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Influence of purity level on the microstructure and some properties of γ Ti-Al intermetallics

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ABSTRACT- The dependence of microstructure, hardness and electrical resistivity on the purity of TiAl alloys with 50, 53 and 56 at.% Al was investigated by comparing materials prepared from high-purity metals by clean processing methods, to materials prepared from commercial metals. Grain size and resistivity were found to depend both on purity and on Al content, whereas hardness varied little with the overall purity, in the investigated range.

INTRODUCTION

Among the intermetallic compounds, TiAl-based titanium aluminides are attractive for elevated-temperature structural applications. Above 50 at.% aluminium, these materials have a single-phase γ structure and are brittle; between 46 and 48 at.% aluminium, the alloys are two-phase ($\alpha_2 + \gamma$) and display some ductility, particularly when they have a purely lamellar structure [1]. The influence of the oxygen content on the ductility of these alloys has been previously investigated [2,3] and the results suggest that an oxygen content below 0.2 wt.% is necessary to keep a certain ductility.

In view of the importance of brittleness in these materials, we have investigated the effects of material purity on the properties of γ TiAl alloys by using high-purity starting materials and clean melting methods. Two series of alloys with a range of aluminium contents, one of commercial purity and one of high purity, were compared with respect to microstructure, to microhardness and to 4.2 K electrical resistivity.

PREPARATION OF MATERIALS

The investigated alloys were TiAl compounds with three different compositions. One of them corresponded to the γ phase (L1₀) stoichiometry: Ti₅₀Al₅₀; the two others were on the Al-rich side with 53 and 56 at.% Al. They were named TiAl₅₀, TiAl₅₃ and TiAl₅₆. Two series of alloys were prepared, depending on the purity of the starting metals. In the first one (CP), commercial titanium and double-electrolysed aluminium were used. The second series (HP) was prepared from very high purity metals: the level of oxygen was very low in titanium and the 99.9999 % aluminium was zone refined. The contents of the main impurities are given in table 1.

The alloys were prepared in two steps:

i) a RF levitation melting, with an exothermic dissolution reaction, followed by casting in a water-cooled copper crucible under pure helium atmosphere (He purity was checked by gas-chromatographic analysis; oxygen and nitrogen contents were ≈ 0.01 vpm). Melting, without contamination, was achieved in a short time (2 min); however solidification was very fast and generally led to a cracked ingot, due to the strong thermal strains.

ii) cracks were suppressed by remelting the ingot under helium gas, in the same conditions as above, by RF induction, on a water-cooled copper boat, and directionally solidifying.

Finally, the alloys were homogenized by annealing in a sealed silica tube back-filled with pure helium gas, for 24 hours in the temperature range 1100-1125°C. The ingots were wrapped in a 40 μ m thick tantalum foil to avoid contamination from silica.

Samples used for X-ray diffraction, micrographic observations, microhardness and electrical resistivity measurement were cut out from the ingots with a diamond saw.

Table 1. Main impurities (wt. ppm) of the starting metals for the commercial purity (CP) and high purity (HP) alloys.

Alloys	CP	HP
Titanium	Ti 40 (sponge) typical: O = 200 to 1200 ppm Fe = 100 to 1100 ppm Si = 50 to 100 ppm	Ti VA31 (refined by iodide process) typical: O = 27 ppm Fe = 107 ppm Zr = 95 ppm
Aluminium	Al 6174 (double-electrolysed) Fe = 3.3 ppm Mg = 17 ppm Si = 120 ppm Zn = 8 ppm	Al H3GS67 (zone-refined) Total: ≤ 1 ppm

MICROSTRUCTURE

Diamond polished sections of the three alloys of each purity level were examined by optical and scanning electron microscopy (SEM), in the as-cast or homogenized state.

In the as-cast condition, TiAl₅₀ showed extensive dendritic zones (figure 1-a); back-scattered electron (BSE) chemical contrast reveals a lamellar structure in these Ti-rich zones (figure 1-b). In TiAl₅₃, thinner Ti-rich dendrites are present (figure 1-c). Finally, in TiAl₅₆ large twinned grains are observed, with no BSE contrast (figure 1-d).

Figure 2 : Example of grain structure of homogenized alloys. Polarised light optical image of HP TiAl₅₀.

In the homogenized state (figure 2), no chemical heterogeneity was detected in any of the alloys. The structure is a twinned equiaxed one, with a grain size increasing with the Al content and with the purity of the material. X-ray diffraction showed that TiAl₅₆ was single-phase γ ; very weak Ti₃Al lines were detected in the low-Al samples.

