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An X-ray diffraction study of defect parameters in a Ti-base alloy

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Abstract. **Detailed studies based on the well established method of Fourier line shape analysis have been made on the X-ray diffraction profile of hexagonal titanium alloy of nominal composition Ti–6·58% Al–3·16% Mo–1·81% Zr–0·08% Fe–0·012% N–0·0078% C. While deformation fault probability, a, has been found to be quite high compared to that of pure titanium, the deformation growth fault parameter, b, shows a negative value ruling out the presence of growth fault in this alloy in the deformed state.**

Keywords. **Cold working; XRD; titanium alloy; microstructural parameters.**

1. Introduction

The X-ray diffraction study of microstructural parameters to reveal the nature of imperfections in metallic alloys is an established technique and is also finding wide applications in the recent studies of cold-worked alloys and nanocrystalline materials (Adler and Wagner 1962; Delhez *et al* 1982; De and Sengupta 1984; Van Berkum *et al* 1993, 1994). Titanium alloys are used extensively in aviation industry as suitable structural material offering an excellent combination of strength and ductility. The present material has been chosen to investigate as to how the fault parameters control the deformation characteristics of this alloy. The microstructural parameters like coherent domain size, deformation fault parameter a, growth fault parameter b and micro strain $<e_L^2>^{1/2}$ have been determined by considering a number of fault unaffected ($H–K = 3N$) and fault affected ($H–K = 3N \pm 1$) reflections.

2. Experimental procedure and method of analysis

A very drastic cold working was performed on the rod of the said composition procured from the Hindustan Aeronautics Ltd., Bangalore, with jewellery file at room temperature. The powdered samples were magnetically separated and sieved through a 250 mesh screen and flat diffractometer sample in the form of briquette suitable for the recording of XRD profile was prepared. A portion of the cold worked sample was vacuum sealed and annealed at 500°C for 10 h to annul the effect of cold work and cooled in air for the preparation of the standard sample required to make the profile analysis.

The XRD profiles for both the cold worked specimen as well as the standard specimen were recorded with CuK_a radiation in a highly stabilized Siemens Kristalloflex-4 X-ray Diffractometer operating at 30 kV, 12 mA and having 1° and 0·05 mm as divergence and receiving slits, respectively.

The detailed line profile analyses for hexagonal system (Warren 1969; Chatterjee and Sengupta 1975; Chattopadhyay *et al* 1990) have been performed making use of the following equations worked out by Warren and Averbach in their method of fourier analysis of imperfections in metallic alloys. The Stoke's corrected (De and Sengupta 1984; Warren 1969) fourier coefficients A_L are related to the size coefficients A_L^S and distortion coefficients A_L^D by

$$A_L = A_L^S\, A_L^D, \tag{1}$$

which can be separated in A_L^S and A_L^D from the log plot of A_L vs $1/d^2$ where d is the interplanar spacing (Warren 1969). Assuming Gaussian distribution of strain, the distortion coefficient A_L^D is given by

$$A_L^D = \exp(-\,2p^2L^2<e_L^2>/d^2), \tag{2}$$

where $L = na_3'$ i.e. undistorted distance between the cells and $a_3' = l/[2(\sin q_2 – \sin q_1)]$, where q_2 and q_1 are the truncation limits equispaced around $2q_0$ (peak position) within which the profiles are expressed as fourier series (Warren 1969) and n the harmonic number. For fault unaffected reflections ($H–K = 3N$), where $H–KL_0$ are the hexagonal indices and initial slope of A_L^S vs L is related as

$$-\,[\mathrm{d}A_L^S/\mathrm{d}L]_{L \to 0} = 1/D, \tag{3}$$

where D is the average domain size (Warren 1969). The

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Table 1. Values of lattice parameters a and c, average domain size, D and r.m.s. strain $<e_L^2>^{1/2}$ for Ti-base alloy.

Alloy composition	Lattice parameters a(Å)	c(Å)	Fault unaffected reflections $H–K = 3N$	D(Å)	r.m.s. strain $<e_L^2>^{1/2}$ at $L = 50$ Å
Ti–88·3502%	3·2367	5·1581	11·0	675	$5{\cdot}3 \times 10^{-3}$
Al–6·58			00·2		
Mo–3·16			01·0		
Zr–1·81					
Fe–0·08					
Ni–0·012					
C–0·0078					

slope of log A_L vs $1/d^2$ gives the measurement of micro-strain $<e_L^2>$ for various values of L.

