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I am submitting herewith a dissertation written by Bimal Kumar Kad entitled "MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF BINARY AND TERNARY ALLOYED TiAl". I have examined the final copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirement for the degree of Doctor of Philosophy, with a major in Metallurgical Engineering.



Dr. Ben F. Oliver
Major Professor

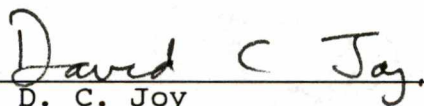
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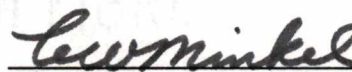


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Vice Provost and Dean
of the Graduate School

MICROSTRUCTURES AND MECHANICAL PROPERTIES OF BINARY AND
TERNARY ALLOYED TiAl

A Dissertation
Presented for the
Doctor of Philosophy
Degree

The University of Tennessee, Knoxville

Bimal Kumar Kad
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ABSTRACT

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1. INTRODUCTION

The TiAl (τ phase) ordered intermetallic compound exists in the Ti-Al system at 773K within the composition range of 50-59.5at% aluminum (1). TiAl, and some dilute alloys with a TiAl base, exhibit superior oxidation and creep properties at 950°C as compared to conventional titanium alloys. In addition, TiAl has a high modulus and low density and thus exhibits significant potential as material for use at elevated temperatures. However, TiAl is currently not in use because of its brittle behavior at temperatures below 600°C. The room temperature tensile ductility is essentially zero and the formability characteristics are poor even at high temperatures.

Efforts are being directed to improve the low temperature ductility of the TiAl compound. Two different approaches to attain this objective would be: (i) developing two phase alloys such that the superior mechanical and high temperature properties of TiAl can be combined with a more ductile matrix providing good formability and (ii) modifying the TiAl single phase with ternary additions thereby attempting to alter the deformation behavior of this compound. The solubility characteristics of several ternary solutes were studied, and based on preliminary results, two ternary solutes, gallium and manganese, were selected for further study.

2. LITERATURE REVIEW

Crystal Structure

TiAl is an ordered compound of the $L1_0$ type, stable up to its melting point (1). It consists of alternate pure aluminum and titanium (001) planes stacked along the c-axis. The structure is face-centered tetragonal with a space group $P4/mmm$, and for a Ti-36.03wt%Al composition, the lattice parameters are $a=0.399\text{nm}$, $c=0.407\text{nm}$, and c/a ratio = 1.02 . Table 1 lists the crystal structures of the different phases in the Ti-Al system, and Table 2 lists the lattice parameters of the different stable phases. Within the range of homogeneity of the TiAl phase, the 'a' parameter decreases linearly, whereas the 'c' parameter increases linearly, with increasing aluminum concentration. The axial ratio c/a increases linearly from 1.017 to 1.026 (2).

Ti-Al Phase Diagram

The most recent compilation of Ti-Al phase diagram was reported by Murray (1), figure 1. The single phase region for TiAl extends between 49 to 55.5 at% aluminum at 500°C . On the aluminum-rich side, the extent of the TiAl field is difficult to determine because of residual coring in the alloys. The uncertainty in the aluminum-rich phase boundary is at least ± 2 at%. At aluminum concentrations lower than 49 at%, TiAl coexists with Ti_3Al (α_2), which is hexagonal Mg_3Cd type. At compositions greater than 56at%Al, TiAl

TABLE 1: CRYSTAL STRUCTURES OF THE PHASES IN Ti-Al SYSTEM

Phase	Homogeneity range, at%Al	Pearson symbol	Space group	structure
α (Ti)	0-45	hP2	P6 ₃ /mmc	A3
β (Ti)	0-47.5	cI2	Im3m	A2
Ti ₃ Al	22-39	hP8	P6 ₃ /mmc	DO ₁₉
TiAl	48-69.5	tP4	P4/mmm	L1 ₀
Ti ₃ Al ₅ (a)	58-63	tP32	I4/mbm	
TiAl ₂	(b)	oC12	Cmmm	
δ	70-72.5	(c)		
TiAl ₃	75	tI8	I4/mmm	DO ₂₂
α TiAl ₃	75	(b,d)		
Al	99.3-100	cF4	Fm3m	A1

(a) Not an equilibrium phase. (b) Not shown on the assessed diagram. (c) Long-period superlattice. (d) Tetragonal. A superstructure of the DO₂₂ lattice.

TABLE 2. LATTICE PARAMETERS OF THE PHASES IN TI-AL SYSTEM

Phase	Composition at% Al	lattice parameters	
		a, nm	c, nm
α TiAl ₃	75	0.3875	3.384
	69-72	0.389	3.392
	72(a)	0.384	3.346
TiAl ₃	75	0.385	0.861
TiAl	(b)	0.40155	0.40625
	(c)	0.3976	0.4049
	46	0.4011	0.4069
	50	0.4004	0.4071
	50	0.3998	0.4076
	55	0.3997	0.4075
	60	0.3999	0.4074
	62	0.3988	0.4081
	52.13	0.3992	0.4073
	55.23	0.398	0.4075
	60.22	0.3975	0.4093
	68.47	0.3957	0.4097
TiAl ₂	67	0.3976	2.436
	67-72	0.398	2.436
	69	0.3917	1.6524
	69	0.392	1.648

(a) Two phase alloys. (b) At Ti₃Al boundary. (c) At TiAl₂ boundary.

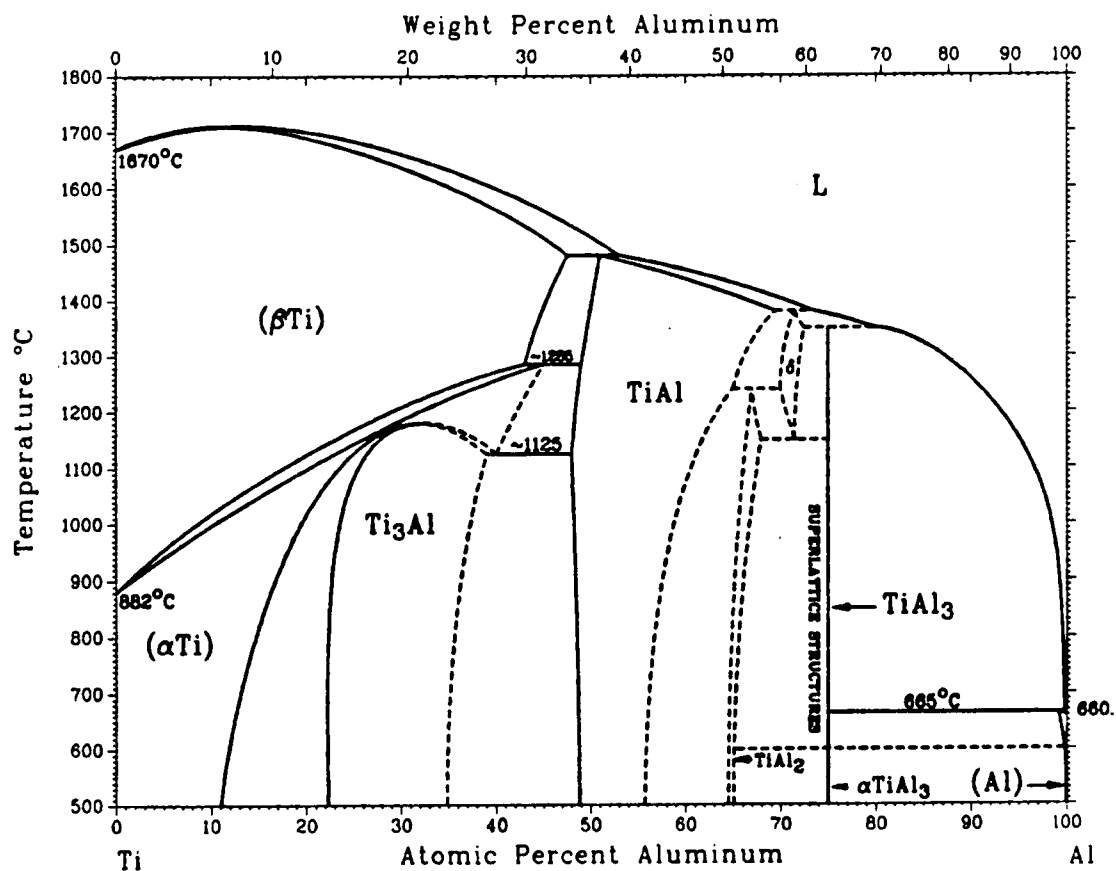


Figure 1. TiAl Phase diagram, Murray's version
 Source: J.L. Murray in "Phase Diagrams of Binary Titanium Alloys", ASM monograph, Ohio, 1987

coexists with TiAl_2 , which is body-centered tetragonal, HfGa_2 type.

In recent years, anomalies have been reported in the high temperature phase diagram in the Ti-50at%Al range. On solidification at various freezing velocities, the phase forming from the melt is α -hcp and not β -bcc as suggested by Murray's diagram (3-6), figure 1. This α is the low temperature hcp solid solution of Ti that is currently being reported as forming directly from the melt (3-6). Though several doubts have been raised as to the metastable existence of α at high temperatures possibly associated with interstitial contamination (3,7), the existence of α has been positively ascertained by high temperature x-ray diffraction measurements (5). This suggests an α +L phase field in the high temperature phase diagram, possibly a peritectic (4). Incidentally, a double peritectic phase field was reported in the original phase diagram by Willey and Margolin (8) in the ASM handbook, figure 2. More recently, Perepezko et al. (9), in a thermodynamic modelling of the Ti-Al system, has further ascertained the nature of the phase diagram, especially pertaining to the existence of the second α +L= τ peritectic, figure 3. Huang et al. (10) and Shull (11), in separate investigations, have reported data on the boundaries of the α + τ phase field.

Within the single phase τ region, several anomalies of second phase particles have been reported. Loiseau et al.

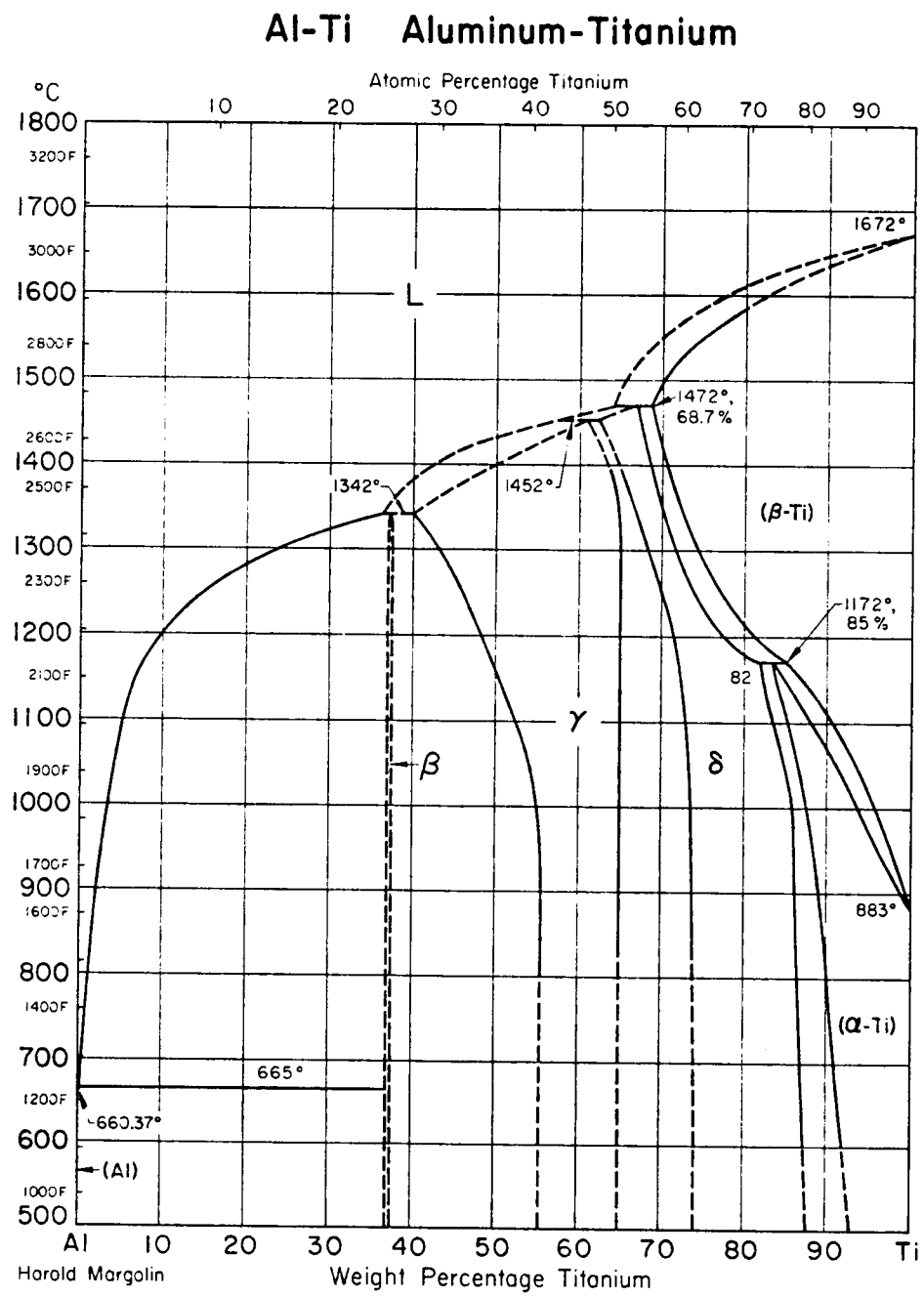


Figure 2. TiAl Phase diagram, Willey's version
 Source: L.A. Willey and H. Margolin in "Metals Handbook",
 ASM Metals Park, Ohio, Vol.8, p.344, 1973.

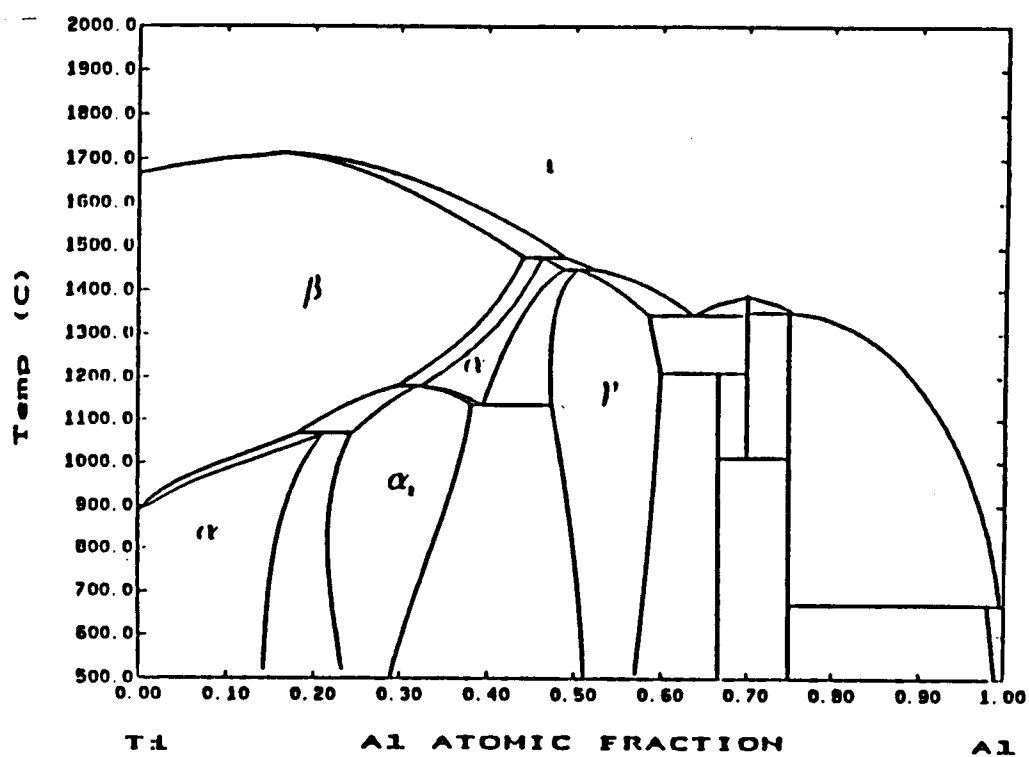


Figure 3. TiAl Phase diagram, Perepezko version
Source: J.H. Perepezko et al. in "MRS Symp. Proc.", Vol.133,
p.127, Boston, MA, 1988.

(12,13) report that there is no composition range, at least at low temperatures, at which TiAl can be obtained as a single phase. They report that a Ti-50at%Al alloy on cooling, decomposed into a mixture of TiAl(τ) and Ti₃Al(α_2) phases. For a Ti-54at%Al composition, τ -TiAl could be obtained as a single phase only at temperatures above 1300°C. At lower temperatures, formation of thin Ti₂Al platelets lying parallel to the (111) TiAl planes was reported (12,13). At heat treatments below 1000°C, these platelets formed particles with a lenticular shape. A hcp structure was proposed with lattice parameters $a=0.300\text{nm}$ and $c=1.4\text{nm}$. Kaufman et al. (14) studied similar particles in TiAl alloys using the Electron Energy Loss Spectra (EELS) method and observed a nitrogen edge in the spectra for the precipitate. Since this precipitate had the same composition and lattice parameters as those described by Loiseau et al. (12,13), it was interpreted as Ti₂AlN. It forms presumably as a result of contamination of the alloy by nitrogen. Kaufman et al. speculated on the fact that the Ti₂AlN phase was observed only in alloys containing greater than 50at%Al. They found only single phase Ti₃Al in their highly nitrogen contaminated Ti-33at%Al alloy. They suggest that all the 'N' is in solution and so the solubility of 'N' in Ti₃Al appears to be much greater than that in TiAl.

Physical Properties of TiAl

Table 3 lists some physical properties of TiAl at ambient temperatures. TiAl has a high stiffness constant comparable to the other conventional titanium base high temperature alloys. Also it has a significantly lower density because of the higher aluminum concentration. TiAl thus is an attractive material by reason of its high specific strength (i.e, ratio of strength to density).

MECHANICAL PROPERTIES OF TiAl

Microhardness Data for TiAl.

Bumps et al. (15) reported that alloys containing 35-49at%Al showed decreasing hardness with an increasing volume fraction of TiAl in the microstructures. The minimum hardness was recorded for the 50at%Al alloy, identified metallographically as single phase TiAl. More recently Tsujimoto et al. (16) reported that hardness decreases with increasing annealing time for γ -TiAl phase, this decrease being attributed to the solution of the harder α_2 -Ti₃Al phase. TiAl is softest about the equiatomic ratio, with hardness increasing at either end with increasing deviation from the Ti/Al = 1:1 ratio (17).

Properties in Compression.

Shechtman et al. (18) reported that polycrystalline specimens of TiAl could be deformed in compression at room temperature to strains of about 8% without any cracking. No compression test data were reported. Tsujimoto et al. (16)

TABLE 3. PHYSICAL PROPERTIES OF TiAl

	TiAl	Ceramic	Metal
Density (gm/cm ³)	3.8	3.2	7.9
Thermal Conductivity (cal/s.cm.K)	0.05	0.07	0.03
Thermal Expansion (X10 ⁻⁶ /K)	10.3	2.8	13.1
Young's Modulus (kgf/mm ²)	17600	31600	22400
Poisson's Ratio	0.28	0.24	0.30
Fracture Toughness (kgf/mm ^{3/2})	42	19	239

reported detailed compression test data on polycrystalline TiAl alloys for a range of temperatures and compositions, table 4, figure 4. A remarkable difference in material performances between the 50at%Al and the 56at%Al alloy is observed, table 4. The strain at fracture exceeds 20% at all temperatures tested in the former alloy while the latter always fractured at strains below 2%. It has been suggested that an increase in hardness of binary TiAl with increasing aluminum (i.e, $Ti/Al < 1$) is accompanied with increasing brittleness of the alloys. Kawabata et al. (19) reported compression test data for single crystal TiAl at several different orientations and temperatures, figure 5. No detail of the exact composition of the alloys was provided. A positive temperature dependence of yield stress with temperature was reported, figure 6. Data on the strain rate sensitivity and the critical resolved shear stress as a function of temperature are shown in figures 7,8.

Properties in Tension.

Limited data are available for the properties of TiAl in tension. Polycrystalline extruded Ti-54.2at%Al alloys were deformed in tension by Lipsitt et al. (20) and their data on strength and ductility are shown in figure 9. Sastry et al. (21) also report tensile properties data for Ti-50.27at%Al alloy.

TABLE 4. COMPRESSION TEST RESULTS AT A STRAIN RATE OF $1.34 \times 10^{-3} \text{ s}^{-1}$.

Temperature (K)	Proof Stress (MPa)	Compression Strength (MPa)	Strain at fracture
a) Ti-50at%Al alloy, As-cast			
R.T	384	1413	22.0
573	377	1544	21.9
773	362	>1216	>24.9
973	353	>880	>52.4
b) Ti-50at%Al alloy, annealed at 1573K for 7.2ks			
R.T	341	>1385	>25.8
573	292	1432	25.2
773	280	>1340	>30.5
973	341	>1069	>41.7
R.T*	288	1575	26.2
* at a strain rate of $2.68 \times 10^{-2} \text{ s}^{-1}$			
c) Ti-56at%Al alloy, annealed at 1573K for 3.6ks			
R.T	320	379	0.4
573	308	439	0.5
773	265	450	2.2
973	309	441	1.1

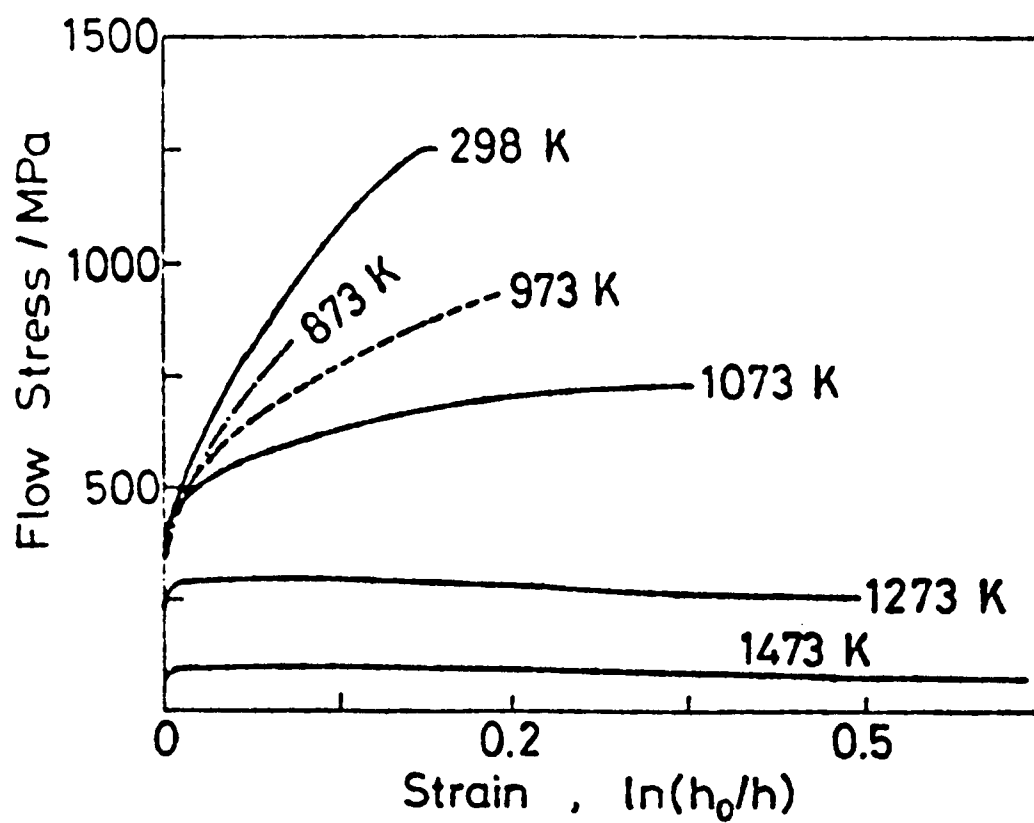


Figure 4. Stress-Strain curves in compression tests of a Ti-51at%Al alloy.

Source: T. Tsujimoto et al. in "Trans. Japan Inst. Metals", Vol.27, No.5, p.341-350, 1986.

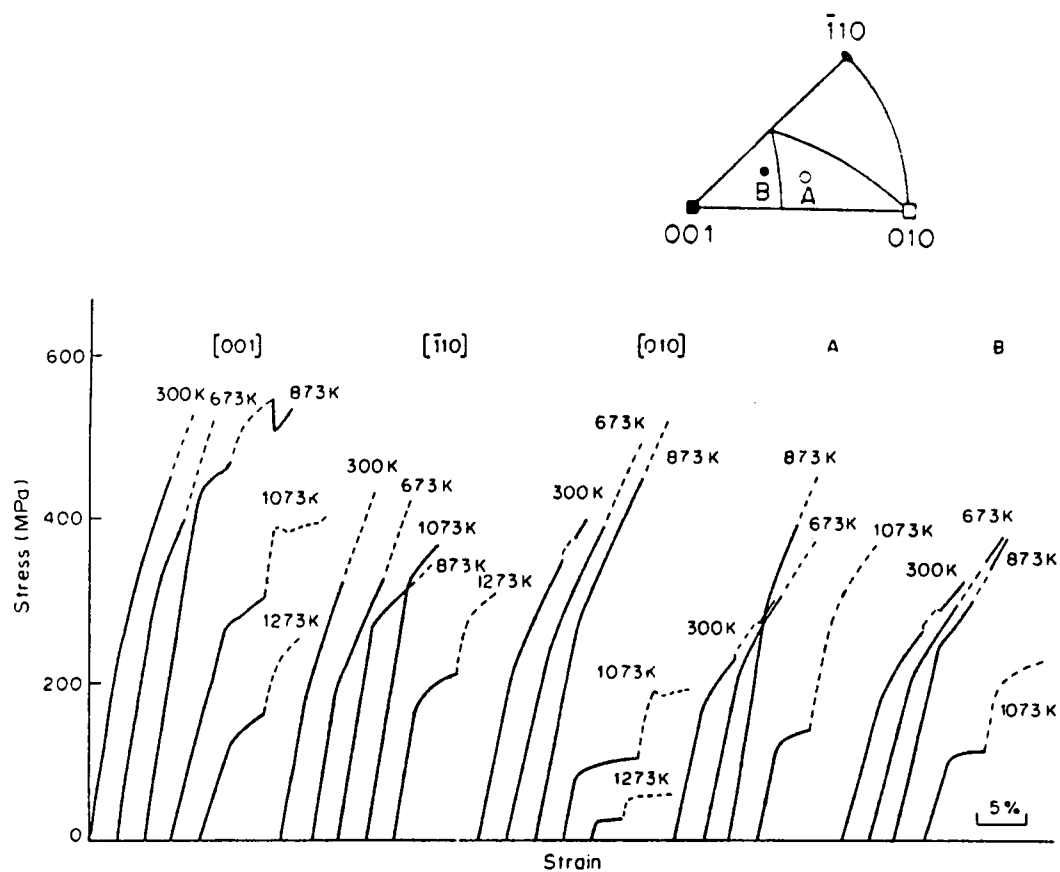


Figure 5. Compressive yield stress data for single crystal TiAl as a function of orientation and composition.
 Source: T. Kawabata et al. in "Acta Met.", Vol.33, No.7
 p.1355-1366, 1985

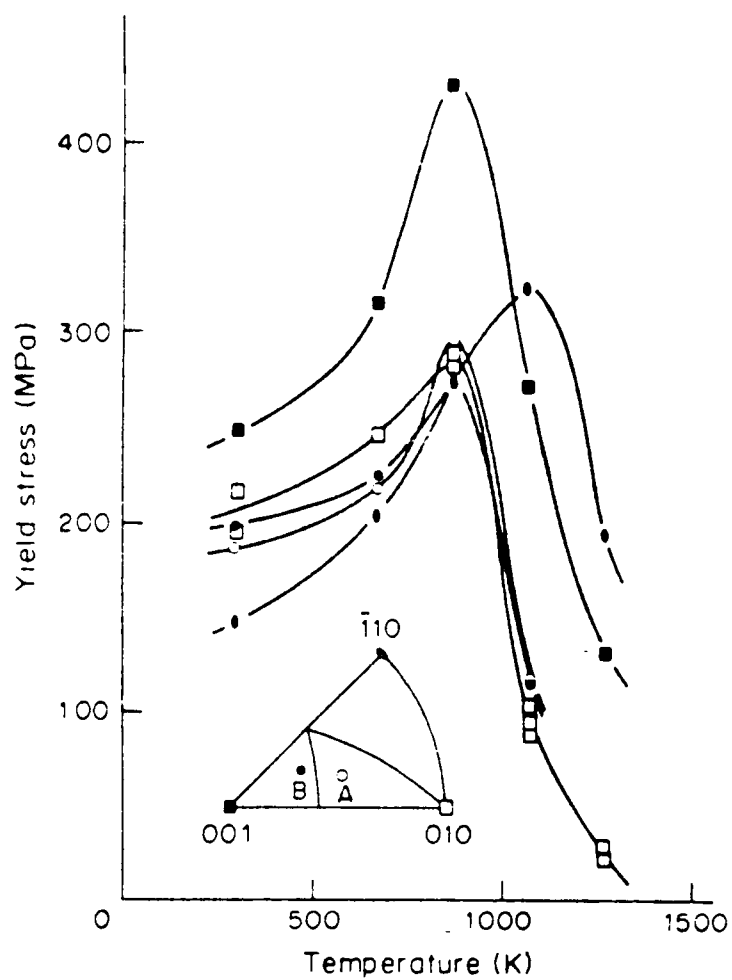


Figure 6. Yield Stress vs Temperature curves in [001], [010] [110], A and B orientations for single crystal TiAl.
Source: T. Kawabata et al. in "Acta Met.", Vol.33, No.7 p.1355-1366, 1985

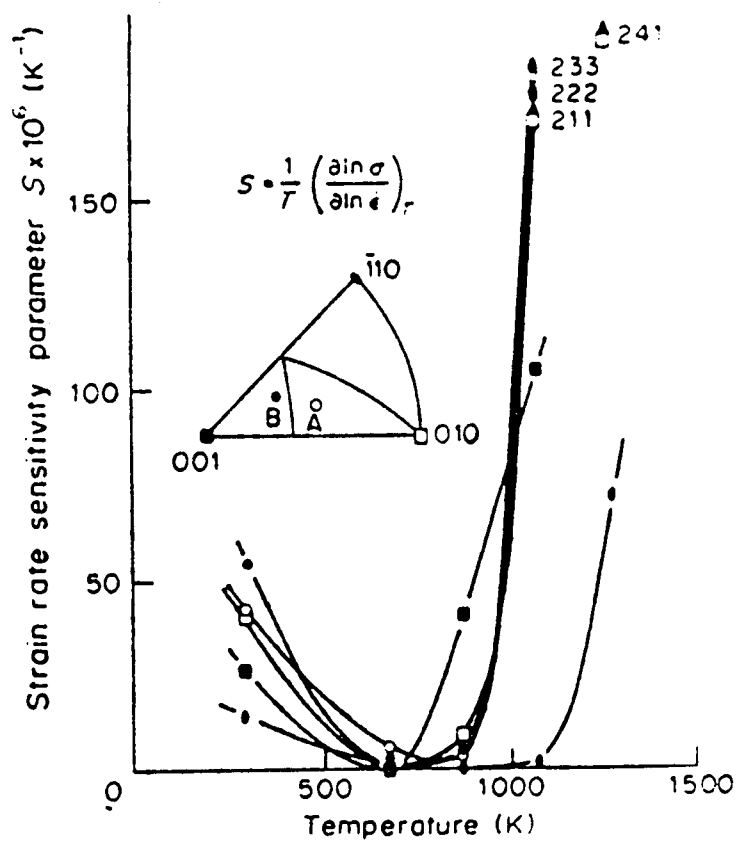


Figure 7. Strain rate sensitivity as a function of temperature and orientation for single crystal TiAl. Source: T. Kawabata et al. in "Acta Met.", Vol.33, No.7 p.1355-1366, 1985

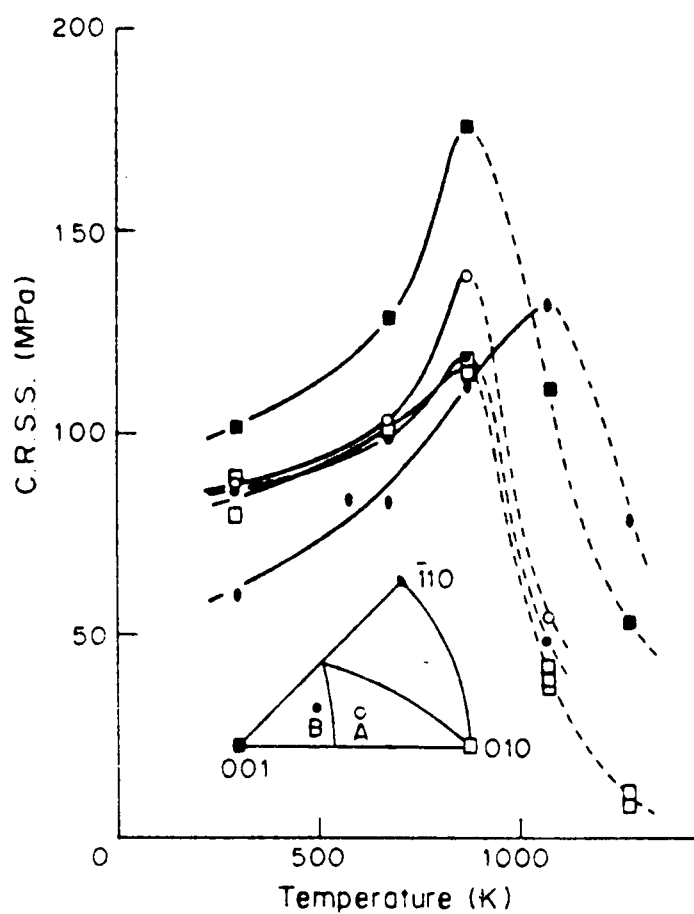


Figure 8. Critical resolved shear stress vs temperature curves in [001], [010], [110], A and B orientations for TiAl single crystal.

Source: T. Kawabata et al. in "Acta Met.", Vol.33, No.7 p.1355-1366, 1985

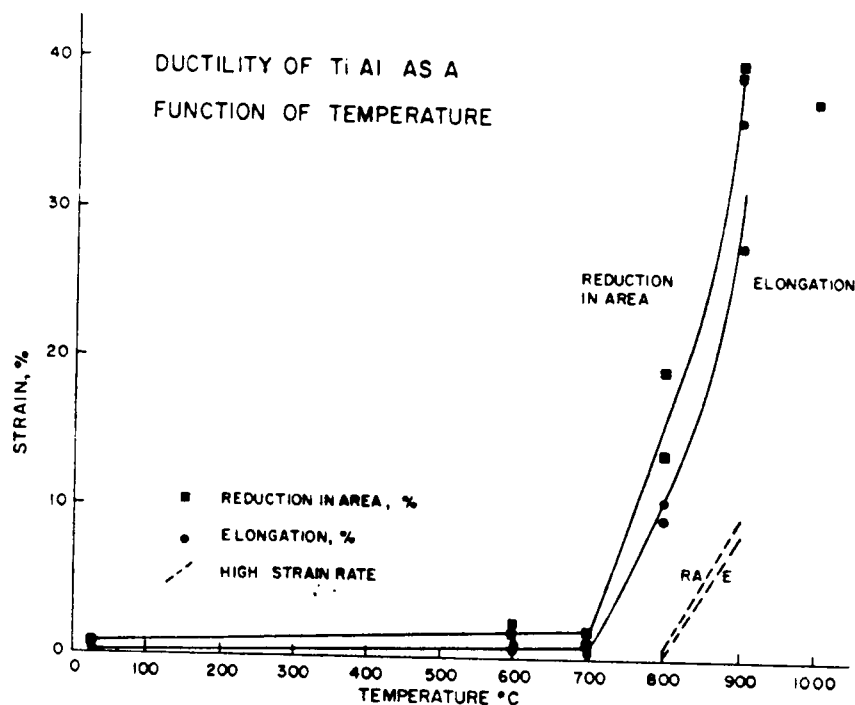
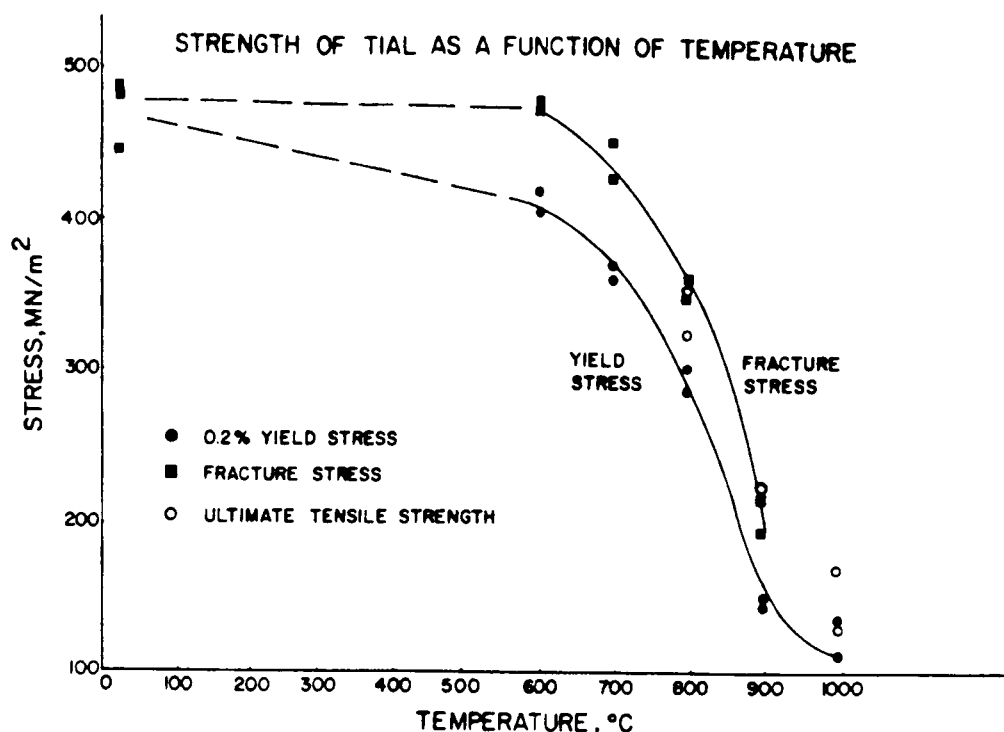


Figure 9. a) Tensile Yield Stress and b) ductility curves for single phase, Ti-54at%Al, polycrystalline alloys, as a function of temperature.

Source: H.A. Lipsitt et al. in "Met. Trans.", Vol.6A, p.1991-1996, 1975

Yield Stress Anomaly in TiAl and its Grain Size Dependence

The yield stress as a function of temperature for TiAl was originally determined by Lipsitt et al. (20). It was shown that, for polycrystalline alloys with an average grain size of $\approx 25\mu\text{m}$, the yield stress at room temperature was 480MPa, and decreased only slightly up to 700°C, above which a marked decrease occurred, figure 9. Kawabata et al. (19) studied the deformation of single crystals of TiAl and report that, in contrast to the results of Lipsitt et al. there is a yield stress anomaly, in that the yield stress peaks at about 600°C, figure 6. Hug et al. (22) contend that this anomaly results from cross-slip of dislocations with Burger's vector (b) given by $b = \frac{1}{2}\langle 101 \rangle$ from the {111} to {100} plane, i.e., Kear and Wilsdorf locks are formed as in the case of $L1_2$ (for example Ni_3Al) compounds. Huang (23) reported no yield stress anomaly for TiAl polycrystalline samples of various compositions with grain sizes varying from 20-50 μm . Vasudevan et al. (24) have reconciled these discrepancies with the following observations. They suggest that First there is a strong dependence of yield stress on grain size at room temperature, given by a Hall-Petch relationship as $\sigma_Y = \sigma_0 + k_Y d^{-0.5}$, where $k_Y = 1.37\text{MNm}^{0.5}$. Second, for Ni_3Al ($L1_2$), where a yield stress anomaly is exhibited, for very fine grained samples this yield stress anomaly is not apparent (25). Since the mechanism responsible for the yield stress anomalies in the two

compounds TiAl and Ni₃Al is considered to be the same (22), this effect is attributed to the strengthening effect of the grain boundaries (24).

Dislocation Structure and Deformation Modes of TiAl

The nature of dislocations in the L1₀ structure was first speculated upon by Marcinkowski et al. (26). Because of alternate Al and Ti (001) planes, two possible $\frac{1}{2}\langle 110 \rangle$ slip vectors exist on the {111} plane. $\frac{1}{2}\langle 110 \rangle$ may be divided into two partials; $1/6[211]$ and $1/6[121]$ with a region of stacking fault and antiphase domain boundary (CSF) between them, but after the second partial the structure is restored, figure 10. $\frac{1}{2}\langle 011 \rangle$ can be divided into two partials $1/6[121]$ and $1/6[112]$. When the leading partial is the $1/6[112]$, there is a stacking fault region between the two partials. However, when the second partial $1/6[121]$ passes, the superlattice is not reformed. A region of antiphase domain boundary is formed. Coupled slip of a second $\frac{1}{2}\langle 011 \rangle$ dislocation will leave the superlattice undisturbed, figure 11. The $\frac{1}{2}\langle 011 \rangle$ dislocations are predicted to slip in pairs, which may be considered a more difficult process. Cass and Spencer (27) report $\frac{1}{2}\langle 110 \rangle$ dislocations and (111) twins as the deformation modes for TiAl. Shectman et al. (18) report that both $\frac{1}{2}\langle 110 \rangle$ and $\frac{1}{2}\langle 011 \rangle$ type dislocations contribute to plastic deformation of TiAl in compression. They observed numerous dislocations of $\frac{1}{2}\langle 110 \rangle$ type and their distribution was relatively random, with only a few examples

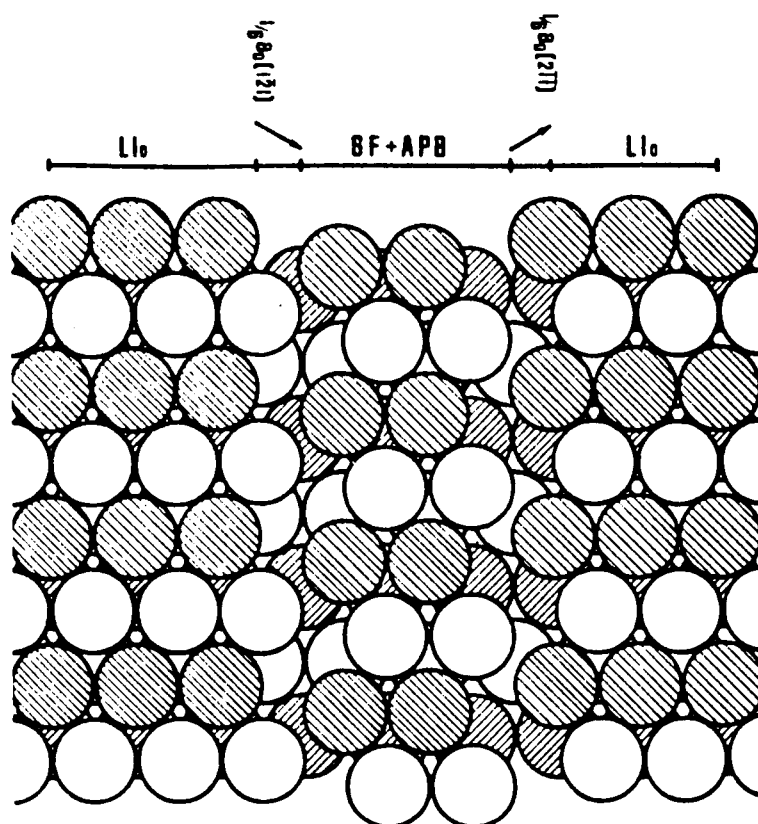


Figure 10. Atomic arrangement on two successive (111) planes of the $L1_0$ lattice for a $\frac{a}{2}[110](111)$ dislocation. Source: D. Shectman et al. in "Met. Trans.", Vol.5, p.1373-1381, 1974.

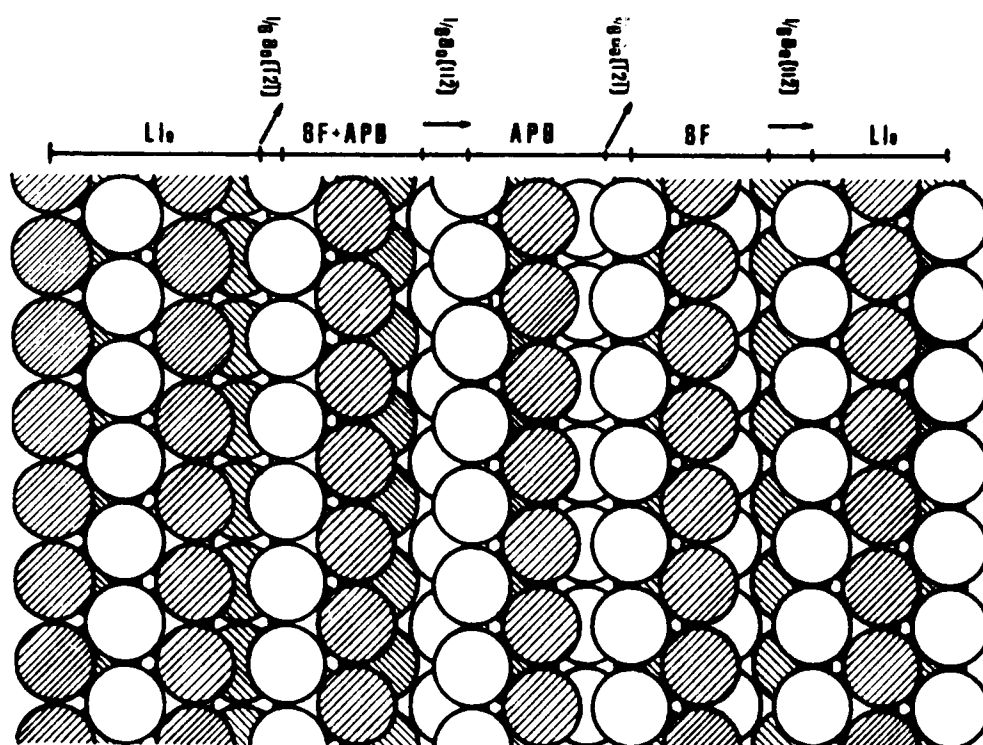


Figure 11. Atomic arrangement on two successive (111) planes of the $L1_0$ lattice showing two $a/2[011](111)$ dislocations. Source: D. Shectman et al. in "Met. Trans.", Vol.5, p.1373-1381, 1974.

of planar groupings. The $\frac{1}{2}\langle 110 \rangle$ dislocations were also a common constituent of subboundaries occasionally found in TiAl. No evidence was observed for the dissociation of the $\frac{1}{2}\langle 110 \rangle$ dislocation into its constituent partials $\frac{1}{6}[211] + \frac{1}{6}[121]$ connected by a ribbon of stacking fault and APB. The $[011]$ dislocations observed sometimes appeared as single lines, complex configurations occurring more randomly. In some cases superdislocations were associated with stacking faults of the $a/3[111]$ type bounded by the $\frac{1}{6}[112]$ and only this type of partial loops. The faults were not formed by the characteristic separation of $\frac{1}{2}\langle 101 \rangle$ dislocation into constituent $\frac{1}{6}[211]$ and $\frac{1}{6}[112]$, but by the movement of the $\frac{1}{6}[112]$ segment only. Doubts were expressed as to whether these faults were formed by the expansion of these loops under the applied shear stress or were drawn out by the pinning of the $\frac{1}{6}[112]$ segment of an $\frac{1}{2}\langle 101 \rangle$ dislocation. Lipsitt et al., (20) report that for room temperature tensile tests, $\frac{1}{2}\langle 110 \rangle$ dislocations are the major mode of deformation, with superdislocations of the $\frac{1}{2}\langle 011 \rangle$ type showing only limited activity, their motion being impeded by the pinning of the trailing $\frac{1}{6}[112]$ partial by an unknown pinning agent. This pinning effect vanishes at temperatures of 700°C and above. The superdislocations are no longer impeded by the trailing $\frac{1}{6}[112]$ partial dislocation dipole and may contribute significantly to plastic deformation. It is suggested that $\frac{1}{6}[112]$ partial dislocations control the plasticity of

TiAl. Mobility of these partials control the mobility of $2/3$ of the dislocations in TiAl as well as the thickening of the $[112](111)$ twins. Thus the temperature range $700-800^{\circ}\text{C}$, where $1/6[112]$ partials become unpinned, would be a turning point in the ductility of TiAl, figure 9. In contrast to this observation, Kawabata et al. (19) on their compression tests on single crystals, have reported that the critical resolved shear stresses in the orientations operating the $\frac{1}{2}\langle 110 \rangle$ dislocations are not always smaller than those in the orientations operating the $a\langle 011 \rangle$ superdislocations. Based on specific orientation $[010]$ compression axis, an orientation which would apply equal critical resolved shear stress to both $[011]$ and $[110]$, they observed that mobility of both dislocations was nearly at the same level. Greenberg (28,29) reported that both sessile and glissile configurations could result from the cross slip of one of the end partials of the $[011]$ superdislocation. Hence the pinning of the $1/6[112]$ partial could be an intrinsic effect. It is known that extrinsic effects, such as a small alloying addition, cause profuse pinning of the $1/6[112]$ partial dislocations. Hug et al. (30) in 1986 reported observing superdislocations of the $\frac{1}{2}\langle 112 \rangle$ type. Greenberg (28,29) had postulated this dislocation for the $L1_0$ structure earlier. The splitting of this superdislocation is discussed in a later paragraph. Kawabata et al., (31) confirmed the presence of $\frac{1}{2}\langle 112 \rangle$ dislocation for TiAl single crystal specimens deformed in compression at -196°C . $\frac{1}{2}\langle 110 \rangle$

type dislocations were rarely observed thereby suggesting that they may be immobile at -196°C .

