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Structural- and mechanical-properties of hot-pressed surface modified Ti_xSi_{100-x} ($x = 62.5$ and 85 at.%) powders synthesized by mechanical alloying

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Abstract

In this paper, we are describing a new procedure for the synthesis of metallic- and intermetallic-matrix composites from powders obtained by mechanical alloying, subsequently coated by magnetron sputtering and compacted by hot isostatic pressing. Mixtures of $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ (numbers indicate at.%) were mechanically-alloyed from elemental titanium and silicon powders in a planetary ball-mill for 50 h and subsequently coated with a titanium thin film. The presence of a metallic layer on the surface of the powders affects the mechanical properties of the hot-compacted samples. The Ti thin film increases the Young modulus and fracture-toughness of the $Ti_{85}Si_{15}$ samples; this effect is not seen in the $Ti_{62.5}Si_{37.5}$ alloy. Hardness is not significantly affected. In the case of the $Ti_{62.5}Si_{37.5}$ alloy, partial oxidation of the coating was observed during the synthesis procedure; this procedure is responsible for the increase in hardness, in the Young's modulus and in the decrease in fracture-toughness.

Keywords: Ti-matrix composites; Ti_5Si_3; Mechanical alloying; Indentation mechanical properties

1. Introduction

Traditional titanium alloys have been used since 1960s in aerospace applications because they are lightweight, corrosion-resistant and have good mechanical properties. However, in recent years, there has been considerable interest in the development of new advanced high-temperature materials for structural applications, mainly in the automotive and aerospace industries. A case in point is titanium-matrix composites (TMCs), which consist of a matrix of titanium or a titanium solid solution, reinforced with ceramic fibres, such as TiC, TiB_2, SiC or Al_2O_3 [1,2]. These alloys have similar density but higher strength and stiffness than the unreinforced Ti alloys. However, their ductility is a major concern, amounting to only about 2%. Moreover, they are not particularly attractive in biaxial loading situations because of the reduced properties when loaded perpendicular to the fiber axis. This disadvantage can be overcome by the use of other processing techniques such as powder metallurgy, rapid solidification, vapour deposition and mechanical alloying (MA) in order to produce isotropic nanocrystalline TMCs with improved mechanical properties. Recently, there has been a great interest in the development of new materials for very high temperature structural applications, especially those based on silicides, e.g. Ti_5Si_3. Once again, the ductility and fracture-toughness of these materials are main problems. Refinement of grain size [3], addition of ternary alloying elements [4] and the development of multi-phase materials [5,6] have been used to improve these properties with rather successful results.

Although MA has been used since long for the production of Ti- and Ti_5Si_3-matrix composites reinforced with ceramic and intermetallic particles, the consolidation of the mixtures is one of the crucial steps to obtaining high-performance materials.

In a recent paper, MA was used for the synthesis of Ti–Mg–Si lightweight alloys [7]. Depending on the chemical composition, nanocrystalline Ti-based alloys reinforced

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with Ti_5Si_3 particles, and $Ti_5Si_3 + Mg_2Si$ intermetallic alloys were obtained. However, the temperature required for a good hot compaction (e.g. hot isostatic pressing (HIP)) of these materials is relatively high, mainly due to the high melting point of the ordered structures. The incorporation of a ductile phase with a low melting point in the grain boundaries of the mechanically-alloyed powders may enhance their consolidation ability even at lower temperatures. In a previous work on the compaction of $Ti_{80}Al_{20}$ and $Ti_{85}Si_{15}$ (numbers indicate average concentration in at.%) powders coated with aluminium [8], it was shown that the use of magnetron sputtering to coat mechanically-alloyed powders is a promising process for good subsequent hot compaction. However, during heating, it was observed that aluminium diffused into the core of the powders leading to the formation of new structural phases. This was explained by the existence of free titanium in the powders which reacted with the aluminium to form Ti aluminides.

In this study, titanium and silicon powders were mechanically-alloyed and subsequently coated with Ti by magnetron sputtering. Hot isostatic pressure was used to consolidate the coated powders in order to obtain dense bulk samples. The aim of the present work is to study the influence of the Ti coating layer on the compaction and mechanical properties of Ti–Si mixtures in order to synthesize high-performance metallic- or intermetallic-matrix composites with increased ductility and toughness. Special attention is focused on the determination of the mechanical properties of the compacted samples, i.e. hardness, Young modulus and fracture-toughness.

