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SINTERABILITY OF MECHANICALLY ALLOYED Ti-37.5Si (at.%) POWDERS COATED WITH A METALLIC THIN LAYER BY SPUTTERING

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Abstract. In this work, a Ti-37.5Si (at.%) alloy synthesized by mechanical alloying from Ti and Si powders was coated with Al by magnetron sputtering followed by hot isostatic pressing. The mechanically alloyed powders consisted of a Ti_5Si_3 intermetallic phase. Al-coating played a significant role in the final properties of the compacts. The $TiAl_3$ phase formed on surface of the coated powders during compaction led to an increase of their sinterability. Values of 21.2 GPa and 5.9 $MPam^{1/2}$ were obtained for hardness and fracture-toughness of the coated sample, which are twice-time larger than the values determined for uncoated sample.

1. INTRODUCTION

In recent years, the research on Ti-based alloys has been specially focused on intermetallics, such as titanium aluminides and titanium silicides. These materials show considerable potential for structural applications. In fact, they have higher corrosion resistance and tensile strength at high temperatures than the traditional titanium alloys. However, their use in industrial applications has been limited mainly due to the low ductility and fracture-toughness at room-temperature. Recently, we have proposed a new method for the production of bulk intermetallics with improved mechanical properties from surface modified mechanically alloyed (MA'ed) powders [1]. In this method, elemental powders are mechanically alloyed and subsequently coated with a ductile thin layer by Physical Vapor Deposition (PVD), before final hot isostatic pressing (HIP). In the present work, Ti-37.5Si (at.%) powders synthesized by mechanical alloying and subsequently coated with Al by magnetron sputtering, were compacted by hot isostatic pressing. The sinterability and mechanical properties of the coated and uncoated samples are compared and discussed.

2. EXPERIMENTAL

Magnetron sputtering was used to deposit an Al thin layer on the surface of Ti-37.5Si (at.%) powders synthesized by mechanical alloying in a previous work [2]. The surface modified powders (Ti-37.5Si + Al sample designation) were consolidated by hot isostatic pressing according to the procedure described elsewhere [1].

Particle size distributions of powder mixture were determined by laser scattering (Cilas 1064 equipment). After consolidation, the cylindrical bars were cut, grounded and polished with diamond down to 3 μm. The microstructure of the hot compacted samples was analyzed by X-ray diffraction (XRD) with Co K_α radiation and scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS). Microhardness tests were

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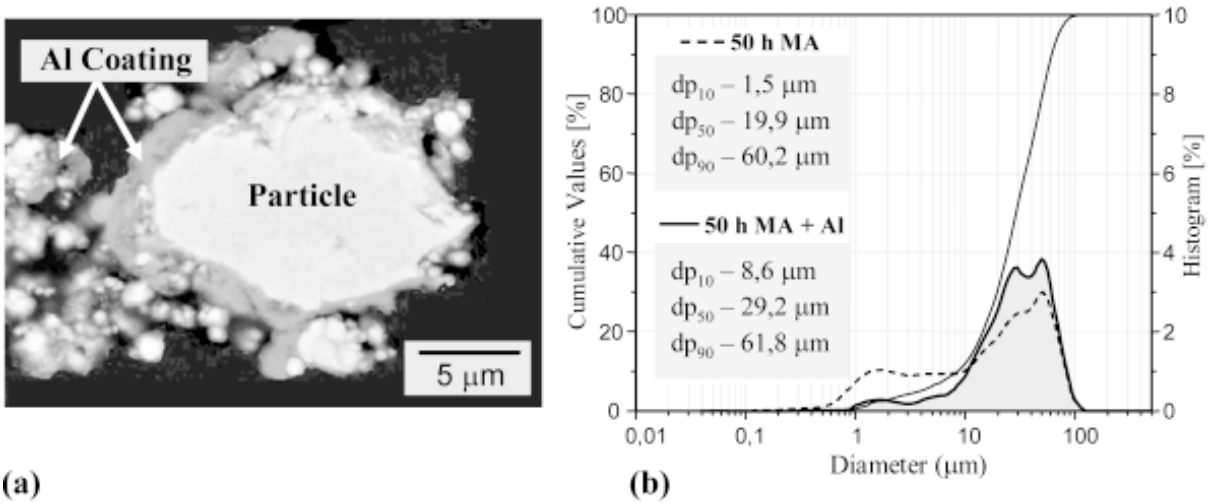

Fig. 1. – (a) Cross section of Ti-37.5Si coated particle. (b) Particle-size distributions of 50 h uncoated MA´ed powders (**- - -**) and MA´ed powders coated with Al (———).

carried out on flat polished surfaces using a Vickers diamond-indenter with a load of 245 mN. Indentation fracture-toughness parameter K_c of the compacts was also determined by Vickers method with a load of 49 N using the Evans *et al.* equation [3]. The open porosity values of consolidated samples were determined by mercury porosimetry, using a Micromeritics 9320 equipment.

