, however, considerable variation with depth below the surface. For specimens shot peened to an intensity of 0.014A2, the ratio corresponding to random orientation was obtained at 0.010 in below the surface and basal mode was predominant upto 0.019 in below the surface. In comparison, for specimens shot peened to an intensity of 0.020A2, the ratio corresponding to random orientation occurred at 0.0015 in below ^{the} the surface and basal mode also changed to ^{the} prismatic mode. For ground specimens, the ratio corresponding to random orientation occurred at a depth of 0.006 in below the surface whilst the mode changed from prismatic to basal at a depth of 0.016 in below the surface.

The similarity between the ratio of % intensity of prismatic/basal planes (Appendix 8 Fig.2) with compressive macro-stresses shown in Fig.34 and measured on the same specimen is to be noted. It is probable that the intensity distribution of the diffraction line profiles, modes of deformation and residual stresses may be interrelated and it is important to understand this relationship. The changes from basal to prismatic mode and vice versa due to shot peening and grinding may have an important bearing on the fatigue or other properties of titanium or titanium alloys.

APPENDIX 8 TABLE 1

Specimen Identity	Condition		% Intensity Surface/ Subsurface	Ratio Prismatic/Basal Planes			
	Process	Variable		$\frac{010}{002}$	$\frac{110}{10\bar{3}}$	$\frac{300}{01\bar{5}}$	Average of 3 results
G2	Grinding	0.001 in/pass Down feed rate	Surface	1.54	1.28	1.50	1.44
			Subsurface 0.003 in	7.75	1.03	0	2.926
			" 0.006 in	1.57	0.65	1	1.07
			" 0.010 in	19.00	3.25	0	7.42
Ti Sheet	Cross rolled	Long.Direction Trans.Direction 45° to long. axis (RHS) 45° to long. axis (LHS)	" 0.016 in	0.55	0.80	0	0.45
			Surface	0.09	0.15	0	0.08
			"	0	0.19	0	0.06
			"	0	0.15	0	0.05
Ti Powder ASTM Card Index			"	0.05	0.19	0	0.08
				1.15	1.06	1.00	1.07

APPENDIX 8 TABLE 2

Specimen Identity	Condition		% Intensity Surface/ Subsurface	Ratio Prismatic/Basal Planes			
	Process	Variable		$\frac{010}{002}$	$\frac{110}{103}$	$\frac{300}{015}$	Average of 5 results
SP2 L H	Shot Peening	Peening Intensity 0.014A2	Surface	0.31	0.35	0	0.25
			Subsurface 0.003 in	0.67	0.82	0.42	0.63
			" 0.006 in	0.83	0.92	0.37	0.71
			" 0.010 in	1.36	1.21	0.50	1.02
			" 0.016 in	1.22	0.72	0.50	0.89
SP3	"	Peening Intensity 0.020A2	Surface	0.25	0.38	0	0.21
			Subsurface 0.001 in	0.38	1.27	0	0.55
			" 0.0015 in	0.82	2.23	0.50	1.18
			" 0.003 in	4.94	4.45	2.75	4.04
			" 0.006 in	0.61	4.97	3.36	2.99
Ti Powder ASTM Card Index			" 0.010 in	50.00	∞	7.50	∞
			" 0.016 in	∞	∞	∞	∞
			" 0.018 in	∞	∞	∞	∞
				1.15	1.06	1.00	1.07

Appendix 8 Fig.2. Subsurface distribution of ratio of prismatic/basal planes in both ground and shot peened Ti-130.

TABLE 1

Static mechanical properties of commercially pure titanium IMI-170 in stress relieved condition

Material	Heat treatment	Direction	Static mechanical properties							E (10 ⁶) lb/in ²	Mean E (x10 ⁶) lb/in ²
			0.1% P.S. tonf/in ²	0.2% P.S. tonf/in ²	0.5% P.S. tonf/in ²	T.S. tonf/in ²	Elong.% 4/a	Stress tonf/in ² at limit of proportion- ality	Mean stress tonf/in ² at limit of proportion- ality		
IMI5023	Stress relieved 540°C - 1/2 hr AC	Longitudinal	25.0	25.3	25.3	32.6	30	23.1		15.0	15.5
		Longitudinal	25.5	25.6	25.9	32.7	29	20.3	22.2	16.4	
		Longitudinal	25.0	25.2	25.6	34.3	27 1/2	22.5		15.1	
		Longitudinal	25.2	25.3	25.6	33.6	27 1/2	22.9		15.5	
		Transverse	25.9	26.1	26.4	32.5	28 1/2	21.9		16.4	
		Transverse	26.5	26.7	26.7	32.6	26 1/2	22.7	23.0	16.4	
		Transverse	26.0	26.2	26.4	32.7	27	24.4		15.7	
		Transverse	25.7	25.9	26.1	32.6	28	23.1		15.6	