2. Experimental details

Powders of Ti (99.5% purity) and Si (99.0% purity) with maximum particles sizes of 100 and 45 μm, respectively, were used as starting materials to obtain mixtures of $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$. The mixtures were synthesized by mechanical-alloying in a planetary ball-mill as described elsewhere [8]. The $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ samples milled for 50 h were subsequently coated with Ti by d.c. magnetron sputtering, with a specific discharge power of 2.62×10^{-2} W/mm^2 for 9 h. The depositions were performed in a pure argon atmosphere (5×10^{-3} Pa) after the chamber evacuation down to a base pressure of 10^{-6} Pa. During the depositions, the powders were continually shaken by vibration and translation movements in order to obtain homogeneous coatings. The powders produced by MA with and without Ti-coating were cold isostatic pressed at 320 MPa and then encapsulated in steel cans lined with Ta foil, degassed at 300 °C for 60 min and sealed. The selected HIP cycle (Fig. 1) consisted of a cold pressurization to about 70 MPa, followed by continuous heating (10 K/min) and pressing (2 MPa/min). The samples were consolidated at a maximum pressure of 150 MPa for 2 h. Cooling and decompression of the vessel were performed simultaneously. After

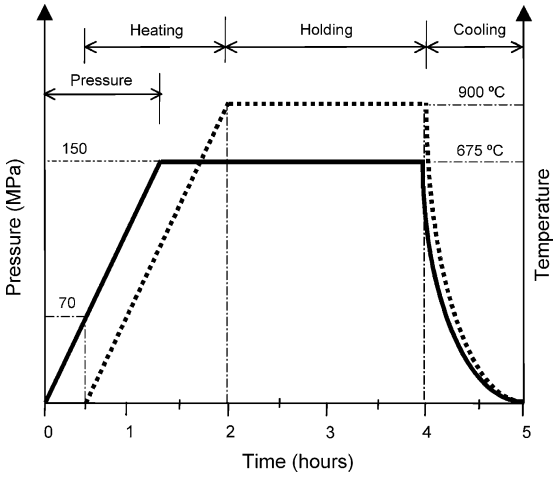

Fig. 1. Pressure and temperature stages used in the HIP cycle (—pressure, · · · temperature).

consolidation, the cylindrical bars were cut, grounded and polished with diamond down to 3 μm. The characteristics of milled powders and hot consolidated samples were determined by the following techniques: X-ray diffraction (XRD) with Co K_α radiation, differential scanning calorimetry (DSC) applying a heating rate of 40 K/min and scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) analysis. The Williamson–Hall method [9] was used to determine grain size of the phases formed during milling and after subsequent heating. This method considers that the final width of an X-ray line is the result of its half-height intensity, the residual strain broadening and the instrumental broadening associated with the X-ray spectrometer. The indented Young modulus was calculated with a 245 mN load, according to the method described in [10]. Ultramicrohardness measurements were carried out on flat polished surfaces using a Vickers diamond-indenter under the same loading conditions. The fracture-toughness K_c was also determined by Vickers indentation but with a 49 N load using the "halfpenny" and Palmqvist crack systems and the Evans et al. and Shetty et al. equations, respectively [11].

Particle size distributions were determined by laser scattering (Cilas 1064 equipment) from a powder suspension in water under mechanical agitation after a 60-s sonication. Sample density was measured according to the Archimedes principle method [12].

3. Results and discussion

3.1. Characterization of the mechanically-alloyed powders

Fig. 2 shows the structural evolution of the $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ mixtures during mechanical alloying. The results show that after 50 h of milling, the $Ti_{62.5}Si_{37.5}$ alloy is mainly composed of the intermetallic Ti_5Si_3 (Fig. 2a) with