Slip Systems in the L10 TiAl Structure

The close packed $\{111\}$ planes are the slip planes in the L10 structure. The Burgers vectors of the dislocations operating on the $\{111\}$ planes are in the order of increasing length; $\frac{1}{2}\langle 110 \rangle$, $\frac{1}{2}\langle 112 \rangle$, $\frac{1}{2}\langle 101 \rangle$.

$\frac{1}{2}\langle 110 \rangle$ is the usual slip vector of the disordered lattice and it joins the first nearest neighbours. $\frac{1}{2}\langle 112 \rangle$ joins the third nearest neighbours. $\frac{1}{2}\langle 101 \rangle$ joins the fourth nearest neighbours.

$\frac{1}{2}\langle 112 \rangle$ and $\frac{1}{2}\langle 101 \rangle$ dislocations are usually called superdislocations since their dissociation produces an APB ribbon. Dislocations with $\frac{1}{2}\langle 110 \rangle$ or $\frac{1}{2}\langle 101 \rangle$ Burgers vectors may slip in two different $\{111\}$ planes while dislocations with $\frac{1}{2}\langle 112 \rangle$ burgers vectors can only slip in one $\{111\}$ plane. The possible splitting sequence of the three types of dislocations are:

$$1/2[110] \rightarrow 1/6[2\bar{1}\bar{1}] + \text{CSF} + 1/6[121]$$

$$[011] \rightarrow 1/6[\bar{1}12] + \text{SISF} + 1/6[121] + \text{APB} + 1/6[\bar{1}12] + \text{CSF} + 1/6[121]$$

As the energy of the APB and CSF are expected to be very high compared to the SISF energy, this might reduce to

$$[011] \rightarrow 1/6[\bar{1}12] + \text{SISF} + 1/6[154]$$

$1/6[\bar{1}12]$ is the only partial generating a stacking fault. Another splitting reaction suggested by Greenberg (29,30) has no stacking fault formation, i.e.,

$$[011] \rightarrow 1/2[110] + 1/2[\bar{1}12]$$

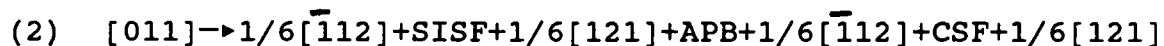
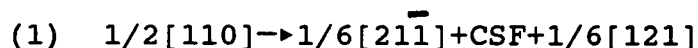
At this step of the decomposition there is only a slight energy drop, since the resulting burgers vectors are orthogonal. However, further dissociation of the $\frac{1}{2}[\bar{1}12]$ segment may provide the driving force for this reaction as shown below.

$$1/2[\bar{1}12] \rightarrow 1/3[\bar{1}12] + \text{SESF} + 1/6[\bar{1}12]$$

The $\frac{1}{2}[110]$ dislocations are never dissociated, since this would produce a complex stacking fault having a high energy. The $[011]$ and $\frac{1}{2}[112]$ dislocations do not split all over the dislocation line, but only locally. This splitting occurs in the slip plane, as described above. In all cases, dissociation of the different superdislocations leads to the same extrinsic faults, limited by the $1/3[112]$ and $1/6[112]$ partials. This dissociation results from the rearrangement of jogs which pin the superdislocations. The rearrangement occurs by cross-slip of one partial of the superdislocation followed by a redissociation in the slip plane. This mechanism leads to an extrinsic faulted loop surrounded by a sessile $1/6[112]$ partial. It must be noted that although extrinsic and intrinsic faults have similar energies, the observation of an extrinsic fault is unusual since its formation requires the gliding of two partials on two successive planes.

As in the $L1_2$ structure, intrinsic/extrinsic superlattice stacking faults, complex stacking faults and antiphase boundaries are potential planar defects in the $L1_0$

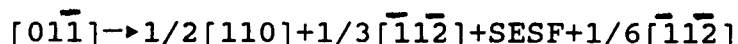
structure. Dislocations $\frac{1}{2}\langle 110 \rangle$ and $\langle 101 \rangle$ in their fully disordered state involve the above mentioned planar defects according to the reactions:



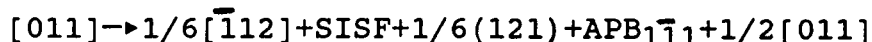
Whereas reaction 1 is restricted to $\{111\}$ planes, reaction 2 may give rise to a non-planar configuration since the APB is liable to stabilize on any low-energy plane Hug et al., (22,30) report that whatever their Burgers vectors (i.e. $\frac{1}{2}\langle 112 \rangle$, $\frac{1}{2}\langle 110 \rangle$ or $\langle 101 \rangle$), there is a tendency for the dislocations to be aligned along the $\langle 110 \rangle$ dense directions, which suggests the influence of strong Peierls forces. Faulted dipoles are frequently observed.

Room Temperature Dissociations

The dissociation reaction for the formation of dipoles was shown as (22):



At the weak beam scale, in the electron microscopy investigations, $\langle 101 \rangle$ dislocations appear to be dissociated into three partials according to the reaction

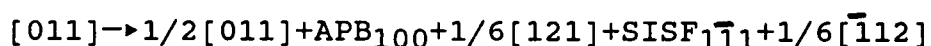


Both planar defects lie on the (111) glide plane.

High Temperature Dissociation

In comparison with the room temperature deformation, TiAl exhibits a different microstructure on straining at

600°C. The dislocations observed are exclusively of the type $\frac{1}{2}\langle 110 \rangle$ and $\langle 101 \rangle$ types. The $\langle 101 \rangle$ dislocations are present as elongated screw dislocations, indicating that they are sessile at the peak flow stress temperature. Faulted dipoles are not observed at elevated temperatures (20). Hug et al. (30) report that, rather than being the consequence of increased mobility of the $\frac{1}{6}\langle 112 \rangle$ partials, as postulated earlier (18,20), the absence of faulted dipoles is ascribed to a change in the core configuration of $\langle 011 \rangle$ superdislocation. Hug et al. (30) report the following dislocation dissociation reaction



The difference from the room temperature dissociation reaction is that the APB lies in a $\{100\}$ plane instead of the $\{111\}$ plane to which its companion SISF belongs. Superdislocation fine structure at 600°C bears similarities to the core structure of superdislocations in the $L1_2$ structure as hypothesized earlier (32). The plastic behavior of $L1_2$ is controlled by the formation of Kear-Wilsdorf locks which require a driving force for the $\frac{1}{2}\langle 011 \rangle$ superpartials to cross slip from the octahedral glide plane to a cube plane. As with the $L1_2$, such a driving force originates in $L1_0$ by the APB energy being more favorable on $\{100\}$ than $\{111\}$. In crystals for which anisotropy is >1 , the elastic anisotropy may reinforce this tendency because of the torque which is exerted on each $\frac{1}{2}\langle 011 \rangle$, out of $\{111\}$ and towards $\{010\}$ (33).

Twinning as a Deformation Mode in TiAl

Cass and Spencer (27) first reported the presence of (111) mechanical twins in TiAl. Similar twins were also reported to be the predominant mode of deformation for CuAu (L10 structure) by Pashley et al. (34). Shectman et al. (18) report the formation of twins in powder extruded specimens, ranging from faults to relatively large twins. These twins were identified as (111)[112] type and only of this type. This is in agreement with Pashley et al. who pointed out that the other potential twin system, (111)[211], would destroy the symmetry of the lattice. Since TiAl has a high APB energy, one may predict that glide elements which produce no disorder within the lattice would predominate. Shectman et al. (18) also reported that growth of twins was confirmed to occur by the movement of the partials at the twin interface, a process which was simulated by heating the specimen in the electron microscope.

Twinning plays an important role in the plastic deformation of TiAl. Only four types of twins are available and the equi-schmid factor lines for them are shown in figure 12. Compressive load along the [001] direction will activate all four twinning systems. However, a tensile load in the [001] or [110] will activate only two twin systems at a time. This fact may contribute to the difference in ductility in tension and in compression. Lipsitt et al. (20) reported cross twinning in powder extruded TiAl in tensile

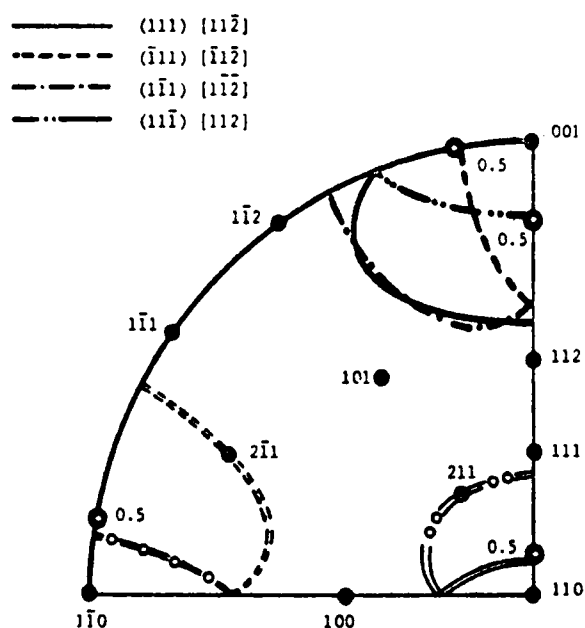


Figure 12. Equi-Schmid factor lines for {111}<112> twinning dislocations. The lines indicate directions along which Schmid factor equals 0.4 and the 0.5 points are also indicated. Single lines are for compression and double lines for tensile load.

Source: D. Shectman et al. in "Met. Trans.", Vol.5, p.1373-1381, 1974.

tests at 700°C. A [112](111) twin crosstwins onto another plane of the same family when it intersects with another twin of the same kind. This process was observed many times, but only over a narrow temperature range. Lenticular twins formed at 800°C. TEM observations indicate that twinning is a major mode of deformation at this temperature. The lenticular twins increased in width with increasing temperature. The twinning strain in TiAl is considered to be fairly low since they are present right from the initial stages of the deformation process. Hug et al. have related their observation of extrinsic faults in TiAl to the easy twinning strain of TiAl. This is in accordance with the earlier observation by Christian (35) that ease of formation of twins is related to the stacking fault energy. More recently Vergnol et al. (36) have shown that twinning is initiated by the nucleation and extension of an extrinsic stacking fault.

Dislocation Mobilities/Immobilities in TiAl Structure

It was shown by Vasudevan et al. (24) that in TiAl there is a strong dependence of yield stress on grain size. In that work it was shown that a reasonably linear variation between yield stress and $d^{-0.5}$, where d is the average grain diameter, existed and from this analysis it was found that the Hall-Petch parameter, K_y , was given by $K_y = 1.37 \text{ MPam}^{0.5}$. This is a relatively large number for this parameter and similar to that of α -Fe (51). Such large values of the

parameter K_y are usually attributed to materials which have a strong locking of dislocations; for example, in α -Fe, the locking is a result of a strong interaction between interstitial impurities and dislocations in the BCC structure (52). Alternatively, a limited number of slip systems, which implies an orientation effect, may be used to account for strong dependencies on grain size, although for such materials, such as the hcp metals, the values of K_y tend to be near to that of Ti (i.e., $K_y=0.41$, (53)). It is concluded that the large values of K_y in TiAl imply that there is a strong locking of dislocations, which inhibits dislocation motion.

The origin of this locking is of interest. One possible cause for the locking of dislocations is core dissociation or spreading (54). For example, Hug et al. (30) have shown that extrinsically faulted dipoles may form by dissociation of dislocations with $b=\langle 101 \rangle$, or $b=\frac{1}{2}\langle 112 \rangle$. Also, as the temperature increases, there is a tendency for cross-slip of dislocations with $b=\langle 101 \rangle$, normally dissociated into a triplet on the $\{111\}$, in to the $\{010\}$ planes, where the core of the partial with $b=\frac{1}{2}\langle 101 \rangle$ subsequently spreads. Although these dislocations with $b=\langle 101 \rangle$ may therefore be involved in dissociations which result in the formation of sessile components or configurations, this does not explain why dislocations with $b=\frac{1}{2}\langle 110 \rangle$ do not contribute to strain at ambient temperatures, as implied by the flow stress anomaly.

An alternative explanation for the locking of

dislocations in this structure has been put forward by Greenberg et al. (43) involving the anisotropy of charge density about Ti atoms. Calculations of the charge densities around Ti atoms have been performed using the linear combinations of muffin tin orbitals method (55). The calculations suggest that significant anisotropy in the charge density about Ti atoms exists, for example, in the {001} layers containing only Ti atoms, such that directional bonding between the Ti atoms along the $\langle 110 \rangle$ directions in these layers develops; in addition, such directional bonding is expected to develop between Ti atoms lying in the $\langle 001 \rangle$ directions. It is concluded that while there is a general tendency to form covalent bonds in the compound, strong directional bonds are expected to form between atoms in any plane that contains only Ti atoms, such as {001}, {110} and {112}; Greenberg et al. (43-45) have made predictions regarding the anisotropy in the Peierls stress that would result from such covalent bonding. Thus, in general, any dislocation segment lying normal to planes with layers containing only Ti atoms, where a significant degree of covalent bonding between the Ti atoms is expected, will have small widths and therefore, on the basis of Peierls-Nabarro theory experience a large Peierls stress. This analysis may be used to predict that certain dislocation segments in TiAl will tend to be sessile or exhibit reduced mobility because of the anisotropy in the Peierls stress. It is expected then that dislocation segments lying close to $\langle 001 \rangle$, $\langle 110 \rangle$ and

$\langle 112 \rangle$ will lie in deep Peierls valleys and therefore tend to be relatively sessile. Greenberg et al. (43-45) point out that the degree of directionality of the bonds depends on the distance between adjacent sections, which means the appropriate interlayer spacing; if this is small, significant Ti-Al interactions between the layers may occur and effectively reduce the covalency. Therefore, the strengths of bonds which develop in sheets of Ti atoms parallel to the three types of planes will vary, and so on this basis the Peierls stresses for dislocation segments parallel to $\langle 110 \rangle$ are expected to be stronger than those for $\langle 112 \rangle$. On the basis of this analysis, the effect of covalency on the Peierls stress of various dislocations may be predicted, as listed in Table 5. As can be seen, screw dislocations with $b = \frac{1}{2} \langle 110 \rangle$ will tend to be sessile with the edge segments less strongly locked. Edge and screw segments of dislocations with $b = \langle 101 \rangle$ are not affected by the directionality of bonding, but 60° and 30° segments lying parallel to $\langle 110 \rangle$ and $\langle 112 \rangle$, respectively, will tend to be sessile, although screw segments will be affected to a lesser degree than those of the edge character. The effect of temperature is to reduce the effective depths of the Peierls valleys by thermal activation and so the various dislocation segments are expected to exhibit increased mobility.

TABLE 5. PREDICTIONS OF THE EFFECTS OF COVALENCY ON THE MOBILITY OF DISLOCATIONS IN TIAL.

Slip System	Character	Line direction	Comments
$\frac{1}{2}\langle 110 \rangle \{111\}$	screw edge	$\langle 110 \rangle$ $\langle 112 \rangle$	Deep Peierls valley Significant valley
$\langle 101 \rangle \{111\}$	screw edge 60° 30°	$\langle 101 \rangle$ $\langle 121 \rangle$ $\langle 110 \rangle$ $\langle 112 \rangle$	Not affected Not affected Deep Peierls valley Significant valley
$\frac{1}{2}\langle 112 \rangle \{111\}$	screw edge	$\langle 112 \rangle$ $\langle 110 \rangle$	Significant valley Deep Peierls valley

The term "deep (or significant) Peierls valley" indicates that the dislocation segment is perpendicular to a plane containing sheets of Ti atoms, the atoms in which are thought to have the tendency to develop strong covalent bonding; deep and significant are used to indicate an approximate magnitude of the Peierls valleys. The term "not affected" is taken to mean that the segment of dislocation is not so oriented.

Ductile Enhancements via Two Phase Microstructures

Several recent reports have cited improved ductility for two phase $\text{Ti}_3\text{Al}(\alpha_2) + \text{TiAl}(\tau)$ alloys (37-40). Kawabata et al. (41) contend that ductility is improved by the use of high purity elements in Ti-48at%Al alloy and the presence of the second phase lamellae of $\text{Ti}_3\text{Al}(\alpha_2)$. Huang et al. (42) observed similar improvements in ductility in Ti-48at%Al alloy when compared to Ti-52at%Al alloy. They also reported observing the $\frac{1}{2}\langle 110 \rangle$ dislocations only in the former composition and not in the latter. This would suggest that the increased number of slip systems available contribute to additional deformation.

Recently attempts have been made to reconcile the difference in dislocation observation as a function of alloy chemistry by using Greenberg's model of covalent Ti-Ti bonds in the L1_0 structure. Greenberg's model (43-45) suggests that an anisotropic state of stress exists in the L1_0 lattice because of the strong nature of the Ti-Ti bonds. This results in significant Peierls valleys for dislocations of the $\frac{1}{2}\langle 110 \rangle$ and $\frac{1}{2}\langle 112 \rangle$ type thereby rendering them sessile. Vasudevan et al. (46) contend that since Ti_3Al has a significantly higher solubility for interstitials than TiAl (14), the presence of Ti_3Al may act in a way to internally getter the interstitials from the TiAl . Hence it will be the effect of reduction in the interstitial content which causes enhanced ductility. Vasudevan et al. (47) have shown that when interstitials are internally gettered from

single phase TiAl by alloying with erbium, dislocations with $b=\frac{1}{2}\langle 110 \rangle$ dominate the deformation microstructure. However, for nominal purity alloys, these dislocations are sessile. Court et al.(48) report that dislocations with $b=\frac{1}{2}\langle 110 \rangle$ are sessile at room temperature because of a large Peierls stress which results from the anisotropy of the charge density about the Ti atoms in TiAl. This anisotropy of the Peierls stress has a less important effect on dislocations with $b=\langle 101 \rangle$. Hence for single phase TiAl of nominal purity, strain is accomplished by limited glide of the dislocations with $b=\langle 101 \rangle$, their activity being limited not only by the covalency but also by the formation of extrinsically faulted dipoles (22) and twinning. Vasudevan et al. contend that Ti_3Al behaves in a similar manner as erbium containing TiAl, and have shown that dislocations with $b=\frac{1}{2}\langle 110 \rangle$ would be more glissile in two phase mixtures $\text{Ti}_3\text{Al}+\text{TiAl}$ than in single phase TiAl.

Ductility Improvements by Ternary Alloying

Limited data are available for ductility enhancement of the single phase γ by ternary additions. However, since improved ductility has been observed for binary two phase alloys, any improvement in ductility by ternary alloying should take into account the microstructure, i.e., single phase or two phase. Unfortunately current data are essentially lacking in this regard. Table 6 lists the combined mechanical properties data compiled for binary and

TABLE 6. MECHANICAL PROPERTIES OF γ TITANIUM ALUMINIDES

Alloy	RT Strength, MPa		Elongation %		BDT °C
	Y.S	U.T.S	RT	800°C	
Ti-54Al	490		<1	10	700
Ti-52Al	350		.1-1.1	20	600
Ti-52Al			1.8		
Ti-48Al	390/520	483	.3-2.1*	40	550
Ti-46Al	501	592	.7		
Ti-44Al	748	748	0.3		
Ti-50Al-1V			1.3		
Ti-48Al-1V	400	507	2.3		
Ti-48Al(1-3)V	520		1.5-3.5		550
Ti-44Al-1V	754		.6		
Ti-48Al(1-3)Mn	500		1.5-3.5		
Ti-48Al(1-3)Cr	520		1.5-3.5		
Ti-48Al-2W	547	616	1.3		
Ti-48Al(1-3)X			<1.8		
(X=Nb, Ta, Hf)					

BDT Brittle-ductile transition temperature.

*Elongation = 2.7% for high purity (300 ppm O₂)

ternary TiAl alloys.

Fracture Behaviour of TiAl

TiAl is a brittle material at ambient temperatures and has a cleavage fracture mode (20). The river pattern observed indicates that fracture initiates at or near the specimen surface. This brittle behavior is observed up to temperatures of 600°C. At 700°C the fracture surface shows both cleavage and scattered indications of ductile fracture. However the major change is in the propensity for separation at the grain boundaries. At 800°C the tendency to intergranular fracture becomes pronounced. Areas that exhibit transgranular failure show considerable evidence of ductility while transgranular cleavage is seen only in scattered grains. At 900°C the fracture is ductile and only a few instances of intergranular separation were observed. The fracture behavior at 1000 °C is similar to that observed at 900°C. This brittle to ductile fracture transition is also evident in figure 9. The ductility of TiAl increases rapidly at temperatures beyond 700°C in accordance with the fracture behaviour changing from brittle to ductile mode. This change in the fracture mode was attributed to the increased mobility of the $\frac{1}{2}[011]$ superdislocations, as described in an earlier section.

Kawabata et al. (49) studied the fracture behavior of TiAl single crystals with the [010] and [001] orientations

by bending tests. Fractography revealed small facets indicative of cleavage fracture on the $\{010\}$, $\{001\}$, $\{110\}$, and $\{101\}$ (or $\{011\}$). Besides, fracture on the $\{111\}$ slip planes was frequently observed. These fracture planes have not been reported in FCC metals and alloys. In TiAl the cleavage fracture initiated at a portion about 50-100 μm beneath the specimen surface. Kawabata et al. imply that cleavage fracture occurs by dislocation reactions and have proposed the possible dislocation reactions (49,50).

3. EXPERIMENTAL PROCEDURE

The experimental procedure is divided into several subsections.

Alloy Preparation

1) For Directional Solidification

The alloys were prepared by arc melting under an argon atmosphere and drop cast into a water cooled cylindrical copper mold 2.5cm diameter X 15cm long. These drop cast bars were used as starting material for directional solidification. The essential parameters of this operation are a slow growth condition (2.0cm/hr maximum growth speed used in this study) of an inductively levitated crucibleless melt. The liquid zone is inductively stirred providing even compositional control across the solid-liquid interface. This mixing also allows for possible nucleation of dissolved gaseous species that may easily nucleate and bubble out. Melt processing was carried out in Pd-purified hydrogen and helium atmospheres.

2) For Preliminary microstructural and mechanical properties

All alloys used in this study were prepared from individual elements of 99.99% or better purity. Alloy buttons were arc cast under a constant 30 cubic feet/hour argon flow. The buttons were remelted at least six times, to ensure better mixing. The weight loss was less than 1 wt% for all alloys accepted for further study. The actual

compositions were assumed to be the same as the intended compositions.

3) For Tensile Mechanical Properties of Ternary Alloys

Alloy specimens for tensile mechanical properties were generated from two sources. For high purity, large grain size material, the alloys were directionally cast in the zone melter as explained above. For polycrystalline tensile specimens, the alloys were drop cast into a water cooled copper mold of 2.5cm X 1.25cm rectangular cross section and 12.5cm height.

Thermo-Mechanical Processing of Alloys

For preliminary studies, arc-cast alloys were wrapped in tantalum foil and encapsulated in silica tubing under a partial pressure of argon. Alloy specimens were subsequently homogenized at 1373K for 100 hours, unless otherwise stated, and air cooled after a gradual withdrawal from the furnace.

For polycrystalline tensile specimens, the alloys were homogenized in vacuum at 1373K for 16 hours. They were then canned in carbon steel and hot forged in 5-10 steps, i.e., soak at 1373K for 1 hour, hot forge and then repeat the process to a total reduction in thickness of 25%.

Optical Microscopy

All optical microscopy was performed on samples etched with a 5%HF+5%HNO₃ mixture based in glycerine or distilled water. A water based etchant has a quicker response and was

preferred for the homogenized alloys that were largely single phase τ .

Transmission Electron Microscopy

TEM investigations were performed on Phillips EM300 and Hitachi S-800 electron microscopes. The compositional analyses in the TEM were performed using the Link system X-ray Energy Dispersive Spectrometer (EDS) on the S-800. TEM thin foils were prepared using an electropolishing solution of 30ml perchloric acid, 175ml n-butyl alcohol and 300ml methyl alcohol ratio. The foil thinning operations were carried out on Tenupol at 40-80V at 223K.

Scanning Electron Microscopy

All SEM investigations were carried out on a Cambridge S-360 Stereoscan microscope. All composition analysis in the SEM were performed using the Link X-ray EDS system. This SEM instrument was also equipped for electron channeling studies performed in this study.

Microhardness Measurements

Microhardness measurements were made on a Kentron microhardness tester using a diamond pyramid indenter at a 100 gram load. To ensure careful handling of the brittle materials, a slow loading rate was used. Typically this implies a 30 second interval while the load is slowly being applied. All hardness indentations were made within the

single phase τ region to monitor the effect of ternary additions on the τ phase.

Tensile Specimen Preparation

The tensile specimens were spark machined from the stock material in the configuration of an ASTM E-8 specification, proportionally reduced to the dimensions as shown in figure 13. Typically the spark machined specimens were a little oversize, and were reduced to their final dimensions, figure 13, by hand polishing to $\approx 9\mu\text{m}$. They were given a final electropolishing treatment in a similar solution as was used for polishing TEM thin foils.

Tensile Testing

All tensile testing reported in this study was performed on an instron equipped with a atmosphere control chamber. The specimens were heated to the testing temperature using a split wound furnace around the specimen gage length. Testing was performed in air for the room temperature tests, and in vacuum for the high temperature tests. Two strain rates, $6.67 \times 10^{-4} \text{sec}^{-1}$ and $3.33 \times 10^{-3} \text{sec}^{-1}$, were used.

Control System for Directional Solidification Process

The directional solidification (DS), also referred to as the zone melt refiner is shown in figure 14. Figure 15 shows a close up view of the inductively levitated and mixed

ASTM E8-66

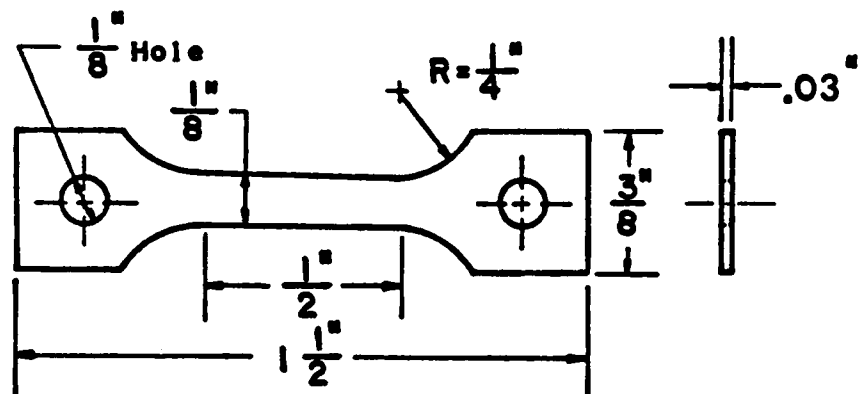


Figure 13. ASTM E-8 specimen geometry used for tensile tests.

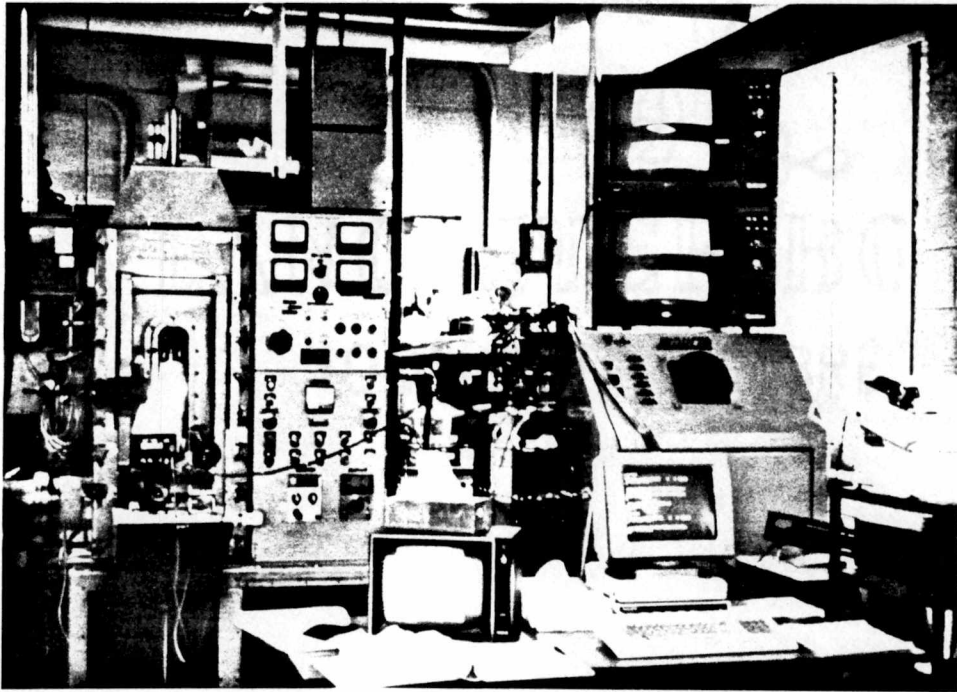


Figure 14. Directional solidification equipment setup.

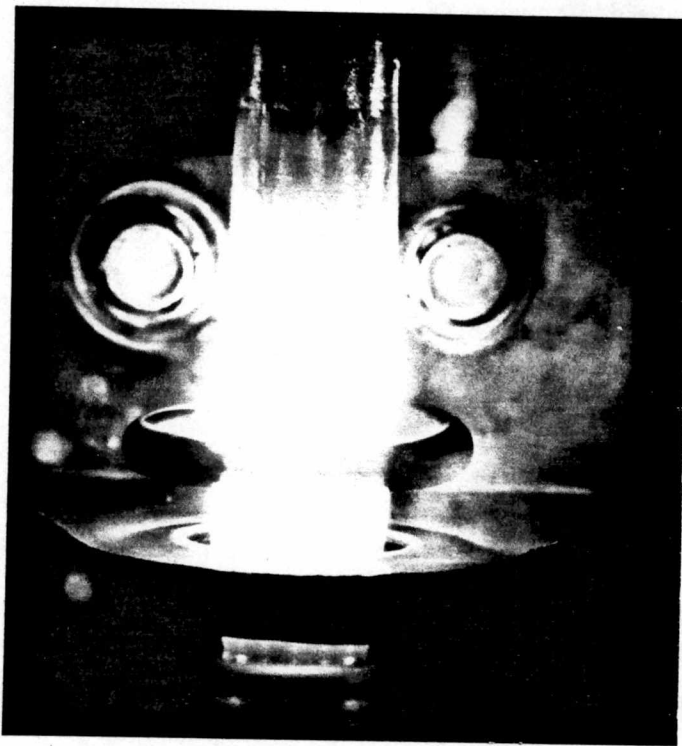
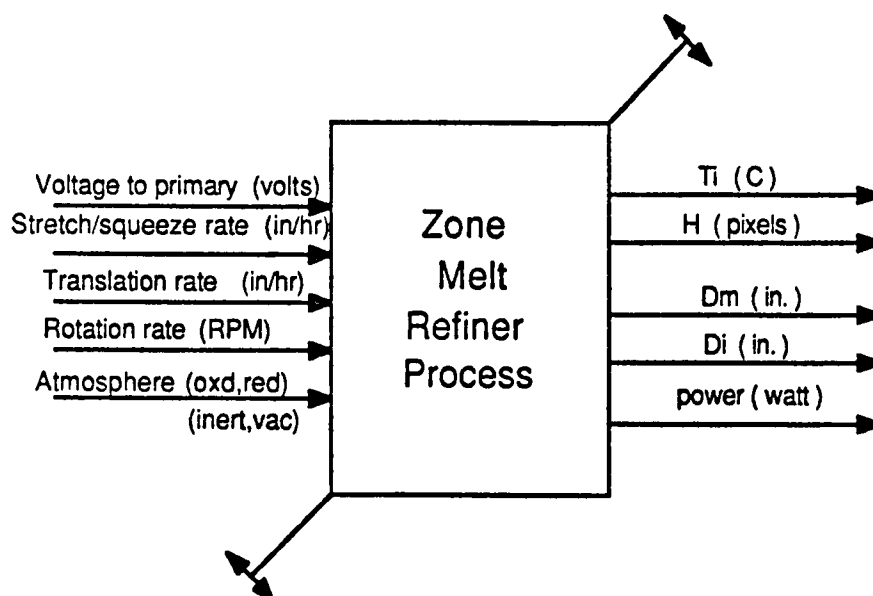


Figure 15. Close up view of the levitated zone.

liquid zone. This equipment is currently controlled through a complex set of computer interfaces (60). For the convenience of the reader, the complicated set of independent/dependent process variables are listed in a schematic, figure 16. The computer inputs are a digital image of the liquid zone (defined as the process), infra-red temperature sensing close to the liquid-solid interface, and the induced power into the process. These variables are shown in figure 17. the exact nature of the computer interface with the process outputs and process inputs is shown in figure 18.

Precise control of solidification processes is required for crystal growth. The rapid response of the computer (30 control cycles/second) provide an accurate control of the freezing behavior and may suppress any instabilities that would otherwise occur in a manually controlled process.

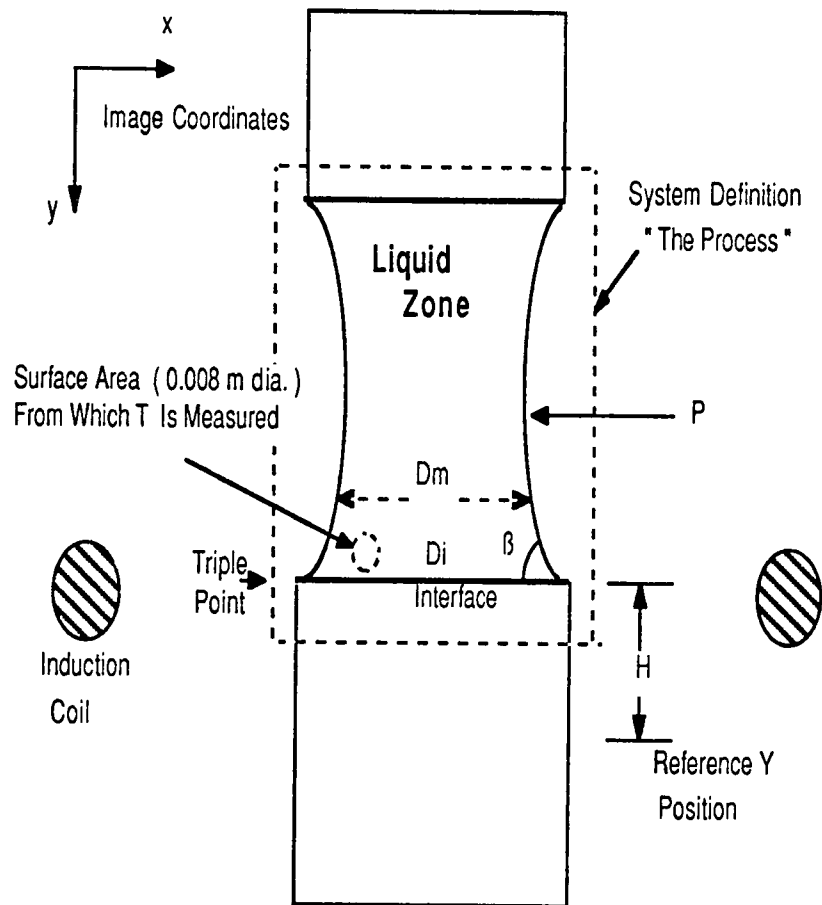
INDEPENDENT/DEPENDENT PROCESS VARIABLES



H = position of interface relative to a reference position
 Dm = melt diameter
 Di = interface diameter
 Ti = interface temperature

Figure 16. Independent/dependent variables for the directional solidification processing equipment.

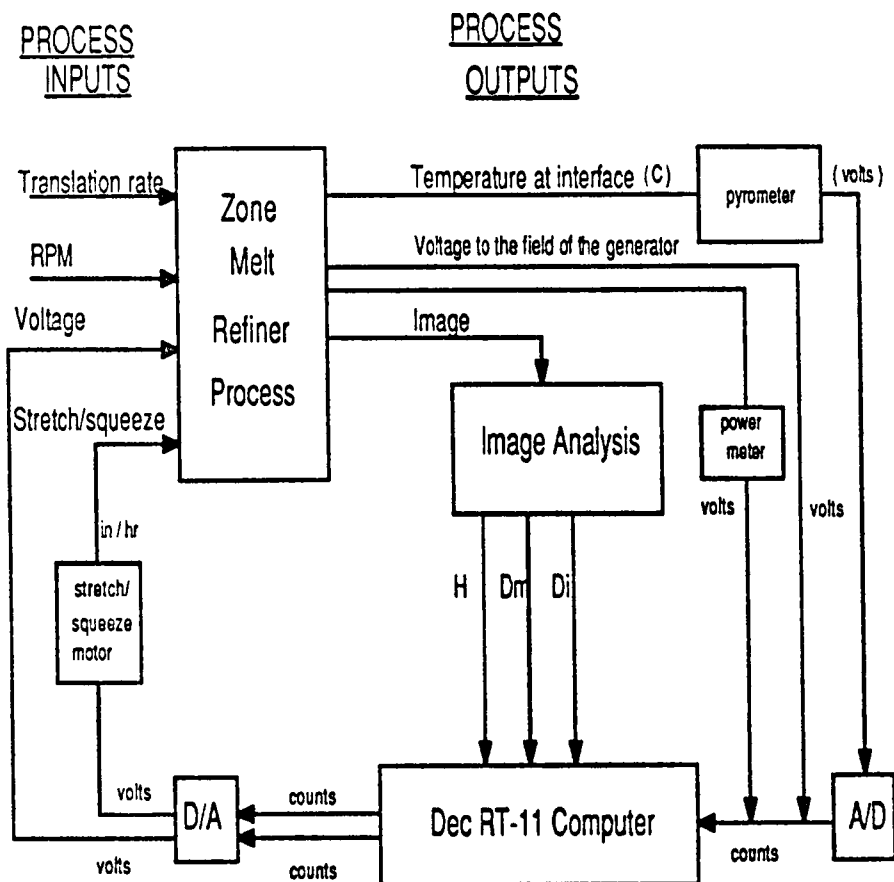
Source: R. D. Reviere et al. in "Materials and Manufacturing Processes", Vol.4, No.1, pp. 103-131, 1989



- D_m = melt diameter taken at some Y position
- D_i = diameter at zone bottom (i. e., interface diameter)
- H = difference between the Y position of the zone bottom and the reference Y position
- β = melt angle at zone bottom
- P = induced power
- T = melt temperature at zone bottom

Figure 17. Formal process definition for the computer interface.

Source: R. D. Reviere et al. in "Materials and Manufacturing Processes", Vol.4, No.1, pp. 103-131, 1989



H = position of interface relative to a reference position
 Dm = melt diameter
 Di = interface diameter
 RPM = rotation rate of solid/liquid interface
 A/D = analog to digital conversion device
 D/A = digital to analog conversion device

Figure 18. Data acquisition and control system for directional solidification using a computer interface.
 Source: R. D. Reviere et al. in "Materials and Manufacturing Processes", Vol.4, No.1, pp. 103-131, 1989

4. RESULTS AND DISCUSSION

The results and discussion chapter is divided into four sections for ease of flow and due to the separate nature of the problems dealt with in this study.

Part 1 deals with attempts to better understand the Ti-Al phase diagram and the role of interstitial impurities, particularly nitrogen, in phase partitioning and in altering the phase boundaries of the Ti-Al phase diagram.

Part 2 deals with preliminary alloy theory with respect to ternary additions. Data on site substitution by a ternary element in the binary Ti-Al alloy system, preliminary mechanical properties, and dislocation observations are presented.

Part 3 deals with microstructures, tensile and fracture properties, and electron microscopy observations for TiAl+Mn and TiAl+Ga alloys.

Part 4 deals with the cleavage fracture behavior and characterization of the crystallographic nature of cracking in these materials.

PART 1.

UNDERSTANDING THE Ti-Al PHASE DIAGRAMDefinition of the Problem

The Ti-Al phase diagram has undergone considerable changes since it was first reported in the ASM handbook (8) by Wiley and Margolin. The most current version edited by Murray and published by ASM in 1987 (2) has also come under severe doubt. The disagreement centers on the high temperature region of 50-55at%Al. The earlier version of the phase diagram (8) indicates a double peritectic, figure 2. The new version (2) shows only a single peritectic $L+\beta=\gamma$ reaction, figure 1. However, recent reports (3-6) have indicated that α can be nucleated directly from the liquid. Hence, there must be an additional $L+\alpha$ phase field in the diagram, as was earlier published by Wiley and Margolin (8). While ASM has not published an errata or supplement to the phase diagram, researchers are now referring to a double peritectic cascade as the correct version. Perepezko et al. (9) have verified the double peritectic by thermodynamic calculations. Huang et al. (10) have reported data on the possible $\alpha+L$ phase boundary compositions, figure 19.

The current study was undertaken to provide solidification structure evidence for this second peritectic. Three alloys, Ti-50at%Al, Ti-52at%Al and Ti-54.1at%Al, were selected to span the range of the second peritectic. In directional solidification, the underlying

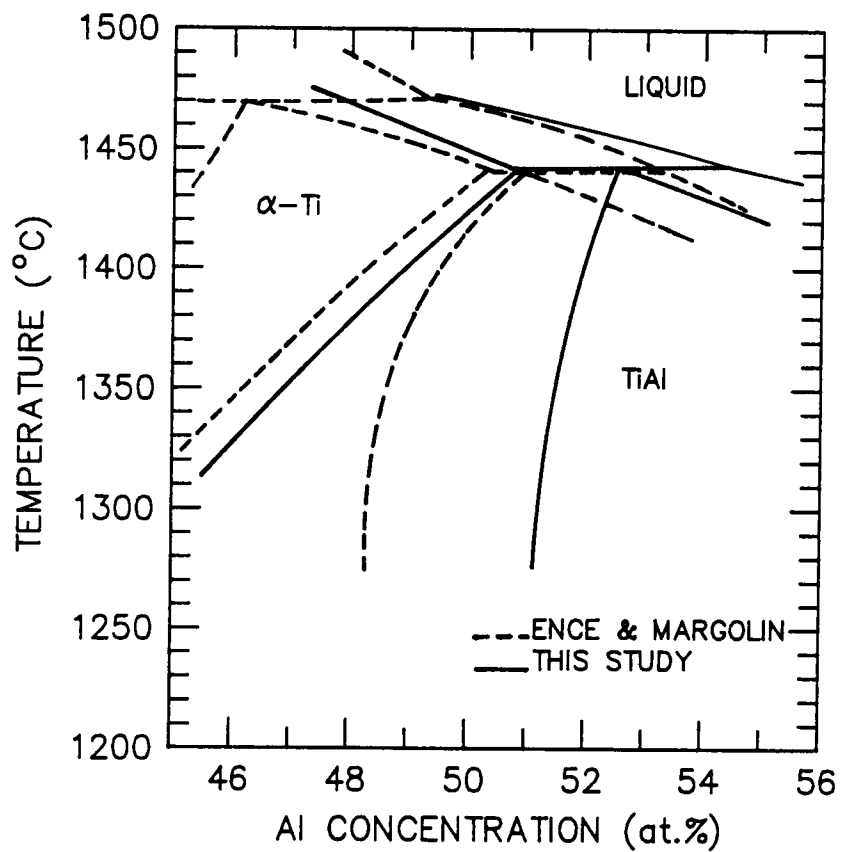


Figure 19. Data on the α +L phase field boundaries, estimated by dilatometry and optical microscopy.

Source: S.C. Huang, P.A. Siemers, "Met. Trans.", Vol.20A, No.10, p.1899-1906, Oct.1989.

factor to establish a peritectic is to select a hypo-peritectic composition which, under planar interface growth conditions, yields a banded microstructure of the bounding phases, normal to the solidification front. A planar interface precludes coupled growth for the peritectic, unlike the eutectic. Since the two phases cannot form simultaneously, they form alternately (56), yielding the phase oscillations. The theoretical and mathematical arguments for such a banded structure are laid out in an elegant paper by Boettinger (56) and that discussion will not be presented here.

For brevity, a schematic peritectic phase field is shown with solidification paths marked for several compositions scanning the phase field in figure 20. Compositions to the left of the peritectic phase field, C_s , solidify as α over the entire length of the bar. Compositions to the right of the peritectic phase field, C_l , solidify as τ over the entire length of the bar. Hyper-peritectic compositions initially solidify as α and, as the interface liquid is enriched in solute to or beyond C_l , it can now solidify as τ . This τ solidification is stable since the liquid interface continues to be solute rich. Hypo-peritectic compositions first solidify as α . When the interface liquid is enriched in solute to or beyond C_l , it can solidify as τ . However the τ solidifying from the liquid has a higher solute content than the average composition of the bar. The net result is that the liquidus is now

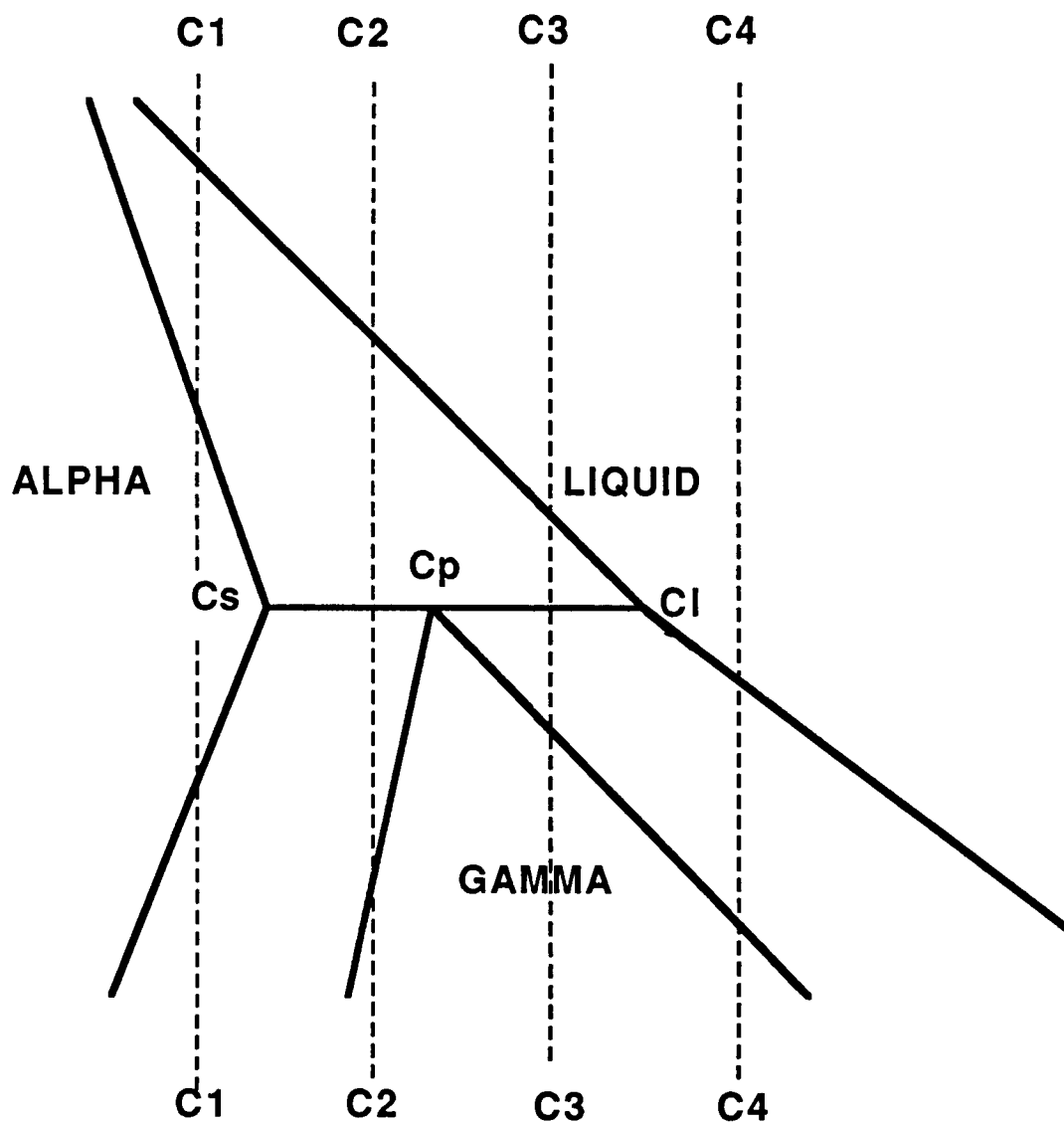


Figure 20. Schematic of solidification paths for a peritectic phase field at different bulk compositions.

continually depleted in solute. As this depletion continues, the liquidus composition follows the liquidus curve to where it can now solidify as α . This periodic compositional fluctuation yields an oscillating microstructure of the α and τ phases perpendicular to the directional growth front.