3. RESULTS AND DISCUSSION

Fig. 1a shows a SEM image of an Al coated Ti-37.5Si particle. It is possible to distinguish the particle core and the coating layer. However, a rather poor coating uniformity is observed as a result of (i) wide particle size distribution, (ii) existence of fine particles which tend to agglomerate during deposition and (iii) particles with no spherical shape.

Comparing the particle size distributions of coated and uncoated powders (Fig. 1b), it is possible to conclude that a considerable decrease of number of particles with smaller diameter and an increase of particles with larger diameter occurred during the deposition process, probably due to particles agglomeration.

XRD analysis performed for the coated powders (Fig. 2) reveals a Ti_5Si_3 intermetallic phase and a fcc-Al phase, which are ascribed to the MA´ed powders and the coating layer, respectively.

Fig. 3 shows SEM images of the morphology of the HIP compacted samples. EDS analysis performed at different points of the Ti-37,5Si + Al sample (Fig. 3b) allowed to conclude that the white areas correspond to the MA´ed particles (Ti-37.5Si), while the grey areas are ascribed to the coating layer.

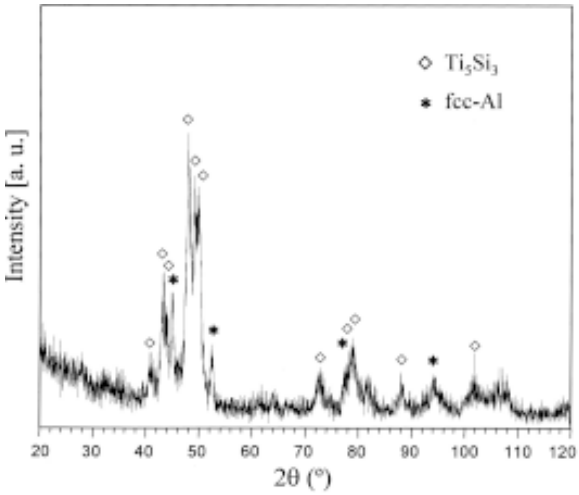

Fig. 2. – XRD pattern of the MA´ed powders coated with Al.

A detailed analysis performed in different zones of the samples showed that pores and microcraks exist in both samples, in particular in the uncoated one. As referred by Hakamoto [4], many problems are encountered during the compaction of Ti_5Si_3 intermetallic bulk materials. The development of microcraks can arise from strain development during consolidation and anisotropy of the Ti_5Si_3 thermal expansion coefficient [5].

Table 1. Properties of uncoated and Al-coated consolidated samples.

Sample	Open porosity (%)	Hardness (GPa)	Fracture-toughness ($MPam^{1/2}$)
Ti-37.5Si (at.%)	16	12.8 ± 0.9	2.9 ± 0.6
Ti-37.5Si + Al (at.%)	9	21.2 ± 0.8	5.9 ± 0.4

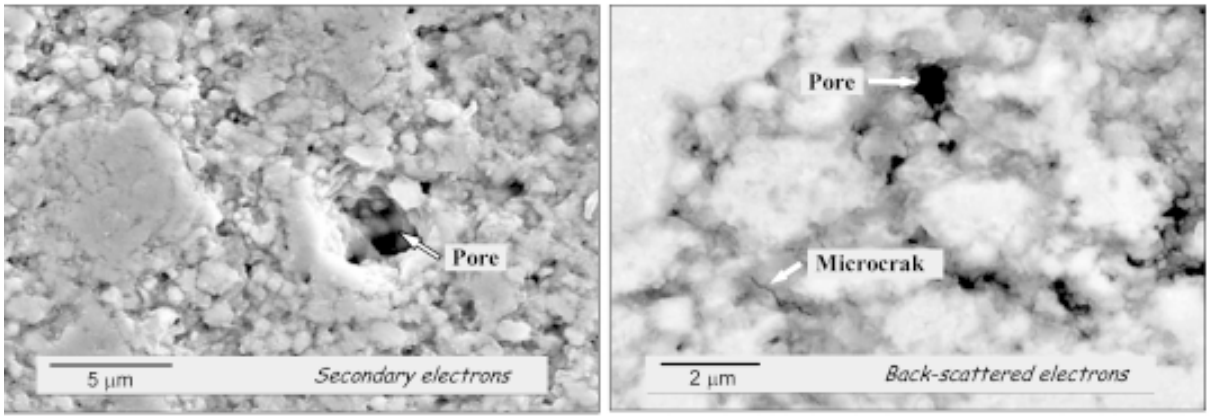

Fig. 3. – Morphology of HIP consolidated samples: (a) Uncoated sample (b) Coated sample.