TABLE 2

Static mechanical properties of 6% Al - 4%V Ti alloy IMI.318A in stress relieved condition

Material	Heat treatment	Direction	Static mechanical properties								
			0.1% P.S. tonf/in ²	0.2% P.S. tonf/in ²	0.5% P.S. tonf/in ²	T.S. ² tonf/in ²	Elong.% 4/a	Stress tonf/in ² at limit of proportion- ality	Mean stress tonf/in ² at limit of proportion- ality	E (10 ⁶) lb/f/in ²	Mean E (x10 ⁶) lb/f/in ²
DD5163	Stress relieved 600°C - 1 hr AC	Longitudinal	59.3	59.6	60.4	63.2	12	56.4	55.0	15.9	16.3
		Longitudinal	61.1	61.3	61.7	65.0	11	57.3		16.5	
		Longitudinal	59.3	60.1	61.0	64.2	12	51.5	49.9	16.5	14.5
		Transverse	56.0	56.5	57.3	64.4	11	51.9		14.7	
		Transverse	54.7	55.6	56.9	63.3	12	51.9		14.4	
		Transverse	55.0	55.4	56.1	64.0	12	45.7		14.5	
		Transverse	55.0	55.6	57.3	63.3	11	50.0		14.5	
		Transverse									

TABLE 3

Surface stresses measured in commercially pure titanium IMI.130 strip TT2

using Co K α radiation and silver calibration powder

Degree of Bend	Stress σ Tonf/in ² Bending Theory (Theoretical)	Stress σ Tonf/in ² strain gauges	Stress Tonf/in ² constrained condition		Stress Tonf/in ² unconstrained condition	
			Tension face Lattice planes Ti (114) Ti (114) Ag (420) Ag (331)	Compression face Lattice planes Ti (114) Ti (114) Ag (420) Ag (331)	Tension face Lattice planes Ti (114) Ti (114) Ag (420) Ag (331)	Compression face Lattice planes Ti (114) Ti (114) Ag (420) Ag (331)
4° 51'	4.5	4.1	+ 5.3 $\pm$ 0.7	+ 5.2 $\pm$ 0.6	- 4.4 $\pm$ 1.0	- 3.9 $\pm$ 0.8
9° 42'	10.0	10.3	+ 7.8 $\pm$ 1.8	+ 8.3 $\pm$ 0.3	- 8.1 $\pm$ 0.5	- 8.8 $\pm$ 1.7
14° 46'	15.8	15.7	+ 13.7 $\pm$ 0.3	+ 14.6 $\pm$ 1.0	- 13.2 $\pm$ 0.3	- 11.9 $\pm$ 1.2
20° 50'	22.7	23.5	+ 13.7 $\pm$ 1.0	+ 15.3 $\pm$ 1.0	- 14.2 $\pm$ 0.4	- 16.6 $\pm$ 0.6
25°	28.2	-	+ 14.8 $\pm$ 0.8	+ 16.1 $\pm$ 0.3	- 14.9 $\pm$ 0.7	- 14.6 $\pm$ 0.5
30° 2'	34.4	-	+ 16.8 $\pm$ 1.6	+ 16.4 $\pm$ 0.1	- 14.2 $\pm$ 1.5	- 14.1 $\pm$ 1.4
Initial surface stress			0 $\pm$ 1.0	-	0 $\pm$ 1.6	-

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 4

Surface stresses measured in commercially pure titanium IMI.130 strip TT3

using Co K α radiation and silver calibration powder

Degree of Bend	Stress Tonf/in ² Bending Theory (Theoretical)	Stress Tonf/in ² constrained condition					
		Tension face Lattice planes Ti (114) Ag (420)		Compression face Lattice planes Ti (114) Ag (420)		Compression face Lattice planes Ti (114) Ag (331)	
4° 51'	-	+ 4.3 ± 0.3	+ 4.6 ± 0.7	- 3.5 ± 1.0	- 3.6 ± 1.0	-	-
9° 42'	10.5	+ 8.6 ± 1.1	+ 9.4 ± 0.6	- 8.0 ± 0.3	- 8.3 ± 0.4	-	-
14° 46'	15.3	+ 12.7 ± 0.5	+ 14.8 ± 1.0	- 13.3 ± 0.6	- 14.5 ± 0.3	-	-
20° 50'	23.3	+ 12.6 ± 0.2	+ 14.2 ± 1.3	- 12.7 ± 0.6	- 13.6 ± 0.9	-	-
25°	28.2	+ 13.2 ± 0.4	+ 14.0 ± 0.2	- 13.1 ± 0.3	-	-	-
30° 3'	32.9	+ 13.6 ± 0.7	+ 15.4 ± 1.1	-	-	-	-
Initial Surface stress		0	0	0	0	0	0