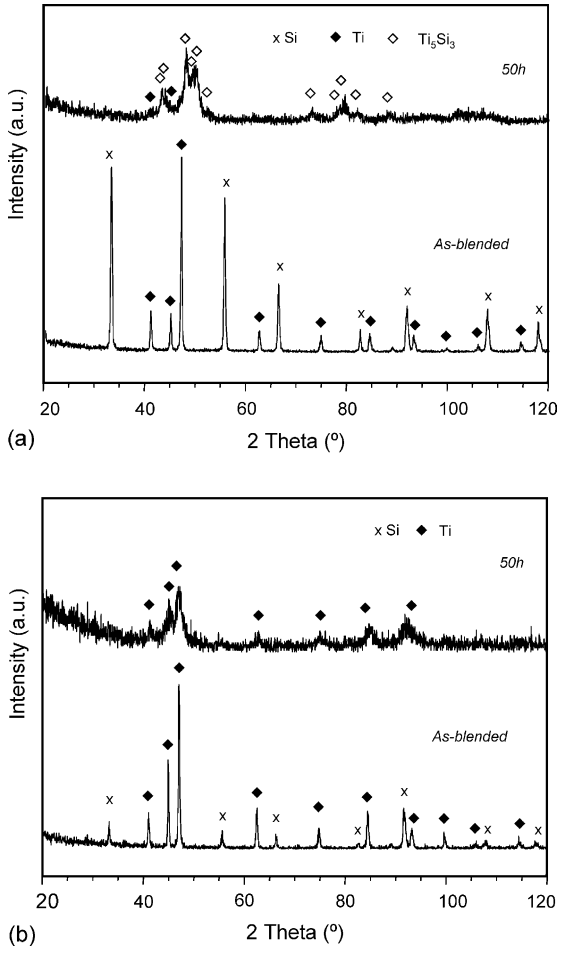

Fig. 2. XRD patterns of the (a) $Ti_{62.5}Si_{37.5}$ and (b) $Ti_{85}Si_{15}$ mixtures as a function of milling time.

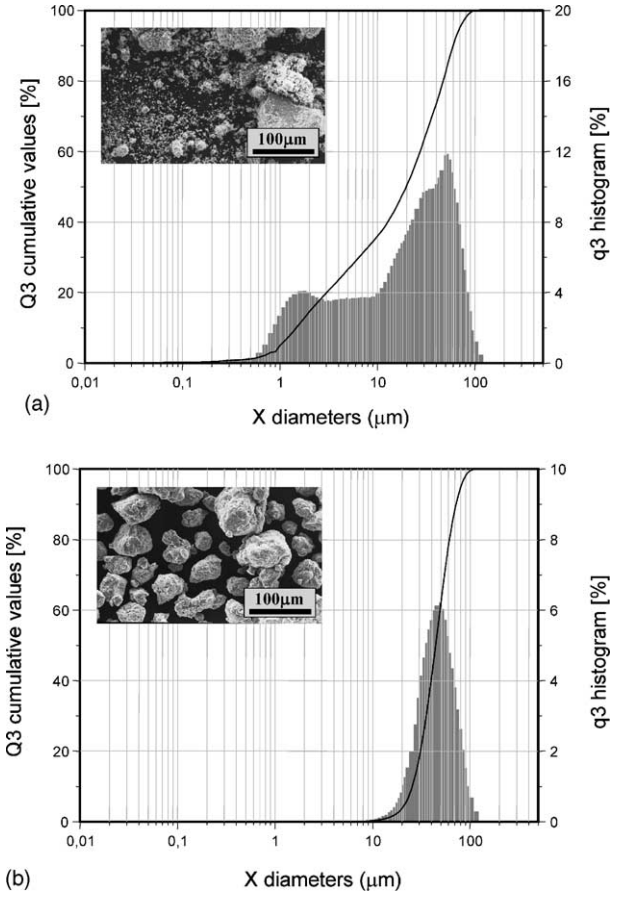

Fig. 3. Particle-size distributions of mixtures (a) $Ti_{62.5}Si_{37}$ and (b) $Ti_{85}Si_{15}$ milled for 50 h.

a grain size of about 8 nm and lattice parameters *a* and *c* of 7.433 Å and 5.140 Å, respectively. These values are slightly lower than the ones indicated in the ICDD card for this phase [13]. Vestiges of α-Ti could also be detected in this sample after milling. The $Ti_{85}Si_{15}$ alloy is composed of α-Ti (Fig. 2b). No Si-diffraction peaks were observed in the diffraction pattern of the $Ti_{85}Si_{15}$ sample milled for 50 h, which means that major portions of this element might be in substitutional positions in the hcp-lattice of titanium. The lattice parameters of this phase were calculated after 50 h milling. The values obtained for *a* and *c* (2.943 Å and 4.666 Å, respectively) are slightly lower than the ones indicated in the ICDD card for the α-Ti phase **(**2.951 Å and 4.683 Å, respectively [14]). Shrinkage of the lattice is in accordance with the substitution of Ti (at. radius = 1.47 Å) by Si (at. radius = 1.17 Å). As with the Ti_5Si_3 phase, the final α-Ti phase is nanometric with a grain size of 17 nm.

