

Influencia de la presión de compactación en compuestos de acero AISI 316 reforzados con carburo de titanio frente al desgaste

Influence of compaction pressure in steel composites AISI 316 reinforced with titanium carbide against wear

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Resumen

En los materiales manufacturados con la técnica de pulvimetalurgia, es importante analizar diferentes condiciones de fabricación que conlleven a obtener un mejor comportamiento del compuesto de matriz metálica (MMC) frente al desgaste, ya que este es uno de los principales problemas en la industria, que afecta en gran parte a los sectores de producción. En este estudio se seleccionó el acero inoxidable AISI 316 como matriz, reforzado con 3, 6 y 9% de carburo de titanio (TiC). Se evalúa el efecto que tiene la presión de compactación (700 – 800 MPa) y el porcentaje de refuerzo. La temperatura de sinterización ($1.200\pm 5^{\circ}\text{C}$), es alcanzada por medio de la generación de una descarga luminiscente anormal de corriente directa, que transfiere energía eléctrica a un medio gaseoso, generando una descarga eléctrica en medio de los electrodos (ánodo y cátodo, donde se sitúa la muestra) en una atmósfera de $\text{H}_2 - \text{N}_2$ con tiempo de permanencia de 30 minutos. Como resultado de la investigación se evidencia que la incorporación de partículas de cerámica (TiC) en matriz de acero austenítico (AISI 316) presenta mejoras significativas en la resistencia al desgaste, obteniéndose el mejor comportamiento cuando el compuesto es compactado a 800 MPa y con contenido de refuerzo del 6%. En estas condiciones de fabricación existe buena interacción entre la matriz y el refuerzo, baja porosidad, mejor densificación con la más alta dureza y bajo coeficiente de fricción.

Palabras clave: *Compuestos de matriz metálica, presión de compactación, pulvimetalurgia, resistencia al desgaste, sinterización por plasma.*

Abstract

In the manufactured materials with the powder metallurgy technique, it is important to analyse the different manufacturing conditions that lead to get a better behaviour of the metallic matrix composite (MMC) against the wear, since it is one of the main industry problems largely affecting part of the production sectors. In this study the stainless steel AISI 316 was selected as a matrix reinforced at 3, 6 and 9% Vol of titanium carbide (TiC). The effect of the compaction pressure (700 – 800 MPa) and the reinforcement percentage were evaluated. The sintering temperature ($1200\pm 5^{\circ}\text{C}$) is reached by means of the generation of an abnormal glow discharge of direct current that transfers electric energy to a gaseous medium, generating an electric discharge between the electrodes (anode and cathode, where the sample is located) in an atmosphere of $\text{H}_2 - \text{N}_2$ and with a residence time of 30 minutes. The investigation results evidence that the ceramic particles incorporation (TiC) in an austenitic steel matrix (AISI 316) demonstrates a significant improvement in the wear resistance, getting the best behaviour when the composite is compacted at 800 MPa and with a reinforcement content of 6%. In these manufacturing conditions there is a good interaction in the matrix and the reinforcement, low porosity, superior densification with the highest hardness and lowest friction coefficient.

Keywords: *Compacting pressure, metal matrix composite, plasma sintering, powder metallurgy, wear resistance.*

1. Introduction

The manufacturing process by powder metallurgy (PM) was selected as the processing method for this research, for the advantages of incorporating a hard phase in a metal matrix through the powder mixture (Xinhong et al., 2006). In this process compounds are prepared by mixing the dry metal powders, sometimes combined with other elements as ceramics or polymers, resulting in the metal matrix composites (MMC), obtaining improved wear resistance, heat resistance and thermal stability. In particular, the compounds of steel matrix become notorious in industrial applications due to their mechanical properties and low cost (Meng et al., 2010).

