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Final Proceedings of The EOARD/IRC-sponsored International Workshop on Gamma Aluminide Alloy Technology

held from 1 to 3 May 1996 at The IRC in Materials for High Performance Applications The University of Birmingham

SECTION TWO

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in

Materials for High Performance Applications

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XD TiAl Alloys as an Example

Department of Materials

L. Christodoulou

Imperial College

London

Microstructural Effects in γ -Titanium Aluminides;

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Influence of Reinforcement on Microstructure

- Refines the grain size
- Grain structure uniformity
- Improved workability
- Modifies kinetics of
- Recrystallization
- Grain growth
- TiB₂ does not appear to change intermetallic phase transformations or shift phase boundaries
- Complex precipitation reactions with nitrides, carbides and in matrices containing "active" transition elements •



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XDTM Processing Applied to TiAl-based Materials

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- For gamma-based materials:
- Investigated tens of reinforcement (borides, nitrides, carbides silicides) /matrix combinations
- For compositions containing V, Cr, Mn, Fe, Co and Ni TiB $_2$ is the stable reinforcement
- For compositions containing Zr, Nb, Mo, Hf, Ta and W the stable reinforcement is (Ti,X)B



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.%v St of Instancy yllaitneses

- Grain retinement occurs at low wolves the subcode of 1.8.



Grain Size versus Reinforcement Loading

CAST XD" TITANIUM ALUMINIDE COMPOSITES SOLIDIFICATION STRUCTURE



SX

- GRAIN REPINEMENT
- MCREASED MICROSTRUCTURAL UNIFORMITY
- STABLE MICROSTRUCTURE
- PARTICLE STRENGTHENING

GO-10.00-7

HOWMET COMPONATION



Microstructural Options

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Fully Lamellar



Fully Lamellar (deformed)



Deformed Lamellar / Equiaxed



Fully Equiaxed

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Duplex Structure



Spheroidized Lamellar



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Extruded

MARTIN MARIETTA

PHASE MORPHOLOGY OF XDTM TI-ALUMINIDES: COMPARISON WITH BASE ALLOY

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Heat Treated Condition



As-Extruded + 1200°C/16 hr Ti-45 a/o Ai (Base Alloy):

Bright Field Im**age Take**n at [110] $_{\gamma}/$ [1120] $_{lpha_2}$ Zone Axis of Lamellae 10µm

WELL FIRE WIN PUT LA REAL

Ti-46AI: Extruded then Aged 5 Hours at 1200°C

Strengthening Contributions



$$* \sigma_{c} = \sigma_{o} + \Delta \sigma_{m,p} + \Delta \sigma_{F}$$

where σ_c = Strength of the composite

= Reference stress = strength of base alloy given identical thermal history 0**0**

 $\Delta \sigma_{m,p}$ = Strength increase of matrix due to presence of particulate (indirect effects)

 $\Delta \sigma_p$ = Strength increase due to particulate (direct effect)

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TiB₂ Reinforcement of XDTM Cast Titanium Aluminides

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- Addition of TiB2 results in strengthening of all titanium aluminide matrices examined
- The response to TiB₂ additions varies with base alloy composition



Direct Strengthening Contributions in XDTM Ti-Aluminides



outering of AD¹¹⁴ Hitanium Aluminide Composites

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Plastic Elongation vs Proeutectoid Gamma



MARIN MARIE TIA

80 <u> 8</u> Primary Gamma (%) 4 8 0 750 800 **0**00 200 650 Yield Strength (MPa)

<u>**Yield Stress vs Proeutectoid Gamma**</u>

نة. ا MARTIN MARIETTA



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Fracture Toughness as a Function of Microstructure



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Creep Trends

- Strong grain size dependence
- Lamellar microstructures superior to equiaxed
- Deformed lamellar particularly poor---"dynamic recrystallization" effectively results in very fine grain size and repeated stage I creep •
- Reinforcements improve creep (all other microstructural features being equal) •

MARIN WARELIN





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Average Particle Equivalent Diameter = 1.85 μ m Standard Deviation = 1.15 μ m MARTIN MARIETTA

Grain Size-Dependent Creep Deformation

TI-47AI

Ti-47AI + 7 v% TiB2 (Particulate / Equiaxed)