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 5

Surface stresses measured in commercially pure titanium IMI.130 strip TT3

using Cu K α radiation and silver calibration powder

Degree of Bend	Stress Tonf/in ² Bending Theory (Theoretical)	Stress Tonf/in ² constrained condition					
		Tension face Lattice planes			Compression face Lattice planes		
		Cu (006)	Cu (302)	Ag (511)(333)	Cu (006)	Cu (302)	Ag (511)(333)
4° 51'	-	+ 4.2 ± 0.4	+ 5.1 ± 1.8		- 3.3 ± 1.1	- 4.8 ± 1.0	
9° 42'	10.5	+ 7.9 ± 0.7	+ 9.2 ± 1.0		- 6.7 ± 0.6	- 10.2 ± 2.4	
14° 46'	15.3	+ 11.1 ± 0.3	+ 14.2 ± 2.4		- 12.2 ± 0.7	- 12.2 ± 0.2	
20° 50'	23.3	+ 13.1 ± 0.7	+ 13.4 ± 1.8		- 12.7 ± 0.6	- 13.6 ± 0.9	
25°	28.2	+ 11.8 ± 0.7	+ 13.6 ± 0.5		- 13.9 ± 0.9	-	
30° 3'	32.9	+ 14.5 ± 0.7	+ 17.6 ± 1.3		-	-	

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 6

Surface stresses measured in commercially pure titanium IMI.130 strip TT3
using Cr K α radiation and silver calibration powder

Degree of Bend	Stress Tonf/in ² Bending Theory (Theoretical)	Stress Tonf/in ² constrained condition	
		Tension face Lattice planes Ti (004) Ag (222)	Compression face Lattice planes Ti (004) Ag (222)
4° 51'	-	+ 4.0 $\pm$ 1.2	- 2.3 $\pm$ 1.0
9° 42'	10.5	+ 7.5 $\pm$ 0.3	- 7.1 $\pm$ 0.6
14° 46'	15.3	+ 9.3 $\pm$ 0.6	- 12.3 $\pm$ 1.8
20° 50'	23.3	+ 12.4 $\pm$ 1.1	- 13.8 $\pm$ 0.1
25°	28.2	+ 13.0 $\pm$ 2.1	- 13.1 $\pm$ 0.5
30° 3'	32.9	+ 9.9 $\pm$ 2.1	-
Initial surface stress		0	0

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 7

Surface stresses measured in 6%Al - 4%V Ti alloy IMI-318A strip BB2

using Co K α radiation and iron calibration powder

Degree of Bend	Stress Tonf/in ² Bending Theory (Theoretical)	Stress Tonf/in ² Strain gauges	Stress tonf/in ² constrained condition			
			Alpha phase	Tension face Lattice planes		Beta phase
			Ti (114) Fe (310)	Compression face Ti (114) Fe (310)	Ti (222) Fe (310)	Compression face Ti (222) Fe (310)
4° 51'	4.3	4.2, 4.7	+ 3.8 ± 0.5	- 3.1 ± 0.3	+ 4.0 ± 0.1	- 3.9 ± 1.0
9° 42'	9.6	9.3, 10.1	+ 7.8 ± 0.3	- 9.8 ± 0.9	+ 8.4 ± 0.2	- 10.7 ± 1.1
14° 46'	14.6	15.0, 15.3	+ 12.5 ± 0.8	- 13.8 ± 0.2	+ 14.8 ± 0.4	- 14.7 ± 2.0
20° 50'	20.6	19.0, 23.7	+ 19.8 ± 0.8	- 20.1 ± 1.5	+ 25.0 ± 1.3	-
25°	26.4	30.0, 28.2	+ 21.9 ± 0.2	- 24.6 ± 0.5	+ 24.4 ± 0.7	-
30° 3'	31.8	-	+ 24.4 ± 0.4	- 31.0 ± 1.9	+ 29.7 ± 1.3	-
35°	37.6	-	+ 33.7 ± 0.3	-	+ 37.2 ± 0.8	-
40°	43.6	-	+ 35.3 ± 0.7	-	+ 39.6 ± 0.8	-
Initial Surface stress			0	0	0	0

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 8
Surface stresses measured in IMI-318A strip CC3 using Co K radiation and Fe calibration powder