The constitutional supercooling stability criterion (57,58) for the stability of a multiphase planar liquid-solid interface states that if

$$G \geq -m_{\alpha}(dC/dz)_{z=0},$$

a planar interface will be stable with respect to the formation of cells or dendrites of α . G is the temperature gradient in the liquid at the interface, m_{α} , defined as positive, is the slope of the α liquidus at C_1 , and $C(z)$ is the composition of the liquid as a function of the distance z from the interface. $(dC/dz)_{z=0}$ can be found from the condition of continuity of mass flux at the interface,

$$-D(dC/dz)_{z=0} = R(C_1 - C^2),$$

where D is the interdiffusion coefficient in the liquid, and the R is the freezing velocity. Combining the two equations, the constitutional supercooling criterion becomes

$$G/R \geq m_{\alpha}(C_1 - C^2)/D$$

The predominant condition to establish a planar solid/liquid interface is to have a sufficiently large G/R parameter. It suffices to say that G/R can be made large by decreasing R , an experimental parameter that can be easily set and controlled. The nature of the experimental setup to perform directional solidification prohibits any contact

temperature sensing devices to monitor G . However, certain equipment parameters can be effectively manipulated to impose a high temperature gradient in the liquid. The liquid zone is inductively heated and highly mixed. Hence in the bulk of the liquid the temperature and the composition are expected to be near uniform. However, the boundary layer adjacent to the solid-liquid interface is not subject to the above condition and the temperature gradient between the solid and the liquid exists only in this finite layer, experimentally determined as $\approx 300\mu\text{m}$ (59). In addition, the solid interface is rotated, which has the net effect of further reducing the boundary layer dimension. This imposes a steeper thermal gradient, G , in the liquid (60).

Results and Discussion

Figure 21 shows the as-cast surface microstructures of the different bars as viewed on the surface by the naked eye. Ti-50at%Al and Ti-54at%Al bars solidified without any breaks in the processed length. However Ti-52at%Al showed occasional breaks in the solidification structure along the processed length.

Breaks in the solidification morphology are readily evident, as noted in figure 22. The bounding phases of the peritectic phase field are α and τ . The α formed from the liquid undergoes a solid state transformation to τ . This solid state transformation occurs as a mix of α_2/τ laths, as observed at room temperature. A low magnification picture of

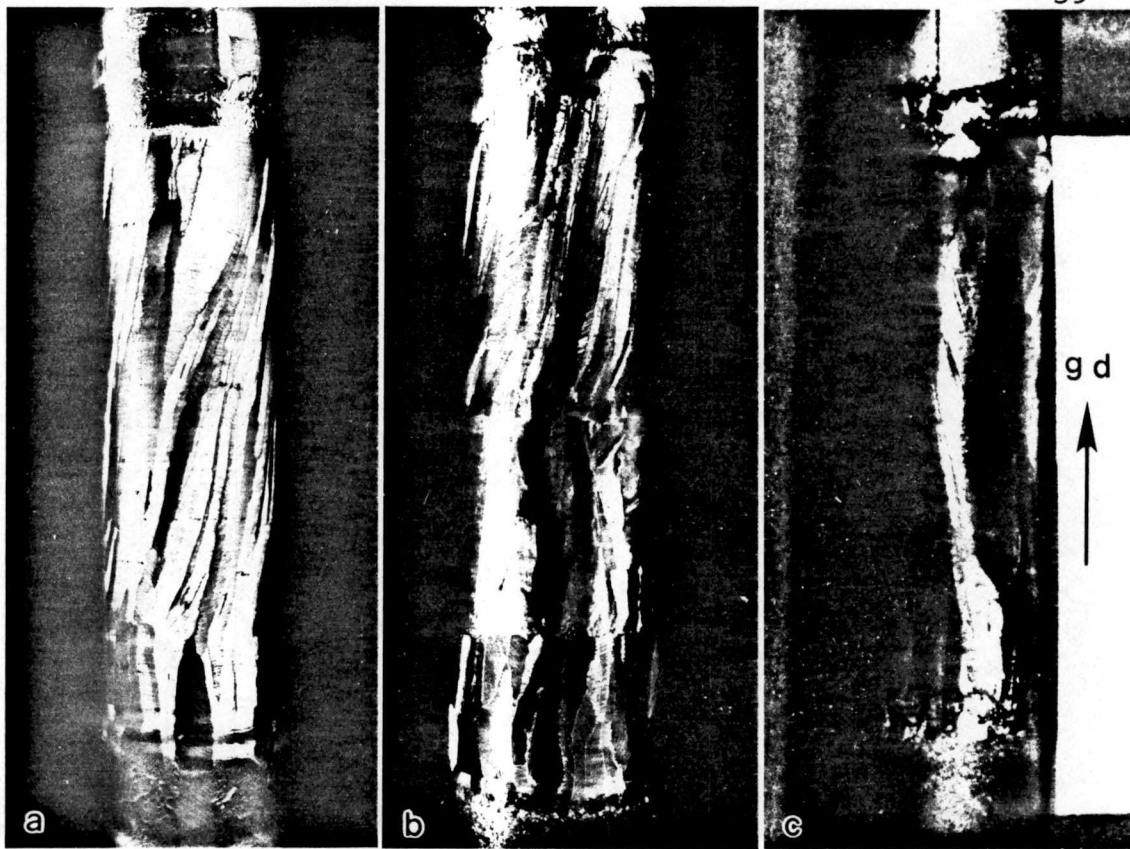


Figure 21. Directionally solidified as-cast bars of a) Ti-50at%Al, b) Ti-52at%Al c) Ti-54at%Al compositions. All bars are grown at $R=0.75''/\text{hr}$ in a helium atmosphere.

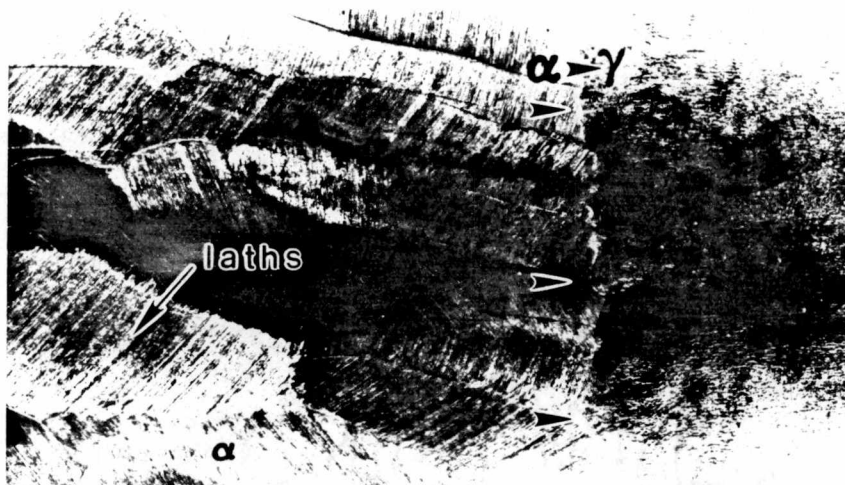


Figure 22. Optical micrograph of the surface of a Ti-52at%Al bar, directionally solidified, showing a $\alpha \rightarrow \gamma$ transition. The α region reveals (111) plane lath striations indicative of $\alpha \rightarrow \alpha_2 + \gamma$ solid state transformation.

the cast surface shows the presence of fine laths in the region solidified as α , figure 22. Note that after a change in solidification morphology, i.e., from α to τ , no such laths are visible optically, the reason being that τ is a stable phase at that composition and does not undergo any solid state transformations on cooling to room temperature. This condition also applies to the Ti-50at%Al and Ti-54at%Al bar. The Ti-50at%Al bar solidified as all α and has α/τ striations all along its zone length. The Ti-54at%Al bar solidified as τ and no α/τ striations were observed, figure 21. To ensure that the high temperature phase is indeed α -hcp and not β -bcc, a shrinkage cavity was formed in the zone length of the Ti-52at%Al bar at the end of the melt processing. Dendrites in the shrinkage cavity reveal the hexagonal symmetry, typical of the hcp structure, figure 23a,b. In addition, faceted growth was often observed, figure 24.

Figure 25 shows the etched macrostructure of the as-cast longitudinal section, of the Ti-52at%Al alloy, with alternating α and τ regions, (τ etches light, α at room temperature exists as $\alpha_2+\tau$ and etches as dark and light). X-ray diffraction confirmed the lamellar regions as τ -TiAl and α_2 -Ti₃Al, figure 26. High magnification views of several regions, including the α/τ interface, are shown in figure 27. Figure 27a shows the initial α formed from the melt (this region now exists as $\alpha_2+\tau$). The striations are the (111) traces of the τ twin plane, the habit plane for the

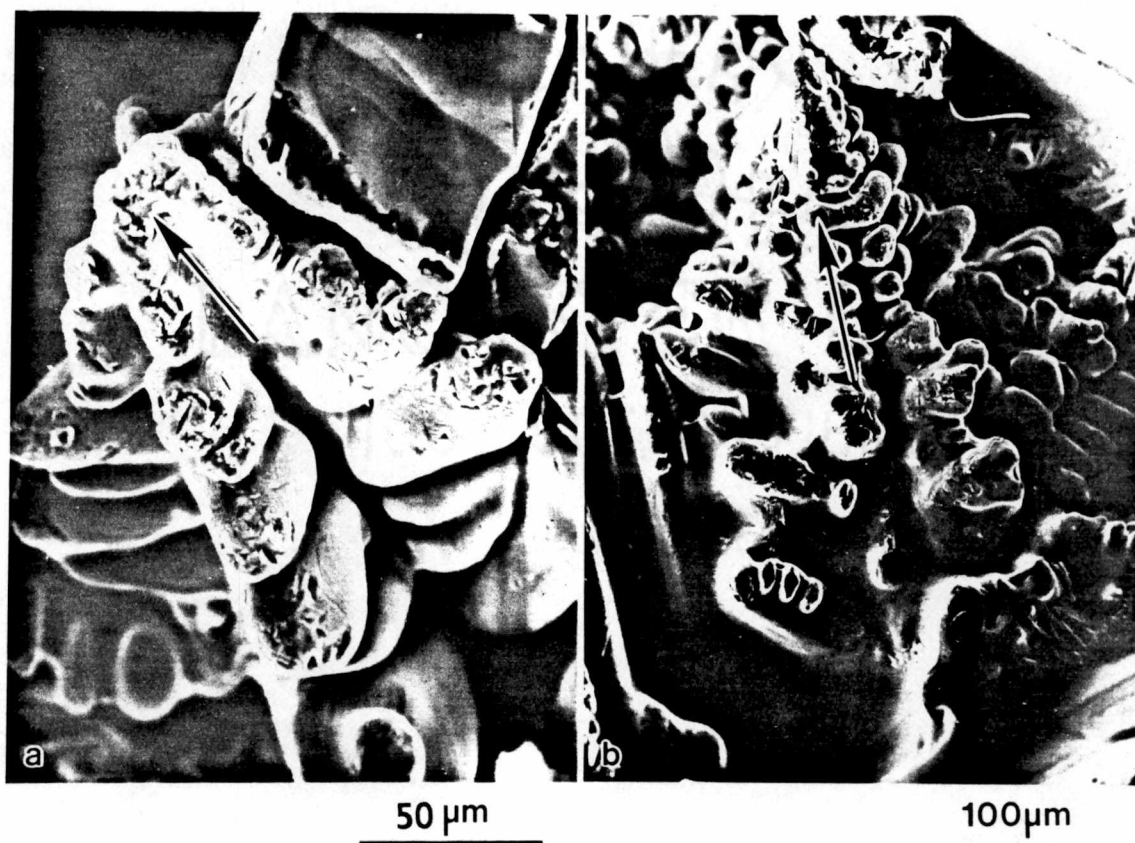


Figure 23. SEM micrograph of α -hcp symmetry dendritic growth in Ti-52at%Al bar. a) the arrow indicates [0001] growth. b) the arrow indicates [1010] growth direction.

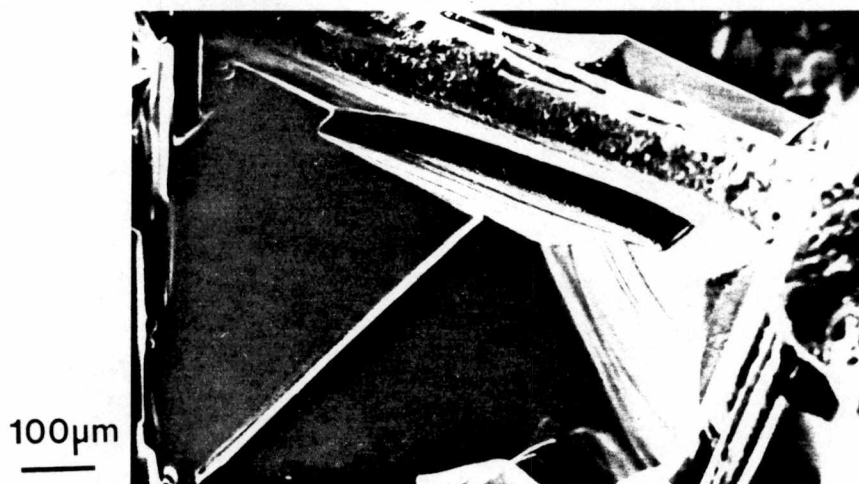


Figure 24. SEM micrograph of faceted growth also observed in the shrinkage cavity.

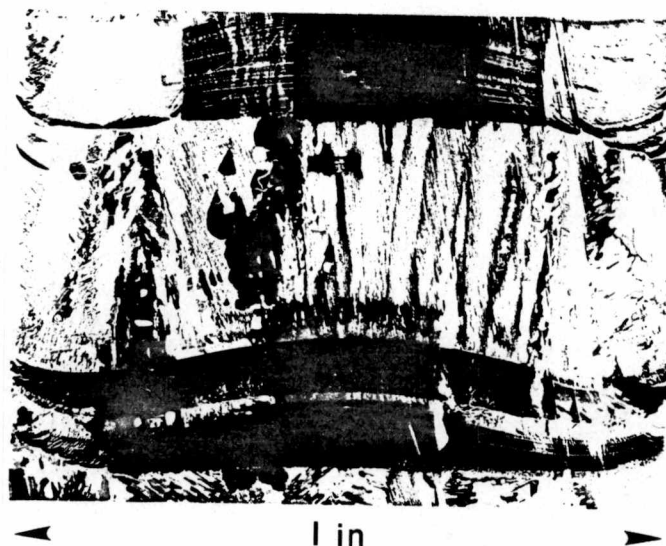


Figure 25. Etched macrostructure of the as-cast longitudinal section showing $\alpha/\tau/\alpha$ oscillations. Processing; reverse pass at $R=0.5''/\text{hr}$ in hydrogen.

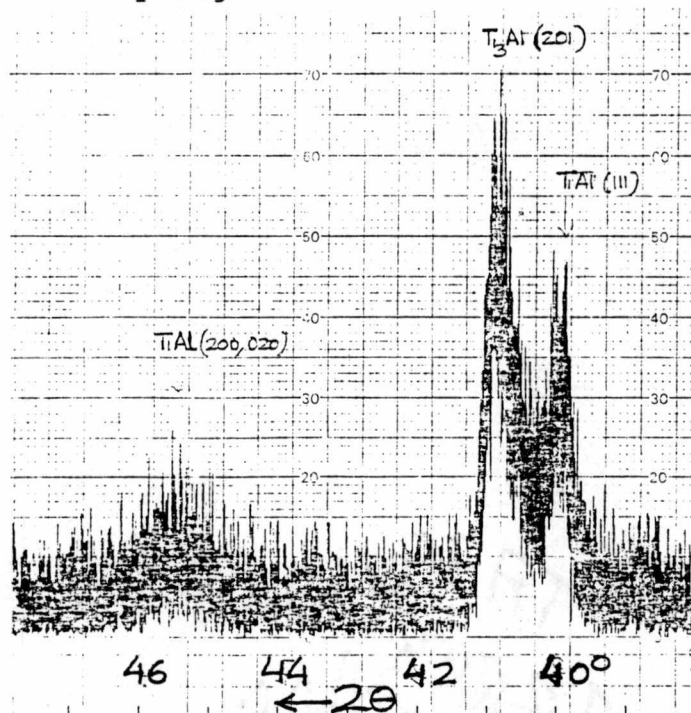


Figure 26. X-ray diffractometer trace identifying the τ -TiAl and α_2 -Ti₃Al peaks for the lath morphology regions.

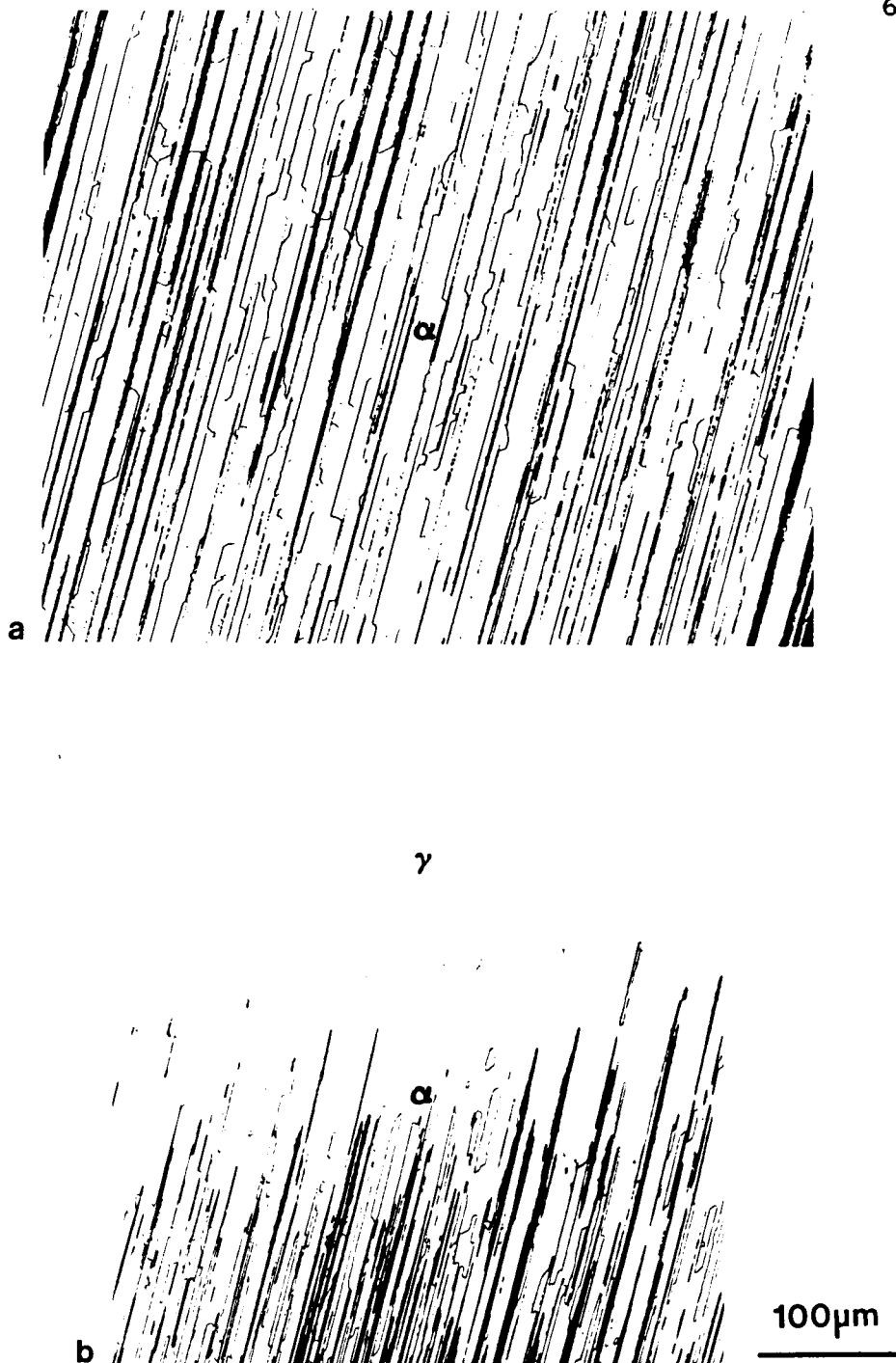


Figure 27. Optical micrograph series of $\alpha/\tau/\alpha$ oscillations along the growth direction of the bar; a) initial α from the melt, now existing as $\alpha_2 + \tau$ laths. b), c) α/τ oscillations. d) random initial orientation of α at a τ/α oscillation. e) Evidence of sustained α growth occurring for $(0001)_\alpha$ //growth direction. f) Evidence of possible breakdown of the flat interface in to a cellular interface.

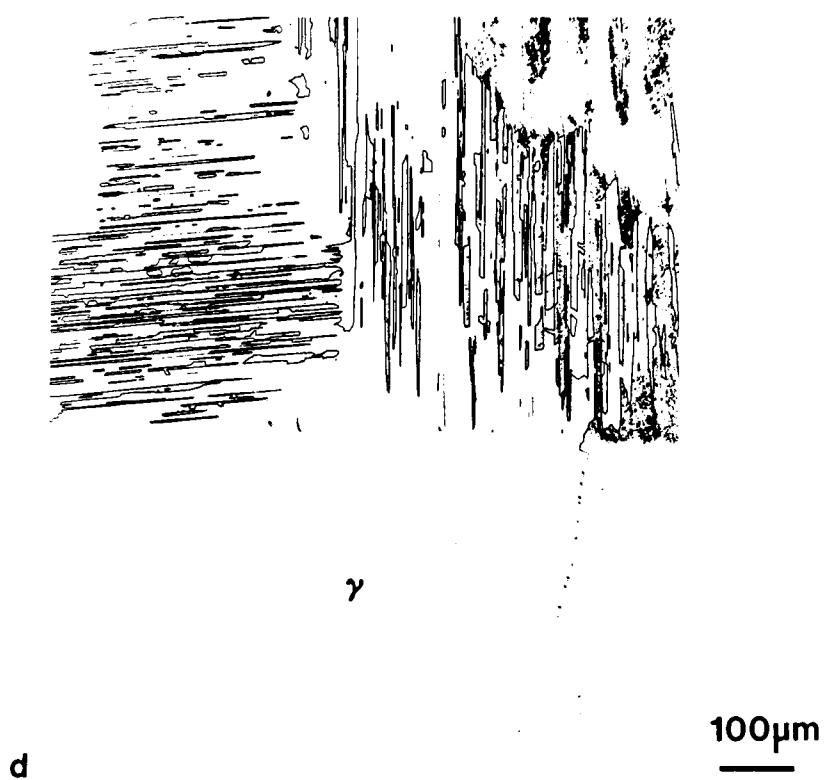
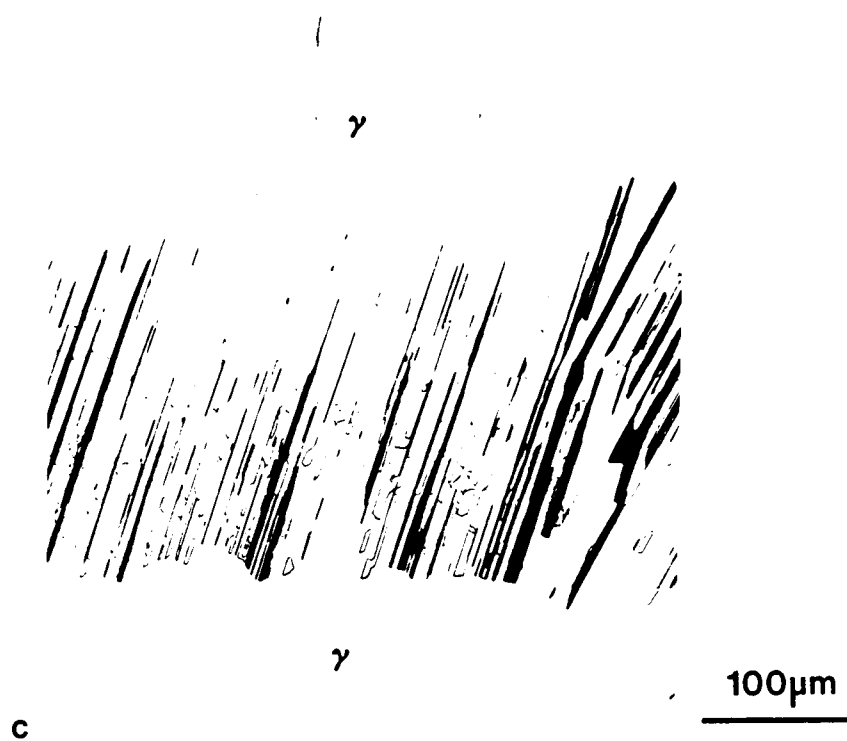


Figure 27. Continued

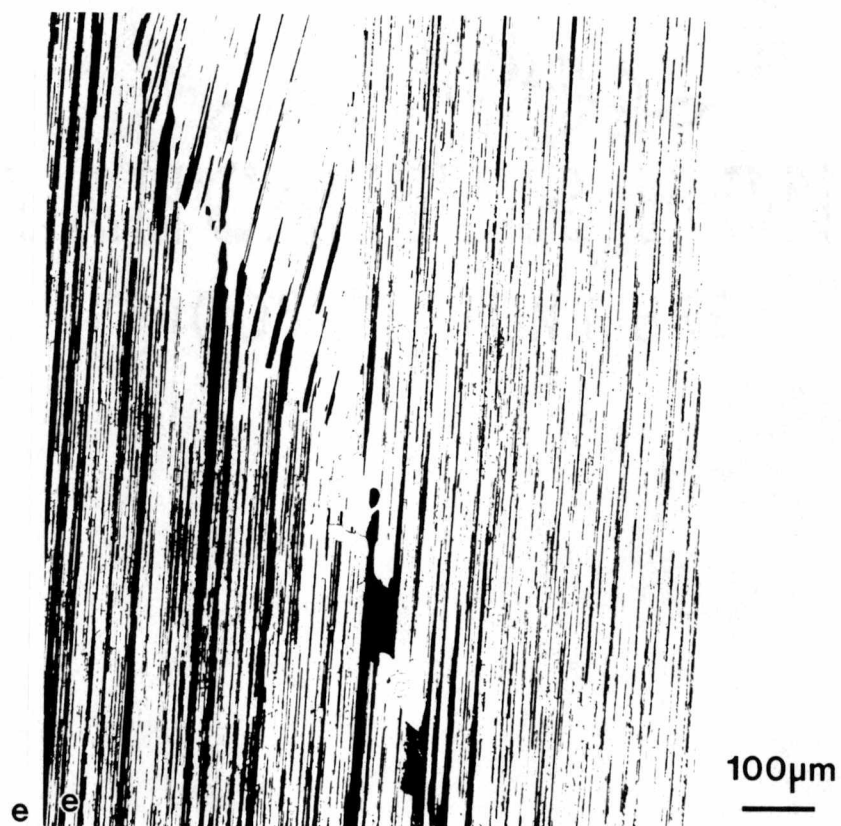


Figure 27. Continued

τ/α_2 interface, and the (0001) basal plane for the α, α_2 phases. Figure 27b,c show the α - τ oscillations. The interface is relatively flat. Note that a perfectly flat transition from $\alpha \rightarrow \tau$ or $\tau \rightarrow \alpha$ is not observed, possibly because local diffusion in the solid may even out the composition somewhat. Upon initial nucleation, the orientation of α is relatively random, figure 27d, where the basal plane is oriented both parallel and perpendicular to the growth direction. However, sustained growth of α occurs only for grains with their basal plane nearly parallel to the growth direction, figure 27e. Figure 27f shows some evidence of a non-planar interface. Interface instability at this point probably promotes a cellular solidification growth morphology. This may be expected. Since events at the liquid-solid interface required to produce oscillating structures must change drastically with time, the assumption of steady state must be violated (56). An oscillation represents a discontinuity at the solid-liquid interface. Hence, the stable growth of the new phase may be preceded by a transient instability. Such instabilities are in part reflected by the wide range of measured temperatures close to the interface, during processing, figure 28, and these temperature-time records show fluctuations that correspond to the banding of the directionally solidified bar.

Back scattered SEM imaging and X-ray EDS analysis were performed on a longitudinal section of the bar across a α/τ interface oscillation. A SEM micrograph of the interface

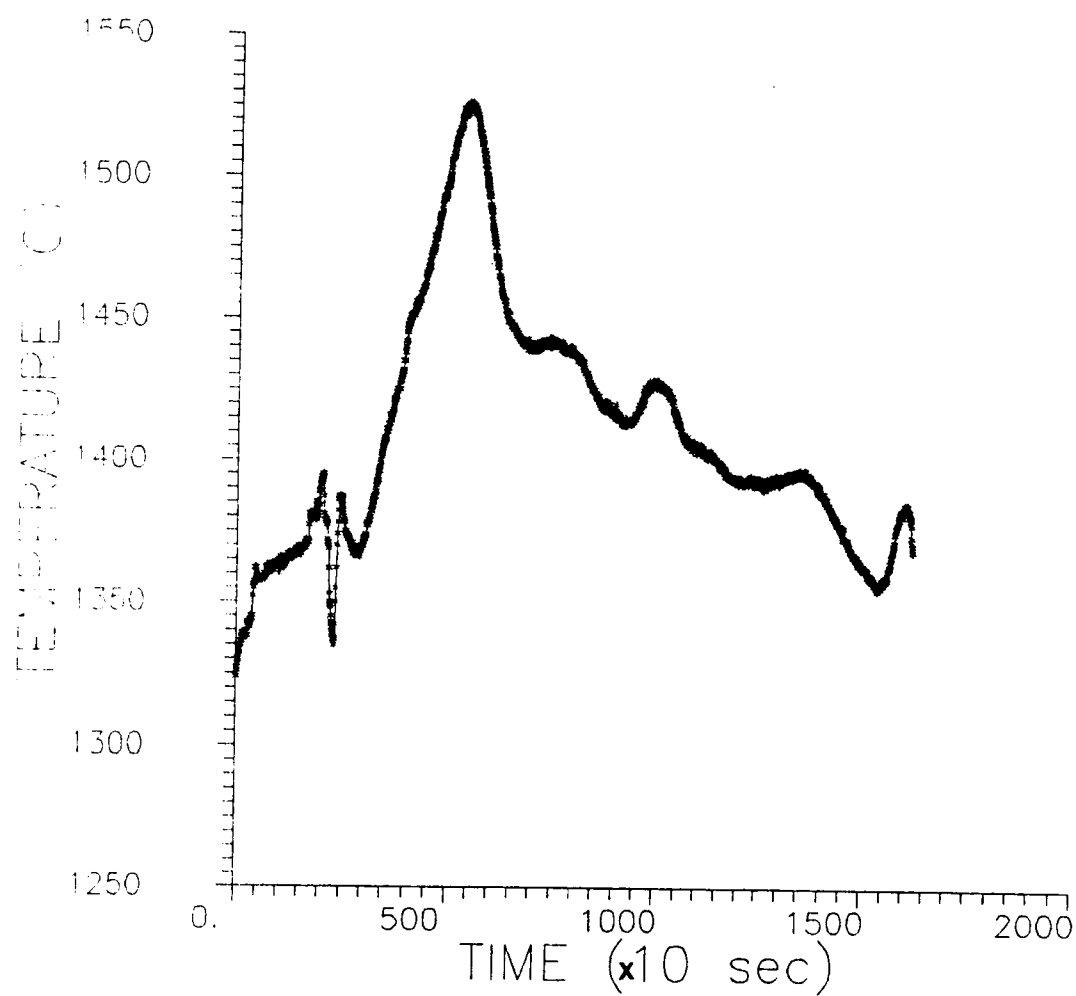


Figure 28. Temperature-time plot for reverse pass, Ti-52at%Al processing run, showing wide fluctuations in temperatures close to the interface.

used for X-ray EDS compositional analyses is shown in figure 29. The mean composition of the α and τ phases, close to the oscillation, as analyzed by X-ray EDS was 50at%Al and 52.05at%Al respectively, figure 29. Farther away from the interface into the single phase τ region, the composition was 52.7at%Al and for the prior α region was 46at%Al. Fine stringers of bright intensity were observed, close to the oscillations, indicating a possibly Ti-rich region. These regions, figure 30, have the shape of the interdendritic or intercellular morphology and were analyzed by X-ray EDS to have a composition of 67at%Al. The presence of such Ti-rich stringers clearly indicates that interface instability occurs along with each $\alpha \rightarrow \tau$ oscillation. This instability is, however, transient and not observed in regions far away from the oscillating interface.

Peculiarities are imminent when the solidification evidence is correlated with the phase diagrams, figure 3,19. An interdendritic liquid is typically characterized as one with a lower melting point and a solute rich composition (aluminum in this case) for a distribution coefficient $K < 1$, as indicated by the phase diagram. Experimental evidence suggests otherwise. The interdendritic regions are Ti-rich and have a higher melting temperature. The Ti-Al phase diagram does not corroborate these experimental observations. Hall et al. (61) have reported similar observations of intercellular Ti-rich Ti_3Al phase for rapidly solidified, hyper-peritectic, Ti-53-55at%Al alloys.

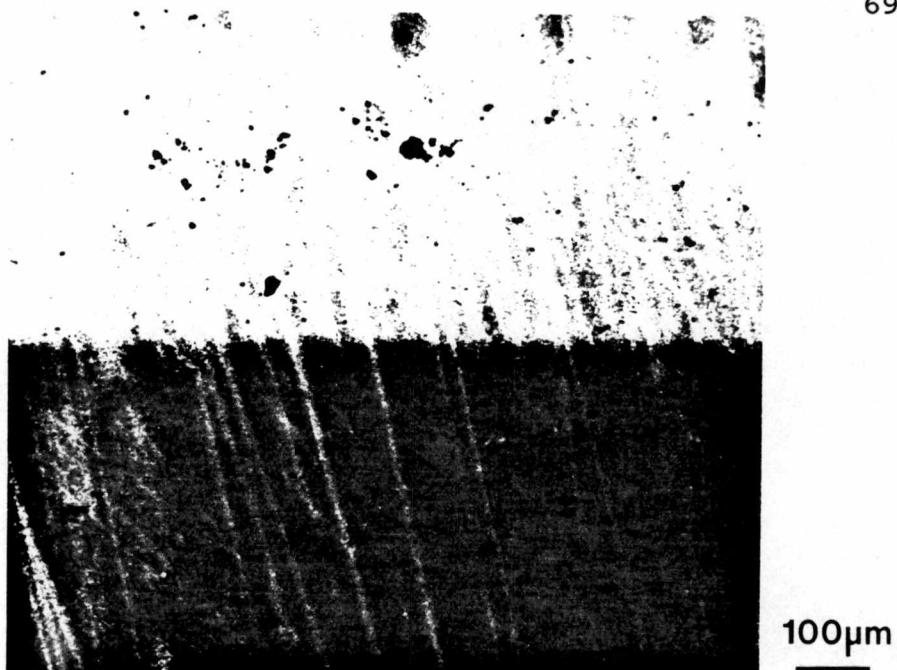


Figure 29. SEM-Back scattered electron image of a α/τ oscillation. α rich in Ti, appears light and τ rich in Al appears dark.

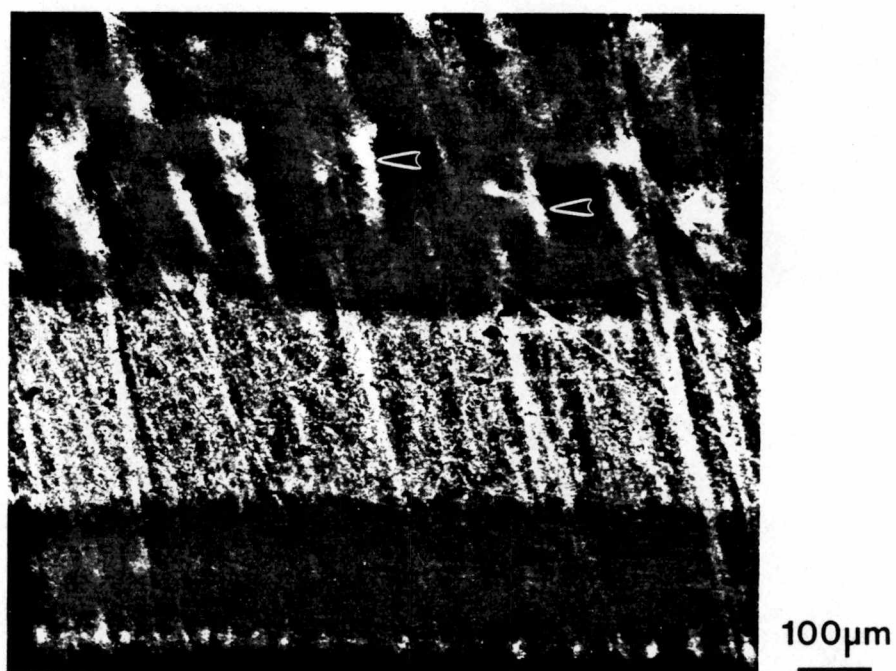


Figure 30. Bright stringers of titanium rich regions close to the oscillating interface.

This effect could be obtained by possible metastable extensions or phase boundary shifts under conditions of rapid solidification. However, in slow growth directional solidification experiments, such large scale undercoolings are not present and the metastability argument is severely weakened. There is some doubt whether these Ti-rich regions are formed from the liquid or during solid state transformations on cooling. Formation of cellular τ with intercellular α_2 was earlier reported (6) as a solid state transformation. While the intercellular segregation of titanium to form α or α_2 may require extensive modification of the existing phase diagram, the solid state transformation does not. The possibility of a solid state transformation yielding such stringers is discussed in a separate section.

Summary and Conclusions

Having established the solidification substructure evidence for the α +L peritectic phase field, some conclusions are drawn as to the possible phase boundary compositions.

- 1) Since the Ti-54at%Al bar solidified as all τ , this composition is past the α +L phase field.
- 2) Since the Ti-52at%Al bar revealed alternating $\alpha \rightarrow \tau / \rightarrow \alpha$ regions, the peritectic composition must be equal to or greater than Ti-52at%Al. For a peritectic composition below Ti-52Al, only one $\alpha \rightarrow \tau$ oscillation will be

observed. The reverse $\tau \rightarrow \alpha$ oscillation will not be observed, as discussed earlier.

EFFECT OF NITROGEN ON SOLIDIFICATION MICROSTRUCTURES

Definition of the problem

Interstitial impurities of carbon, nitrogen and oxygen are considered to have a detrimental effect on the mechanical properties of τ base material. Additionally, recent papers (46,47) have outlined the $\frac{1}{2}\langle 110 \rangle$ dislocation immobilization as a possible consequence of interstitial contamination. Hence, improved ductility of the two phase $\alpha_2 + \tau$ alloys is being attributed to the effective gettering of the interstitials from the τ phase by α_2 .

It was attempted to identify the role of α and α_2 in gettering interstitials from the melt as well as in the solid state. Towards this end only one interstitial, nitrogen, was selected to test this hypothesis. The choice for nitrogen is based on its low solubility in τ .

Kaufman et al. (14) reported a ternary Ti_2AlN precipitate, hexagonal ($a=0.304$ nm, $c=1.389$ nm), in single phase τ -TiAl alloys. They reported that for single phase τ , a 40ppm N impurity was sufficient to precipitate the nitride phase, whereas for two phase $\alpha_2 + \tau$ alloys no nitrides were observed up to a N impurity of 2080ppm. The high nitrogen impurity in the two phase alloy was attributed to a leak in the encapsulation during homogenization of the as-cast specimens. Absence of nitrides in the two phase alloy was

explained as α_2 having a high solubility for nitrogen and presumably, all the N partitioned to α_2 . While this is feasible for two phase $\text{Ti}_3\text{Al}+\text{TiAl}$ as-cast alloys being homogenized in a nitrogen contaminated atmosphere, it does not explain what happens during significant nitrogen contamination in the melting stage. In the latter case, for alloy compositions within the range of the suggested $\alpha+\tau$ phase field, τ forms from the melt by a peritectic reaction $L+\alpha=\tau$ and no α_2 exists at this temperature.

Two possibilities exist in this case. First, if α forming from the liquid has a low N solubility, it will attempt to reject excess nitrogen into the liquid. Under a high freezing rate, α is liable to have some supersaturation of N as it solidifies. Second, if α has a high solubility for N, i.e., at least higher than that in τ , then α will have some N absorption as it solidifies. However at the peritectic interface where $L+\alpha=\tau$ reaction occurs, there may be some supersaturation of N. This N will precipitate Ti_2AlN provided the time constraints to the diffusion parameters are satisfied.

The alloy composition Ti-54at%Al was selected so that irrespective of any cast structure, homogenization should produce single phase τ , thereby enabling us to study the effect of nitrogen and nitride precipitation in α and α_2 . It was intended that regions of nitride precipitation might reveal the partitioning behavior of N in α , α_2 , and τ . Since $\text{Ti}_3\text{Al}(\alpha_2)$ forms by a coherent ordering reaction from

the disordered α -Ti solid solution, it is my contention that both α and α_2 may have a high solubility for nitrogen. Hence, under arc casting conditions, the formation of α (disordered hcp-Ti solid solution) acts as a sump for nitrogen. In effect this is somewhat indicated for a binary Ti-N phase diagram, figure 31, where N is a potent α stabilizer with a partitioning coefficient >1 .

Results and Discussion

Table 7 lists the various compositions and processing conditions for the melts. The alloys are referred to as the binary Ti-54at%Al alloy and the N alloys. The N alloys were significantly harder than the binary alloys, indicating the hardening effects of N contamination. Figure 32 shows the as-cast optical microstructure of the binary TiAl and the TiAl+N alloys. The difference in the nucleation and growth morphology is readily apparent. The binary alloy shows stable growth of the α dendrites. These are formed by few nucleation events and grow to large dimensions, figure 32a. The TiAl+N alloys show a vast number of independently nucleated α colonies, figure 32b. Optical microscopy revealed a peritectic reaction product for both the TiAl and the TiAl+N alloys. A high magnification view, figure 33, of the N alloy reveals a nucleating particle present in almost each of the prior α dendrites. These particles were subsequently identified as containing nitrogen. On melting, in a nitrogen environment, N forms nitrides in the liquid

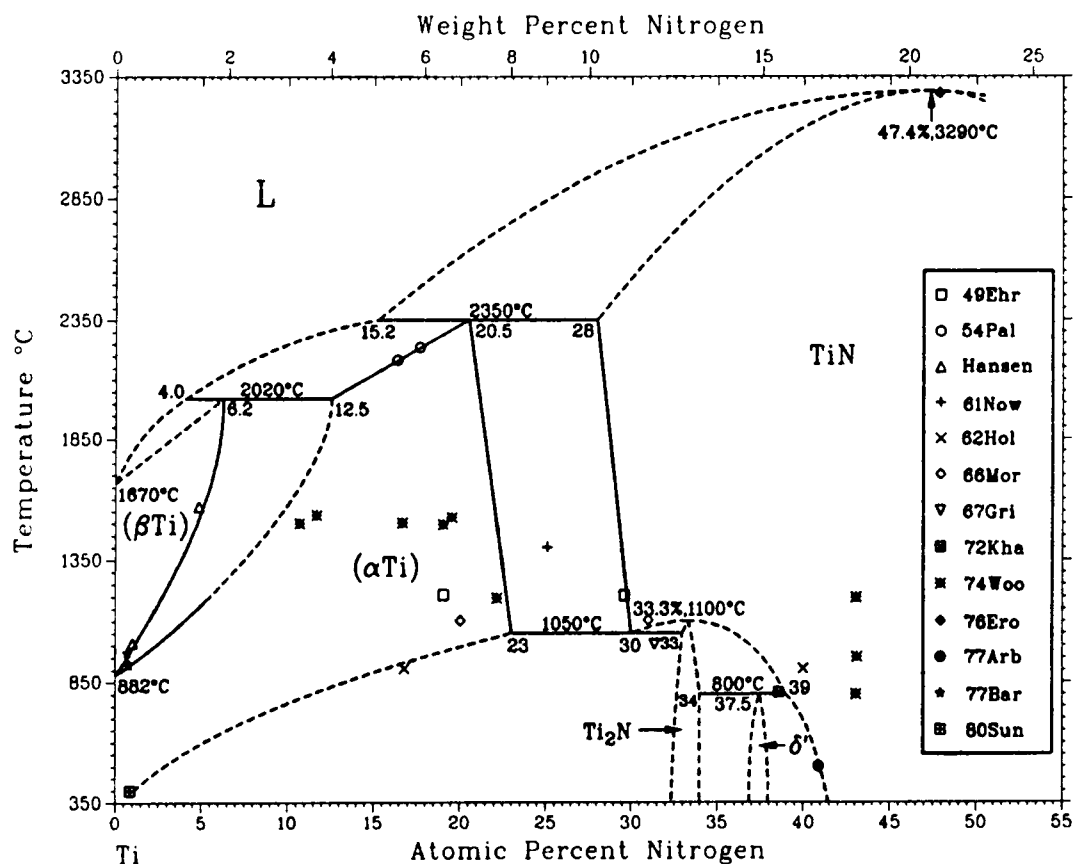


Figure 31. Binary Ti-N phase diagram indicating the partitioning coefficient of N in α as >1 (i.e., $C_S/C_L > 1$). Source: J.L. Murray in "Binary Phase Diagrams of Titanium Alloys" ASM monograph, Ohio, 1987

TABLE 7. ALLOY COMPOSITIONS AND PROCESSING CONDITIONS FOR TiAl AND TiAl+N ALLOYS.

Composition	treatment	N content	DPH Kg/mm ²
Ti-54at%Al	arc-cast	nominal purity	275
	homogenized	N≈200-500ppm	260
Ti-54at%Al-N	arc cast	N=4000ppm	368
	homogenized	"	366

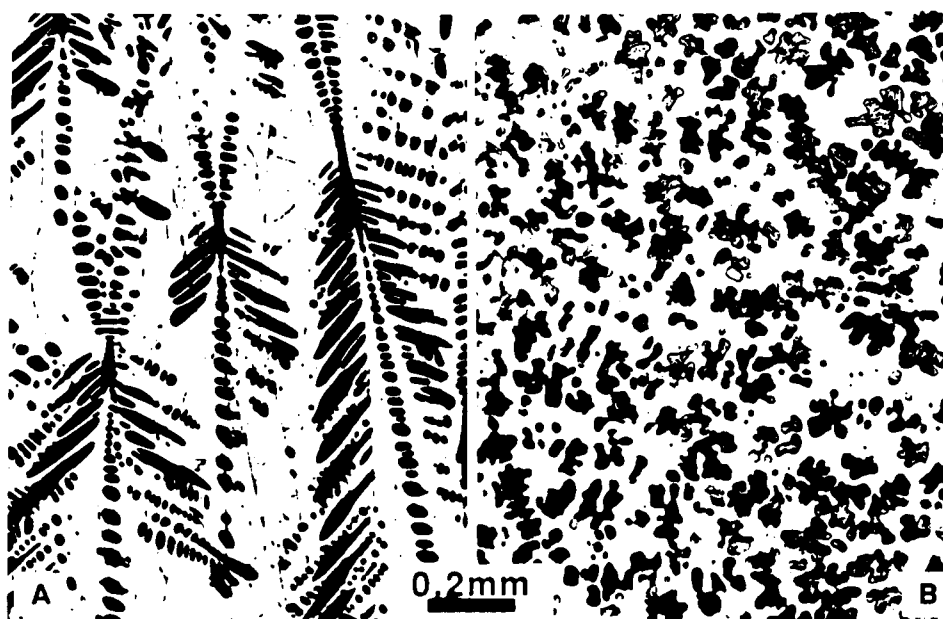


Figure 32 Optical micrographs of the as-cast alloys. a) Ti-54at%Al b) Ti-54at%Al+N.

