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High-performance, low-cost titanium metal matrix composites

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ABSTRACT

Titanium-based discontinuously reinforced metal matrix composites were obtained by mechanical alloying and spark plasma sintering. Titanium sponge was used as a raw material to minimize costs, and a small amount of calcium was added as a process control agent. α and β Titanium matrices were obtained by the use of two different compositions, and 3 wt% carbon black was used as the carbide former.

The milled powder was then sintered at low temperature and low pressure. An *in-situ* precipitation occurred involving a fine and well dispersed distribution of titanium carbide in a titanium matrix. Depending on the starting composition, the volumetric quantity of titanium carbide was between 23 and 31 vol%. Despite the high ceramic loading, all of the samples could withstand tensile loading with distinct plastic deformation. The best samples showed a yield stress of 1013 MPa, Young's modulus of 137 GPa, an ultimate tensile stress of 1181 MPa and a strain at fracture of 5%. These results were obtained without any other additional heat treatment or hot deformation step.

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1. Introduction

Metal matrix composites (MMCs) are an interesting type of metal that combine the distinct characteristics of two or more materials, resulting in physical and mechanical properties that are otherwise impossible to obtain [1,2]. Within the MMC family, discontinuously reinforced MMCs (DRMMCs) based on aluminum, magnesium or titanium are very interesting because of their potentially widespread use [3,4]. Due to their isotropic behavior, DRMMCs can replace standard alloys with materials with better physical/mechanical properties without the need to redesign the object, unlike fiber-reinforced MMCs, which have impressive physical/mechanical properties but are also distinguished by anisotropic behavior.

Discontinuously reinforced aluminum (DRA), the most commonly used MMC, is composed of ceramic particles, usually silicon carbide, embedded in an aluminum matrix. The identity and dimensions of the ceramic particles, the volumetric quantity of the reinforcement and the type of aluminum matrix determine the final physical/mechanical properties. The reinforcement is usually between 2 and 100 μ m in size with a volumetric quantity between 1 and 60 vol%. Small reinforcement dimensions provide better mechanical properties and thermal stability, whereas large dimensions and high volumetric quantity improve wear resistance.

The best DRA mechanical properties are obtained by the powder metallurgy approach to MMC production, which strongly reduce the formation of unwanted compound layers between the reinforcements and the matrix during processing. The formation of these unwanted products, *e.g.*, Al_4C_3 in the case of silicon carbide as the reinforcement, is much higher when the liquid phase is present. The powders are mixed and compacted to form a fully dense billet by hot isostatic compaction. Billets are available for the direct manufacturing of components or fabrication by forging, extrusion or rolling techniques. The selection of the process route depends on the property requirements, component shape and resulting process cost.

The use of powder metallurgy limits the dimensions and volumetric quantity of the reinforced particles. For geometrical reasons, the dimensions of the reinforcement, the quantity and the dimensions of the aluminum powder are strictly correlated: *i.e.*, fine silicon carbide (2-3 µm) cannot be well mixed with highvolumetric-quantity aluminum powder (over 20 vol%) because each aluminum powder particle would be covered by the silicon carbide, inhibiting the metallic contact and thereby preventing sintering. A very fine aluminum powder would be needed, below $d_{50} = 10 \,\mu\text{m}$, but its use is impracticable because of handling difficulties and explosion risk. Of course, thermomechanical processing, such as extrusion, forging and rolling, leads to a much uniform distribution, but not indefinitely so. If a high reinforcement loading of fine particles is desired, high-energy mixing of the powders is necessary, which inserts the reinforcement particles inside the aluminum powder. The resulting MMC powder can be

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processed with the standard schedules to produce spatially homogeneous loading in the aluminum matrix.

DRMMCs obtained by powder metallurgy are also distinct in that they can be produced by *ex-situ* or *in-situ* routes. In the first (*ex-situ*) case, the reinforcement particles are directly introduced without modifying their chemical composition during the processing cycle. In the second (*in-situ*) case, chemical compounds are introduced and converted through a specific step, namely, sintering or a heat treatment, in the final reinforcements. Good examples are the *ex-situ* introduction of SiC in an aluminum matrix and the *in-situ* introduction of carbon black and its conversion in Al₄C₃.

The same considerations can also be made in the case of discontinuously reinforced titanium (DRTi) but with more limitations due to the high reactivity of titanium, high-temperature processing and high-energy mixing problems.