Degree of Bend	Stress tonf/in ² Bending Theory	Stress ² tonf/in ² Strain Gauges	Stress tonf/in ² Constrained Condition				Residual Stress tonf/in ² Unconstrained Condition			
			Alpha Phase Lattice Planes		Beta Phase Lattice Planes		Alpha Phase Lattice Planes		Beta Phase Lattice Planes	
			Ti (114) Fe (310)	Ti (222) Fe (310)	Ti (114) Fe (310)	Ti (222) Fe (310)	Ti (114) Fe (310)	Ti (222) Fe (310)	Ti (114) Fe (310)	Ti (222) Fe (310)
			Tension Face	Compression Face	Tension Face	Compression Face	Tension Face	Compression Face	Tension Face	Compression Face
4° 51'	4.6	5.2	+ 4.5 ± 0.5	- 4.0 ± 0.2	+ 4.4 ± 0.6	- 5.1 ± 1.0	-	-	-	-
9° 42'	9.4	11.2	+ 7.1 ± 0.2	- 9.7 ± 0.6	+ 8.5 ± 1.5	-12.1 ± 0.3	-	-	-	-
14° 46'	14.9	17.0	+13.8 ± 1.1	-14.5 ± 0.6	+17.3 ± 1.4	-13.4 ± 0.7	-	-	-	-
20° 50'	21.4	23.4	+19.2 ± 1.4	-19.8 ± 0.7	+19.6 ± 1.0	-	-	-	-	-
25°	26.2	28.3	+25.2 ± 0.5	-26.1 ± 0.8	+24.6 ± 3.0	-	-	-	-	-
30° 3'	31.9	34.0	+26.1 ± 0.7	-32.0 ± 0.6	+28.6 ± 2.2	-	-	-	-	-
35°	37.8	38.9	+33.9 ± 0.5	-	+34.7 ± 0.9	-	-	-	-	-
40°	43.8	-	+40.3 ± 0.6	-	+39.8 ± 0.5	-	-0.5 ± 0.3	+ 0.4 ± 0.3	-3.5 ± 0.3	+ 2.8 ± 0.5
45°	49.7	-	+41.5 ± 0.2	-	+49.2 ± 1.0	-	-2.4 ± 0.6	+ 6.2 ± 0.3	-5.2 ± 2.0	+11.4 ± 0.7
50°	55.1	-	+43.2 ±* 1.0	-	+49.5 ± 5.0	-	-2.6 ± 0.9	+ 9.0 ± 0.9	-	+13.0 ± 2.0
55°	61.8	-	+43.1 ± 1.0	-	+50.2 ± 2.0	-	-3.9 ± 0.4	+10.0 ± 0.5	-9.4 ± 1.2	+15.2 ± 0.5

- ve sign indicates compressive stress
+ ve sign indicates tensile stress

TABLE 2

Stress factor calibration data for IMI.130 using diffractometer

Degree of Bend	Stress tonf/in ² Bending Theory (Theoretical)	$\Delta 2\theta$ Shift + Instrumental Correction Factor					
		Combined use of variable jig (Photo 21) and bending jigs (Fig. 2)			Variable jig		
		Constrained Condition		Unconstrained Condition		Constrained Condition	
		Tension Face	Compression Face	Tension Face	Compression Face	Tension Face	Compression Face
4° 51'	4.3, 4.5	+ 0.07	- 0.20	-	-	+ 0.12	- 0.13
9° 2'	10.3, 10.0	+ 0.28	- 0.29	-	-	+ 0.33	- 0.22
12° 30'	11.7	+ 0.22	- 0.37	-	-	+ 0.35	- 0.32
14° 46'	15.8, 15.9	+ 0.43	- 0.47	-	-	+ 0.49	- 0.36
20° 50'	22.8, 22.7	+ 0.37	- 0.69	- 0.35	+ 0.01	-	-
25°	28.2	+ 0.43	- 0.59	- 0.24	+ 0.02	-	-
30° 3'	34.4	+ 0.26	-	- 0.30	+ 0.09	-	-

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 10

Stress factor calibration data for IMI 318A using diffractometer

Degree of Bend	Stress tonf/In ² Bending Theory (Theoretical)	$\Delta 2\theta$ shift + Instrumental Correction Factor			
		Constrained Condition		Unconstrained Condition	
		Tension Face	Compression Face	Tension Face	Compression Face
4°5'	4.5	+ 0.13	- 0.20	-	-
9°42'	9.5	+ 0.39	- 0.31	-	-
14°40'	14.8	+ 0.42	- 0.46	-	-
20°50'	21.0	+ 0.78	- 0.76	-	-
25°	26.3	+ 0.91	- 0.94	-	-
30° 3'	31.8	+ 1.08	-	-	-
35°	37.7	+ 0.78	-	- 0.058	- 0.112
40°	43.6	+ 1.42	-	- 0.035	+ 0.005
45°	49.7	+ 1.60	-	- 0.036	+ 0.057
50°	55.1	+ 1.51	-	- 0.150	+ 0.04
55°	61.8	+ 1.42	-	-	-
Initial $\Delta 2\theta$ Shift Flat Strip		- 0.007	-	-	-

- ve sign indicates compressive stress

+ ve sign indicates tensile stress

TABLE 11

Residual macro- and micro-stress measurement both surface and subsurface in IMI-130 deformed by uniaxial tension

