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LA-UR -90-25

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DE90 006490

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FEB 09 1990

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TITLE. DEFORMATION AND FRACTURE OF TiAl + W AT ELEVATED TEMPERATURES

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SUBMITTED TO. **To be presented at and published in the proceedings of
Fourth International Conf. on Creep and Fracture of Engineering
Materials and Structures; 1-6 April 1990, University College,
Swansea, U.K.**

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Deformation and Fracture of TiAl + W at Elevated Temperatures

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1.0 SUMMARY

The ternary alloy, Ti - 49 at% Al - 2 at% W, was produced using Rotating Electrode Powder (REP) compacted by hot extrusion. The tensile properties of this alloy (strength, ductility and fracture mode) were studied from room temperature to 900°C. Constant load creep properties were measured from 700 to 900°C and analyzed using conventional power law equations to calculate the stress exponent and activation energy. These parameters were approximately 4 and 400 KJ/mole respectively. TEM examination showed that the W was held in solid solution during the tensile and creep deformation when the material was tested in the 'as-extruded' condition. These results are interpreted as evidence for solid solution strengthening of the TiAl matrix by the W solute.

2.0 INTRODUCTION

Advanced aerospace systems are putting increased emphasis on lighter weight structures at the same time that they are requiring higher operating temperatures from the materials comprising those structures. Ordered intermetallic alloys have been the subject of intense interest due in large part to their retention of attractive properties at elevated temperatures. As is well documented in the literature, a lack of low temperature ductility and toughness is their major deficiency. The alloys based on the ordered compound TiAl are no exception [1; 2; 3; 4]. This compound has a low density,

oxidation resistance to 850°C and an elastic modulus significantly greater than either of its elemental constituents [5].

Ternary elements are known to have dramatic effects on the physical and mechanical properties of intermetallic compounds. While some progress has been made in understanding the alloying effects on low temperature ductility in TiAl [6], less information is available in the literature concerning ternary effects on high temperature deformation behavior. Since the material under study was made by powder metallurgy techniques, specifically the REP process using a tungsten electrode to maintain an arc on the spinning ingot, it seemed appropriate to delineate the potential effects of W ternary additions on TiAl. Although W has not been found in large quantities in previous alloys in bulk chemistry [3; 4], unintentional inclusions of W could cause the local chemistry to approach several atomic percent. Some data on the mechanical properties of TiAl+W, in a microstructural condition approaching equilibrium, have already been discussed [7; 8]. This paper will present data on the same alloy, nominally Ti- 49 at% Al- 2 at% W, except that the majority of the ternary W addition has been held in solid solution instead of heterogeneously precipitated on internal surfaces. Both uniaxial tensile data and tensile creep properties have been determined from room temperature to 900°C.

3.0 EXPERIMENTAL PROCEDURE

The prealloyed ingots were cast into iron pipe to become a suitable form for conversion to powder by Nuclear Metals, Inc. using the REP process. The chemical composition of the resulting powder is listed in Table I. Following a sieve to -35 mesh, the powders were sealed in Ti-6Al-4V extrusion cans, evacuated and extruded. Extrusion conditions were varied in order to explore the limits of formability and their effects on the dynamically

Table I. Chemical composition of the TiAl + W alloy (wt%).

Ti	Al	W	Fe	Cu	Mo	Ω	N	H	C
bal	32.3	9.77	0.12	0.049	0.049	0.064	0.012	0.002	0.014

recrystallized grain size and resulting properties of the product. The billet preheat temperature varied from 1400°C to 1260°C with the extrusion ratio being either 26:1 or 40:1. The extruded rod was machined into tensile specimens to the configuration described elsewhere [4] using low stress grinding techniques. The same specimen geometry was used for both tensile and creep testing.

