Development of a sintering methodology through abnormal glow discharge for manufacturing metal matrix composites

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Abstract. In this study, a sintering methodology is presented by using abnormal glow discharge to metal matrix composites (MMC), consisting of 316 steel, reinforced with titanium carbide (TiC). The wear behaviour of these compounds was evaluated according to the standard ASTM G 99 in a tribometer pin-on-disk. The effect of the percentage of reinforcement (3, 6, and 9%), with 40 minutes of mixing in the planetary mill is analysed, using compaction pressure of 700 MPa and sintering temperature of 1,100°C±5°C, gaseous atmosphere of H₂ - N₂, and sintering time of 30 minutes. As a result of the research, it shows that the best behaviour against wear is obtained when the MMC contains 6% TiC. Under this parameter the lowest percentage of pores and the lowest coefficient of friction are achieved, ensuring that the incorporation of ceramic particles (TiC) in 316 austenitic steel matrix significantly improves the wear resistance. Also, it is shown that it is possible to sinter such materials using the abnormal glow discharge, being a novel and effective method in which the working temperature is reached in a short time.

1. Introduction

In search of new materials with improved properties, an inquiry is made about the behaviour of metal matrix composite (MMC) materials that can provide properties that are not possible to obtain with only one of the original materials (matrix or reinforcement). One of the most used techniques for the manufacture of composite materials is the powder metallurgy techniques (PM). In this study, type 316 stainless steel is used as the matrix, given that it is one of the most used to present excellent mechanical properties, corrosion resistance and thermal conductivity [1]. Although these steels exhibit low wear resistance, superior properties are achieved if particles as reinforcing material are added and if the sintering method of the parts [2] is varied. Reinforcement particles commonly used in MMC are oxides (Al₂O₃, ZrO₂), carbides (TiC, WC) and borides (TiB₂, CrB₂). Among these reinforcements, the TiC is considered a good alternative to be used to present high melting point and being chemically stable at high temperatures. Its pure structure allows the combination of ionic, covalent and metallic links; for that reason, it combines the physical properties of the ceramic and the electrical properties of metals, such as high hardness and high thermal conductivity [3]. Some studies [4] suggest that the reason for that studied steel (316) is sintered in inert atmospheres, at high temperatures is to thereby achieve a better performance. For the development of this research, the MMC are sintered by generating an abnormal glow discharge in H₂ – N₂ atmosphere, which is a recent technique that represents some decrease in the time required for the sintering of samples and lower power consumption, because heating is performed directly by the bombardment of ions and neutral atoms on the surface of the cathode where the sample [5] is usually located. Considering that the material wear...
is a major problem in industry, which largely affects the production sectors [6], the objective of this work is to study the wear behaviour of MMC manufactured with 3, 6 and 9% (vol.) of TiC. Thus useful information to ensure that the incorporation of ceramic particles (TiC) in austenitic steel matrix (316) may lead to an improvement in wear resistance.

2. Experimental

The metal matrix composite is obtained through conventional PM using powders of austenitic 316 stainless steel and metal matrix, with the following chemical composition: 17% Cr, 12% Ni, 2.5% Mo, 2% Mn, 1% Si, 0.08% C and Fe (balance), using as reinforcement titanium carbide TiC (99.95% purity), with average particle sizes for the steel 106.140 µm and for the TiC 4.056 µm.

The mixture is performed in a planetary mill (RESTCH), consisting of two stainless steel vessels with balls of the same material of 10mm in diameter with a ball/loading ratio of 4:1, for 40 minutes at 180rpm; the mixtures are prepared with three different amounts of reinforcement 3, 6 and 9% (vol.). The homogeneity of the mixtures was checked by scanning electron microscopy. The samples are uniaxially compacted at 700MPa in a hydraulic press (ELE International) of 1000KN capacity, using a matrix to which was applied zinc stearate on the wall as a lubricant to obtain a uniform density, resulting in green preforms with approximate dimensions of 12mm diameter and 4mm thick. These preforms were sintered by means of plasma abnormal glow discharge of direct current in an atmosphere of H2–N2, under the following operating parameters: pressure 3torr, flow rate of 2cm³/min, voltage 534V, 160mA current and heating rate of 100°C/min, conditions that reach the temperature of (1,100°C±5°C), with a stay of 30 minutes, ensuring the presence of a stable plasma. Cooling is performed in the same sintering atmosphere under the same flow conditions [4, 6].

Characterization of the interface between TiC particles and the matrix is performed by chemical microanalysis, to thereby images are taken in the scanning electron microscope at 5000X, in the mode of backscattered electrons with an acceleration voltage of 20kV and spot size of 40.