Specimen Identity	Specimen Type Fig.1	% Applied Strain Uniaxial Tension	Stress Measurement: Surface/ Subsurface	Residual Stresses						Wagner and Aqua method 2 tonf/in ²
				Macro-stresses			Diffractionmeter (Cu K α radiation)			
				Back reflection (Co K α radiation)		$\Delta 2\theta$ Shift + Instrumental correction	Stress Longitudinal Direction	Subsurface Corrected Stress Value tonf/in ²		
				Longitudinal Direction Stress ² tonf/in ²	Transverse Direction Stress ² tonf/in ²					
TP1	S1	4 $\frac{1}{2}$	Surface	-1.9 $\pm$ 0.2	-2.9 $\pm$ 1.2	-	-	-	-	
TP3	S1	10	Surface	-5.7 $\pm$ 1.2	-2.2 $\pm$ 0.3	-	-	-	-	
TP2	S1	15	Surface	-5.4 $\pm$ 0.8	-2.1 $\pm$ 0.3	-	-	-	-	
TS1	S3	10	Surface	-4.9 $\pm$ 0.2	-1.4 $\pm$ 0.5	-0.36	-12.1	-	8.1	
"	"	"	Subsurface 0.0005 in			-0.20	-6.7	-	12.7	
"	"	"	" 0.002 in			-	-		10.4	
"	"	"	" 0.003 in			-0.36	-11.91	-9.5	10.1	
"	"	"	" 0.0065 in			-0.285	-9.41	-6.5	13.4	
"	"	"	" 0.0125 in			-0.17	-5.48	+3.9	11.2	
"	"	"	" 0.0145 in			-0.36	-11.8	-18.0	11.4	

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 12

Residual macro- and micro-stress measurement normal to the grinding direction in ground IMI.130

Specimen Identity	Condition		Stress measurements Surface/ Subsurface	Macro-stress			Micro-stress tonf/in ²		Crystallite size A ^o	
	Process	Variable Down feed rate		$\Delta 2\theta$ Shift + Instru- mental Correction	Stress tonf/in ²	Corrected subsurface stress value tonf/in ²	Hall and Williamson method	Wagner and Aqua method	Hall and Williamson method	Wagner and Aqua method
G1	Grinding	0.0005 in/pass	Surface	-0.52	-17.7	-	8.6	17.3	274	264
G2	"	0.001 in/pass	Surface	-0.61	-20.7	-	15.2	20.5	371	253
G3	"	0.002 in/pass	Surface	-0.13	-4.4	-	11.2	18.8	280	224
G5	"	0.003 in/pass	Surface	-0.35	-11.9	-	18.5	25.4	202	145
G2	"	0.001 in/pass	Subsurface 0.0005 in	-0.45	-15.3	-15.2	7.1	9.1		
"	"	"	" 0.0015 in	-0.07	-2.4	-2.0	4.6	6.0		
"	"	"	" 0.003 in	+0.16	+5.4	+5.3	1.1	2.0		
"	"	"	" 0.006 in	-0.13	-4.4	-4.9	1.6	3.1		
"	"	"	" 0.010 in	-0.03	-1.0	-0.13	1.1	1.8		
"	"	"	" 0.016 in	-0.07	-2.4	-2.4	2.5	3.4		

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 13

Residual macro- stress measurement parallel to the grinding direction in ground IMI 130

Specimen Identity	Condition		Stress measurement surface/sub surface	Macro - stress		
	Process	Variable Down feed rate		$\Delta 2 \theta$ shift + Instrumental correction	stress tonf/in ²	Corrected sub surface stress value tonf/in ²
G1	Grinding	0.005 in/pass	Surface	- 0.07	- 2.4	-
G2	"	0.001 in/pass	Surface	- 0.45	-15.3	-
G3	"	0.002 in/pass	Surface	+ 0.08	+ 2.7	-
G5	"	0.003 in/pass	Surface	- 0.22	- 7.5	-
G2	"	0.001 in/pass	Sub Surface 0.003 in	+ 0.08	+ 2.7	+ 3.4
"	"	"	" " 0.006 in	- 0	0	- 2.3
"	"	"	" " 0.010 in	+ 0.14	+ 4.8	+ 5.1
"	"	"	" " 0.016 in	- 0.035	- 1.2	- 2.4

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 14

Residual macro- and micro- stress measurement in vapour blasted, turned milled and shot peened IMI 130