The particle size distributions of the mechanically-alloyed Ti_xSi_{100-x} ($x = 62.5$ and 85 at.%) mixtures are shown in Fig. 3. As can be seen, the $Ti_{85}Si_{15}$ exhibits a unimodal distribution ranging from 6 to 125 µm, whereas $Ti_{62.5}Si_{37.5}$ presents a bimodal distribution in the range of 0.1–125 µm and a considerable amount of fine particles. In fact, the elements Ti and Si have different ductilities, Ti–Si being considered a ductile–brittle system. During the early stages of milling, the ductile Ti powders are flattened and covered with finely crushed brittle Si powders, resulting in a continuous refinement of the particles by fracturing. However, the higher the titanium percentage, the higher the ductility of the mixture, and consequently, the lesser intensified fracturing process, thus leading to larger final particle sizes. However, after at a certain time, no further refinement of particles occurred and both alloys attained a steady-state equilibrium.

3.2. Thermal behavior of coated and uncoated mechanically-alloyed powders

After milling, a portion of each powder was coated with titanium by magnetron sputtering and their thermal behavior analyzed at increasing temperatures up to 1000 °C. The coated and uncoated powders were submitted to DSC runs followed by XRD-analysis at room temperature after cooling. The results obtained from the $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ samples are presented in Fig. 4a–d. Some small exothermic peaks could be detected in the DSC curve of the $Ti_{62.5}Si_{37.5}$

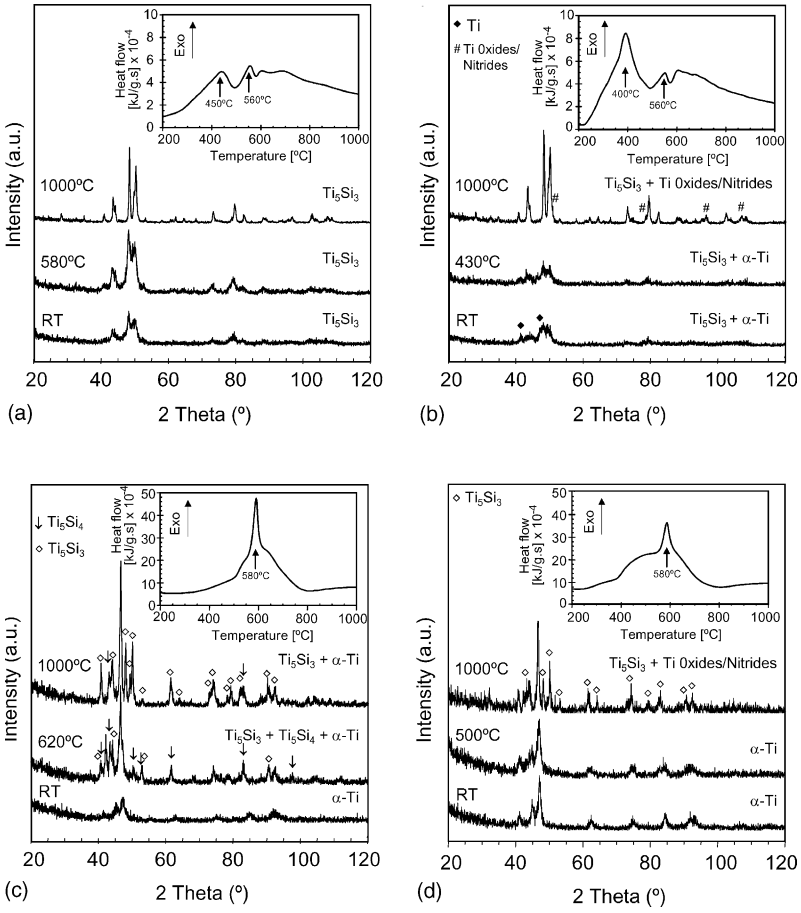

Fig. 4. DSC curves and XRD patterns of the (a) $Ti_{62.5}Si_{37.5}$ (b) $Ti_{62.5}Si_{37.5}$ + Ti, (c) $Ti_{85}Si_{15}$ and (d) $Ti_{85}Si_{15}$ + Ti mixtures as a function of temperature.