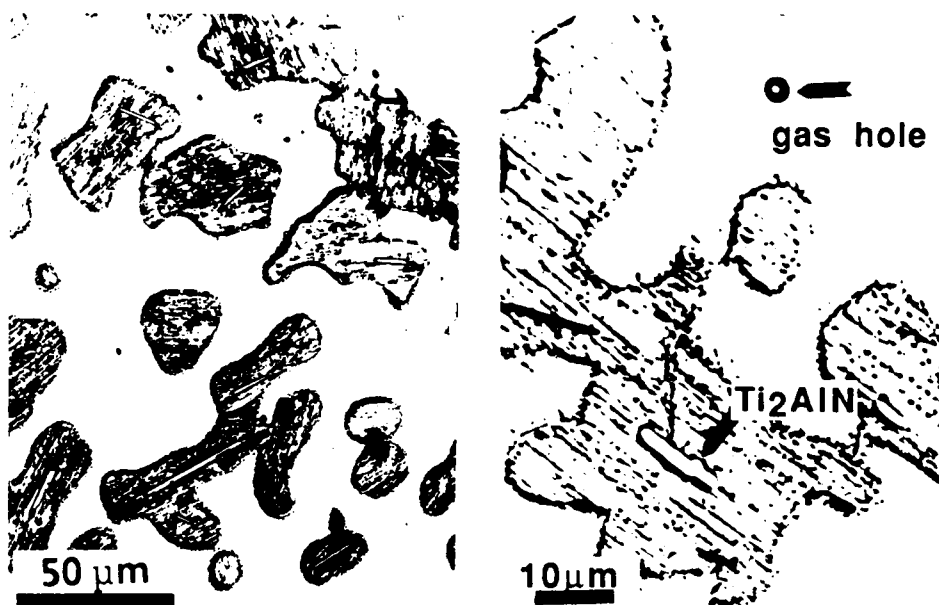


Figure 33. High mag optical micrographs of TiAl+N alloy indicating the presence of nucleating particles present inside the prior α colony.

that act as nucleation sites for α and presumably solidification occurs with little undercooling. This is indeed the case since no large scale α dendritic growth was observed, figure 32b. An interesting feature of the N alloys was the presence of several gas holes in the interdendritic τ regions. This observation suggests that τ is unlikely to absorb a significant amount of nitrogen. This nitrogen build-up in the liquid presumably nucleates gas bubbles. The homogenized alloys revealed a single phase microstructure for the TiAl alloys, figure 34. In sharp contrast, the N alloys, following homogenization, revealed globular particles at the peritectic rim as well as inside the rim, figure 35. Figure 36 shows SEM micrographs of the as-cast and the homogenized TiAl+N alloy, with essentially identical features as the optical microstructures. Extraction replicas were prepared from the as-cast TiAl+N alloy to determine if these particles were nitrides or carbides. Since Ti_2AlN has identical lattice parameters to Ti_2AlC (62), diffraction evidence would be inconclusive. Extraction replica X-ray EDS analyses confirmed the presence of nitrogen in these particles, figure 37. In addition, the Ti and Al peaks were quantified and the atomic ratio was $\text{Ti}/\text{Al}=2$. From a $[0001]$ diffraction pattern obtained for the precipitates, figure 38, the precipitate was identified as Ti_2AlN .

TEM investigation of thin foils of the as-cast material also revealed nitride particles at the peritectic rim, figure 39. This is explained by the fact that some $\alpha+\text{L}=\tau$

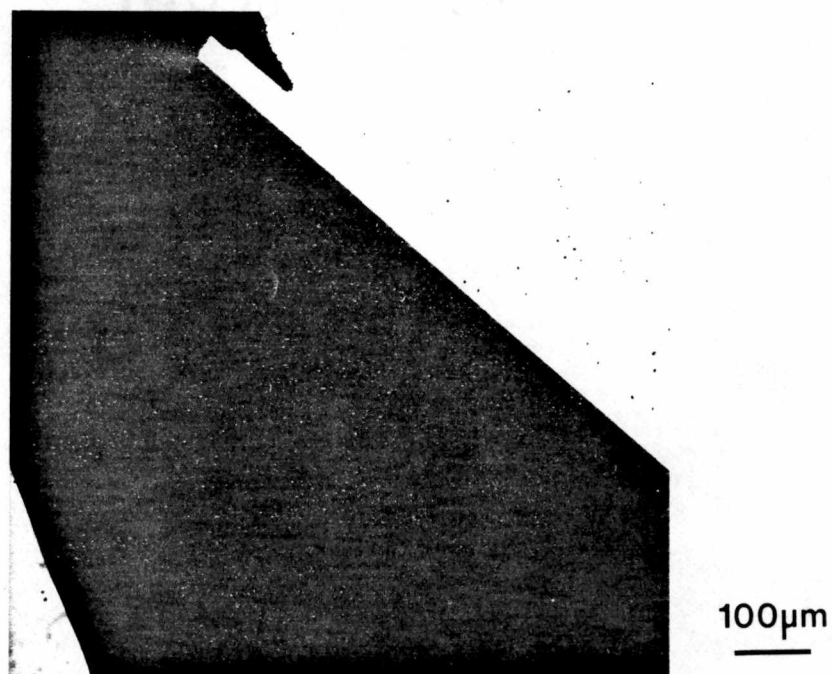


Figure 34. Optical micrograph of binary TiAl alloy showing a single phase microstructure following a homogenization treatment.

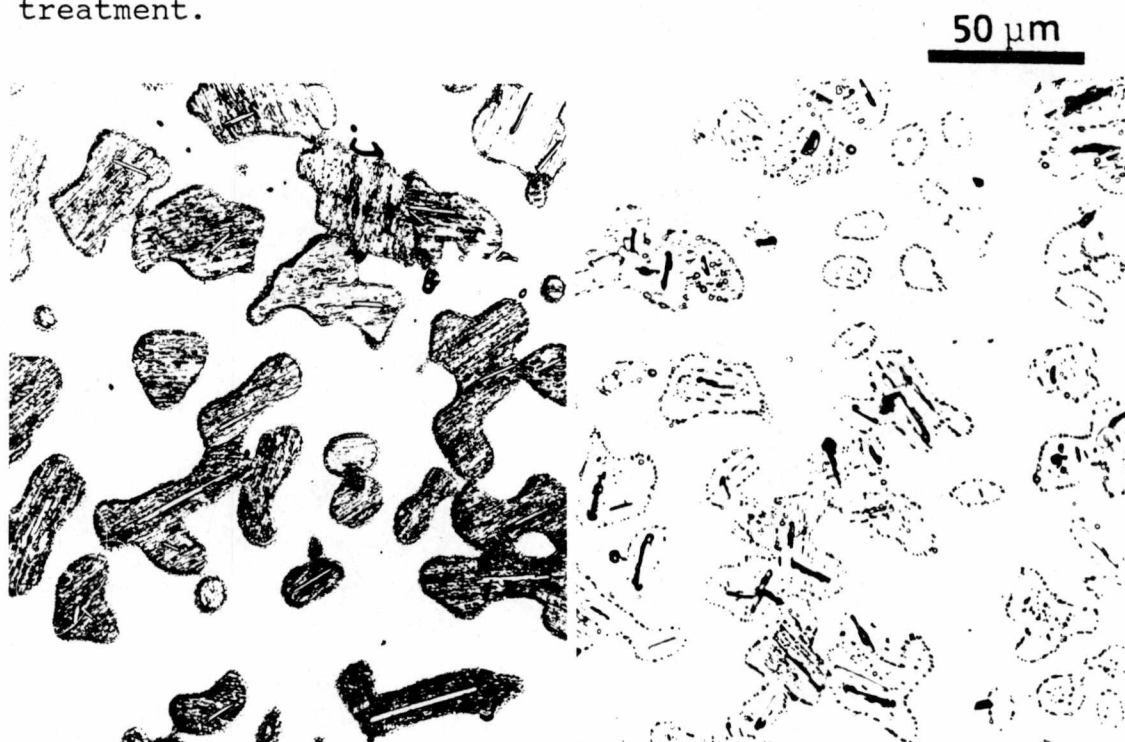


Figure 35. Optical micrographs of the TiAl+N alloys. a) as-cast b) after homogenization at 1523K for 5 hours.

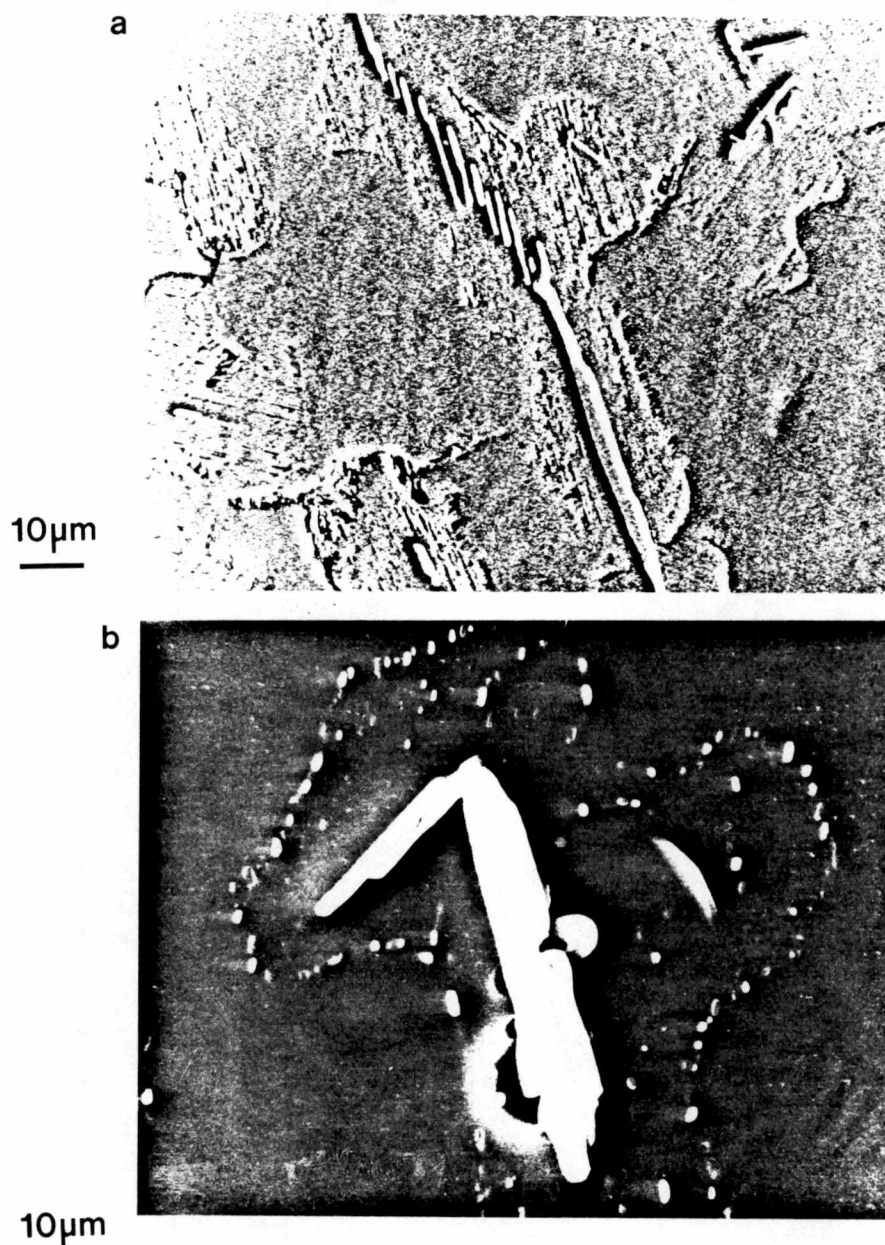


Figure 36. SEM micrographs of the TiAl+N alloys. a) as-cast b) after homogenization at 1523K for 5 hours. The distribution of the particles at the rim suggests that they form at the peritectic rim (i.e., the α/τ interface).

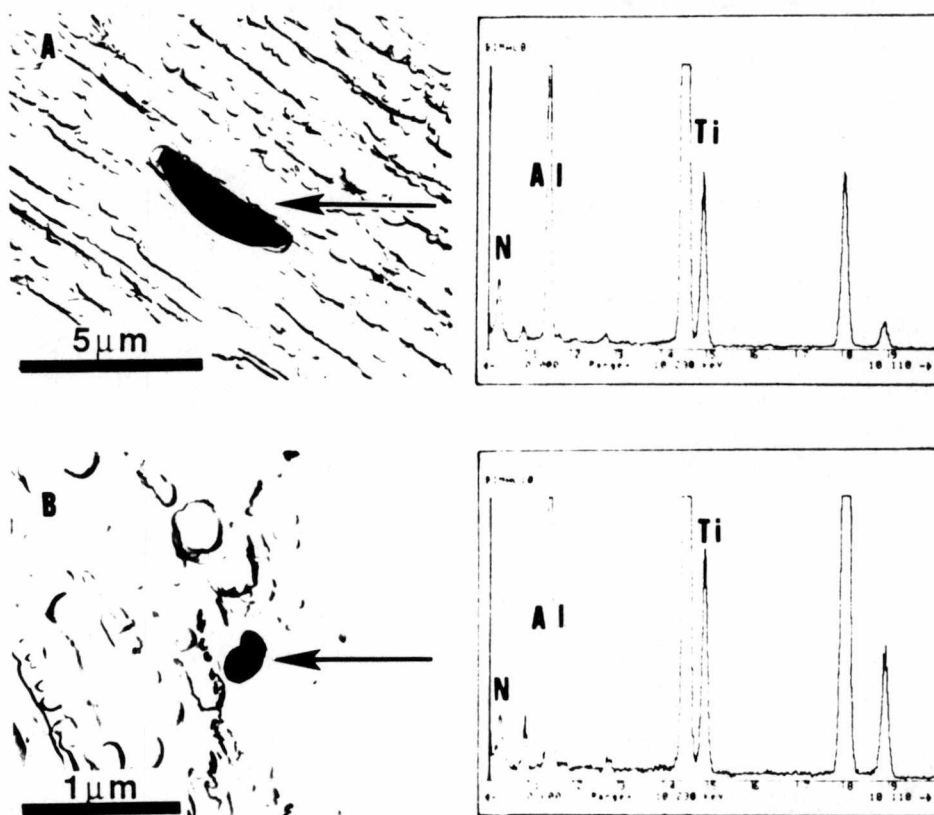


Figure 37. X-ray EDS spectra for a) the nucleating particle for the α colony and b) particle at the peritectic rim indicating a nitrogen peak.

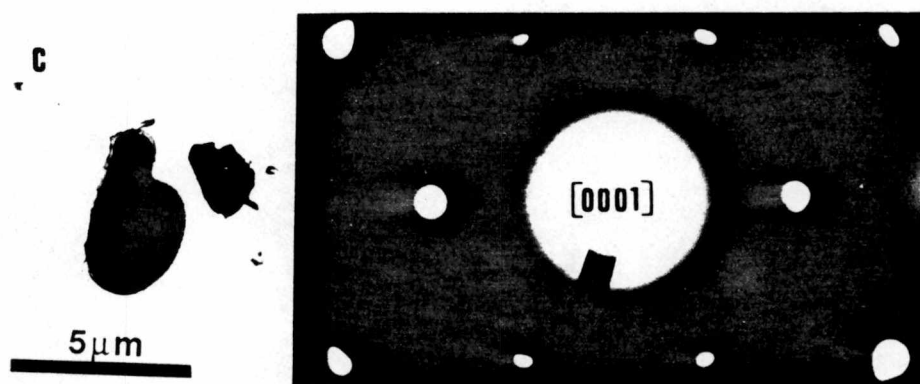


Figure 38. [0001] diffraction pattern corroborating the Ti_2AlN lattice parameters.

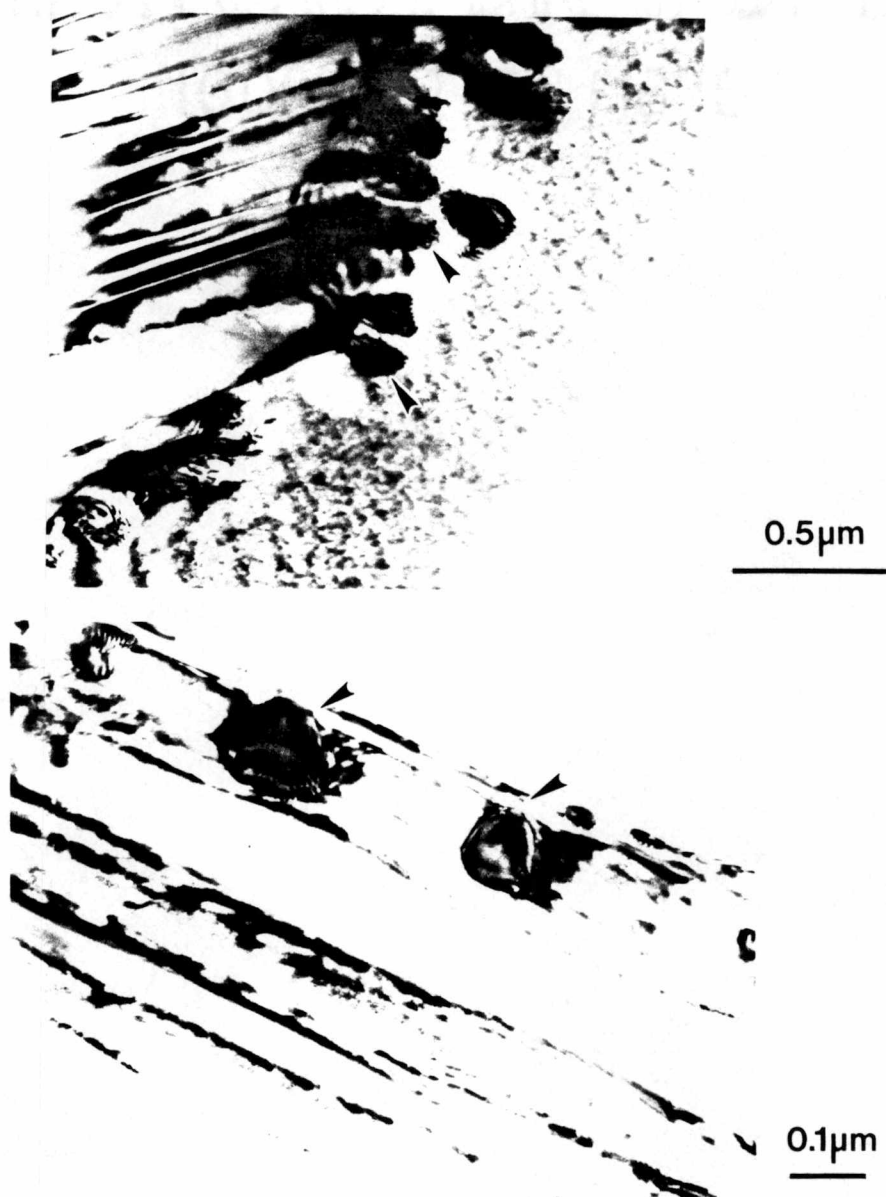


Figure 39. TEM micrographs of as-cast Ti-54at%Al+N alloy indicating the presence of precipitate particles at the peritectic rim

reaction occurred at the rim, and for α having a higher solubility for N than τ , the excess N forms nitrides at the peritectic rim. On homogenization the N possibly diffuses to these pre-existing precipitates and they grow to large dimensions, clearly visible by optical microscopy, figure 35. This indicates that α does indeed have a higher solubility for N than τ and that nitrides will form whenever there is a $L+\alpha=\tau$ reaction occurring in a N containing melt.

It was apparent that N was present in the pro-peritectic α region both in the as-cast and homogenized alloys. A detailed look at the replicas as well as TEM thin foils of as-cast alloys revealed only one incident where a nitride particle was observed in the prior α region. It is possible that all the N in α partitions to the α_2 phase during the eutectoid reaction, as suggested by Kaufman et al.(14). To elucidate this point, TEM studies were extended to homogenized alloys to study the pro-peritectic region. Such pro-peritectic regions were easily identified as they were decorated by a large volume fraction of globular nitride particles, figure 36. This region contains arrays of nitride needles all aligned in a single direction with an orientation relationship with τ -TiAl as

(0001)ppt// (111)TiAl, and [1120]ppt//[110]TiAl in agreement with Kaufman et al.(14), figure 40. In addition, TEM bright and dark field micrographs revealed the precipitation of Ti_2AlN at the α_2/τ interface in a partially homogenized specimen, figure 41. Precipitation of oriented

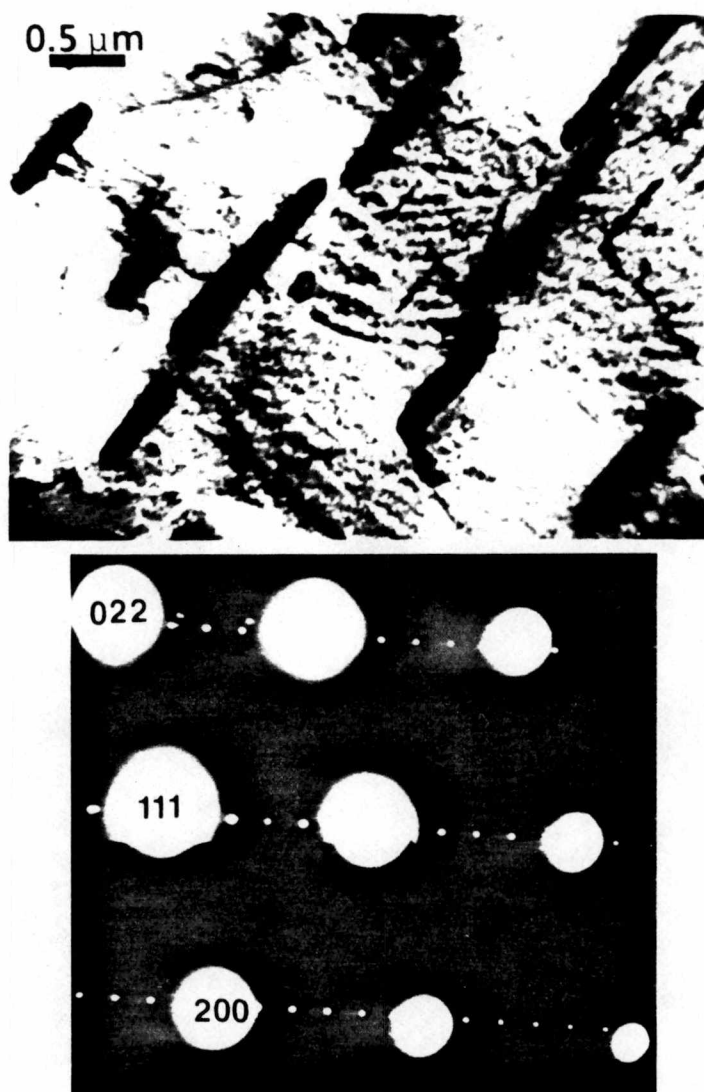


Figure 40. TEM micrograph of homogenized Ti-54at%Al+N alloy
a) indicating the presence of Ti_2AlN precipitates within the
prior α region b) diffraction pattern confirming the
orientation relationship between Ti_2AlN and $\tau\text{-TiAl}$

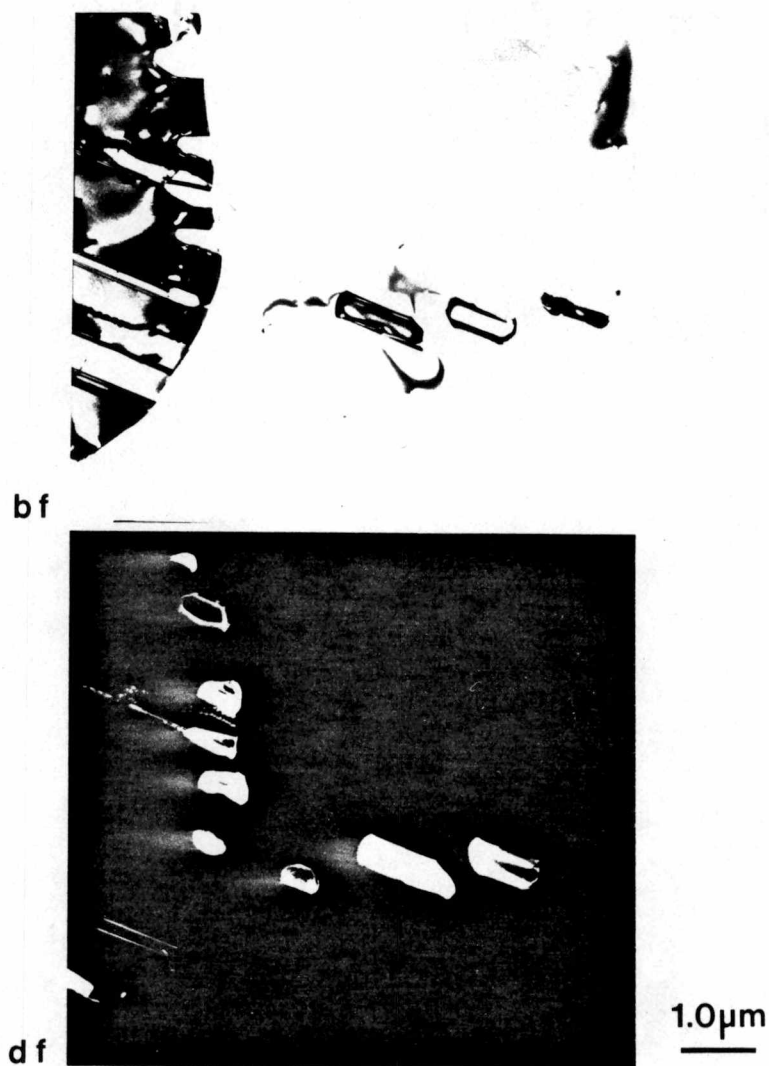


Figure 41. TEM, BF and DF micrographs indicating the growth of Ti_2AlN particles at the α_2/τ interface in a partially homogenized specimen.

needles inside the prior α region clearly illustrates that both α and α_2 have a higher solubility for nitrogen than τ . Hence Ti_2AlN precipitation occurs by the dissolution of $\alpha_2 \rightarrow \tau$. It is noteworthy that the prior α region was the dominant site for nitride precipitation. Intragranular nitride precipitation in the τ formed from the melt was also occasionally observed, suggesting that N largely partitions to the α phase.

Summary and Conclusions

The following solidification sequence is proposed for a nitrogen containing melt:

$L + N = L(\text{lower } N) + \text{Ti}_2\text{AlN particles}$

$L = \alpha$ disordered nucleating on Ti_2AlN particles

$L + \alpha = \tau$

$N + L + \alpha = \text{Ti}_2\text{AlN}$ at the peritectic rim.

$L = \tau$

$\alpha = \alpha_2 + \tau$, the N probably partitioning to α_2

On annealing, $\alpha_2 = \tau$ and the N forms Ti_2AlN .

This indicates that τ has a lower solubility for N than either α or α_2 . This locates the sites and morphology of nitride precipitation. A peritectic reaction yields globular particles and their distribution is limited to the peritectic rim. Nitride precipitation in the solid state from α_2 is fine and oriented with respect to τ . Thus the prior α regions are the dominant sites for Ti_2AlN precipitation. It would be interesting to attempt better

control of the nitride precipitation in the solid state for beneficial creep properties. The alloy chemistry may be altered to have a varying volume fraction of the peritectic product. Irrespective of this volume fraction effect the particles once formed at the rim always remain and their growth is aided by subsequent homogenization treatment.

EFFECT OF NITROGEN ON EXTENSION OF THE α +L PHASE FIELD.

Definition of the problem

In earlier directional solidification experiments, a Ti-54at%Al alloy solidified as TiAl(τ) directly from the liquid, figure 42. The directionally solidified bar had a <40ppm N impurity content. No initial transient Ti₃Al(α) was observed. However, the starting ingot had a two phase microstructure, figure 43, the phases being Ti₃Al(α_2) and TiAl(τ) as identified by room temperature X-ray diffraction experiments, figure 44. It is surprising that a vast difference exists in as-cast microstructures for the two different processing conditions. The following segment attempts to explain the discrepancy with respect to processing conditions and possible metastability in the α +L phase field.

Results and Discussion

This discrepancy in as-cast microstructures for the two different processing conditions may be explained by the



Figure 42. Directionally cast optical microstructure of Ti-54.1at%Al alloy showing single phase γ . N impurity content for the processed bar <40ppm.

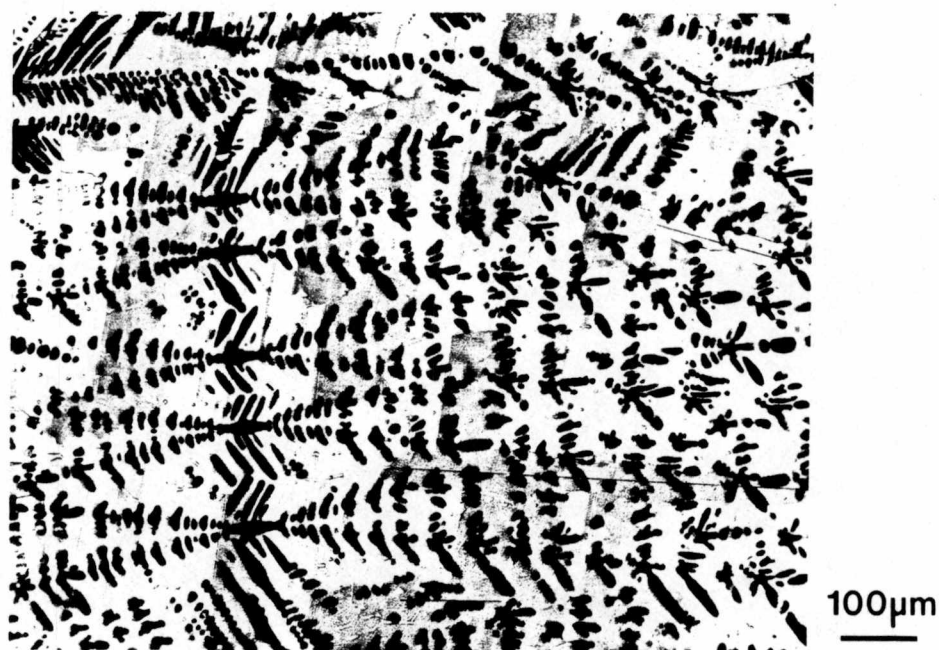


Figure 43. Starting ingot microstructure of drop cast Ti-54at%Al alloy used in directional solidification experiments. N impurity content 200-500ppm.

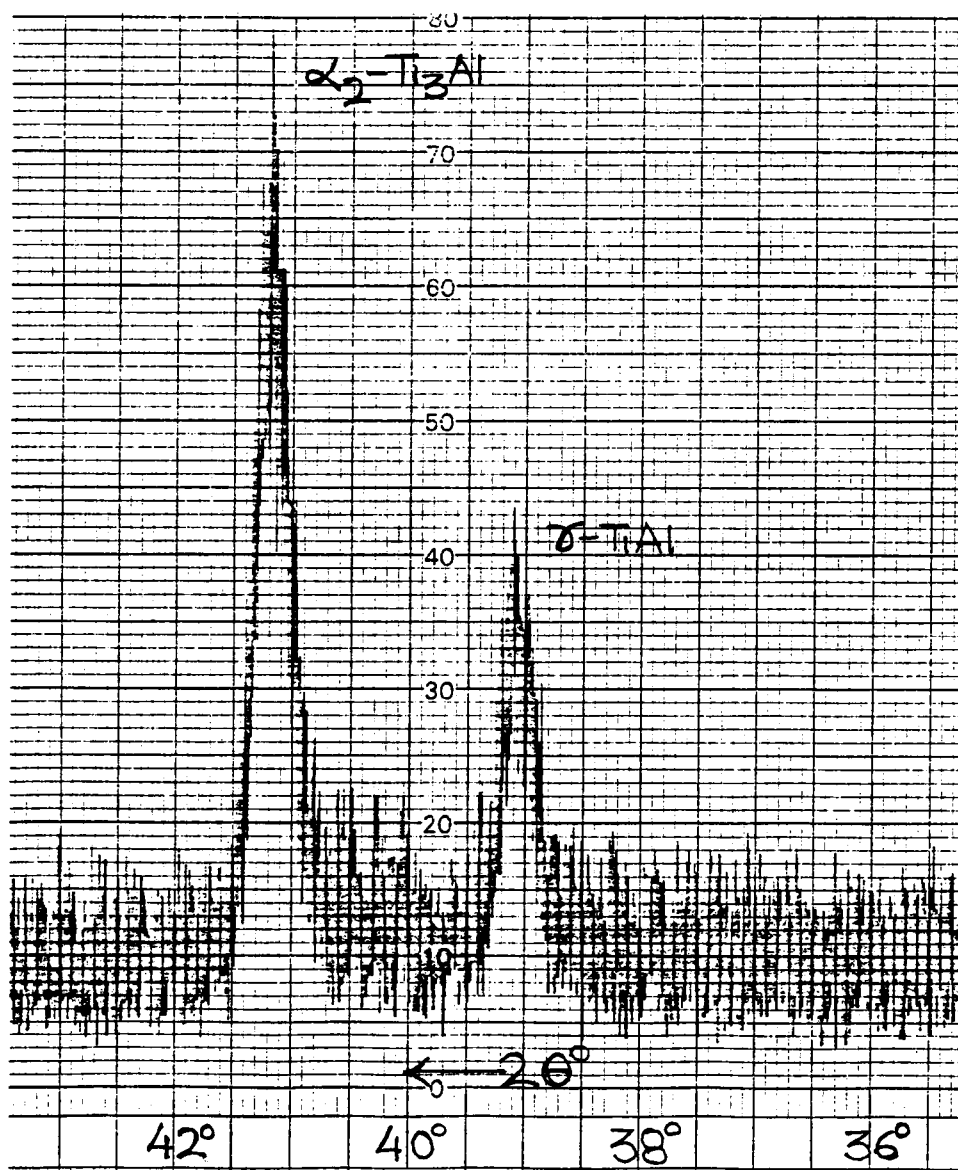


Figure 44. X-ray diffraction evidence indicating the two phase $\alpha_2 + \gamma$ nature of the drop cast Ti-54at%Al ingot.

difference in solidification velocity and the nature of turbulence during solidification for the two different processing conditions. For directional solidification, a slow growth condition in a turbulent melt is maintained. For arc-cast materials, solidification proceeds at a higher rate in a quiet melt (once the arc is cut off there is no further mixing turbulence).

For slow growth solidification processing, local equilibrium at the growing interface is obtained, neglecting the small finite undercooling required to nucleate the γ phase. This indicates that a Ti-54at%Al composition is beyond the α +L phase field. However, α dendrites in the initial drop-cast ingot suggests solidification on the metastable extension of the α +L phase field to higher aluminum concentrations. Similar α dendrites have been reported (6) in arc-cast Ti-55at%Al compositions (figure 7d of this reference). A growth interface exists in directionally solidified melts at all times. To ensure that the variation in microstructures was not influenced by this interface, similar conditions were simulated in arc melting. α was still nucleated at the solid interface.

It can be argued that γ has a significant nucleation barrier and an undercooled melt may preferentially form α in arc melted buttons. Additionally, since α has a high solubility for both nitrogen and oxygen, it is possible that metastable formation of α is favored by a large interstitial content in the liquid. The volume fraction of α increases

with increasing nitrogen content, see figures 32,42. It was earlier shown that for a high nitrogen containing melt, Ti_2AlN particles were formed in the melt and α nucleated on these nitride particles, and not τ , with little undercooling. The preferential nucleation of α in a nitrogen contaminated liquid possibly reflects the ability of the melt to lower its nitrogen content. For arc melting conditions with high freezing velocities, α formation effectively getters the nitrogen from the melt as its partitioning coefficient >1 . Upon homogenization to obtain single phase τ , this excess nitrogen precipitates as Ti_2AlN , because of the lower solubility of N in τ . This is indeed the case for homogenized TiAl+N alloys, where the nitrides are predominantly localized near and within the prior α regions, figures 35, 36, 40.

It is evident that α can be nucleated under conditions of fast growth velocities and nominal nitrogen contents. It is possible that these two effects are interdependent. A rapidly freezing melt presents a kinetic barrier for the escape of the interstitials dissolved in the liquid. It is argued that the inability of the liquid to reject interstitials promotes α formation. This point is supported somewhat by the directional solidification slow growth experiments, where the interstitial content is significantly reduced upon processing (63) and only τ is formed. This interstitial rejection is aided by the inductively stirred liquid zone.

Summary and Conclusions

Based on the experimental observations, the following conclusions are drawn:

1. Under directional solidification of slow growth and minimal interstitial content, a Ti-54at%Al composition is beyond the α +L phase field and solidifies as single phase τ .
2. Under arc-melting of fast growth conditions, where comparatively larger undercoolings are expected to prevail, α can be preferentially nucleated from the melt. This then suggests a possible nucleation barrier to τ .
3. In N containing melts, where a heterogeneous nucleation agent (Ti_2AlN) exists, α is still preferentially nucleated and getters the melt of nitrogen.

Finally, while this effect has been elucidated only for nitrogen, similar effects may exist for oxygen since it too preferentially partitions to the α phase.

PART 2.

PRELIMINARY TERNARY ALLOYING EXPERIMENTSDefinition of the problem

Binary TiAl is brittle, but there is a slight ductility if a small volume fraction of Ti₃Al is present. Presumably there are few active slip systems contributing to the deformation at ambient temperatures. Attempts to ductilize TiAl is currently a subject of intense interest for its potential high temperature applications. Two different approaches to attain this objective would be: (i) developing two phase alloys such that the superior mechanical and high temperature properties of TiAl can be combined with a more ductile matrix providing good formability and (ii) modifying the TiAl single phase with ternary additions thereby attempting to alter the deformation behavior of this compound. Two phase microstructure development is a problem of modifying the chemistry to obtain varying volume fractions of $\alpha_2 + \gamma$ microstructures. The method of selecting suitable ternary additions for modifying deformation behavior is however an obscure one. No clear rationale was available as to the choice of a particular ternary addition at the beginning of this study. It was intended to study several ternary additions at least in the preliminary stages to develop certain comparisons between microhardness measurements and microstructures. Microhardness measurements are a rough guide to a materials resistance to plastic

deformation, and in the preliminary studies were used extensively to rank ternary additions. An important condition imposed was to choose ternary additions that would not promote ternary compound formation. Towards this end, the choice was geared towards elements that exhibited extended solubility in either titanium or aluminum.

Research efforts were focused on developing a suitable ternary alloying program for TiAl based on certain physical concepts developed from elemental properties like atomic and ionic radii, electronegativity parameter, valency and melting points of the elements to be added.

Fundamental insight into using such elemental property information towards alloying and structure determination was first laid down by Villars (67). He used a systematic elimination procedure to find three atomic property expressions which could be used to distinguish the crystal structures of AB intermetallic compounds. The three expressions giving the best separation are:

- a) $(r_s + r_p)^{z_{AB}} = (r_s + r_p)^{z_A} - (r_s + r_p)^{z_B}$ of the difference of Zunger's (68) pseudopotential radii sums.
- b) $X_{AB}^{MB} = X_A^{MB} - X_B^{MB}$ of the Martynov-Batsanov (MB) electronegativity difference, and
- c) ΣVE_{AB} of the number of valence electrons (VE).

It must be noted that TiAl did not fall in the calculated AuCu (L10) domain, but appears in the BCr (Oc8) domain which also has structures of AlHf, AlTh, AlY, AlZr, etc.

Later these expressions were modified and new parameters

substituted as

- a) $(r_s + r_p)^z_{AB} = (r_s + r_p)^z_A - (r_s + r_p)^z_B$ of the difference of the pseudopotential radii sums.
- b) the ratio T_A/T_B of the melting temperatures of the constituent elements with $T_A > T_B$ and
- c) VE_{AB} of the difference between the number of valence electrons.

Elements in combinations of the solubility types are characterized by a close similarity of their relevant physical or calculated atomic properties. One such property chosen was the difference in the Zunger pseudopotential radii sums (68), taken to be less than 10%. Accordingly the following solubility types were established for titanium; Ti-Nb, Ti-V, Ti-Zr, Ti-Hf, Ti-Mo, Ti-Ta and aluminum; Al-Ga, Al-Si.

Nb and V ternary additions were selected to substitute for titanium and Ga was chosen to substitute for aluminum. Another addition, Mn, not derived from the above considerations, was also selected for ternary alloying.

Results and Discussion

Ternary additions of 5at% were made to different TiAl compositions with a varying Ti/Al ratio. The microhardness data is shown for a series of compositions, figure 45, for the binary TiAl and TiAl+X alloys. Optical microstructures of TiAl, TiAl+Nb, TiAl+V and TiAl+Ga alloys are shown in figure 46-49 respectively. A hardness minima was established

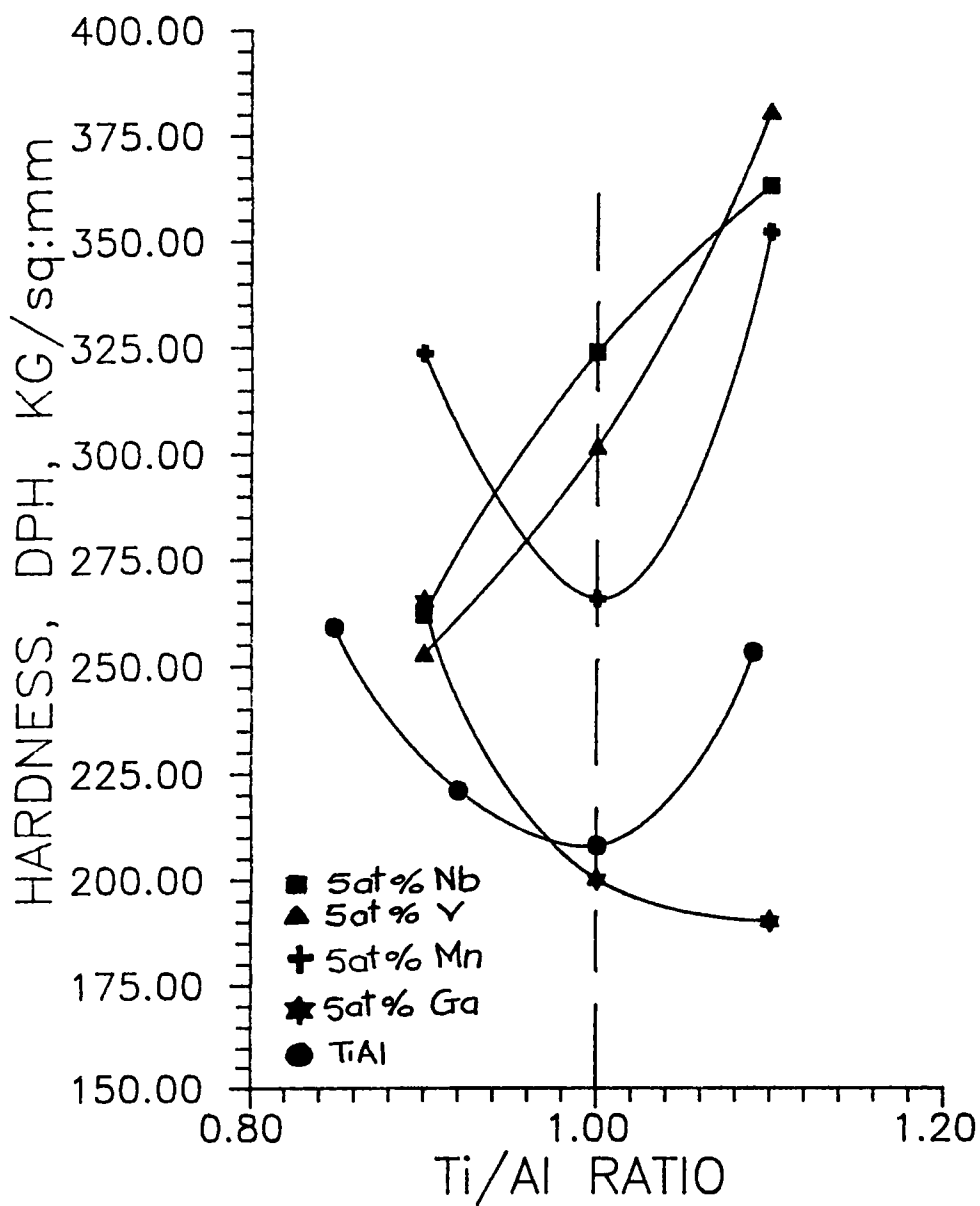


Figure 45. Microhardness values of selected binary TiAl and ternary alloyed TiAl with a varying Ti/Al ratio.