To analyse the properties achieved in the MMC the following are determined: The density of the samples by the Archimedes method; porosity, with the software IQMaterials, version 2, which quantifies the amount of pores in the MMC with the images obtained in the scanning electron microscope, under the guidelines of ASTM E112 standards, E1382 and E562; wear, with the pin-on-disk test according to ASTM G 99 standard in a tribometer (NANOVEA TRB), using as contramaterial a steel pin of 6mm diameter and hardness of RC 71.4. The tests are carried out at room temperature without lubrication. The applied load is of 5N, with sliding speed of 0.1m/s, footprint diameter of 3.5 mm and sliding distance of 377m. The friction coefficient is measured continuously during the test and the wear is determined by the wear coefficient k: k(m²/N)= loss of material volume (m³)/[applied load (N)*Sliding distance (m)].

With the obtained results, the effect of the percentage of reinforcement against wear produced in the MMC when sintered through abnormal glow discharge is evaluated.

3. Results and discussion

In Figure 1, the chemical EDX microanalysis is shown, performed with the samples by Line Scan, in order to characterize the interface between TiC particles and the matrix, indicating the start (P₀) and final (Pₚ) points of the line scan.

For all MMC made with different percentages of reinforcing the behaviour is similar. Observing that chromium oxide appears as a continuous layer defining TiC particles; a situation that is evidenced by the presence of peaks of O and Cr located on one or both sides of the region comprising the reinforcing particle. Likewise, the elements comprised by the matrix are shown: Fe, Cr, Ni, Mo and Si, which are located around the particle and no direct chemistry interaction with the elements that constitute the reinforcement is evidenced. Considering that titanium carbide is a chemically stable reinforcement that does not react with the matrix, ZiFei Ni, et. al [7], hence the addition of TiC aids to the grain refinement of the austenite matrix without forming a new phase in the microstructure, and improves the mechanical properties and wear resistance [8]. The sintered samples exhibit the
following densities: steel PM (6.07g/cm³), MMC 3% TiC (5.58g/cm³), MMC 6% TiC (5.45g/cm³) and MMC 9% TiC (5.31g/cm³). It is evident that when reinforcing the stainless steel with titanium carbide a decrease in density occurs due to the lower specific weight of the reinforcing material. The relative density is maintained around 70% in all compounds.

In Figure 2, the analysis of images obtained in a scanning electron microscope of the sintered samples by using the software IQMaterials, Version 2, is presented, showing that the MMC exhibiting lower percentage of pores is the composite reinforced with 6% TiC, which is possibly due to the fact that with this amount of reinforcement there is better interaction between the matrix and reinforcement.

The effect of sintering on the mechanical properties is evaluated, emphasizing on the friction coefficient and wear in the manufactured compounds with different percentages of reinforcement, see Table 1. It is noted that the lower coefficient of friction is presented in the MMC made with 6% of TiC and, as expected, it exhibits less wear. Also, it shows that by increasing the percentage of reinforcing the compound undergoes greater wear, which can be caused by the TiC tendency to agglomerate around itself, preventing any proper interaction between the matrix and the reinforcing; an effect that is observed in Figure 1(C), causing that the reinforcing be removed from the material and help generate three-body abrasive wear.

![Figure 1. Line Scan performed at MMC reinforced: (A) (3% TiC), (B) (6% TiC), (C) (9% TiC).](image-url)
VARIABLE A (3% TiC) B (6% TiC) C (9% TiC)
% of Poros (red) 4.6203 2.2205 4.934

Figure 2. Percentage of porosity in the sintered MMC, obtained with software IQMaterials.

Table 1. Effect of the percentage of reinforcement in the friction coefficient and wear coefficient.

| % TiC | Coefficient of friction | Wear coefficient (m³/N.m) |
|-------|-------------------------|---------------------------|
| 0     | 0.7441                  | 8.29 X 10⁻⁹               |
| 3     | 0.5956                  | 6.43 X 10⁻⁹               |
| 6     | 0.4471                  | 3.68 X 10⁻⁹               |
| 9     | 0.6469                  | 7.73 X 10⁻⁹               |

4. Conclusions
The best condition for manufacturing the MMC in the percentage of reinforcement is obtained when the compound contains 6% of TiC. Under this parameter the smaller pore size and lower coefficient of friction was obtained by ensuring the incorporation of ceramic particles (TiC) in austenitic steel matrix (316) representing improvements in wear resistance. In materials produced with the powder metallurgy technique it is important to analyse different manufacturing conditions that lead to obtain better MMC behaviour against wear; for this reason it is recommended to compact the preforms in green at pressures greater than 700MPa and sinter at temperatures above 1,100°C.

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