In this research a 316 stainless steel type is used as a matrix, taking into account that this is one of the most used stainless steels, mainly in the automotive industry for engine systems and transmission for presenting excellent mechanical properties and corrosion resistance (Akhtar et al., 2009; Velasco et al., 2003). Although these steels show low resistance to wear, if particles as reinforcement material are added and if the sintering method is modified an improvement of those properties is achieved (Akhtar et al., 2009). The reinforcing particles used were titanium carbide (TiC), for the properties it has as: hardness, a high melting point and good thermodynamic stability that make it suitable to reinforce matrices of iron and steel. (Xinhong et al., 2006; Moreira, 1999; Zhong et al., 2011; Doğan et al., 1999). The compacts were sintered by generating a glow discharge or plasma of direct current, which transfers electrical energy to a gaseous medium, generating an electrical discharge between the electrodes (anode and cathode) in an atmosphere of $H_2 - N_2$. Recent technique that represents a decrease in the time required for the sample sintering process and a lower energy consumption, since the heating is performed directly by bombarding ions and neutral atoms on the cathode surface, where the sample is generally located. Obtaining better results than those achieved when the samples are sintered by conventional methods, since in this case is shown a wide range of size distribution of

grains and large morphological heterogeneity, with the presence of higher internal stresses that generate higher content of microcracks in the grains of the material, leading to a decrease of the mechanical properties (Benavente, 2015).

The aim of this research is to study the wear resistance behaviour of MMC manufactured with 3, 6 and 9% (vol.) of TiC, using compaction pressure of 700 and 800 MPa, in order to evaluate the best manufacturing conditions of the compounds that can ensure that the addition of ceramic particles (TiC) to austenitic steel (316) matrices leads to an improvement in wear resistance.

2. Materials and methods

The stainless steel 316 type was selected as the matrix, supplied by SULZER-METCO, with the chemical composition shown in Table 1. Considering that it is a material with high production, since it is present in a wide range of applications in different branches of the industry. And as reinforcement, titanium carbide (TiC) for the properties it has, as high hardness, high melting point and good thermodynamic stability that makes it suitable to reinforce matrices of iron and steel (Xinhong et al., 2006; Zhong et al., 2011; Doğan et al., 1999; Onuoha et al., 2013), this powder was supplied by SIGMA-ALDRICH (99,95% purity).

Table 1. Chemical composition of steel AISI 316.

Cr	Ni	Mo	Mn	Si	C	Fe
17	12	2.5	2	1	0.08	equilibrium
%	%	%	%	%	%	

The morphology was determined from the form factor obtained with the scanning electron microscope, JEOL brand. Model JSM 6490LV, Sonda EDS (energy dispersive spectrometry X-ray), Oxford Instruments brand, Model: IncaPentaFETx3.

The mixture (316 steel with 3, 6 and 9% Vol. TiC) is performed in a planetary mill RESTCH brand, consisting of two stainless steel vessels with balls

of the same material of 10 mm diameter, with a ball\ load ratio of 4: 1, during 40 minutes at 180 rpm. The powder mixture is carried out in order to obtain a homogeneous distribution of the elements constituting the alloy, avoiding the formation of aggregates, segregation phenomena and effects that change the physical characteristics and modify the behaviour when compacting the powders (Moreira, 1999; Cardenas, 2012), giving them consistency and shape, so that it can produce a piece in "green" suitable for handling.

Once each of the mixtures was made, the homogeneity was checked by scanning electron microscopy at magnifications of 250X in backscattered electron mode, with an acceleration voltage of 20 kV. Also, the particle size was determined by laser granulometry analysis on the computer MALVERN INSTRUMENTS, with diffraction technique.

The mixtures are uniaxially compacted at 700 and 800 MPa in the hydraulic press ELE International brand of 1000 KN capacity, using a matrix to which zinc stearate was applied as a lubricant to achieve a uniform density (Doğan et al., 1999) and easy removal of the matrix. The dimensions of the preformed in green obtained for this research are of 12mm diameter and 4mm thick.