alloy with no titanium coating (Fig. 4a). These peaks are not ascribed to any phase transformation. In fact, no other peaks besides those of the Ti_5Si_3 intermetallics were detected in the XRD-diffraction patterns of this sample after milling and subsequent heating up to 1000 °C. The only feature to notice is the grain-size increase of the intermetallic phase, in particular for 1000 °C. Therefore, the exothermic peaks must be ascribed to structural and stress relaxation of the milled samples as reported by other authors [15,16], and grain size increase in the intermetallic phase.

If one compares the DSC curves obtained for the coated and uncoated samples of this system, the only differences to report are the shift of the exotherm for lower temperatures and the increase in its intensity (Fig. 4b). Once more, this DSC peak has no correspondence to any phase transformation and it must be the result of the already-mentioned structural and stress relaxation of the milled powders, as well as the presence of the Ti coating. Moreover, it is quite conceivable that there is a stress release at the powders/coating interface during heat treatment, the magnitude at a first sight seems to be of minor importance, since the powder/coating interface is several orders of magnitude smaller than the grain boundary area. At 430 °C, the Ti-diffraction peaks are still present in the XRD pattern of the coated $Ti_{62.5}Si_{37.5}$ sample, which means that Ti did not react with the Ti_5Si_3 phase to give rise to other intermetallic phases. After 1000 °C, some peaks emerged, probably associated to the formation of Ti-oxides and/or Ti-nitrides. The coated and uncoated $Ti_{62.5}Si_{37.5}$ samples heated up to 1000 °C are formed by Ti_5Si_3 + Ti-oxides/nitrides and Ti_5Si_3, respectively, which indicates that the Ti coating tends to be oxidized/nitrided at high temperatures. The presence of oxygen/nitrogen is probably due to the level of atmospheric control during MA. Although the vials were filled with hydrogenated argon, complete protection from the atmosphere is difficult to achieve. The results obtained for the $Ti_{85}Si_{15}$ samples with and without coating do not vary significantly. In both cases, the DSC curves revealed an exothermic peak close to 580 °C due to the formation of the Ti_5Si_3 intermetallic from the Ti(Si) solid solution (Fig. 4c and d). Additionally, a broad exothermic peak between 400 and 800 °C is also visible in both DSC curves which might be the result of stress relaxation and grain growth, as mentioned before. The final structure of these samples after heating up to 1000 °C is composed by Ti_5Si_3 and Ti oxides/nitrides in the case of the coated sample. Vestiges of these oxides/nitrides could also be detected at relatively low temperatures, 500 °C in the $Ti_{85}Si_{15}$ samples. It should be pointed out that these samples are richer in titanium than the ones of the $Ti_{62.5}Si_{37.5}$ alloy which might