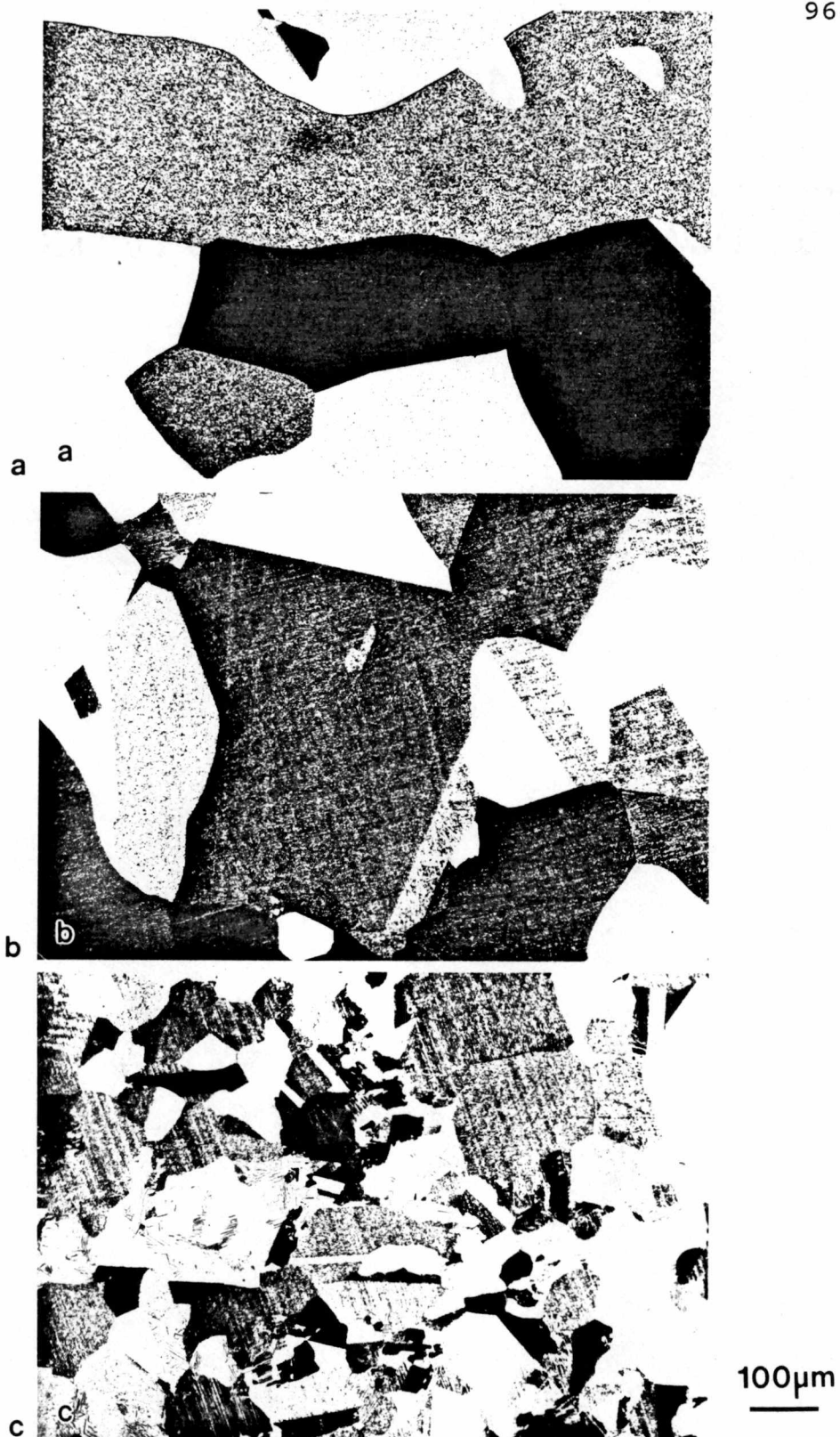


Figure 46. Optical microstructures of binary TiAl with a varying Ti/Al ratio. a) Ti/Al=0.9 b) Ti/Al=1.0 c) Ti/Al=1.1

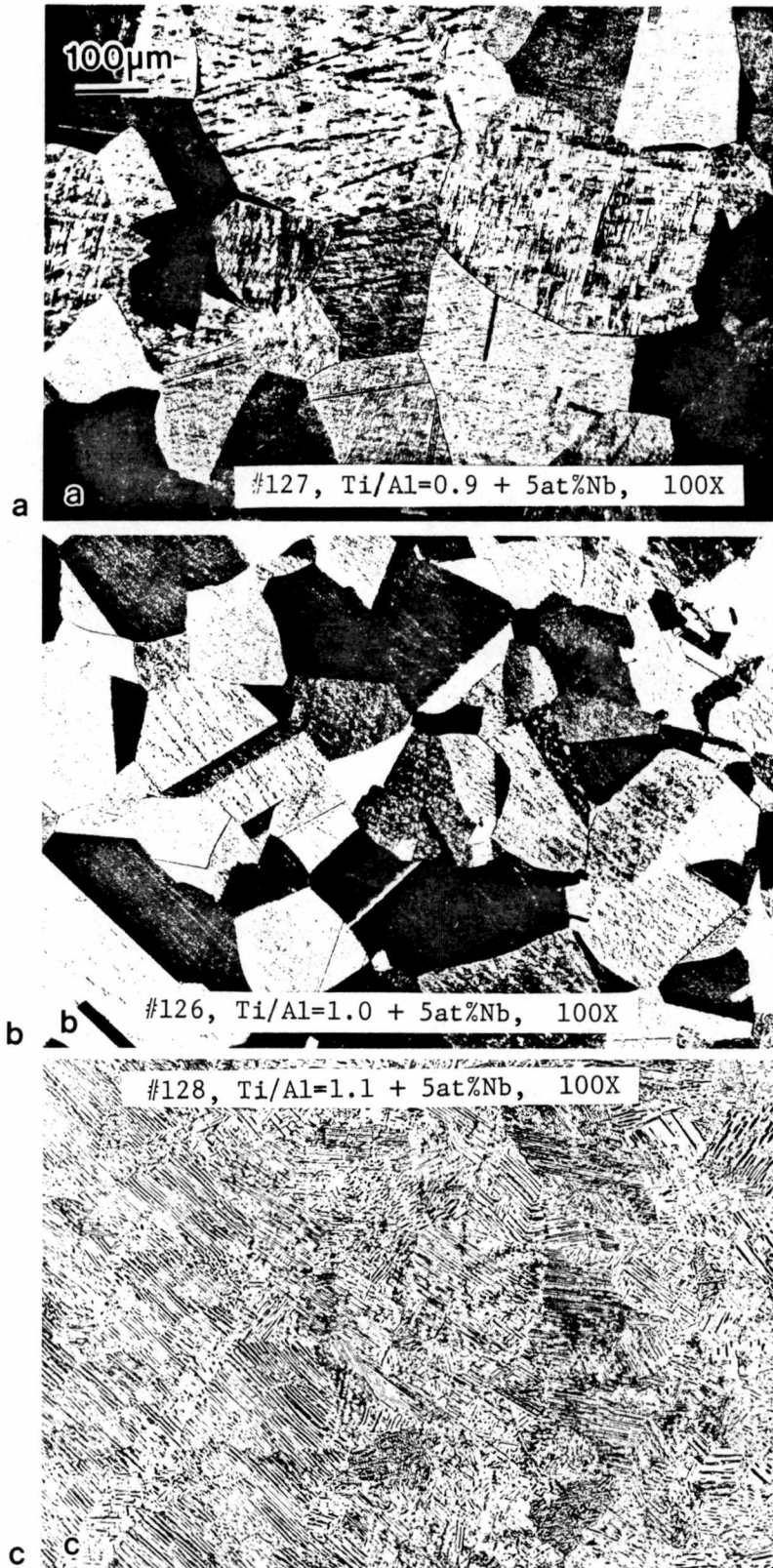


Figure 47. Optical microstructures of TiAl+5at%Nb with a varying Ti/Al ratio. a) Ti/Al=0.9 b) Ti/Al=1.0 c) Ti/Al=1.1



Figure 48. Optical microstructures of TiAl+5at%V with a varying Ti/Al ratio. a) Ti/Al=0.9 b) Ti/Al=1.0 c) Ti/Al=1.1

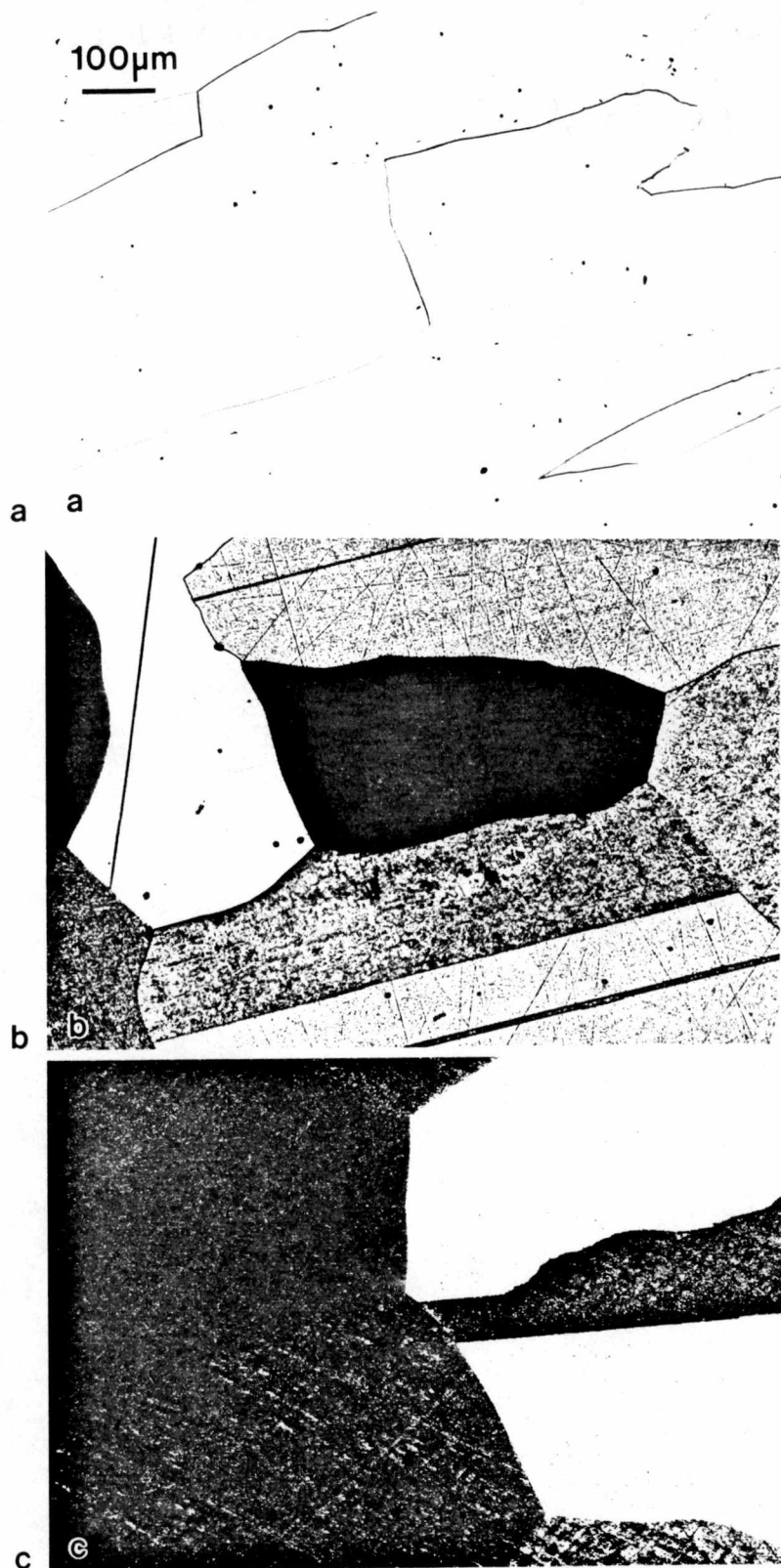


Figure 49. Optical microstructures of TiAl+5at%Ga with a varying Ti/Al ratio. a) Ti/Al=0.9 b) Ti/Al=1.0 c) Ti/Al=1.1

near the equiatomic Ti/Al ratios for binary TiAl alloys.

Optically the alloys with Ti/Al ratio >1 were single phase TiAl and those with Ti/Al ratio <1 were two phase τ -TiAl + α_2 -Ti₃Al. TiAl is brittle despite its low hardness values.

Since hardness is somewhat a measure of the material's resistance to deform plastically, it was envisaged that any ternary additions that decrease the hardness of TiAl may be of interest for further study. Figure 45 shows that Nb and V additions decreased the hardness towards the hypo Ti/Al ratios whereas Ga decreased towards the hyper Ti/Al ratios. Several interesting points are evident from this plot.

First, the ternary elements reduced the hardness in the direction in which they preferentially substitute. For example both Nb and V were expected to substitute for Ti and Ga for Al. Hence if the Ti/Al ratios are modified as effective $\text{Ti} + \text{X} / \text{Al} + \text{Y}$, the resulting hardness plot shows a minimum at $\text{Ti} + \text{X} / \text{Al} + \text{Y} = 1$. Here $\text{X} = \text{Nb}, \text{V}$ and $\text{Y} = \text{Ga}$. This trend was verified using microstructural information when compared to the TiAl phase diagram. TiAl(τ) has a τ/α_2 phase boundary at roughly 50at%Ti, as verified by a two phase $\alpha_2 + \tau$ microstructure for 50.5at%Ti alloy. When the effective Ti (i.e. $\text{Ti} + \text{X}$) exceeded 50at%, two phase microstructure were observed, figures 47b,c and 48b,c. Note that with ternary additions a slight shift in the τ/α_2 phase boundary either to a Ti-rich or Al-rich region is possible. In the event of a significant phase boundary shift, a small ternary addition may not reveal the exact nature of the substitution.

However, a 5at% ternary addition revealed preferential substitution effects as mentioned above. It is observed that this simple technique provides sufficient information as to the nature of the substitution.

Note that even though Nb and V reduced the hardness values when substituted for Ti, they were still higher than the binary alloys of equivalent compositions. In contrast, Ga when substituted for Al reduced the hardness values when compared to equivalent binary compositions.

To better establish the hardness minimum for TiAl+Ga alloys, a series of alloys were prepared keeping Ti at 50at% and adding Ga from 2 to 10at%. The hardness plot shows a shallow minimum at about 5at%Ga addition, figure 50. Optical microscopy revealed a single phase TiAl(τ) for up to 7at% Ga, with a small volume fraction of second phase observed at a 10at%Ga addition.

Mn Addition

The Mn addition was made following reports of its ductilizing effect by Tsujimoto et al. It was suggested that Mn increases the twinning tendency. A 5at%Mn addition was made at varying Ti/Al ratios and the hardness data and microstructures are reported, figure 51. All the microstructures with a Mn addition showed varying amounts of second phase present, figure 51. The Mn addition at equiatomic Ti/Al ratio revealed the lowest hardness accompanied by a extensively twinned microstructure, figure

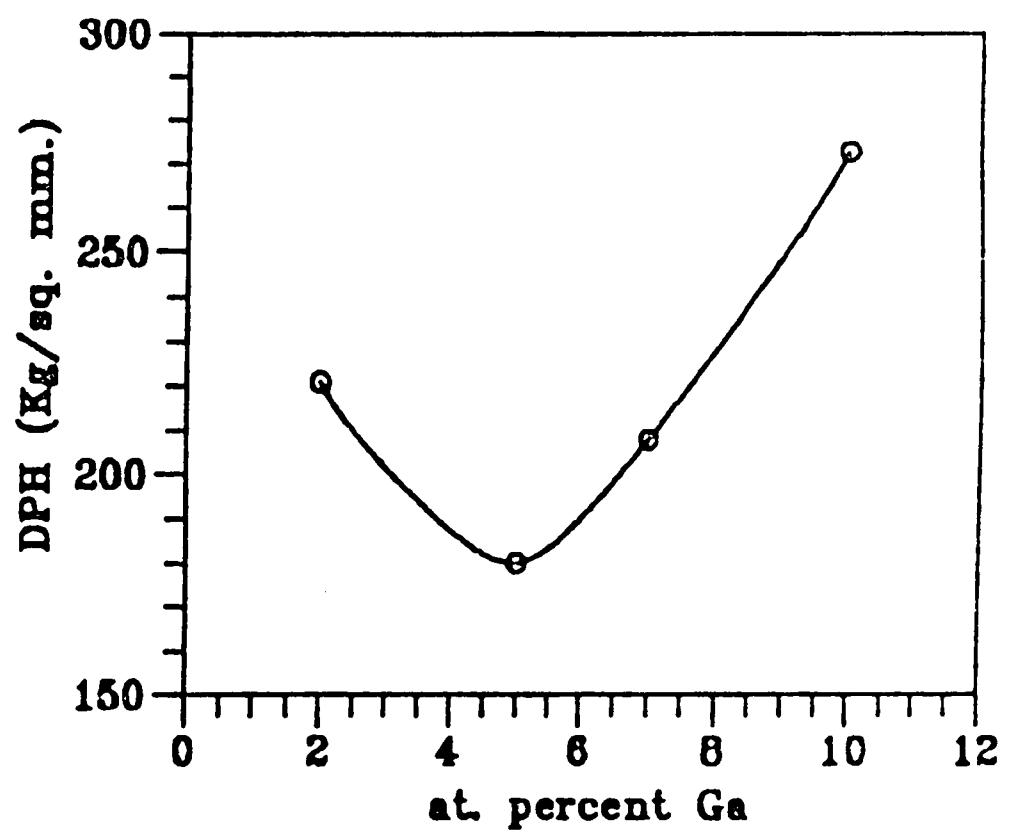


Figure 50. Microhardness plot for Ti-50at%/(Al+Ga)-50at% for increasing addition of gallium.

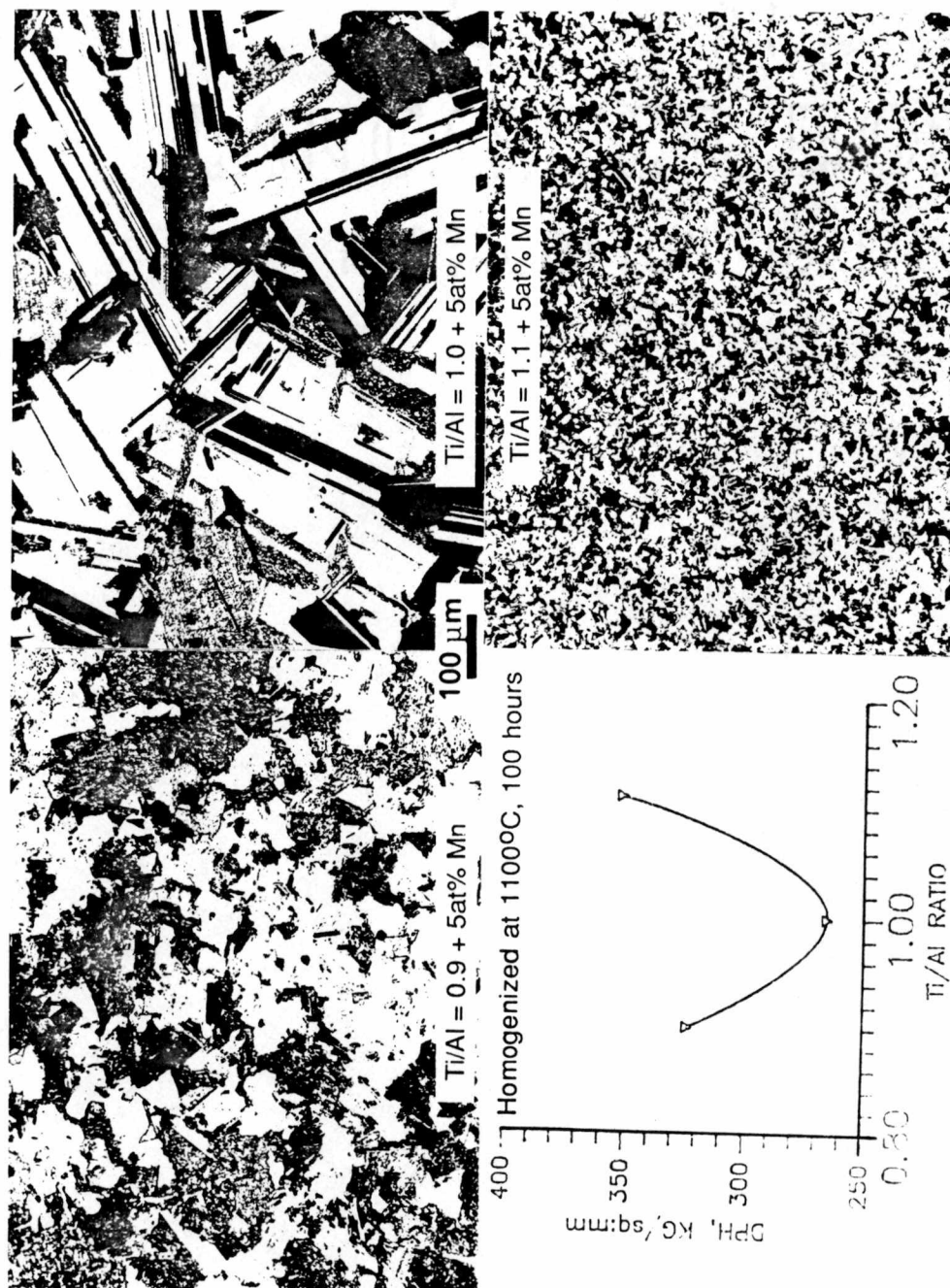


Figure 51. Optical microstructures and microhardness values of selected TiAl+5at%Mn alloys with a varying Ti/Al ratio.

51. Note that the substitution scheme developed above does not provide any clues as to the nature of Mn substitution. The development of the microstructure with increasing Mn additions is shown in figure 52. The Mn addition was selected for further study, due to its microstructural modifications. To better establish the hardness minima, Mn additions 1-10at% were added, keeping Ti/Al=1. The resulting hardness plot is shown in figure 53. A micro addition of 1at%Mn significantly hardened the alloy. At 3.8%Mn twinning was observed accompanied with a slight drop in hardness. Clearly a 5at%Mn addition was found to be optimum from a hardness standpoint and was selected for further study.

Electron Microscopy Observations

Electron microscopy observations were performed on compression specimens of the homogenized alloys primarily to monitor the presence of $\frac{1}{2}[110]$ dislocations. Since $\frac{1}{2}[110]$ perfect dislocations are the easy deformation mode, as compared to the super dislocations $\langle 011 \rangle$ and $\langle 101 \rangle$, their presence and mobility will enhance the deformation characteristics of the material. For the purpose of this thesis only selected micrographs are presented to indicate the presence of $\frac{1}{2}[110]$ dislocations.

1) Electron Microscopy Observations for TiAl+Ga Alloys

Contrary to optical microscopy results of TiAl+Ga alloys being single phase, thin laths of Ti_3Al laths were

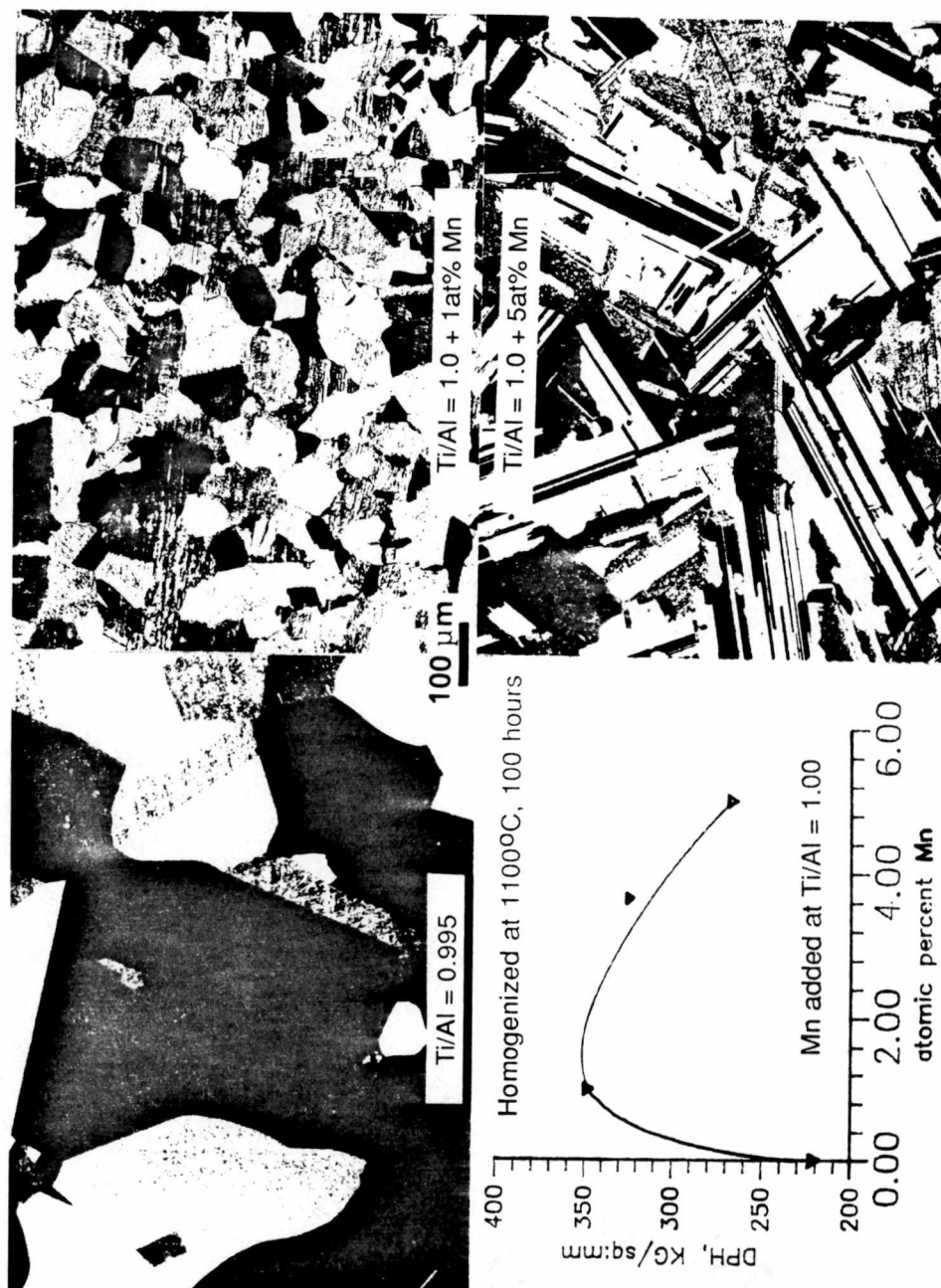


Figure 52. Microhardness data and optical microstructures for $\text{Ti/Al}=1+\text{varying at\%Mn}$ addition.

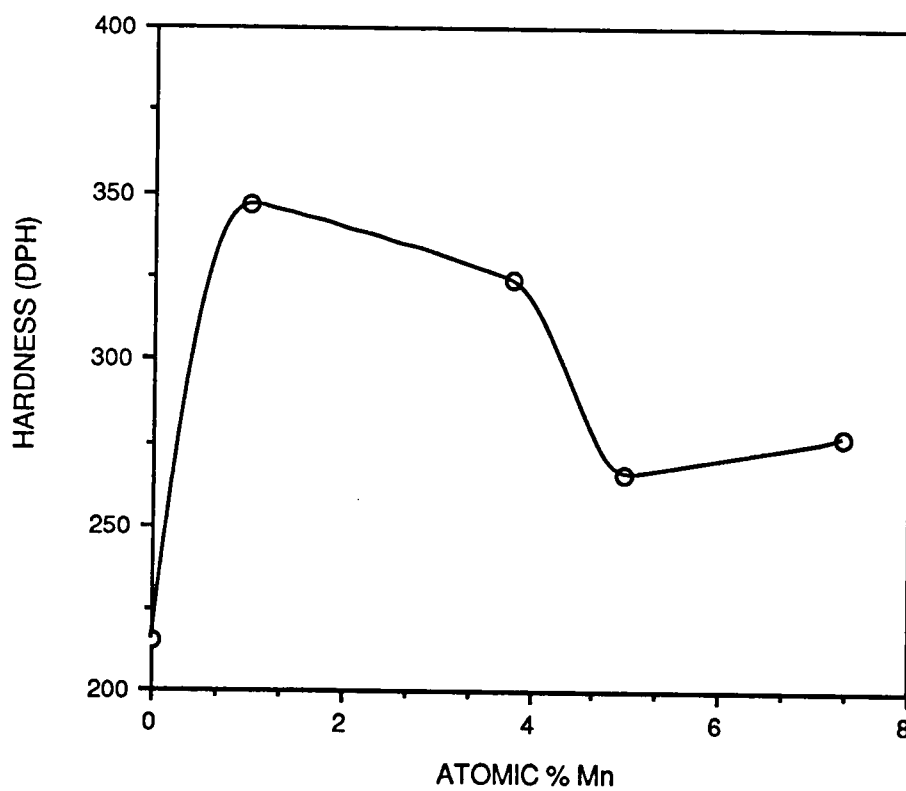


Figure 53. Microhardness plot for Ti/Al=1+varying at%Mn additions indicating a hardness minima at 5at% Mn.

observed occasionally in homogenized alloys. Hence the material was not truly single phase τ . Deformed compression specimens show the presence of $\frac{1}{2}[110]$ dislocations, figure 54. Since $\frac{1}{2}[110]$ dislocations display a characteristic invisibility for a diffracting vector $g=002$ based on $g.b=0$, this condition is used to characterize $\frac{1}{2}[110]$ dislocations as shown in figure 54. The detailed dislocation observations are reported elsewhere (17) by the author, a copy of which is reproduced in appendix 1.

2) Electron Microscopy Observations in TiAl+Mn Alloys

Figure 55 shows selected micrographs from a $g.b$ analysis performed on specimens deformed in compression. All micrographs are shown with $B=[011]$. For a diffracting vector $g=200$, dislocations A,B and stacking faults C are visible. Dislocation A is representative of all horizontally oriented dislocations. It shows a $g.b=0$ type contrast for $g=11\bar{1}$ and 002 . Additionally it shows a $g.b=1$ for 200 , $1\bar{1}1$ and $g.b=2$ for $3\bar{1}1$. Its burgers vector was identified as $\frac{1}{2}[110]$. Dislocation B shows $g.b=0$, near invisibility for $1\bar{1}1$ and $g.b=2$ type contrast for $11\bar{1}$ and $3\bar{1}1$. It was identified as $a[101]$. The stacking faults were bounded by a drawn out loop of $a/6\langle 112 \rangle$ partial. Both bounding partials for the stacking fault are invisible for $g=3\bar{1}1$. These faults are $a/3\{111\}$ and of intrinsic nature. Dipoles D are also observed. Additional dislocation observations in TiAl+Mn compression specimens have previously been reported elsewhere (65) by the author.

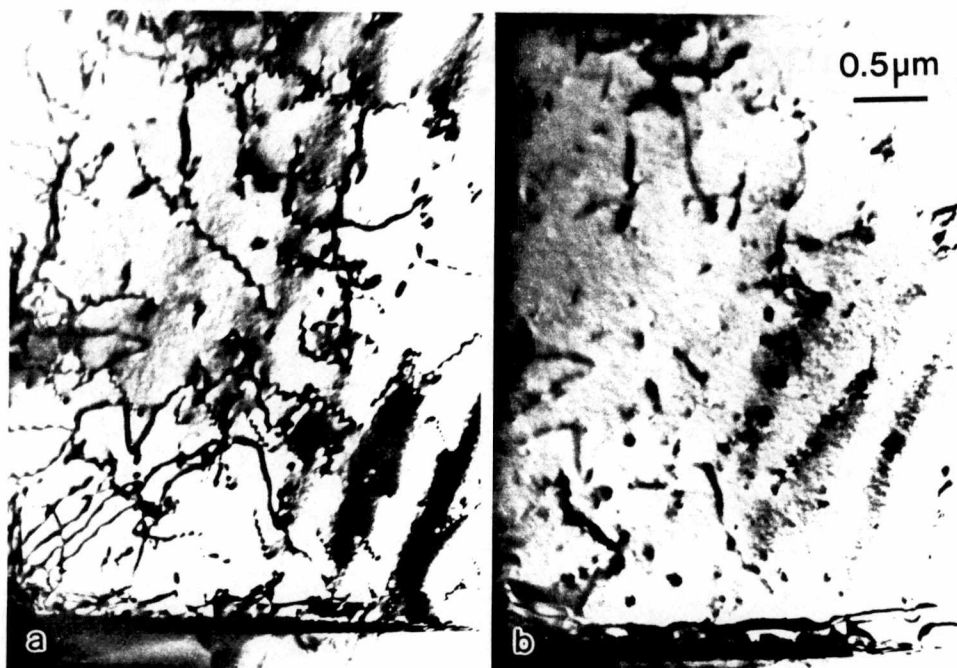


Figure 54. TEM micrographs of $\frac{1}{2}[110]$ dislocations in Ti-45at%Al5at%Ga deformed in compression. a) diffracting vector $g=020$ b) diffracting vector $g=002$. Beam direction $=100$.



Figure 55. TEM micrographs of dislocation observations in TiAl+Mn alloy deformed in compression.

Summary and Conclusions

Based on the preliminary studies the following conclusions are drawn:

- 1) Gallium exhibits an extended solubility range in γ -TiAl and softens the TiAl matrix over a 2-5at% addition range
- 2) Mn promotes extensive twinning in TiAl with a significant softening accompanying the twinned microstructure
- 3) Both the gallium and manganese modified TiAl alloys show an abundance of $\frac{1}{2}[110]$ dislocations which are expected to aid the deformation behavior of TiAl.

For the purpose of alloy development and further tensile testing, the compositions of Ti-45at%Al5at%Ga and Ti-47.5at%Al5at%Mn were selected. Part 3 reports the mechanical properties of these specific compositions unless otherwise stated.

PART 3.

MICROSTRUCTURES AND MECHANICAL PROPERTIES OF TERNARY
ALLOYED TiAlDefinition of the Problem

A review of the literature presents interesting problems towards the design of TiAl alloys with appreciable room temperature ductility. Most notable is the increment in ductility of two phase $\text{Ti}_3\text{Al}(\alpha_2)+\text{TiAl}(\tau)$ alloys over single phase $\text{TiAl}(\tau)$ alloys. The poor ductility in single phase $\text{TiAl}(\tau)$ alloys has commonly been attributed to a nominal interstitial content of carbon, nitrogen and oxygen, possibly present in the alloys as a result of poor processing control. Increased ductility of two phase $\text{Ti}_3\text{Al}(\alpha_2)+\text{TiAl}(\tau)$ alloys is being attributed to the presence of $\frac{1}{2}\langle 110 \rangle$ dislocations contributing to plastic deformation. An important question as to why $\frac{1}{2}\langle 110 \rangle$ dislocations are present in two phase alloys and not in single phase alloys was addressed recently by Vasudevan et al. (46)., where it was shown that $\text{Ti}_3\text{Al}(\alpha_2)$ acts as to internally getter the $\text{TiAl}(\tau)$ of its interstitial content, thereby making the $\frac{1}{2}\langle 110 \rangle$ dislocations mobile. The exacerbation of deep Peierls valleys or the stronger locking of the $\frac{1}{2}\langle 110 \rangle$ dislocations by the interstitial impurities renders them sessile. Vasudevan et al. (47) have shown in a separate investigation that for single phase alloys $\text{TiAl}(\tau)$, when the interstitial oxygen content is gettered internally by erbium, $\frac{1}{2}\langle 110 \rangle$

contribute to plastic deformation.

Based on the these earlier observations, several factors were designed to be included in the current study. Firstly it was attempted to employ means to produce high purity alloys with the lowest possible interstitial content. Towards this end we have used the directional solidification equipment to generate large grained material, processed in Pd-purified hydrogen atmosphere, which yields extremely low levels of interstitial contamination (63). Secondly, since there is a large anomaly between reported plastic strains in compression and tension, it was desired to perform tensile tests exclusively to ascertain the ductility parameters. Compression tests have been used, as reported in the earlier chapter, primarily to rank different alloys and to characterize the deformation behavior. For alloys that exhibited ambient temperature ductility, tensile tests were extended to high temperatures to monitor the strength at the intended application temperatures for these alloys. Thirdly, it suffices to say that an important aspect of this study was to compare two phase alloys to single phase alloys. An important point to be noted here is that the increase in ductility in two phase alloys could possibly be attributed to a varying composition of TiAl alloys to produce single phase and two phase alloys, i.e., the contribution of alloy chemistry to ductility cannot be ruled out. It was attempted to study the same alloy under different thermomechanical treatments to produce varying microstructures. Such a study

may better define the role of alloy chemistry in contributing to the ductilizing effect.

Results and Discussion

Interpretations of Microstructures in Directionally Solidified TiAl+Ga Alloys.

1) As-cast Alloy

The development of solidification microstructures in a directionally solidified bar are of special interest. Firstly because of the slow growth nature of the process, the solidification path follows the equilibrium phase boundaries (i.e., neglecting the finite undercooling required to nucleate the solid phase). Secondly due to the nature of an essentially uniaxial heat flow, the solid state transformations follow closely behind the solid-liquid interface. In the initial stages of directional growth, the liquid composition at the solid-liquid interface is the same as the bulk composition. The solid forming from this liquid is of much lower solute content than the bulk solid composition, figure 56. As the liquid composition at the interface is enriched in solute as a result of finite growth, a steady state condition is approached where the composition of the freezing solid is the same as that of the bulk composition. At the end of zone processing, there is a solute rich solid. The resulting solid composition along the length of the processed bar is shown in figure 56. The general microstructures as observed along the length of the

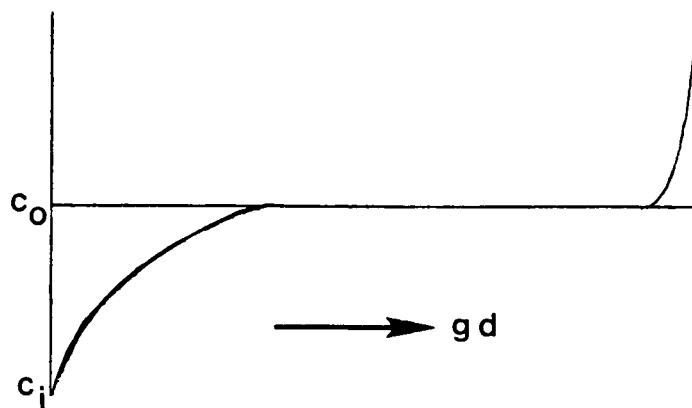
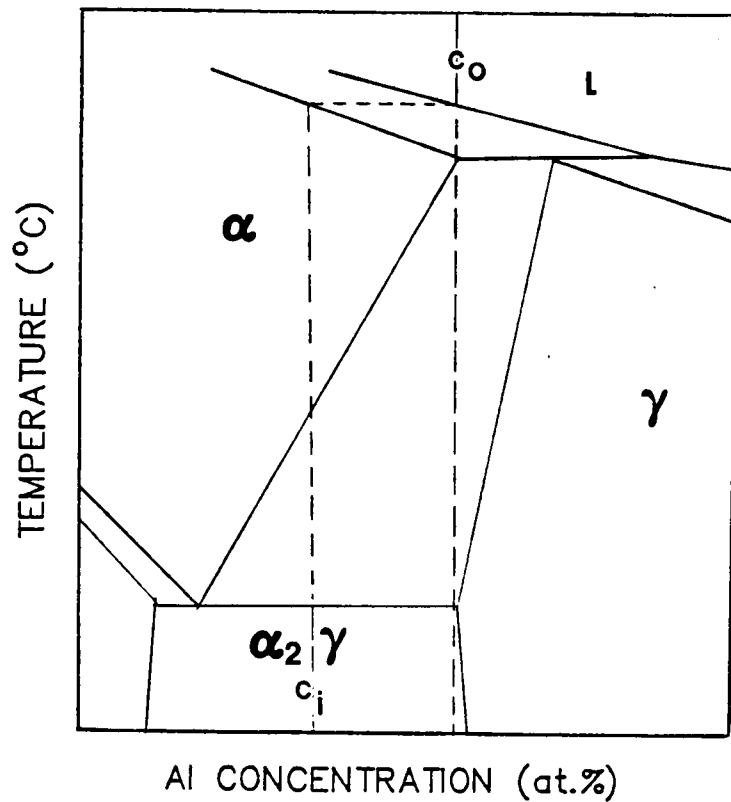


Figure 56. Schematic Ti-Al phase diagram with compositional profiles along the length of the directionally solidified bar. C_0 =bulk composition, C_i =initial solid composition, gd=growth direction.

bar are shown in figure 57. Clearly, a difference is observed in the resulting microstructures along the length of the processed bar. The most notable difference is in the transition from predominantly α_2/τ lath structures in the initial transient zone to a cellular morphology, as observed in the middle portion of the bar continuing all the way to the last zone. In this case the cells are τ with α_2 as intercellular. An increase in the volume of intercellular phase is observed in the last zone, figure 57. Note that a similar intercellular morphology was observed for the Ti-52at%Al alloy during directional solidification, figure 30. The general condition for the presence of this morphology was a gradual enrichment of solute at the solid-liquid interface, resulting in a net increase in solute composition of the solid, as detailed in part 1. In part 1, it was shown that the solidification morphology changes from a α_2/τ lath morphology to a α_2/τ cellular morphology to single phase τ . Since the composition of the bar solidified here is lower in solute (i.e., 45at%Al as compared to 52at%Al or 45at%Al+5at%Ga=effective 50at%Al as compared to 52at%Al) the solidification microstructures will not go to complete τ , as confirmed by our observations. Hence the essential problem here is to understand the transition from lath to a cellular morphology. Attempts are made to reconcile this transition as a solid state transformation by following microstructures along the length of the bar.

The initial transient zone length of the solidified bar

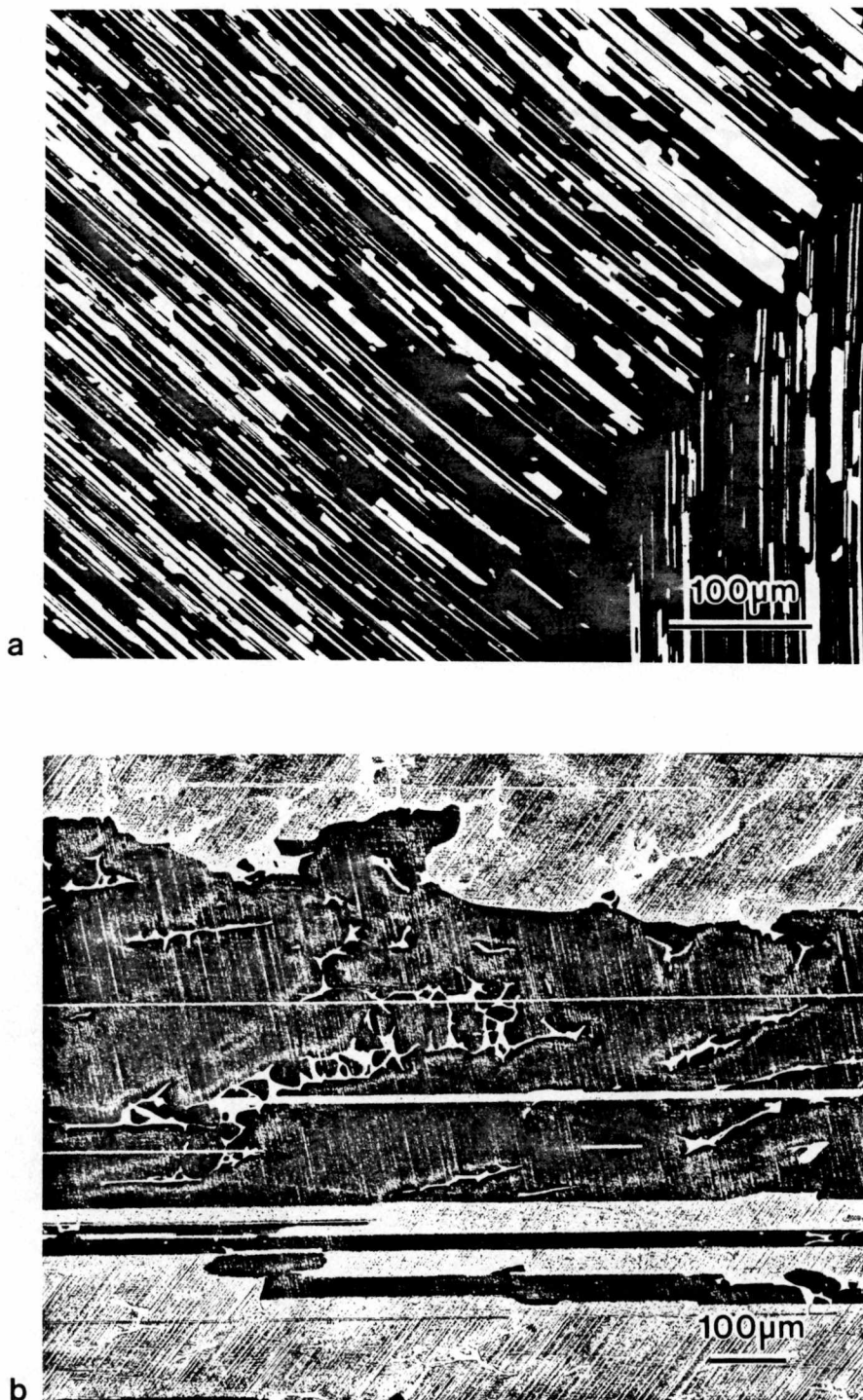


Figure 57. General nature of the microstructures along the length of the directionally solidified bar. a) Bottom region, transverse section b) middle region, longitudinal section c) top region, transverse section.



Figure 57. Continued

provides important clues to the nature of transformations occurring in the solid state. Working from the Ti-Al phase diagram, neglecting the deviation in the phase boundaries due to the presence of gallium, the Ti-45at%Al-5at%Ga liquid initially solidifies as α , with a small volume fraction of τ segregate at the boundaries, presumably forming either by a peritectic reaction or by a cellular liquid-solid interface. This α solid from the liquid undergoes two solid state transformations; $\alpha \rightarrow \tau + \alpha$ and then $\alpha \rightarrow \alpha_2 + \tau$, figure 2. For the solute lean solid, the $\alpha \rightarrow \tau$ transformation initiates at a lower temperature and then remains in a $\alpha + \tau$ two phase field during the cooling regime, figure 56. The remaining α then transforms to a $\alpha_2 + \tau$ mixture (first $\alpha \rightarrow \alpha_2$, then $\alpha_2 \rightarrow \alpha_2 + \tau$) with a $(0001)\alpha_2 // (111)\tau$ and $[1120]\alpha_2 // [110]\tau$ orientation relationship. As the freezing solid is enriched in solute, (i.e., freezing solid composition approaches bulk composition) the $\alpha \rightarrow \tau + \alpha$ transformation temperature increases and more of the prior α transforms to τ before cooling to 1398K, the eutectoid transformation temperature. At 1398K the remaining α first orders to α_2 and then decomposes according to the eutectoid reaction. Microstructures from the initial transient region of the bar clearly indicate this behavior, figure 58. It appears that the initial low temperature $\alpha \rightarrow \tau$ transformation may also have an identical $(0001)\alpha // (111)\tau$ orientation since all the α_2 and τ laths have a parallel orientation in the prior α grain, figures 58c,d. For the steady state solid forming from the liquid,

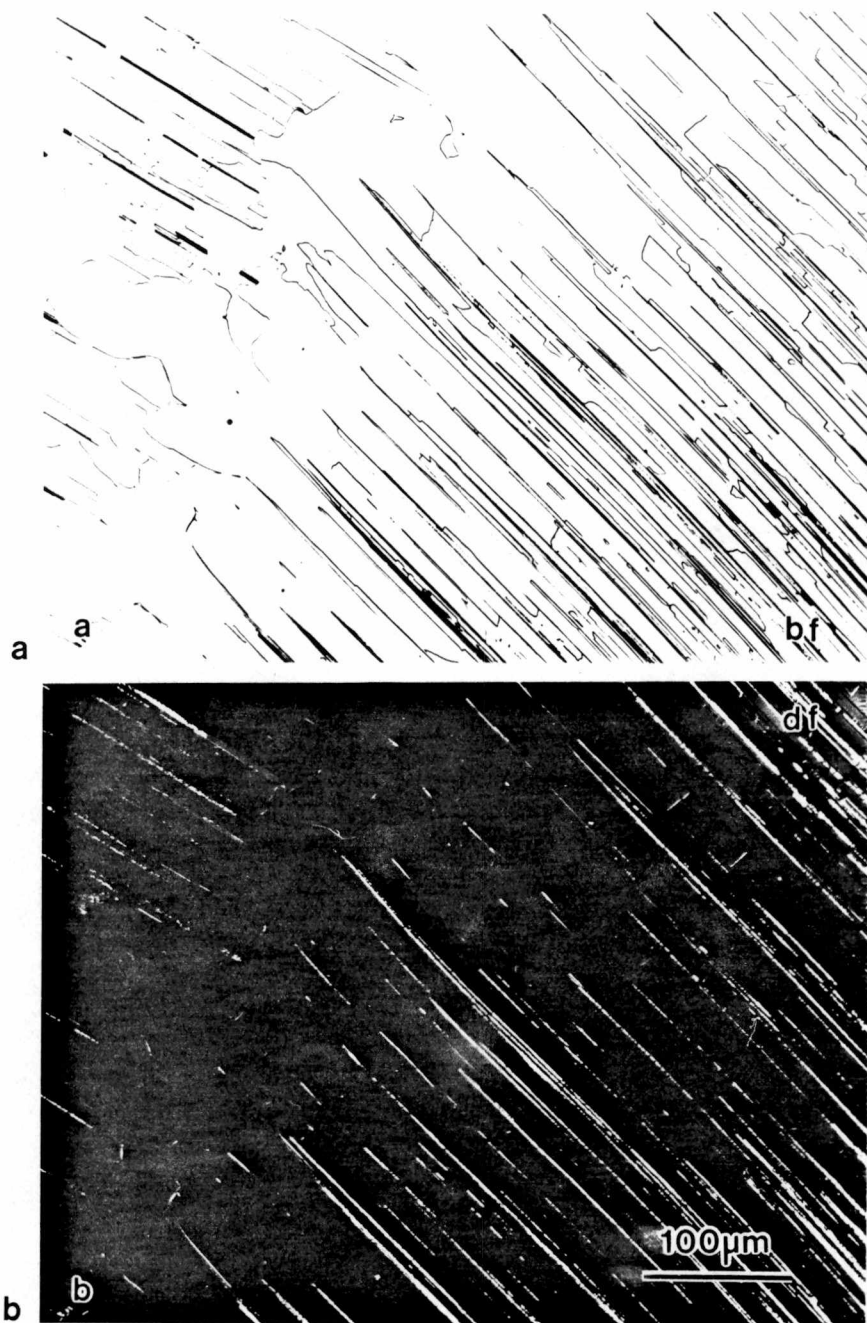


Figure 58. Optical micrographs from the initial transient region of the bar showing the gradual development of the microstructure for $\alpha \rightarrow \tau$ and $\alpha_2 + \tau$ transformations. a) Optical bright field and b) dark field images showing α_2 laths. c) τ laths originating from the α boundary into the α nearly parallel to the traces of (111) planes d) coarse τ growth into the α region.