All testing was conducted in air in a three zone clamshell furnace where temperature gradients were limited to $\pm 1^{\circ}\text{C}$ over the gage length. Temperature stability ($\pm 1^{\circ}\text{C}$ of setpoint) was attained in the furnace for at least 30 minutes prior to beginning the test in order to ensure temperature uniformity. Tensile testing was conducted on a screw-driven Instron at an initial strain rate of $3 \times 10^{-4} \text{ sec}^{-1}$. Specimen extension was measured by an LVDT (outside of the furnace hot zone during elevated temperature tests) calibrated so that small plastic extensions (<1% plastic elongation) could be measured accurately. The elongations reported are the uniform plastic elongation measured either to the ultimate stress or to the fracture stress whichever occurred first. The yield stress reported uses the 0.2% plastic offset criteria while the fracture stress reported is a true stress using the final diameter measured after fracture. Creep testing was conducted on a constant load type frame employing automatic load arm leveling and continuous monitoring of strain with a single LVDT outside the furnace.

4.0 RESULTS

4.1 Microstructures Tested

The small diameter product ($\leq 1.25 \text{ cm}$ diameter) was air cooled following the high temperature extrusion. This processing schedule resulted in a fine recrystallized grain size. Figure 1 shows the optical and transmission electron microscopy (TEM) of the ternary TiAl containing W following extrusion at 1400°C and 26:1 reduction. The primary elements of the microstructure are the equiaxed TiAl (γ) grains coexisting with the colonies of lamellae comprised of Ti_3Al (α_2), γ , and twin related γ [3; 4]. Energy dispersive x-ray analysis in the TEM identified W rich precipitates which were located primarily in the equiaxed γ grains. Comparison of this microstructure with the near equilibrium heat treated condition previously discussed [7; 8] clearly illustrates that the γ phase, in the 'as-extruded' condition, is supersaturated in W.

Altering the extrusion conditions resulted in refinement of the dynamically recrystallized grain size without changing the fundamental constituents of the microstructure. Observation at higher magnifications did not show any differences, i.e. the W was primarily in solid solution following all extrusion conditions.

4.2 Tensile Data

Table II summarizes the tensile test results of material extruded from 1400°C with a reduction ratio of 26:1. At least two specimens were tested at each temperature. While the ductilities measured were always modest, the ternary alloy averaged 0.7% uniform plastic elongation at room temperature.

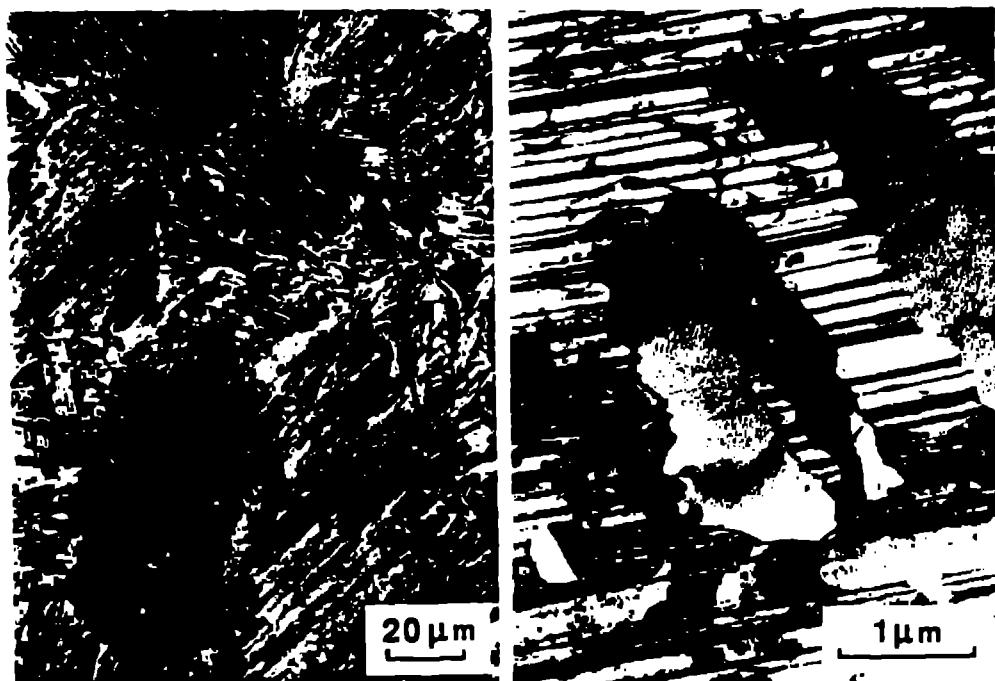


Figure 1. Optical and TEM bright field microstructures following extrusion at 1400°C - 26:1 reduction.