Assuming the average domain size, D and micro strain $<e_L^2>$ thus determined from fault free reflections to remain uniform, the distortion coefficients (A_L^D) for faulted reflections are calculated using (2) and size coefficients (A_L^S) for faulted reflections are separated from Stoke's corrected fourier coefficients A_L for faulted ($H–K = 3N \pm 1$, L_0 odd or even) reflections (1). The initial slope of these A_L^S vs L is then given as (Warren 1969; Chatterjee and Sengupta 1975; Van Berkum *et al* 1994)

$$-[dA_L^S/dL]_{L\to 0} = 1/D_e = 1/D + |L_0|\, d(3a + b)/C^2 \quad \text{for } L_0 \text{ even}, \tag{4}$$

$$-[dA_L^S/dL]_{L\to 0} = 1/D_e = 1/D + |L_0|\, d(3a + 3b)/C^2 \quad \text{for } L_0 \text{ odd}, \tag{5}$$

where D_e is the effective domain size in the faulted planes, D the average domain size obtained from fault free reflections, a and b are respectively the deformation fault and growth fault parameters, and c the hexagonal axis ($c = 2d_{00\cdot2}$).

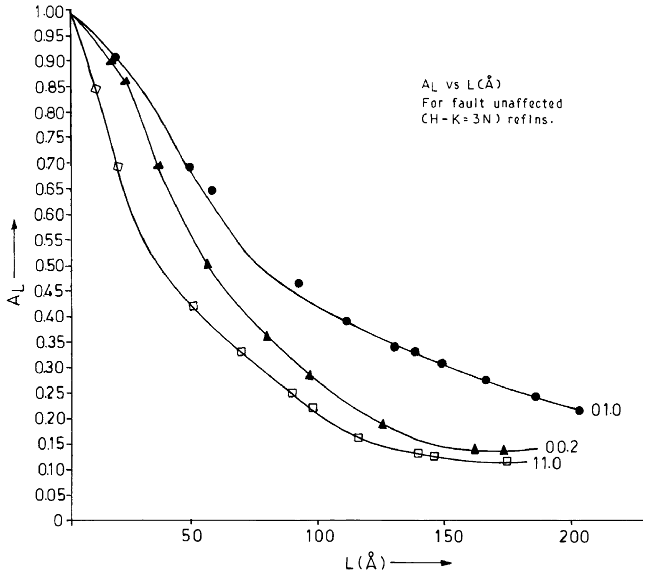

Figure 1. A_L vs L(Å) for fault unaffected $H–K = 3N$ reflections.

3. Results and discussion

The nominal composition of the titanium base alloy under investigation is Ti–6·58% Al–3·16% Mo–1·81% Zr–0·08% Fe–0·012% Ni–0·0078% C. From the peak position ($2q_0$) for all the observable reflections both belonging to fault unaffected and fault affected reflections, the lattice parameters have been calculated and are shown in table 1. This shows a c/a ratio of 1·594 which is same as other Ti-base alloys studied before (Sen *et al* 1995, 1997, 1998) and also as pure titanium (Chatterjee and Sengupta 1975).

The Stoke's corrected fourier coefficients A_L for fault unaffected ($H–K = 3N$) and fault affected ($H–K = 3N \pm 1$) for L_0 odd or even are shown in figures 1 and 2, respectively. The A_L^S and A_L^D coefficients ($A_L = A_L^S\, A_L^D$) have been separated from the plot of log A_L vs $1/d^2$ for fault unaffected reflections. The plot of log A_L vs $1/d^2$ is shown in figure 3. The intercepts give A_L^S and slope gives the strain. The average domain size, D calculated by using (3) from A_L^S of fault free $H–K = 3N$ reflections is obtained as 675 Å, which is slightly higher than that of pure titanium (Chatterjee and Sengupta 1975) and is of the same order as those for titanium alloys (Sen *et al* 1995, 1997, 1998). The r.m.s. strain $<e_L^2>^{1/2}$ has been calculated from the slope of log A_L vs $1/d^2$ for fault unaffected reflections and its variation with L is shown in figure 4. The value of $<e_L^2>^{1/2}$ at $L = 50$ Å is $5{\cdot}35 \times 10^{-3}$, which is also slightly higher than that for pure titanium but of the same order as that for titanium alloys (Sen *et al* 1995, 1997, 1998). Assuming the r.m.s. strain $<e_L^2>^{1/2}$ to be uniform both for fault unaffected and fault affected reflections, the A_L^D values of fault affected reflections were calculated and the A_L^S coefficients have been sorted out from A_L coefficients of fault affected reflections. The plot of A_L^S vs L for fault affected reflections is shown in figure 5. The initial slopes of A_L^S vs L give corresponding effective domain size, D_e (Å). The values of D_e are listed in table 2 along with the compound fault parameters ($3a + b$) when L_0 is odd and