Suggests development of a more-extensive dislocation population in lamellar matrices

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MLL JIM DO NILLICA

1000

100

2

10 ⁶

200

160

120

8

9

0.0

0.5

Time, h

Initial Stress, MPa

Mechanisms of Creep Deformation

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 $\dot{\boldsymbol{\epsilon}}_{\text{ss}} = \dot{\boldsymbol{A}}_{\text{o}} \cdot \boldsymbol{exp} \left(\textbf{-Q}_r \; / \textbf{RT} \right) \cdot \boldsymbol{\sigma}^n$

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Lamellar (proposed)	≈ 3 - 4	≈ 300 - 350 kJ	=f(λ ⁿ ,ρ, d ^p)?
	Stress Exponent, n	Activation Energy, Q _{cr}	Structure Constant, A
Equiaxed	8-6 ≈	≈ 300 - 350 kJ	= A ₁ • (b / d) ²

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* For TiAl: Martin et al., 1981;
Kampe et al., 1989;
Takahashi et al., 1991

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May 1, 1996

Joint EOARD / IRC International Workshop on Gamma Aluminide Alloy Technology

Preliminary results on point defects, atomic mobility and creep in model TiAl compounds

Centre d'Etudes de Chimie Métallurgique (C.N.R.S.) Address: 15, rue Georges Urbain, 94407 Vitry-sur-Seine Cedex, France Phone : + 33.1.46 87 35 93 Fax : + 33.1.46 75 04 33 Telex : 265 586 F

Types of properties investigated

- Vacancy properties From recovery of irradiation-produced vacancies
- Atomic mobility From kinetics of atomic order variations atomistic mechanism: controlled by vacancy concentration and vacancy migration

• High temperature deformation (creep)

Connected with dislocation climb: directly controlled by self-diffusion, i.e. atomic mobility

MATERIALS

1) High purity and commercial purity binary alloys,

prepared from high purity or commercial purity Ti and AI

Composition: $Ti_{1-x}Al_x$ with x = 48-50-51.5-53-54.5-56 at%

Processing route

Levitation melting and drop casting + remelting on cooled copper boat and directional solidification

Impurity determination in starting materials:

by activation analysis Metallic impurities: neutron irradiation

Oxygen: charged particle (³He ions) irradiation

	Impurity content	(wt ppm)
Metal	Commercial Purity	High Purity
Titanium	Ti 40 (sponge)	Ti VA31
	\$	(refined by iodide process)
	$O = 2500 \pm 200$	O = 27
	typical:	
	Fe = 100 to 1100	Fe = 107
	Si = 50 to 100	Zr = 95
Aluminium	Al6174 (double-electrolysis)	Al H3GS67 (zone refined)
	Fe = 3.3	
	Mg = 17	Total ≤ 1
	Si = 120	
	Zn = 8	

2) Quaternary alloy: Ti48Al48Nb2Mn2 (CEASI), for creep tests

Processing route: plasma casting + melting on cooled copper boat and directional solidification.

MATERIALS CHARACTERIZATION

Chemical

Bulk concentrations

Determined by Atomic Emission Spectroscopy (ICP - AES)

In high-purity binary TiAl alloys: 50 - 56 at% Al: bulk concentrations consistent with nominal composition (deviations < 0.3 at%, usually 0.1 at%)

4

Homogeneity

Characterized by local X-ray emission (SEM - EDX)

 Al-rich binary TiAl alloys, 50 - 56 at% Al (homogenized 24 h at 1125 °C)

> Ti-56 at% AI is homogeneous (instrumental standard deviation = 0.25 at%)

- less Al-concentrated alloys: some residual heterogeneity (standard deviation of concentration = 0.5 to 1.4 at%)
- Ti-48 at% Al alloys (high purity and commercial purity) and Ti₄₈Al₄₈Nb₂Mn₂:

concentration fluctuations (after directional solidification) in Ti₄₈Al₄₈Nb₂Mn₂.