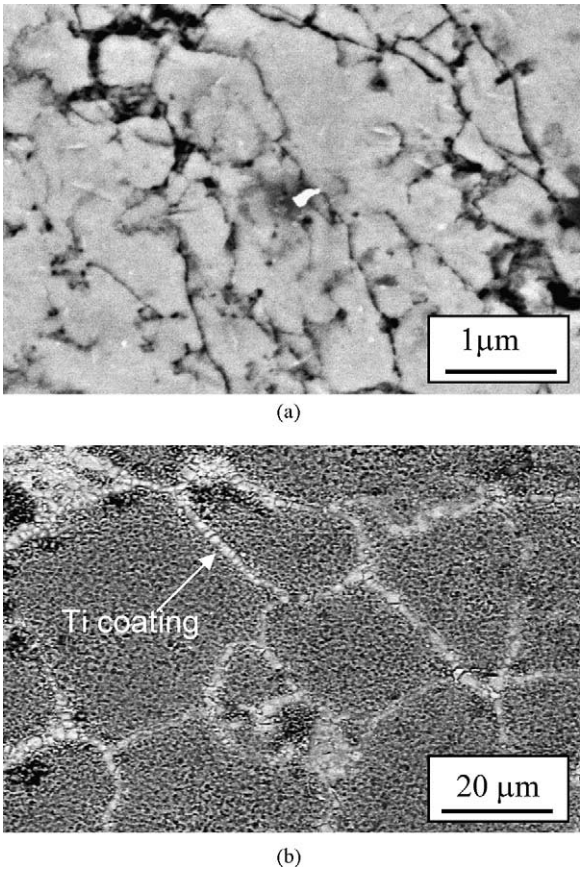

Fig. 5. Back-scattered electron SEM images of Ti-coated (a) $Ti_{62.5}Si_{37.5}$ and (b) $Ti_{85}Si_{15}$ samples.

explain the more intense oxide/nitride peaks observed in this case.

3.3. Hot isostatic pressing consolidation

Four samples were submitted to HIP (one sealed capsule of each mechanically-alloyed sample with and without Ti coating). Fig. 5a and b show SEM images obtained from the consolidated coated samples. As can be seen, the $Ti_{62.5}Si_{37.5}$ sample (Fig. 5a) consists of grains with different sizes and unclear interfaces, whilst the $Ti_{85}Si_{15}$ sample (Fig. 5b) is composed of bigger grains of some tens of micrometers and broad interfaces ($\approx 2\,\mu m$) (see Fig. 6a). Fig. 6b shows the results of the EDS analysis performed at the interface and powder core of the hot-pressed $Ti_{85}Si_{15}$ sample. The signal obtained at the interface arises from titanium, meaning that during heating, there was no diffusion from the powder to the coating. In the powder core, peaks from Ti and Si were obtained in accordance with the chemical composition of the powders.

During HIP, there was grain growth of the mechanically-alloyed phases. However, the nanometric size of the intermetallic phase is maintained during consolidation as a result of the high level of dislocations density, existing in the mechanically-alloyed powders together with the high

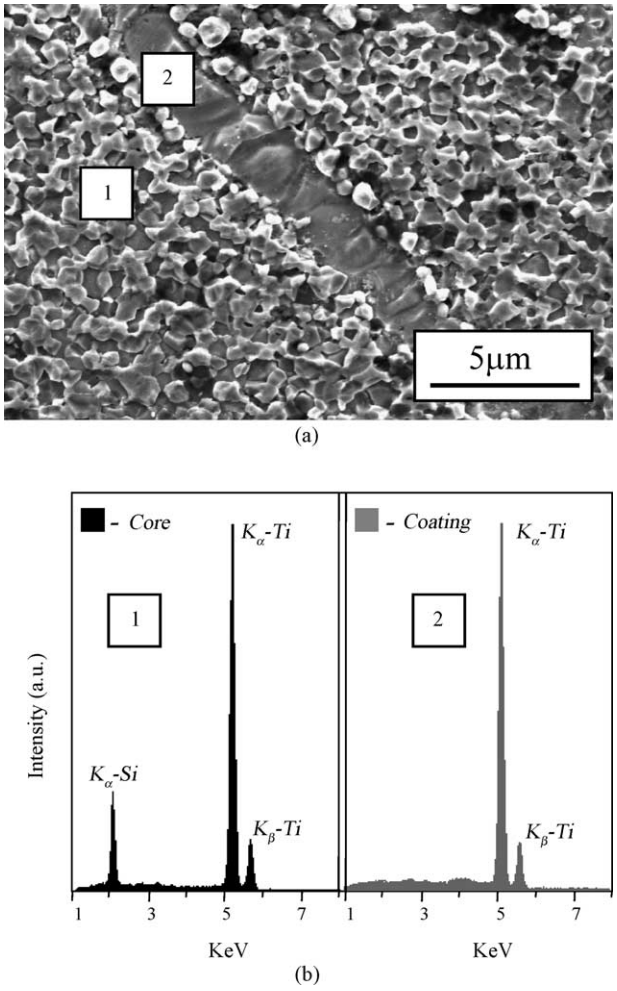

Fig. 6. Compacted $Ti_{85}Si_{15}$ grains with Ti at the interface. (a) Secondary electrons SEM image, (b) EDS analysis.

pressure and relative low temperature used in HIP. After consolidation, the grain size of the Ti_5Si_3 phase of the $Ti_{85}Si_{15}$ samples (with and without coating) is lower than the grain size of the $Ti_{62.5}Si_{37.5}$ sample. This can be explained by the fact that in case of the former system, the Ti_5Si_3 intermetallic emerges during heating (Fig. 4c and d), while in the case of $Ti_{62.5}Si_{37.5}$, the intermetallic is formed during MA (Fig. 4a and b), i.e. already exists before the consolidation process.