Table II. Tensile data for TiAl+W; extruded at 1400°C with a reduction of 26:1; tested 'as-extruded'; average of multiple tests.

Temp. (°C)	Yield Stress (MPa)	Fracture Stress (MPa)	RA (%)	Plastic Elong. (%)	# of Tests (avg.)
25	599	737	1.3	0.7	5
260	524	692	2.0	1.0	2
500	582	829	3.9	1.5	2
700	528	800	4.9	3.2	2
800	488	733	8.5	5.1	2
900	385	505	8.0	2.3	2

Table III lists the tensile data for the W modified ternary alloy following the more aggressive extrusion conditions. Only a limited amount of tensile data is presented; specifically at 25 and 700°C. Comparison with Table II clearly indicates that the fine grain sized material showed increased yield and fracture strengths. The effect of these processing conditions on the ductility is difficult to discern from the limited data available. However, the data suggests that the standard extrusion condition (1400°C with 26:1

reduction) may result in a lower ductility, at both room temperature and 700°C, at least compared to the extrusion processed at 1340°C with 40:1 reduction in area.

Table III. Tensile data for TiAl+W as a function of extrusion parameters; tested 'as-extruded'; average of multiple tests.

Extrusion (°C-Ratio)	Temp. (°C)	Yield (MPa)	Fracture (MPa)	RA (%)	Elong. (%)	# of Tests (avg.)
1340-40:1	25	718	864	1.6	1.0	2
	700	564	902	7.7	4.2	2
1315-40:1	25	740	813	0.7	0.8	2
	700	583	866	7.6	5.3	2
1260-40:1	25	-	868	1.1	0.1	2
	700	680	856	6.5	3.8	2
1260-26:1	25	-	821	0.2	-	3
	700	649	827	4.2	3.0	2

4.3 Creep Data

Table IV summarizes the constant load creep results. The specimens were tested following air cooling from the extrusion conditions. Only two of the extrusion conditions discussed above were tested in creep. A limited number of specimens were available for the finer grained (lower extrusion temperature) material. Inspection of the similar creep conditions shows that the fine grain sized material had identical creep properties at 700°C to the material extruded at higher temperature but became progressively inferior at increasing temperatures. Comparison should be made between the 850°C/173 MPa creep test results where the time to 1% strain is shorter for the fine grain size material (29.5 versus 96 hours) although it exhibited a lower minimum strain rate (3×10^{-4} versus 8×10^{-4} hr⁻¹). Since the continuously measured strain-time curves for this alloy showed conventional primary and secondary creep stages, this anomaly can be explained by noting that the strain which occurred in the primary stage of creep for the fine grained material was greater than that observed in the coarser grained extrusion.

Conventional power law analysis of the second stage creep rate of this alloy in the large grained condition is possible even with the limited data in Table IV [9]. In this approach, the minimum strain rate is used to determine

Table IV.

Creep data for TiAl+W as a function of extrusion parameters; tested in the 'as-extruded' condition.