The preforms in green were sintered by plasma generated by the abnormal glow discharge of direct current, Figure 1, at an atmosphere of $H_2 - N_2$, under the following operating parameters: pressure 3 torr, voltage 534 V, current 160 mA heating rate $100^\circ C / min$, conditions under which the work temperature ($1200^\circ C \pm 5^\circ C$) is reached. This is measured by a thermocouple, with a stay of 30 minutes, ensuring the presence of stable plasma. Cooling is performed in the same sintering atmosphere with the gas flow used ($2cm^3 / s$) (Abenojar et al., 2003).

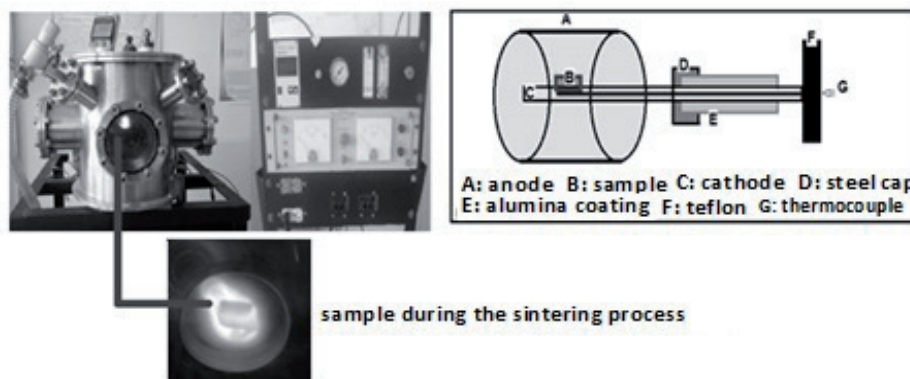


Figure 1. Sintering equipment by abnormal glow discharge

Sintered compounds were characterized in their physical, chemical and technological properties as follows:

Density: by using Archimedes' principle, according to ASTM B962-08 (Containing et al., 2013) standard, and using isopropyl alcohol as a fluid.

Porosity: using the software IQMaterials version 2. The number of pores present in the MMC is determined with the images obtained in the scanning electron microscope, according to these three standards, the ASTM E112 (Methods, 2014),

the E1382 (Documents, 2014) and the E562 (Conshohocken, 2014th).

Hardness: according to ASTM B 933 (Method, n.d.) and B 934 (Depth & Stage, 2004) standards, using 100gf (0.9807 N) in the digital microdurometer QV - 1000 DM model.

Wear: evaluated by pin-on-disk test according to ASTM G 99 (Conshohocken, 2014b) standard, in a tribometer TRB NANOVEA brand, using as contramaterial steel pin of 6 mm diameter and 71.4 RC hardness. The tests are carried out at room

temperature without lubrication. The load applied is 5 N, with sliding speed of 0.1m/s, track diameter of 3.5 mm and sliding distance of 377 m. The friction coefficient is measured continuously during the test and the wear is determined by the wear coefficient k : $k \text{ (m}^2\text{/N)} = \text{loss of material volume (m}^3\text{)} / [\text{applied load (N)} * \text{sliding distance (m)}]$.

3. Results and discussion

The figure 2A shows the form factor having the particles of steel, which reveals a value of 1.61 (0.44). The morphology of the matrix is elongated (oval), as shown in Figure 3A. Also, the form factor reveals that the TiC particles have a value of 1.27 (0.20), directly linked to its polygonal and symmetrical morphology, as shown in Figures 2B and 3B.

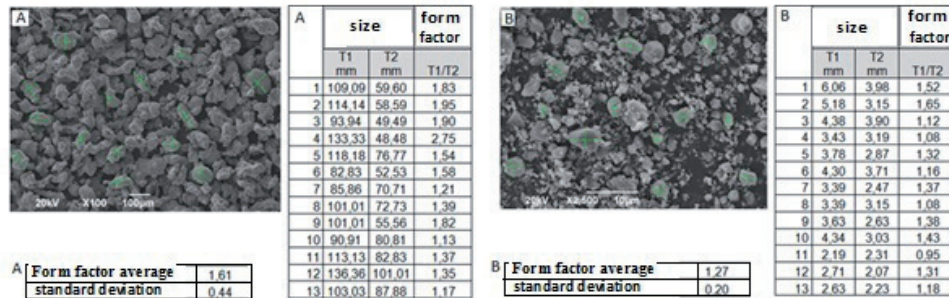


Figure 2. 316 steel Form factor (A). TiC Form Factor (B).