($3a + 3b$) when L_0 is even. The effective domain sizes are found to be sufficiently low indicating the presence of deformation faults. The deformation fault parameter a and growth fault parameter b have been calculated from the compound fault parameter values for various reflections and are shown in table 2. It has been found that the value of a is quite high for this alloy compared to that of pure titanium (Chatterjee and Sengupta 1975). The increase in the value of a from pure titanium by an order of magnitude (from $8{\cdot}1 \times 10^{-3}$ to 103×10^{-3}) is due to the

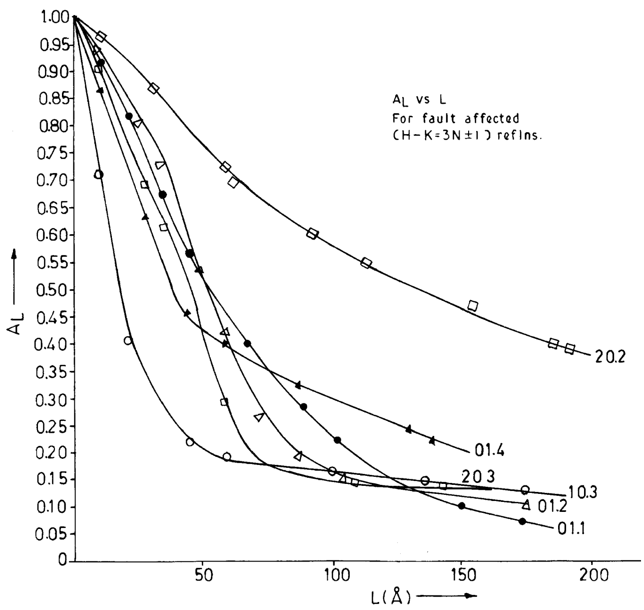

Figure 2. A_L vs L(Å) for fault affected H–$K = 3N \pm 1$ (L_0 even and odd) reflections.

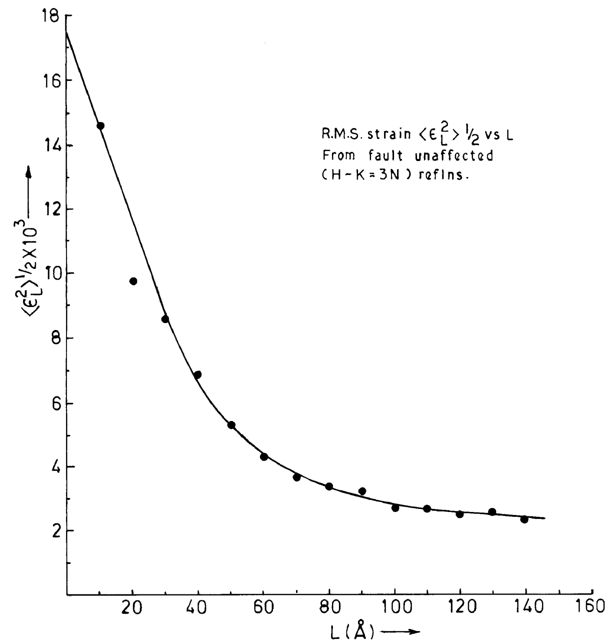

Figure 4. $\langle e_L^2 \rangle^{1/2}$ vs L(Å) for fault unaffected reflections.

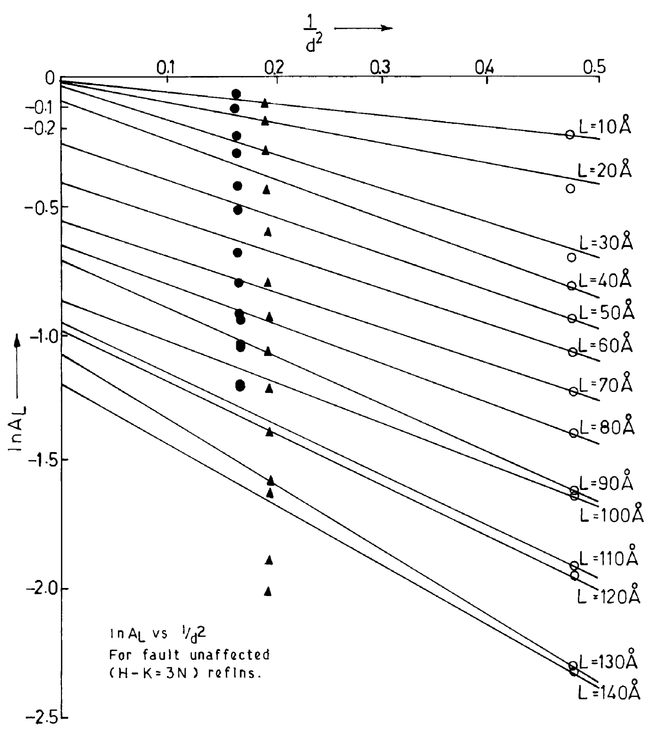

Figure 3. lnA_L vs $1/d^2$ for fault unaffected reflections.