CHEMICAL ANALYSIS

Polished sections were microprobe analysed by energy dispersive X-ray spectroscopy (EDX), using pure elements standards (Ti VA and zone melted Al). Several sets of standards spectra (2 to 6 in a day) were used for determining concentrations, to take into account the variations of the beam current, and results were averaged. A number of spot analyses were taken on each sample, either randomly for homogenized samples or on each contrast area for as-cast samples. The results were averaged for each area and the estimated standard deviations compared with the intrinsic standard deviation of the method, measured in the same conditions with the same sets of standards (0.1 to 0.25 at.%). It was thus possible to assess chemical heterogeneity. General results for homogeneous samples are presented in table 2 together with results of bulk analyses by atomic emission spectroscopy excited by inductively coupled plasma (ICP-AES): it can be seen that CP and HP TiAl₅₆ are homogeneous in the as-cast as well as homogenized states whereas homogenized CP and HP TiAl₅₀ and TiAl₅₃ present standard deviations slightly larger than that of the method, indicating some concentration variation. CP and HP as-cast TiAl₅₀ and TiAl₅₃ are heterogeneous and some results are given in figure 1: chemical heterogeneity seems stronger in TiAl₅₃.

Table 2. Aluminium content (at.%) of the two series of alloys determined by SEM-EDX. (s is the estimated standard deviation and N the number of spot analyses).

Alloys	TiAl ₅₀		TiAl ₅₃		TiAl ₅₆	
	CP	HP	CP	HP	CP	HP
As-cast	Heterogeneous	Heterogeneous	Heterogeneous	Heterogeneous	56.1 s = 0.13 N = 18	55.0 s = 0.3 N = 31
Homogenised	50.0 s = 0.7 N = 22 (49.9 from ICP-AES)	50.0 s = 1.1 N = 48	53.1 s = 0.7 N = 18 (53.1 from ICP-AES)	52.6 s = 0.5 N = 34	56.1 s = 0.25 N = 30 (56.1 from ICP-AES)	55.7 s = 0.25 N = 28

MICROHARDNESS

The effect of the homogenization treatment was investigated as a function of purity level and of composition (figure 3). Vickers microhardness was measured under a 100 g load. Figure 3 shows that:

- i) hardness increases with aluminium content.
- ii) homogenization decreases strongly hardness of the stoichiometric TiAl₅₀ alloy and is less effective in the Al-rich alloys.
- iii) there is no strong effect of the overall purity in the investigated range of impurity level; however, the tendency would be a small decrease of hardness with increasing purity of the materials.

The microhardness determined by Kawabata et al. [3] on Ti-Al alloys of the same composition as the present ones and containing ≈ 300 wt. ppm of oxygen are included in figure 3. Their values are slightly lower than ours; however no firm conclusion can be deduced from this comparison, since the load used in their measurements was not specified (microhardness is lower under higher loads: compare values under 100 and 300 g for TiAl₅₀ in figure 3, right).

ELECTRICAL RESISTIVITY

Residual resistivity of the alloys was determined from DC electrical resistance measurements performed at 4.2 K on rod-shaped samples (1×1×10 mm³). After cutting out from the ingot, the samples were annealed for 10 hours at 950 or 1000 °C under a 10⁻⁶ Pa vacuum and furnace cooled. Figure 4 shows the electrical resistivity evolution as a function of composition in the two series of alloys:

- i) residual electrical resistivity increases with the impurity content in TiAl₅₀ and TiAl₅₃. However no change was detected in the Al rich TiAl₅₆ alloy. This material is very brittle and the

possible contribution of microcracks to the resistivity could be more important than the contribution of residual impurities.

ii) residual resistivity is lowest ($\approx 8 \mu\Omega\cdot\text{cm}$) in HP stoichiometric TiAl_{50} and strongly increases with deviation from stoichiometry (up to $78 \mu\Omega\cdot\text{cm}$) in the Al-rich alloy. The same trend has also been observed in Ni_3Al compounds [4-5].

Figure 3. Vickers microhardness (under 100 g) as a function of composition in CP (open symbols) and HP (full symbols) Ti-Al in two structural states: as-cast (left) and homogenized at 1100 - 1125 °C (right).

Figure 4. Variation of the residual electrical resistivity (determined at 4.2 K) as a function of Al content in the CP and HP Ti-Al alloys.

CONCLUSION

Ti-Al alloys with 50, 53 or 56 at% Al have been prepared from commercial purity or from high-purity metals.

In the as-cast condition, the volume fraction of Ti-rich dendrites decreased with increasing Al contents, and vanished in the 56 at.% Al alloy. The chemical heterogeneity also decreased for the higher Al content. After homogenizing at 1100°C, all samples displayed a structure of twinned nearly-equiaxed grains, the size of which increased with Al concentration and purity level. X-ray diffraction detected the presence of a small amount of α_2 phase in the low-Al alloys.

Vickers microhardness increased in all cases with the Al content. Homogenizing resulted in an important decrease of hardness of the Ti- 50 at.% Al alloy, probably connected with the removal of the lamellar structure. No significant influence of impurity content on hardness was detected, in the investigated purity range.

Low-temperature electrical resistivity increased with Al concentration (i.e. with the deviation from TiAl stoichiometry); it also increased with the impurity content of the samples.

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