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Microstructural and mechanical comparison of Ti + 15%TiCp composites prepared by free sintering, HIP and extrusion

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ABSTRACT

Ti + 15%TiCp composites were prepared by three different powder metallurgy techniques: free sintering, HIPing and direct powder extrusion. The microstructures and textures were characterized by EBSD while the Ti/TiC reaction, which takes place during heat treatment, was characterized using X-ray diffraction and Rietveld analysis. The mechanical properties were obtained by the mean of tensile tests.

TiC particles effectively reinforce the strength of the Ti matrix by at least 40% indicating the Ti/TiC reaction did not deteriorate the mechanical properties. The correlation between the microstructural features and the mechanical properties is discussed and the processes are compared. Direct powder extrusion was shown to be the most suitable process to obtain ductile composites ($\varepsilon = 4.6\%$).

1. Introduction

This research was performed in the environmental context of reducing the weight of materials used in the domain of transports. A lot of attention has been paid to metal matrix composites (MMCs) in this regard. Aluminum MMCs are now well established for applications with moderate loadings and temperature [1]. In order to extend the use of MMCs to more severe conditions, a lot of research has been dedicated to the development of Particle Reinforced Titanium Matrix Composites (PR-TMCs) [2]. These materials have considerable potential for use in aerospace applications due to their high specific mechanical properties [1].

TiC particles improve mechanical properties such as tensile strength, Young's modulus and wear resistance [3]. However, adding ceramic particles significantly reduces the ductility of PR-TMCs which has greatly limited their application to the industry up to now [4,5]. For this reason, most PR-TMCs produced as of today rarely exceed 10 vol.% reinforcement and tend to be almost brittle [6]. Nevertheless, higher ceramic loading is attractive to further enhance the composite properties in particular wear resistance and high temperature properties [7–9].

TMCs have mainly been prepared by Powder Metallurgy (PM) as it gives the possibility to tailor the microstructure [10]. Recently authors have preferred the in situ route (in situ formation of the TiC particle from the reaction of carbon with the matrix) over ex situ (direct introduction of TiC particle) because of the chemical reaction occurring between Ti and TiC (TiC exists over a large range of stoichiometry, from $TiC_{0.5}$ to TiC_1) during heat treatment [6]. However we consider ex situ to be a valuable route to obtain composites with enhanced mechanical properties as long as the reaction is understood and limited.

In order to maximize the reinforcement effect, Ti + 15 vol.% TiC were produced using three different PM processes: free sintering, hot isostatic pressing (HIP) and direct powder extrusion. This work will aim on the one hand at comparing the composites obtained from a microstructural and a mechanical point of view, in order to find the most suitable process to produce ductile Ti + 15 vol.% TiC composites. On the other hand, this work will discuss the interaction between Ti and TiC to validate the use of the ex situ route.

2. Experimental procedures

The composites produced in this research consist of a pure Ti matrix (Grade 2) reinforced with 15 vol.% TiC particles. The spherical Ti powder was obtained from Electrode Induction-Melting Gas

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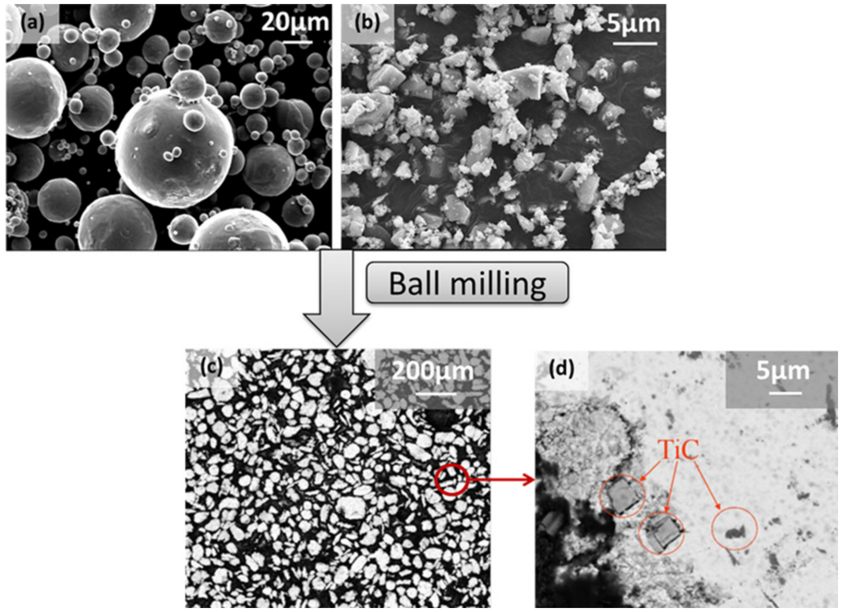

Fig. 1. Composite powder preparation: (a) Ti powder, (b) TiC powder, (c) and (d) milled composite powder.