Titanium is extremely prone to dissolving other materials and tends to pick up interstitial elements, such as oxygen, nitrogen and hydrogen, which must be closely controlled to obtain a highquality microstructure and good mechanical properties, especially toughness. The high reactivity of titanium limits the reinforcement that can be used because the introduced ceramic powders evolve during the high-temperature processing [5]. For example, sintering a mix of silicon carbide powder and titanium powder produces a mixture of TiC, Ti₅Si₃ and SiC embedded in a titanium matrix because some of the silicon carbide is dissolved by the titanium and an in-situ formation of non-stoichiometric unstable compounds takes place, undermining the alloy stability and its mechanical properties. Oxide and nitride must be avoided for the same reasons and because the titanium matrix cannot withstand oxygen levels above 0.4 wt% or nitrogen levels above 0.05 wt % [6]. Carbides and borides can be included provided that the bound elements (e.g., TiB₂, TiC, ZrB₂, and ZrC) are good candidates because dissolution by the titanium matrix forms stable monoboride or carbide and the metallic elements form a solid solution. Elemental carbon, elemental boron and B₄C are probably the best candidates because they form in-situ TiC or TiB without any other harmful compounds and are added to the matrix in low quantities [7–9]. For example, if a DRTi with 20 wt% TiC is wanted, one can choose the *ex-situ* or *in-situ* route. For simplicity, it is assumed that only the stoichiometric TiC is formed; therefore, the *ex-situ* route requires the mixing and processing of 80 wt% pure titanium and 20 wt% TiC, whereas the *in-situ* route requires only 4 wt% carbon and 96 wt% titanium which are needed to produce the same TiC quantity. This point is very important from the perspective of harmful element introduction, especially oxygen. Titanium powder has an oxygen content of approximately 0.15 wt%, titanium carbide approximately 1 wt% and graphite or carbon black, as a carbon carrier, approximately 0.5 wt%. Therefore, the starting mixture of the *ex-situ* DRTi has an oxygen content of 0.32 wt%, while the oxygen content will be 0.164 wt% for the in-situ DRTi. Considering that the oxygen content inevitably increases during processing [10], it is easy to obtain DRTi with 0.4 wt% and 0.2 wt% for the ex- and in-situ routes, respectively. At the end, the ex-situ DRTi has a high level of oxygen, which is deleterious for toughness and even more for corrosion resistance [6]. Due to the different coefficients of expansion for titanium and titanium carbide, all of the composites are internally stressed, and it is known that titanium with oxygen contents in excess of 0.2 wt% is prone to stress corrosion. Therefore, composites obtained by in-situ route with a low oxygen content are always preferable.

Nevertheless, the problem of obtaining a uniform distribution of fine reinforcement in the titanium matrix remains. This problem is even worse for titanium matrices than for aluminum matrices because the finest titanium powder is $d_{50}=20 \,\mu\text{m}$. Below this number, the oxygen and nitrogen contents of the powder would be over the tolerable limit.

The uniform distribution of the reinforcements, or the elements that will be converted *in-situ* to become reinforcements, can be enhanced by high-energy mixing; however, this approach suffers from high contamination and low process yield [11,12].

A different approach for obtaining a high loading of fine hard particles is the *in-situ* precipitation of a supersaturated solid solution (SSS). Using this method, liquid titanium with a high carbon or boron content is atomized to obtain a fine powder with carbon or boron in solid solution. During sintering and/or thermal treatment, the precipitation of TiC or TiB then occurs for thermodynamic reasons [13]. The high cooling rate during atomization allows powders to be obtained at room temperature with carbon or boron contents above those stated by the phase diagram without the formation of precipitates. Unfortunately, the carbon or boron content in the liquid titanium cannot be in excess of the eutectic point (1.7 wt% for boron and 0.7 wt% for carbon for pure titanium) because large primary carbides or borides form in the melt and clog the nozzle.

To overcome these problems, mechanical alloying (MA) can be used [12]. Mechanical alloying, which is a solid-state process consisting of the repetitive and continuous plastic deformation of a powder, can process virtually any composition. However, it has been rarely used for titanium because of low process yield and contamination. Lately, it has been demonstrated that the introduction of a low quantity of calcium or rare earth metals makes mechanical alloying easy and stable for titanium alloys [14,15].

This paper reports efforts to obtain a DRTi with high TiC loading and a fine microstructure by mechanical alloying and spark plasma sintering (SPS). MA was used to obtain a carbon-supersaturated titanium powder, while SPS was used to obtain bulk samples quickly and at low temperature. Two compositions were produced: Ti5Sn3C and Ti13Cr5Sn3C. Tin was added to reduce cold welding phenomena and increase the α phase hardness, while chromium was added to understand the behavior of a β -stabilizing element. Moreover, chromium is beneficial for corrosion and high-temperature oxidation [6]. As a starting metal, to obtain a low-cost DRTi, Kroll titanium sponge instead of titanium powder was used [14].

2. Experimental procedure

As raw materials, Kroll titanium sponge (Toho, -3 mm, purity 99.8%), pure chromium (Exotech Inc., $-150+45 \mu$ m, purity 99.9%), tin (Alfa Aesar, -16 mesh, purity 99.5%) and carbon black (Alfa Aesar, 42 nm, purity 99.9+%) were used.

The raw materials were weighed to produce two alloys with the compositions Ti5Sn3C and Ti13Cr5Sn3C (all of the compositions are expressed in weight). The mechanical alloying was performed in a Fritsch Pulverisette 6 with a jar volume of 500 ml. A ball-to-powder ratio of 20:1, steel ball diameter of Ø15 mm, rotational speed of 500 rpm and milling time of 80 min were used. The jar was evacuated to a low vacuum, and 0.1 wt% calcium was used as a processing control agent (PCA) for reasons provided elsewhere [14,15]. After ball milling, the powders were cooled to room temperature in the jar over 2 h. The jar was always open to air, and the powders were stored for several days in bottles with air.

The powders were sieved to particle sizes below $180 \ \mu m$, and the process yield was calculated as the ratio of the sieved powder to the amount of powder introduced into the jar.

Oxygen and nitrogen analysis was performed using a LECO TC400 system. The diffraction patterns were collected using a Cu K α (λ =1.5418) source and an image plate detector over the 2 θ range of 30–110° in reflection geometry. The experimental spectra were elaborated with the Rietveld method using the Materials

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