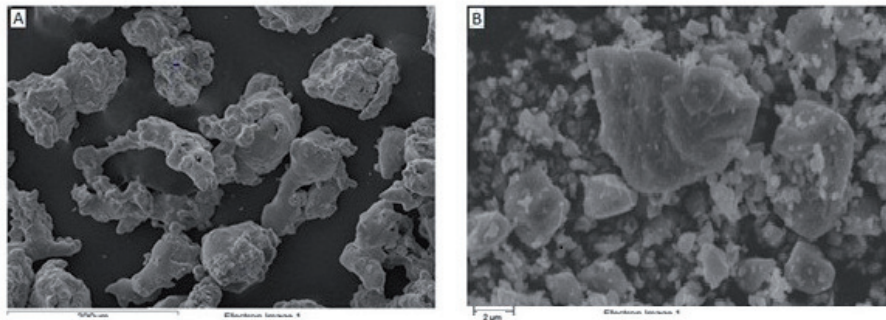


Figure 3. Morphology of 316 steel (A). Morphology of Titanium carbide (B).

The homogeneity of the mixtures is checked with scanning electron microscopy, as shown in Figure 4.

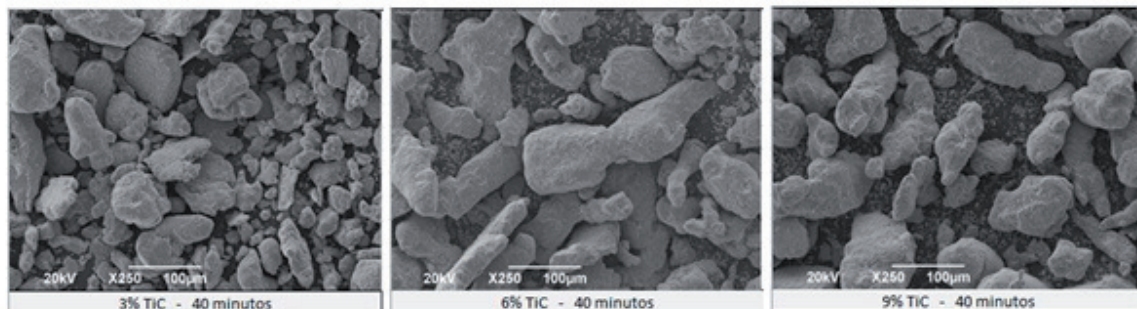


Figure 4. Homogeneity of mixtures.

This figure shows the behaviour of the overall distribution of the TiC particles regarding the stainless steel matrix at the mixing process. For compositions of 3% TiC, small particles are not seen along image, indicating that these were incorporated within the matrix, due to continuous impacts provided by the grinding bodies (balls) and the plastic nature of the matrix. Meanwhile, the chemical composition of 6% TiC, reveals the presence of a low amount of small particles that are isolated from large particles corresponding to a

part of the reinforcement that was not incorporated into the matrix. Finally, the chemical composition of 9% TiC, shows the presence of reinforcing particles that were not embedded within the matrix.

Figure 5 corresponds to the distribution of particle sizes achieved in each of the mixtures, important parameter, as it influences both the fluidity and density of the particles and the final porosity of the product (Moral, 2008).