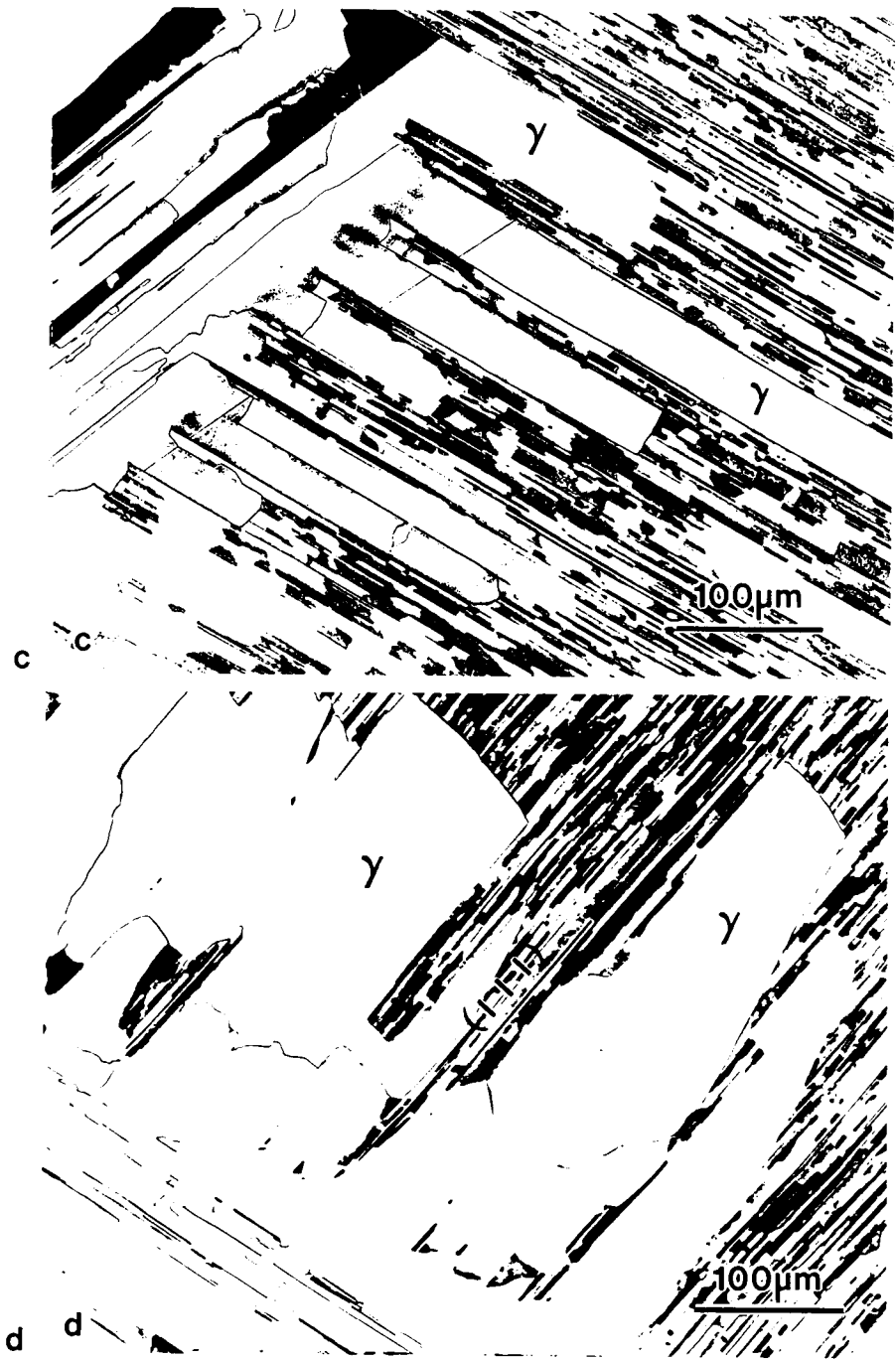


Figure 58. Continued.

figure 57b shows that $\alpha \rightarrow \tau$ transformation is almost complete. The microstructures show very little α_2 remnants, some in the lath orientation widely spaced, figure 59, and the rest intercellular, figure 60. It is my contention that, for a low $\alpha \rightarrow \tau$ transformation temperature, the τ laths grow from the boundary inwards into the prior α region. This τ may either nucleate independently at the grain boundary or grow on pre-existing τ at the α boundary. Presumably this τ grows nearly parallel to the basal plane of the α region, growth occurring by the forward motion of the plate into the α region and also by the thickening of the individual τ plates. The solute redistribution process in this case implies a rejection of titanium ahead of the τ plate and sideways into the α region. Note that for a lower transformation temperature, diffusivity is limited and growth of τ may be favorable by the sideways rejection of titanium. Upon cooling to the eutectoid transformation temperature, α undergoes a coherent ordering transformation to α_2 . This α_2 partially transforms to τ , following the eutectoid transformation path. This α_2 (DO₁₉ structure) possibly decomposes to $\alpha_2 + \tau$ by the Blackburn mechanism (64). This mechanism suggests that in changing the basal plane stacking from the usual pattern of ABAB to ABC, stacking faults in DO₁₉ are created, which are equivalent to monolayers of the L1₀ τ structure. It is suggested that these stacking faults are the nucleation mechanism for τ in α_2 , which may subsequently grow. TEM observations show few



5 μ m

Figure 59. TEM micrographs from the middle region of the bar indicating the lath distribution of α_2 .

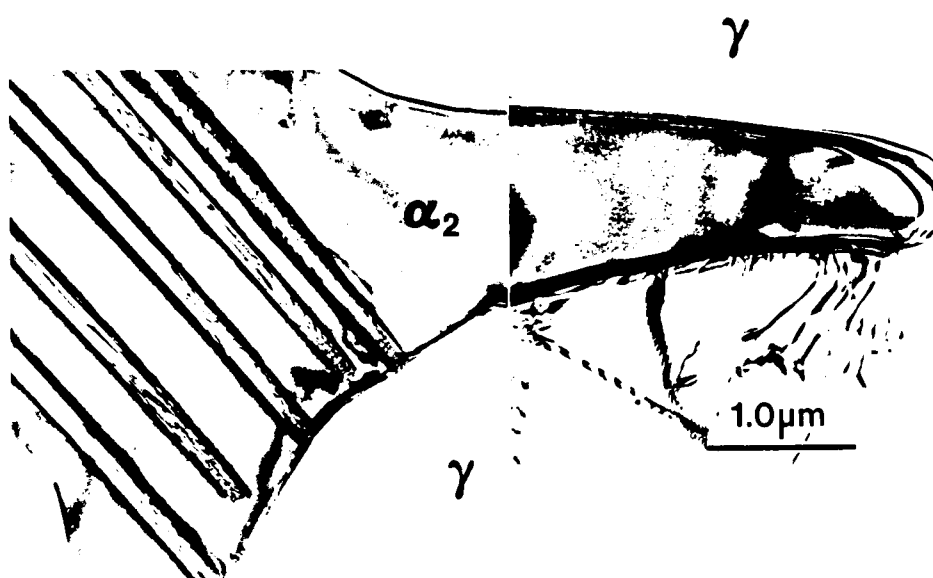


Figure 60. TEM micrographs from the middle region of the bar indicating the cellular morphology of α_2 .

instances of τ plates inside the α_2 regions. These τ plates are nucleated by the formation and growth of stacking faults inside the α_2 region as suggested by Blackburn, figure 61. The question as to why τ plates in α_2 are observed only sparsely can be addressed by the following. First, the slow growth and slow cooling experimental setup allows sufficient time for enrichment of the remaining α such that it approaches the equilibrium composition of α_2 . Second, τ formed into the prior α region may continue to grow into α_2 . This is possible because of the coherent ordering nature of the $\alpha \rightarrow \alpha_2$ transformation such that the orientation of α and α_2 remains identical.

For steady state solidification conditions, the α_2 intercellular morphology may be a result of rapid growth on the (111) plane of the adjacent τ regions. With time and diffusion conditions being satisfied, the α_2 laths may disappear completely leaving only adjacent regions of τ or twinned τ , figure 62. Alternately, under steady state solidification, the $\alpha \rightarrow \tau$ transformation will initiate at a higher temperature. Since, diffusion is expected increase at higher temperatures, several τ cells could originate and grow such that the adjacent regions are continuously enriched in titanium to give intercellular α_2 . It would be worthwhile to incorporate McCollough's (6) observation that in arc cast Ti-50at%Al alloys a cellular morphology was observed in a small region between the α dendrite and the τ segregate. This can be explained by considering a cored

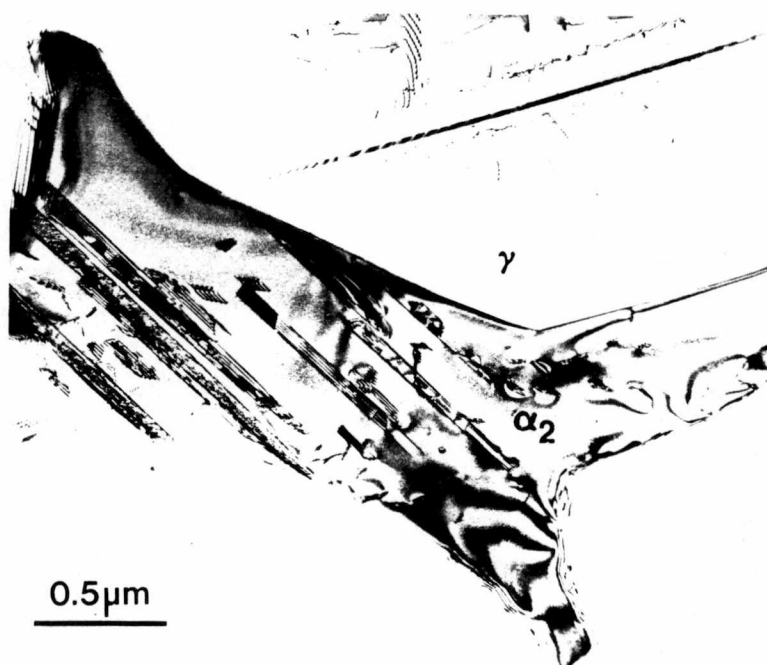


Figure 61. TEM micrograph of the nucleation and growth of τ stacking faults in α_2 .

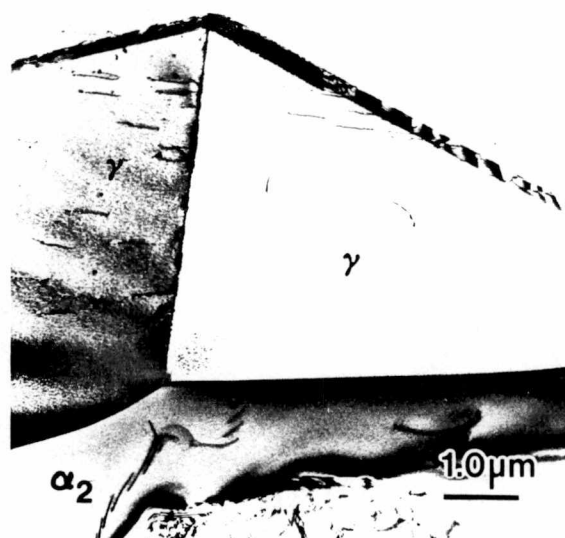


Figure 62. Adjacent regions of τ with α_2 as the intercellular phase.

dendrite. The outer layers of the dendrite are expected to be solute rich and undergo transformation at a higher temperature. This transformation is then predominantly cellular. However, the core of the dendrite is solute lean and will undergo transformation at a much lower temperature. This transformation, McCollough reports, occurs as lath structures, as has been observed here in the initial transient length of the bar, figure 57. Hence the formation of lath structures or cellular structures may be effected by an increase in the $\alpha \rightarrow \gamma$ transformation temperature which has to come from the increasing solute content of the solid. This ties in with the microstructural evidence for the Ti-52at%Al bar where an increase in solute content occurs concurrent with a cellular morphology. An important difference, of course, is that for the Ti-52at%Al bar the cellular morphology occurs as the solid composition changes from 50at%Al to $\approx$ 52at%Al. For the Ti-45at%Al5at%Ga alloy this cellular morphology is observed consistently in the steady state growth region. This effect could be incorporated by a shifting of the $\alpha \rightarrow \alpha + \gamma$ phase boundary towards higher titanium, possibly a ternary addition effect, with the net result of increasing the $\alpha \rightarrow \gamma$ transformation temperature to yield a diffusion dominated emergence of the cellular morphology.

Note that the predominance of cellular morphology depends on the low cooling rates as implied inherently in slow growth processing. It is likely that by imposing a

faster cooling rate, the formation of cells may be suppressed, resulting in a non equilibrium formation of α_2/τ structures.

The predominant crystal growth direction for this bar was [011]. Variations upto 15° off the growth direction were observed over the cross section of the solidified bar, as measured by tilt angles required in the TEM to obtain a [011] pole SADP, for the transverse specimens.

The tensile specimens were cut from the middle of the directionally solidified bar to ensure uniform composition in the tensile specimen gage length.

2) Homogenized TiAl+Ga Alloys

Upon homogenization at 1373K for a 100 hours, the microstructure of directionally solidified alloy shows a significant reduction in the volume fraction of α_2 . The distribution of α_2 is primarily as a discontinuous phase at the τ grain boundaries and sometimes within the τ grain, figure 63. The alloy however did not go to complete single phase as was observed for polycrystalline alloys. A possible reason is that due to the coarseness of the microstructure and the large diffusion distances involved, the temperature-time combination was not long enough to completely equilibrate the structure. An interesting feature of the remnant α_2 is its stringer like shapes, with a limited tendency to spherodize, figure 64. This could be the case for a low driving force in terms of surface energy criteria.

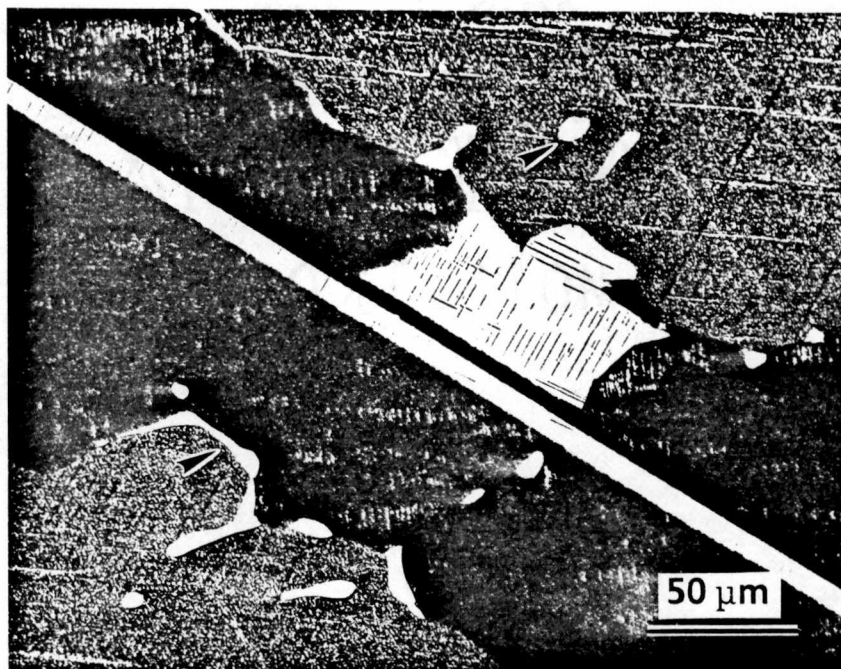


Figure 63. Optical micrograph of the homogenized structure showing scattered α_2 remnants (arrows), both at the τ grain boundaries and inside the τ grain.

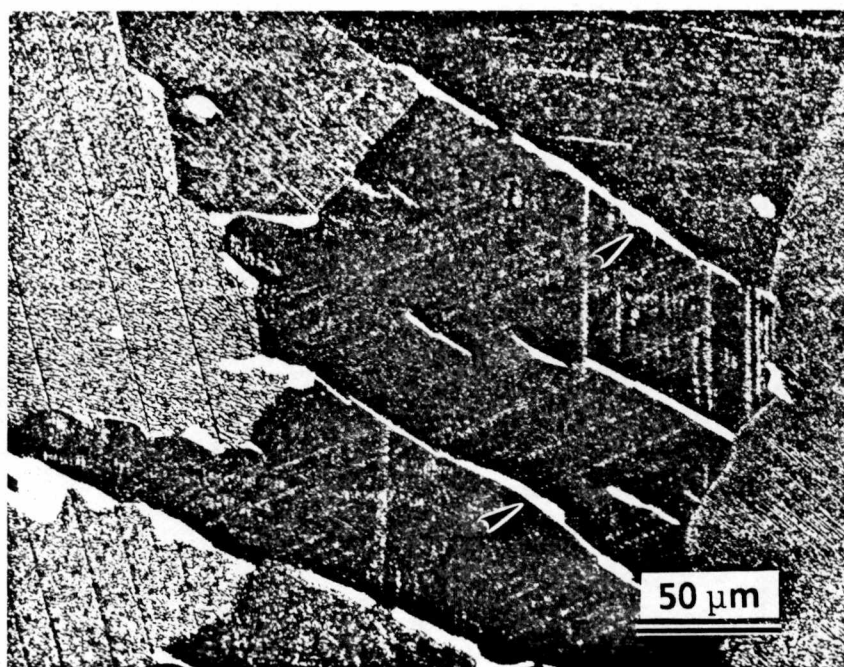


Figure 64. Stringer like shapes of α_2 in the homogenized alloy.

The tensile specimens for the homogenized bar were cut from the middle portion of the bar where the composition was essentially the same as the initial bulk composition.

Room Temperature Tensile Ductility of Directionally Solidified TiAl+Ga Alloy

1) As-cast Alloy

Table 8 lists the strength and ductility parameters of specimens tested at room temperature. A maximum of 3.72% plastic strain was observed in these specimens.

2) Homogenized TiAl+Ga Alloy

Table 8 lists the strengths and Ductility parameters of the homogenized alloys. The mechanical properties in general were significantly poorer than the as-cast alloy. Even at a lower strain rate of testing, the best ductility (i.e. 1% plastic strain) obtained was lower than the lowest ductility obtained (i.e., 1.66% plastic strain) for the as-cast material. A striking difference was the presence of microcracks in the homogenized material. These cracks were also present in the gage length of the tensile specimen, figure 65. The poor ductility observed following homogenization can possibly be attributed to the presence of such cracks. These pre-existing cracks were not localized near the α_2 phase, instead occurring in the γ region. It has been observed (see part 4) that single phase γ alloys show an increased propensity of crystallographic cracking. While

TABLE 8. ROOM TEMPERATURE TENSILE RESULTS OF DIRECTIONALLY SOLIDIFIED TIAL+GA ALLOYS.

Specimen No.	Yield Stress	Fracture Stress	Plastic Strain%
1) As-cast material, Strain rate= $3.33 \times 10^{-3} \text{sec}^{-1}$.			
ZM/AC-1	24.4Ksi	27.59Ksi	2.96
ZM/AC-2	25.6	28.98	3.72
ZM/AC-3	28.9	37.3	1.66
ZM/AC-4	-----	30.8	2.00
2) Homogenized at 1373K for 100 hours,			
ZM/H-1*	26.0	26.5Ksi	0.30
ZM/H-2*		20.8	
ZM/H-3**		21.5	
ZM/H-4**	22.2	22.9	1.00

* Strain rate= $3.33 \times 10^{-3} \text{sec}^{-1}$.** Strain rate= $6.67 \times 10^{-4} \text{sec}^{-1}$

ASTM specification E-8 specimen, reduced proportionally

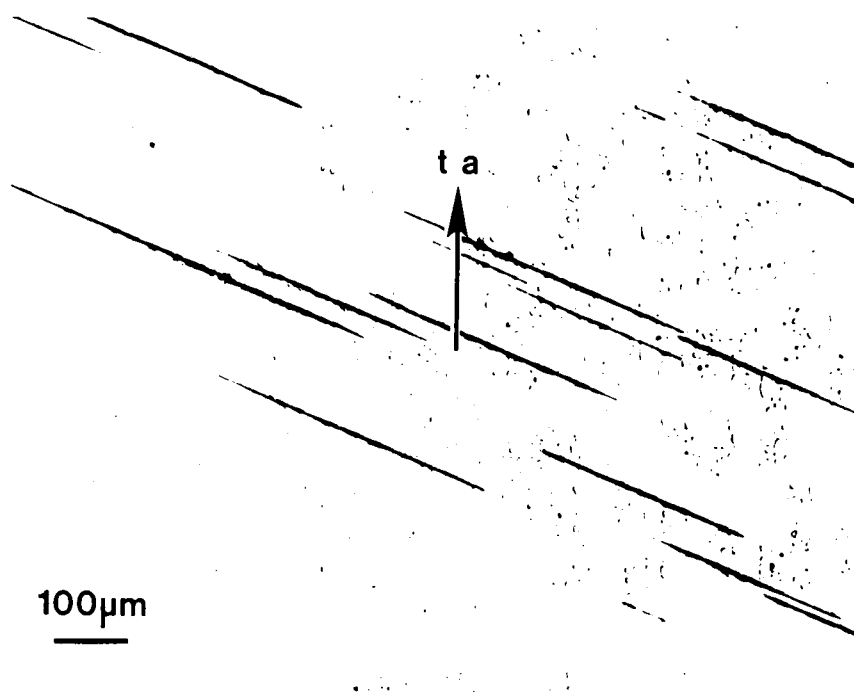


Figure 65. Pre-existing crystallographic cracks in the homogenized alloy, as observed in the gage length of the tensile specimen. tensile axis = ta.

the exact origin of these cracks is not clear, the propensity of crack formation dictates the need for careful handling, thereby, seriously undermining the potential of single phase τ as being a damage tolerant material. An alternative explanation for the low ductilities is the presence of α_2 in the specimen microstructures. The distribution of α_2 is shown in figures 63,64. Since the net effect of the homogenization treatment was to reduce the volume fraction of α_2 , the low ductility of the homogenized specimens can be correlated to a second phase effect. However, the presence of preexisting cracks in the homogenized specimens clouds this issue.

Deformation Behavior of Directionally Solidified TiAlGa

The deformation bands on the as-polished and tensile strained material are shown in figure 66. The deformation bands are continuous through the α_2 , τ and twinned τ regions. An interesting feature is the evidence of α_2/τ interface cracking, and the cracking associated with the α_2 regions, figure 66. This Ti-rich regions are readily evident in a BSE micrograph where the α_2 -Ti₃Al laths and intercellular regions appear bright, figure 67, 68. Seemingly, even though the volume fraction of α_2 is responsible for improving ductility, the cracks are almost always associated with the α_2 region, figure 67. These cracks propagate some distance into the τ and stop, thereby indicating that for as-cast material the τ phase is a good

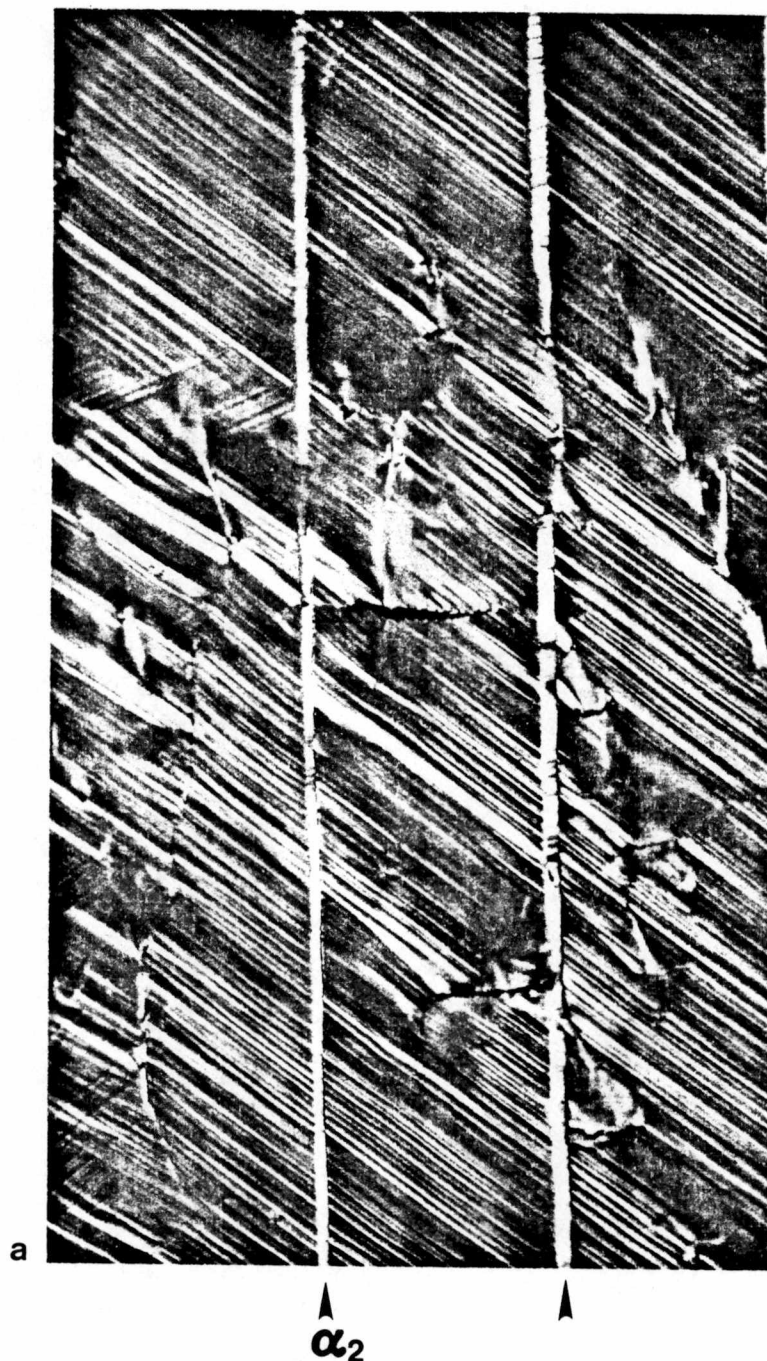


Figure 66. Optical micrographs of the tensile strained as-cast specimens. a) showing the deformation bands in single grain of τ with a few α_2 laths and intercellular α_2 b) across twinned τ regions and c) association of the cracks with the intercellular α_2 regions. In all micrographs the growth direction and the tensile direction is parallel to the long edge of the micrograph.

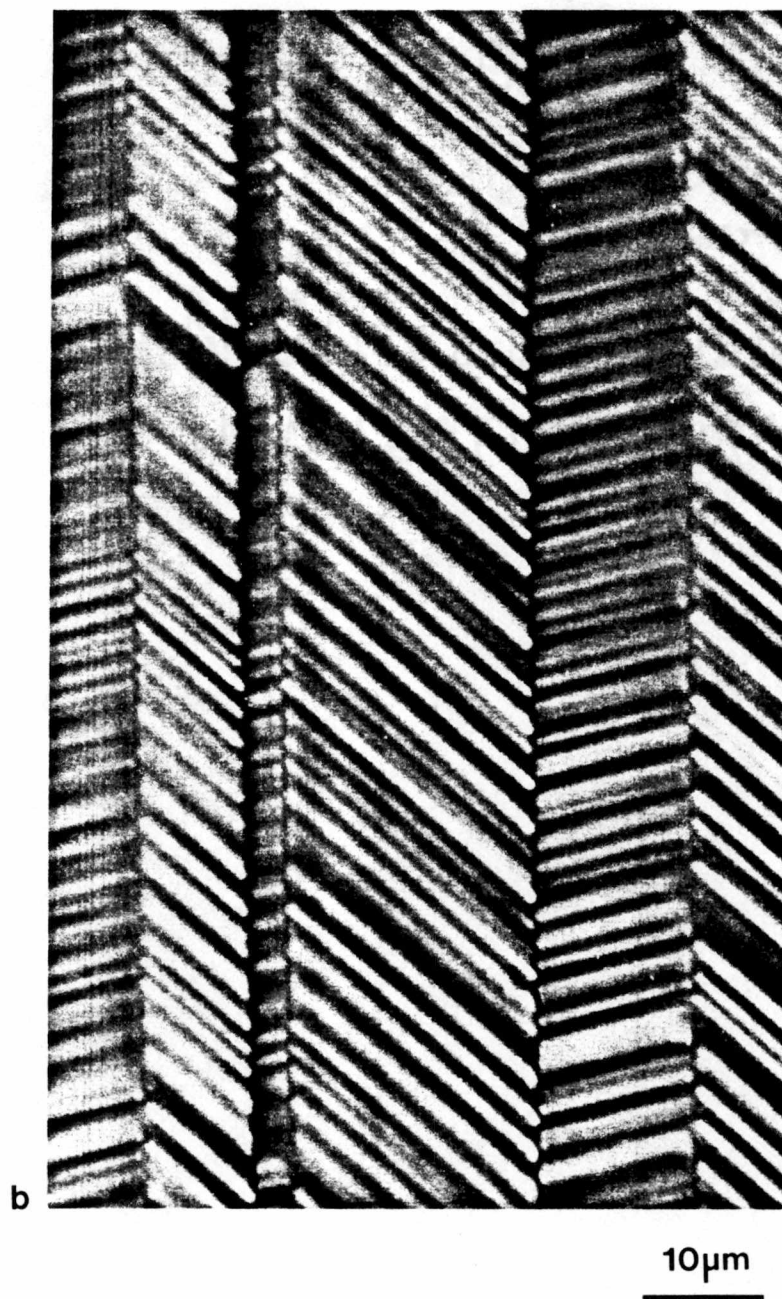


Figure 66. Continued.



Figure 66. Continued.

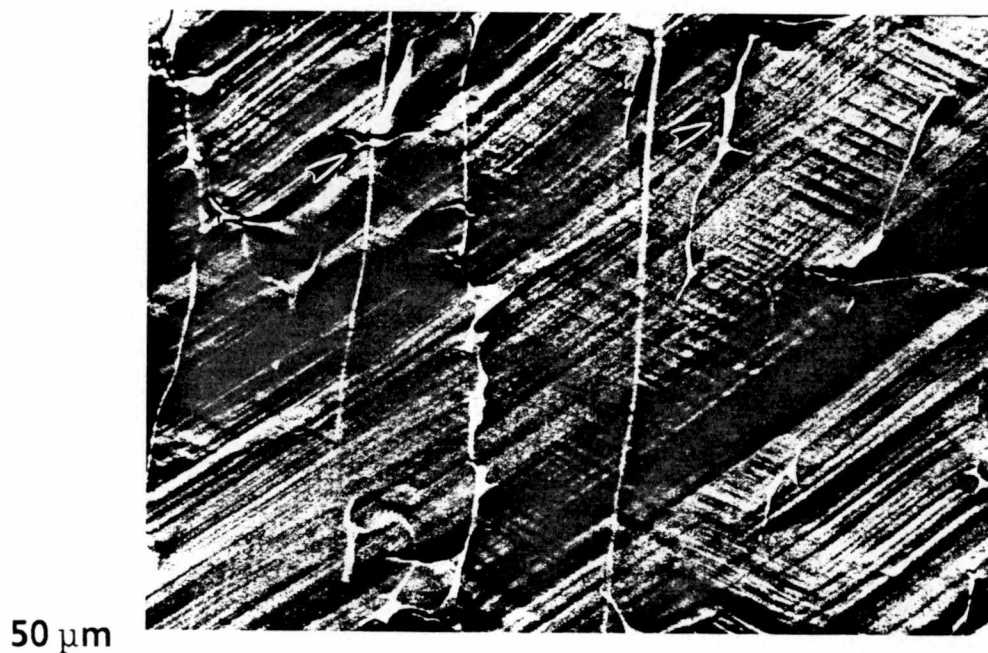


Figure 67. BSE image of the tensile strained as-cast material showing the α_2/τ interface cracking (arrows) and the association of cracks with the α_2 regions predominantly.

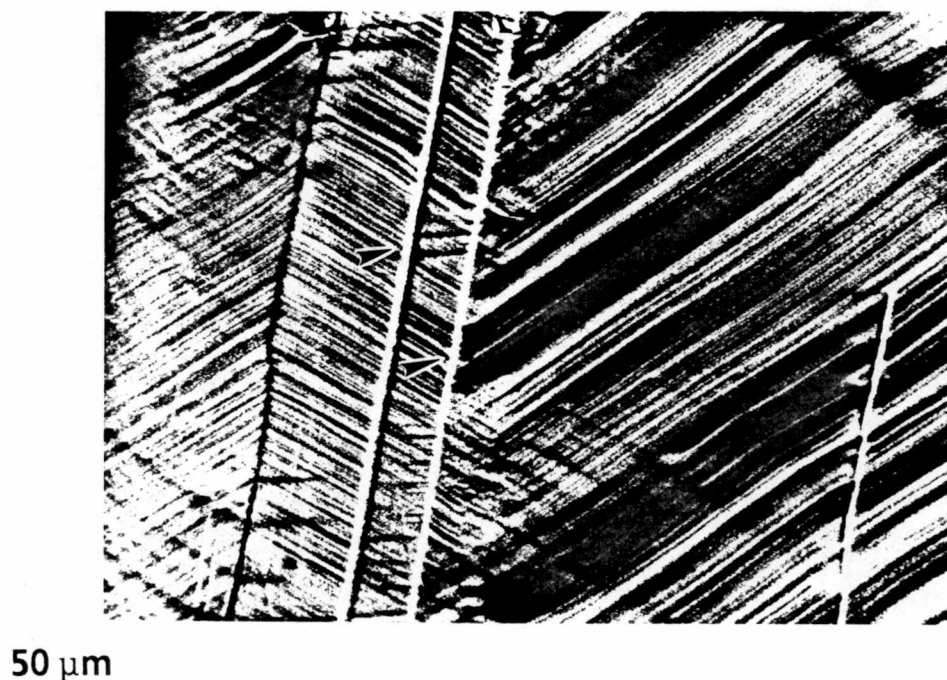


Figure 68. BSE image of the tensile strained as-cast specimen indicating the variation in orientations of the τ phase across the α_2 laths.

crack arrestor. The variation in ductilities in the as-cast specimen is most likely related to the local non-uniform distribution of α_2 in the gage length of the tensile specimen. Figure 68, a back scattered electron micrograph of the strained specimen, indicates that the regions of τ may or may not be twinned across an α_2 lath. Both combinations are observed in this micrograph. The arrangement of twinned τ morphology across a α_2 lath is better elucidated in the sequence of electron diffraction micrographs as shown in figure 69.

Multiple slip/twinning is observed at the twin and second phase interfaces suggesting the deformation behavior may be different close to the interface. Regions close to the fracture edge show abundant deformation bands, figure 70, suggesting that multiple slip systems and twinning modes are operative. Note that we have not differentiated between slip and twin deformation here. Preliminary TEM studies on compression specimens revealed a high density of dislocations and micro-twinning. Figure 71 shows the abundant microtwinning observed in twinned τ structures. The multiple τ orientations and microtwinning is better elucidated in the sequence of electron micrographs shown in figure 72. Since an abundance of dislocations as well as microtwinning was observed in TEM specimens, we have attributed the observation of deformation bands on the tensile specimens to a combined effect of slip and twinning.

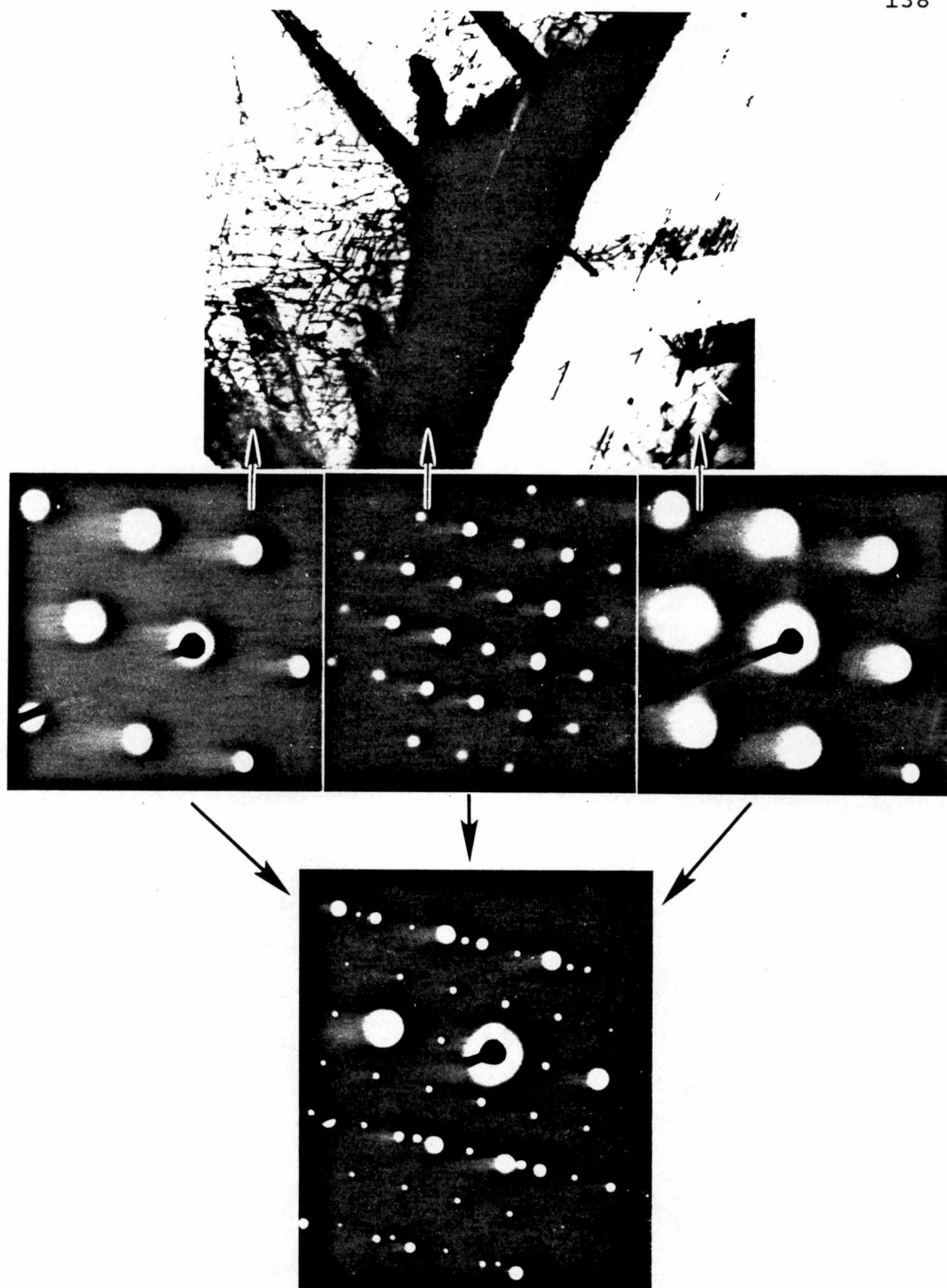
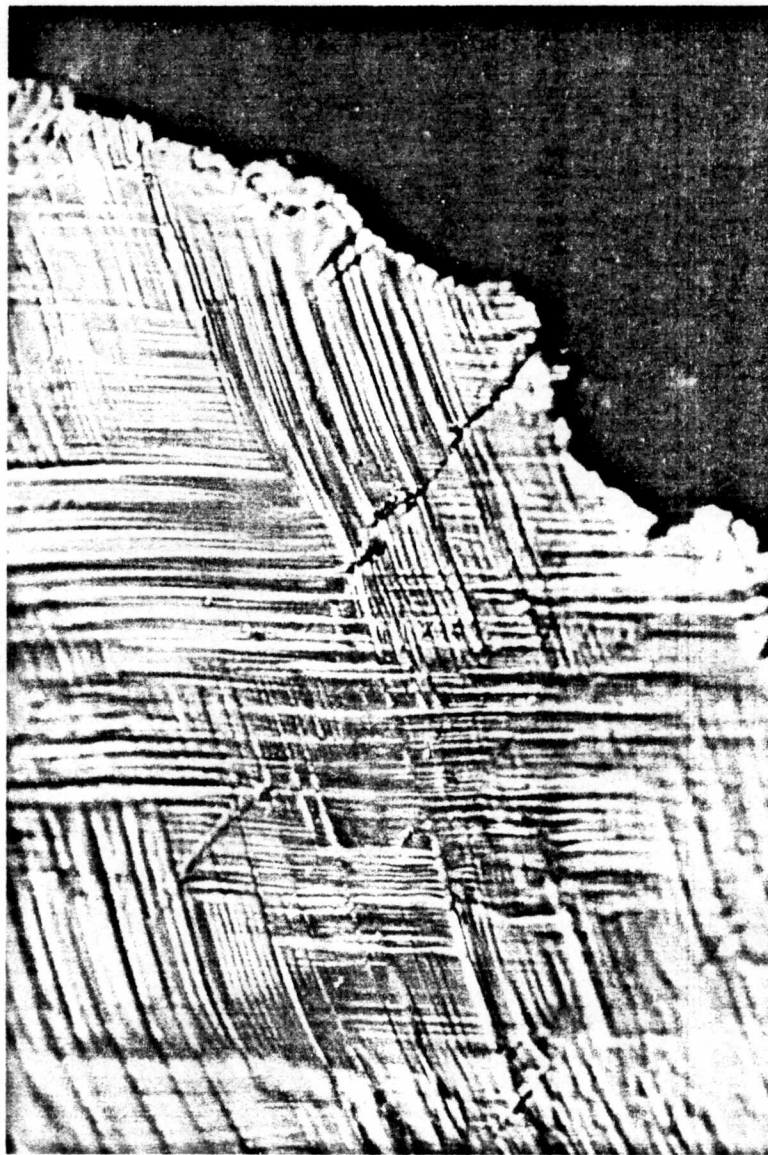


Figure 69. TEM micrographs of the sequence of τ orientations across the α_2 lath.



10 μ m

Figure 70. Multiple orientations of the deformation bands close to the fracture surface of the tensile strained as-cast specimen.

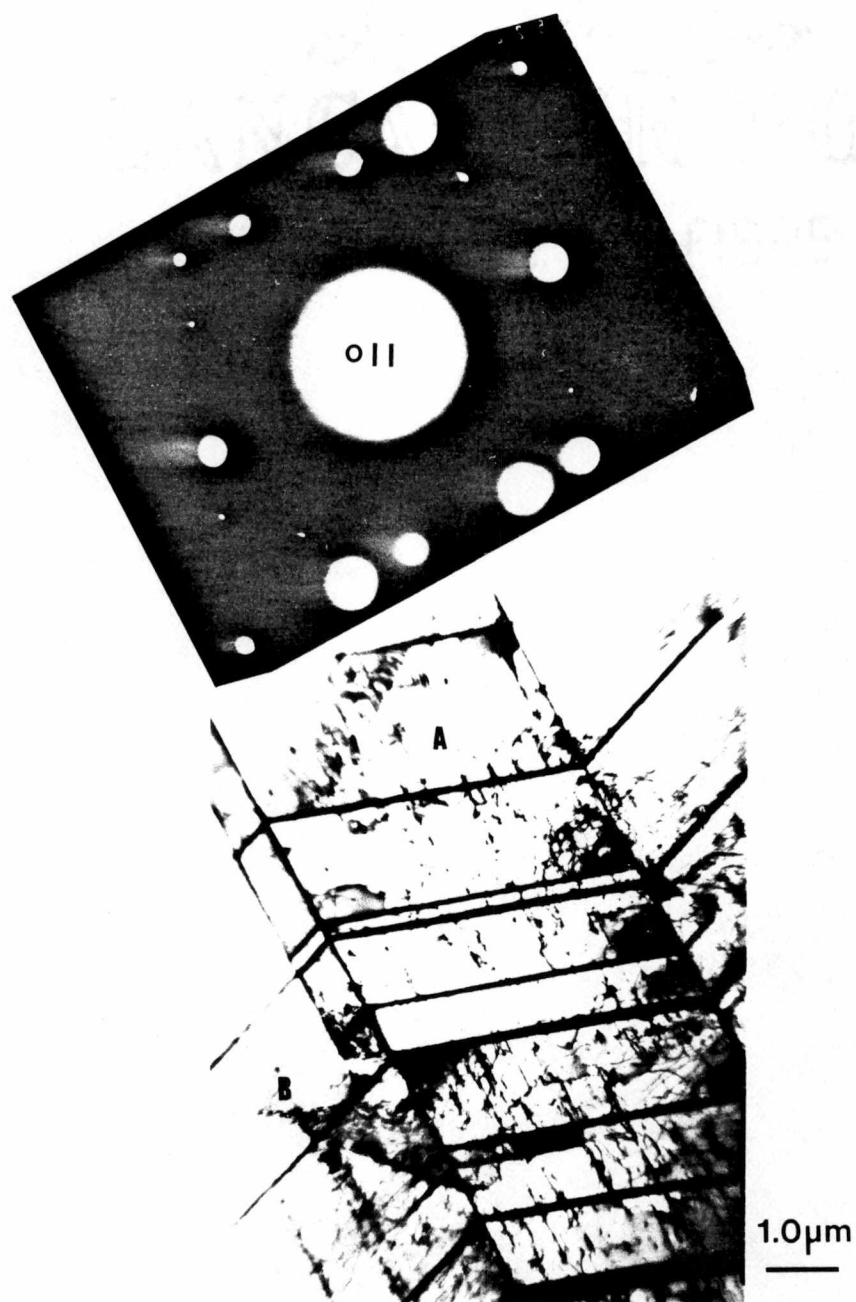


Figure 71. Microtwinning in as-cast compression specimens.

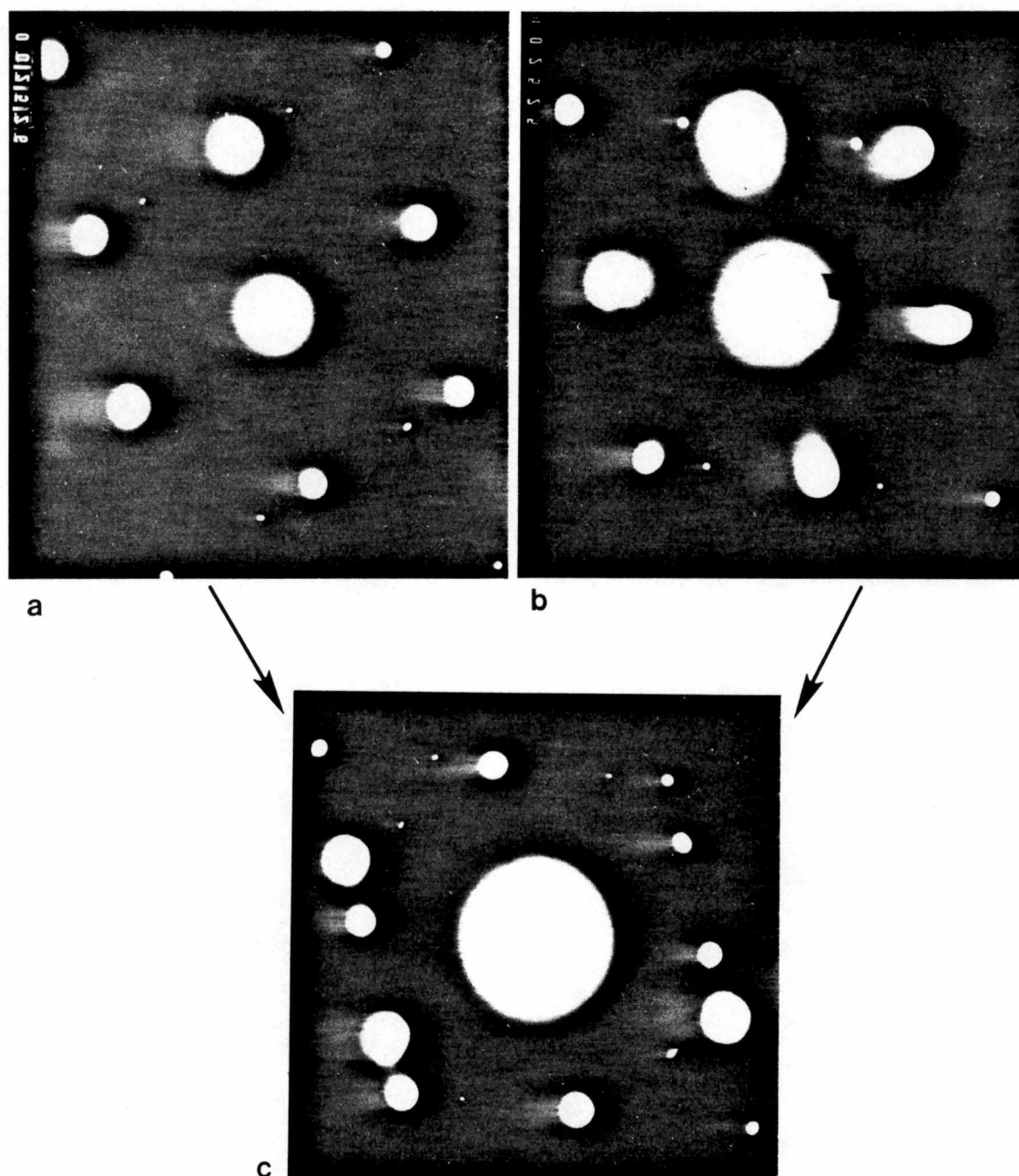


Figure 72. SADP micrographs elucidating the twinned γ morphology and the microtwinning as observed in as-cast compression specimens, figure 71. a) SADP from region marked A b) SADP from region marked B c) combined SADP from A and B as shown in figure 71.