Specimen Identity	Condition		Macro- stress		Micro-stress tonf/in ²		Crystallite size A°	
	Process	Variable	$\Delta 2\theta$ shift + instrumental correction	Stress tonf/in ²	Hall and Williamson method	Wagner & Aqua method	Hall and Williamson method	Wagner & Aqua method
VB1	Vapour blasting	-	- 0.66	- 22.4	22.0	26.3	372	217
T1	Turning	-	- 0.34	- 11.6	10.2	14.2	390	278
FEMI	Face 2nd milling	-	- 0.45	- 15.3	17.0	22.0	369	242
SP1	Shot peening	Peening Intensity 0.008 A2	- 0.70	- 23.8	12.9	19.1	281	213
SP2	"	" 0.014 A2	- 0.86	- 29.2	-	-	-	-
SP3	"	" 0.020 A2	- 0.83	- 28.2	16.4	22.4	323	219
SP2 LH	"	" 0.014 A2	- 0.17	- 5.8	18.5	23.4	449	279
SP2 A3	"	" 0.014 A2 + annealed 300°C 1 hr	-	-	14.9	18.1	502	458
SP2 A5	"	" 0.014 A2 + annealed 500°C 1 hr	-	-	7.8	5.2	656	602

Hardness
191 HV10Hardness
190 HV10Hardness
177 HV10Hardness
128 HV10

"

"

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 15

Residual macro- and micro- stress measurement with depth below surface in shot peened IMI 130

Specimen Identity	Condition		Stress Measurement Surface/Sub Surface	Macro-Stresses			Micro-Stress Tonf/in ²
	Process	Variable		$\Delta \sigma$ Shift + Instrumental Correction	Stress Tonf/in ²	Corrected sub surface stress value Tonf/in ²	
SP 2 LH	Shot Peening	Peening Intensity 0.014 A2	Surface	- 0.17	- 5.8	-	23.4
			Sub Surface 0.001in	- 0.13	- 4.4	- 4.4	-
			" " 0.0015in	- 0.10	- 3.4	- 3.4	-
			" " 0.003in	- 0.09	- 3.01	- 3.0	9.31
			" " 0.006in	- 0.12	- 4.1	- 3.9	4.00
			" " 0.010in	- 0.01	- 0.3	- 0.01	1.6
			" " 0.016in	- 0.15	- 5.1	- 5.1	1.6
			Surface	- 0.83	- 28.2	-	23.4
			Sub Surface 0.001in	- 0.91	- 30.9	- 30.8	25.7
			" " 0.003in	- 0.44	- 15.0	- 14.4	20.3
SP 3		Peening Intensity 0.020 A2	" " 0.006in	- 0.72	- 24.5	- 24.0	22.2
			" " 0.010in	- 0.51	- 17.3	- 16.7	15.0
			" " 0.016in	- 0.32	- 10.9	- 9.3	3.6

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 16

Residual macro-stress measurement in shot peened IMI 318A

Specimen Identity	Condition		Stress Measurement surface/sub surface	Macro-Stress		Hardness HV 10	
	Process	Variable Peening Intensity		$\Delta 2\theta$ shift + Instrumental Correction	Stress Tonf/in ²		
G16		0.008 A2	Surface	- 0.85	- 25.5	310	
G29		0.014 A2	Surface	- 1.01	- 30.3	320	
E13		0.018 A2	Surface	- 1.04	- 31.2	313	
G29			Sub Surface 0.002in	- 0.19	- 5.7		Opposite side unpeened
E13			" " 0.005in	- 0.16	- 4.8		surface " "

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 17

Residual macro-stress measurement with depth below surface in shot peened IMI 318A

Specimen Identity	Condition		Strain measurement surface/sub surface	Macro Stress		Stress tonf/in ²	Stress tonf/in ² Corrected sub surface value
	Process	Variable		$\Delta\epsilon$ shift + Instrumentation Correction	Stress tonf/in ²		
E13	Shot Peened	Peening Intensity 0.018 A2	Surface	- 1.04	- 31.2		
			Sub Surface 0.001in	- 1.14	- 34.2	- 33.8	
			" " 0.002in	- 1.40	- 42.0	- 41.2	
			" " 0.003in	- 1.49	- 44.7	- 43.2	
			" " 0.005in	- 1.50	- 45.0	- 42.4	
			" " 0.0085in	- 0.68	- 20.4	- 16.2	
			" " 0.0115in	- 0.05	- 1.5	+ 0.7	
			" " 0.014in	- 0.09	- 2.7	- 2.8	
			" " 0.020in	- 0.08	- 2.4	- 1.7	

-ve sign indicates compressive stress

+ve sign indicates tensile stress

TABLE 18

Residual macro-stress measurement with depth below surface in shot peened IMI 318A

Specimen Identity	Condition		Strain measurement surface/sub surface	Macro-stress		Stress tonf/in ²	Stress tonf/in ²
	Process	Variable		$\Delta 2\theta$ shift + Instrumentation Correction	Stress tonf/in ²		
G29	Shot Peened	Peening Intensity 0.014 A2	Surface	- 1.01	- 30.3		
			Sub Surface 0.001in	- 1.46	- 43.8	- 43.4	
			" " 0.002in	- 1.43	- 42.9	- 41.8	
			" " 0.003in	- 1.77	- 53.1	- 51.5	
			" " 0.005in	- 1.56	- 46.8	- 43.6	
			" " 0.0085in	- 0.34	- 10.2	- 5.7	
			" " 0.0115in	- 0.07	- 2.1	- 1.3	
			" " 0.014in	- 0.12	- 3.6	- 3.4	
			" " 0.020in	- 0.06	- 1.8	- 1.0	

-ve sign indicates compressive stress

+ve sign indicates tensile stress

S₁

S₂

No. 11 Drill
holes to be
jig drilled

S₃

Fig.1. Drawings of S₁, S₂ and S₃ tensile test pieces.