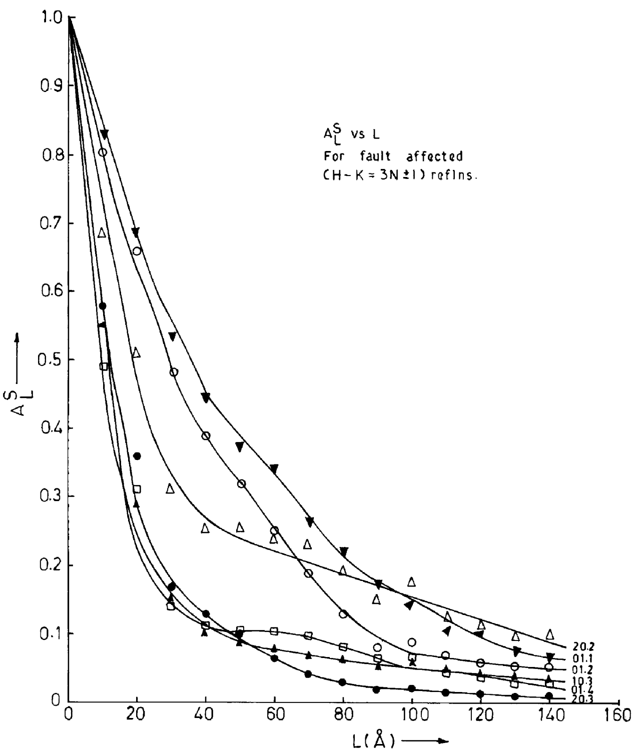

Figure 5. A_L^S vs L(Å) for fault affected reflections.

Table 2. Values of effective domain size, compound fault parameters, deformation fault, *a* and twin fault, *b* parameters for Ti-base alloy.

Fault affected reflections $H–K = 3N \pm 1$	D_e(Å)	Compound fault parameters (× 10^3) $3a + b$ L_0 = odd	$3a + 3b$ L_0 = even	$a \times 10^3$	$b \times 10^3$
20·3	24·0	362·8		103·4	– 37·1
10·3	23·0	281·2			
01·1	62·0	175·0			
01·2	49·0		146·71		
20·2	37·0		299·97		
01·4	19·5		312·50		

effect of solute. But when the value of *a* obtained for this alloy is compared with that in Ti–Al (~ 90×10^{-3}) and Ti–Zr (~ 100×10^{-3}), it is found that the usual effect of Al as solute which is to increase the faulting tendency undergoes a reversal in the presence of other solute elements like *b.c.c.* Mo (3·16 wt.%) and shows a decreasing trend. It is known that *h.c.p.* structure while undergoing faulting, the close packed basal planes change the stacking sequence from AB AB . . . to ABC ABC . . . similar to that for the stacking sequence of (111) planes in *f.c.c.* systems. The addition of Al (*f.c.c.*) has been observed to enhance the formation of this faulting sequence as in Ti–Al alloy (Sen *et al* 1995, 1998), and zirconium also lowers the stacking fault energy in titanium due to their size difference (Sen *et al* 1998). In the present alloy where both Al and Zr are present as alloying elements, their additive effect towards the increase of deformation faulting is not as pronounced as expected. This may be due to the presence of Mo (*b.c.c.*), whose sole effect is probably to suppress the faulting and increase the stacking fault energy to some extent. The growth fault parameter *b* has been observed to be negative which is similar to the earlier cases for pure titanium and also Ti base Al and Zr alloys. This shows that growth faults are totally absent in this hexagonal system as is also observed in other hexagonal structures (Chatterjee and Sengupta 1975; Chattopadhyay *et al* 1990; Sen *et al* 1995, 1997, 1998). So, this alloy also, like other titanium base alloys investigated by similar technique (Sen *et al* 1995, 1998) can be effectively used in the aviation industry due to its rapid strain hardenability.

4. Conclusion

Alloying of titanium with Al and Zr increased its deformation faulting tendency. The growth fault parameter *b* however, was negligible as was observed in various other hexagonal systems.

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