Atomization (EIGA) and has a mean particle size of 70 μm (Fig. 1(a)). The mean size of the angular TiC particles is 2.6 μm (Fig. 1(b)). The two powders were ball milled under inert atmosphere for several hours in order to reduce the grain size and homogenize the TiC distribution. Batches of about 550 g were prepared.

The resulting composite powder was either free sintered, hot isostatic pressed (HIP) or extruded:

- For free sintering, the powder was pressed at 600 MPa by cold uniaxial pressing followed by sintering at 1375 °C for 3 h under Ar atmosphere. Cylinders of about 10 mm in diameter and 10 mm in height were obtained.
- In the case of HIP, the powder was encapsulated, degassed and gas-proof sealed before being heated up to 920 °C for 2 h under a load of 100 MPa. Cylinders of about 25 mm in diameter and 55 mm in height were obtained.
- For extrusion, steel cans were filled with powder, degassed and gas-proof sealed. The cans were then heated to 920 °C for 1 h before extrusion. The extrusion ratio is approximately 4. Bars about 1500 mm long and 15 mm in diameter were obtained.

Milling, HIPing and direct powder extrusion of the PR-TMCs was developed and performed by Mecachrome. Up to now, extrusion was generally used as a post treatment in order to refine the microstructure and homogenize the particle distribution in PR-TMCs obtained by HIP or casting [11]. However, recent research examples have shown good results for composites prepared by direct powder extrusion [12].

A flow chart of the preparation processes is presented in Fig. 2 and the experimental conditions are summarized in Table 1. For easier notation, the free sintered, HIPed and extruded composites are referenced as TMC 1, TMC 2 and TMC 3 respectively.

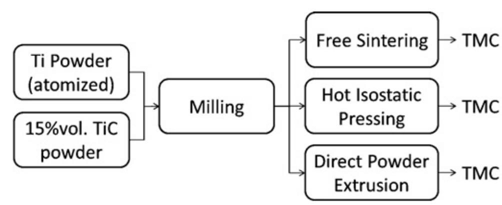

Fig. 2. Processing flow chart.

Surface preparation was performed using standard mechanical grinding (SiC papers up to 2500) and polishing techniques (from 6 μm to 1 μm diamond). Before electron backscattering diffraction (EBSD) analysis, the samples were further polished with colloidal SiO_2 on a vibrating cloth. The microstructures were examined on a Zeiss SUPRA 55VP FEG-SEM coupled with EBSD. HKL Channel5 software was used to analyze the maps obtained by EBSD and determine the grain size and textures of all samples. In the analysis, subgrain boundaries were defined as crystallographic disorientations ranging between 2° and 15° and grain boundaries as disorientation values larger than 15°.

Phase characterization of the samples was performed by X-ray diffraction using Bragg–Brentano geometry by means of a Philips PANalyticak X'Pert Pro X-ray diffractometer, equipped with a curved graphite monochromator (CuKα_1 radiation λ = 1.540562 Å). The program BRASS [13] was used for the Rietveld analysis of the samples to obtain the size of TiC crystallites. The first phase characterization tests performed demonstrated that X-ray experiments on the bulk composites were not pertinent because of strains induced by the matrix on the carbide phase (because of different thermal expansion coefficients). Therefore, to circumvent this pitfall, the metallic matrix was dissolved in a concentrated bath of cooled fluorhydric acid. By doing so, the titanium matrix was

Table 1
Summary of experimental conditions.

Reference	Matrix	Reinforcement	Processing	Temperature (°C)	Holding time (h)
TMC 1	Ti	15 vol.% TiC	Free sintering	1375	3
TMC 2	Ti	15 vol.% TiC	HIP	920	2
TMC 3	Ti	15 vol.% TiC	Extrusion	920	1