Fig.2. Drawing of jig to bend strips into circular arc of radius r for the given angle θ .

θ	$= 5^\circ$
θ	$= 10^\circ$
θ	$= 15^\circ$
θ	$= 20^\circ$
θ	$= 25^\circ$
θ	$= 30^\circ$
θ	$= 35^\circ$
θ	$= 40^\circ$
θ	$= 45^\circ$
θ	$= 50^\circ$
θ	$= 55^\circ$

(a)

(b)

Fig. 3 Constitutional diagrams:-

(a) titanium-aluminium system.

(b) titanium-vanadium system.

Fig. 4. Subsurface microhardness traverses on shot peened Almen strip in commercially pure titanium IMI.130

Fig. 5. Subsurface microhardness traverses on shot peened blanks (3in x 0.75in x 0.5in) in commercially pure titanium IMI.130.

Fig. 6. Subsurface micro-hardness traverses on shot peened blanks (3 in x 0.75 in x 0.5 in) of IMI.130.

Fig. 7. Subsurface micro-hardness traverses on shot peened blanks (3 in x 0.3 in x 0.5 in) of IMI 318A.

Fig. 8. Surface stresses measured by strain gauges and calculated by bending theory on bending strips FT2 in IMI.150.

Applied stress	Ag	Ti
(420)	(114)
Applied stress	(331)	(114)

Fig.9. Relationship between surface stress and strain on bending strip TT2 of IMI.130 using Co K α radiation and Ag calibration powder.

Radiation

Plane
Ag Ti Co K α

(420) (114)

Cu K α

(511) (006)

Cu K α

(511) (302)

Cr K α

(222) (004)

Fig. 10. Relationship between applied stress and strain on bending strip TP3 of IMI 430, using Cu K α , Co K α and Cr K α radiations and Ag calibration powder.

Fig.11. Relationship between applied surface stress and strain on bending strip TT3 of IMI.130 using Cu K α radiation and Ag calibration powder.

Fig.12. Relationship between applied surface stress and strain on bending strip TP3 of IMI.430 using Co K α radiation and Ag calibration powder.

Fig.13. Relationship between applied surface stress and strain on bending strip TT3 of IMI.150 using Cr K α radiation and Ag calibration powder.

Radiation Planes		
	Ag	Ti
○—○	Co K α	(114)
□—□	Cr K α	(004)
△—△	Cu K α	(302)
▲—▲	Cu K α	(006)

Fig.14. Effect of beam penetration using different X-ray wave lengths on applied surface stress and strain on bending strip Ti3 of DI.130 in 5° and 10° bending jigs.

Ag {420} (114)
 Ti {222} {004}
 {511} {500}
 {511} {006}

Co K α
 Cr K α
 Cu K α
 Cu K α

○ □ △ △
 — — — —

Fig. 15. Effect of beam penetration on surface stress and strain on
 Depth of beam penetration - 0.001 mm

O Co K α { 114 }
 □ Cr K α { 004 }
 △ Cu K α { 302 }
 ▲ Cu K α { 005 }
 AG { 420 }
 { 222 }
 { 511 }
 { 511 }
 TT

Fig.16. Effect of beam penetration using different X-ray wave lengths on applied surface stress and strain on bending strip Ti-3Al-3V in 20° bending jig.

Fig. 17. Effect of beam penetration using different X-ray wave lengths on applied surface stress and strain on bonding strip T13 of III-130 in 25° bending jig.

Radiation	$\frac{AG}{T1}$
○—○ Co K α	(420) (114)
□—□ Cr K α	(222) (034)
△—△ Cu K α	(511) (302)
△—△ Cu K α	(511) (005)

Fig. 18. Effect of beam penetration using different X-ray wave lengths on applied surface stress and strain on bending strip Ti3 of IMI.130 in 30° bending jig.

Fig.19. Surface stresses measured by strain gauges and calculated by bending theory on bending strip BB2 of IMI.318A.

Fig.20. Relationship between applied surface stress and strain on bending strip BB2 using Co K γ radiation and Fe calibration powder for alpha and beta phases in IMI.318A.

Fig.21. Surface stresses measured by strain gauges and calculated by bending theory on bending strip CG3 of IMI.318A.

Fig.22. Relationship between applied stress and strain on bending strip CO3 in alpha and beta phases of IMI 318A using Co K α radiation and Fe calibration powder.