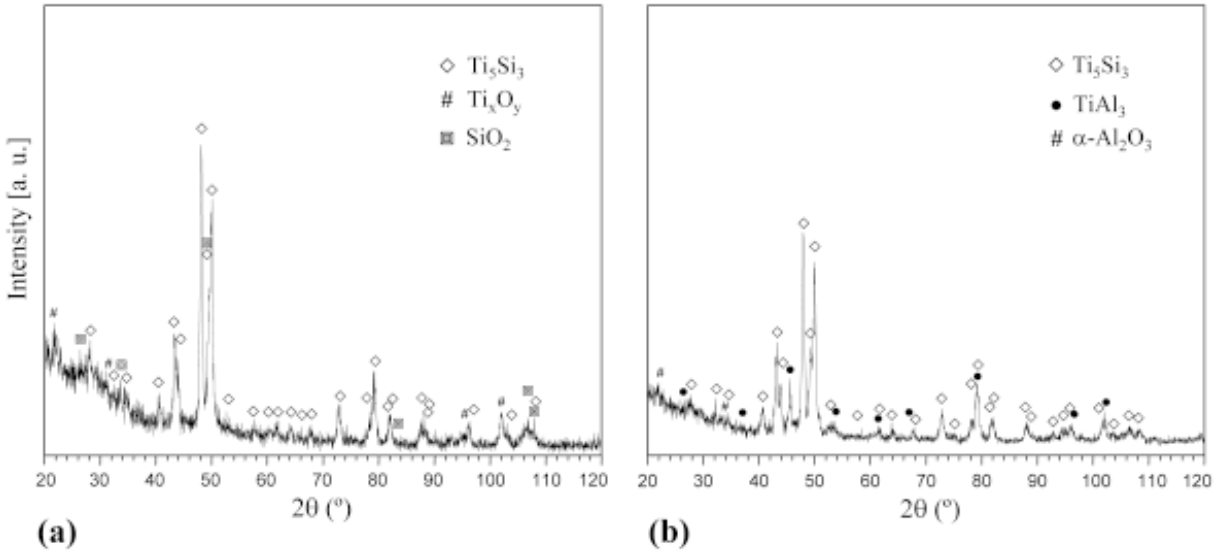

Fig. 4. – XRD patterns of the consolidated samples: (a) uncoated sample, (b) coated sample.

Fig. 4 shows the XRD patterns of the compacted samples. Ti-37.5Si compact is mainly formed by Ti_5Si_3 intermetallic phase and vestiges of Ti-oxides and SiO_2. The incorporation of oxygen in Ti-based systems is a well known problem [6] and it is difficult to prevent, in particular when MA is used for their production from small size powders. Ti-37.5Si + Al compact is formed by Ti_5Si_3 and $TiAl_3$ intermetallic phases and vestige of α-Al_2O_3. Ti_5Si_3 phase is present in MA´ed powders core, while $TiAl_3$ was formed during compaction from the reaction of Ti and Al coating. It is conceivable that residual Ti (α-Ti phase) might exist after mechanically alloying, which was not confirmed by XRD analysis. The

stronger diffraction lines of α-Ti and Ti_5Si_3 phases are positioned at 2θ = 47° and 47.83°, respectively, which turned the analysis difficult to perform.

The open porosity, hardness and fracture-toughness values of the compacted samples are present in Table 1. The uncoated sample (Ti-37.5Si) has a relative high open porosity of 16% confirming the low sinterability of the Ti_5Si_3 intermetallic powder. The Al coating led to a significant increase of sinterability of the powders as, for the same consolidation parameters, the open porosity of the Ti_5Si_3 + Al sample compact is of 9%. This fact can be explained by high plasticity of the Al-layer. At the beginning of the HIP cycle (T < 600 °C), Al exists as an fcc elemental phase [2] and, as the result of its high ductility, it flows into the open cavities between the particles. When the temperature reaches 600ºC, Al coating gives rise to the $TiAl_3$ intermetallic phase and, therefore, the plasticity of the coating layer decreases. However, as was observed, this phase also contributed to the improvement of sinterability of the Ti_5Si_3 powders, which can be explained by its lower hardness [7] and melting point (1342 °C against 2124 °C, for the Ti_3Al and Ti_5Si_3 phases, respectively).

A hardness value of 12.8 GPa was obtained for the Ti-37.5Si sample which is consistent with its phase composition and microstructure. Moreover, this value is within the range reported in the literature for Ti_5Si_3 bulk samples with microcracks [5]. A significant hardness increase was observed for the Al-coated sample. Since the Ti_5Si_3 phase is harder than the $TiAl_3$ phase, this hardness increase can be explained by the porosity decrease and/or by reduction of microcracks density. Moreover, chemical reaction in layered powder might also contribute for this phenomenon as inter-particle bounding in two-phase sinter can be stronger than in on-phase sample.

Fracture-toughness of the uncoated sample (K_c= 2.9 MPam$^{1/2}$) can be considered low for industrial applications. Nevertheless, this value is of the same magnitude of the ones determined in other studies for intermetallic materials produced by similar methods [8]. The fracture-toughness of the Ti-37.5Si + Al sample (K_c= 5.9 MPam$^{1/2}$) is significantly higher than the one of the uncoated sample which might be explained by the $TiAl_3$ interface among the coated particles, less oxygen contamination and lower open porosity.

4. CONCLUSIONS

In this work it was demonstrated that the surface modification of mechanically alloyed Ti-37.5Si powders by the deposition of an Al coating prior compaction improved the sinterability of the Ti_5Si_3 intermetallic phase. The coating acted as an activating sintering element, reducing the size and number of inter-particle's pores and microcracks. Therefore, better mechanical properties were obtained for the Ti-37.5 + Al compact sample.

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