3.4. Indentation mechanical properties

Hardness, Young modulus and fracture-toughness of the hot-compacted samples were determined by hardness tests. The results are presented in Table 1. Both measured and theoretical density values of the samples are also listed in this table. The theoretical densities were calculated, taking into account the respective overall composition and phases of each sample. Therefore, the percentages of theoretical density achieved during consolidation of the $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ samples are of 91.8% and 99.9%, respectively. In both cases, the deposition of the Ti-coating on the surface of the milled

Table 1
Density, hardness, Young modulus and fracture-toughness of the Ti–Si samples with and without Ti coating

	Density (g/cm^3)	H (GPa)	E (GPa)	K_c (MPa.m$^{1/2}$)	
				halfpenny	Palmqvist
$Ti_{62.5}Ti_{37.5}$	4.025 (4.385*)	12.8 ± 0.9	203 ± 13	2.9 ± 0.6	3.1 ± 0.5
$Ti_{62.5}Ti_{37.5}$ + Ti	4.386	22.0 ± 3.0	290 ± 20	1.6 ± 0.2	2.4 ± 0.2
$Ti_{85}Si_{15}$	4.446 (4.451*)	10.9 ± 0.7	191 ± 7	2.2 ± 0.4	2.9 ± 0.3
$Ti_{85}Si_{15}$ + Ti	4.531	10.8 ± 0.8	210 ± 14	3.7 ± 0.1	4.0 ± 0.1

* Theorical values calculated on the basis of the overall composition and structural phases.

powders prior to consolidation leads to the increase in their density. However, it is difficult to form a conclusion about the influence of the Ti-coating on the percentage of theoretical density of the coated samples since it does not form a homogenous layer, and thus, any calculation based on the chemical composition and structural phases would be erroneous. Moreover, some portions of the coating are oxidized which makes the calculations even more difficult to perform.

With respect to mechanical properties, average hardness values of 12.8 and 10.9 GPa were obtained for the $Ti_{62.5}Si_{37.5}$ and the $Ti_{85}Si_{15}$ samples, respectively. These values are consistent with their structures (Ti_5Si_3 and Ti + Ti_5Si_3, respectively) and are within the range reported in the literature [17–19]. Min et al. [17] reported the hardness of Ti_5Si_3 with an average grain size of 5–6 μm to be 11.65 GPa under an indentation load of 1.96 N. Under identical loading conditions, Thom et al. [18] measured the hardness of a fine-grained (1–2 μm) and microcrack-free Ti_5Si_3 synthesized by mechanical-alloying followed by HIP. The authors reported a hardness value of 17.1 GPa, much higher than the value of 9.1 GPa, obtained by Frommeyer et al. [19] in a Ti_5Si_3 sample with microcracks. According to these authors, coarse-grained materials containing a larger number of microcracks have lesser hardness than microcrack-free fine-grained materials.

When comparing the compacts consolidated from Ti-coated Ti–Si powders, the hardness obtained for the coated $Ti_{62.5}Si_{37.5}$ alloy is significantly higher than that obtained for the uncoated alloy (22.0 against 12.8 GPa). This is not found to be true in the case of $Ti_{85}Si_{15}$ compacts. In the former case, before deposition, the particles have a median value of about 19 μm as opposed to 44 μm for the $Ti_{85}Si_{15}$ alloy (see Fig. 3). Therefore, it is likely that in the case of the Ti-coated $Ti_{62.5}Si_{37.5}$ sample, the measured hardness values are influenced by the Ti coating which, as mentioned before, contains a high percentage of oxygen (Fig. 7). In fact, some oxides and nitrides were detected in the XRD pattern of this sample, located in the grain boundaries of the powders (Fig. 4b).