Extrusion (°C-Ratio)	Temp. (°C)	σ_{initial} (MPa)	$t_{1\%}$ (hrs.)	$\dot{\epsilon}_{\text{min}}$ (hrs. ⁻¹)	Life* (hrs.)	Elong.* (%)
1400-26:1	700	345	-	1.5×10^{-5}	(100)	(0.16)
	700	414	273	2.5×10^{-5}	(311)	(1.1)
	750	173	-	2.6×10^{-6}	(527)	(0.6)
	760	276	260	2.8×10^{-5}	(288)	(1.1)
	850	173	96	7.9×10^{-4}	404	9.4
	900	104	145	4.7×10^{-5}	(149)	(1.0)
	750	345	**	9.0×10^{-5}	**	**
	800	"	**	4.7×10^{-4}	**	**
	850	"	**	2.5×10^{-3}	**	**
1340-40:1	700	414	287	2.7×10^{-5}	(330)	(1.2)
	850	173	29.5	2.9×10^{-4}	154	13.7
	900	104	38	2.4×10^{-4}	(42)	(1.1)

* Most tests were interrupted prior to failure. The time and strain at interruption are shown in parentheses.

** One specimen held at constant stress with the temperature successively increased in order to determine the activation energy.

the stress exponent, n, and the activation energy, Q, using the relation:

$$\dot{\epsilon} = A \sigma^n \exp(-Q/RT) \quad (1)$$

Assuming that the step-wise increase in temperature at constant stress resulted in the true value of the minimum strain rate, the activation energy can be determined as shown in Figure 2. The value found, 400 KJ/mole, is slightly greater than the activation energy for the same alloy in the heat treated condition [8]. Determination of the stress exponent is hampered by the limited data in that only pairs of tests were conducted at constant temperature and different stresses. In the temperature range studied, 700 to 850°C, the estimated value of the stress exponent is in the range of 4 to 5, as noted for this alloy in the equilibrium condition [8].

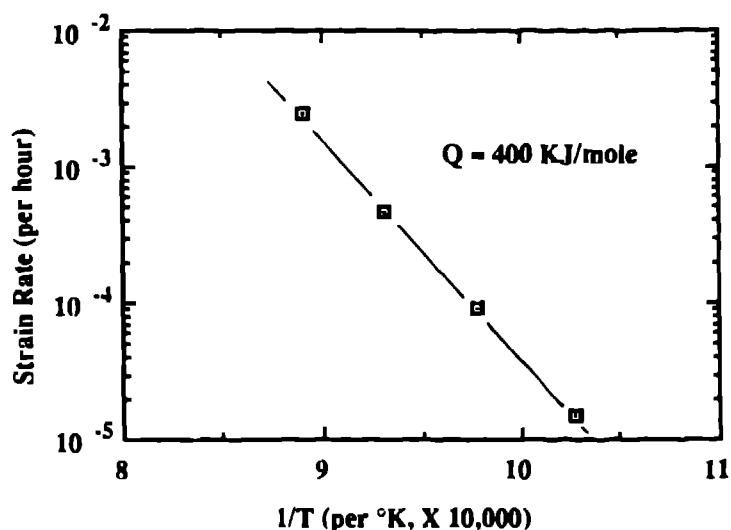


Figure 2. Creep activation energy for TiAl + W 'as-extruded'.

5.0 DISCUSSION

5.1 Tensile Deformation

Comparison of the yield and fracture strengths of the TiAl+W with the single phase γ reported earlier [3; 4], indicates that the W modified alloy has a higher yield strength. This may be due to either the modest microstructural refinement associated with W additions or the effects of solid solution W in the TiAl matrix. Microstructural refinement is unlikely to be the sole source of this strengthening. The impact of solid solution strengthening on flow stress at elevated temperatures is not usually considered to be large [10]. The major factor in the current case is the ordered nature of the matrix which may alter the importance of both microstructural scale and solid solution effects; limited fundamental work has been done on the additivity of these strengthening mechanisms in intermetallic compounds [10; 11].

The ductility of the TiAl+W alloy does not show the marked ductile-brittle transition observed in the binary TiAl studied previously [4]. This again may be due to the effect of the matrix being saturated in W which could be inhibiting elevated temperature dislocation motion. This observation is consistent with the implication of solid solution strengthening by W in the TiAl lattice being significant, even at relatively high temperatures.