Fracture Behavior of Directionally Solidified TiAl+Ga alloy1) As-cast Alloy

In the above segment the deformation behavior was reported for the different morphologies of τ , figure 67-69. The fracture surfaces associated with such deformed specimens are reported here. Figure 73 shows the fracture surface of specimen no. ZM/AC-2 which showed the highest ductility. Optically this specimen showed a large τ grain in the gage length along with the cellular structure as was shown in figure 66a. The cellular nature of the cross section is readily evident, figure 73, and it appears that the intercellular morphology is almost continuous. The specimen shows extensive cracking, almost continuous at the α_2/τ interface, i.e., at the intercellular boundaries. This behavior is more readily evident in figure 74. Additionally, the fracture surface also reveals interface cracking at the τ twin plane, figure 75. This implies that the (111) twin plane may be a weak plane for shear failure. The surface deformation is of the fine lath type, crystallographically oriented and changes orientation at the twin boundary, figure 75. There is a possibility that these features are associated with extensive micro-twinning as was observed in TEM, figure 71. This point will be dealt in detail in the next chapter. Figure 76 shows the fracture appearance of the specimen ZM/AC-4. The surface shows large parallel laths of twinned regions. These individual τ laths are about $200\mu\text{m}$ thick. It is interesting to note that the alternate τ

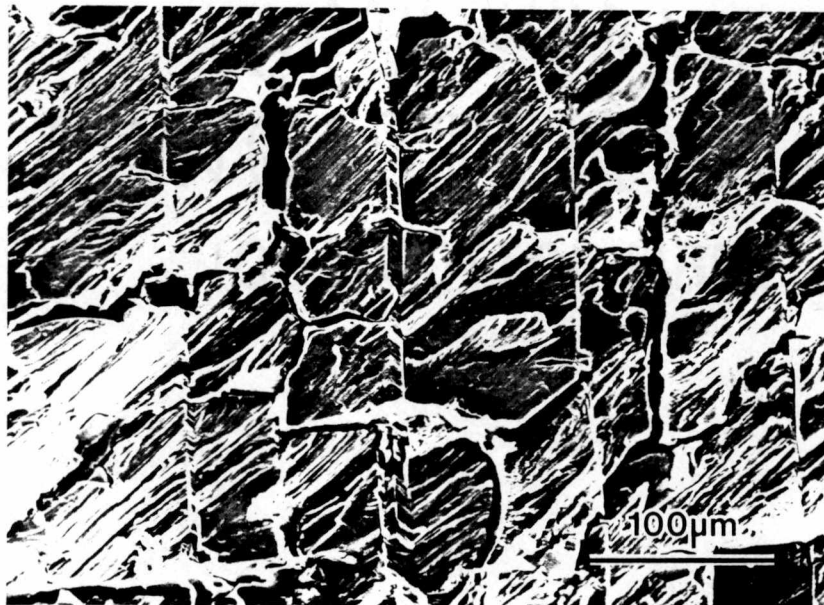


Figure 73. Fracture surface appearance of ZM/AC-2 tensile specimen



Figure 74. α_2/r interface cracking in the tensile specimen.

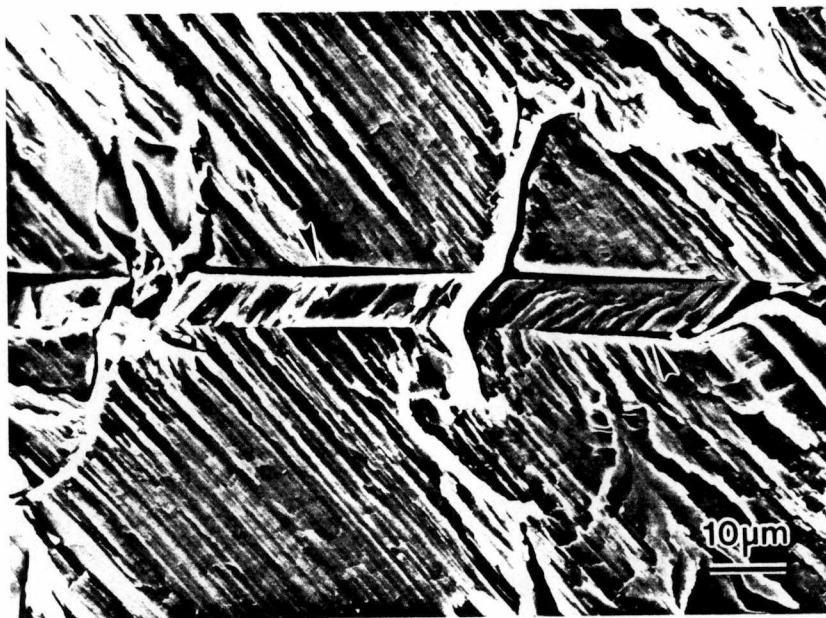


Figure 75. (111) τ twin plane separation observed in tensile specimens



Figure 76. Fracture surface appearance of ZM/AC-4 tensile specimen.

regions show a difference in fracture surface morphology. Alternate bands show typical cleavage behavior. This is reconciled with the fact that the twin plane was not aligned parallel to the tensile axis. Hence alternate bands were subject to different magnitudes of shear stresses for slip (i.e., Schmid factor increases in one τ orientation and decreases in the twinned τ orientation). An off-axis orientation of the twin plane is likely to bring a (111) plane perpendicular to the tensile axis. This would limit the process of slip (because of high Schmid factor) and promote (111) cleavage. Both specimens ZM/AC-3 and ZM/AC-4 had identical fractures, and the above reasoning could possibly contribute to low ductilities observed in these two specimens. Additionally, the fracture surface showed very little α_2 stringers, figure 77, suggesting in part that a second phase volume effect may be operative.

2) Homogenized alloy

Figure 78 shows typical brittle transgranular fractures observed in the tensile strained specimens. Figure 78b shows a pre-existing crack in the specimen gage length, possibly responsible for the low ductility failures observed for the case of the homogenized alloy.

Tensile Ductility data for Polycrystalline TiAl+Ga Alloys

Figure 79 shows the forged and recrystallized nature of the alloys. While some inhomogeneity was observed in grain

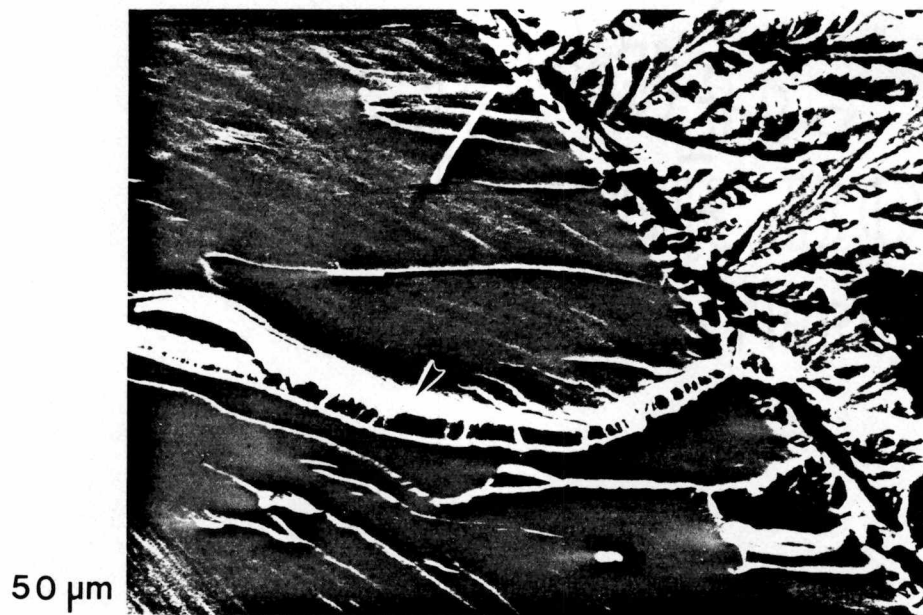


Figure 77. α_2 stringer (arrow) in ZM/AC-4 tensile specimen.

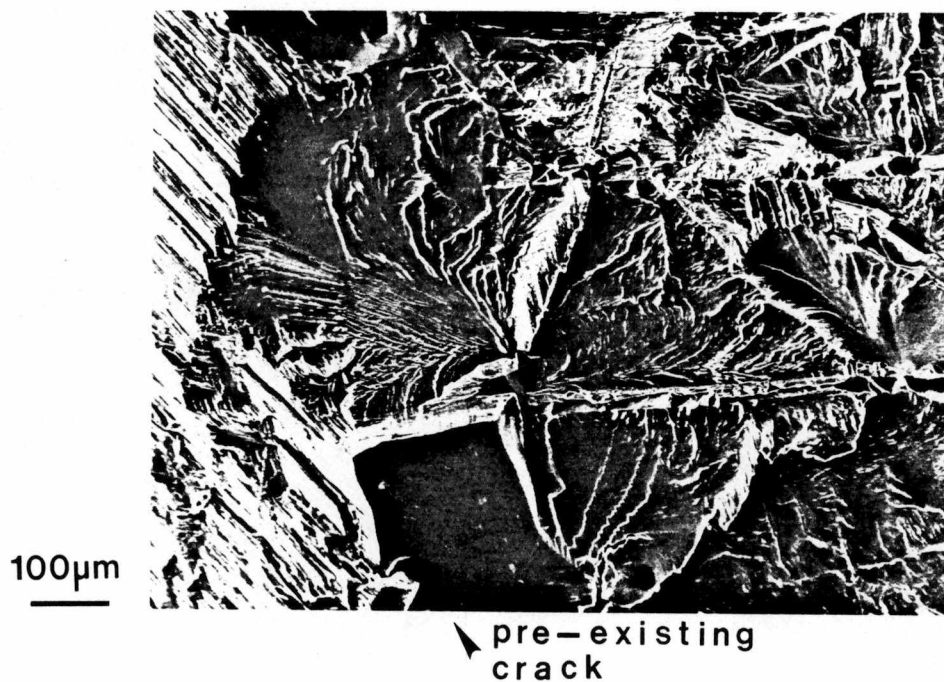


Figure 78. Fracture surface appearance of homogenized alloy, directionally solidified.

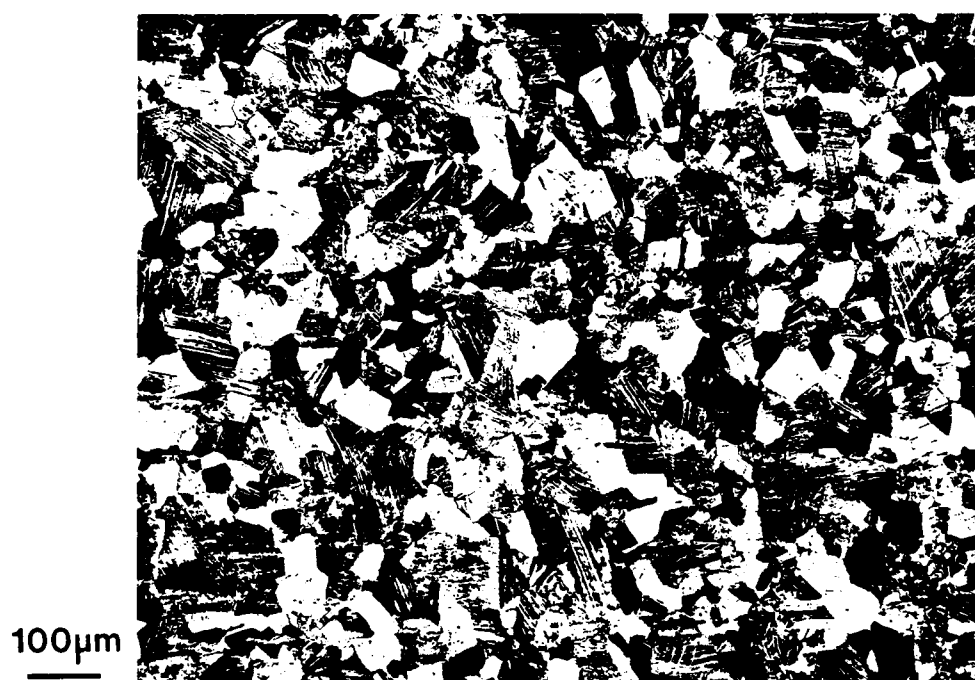


Figure 79. Optical micrograph of forged and recrystallized structure in polycrystalline TiAl+Ga alloys.

size over the cross section of the forged thickness, the average grain diameter size was estimated to be $\approx 50\text{--}75\mu\text{m}$. Table 9 lists the strengths and tensile ductility data for polycrystalline alloys. A limited plastic strain of 0.4% was observed at room temperature. However, since the samples broke outside the gage length, near the hole, this is not a viable test. Brittle fractures initiate from pre-existing cracks at the holes, as a result of spark machining. This is surprising since the directionally solidified (DS) specimens were also identically spark machined. Optical examination of DS specimens revealed that some cracks were present at the hole but did not propagate. It is possible that the poor processing control in terms of interstitial content in polycrystalline alloys makes them more susceptible to cracking. Additionally, the high yield stress observed for the case of polycrystalline will impose a higher stress for crack propagation prior to deformation. It follows that the critical flaw length for failure in these materials is very small, of the order of $10\text{--}20\mu\text{m}$. At higher temperatures, 673K and upwards, the samples all broke in the gage length and the ductilities are as reported. There is an appreciable increase in plastic strain to failure in the temperature range of 673-773K and another significant one in the temperature range of 873-973K. No yield stress anomaly is observed over the temperature range. Since the average grain diameter of the forged and recrystallized material is $\approx 50\text{--}75\mu\text{m}$, it presumably is below the threshold diameter size to

TABLE 9. TENSILE PROPERTIES OF POLYCRYSTALLINE TiAl+Ga ALLOY.

Temperature K	Yield Stress (KSI)	Tensile Stress (KSI)	Plastic Strain % (ϵ_p)
Room Temp			0.4
673	57.00	68.40	1.40
773	54.20	83.90	3.80
873	57.50	82.30	4.20
973	52.90	79.70	15.20

The alloys were canned and forged at 1373K to 25% reduction in thickness. Tensile specimens of ASTM E-8 specification, proportionately reduced, were spark machined and polished down to $9\mu\text{m}$ and electropolished. Cross head velocity 0.05cm/min. Strain rate= $6.67 \times 10^{-4} \text{sec}^{-1}$. tested in vacuum.

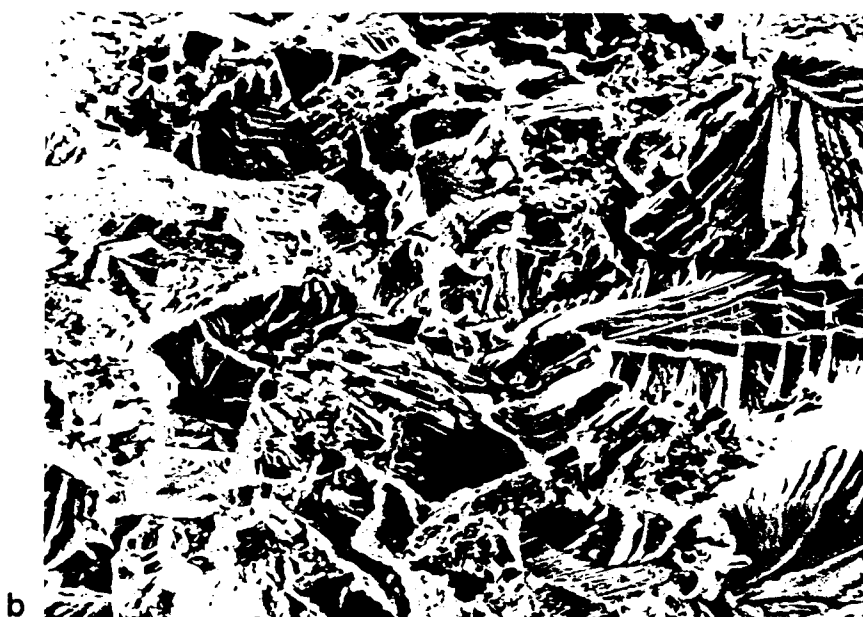
exhibit a yield stress anomaly as explained by Vasudevan et al. The alloy exhibits little loss in yield strength up to 973K, and good ductility at least at 673K and above. Hence, it offers suitable potential for high temperature application. However the room temperature ductility was low.

Fracture Behavior of Polycrystalline TiAl+Ga Alloys

Figure 80 shows the observed fractures for a range of temperatures. The fractures are of the brittle transgranular type and only minute changes in the fracture mode are observed over the entire temperature range (ambient-973K). This is surprising since the ductility changes appreciably over the temperature range. One interesting feature is the appearance of plate-like fractures and mottled dimple fractures as the test temperature increases. The plate like fractures are associated with the $\alpha_2 + \tau$ lath structures or twinned τ structures as shown in a high magnification view, figure 81. Note that some separation is occurring at the (111) twin plane for specimens deformed at 873K. This behavior was also observed in as-cast DS material at roughly equivalent plastic strains. It is possible that some plastic strain is being accommodated by shear deformation parallel to the twin planes (66). Figure 82 shows yet another view of such fractures. Note that regions of smooth fractures, possibly (111) twin planes and dimple fracture appear adjacent to each other. This is reconciled with the fact that dimpled appearance is derived from fractures across the

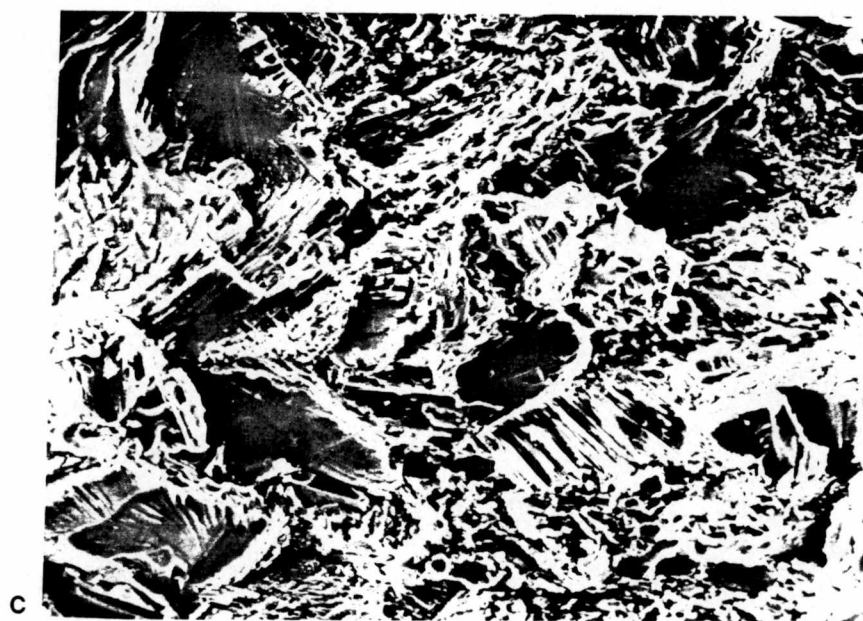


100μm



50μm

Figure 80. Fracture behavior of polycrystalline TiAl+Ga alloys tested at different temperatures. a) room-temperature b) 673K, c) 873K, d) 973K



50μm



50μm

Figure 80. Continued

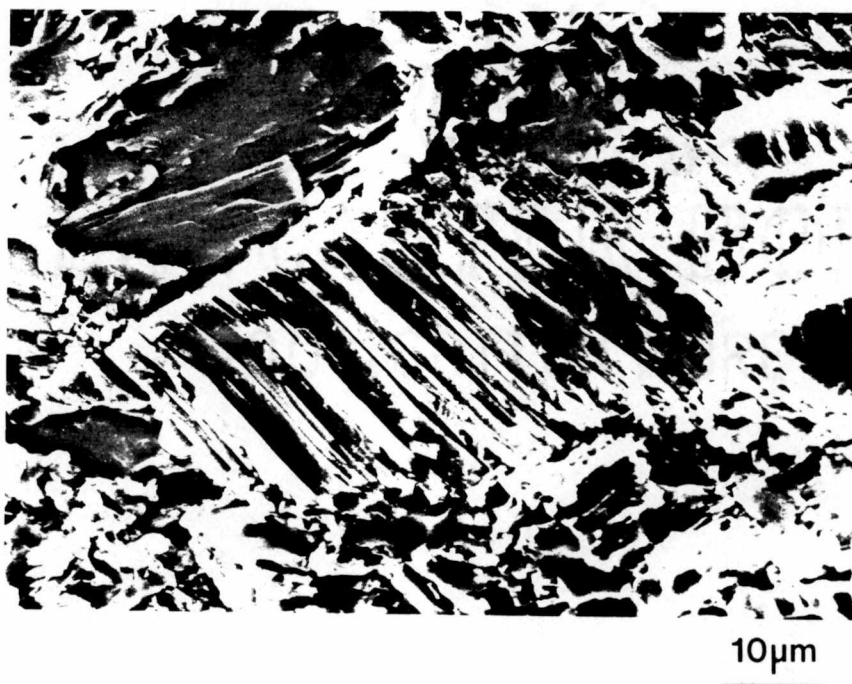


Figure 81. Plate like fractures indicating (111) twin plane separation in specimen strained at 873K

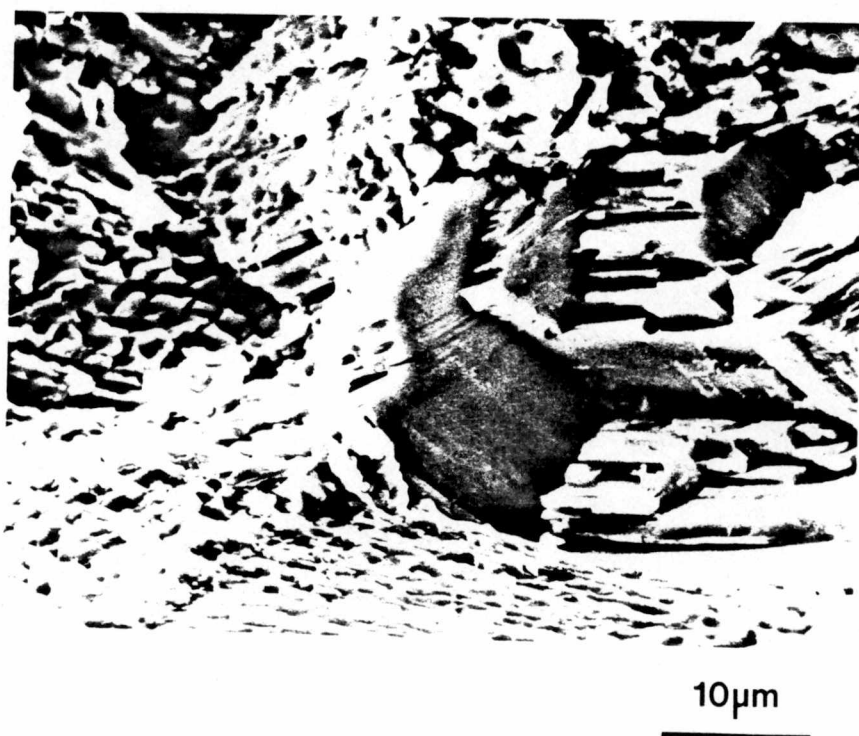
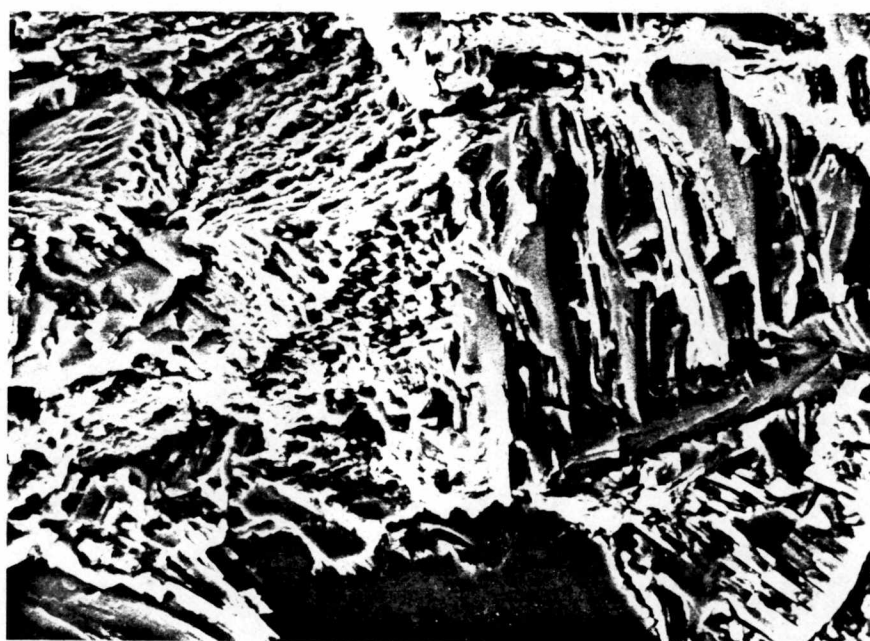


Figure 82. Smooth plate and dimpled fracture surface appearance for specimen strained at 873K

laths, and smooth appearance parallel to the laths. This is shown clearly in specimen deformed at 973K, figure 83. In general, it has been observed that the increase in ductility was somewhat concurrent with the occurrence of plate/lath type features in the microstructure. An interesting point is the observation of smooth facets even at 973K, suggesting weak cleavage planes or low interface strengths of lath type structures.

Mechanical Properties Data for TiAl+Mn Alloys

The tensile ductility data for polycrystalline alloys, figure 84, shows that TiAl+Mn alloys have limited ductility over the entire temperature range of testing, i.e., from room temperature to 973K. The brittle to ductile transition temperature (BDTT) is assessed (20) as $\approx 923\text{K}$ for binary single phase TiAl(τ) alloys. Since no such sharp increase in ductility was observed even at 973K, it is concluded that Mn addition effectively increases the BDTT. Note that at all temperatures of testing, the TiAl+Mn alloy shows superior strength but poorer ductilities when compared to TiAl+Ga alloy. While this alloy exhibits better potential in terms of high temperature strength, its room temperature ductility is poor. Since this study was essentially geared towards improving room temperature ductility, further work on TiAl+Mn alloy was stopped.



10 μ m

Figure 83. Smooth plates and dimple fracture morphology in specimen strained at 973K.

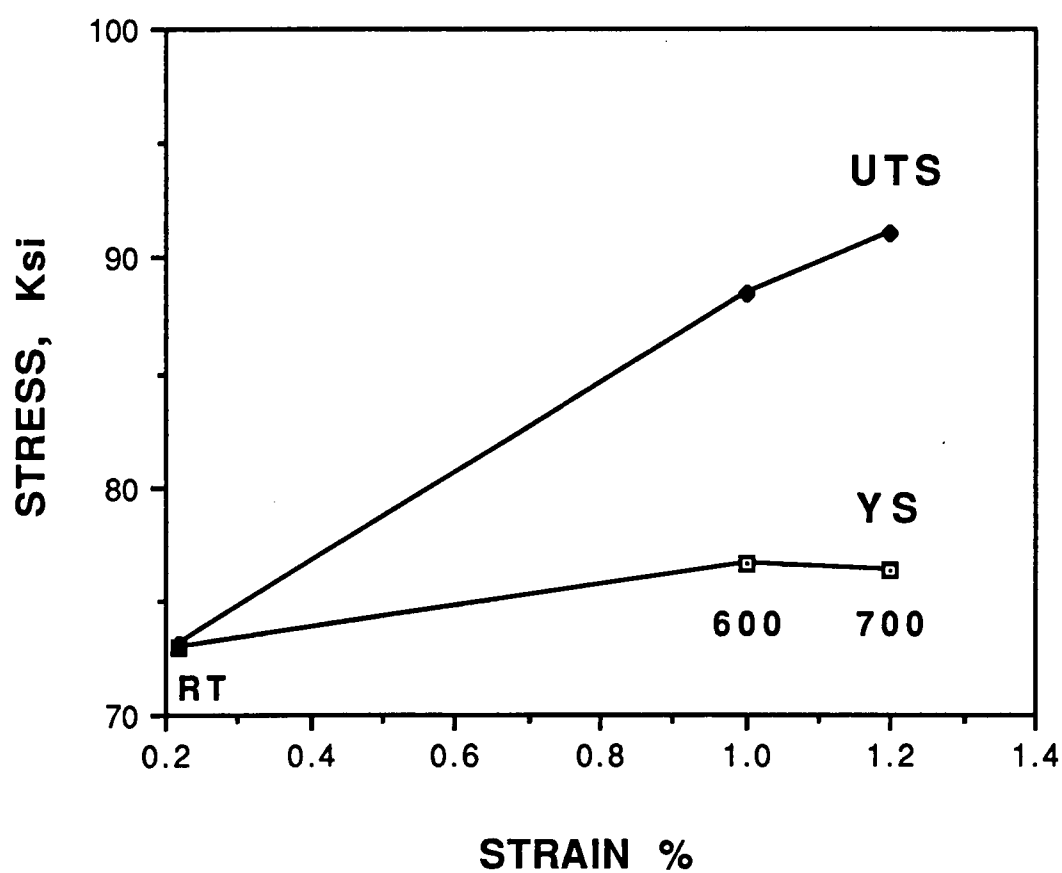


Figure 84. Strengths and ductility data for TiAl+5at%Mn polycrystalline alloy as a function of temperature.

Summary and Conclusions

Based on the tensile properties of TiAl+Ga and TiAl+Mn alloys, the following conclusions are drawn.

- 1) TiAl+Mn exhibits poor ductility up to 973K, and shows little promise for enhancing room temperature ductility.
- 2) TiAl+Ga exhibits appreciable ductility in the as-cast condition for large grain size material, and shows promise for further development to enhance its ductility.
- 3) The origin of cracking in as-cast TiAl+Ga alloy is largely associated with intercellular α_2 . This cracking is manifested both as interface cracking and through cracking.
- 3) Polycrystalline TiAl+Ga alloy exhibits good high temperature strength and ductility, indicating that gallium addition has no detrimental effect on the high temperature properties of TiAl.
- 4) (111) twin plane separation occurs at higher strains, over a range of temperatures..

It can only be surmised that the second phase has a possible effect on the ductility. If indeed this is true, then the nature of the α_2/τ interface is very important. Possibly, the coherent/incoherent nature of the interface will dictate the nature of cracking. It suffices to say that it is very important to study the nature of the interface to better understand the increase in ductilities for two-phase structures.

PART 4.

CLEAVAGE FRACTURE BEHAVIOR OF TiAl ALLOYSDefinition of the problem

This study was necessitated by the observation of cleavage type crystallographic fractures in TiAl, TiAl+Mn and TiAl+Ga alloys. In binary Ti-54at%Al and Ti-56at%Al alloys directionally solidified as single phase τ , a significant density of crystallographic cracks is observed at ambient temperatures, figure 85. These cracks appear either on cooling to room temperature, or during the metallographic specimen preparation. While the origin of this cracking may be disputed, it does suggest the propensity of cracking in this material and hence the poor ductility. Alloys that were directionally solidified as $\alpha_2 + \tau$ two-phase alloys showed no such cracks. However, these two-phase alloys, when homogenized to eliminate the α_2 phase largely, exhibited crystallographic cracking, figure 65. This crystallographic cracking, it is presumed, is responsible for the poor ductility in homogenized alloys. This cracking may also contribute to large anomalies of plastic strains reported for single phase materials in tension and compression. In compression the net effect is to close the crack and thus favor plastic deformation.

Cleavage type facets were also observed in Ti50/Al45/Ga5 polycrystalline alloy specimens, fractured in tension, at high temperatures 873-973K. While a large

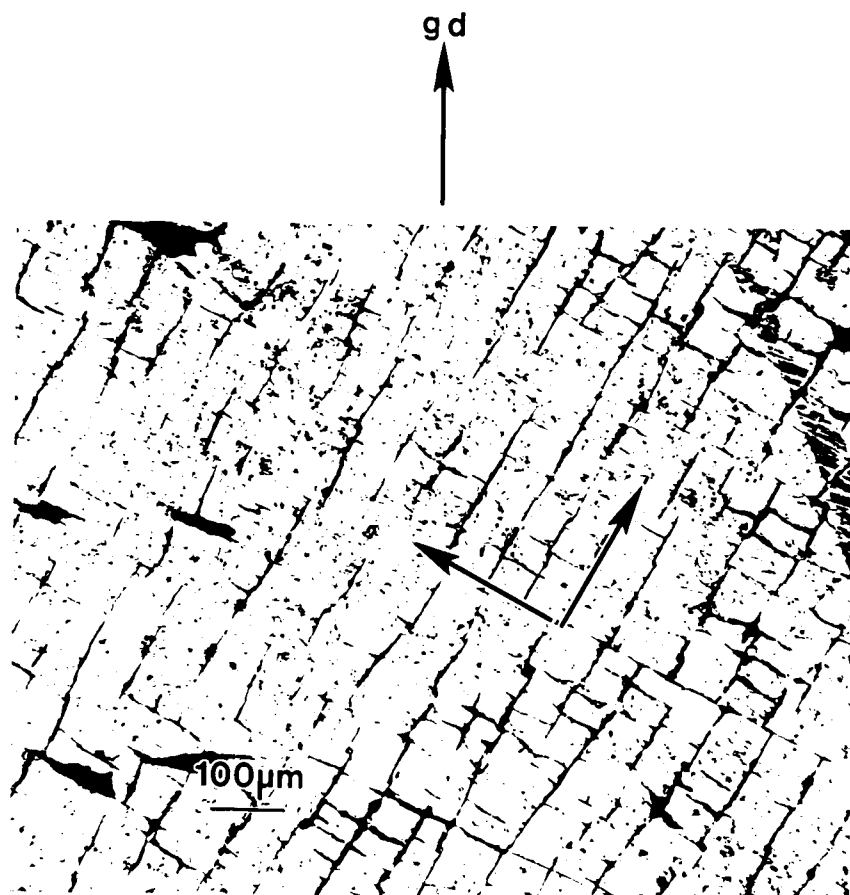


Figure 85. Occurrence of <100> crystallographic cracks in directionally solidified Ti-54at%Al alloys.

increase in plastic strain was observed at these high temperatures, the fractures were still of the brittle transgranular type, figures 81-83. For TiAl+Mn alloys a peculiar tendency of fracture was associated with the annealing twins in the microstructure, figure 86.

TiAl has the low symmetry, ordered $L1_0$ structure with limited slip systems, and is consequently brittle at ambient temperatures. Kawabata et al. (49) identified multiple cleavage planes in TiAl as $\{100\}$ $\{110\}$ and $\{111\}$ with the aid of stereo microscopy. They reported such observations on bend tested, single crystals with a $[010]$ and $[001]$ orientations. This is surprising, since for oriented specimens the maximum normal stress was applied on the $\{010\}$ and $\{001\}$ planes. Incidence of fracture on $\{110\}$ and $\{111\}$ planes suggests the possibility that fractures on these planes may initiate at lower cleavage stresses. According to the maximum normal tensile stress theory, for cleavage, the resolved normal stresses in the $\{110\}$ and $\{111\}$ planes are $\sigma_0 \sin 45.0^\circ$ and $\sigma_0 \sin 54.7^\circ$ respectively. It is possible that $\{110\}$ and $\{111\}$ planes have lower cleavage strength. Fracture of specimens in the $\langle 110 \rangle$ and $\langle 111 \rangle$ orientations may support this hypothesis. This study reports fractography investigations carried out using electron channeling patterns.



100μm



10μm

Figure 86. Optical microstructure a) and Cleavage type facets b) observed in TiAl+Mn alloys.

Experimental Technique for Electron Channeling

Selected area electron channeling patterns were used to identify the cleavage planes in single crystals of Ti-54at%Al, Ti-56at%Al, and polycrystalline TiAl+Mn alloy. The TiAl+Ga polycrystalline material was not used since it fractures with some plastic deformation and renders observing electron channeling experimentally more difficult. The single crystals used in this study were prepared using the directional solidification technique and the crystal growth direction was reported to be 90° off [011] (63). Samples 3-5mm thick were cut in the transverse direction from the processed bar using a slow speed diamond saw. Also 3 mm square rods were discharge machined along the growth direction for bend tests. No attempt was made to correct for the slight off-axis growth of the crystal. Samples were fractured by three point bending. For TiAl+Mn alloy specimens, thin slices about 15mm X 5mm X 1mm were cut using the slow speed diamond saw, polished and fractured in three point bending.

The fracture specimens were observed in a Cambridge Stereoscan 360 SEM equipped to take selected area channeling patterns. While a channeling pattern was easily obtainable in the as-fractured condition, it was observed that a 10-15 second electropolishing treatment (the electropolishing solution is the same as that used for TEM specimen preparation) greatly enhanced pattern detail. Results are reported for fractured specimens after these preparation

techniques.

SACP is a relatively simple procedure to identify cleavage planes in brittle materials since they fracture without any significant plastic strain. The most difficult part of SACP is to align the fracture plane normal to the electron beam. In the current study this was facilitated by using several different methods. First the individual cleaved facets selected were large enough to simply align it using light reflection techniques. This was done by clamp holding the specimen to a ball joint mount and tilting till the entire facet was in sharp focus under a light microscope. A SEM specimen stub was thickly coated with fast drying cement and raised on a vertical stage till it just made contact with the specimen. This set up was allowed to set for about 30 minutes. This alignment was supplemented by slight tilting in the SEM till the distance between two points on the topographic detail was the maximum. A simple alignment by light reflection technique was sufficient, and in all cases less than 2^0 tilt was required to bring a low index pole into the center of the pattern.

Results and Discussion

SACP of Single Crystals of TiAl

Figure 87 shows the SEM micrographs of the faceted fractures observed for the transverse specimen examined. It was apparent that the specimen fractured in a transgranular

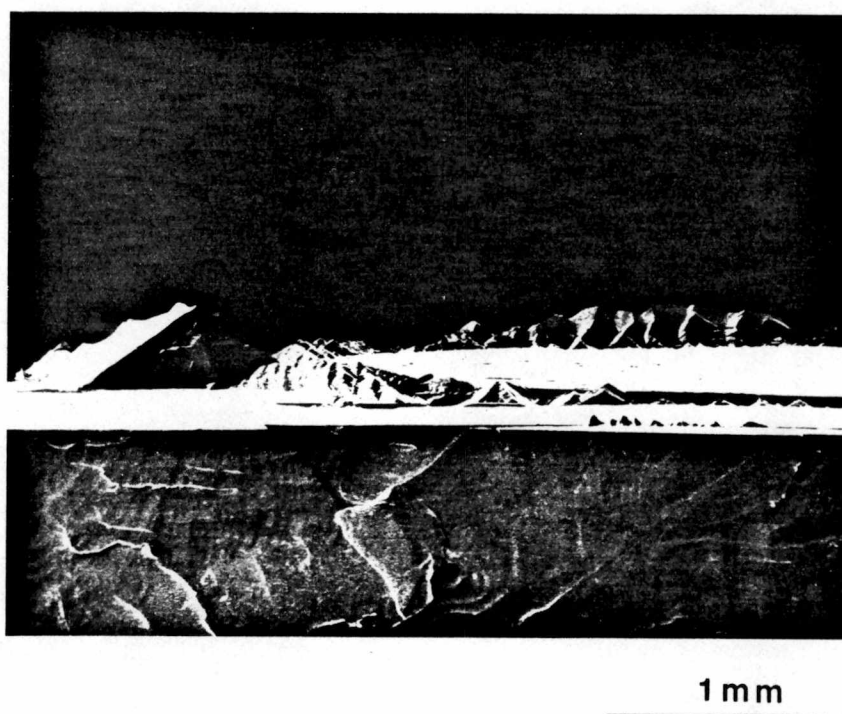


Figure 87. SEM micrograph of fracture facets in Ti-56at%Al single crystal transverse specimen.

cleavage mode and the large flat facets were essentially one cleavage plane. Figure 88 show a close-up view of the facet and the SACP obtained. This facet was so flat that the entire area (1.5mm X 2mm) could be maintained in focus under a light microscope. A 0.8° tilt was required to bring the $\{111\}$ pole into the center of the channeling pattern. Once having obtained this pattern, the specimen was moved using the X-Y stage in the SEM to bring several other points under the beam for channeling. Similar $\{111\}$ patterns were observed without any further adjustment indicating that the entire facet was of the $\{111\}$ type. Note in figure 88 the top left straight line (i.e., the edge of the specimen close to (011) trace) matches very closely with one pair of (220) channeling band on the SACP with a 7° variation. Since no rotation correction is required between the image and the channeling pattern, this band then identifies the actual growth direction as 7° off $[011]$, which is in good agreement with the 9° offset reported earlier (63). Figure 89 shows a high magnification view of the other facet and the SACP obtained. This facet was at an angle of about 55° with the $\{111\}$ cleaved facet, identified in figure 88. The intersection between the two facets is $[110]$. This facet was not as smooth as the $\{111\}$ facet throughout and alignment of a small region was carried out using topographic detail. SACP pattern identifies the fracture cleavage plane as $\{100\}$ type. This facet was also nearly parallel to the growth direction. For the case of transverse specimen orientation,

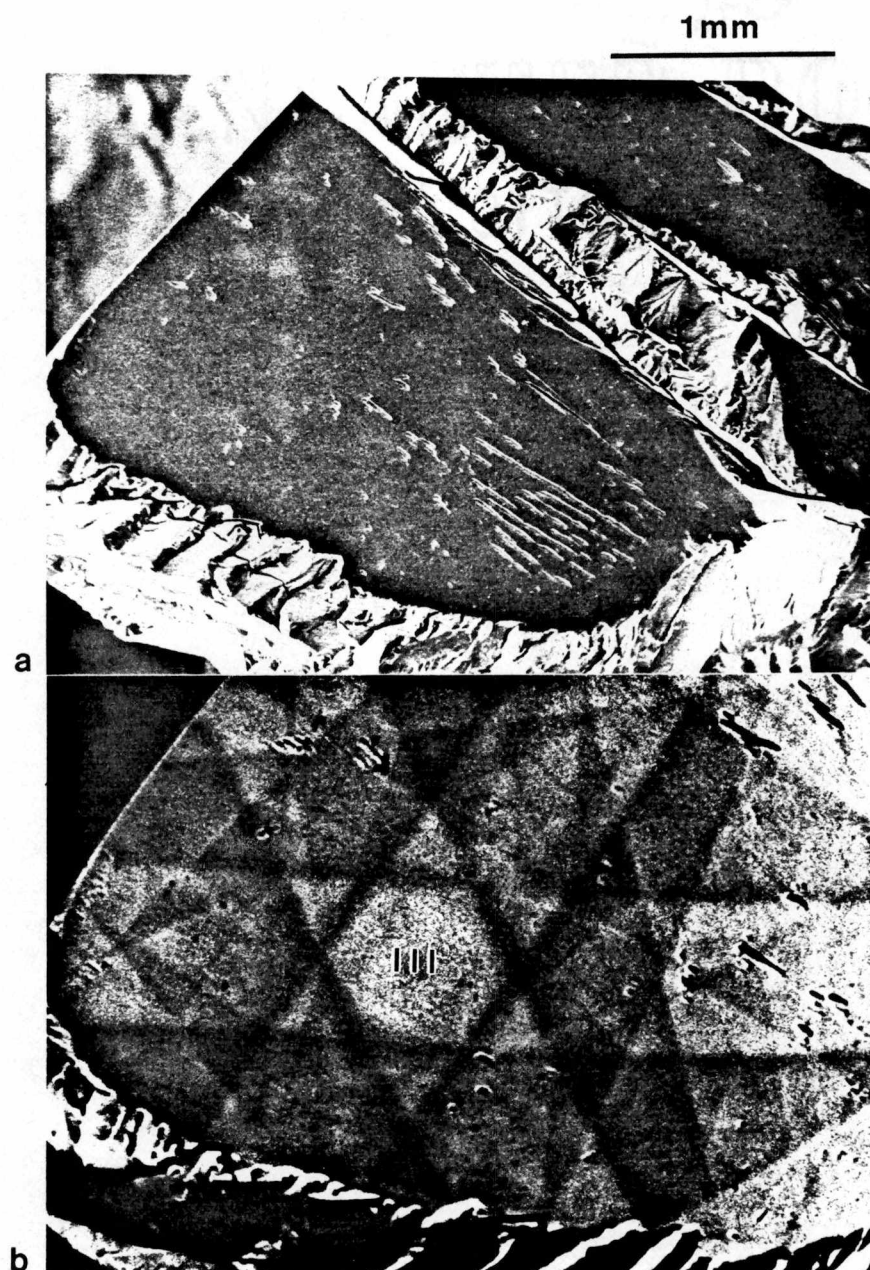


Figure 88. {111} type SACP pattern obtained for the facet.
a) facet b) SACP

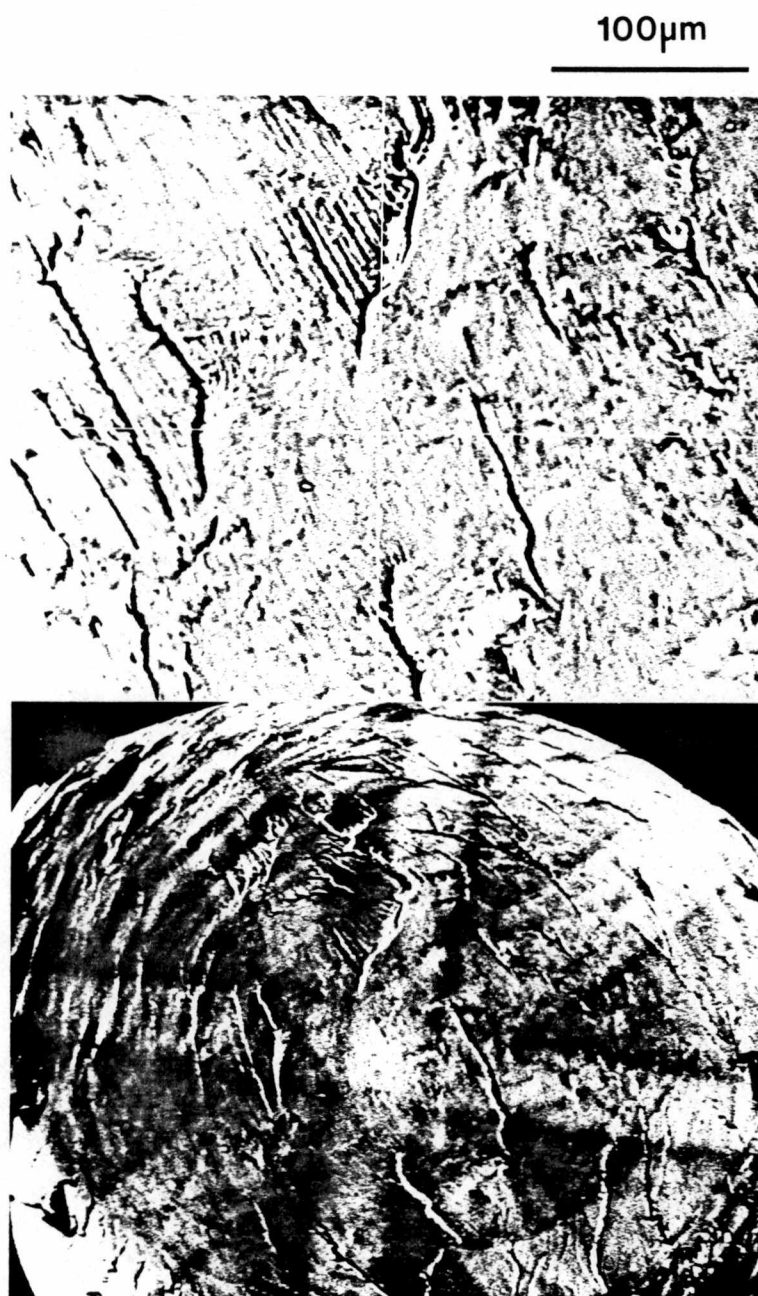


Figure 89. $\{100\}$ type SACP pattern obtained for the facet.
a) facet b) SACP

both {100} and {111} planes lie in the thickness direction (i.e., the growth direction of the crystal) of the specimen. Depending on the actual orientation of the specimen disc while fracturing, either {111} and/or {100} planes will experience maximum bending stresses for cleavage as shown in Table 10. This may explain the variation in the surface appearance of the {111} and {100} facets. Attempts were made to use several transverse specimens to fully characterize the cleavage behavior. Figure 90 shows the mixed nature of {100} and {111} cleavage facets occurring in a coupled manner. The cleavage facets alternate between {100} and {111} suggesting that the propensity for cleavage on either of these family of planes may be identical. Note that both {111} and {100} facets are of the smooth cleavage type unlike figure 88,89. This coupled fracture behavior was frequently observed for samples cut in the transverse orientation.

It was interesting to note that for specimens in the transverse orientation no large facets were present that could be identified as {110} even though four such possible planes exist at 60^0 angle to the growth direction. This {110} cleavage fracture was observed for specimens cut along the growth direction (i.e., longitudinal specimens), where the {110} planes were expected to experience the maximum bending stress. However, in this case both {100} and {110} fracture facets were observed, figure 91. The dominant appearance of {100} cleavage facets in this case suggests

TABLE 10. COMPARISON BETWEEN THE APPLIED NORMAL TENSILE STRESS AND THE SHEAR STRESS FOR SLIP FOR SPECIMENS IN DIFFERENT ORIENTATIONS.

Specimen Orientation	Applied normal stress	Applied shear stress	Schmid Factor
[001]	σ_0 for (001) $\sigma_0 \sin 45.0$ for (110) $\sigma_0 \sin 54.7$ for (111)	$\sigma_0 \cos 54.7 \cos 35.26$	0.472
[110]	σ_0 for (110) $\sigma_0 \sin 45.0$ for (100) $\sigma_0 \sin 35.3$ for (111)	$\sigma_0 \cos 35.26 \cos 60$	0.408
[111]	σ_0 for (111) $\sigma_0 \sin 54.7$ for (100) $\sigma_0 \sin 35.3$ for (111)	$\sigma_0 \cos 35.26 \cos 70.53$	0.272

The competing nature between fracture and slip may indicate whether slip will occur prior to fracture or not.