· Ti is fairly homogeneous

Al fluctuations of amplitude typically \approx 2 at% Nb and Mn: fluctuations anti-correlated to those of Al

MATERIALS CHARACTERIZATION

4

Structure

Methods:

Optical, SEM, TEM observations, X-Ray diffraction

Results

 Al-rich binary alloys 50-56 at% Al, homogenized 24 h at 1125°C:

single phase γ , equiaxed grains

(in the Ti-50at%Al, presence of a very small amount of γ lamellae and α_2 particles)

• Ti-48at% AI (high purity and commercial purity), Ti48-AI48-Nb2-Mn2

(after directional solidification)

- large grains of lamellar $\gamma+\alpha_2$ structure, elongated in the direction of solidification

1. VACANCY MIGRATION

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Experimental

Materials: single-phase γ TiAl

Homogenized high purity alloys composition: 50 to 54.5 at% Al

Production of point defects

low temperature (21 K) irradiation with 2.5 MeV electrons

Defect elimination

investigated by

residual electrical resistivity measurements, during isochronal anneals at increasing temperatures, in range 21 K - 800 K







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1. Vacancy migration

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Results

2 main recovery stages, at ≈ 80 K and at ≈ 440 to 510 K

 Stage I assigned to close-pair recombination and self-interstitial migration position nearly independent of alloy composition

 Stage III assigned to vacancy migration (to be confirmed by positron lifetime measurements) shifted to higher temperature in alloys with higher AI content

Conclusion

Vacancy mobility decreases

when Al concentration increases

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2. ATOMIC MOBILITY

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Materials: single-phase γ TiAl

Homogenized high purity alloys composition: 50 to 56 at% Al

Method

Analysis of atomic order changes resulting from heat treatments (monitored by residual electrical resistivity measurements)

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Qualitative data: from isochronal annealing curves

Quantitative determination of characteristic parameters (jump frequencies, activation enthalpies) from kinetics of atomic order relaxation

Typical isochronal curve

9 Order changes during isochronal thermal Theatments intermetallic compounds en TiAl 12 TIAI50 T 11 TIAISO T X ρ 4.2K (μΩ.cm) 10 . Same behaviour as in Niz Al 9 8 . Reversible resisturity variations 818K 7 LRO changes due to T(K) 400 1000 600 800 ٩. 1200 23 · Composition dependence: T 🛪 TiAI51.5 TiAI51.5 Т × 22 p 4.2K (Jit2.cm) - amplitude of relatinty 21 Variation 20 7941 19 - shift to lower temperatures T (K) 18 of atomic mobility onset (To) 400 600 800 1000 1200 . 42 T 7 T ¥ TIAI53 with increasing Al content 41.9 TIAI53 41.8 (IID.cm) 41.7 41.6 41.5 . Atomic mobility becomes 41.4 To - 733 K 41.3 faiter when deviating. Т (К) 41.2 400 600 800 1000 1200 Stoicheometry 64.6 TiAI54.5 トア 64.4 TiAI54.5 KT stoicheometry • .2K (μΩ.cm) AL 64.2 64 atomic mobility IS 63.8 63.6 smaller than in NiAP Ta:720 63.4 (shift by GOK) 63.2 400 600 800 1000 1200

COMPOSITION DEPENDENCE OF ATOMIC MOBILITY AND VACANCY PROPERTIES

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When AI concentration increases,

• Atomic mobility (i.e. atomic jump frequency: v at) increases

> (consistent with results of Oikawa et al. (1992) creep rate increases with off-stoichiometry)

Vacancy mobility (i.e. vacancy jump frequency: vv)
decreases

 Consequently, vacancy concentration c_V appears dependent on TiAl composition. Since

vat ~ $c_V.v_V$,

cv increases with increasing AI content (in agreement with vacancy formation enthalpy calculations of Badura and Schaefer, 1993)

3. CREEP

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Materials: two-phase Ti-48 at% AI alloys

High purity binary Ti-48 at% AI alloy Commercial purity binary Ti-48 at% AI alloy Quaternary Ti₄₈Al₄₈Nb₂Mn₂ alloy (CEASI)

Processed by melting on water-cooled copper boat and directional solidification

Microstructure in all 3 alloys, large elongated grains of lamellar $\gamma + \alpha_2$ structure

Creep tests at 800 °C under high vacuum compressive stress = 260 MPa

Results

Stationary creep

• in commercial binary and quaternary alloys,

creep rates are: similar (consistent with results of Hayes and McQuay, 1994)

duration of stationary creep shorter in quaternary than in commercial purity binary alloy

• in high purity binary alloy, rate is one order of magnitude larger

Possible mechanism

Impurity (oxygen ?) strengthening of γ phase



Micro and Macro Segregation Patterns in a PACH Melted Near Gamma Titanium Aluminide

Objectives

To identify the physical and operational parameters which influence solute partitioning and macro-segregation in PACH melted titanium aluminides.