5.2 Creep Deformation

The activation energy for this alloy, in the condition where the γ is supersaturated in W, is only slightly higher than that noted for the heat treated condition where the W has precipitated on internal surfaces [8]. In addition, the stress exponent (as determined by the minimum of only two data points)

is in the same range as noted in the previous study [8]. Since the minimum strain rates and $t_{1\%}$ values were significantly lower for the supersaturated condition, analysis using equation (1) to determine 'n' and 'Q' and therefore explain the increased creep resistance is unsatisfactory. The only parameter that is left in equation (1) to explain the difference in the conditions is the pre-exponential factor 'A'. It is possible that solid solution strengthening could have a linear impact on the strain rate without affecting the stress or temperature sensitivity. Extensive TEM examination of specimens following creep failed to show precipitation of W rich particles on dislocations. Precipitation on dislocations is usually evident in the dislocation strain field as a 'wavy' line [12; 13]. Figure 3 shows a typical γ grain having both ordinary and superdislocations following creep deformation and such contrast is not observed. Therefore, dynamic strain aging cannot be used to explain the strengthening observed in this alloy leaving solid solution strengthening as the only remaining mechanism to rationalize the creep resistance.



Figure 3. TEM bright field micrograph following creep at 700°C - 345 MPa.

6.0 CONCLUSIONS

From these results, we can conclude the following:

1. Microstructural refinement through thermal-mechanical process manipulation can increase the tensile strengths up to 700°C.
2. The addition of W to TiAl can lead to solid solution strengthening, as evidenced by both tensile and creep properties to 900°C, compared to a similar binary alloy.

3. The precipitation kinetics for formation of the W-rich precipitates is slow enough that long time exposure during creep deformation does not cause nucleation and growth of the equilibrium phase, even on heterogeneous sites such as dislocations.

7.0 REFERENCES

- [1] J. W. Kim, *J. of Metals*, **41**, (1989), p.24.
- [2] H. A. Lipsitt, in Ordered Intermetallic Compounds; editors C. C. Koch, C. T. Liu and N. S. Stoloff, Materials Research Society Vol. 39, 1985, p. 351.
- [3] D. Shectman, M. J. Blackburn and H. A. Lipsitt, *Metal. Trans.*, **5**, (1974), p.1373.
- [4] H. A. Lipsitt, D. Shechtman and R. E. Schafrik, *Metall. Trans.*, **6A**, (1975), p.1991.
- [5] R. E. Schafrik, *Metal. Trans.*, **8A**, (1977), p.1003.
- [6] T. Hanamura, R. Uemori and M. Tanino, *J. Mater. Res.*, **3**, (1988), p.656.
- [7] P. L. Martin, H. A. Lipsitt, N. T. Nufer and J. C. Williams, in Titanium 80. TMS-AIME, 1980, p.1245.
- [8] P. L. Martin, M. G. Mendiratta and H. A. Lipsitt, *Metall. Trans.*, **14A**, (1983), 2170.
- [9] Evans and Wilshire, Creep of Metals and Alloys, Institute of Metals, 1985.
- [10] R. L. Fleischer, D. M. Dimiduk and H. A. Lipsitt, "Intermetallic Compounds for Strong High-Temperature Materials: Status and Potential", *Annu. Rev. Mater. Sci.*, **19**, (1989), p.231.
- [11] A. J. Ardell, "Precipitation Hardening", *Metall. Trans.*, **16A**, (1985), p.2131.
- [12] P. L. Martin, D. H. Carter, R. M. Aikin, Sr., R. M. Aikin, Jr. and L. Christodoulou, "Creep Behavior of Alloys Based on TiAl Containing TiB₂ and TiN Particulates", to be published in, Proceedings of the Fourth International Conference on Creep and Fracture of Engineering Materials and Structures, Swansea, April 1990.
- [13] R. M. Allen and J. B. Vander Sande, "The Oriented Growth of Precipitates on Dislocations in Al-Zn-Mg Part I. Experimental Observations", *Acta Metall.*, **28**, (1980), p.1185.