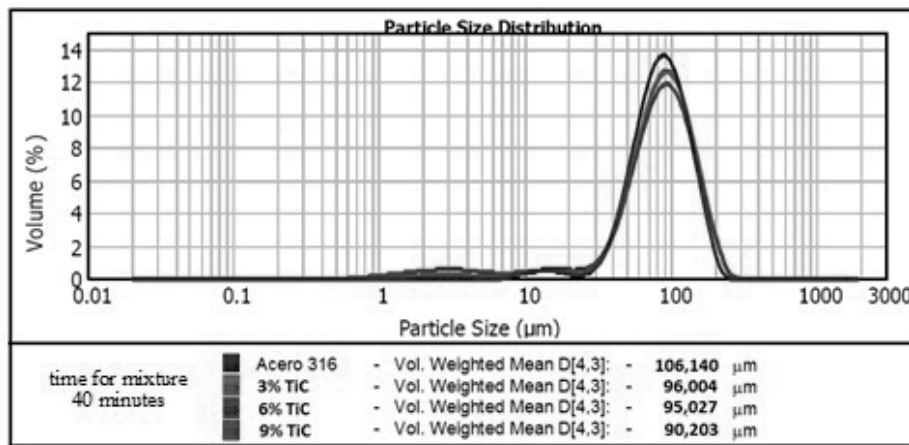


Figure 5. Particles size distribution for mixtures.

The mix of stainless steel and titanium carbide generates a low decrease in the average particle size as the percentage of reinforcement increases (TiC), as seen in Figure 5; this is due to the difference in sizes between the stainless steel and the titanium carbide (106.140 µm and 4,056 µm, respectively) and to the malleable nature of the stainless steel (matrix). Thus, when the powders are subjected to the mixing process, the particles of TiC are mechanically attached to the surface of the matrix, this fact makes the material lose continuity that with the permanent subjection to cyclic stresses (bumps of the grinding bodies), internal cracks are generated, which results in the sectioning of the material and therefore in reducing its size.

During sintering physicochemical interaction processes between particles occur. At this stage

the compressed mixture acquires resistance and ultimate strength. The preforms are introduced into a plasma reactor with atmosphere and temperature controlled (Figure 1). With this atomic diffusion of the material and unity between the different powders are achieved, reached during the compaction process strengthening metallurgical bonds to form a uniform piece with special properties (Kurgan, 2013). Now the behaviour of the density, porosity and hardness selected is analysed in order to evaluate the effect these variables have in the mixtures wear resistance, when compaction pressures of 700 and 800 MPa are used.

The table 2 shows the density achieved by the steel and each MMC manufactured under different percentages of reinforcement, compacted at 700 and 800 MPa.

Table 2. MMC Density reinforced with 3, 6 and 9% of TiC, compacted at 700 to 800 MPa.

Content (TiC) (%)	700 MPa		800 MPa	
	Density (g/cm ³)	Density relative (%)	Density (g/cm ³)	Density relative (%)
0	6,30	79,13	6,70	84,17
3	5,77	73,36	6,10	77,54
6	5,81	73,79	6,32	81,27
9	5,13	65,14	5,17	65,71

According to the results, when reinforcing stainless steel the density is lower in the MMC that contain the largest amount of reinforcement (9% TiC), showing the effect of compaction pressure when evaluating the density achieved, since the pressure increases from 700 to 800 MPa, an increase in density occurs due to the greater plastic deformation experienced by the particles (Ceschini et al., 2006).

The relative density distribution is determined from the density of the sintered regarding the theoretical density (Ertugrul et al., 2014). It is observed that when increasing the percentage of reinforcement, the relative density decreases because adequate packing between the particles

does not occur, a fact that can generate the formation of higher porosity in the composite and lower density of the reinforcement.

In the manufactured materials using the powder metallurgy technique it is important to analyse different manufacturing conditions that lead to obtain lower porosity, this is a very important variable, as it affects the mechanical properties of the compounds made by this forming technique.

Figure 6 shows the number of pores present in each of the compounds and Table 3 shows the percentage of porosity, determined by the software IQMaterials version 2, for the compounds compacted at 700 and 800 MPa.