Fig.24. Diffractometer traces of silicon disc showing first check for:
 (a) instrumental correction with sample positions $\psi = 0^\circ$, $\psi = 45^\circ$
 (b) radial alignment with sample position $\psi = 45^\circ$.
 The position where $K\alpha_1$ would normally occur is also shown.

Fig. 25. Diffractometer traces for determining instrumental correction with sample position $\psi = 0^\circ$, $\psi = 45^\circ$:

(a) stress free silver powder

(b) annealed alpha titanium strip.

The position where $K\alpha_1$ would normally occur is also shown.

(a)

(b)

Fig.26. Stress factor calibration for IMI.130 and IMI.318A using diffractometer:

- (a) Combined use of variable jig Photo 21 and bending jigs Fig.2.
- (b) Variable jig only.

Fig.27. Determination of X-ray elastic constants for 213 reflection in IMI-130.

Fig. 26. Determination of x-ray elastic constants for the 213 reflection in IMI.318A.

- a: 0.336
- b: 0.349
- c: 0.406-0.266
- d: 0.431-0.266
- e: 94.95%
- f: 8.0

Fig. 29. β^* versus d^* for the shot peened IMT.130.
(Peening Intensity 0.008A2)

Fig. 30. $(\beta^*)^2$ versus $(d^*)^2$ for shot peened IMI.130.
(Peening Intensity 0.008A2)

Fig.31. Variation of longitudinal and transverse surface residual macro-stresses in IMI.130 deformed plastically in uniaxial tension.

Fig.32 Residual sub-surface macro and micro-stress distribution in IMI 130 deformed by 10% plastic strain in uniaxial tension

Fig.33. Residual macro- and micro-stress distribution in ground IMI.130 with variation in down feed rate/pass.

Fig.34 Residual sub-surface macro-stress distribution in both ground and shot peened IMI 130

Fig35 Residual sub-surface micro-stress distribution in both ground and shot peened IMI 130

Fig. 36. Residual subsurface macro-stress distribution in the grinding direction in ground IMI.430.

Fig. 37. Residual macro- and micro-stress distribution in shot peened IMI.130 and IMI.318A with variation in peening intensity.

Fig. 39. Effect of annealing temperature on micro-stresses and crystallite sizes in shot peened IM.130; peening intensity 0.01442.

Fig.39 Residual sub-surface macro-stress distribution in shot peened IMI 318A

Etched*

Photo 1. Satisfactorily ground Hylite 51 titanium alloy surface.

Etched*

Photo 2. Abusively ground Hylite 51 titanium alloy showing brittle outer skin and deformation in the surface region.

Etched*

Photo 3. Turned Hylite 51 titanium alloy showing deformation in the surface region.

Photo 4.

Etched*

x 75

Grain structure of commercially pure titanium
IMI.130

Photo 5.

Etched*

x 400

Grain structure of 6%Al-4%V Ti alloy IMI.318A

Etchant

HNO₃ 7%

HF 3%

H₂O 90%

Photo 6*

x 400

Shot peened IMI.130 showing density of twins
in damaged region: peening intensity 0.014A2.

Photo 7*

x 400

Ground IMI.130 showing crack initiation at
twin interfaces: down feed 0.003 in/pass.

Photo 8*

x 1000

Crack at twin
interface

Ground IMI.130: down feed 0.003 in/pass.
Magnified view of Photo 7.

*Polished on Gamma alumina pad impregnated
with oxalic acid.

Photo 9

x750

Photo 10

x2900

Stereoscan micrographs of ground IMI.130:
down feed 0.0005 in/pass.

Photo 11

x750

Photo 12

x2900

Stereoscan micrographs of ground IMI.130:
down feed 0.001 in/pass.

Photo 13

x700

Photo 14

x2800

Stereoscan micrographs of ground IMI.130:
down feed 0.002 in/pass.

Photo 15

x700

Photo 16

x2800

Stereoscan micrographs of ground IMI.130:
down feed 0.003 in/pass.

Photo 17

x 60000

Electron micrograph showing high dislocation density in shot peened IMI.130. The straight and diffuse bands probably correspond to deformation twins: peening intensity 0.014A2.

Photo 18

x 60000

Electron micrograph showing the initial stages of sub-structure formation in shot peened IMI.130: peening intensity 0.014A2.

Photo 19

x 40000

Electron micrograph of ground IMI.130. The dislocations are arranged in poorly developed sub-structure grain boundaries: down feed 0.0005 in/pass.

Bending Jig

Photo 20. General view of Raymax X-ray crystallographic unit and bending jig to deform the strips for stress analysis.

Photo 21. General view of diffractometer and bending jig to deform the strips for stress analysis.

Photo 22. Bending jig and IMI 130 strip
in silica tube under vacuum.

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