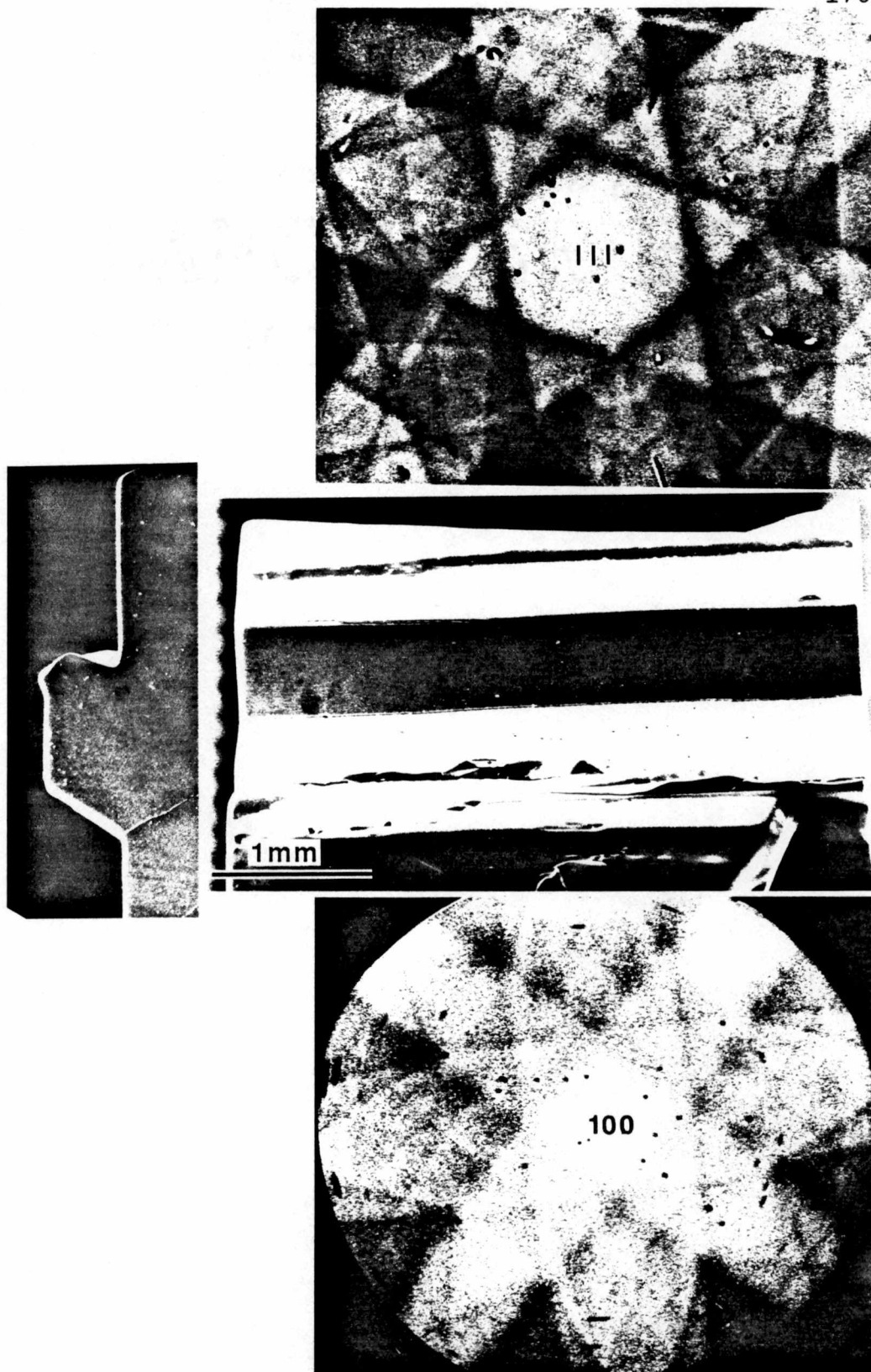


Figure 90. Coupled $\{111\}$ and $\{100\}$ cleavage facets observed in Ti-56at%Al single crystal transverse specimen.

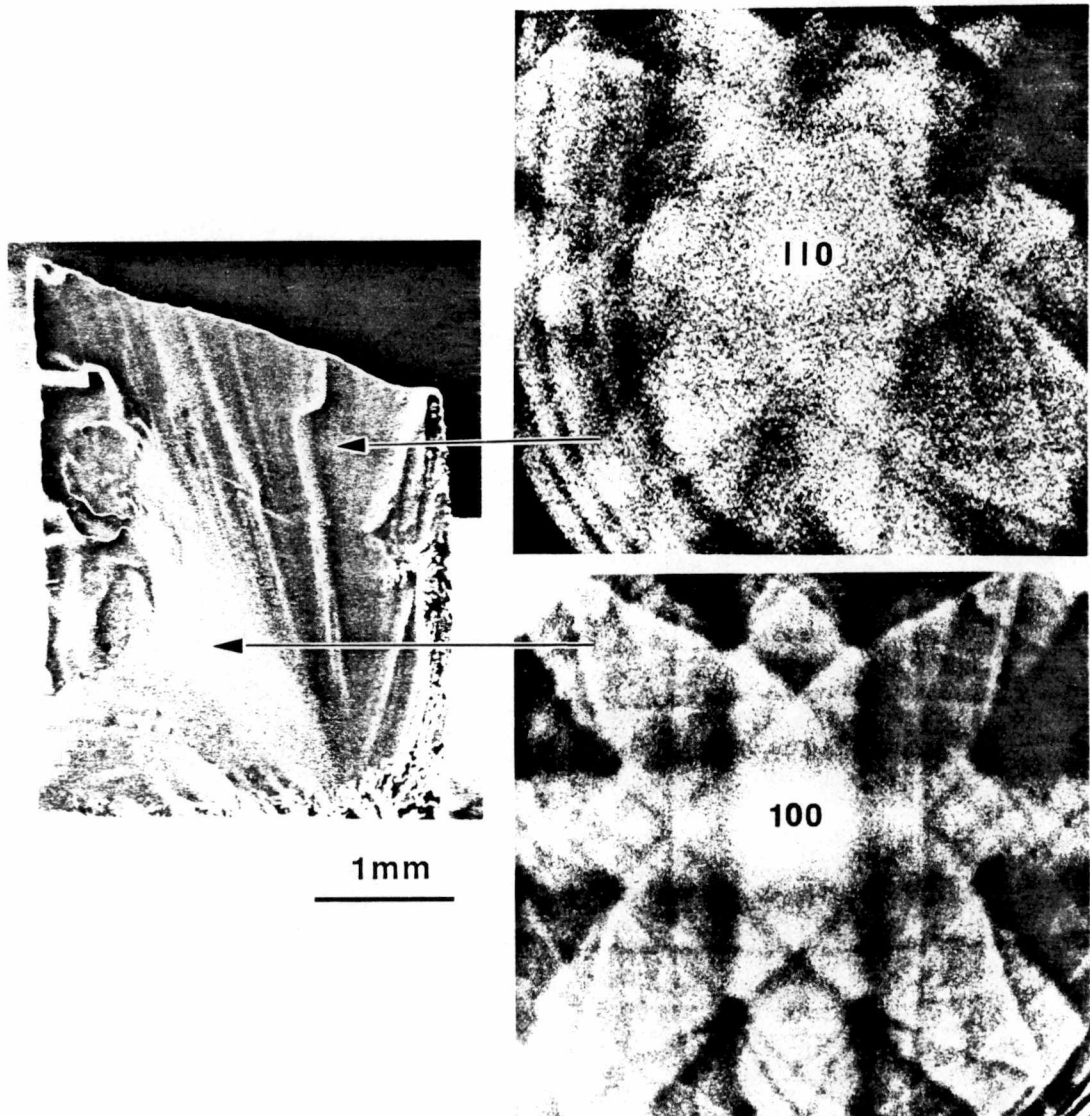


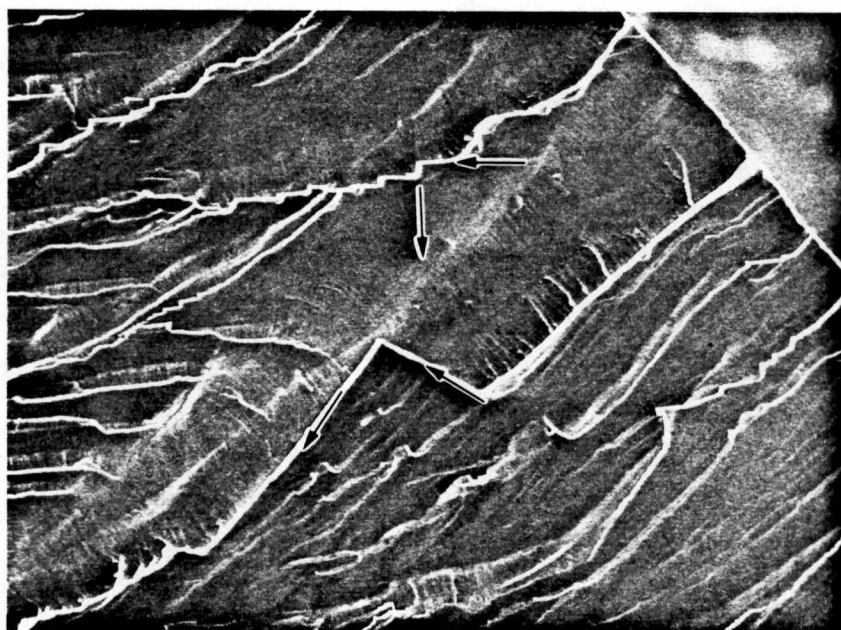
Figure 91. {110} and {100} cleavage facets observed in Ti-54at%Al single crystal longitudinal specimen.

the relative ease of cleavage on such planes in comparison to $\{110\}$, even at lower applied normal stresses, see table 10. The $\{111\}$ facets were not observed for the longitudinal specimens. Note that for a $\{110\}$ orientation the normal stress on the $\{111\}$ is lower than that for $\{100\}$.

A common feature of the $\{100\}$ type fractures was the occurrence of steps and traces of other crystallographic directions, figure 92. These were identified as $\{100\}$ and $\{110\}$ type by matching with the $\langle 200 \rangle$ and $\langle 220 \rangle$ channeling bands from the SACP.

Summary and Conclusions

There are at least three kinds of cleavage planes in TiAl alloy: $\{100\}$, $\{110\}$ and $\{111\}$. The $\{110\}$ is not one of random occurrence, i.e the $\{100\}$ and $\{111\}$ dominate in a decreasing order. $\{110\}$ not being randomly observed can be possibly attributed to the strong directionality of bonding reported (45,46) in the $[110]$ directions. Not knowing the cleavage strengths of these planes, the current results serve to rank the cleavage planes in an order of increasing propensity to occur. This rank is of the $\{110\}$, $\{111\}$, $\{100\}$ order. Both $\{111\}$ and $\{110\}$ fractures occur in conjunction with $\{100\}$. Hence the highest order for $\{100\}$. Note that the experimentally reported CRSS, in compression, for $[110]$ is lower than both $[001]$ and $[010]$ (19), figure 8. This might reflect the tendency for the $[110]$ to deform rather than cleave. Observation of $\{110\}$ steps and traces in $\{100\}$



100 μ m

Figure 92. Crystallographic orientation of $\langle 100 \rangle$ and $\langle 110 \rangle$ steps and traces observed on the $\{100\}$ fracture facet.

facet, figure 92, suggests that while $\{110\}$ may be a potential crack initiation plane, it may not be a suitable crack propagating plane. This agrees well with the fracture evidence for the longitudinal specimen, where $\{110\}$ occurs along with $\{100\}$ plane fracture. $[001]$ has the highest CRSS and hence reflects the tendency to cleave. A $\{111\}$ plane when aligned normal to the tensile direction offers the lowest schmid factor, see table 10, than any other low index plane. Intuitively, this represents a more difficult process to accommodate deformation (because of low Schmid factor) and hence cleave.

Finally it has been shown that multiple planes of cleavage exist in binary TiAl. For polycrystalline alloys multiple cleavage planes, combined with multiplicity of planes factor, represents a formidable problem towards enhancing the ductility. The low incidence of $\{110\}$ cleavage suggests that directionally solidified material with a $[110]$ growth direction will be amenable to plastic deformation, as has been reported for the TiAl+Ga alloy.

Twinning Related Cleavage Facets in TiAl+Mn Alloy

The extensively twinned homogenized structure is shown in figure 86. SEM examination of the fracture surfaces, figure 86b, indicated a predominantly brittle fracture, transgranular type, with some ductility apparent at the tension side of the specimen. A number of step striations all oriented parallel in a grain, were observed. These

striations were linked to the twinned matrix on the etched surface, figure 93. Optical examination of the etched surface revealed sawtooth edges at the fracture edge, figure 93. The intersection angle between the fracture planes and the matrix twin interface were identical, within limits of experimental error, for both the matrix and the twin region (i.e., the fracture planes are symmetrical across the matrix-twin interface). This is shown schematically in figure 93.

Precise tilting experiments were carried out for stereo imaging to determine the included angle between the facets. The sample was mounted at 0° tilt and tilted up to 30° in five steps of 6° each. At each step the image was refocused using only the z-stage control for preserving magnification. Figure 94 shows selected micrographs from the series. Table 11 lists the measurements of projected widths of the facets and the calculations for determining the included angle. The included angle was calculated as 143° , which is in good agreement with the theoretical value of 141° between $\{111\}$ planes across the matrix-twin interface. Figure 95 shows an oblique view of layered $\{111\}$ facets fracturing across the twin plane in the specimen. The oblique view indicates the large included angle between the facets. In all cases where the included angle was measured as approximately 140° , no matrix-twin interface fracture was ever observed. However, in one isolated case the included angle could not be reconciled as 140° , figure 96. Interface cracking was

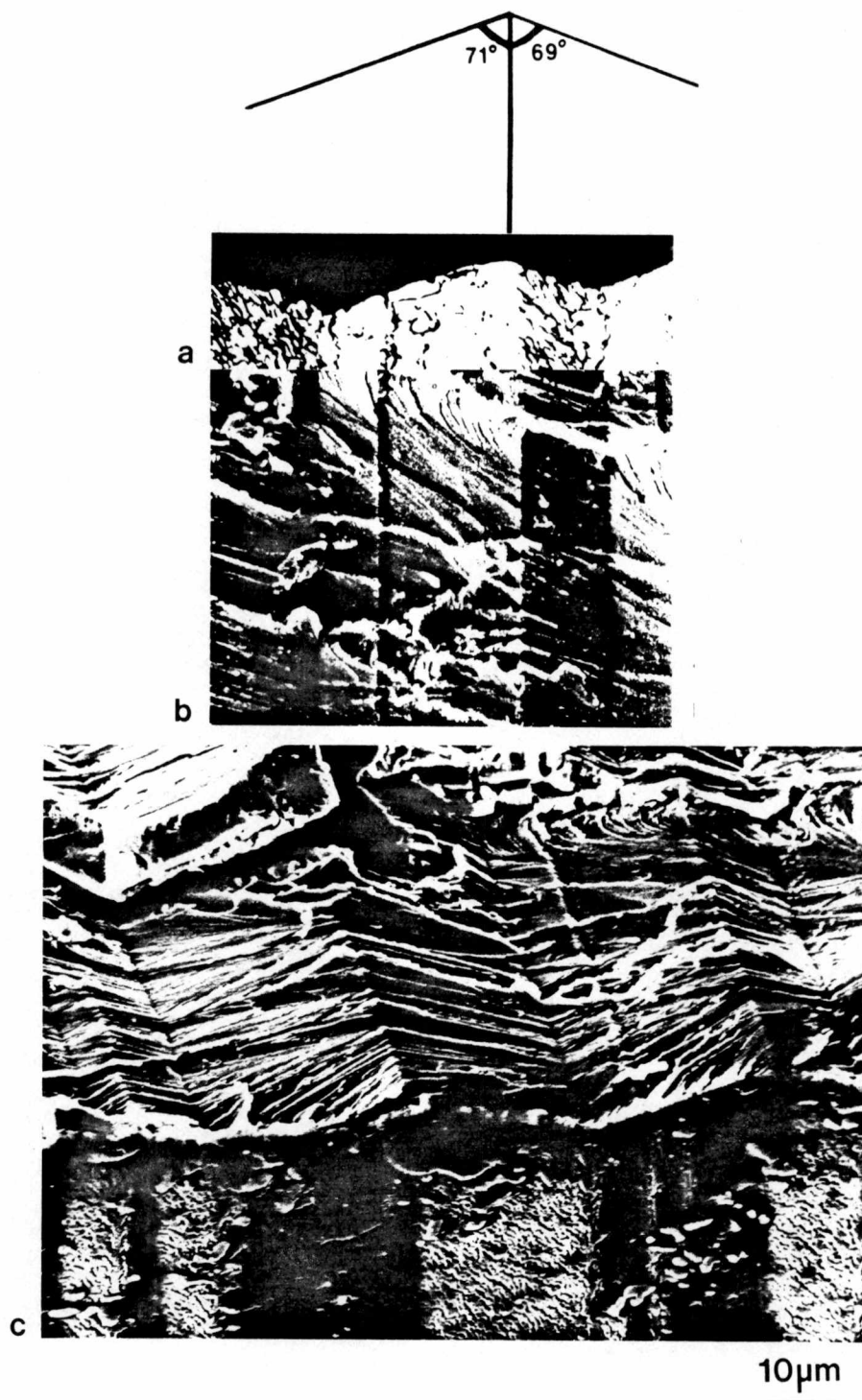


Figure 93. Optical and SEM micrographs of adjacent fracture facets. a) optical view of the etched surface showing the large included angle. The schematic gives the actual angles measured from the optical micrograph b) SEM view of the fracture surface viewed from the top. c) Oblique SEM view showing both the etched and the fracture surface.

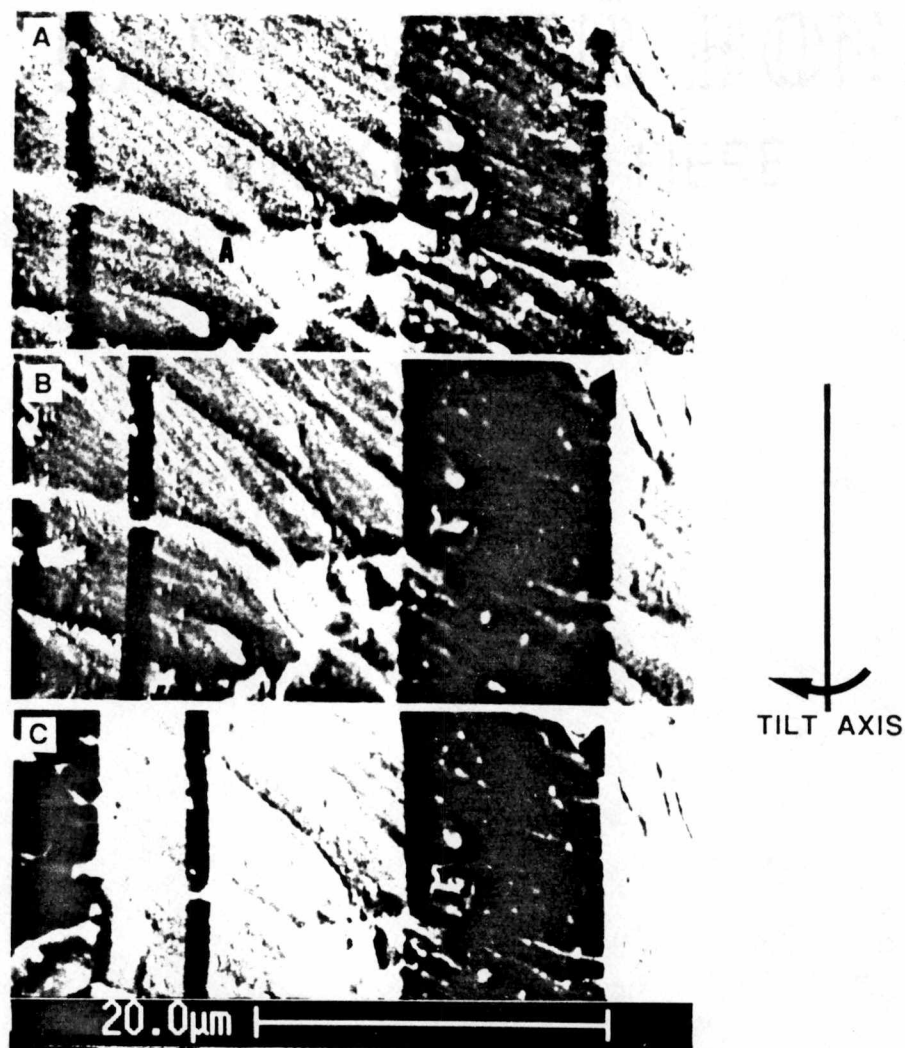


Figure 94. SEM images of the tilting sequence a) 0° tilt b) 18° tilt c) 30° tilt. The tilt axis was aligned parallel to the intersection line. Note that the facet A was tilted away from the plane normal to the optic axis and facet B was tilted towards the normal and then away from it during the 30° tilt sequence.

TABLE 11. ANALYSIS OF PROJECTED WIDTHS OF FACETS ACROSS THE TWIN INTERFACE AS A FUNCTION OF TILT ANGLE.

Tilt Angle	Projected widths of the facets (mm)	
	left	right
0^0	$18.0 = W_l \cos \theta$	$11.5 = W_r \cos \alpha$
18^0	$14.5 = W_l \cos (\theta + 18)$	$12.0 = W_r \cos (\alpha - 18)$
30^0	$11.5 = W_l \cos (\theta + 30)$	$11.4 = W_r \cos (\alpha - 30)$

Based on the anticlockwise convention of tilting from the normal to the electron beam, the left facet (W_l) is continuously decreasing (i.e., its angular rotation is continuously away from the normal and θ is increasing) in its projected width and the right facet (W_r) first increases (i.e., approaches normal to the electron beam) then decreases (i.e., moves away from the normal and α is decreasing) in width. The angles θ and α were calculated using simultaneous equations for the three tilt positions and averaged. $\theta_{avg}=25.33$ and $\alpha_{avg}=11.43$ hence, the included angle $=180-25.33-11.43=143.24$ degrees.



Figure 95. SEM oblique view of layered $\{111\}$ plane fractures indicating the large included angle.

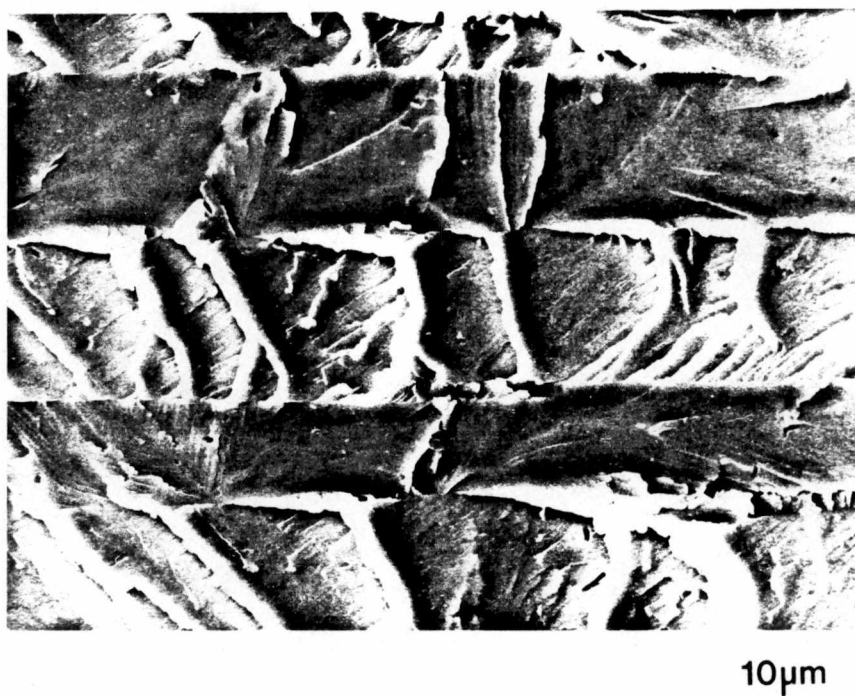


Figure 96. An isolated case observed where the facets are not symmetrical across the matrix-twin interface. Note interface cracking in this case.

observed in this particular case.

Having established the included angle, the sample was electropolished to remove the Bilby surface damage layer and the specimen repositioned inside the SEM, such that $\theta \approx \alpha$, for taking channeling patterns. A [112] type pole was observed for this orientation for both the matrix and the twin. For this orientation the (111) twin plane is exactly parallel to the electron beam and the common direction for both the matrix and the twin should be of the [112] type, as experimentally observed, figure 97. The facets were tilted about the $\langle 110 \rangle$ intersection line over a large range of tilts both clockwise and anticlockwise to generate [111] and [100] channeling patterns, figure 97. The facet was normal to the beam for the [111] channeling pattern observed.

Having ascertained the crystallographic nature of cracking associated with the twins in TiAl+Mn alloys, we now attempt to explain the origin for such fractures. The following dislocation reactions have been proposed (49,50) for observed fractures on (111) slip planes in TiAl alloys.

$$1/6[112](111) + 1/2[110](111) = 1/3[111](211) \quad \text{FP (111)}$$

$$1/2[112](111) + 1/6[112](111) = 2/3[111](112) \quad \text{FP (111)}$$

Note that the $1/6[112]$ partial dislocations are the twinning dislocations in L1₀ alloys. An obvious fact apparent is that alloys where [112] dislocations show increased mobility, or where the twinning tendency is high, as is the case for TiAl+Mn and TiAl+Ga alloys, the material may be susceptible to fracturing on the (111) planes.

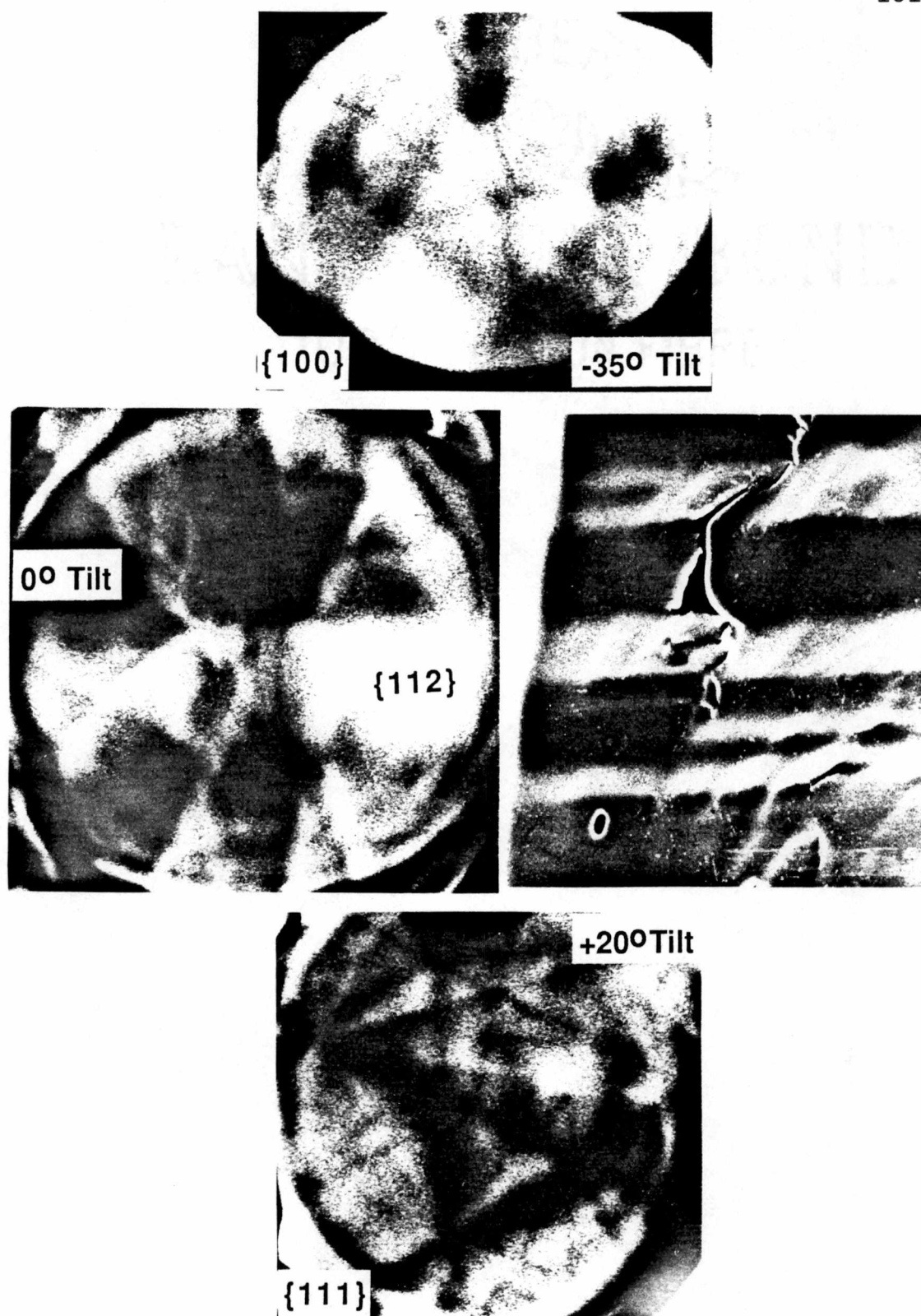


Figure 97. SACP of the twin facets over a range of tilting angles.

A fracture path argument is proposed for the specific fractures observed here. Consider, for simplicity, a single crystal of TiAl+Mn with a matrix-twin interface, figure 98, with the twin plane aligned parallel to the tensile axis. From Griffith's Criterion, strain energy of fracture can be assumed to be transformed into the energy of the surfaces that are created by fracture. Since TiAl fractures on several different planes, the fracture planes most likely to be observed should be one with the highest resolved normal stress, and possibly combined with the least amount of strain energy expended to create surfaces. Considering figure 98, this would indeed be a $\{111\}$ plane both for the matrix and the twin. The shortest path actually is the $\{112\}$ plane common to both the matrix and the twin. However, in binary TiAl single crystals it was never observed and nor does it occur in TiAl+Mn alloys. Symmetrical fractures across the interface of $\{111\}$, $\{100\}$, $\{110\}$ type will have an included angle of 141.06° , 109.4° and 70.53° , respectively. Among the three observed fracture planes for TiAl, symmetrical $\{111\}$ planes offer a combination of maximum resolved normal stresses and the shortest available path. It is noteworthy that even for cases where the twins were at sufficiently large angle to the tensile axis, the fractures were still reconciled to an included angle of about 140° .

It must be mentioned that there exist two combinations of non-symmetrical fractures across the matrix-twin interface that may satisfy the shortest path argument in

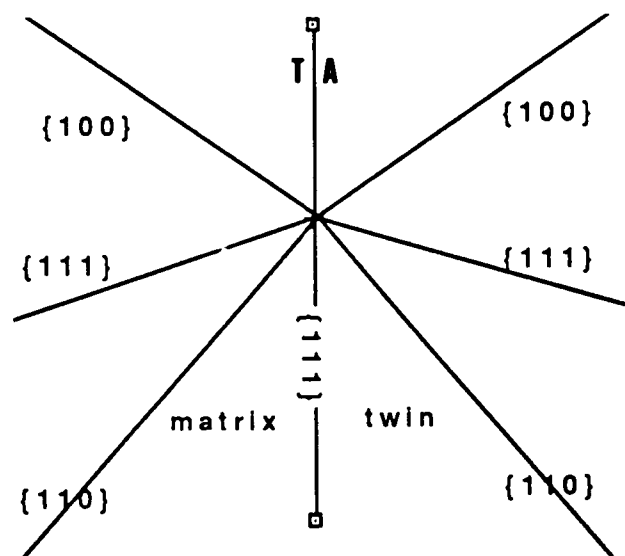


Figure 98. Schematic of a single crystal with a twin interface aligned parallel to the tensile axis. Angular traces for symmetrical fractures across the matrix-twin interface are mapped.

certain orientations of the twin plane with respect to the bending axis. These will be a combination of (100)/(110) and (100)/(111) planes with an included angle of 160.56° and 164.17° . Neither of these combinations have been observed. This may be in part because the large included angles present a somewhat flat perspective in the SEM stereo imaging experiments, thereby not easily detectable.

Summary and Conclusions

It has been shown that the symmetrical fractures across the twin boundary are of the {111} type. In part, our results also shows that {112} is not the common fracture plane. It is evident that twinned structures deflect the crack consistent with the matrix-twin orientation relationship. For the case of twin plane parallel to the tensile axis (this was indeed the case for TiAl+Ga DS alloys) such fractures would result in an increase in path length. This intuitively implies a larger plastic work term for fracture.

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APPENDIX

DEFORMATION BEHAVIOR OF HIGH TEMPERATURE INTERMETALLIC TiAl ALLOYED WITH GALLIUM

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ABSTRACT

TiAl (γ) intermetallic has been investigated with ternary additions of up to 10 at% Ga. Optical microscopy reveals that gallium can be retained in solution to at least 7 at%. Microhardness data reveals that Ga substituted for Al such that $Ti/(Al+Ga)=1$ causes solution softening. Deformation behavior of the single phase alloy in compression is reported. Perfect dislocations of the $a/2[110]$ type and superdislocations of the $a[011]$ type and $a/2[112]$ type have been observed. TEM results indicate that primary deformation is contributed by $a/2[110]$ type dislocations and twinning on (111) planes with wavy slip optically observed.

INTRODUCTION

TiAl is brittle at ambient temperatures. Considerable work has been devoted to understanding the mechanism of this brittle behavior [1-7]. Researchers have attempted to improve the ambient temperature ductility by ternary additions to TiAl. Most notably Ag, V, Nb, Cr and Mn additions have been reported to improve room temperature ductility [5,8-11]. Micro additions of Ni and B also enhance the ductility and castability of the TiAl base alloys [12].

Current research reports on the modified deformation behavior of TiAl when alloyed with Ga. Efforts were limited to improve the room temperature properties while still maintaining a single phase, $L1_0$ compound. Additions of Nb, V, Mo, Mn and Ga were chosen to substitute for either Ti or Al. Ga proved to be most promising based on preliminary hardness measurements and compression tests.

EXPERIMENTAL PROCEDURE

Alloying Guides

- The choice for gallium as an alloy addition was based on the following: Ga exhibits a good solubility in both Al and Ti. Hence, Ga additions might still retain a single phase microstructure.
- Ga forms an $L1_0$ compound with Ti as does Al. Intuitively, Ga may substitute preferentially for Al in the matrix while still retaining the $L1_0$ structure since Ga belongs to the same periodic group as Al and the electron/atom ratio is conserved.
- TiAl has similar lattice parameters ($a=0.3997$ nm, $c=0.4075$ nm, $c/a=1.0195$) to TiGa ($a=0.3970$, $c=0.3970$, $c/a=1.00$) [13]. It was thought that Ga additions may decrease the tetragonality of the TiAl lattice and enhance the plastic deformation behavior.

Experimental Techniques

The alloys were prepared from the individual elements of 99.99% or better purity. Alloy buttons were made in an enclosed chamber on a water cooled copper hearth using a non-consumable tungsten electrode under a constant 30 cubic feet/hour argon flow. The buttons were remelted at least

six times, turning the button after each melt pass for better mixing. The weight loss was less than 1% for all melts accepted for further study.

The alloy buttons were wrapped in Ta and encapsulated in silica tubing and evacuated to 10^{-6} torr pressure, purged with argon, then sealed under a partial pressure of argon. The samples were homogenized at 1100°C for at least 100 hours. Microhardness measurements were made using a diamond pyramid indenter with a 100 gram load. 3 mm diameter rod specimens were spark machined for TEM studies. Thin slices of this rod were cut with an H/D ratio of at least 0.3 for compression testing. TEM specimens were jet polished with a solution of perchloric acid, ethanol and butoxy ethanol at -100°C . TEM observations were made in a Phillips EM 300 instrument.

RESULTS AND DISCUSSION

Microhardness and Optical results

Figure 1 shows microhardness data for binary TiAl and TiAl + 5 at% Ga with a varying Ti/Al ratio. TiAl has a hardness minimum near the 50:50 ratio whereas the gallium alloyed TiAl has lower hardness values at Ti/Al ratios greater than 1. The addition of Ga, substituting preferentially for Al, softens the matrix. Optical metallography reveals a single phase microstructure for all the compositions shown, Figure 2. Optical data had earlier indicated that all alloys containing greater than 51 at% Ti, the microstructure contained two phases irrespective of the ternary addition. Hence, a second set of hardness data was obtained for increasing Ga additions to TiAl, holding the Ti content at 50 at%, Figure 3. Results show that Ga softens TiAl while still retaining a single phase up to 7 at% Ga. A sharp increase in hardness at 10 at% Ga was accompanied with a two phase microstructure.

FRACTOGRAPHY

Three point bend tests were performed at room temperature. Fractography revealed an increasing tendency to intergranular failure with the increasing Ga content, Figure 4. At 10 at% Ga, the fracture was approximately 15% intergranular.

X-RAY DIFFRACTION

X-Ray data of powder specimens is shown in Figure 5. Results show that an increasing Ga content decreases the tetragonality of the Ll_0 lattice.

Based on the combined results of hardness, fractography and X-Ray data, an optimum composition of Ti-50/Al-45/Ga-5 was selected for deformation studies. A thin slab of this alloy was polished, etched and deformed 7.7% in compression. The polished surface reveals wavy slip lines, Figure 6.

TEM STUDIES

Three different dislocations were identified in this alloy. These were the $a/2[110]$ based on invisibility criterion for (002) and (113) diffracting vectors; $a[011]$ based on invisibility for (111) and (311) diffracting vectors; and $a/2[112]$ based on invisibility for (220) and (311) diffracting vectors. These dislocations are labelled as AA, BB, CC respectively, Figure 7. For lack of space only selected micrographs are presented. These dislocations modes have been previously reported for TiAl alloys [2,3,7]. Figure 8 shows dislocation activity in a specimen loaded to 600 lbs. These

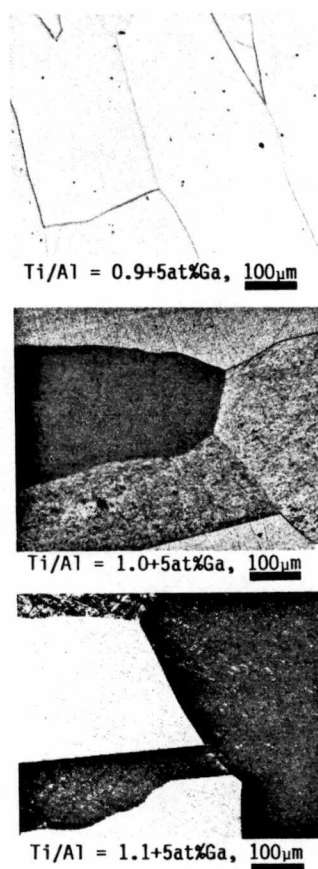


Fig. 2

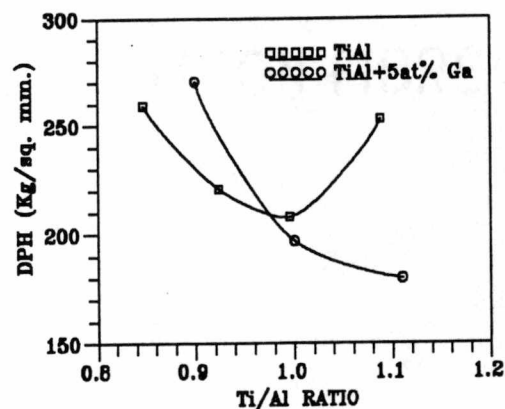


Fig. 1. Microhardness as a function of Ti/Al ratio for the intended compositions.

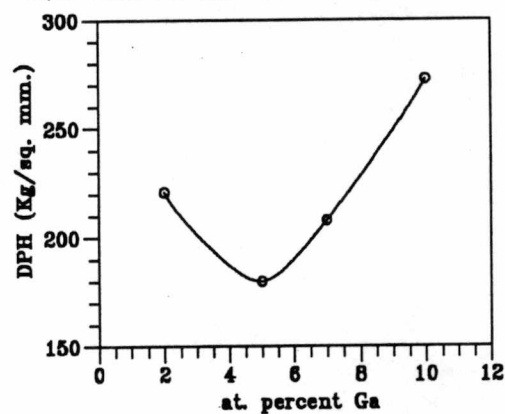


Fig. 3. Microhardness data for the intended Ti = 50at%/(Al+Ga) = 50at% compositions.

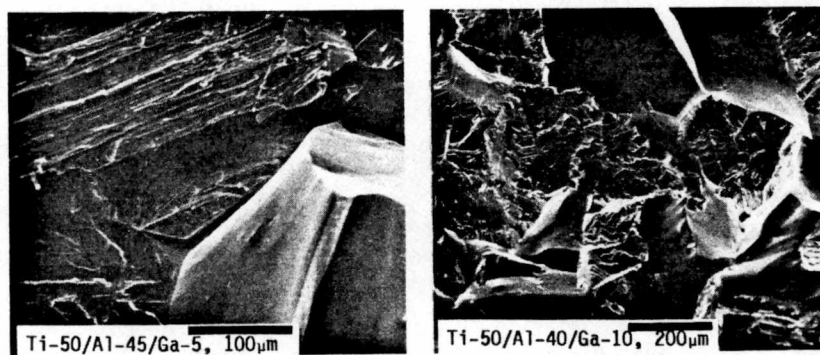


Fig. 4. Mixed mode fractures obtained in 3 point bend tests. Note increased intergranular mode at high Ga content.

240

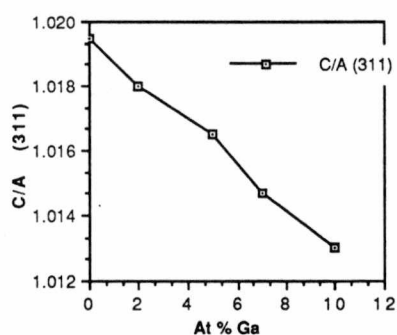


Fig. 5. Ll_0 lattice tetragonality as function of Ga keeping Ti at 50at%.



Fig. 6. Wavy slip in bulk specimen deformed 7.7% in compression.

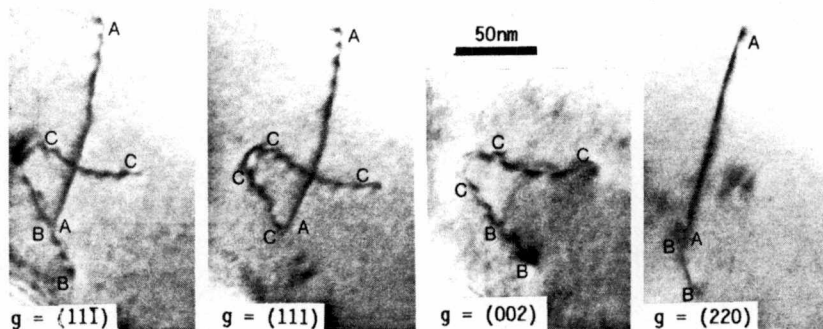


Fig. 7: Dislocation analysis.

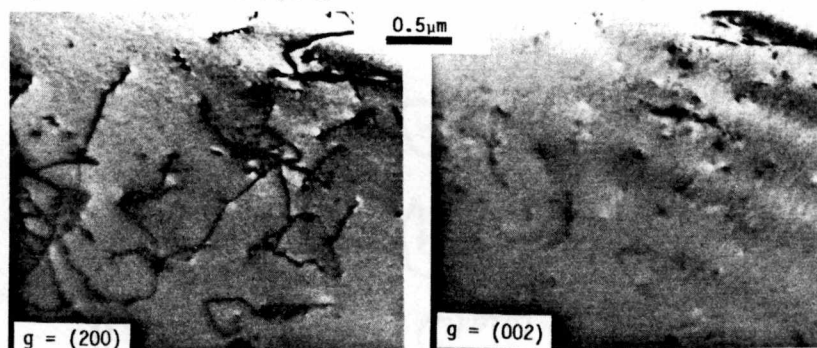


Fig. 8. Specimen loaded to 600 lbs., predominant activity of the perfect $a/2$ $[110]$ type dislocations.

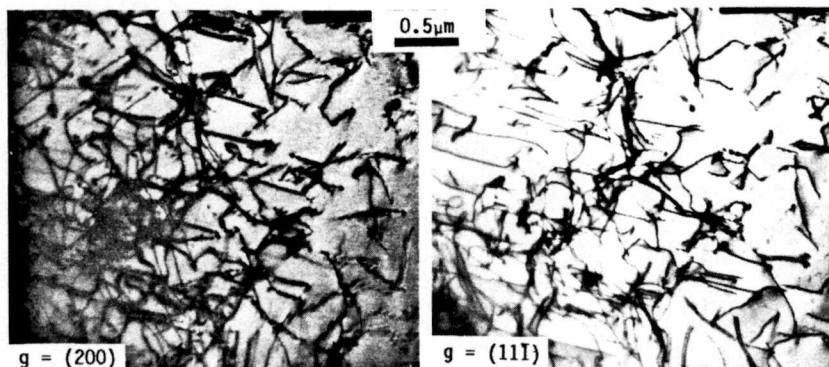


Fig. 9: Specimen loaded to 800 lbs., dislocation tangles.

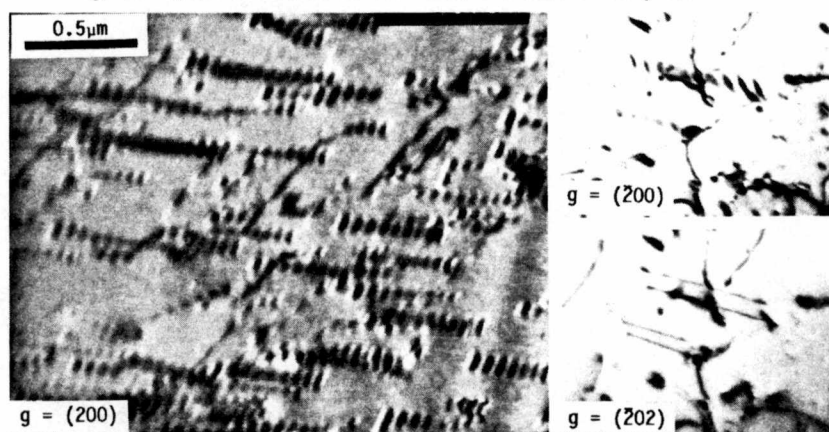


Fig. 10. $a/3$ (111) stacking faults bounded by $a/6$ [112] type partial dislocations.

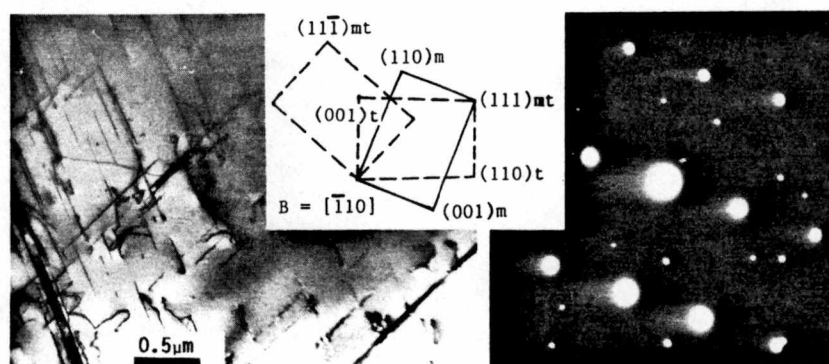


Fig. 11. Twinning on (111) and (111) axes as shown in the SADP and indexed patterns.

dislocations were identified as $a/2[110]$ type perfect dislocations based on their characteristic invisibility for a (002) diffracting vector. Twins are observed frequently with a (111) twin axis. Superdislocations of the type $a[011]$ and $a/2[112]$ were not observed in this specimen. Figure 9 shows dislocation tangling in specimen loaded to 800 lbs. In addition to the tangles, superdislocations are observed. Figure 10 shows stacking fault formation in this specimen which is a further indication of superdislocation activity at this load [3]. Twinning tendency also increases. Figure 11 shows twins with (111) and (11 $\bar{1}$) axes. Thus, $a/2[110]$ dislocations and twinning contribute to the plastic deformation of the alloy at lower loads. At higher loads these dislocations tangle thus restricting further movement. At this stage only the superdislocations become active.

CONCLUSIONS

- 1) Gallium softens the TiAl matrix.
- 2) Gallium addition influences the fracture mode.
- 3) Gallium reduces the tetragonality of the TiAl L_{10} matrix.
- 4) Gallium introduces macroscopic wavy slip in TiAl under compressive loading.
- 5) All three dislocation modes are present with primary deformation contributed essentially by perfect dislocations of $a/2[110]$ type.

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VITA

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