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C. Hallward



R&D Programme on Evaluation and Minimisation of Compositional Variations in PACH Melted Titanium Aluminides.

- Microstructural and SEM studies to determine the extent of variation in local solidification conditions and to ascertain the levels of microsegregation.
- Development of quantitative chemical analysis procedures (GD-OES) for assessing the levels and the spatial variations in alloy composition across the ingot.
- Macrostructural and analytical studies to determine the correlations between process parameters, macrostructural development and macro-segregation.
- The development of process monitoring and control procedures to minimise periodic and / or random variations in melting and casting conditions.

Microstructural and SEM Studies

Objectives:

To determine the effects of process conditions on local solidification time and the extent of microsegregation.

Experimental:

- Grain orientation measurements to determine the depth and shape of the melt pool and its variation with casting conditions.
- SDAS measurements to determine the extent of variation in local solidification time.
- Vertical float zoning to simulate the effects of different solidification rates on SDAS and solute partitioning.
- EDX studies to determine the effects of different solidification and casting conditions on micro-segregation.

Macro / Microstructures Observed in PACH Melted Ti-48AI-2Mn-2Nb

Equiaxed Chill Zone

(Mid-Radius / Centre) Cored-Columnar





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Optical micrograph and EDX analysis plots showing the spatial variations in Al. Mn and Nb within the columnar / dendritic region of PACH melted Ti-48Al-2Mn-2Nb.

SEM Back Scattered Electron Images Showing Dendrite Growth Morphology Within the Columnar Zone



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EDX profiles showing compositional variations across the secondary dendrite arms of a hexagonal dendrite observed in float zone melted Ti-48Al-2Mn-2Nb (sample number B1 / Zoning Rate ≈ 0.08 mm/s).

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EDX data showing correlations between Al, Nb and Mn segregation in float zoned Ti-48Al-2Mn-2Nb alloy.

EDX Data Obtained from Ingot and Float Zone Melted Ti-48Al-2Mn-2Nb Showing Variations in Al Concentration and Extent of Inter-Dendritic Segregation.

Bar N°	Zoning Ratc (mm/min)	Mcan	Мах	Min	Range	Std Dev	Partition Coefficient (C/Co)
11	N/A	48.45	50.52	45.17	5.36	1.09	0.94 - 1.05
N/A	4.8	48.14	51.16	46.29	4.88	1.24	0.96 - 1.06
V/N	9.6	47.57	49.41	45.28	4.13	1.03	0.94 - 1.03
N/A	15.0	47.55	48.71	45.90	2.81	0.66	0.96 - 1.01

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Glow Discharge Optical Emission Spectrometry

Problems:

Assumes that the intensity of an element x of concentration C_x is linearly dependent on the emission yield k_x and the sputter rate q. Problems arise in that the sputter rate is matrix dependent and the emission yield is dependent on all three lamp parameters (voltage, current and argon flow rate). These parameters are not separately variable.

Experimental:

- Production of chemically homogeneous samples with varying alloy contents for the determination of calibration curves.
- Operational studies to determine voltage, current, flow rate correlation's and the effects on emission yield.
- Determination of sputter rate variations as a function of operating conditions and matrix composition.
- Determination of optimum operating conditions and calibration procedures.



Schematic illustration showing the layout and principal operating features of the LECO GDS Spectrumat 750 Glow Discharge Optical Emission Spectrometer.

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∆ Ti-3V-14A1-20Nb-2Mo

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- □ Ti-48Al-2Mn-2Nb
- ♦ Ti-48Al
- O Ti-52Al





GD-OES calibration curves for Aluminium in Titanium and Titanium / Aluminium alloys showing the effects of variations in Argon flow rate. Excitation voltage and lamp current were controlled so as to maintain a constant applied power of approximately 40 watts.