The measured Young modulus follows the same trend as hardness, i.e. the higher the hardness, the higher the Young modulus. However, the value obtained for the monolithic Ti_5Si_3 compound (203 ± 13 GPa) is higher than that reported in the literature [20,21]. Using resonance frequency technique, Rosenkranz et al. [20] obtained a value of 160 GPa for a sample prepared by reaction sintering in vacuum. Kumar [21] reported a value of 150 GPa for the Ti_5Si_3 compound.

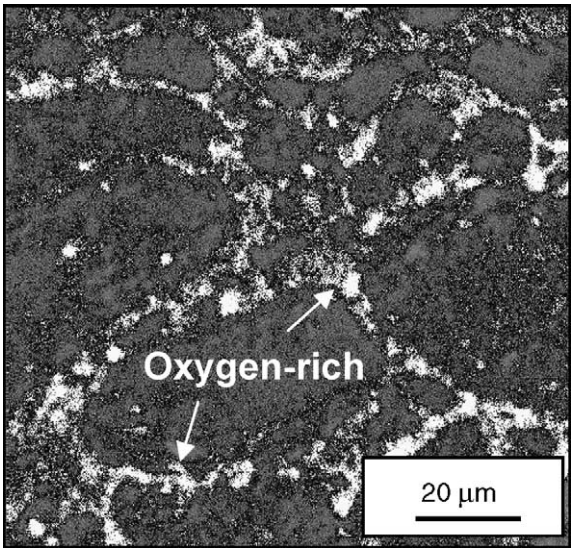

Fig. 7. Oxygen k_α X-ray map showing the oxygen distribution in the Ti-coated compacted $Ti_{62.5}Si_{37.5}$ sample.

The Ti coating seems to play different roles in the fracture-toughness of the compacts. In the case of the $Ti_{85}Si_{15}$ alloy, the coating significantly increases the fracture-toughness of the compacts. Values between 2.2–2.9 MPa.m$^{1/2}$ were obtained (depending on the method used) for the uncoated samples and in the range 3.7–4.0 MPa.m$^{1/2}$ for the Ti-coated samples. However, the same is not true for the $Ti_{62.5}Si_{37.5}$ alloy in which a decrease of K_c is observed for the compacts obtained from coated powders. Once again, this might be the result of the presence of a Ti-oxidized layer in the particles' boundaries. The values obtained for indentation fracture-toughness of the Ti_5Si_3 compound are in the range reported in the literature (see for instance Mitra [6] and the references therein).

4. Conclusions

(i) A method for surface modification of mechanically-alloyed intermetallic powders has been developed,

which involves the use of sputtering prior to consolidation.

(ii) Milling of $Ti_{62.5}Si_{37.5}$ and $Ti_{85}Si_{15}$ samples gave rise to nanostructured mixtures formed mainly by the Ti_5Si_3 intermetallic and Ti(Si) solid solution, respectively. Vestiges of α-Ti were also detected in the XRD pattern of the Si-richer sample ($Ti_{62.5}Si_{37.5}$). No phase transformations in the powder core occurred during heating of $Ti_{62.5}Si_{37.5}$ samples up to 1000 °C. The only structural feature observed was grain growth in the Ti_5Si_3 phase. The titanium coating tended to become oxidized/nitrided at high temperatures. Concerning the $Ti_{85}Si_{15}$ sample, the mechanically-alloyed Ti(Si) solid solution partially transformed to α-Ti + Ti_5Si_3 close to 580 °C.

(iii) The deposition of a titanium coating on the mechanically-alloyed Ti_xSi_{100-x} ($x = 62.5$ and 85 at.%) powders gives rise to composite materials. The presence of a metallic layer on the surface of the powders has different effects on the mechanical properties of the hot-compacted samples. In contrast to the $Ti_{62.5}Si_{37.5}$ alloy, it increases the Young modulus and fracture-toughness of the $Ti_{85}Si_{15}$ samples. Hardness is not affected by the presence of Ti at the interfaces in this alloy. In the case of the $Ti_{62.5}Si_{37.5}$ alloy, partial oxidation of the coating was observed, which is responsible for the increase in hardness and Young modulus and the decrease in fracture-toughness.

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