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XRF analysis results showing showing compositional variability in transverse sections taken from Bar N° 11

Slice N°	AI (WI %)	Mıı (Wt %)	Nb (WI %)	Al (At %)
1	- 32.7	2.86	4.99	47.36
2	34.10	2.83	4.78	48.92
9	33.50	2.80	4.91	48.26
4	32.70	2.90	5.14	47.40
5	33.40	3.01	5.06	48.18

Comparison of XRF and GD-OES Analysis Results for Bar N° 11

Mcthod	Mcan	Maximum	Minimum	Range
XRF	48.02	48.92	47.36	1.56
GD-OES	47.70	50.68	46.42	4.26

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Macrostructural and Analytical Studies

Objectives:

To determine the effects process parameters, and in particular the role of transient variations in melting and casting conditions, on macrostructural development and macro-segregation in PACH melted Titanium Aluminide.

Operational Parameters:

- The frequency and rate of transfer of liquid metal between the hearth and the crucible.
- The height, trajectory and power of the crucible and hearth torches.
- The rate of delivery of the feedstock to the hearth.
- The rate of ingot withdrawal from the crucible and the extent of mould oscillation.
- The ingot top position and the level of liquid metal within the crucible.
- The extent of electro-magnetic stirring.







Aluminium Concentration Variations in As-Cast and Isothermally Forged Ti-48Al-2Mn-2Nb

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GD-OES Contour Map Showing Longitudinal Variations in Al Concentration as a Function of Distance From Dove-Tail (Ingot Bottom).







Withdrawal Rate (mm/s)

Data acquisition data obtained for Bar 81 showing transient variations in ingot withdrawal conditions within the near-dovetail region (ingot bottom)

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Crucible Torch Current (amps)

Parameters contributing to banding in PACH melted Ti-48Al-2Mn-2Nb;

- Inhomogeneous feed-stock.
- Intermittent pouring of liquid metal from the hearth to the crucible.
- Fluctuations in the height of liquid metal in the crucible.
- Variations in withdrawal rate.
- Changes in the height, pattern and power of the crucible and hearth torches.

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Process Monitoring and Control

- Casting rate control through in process monitoring of the level of liquid metal in the crucible and the control of ingot withdrawal and the rate of delivery of the feed-stock to the hearth.
- Temperature distribution within the hearth and crucible through thermal imaging and the associated control of toirch trajectories and operating parameters.
- Optimisation of stir coil design and operational parameters.

Plasma Melting

Process Control





Bar 109 - 48-2-2, bar feedstock

Summary of GD-OES Results Showing Effect of Improved Process Control on Aluminium Concentration Variations and Levels of Macro-Segregation

Bar N°	Mean	Max	Min	Range	Std Dev	Comments
11	47.70	50.68	46.42	4.26	0.71	Withdrawal rate & torch current trials. (Compacts, no casting rate control)
16	47.99	49.86	44.70	5.16	0.72	Withdrawal rate & torch current trials. (Compacts, no casting rate control)
26	47.88	49.88	45.26	4.62	0.97	(Compacts, no casting rate control)
81	47.93	50.02	44.42	5.60	1.15	Cast start-up / dove-tail region. (Compacts, no casting rate control)
16	47.93	48.44	46.72	1.73	0.23	Torch current trials. (Bar stock, level detection & casting rate control)
11	48.45	50.52	45.17	5.36	1.09	EDX Micro-Segregation Data. (Inter-Dendritic)
V/N	47.81	51.16	45.28	5.88	0.83	Float Zoned (4.8 -15.0 mm/min). (Interdendritic)

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Conclusions

- Despite partition coefficients close to unity (C/C₀ ≈ 0.94 -1.05) TiAl based alloys exhibit high levels of inter-dendritic segregation, primarily because of the high Al concentrations.
- GD-OES analysis has shown that these levels of segregation can occur on the macro scale and are often typified by the development of alternating bands of Al enrichment and depletion extending along the length of the ingot. These features are not detected using XRF analysis
- Macrosegregation patterns observed in cast TiAl ingots are retained after forging and have been linked to problems with cracking during forging and changes in heat treatment response during subsequent thermal processing.
- Macrosegregation is found to be most severe in ingots where transient variations in withdrawal rate are at a maximum.