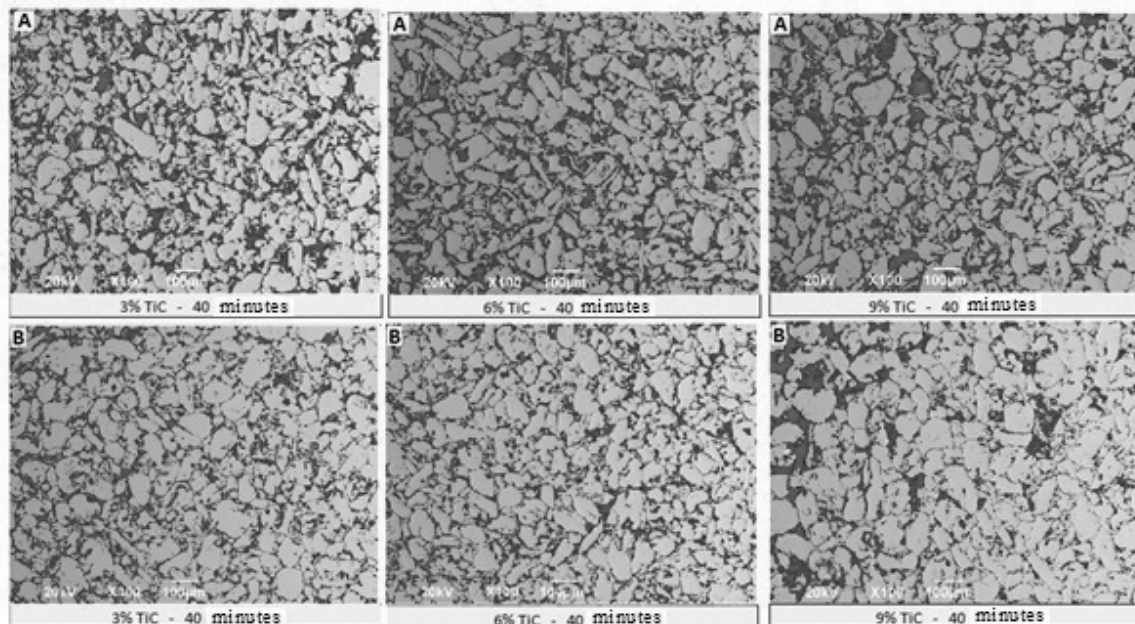


Figure 6. Illustration of the pores present in the MMC compacted at 700 MPa (A) and 800 MPa (B).

Table 3. Percentage of porosity in MMC obtained with the software IQMaterials. Compacted at 700 MPa and 800 MPa.

Compacting pressure	700 MPa			800 MPa		
	3	6	9	3	6	9
% TiC	3	6	9	3	6	9
% pores (red colour)	4,9	4,0	5,7	3,7	2,6	5,2
% reinforcement (blue colour) and % matrix (grey colour)	95,1	96,0	94,3	96,3	97,4	94,8

A porous structure is formed when the surfaces of the added particles do not interact appropriately with the matrix (Liu et al., 2001). This effect results in the detriment of the physical and mechanical properties of the sintered. According to the above, and taking into account the results obtained it is evidenced that the preforms with 6% TiC contain the lowest percentage of pores. Retaking Figure 4, which shows that this mixture shows the presence of a low amount of particles that are isolated, which correspond to portion of reinforcement that is not incorporated within the matrix. These are particles that contribute to be located between the grains at the time of compaction causing a decrease in porosity. For preforms containing 9% TiC, the large quantity of particles that were not embedded within the matrix in the compaction process are agglomerated generating a high percentage of porosity. When the mixture contains 3% of TiC, all the reinforcement is incorporated into the

matrix, by the continuous impacts of the grinding bodies, for that reason, at the time of compaction there is no possibility of presence of additional particles that can help reduce porosity. Also, when the pressure increases, better packing and decrease in the percentage of porosity occurs, due to the formation of new contacts between particles. Lower compaction pressure causes a decrease in the plastic deformation experienced by powders; therefore there are larger clearances between the grains that in many cases do not generate a good retention of the particles of TiC, which is why they break off during handling of the sample.

Figure 7 shows the effect of the percentage of reinforcement and compaction pressure in each of the compounds on the microhardness, measured with "Vickers" scale, considering that this mechanical property can predict the behaviour of the sintered against wear resistance.

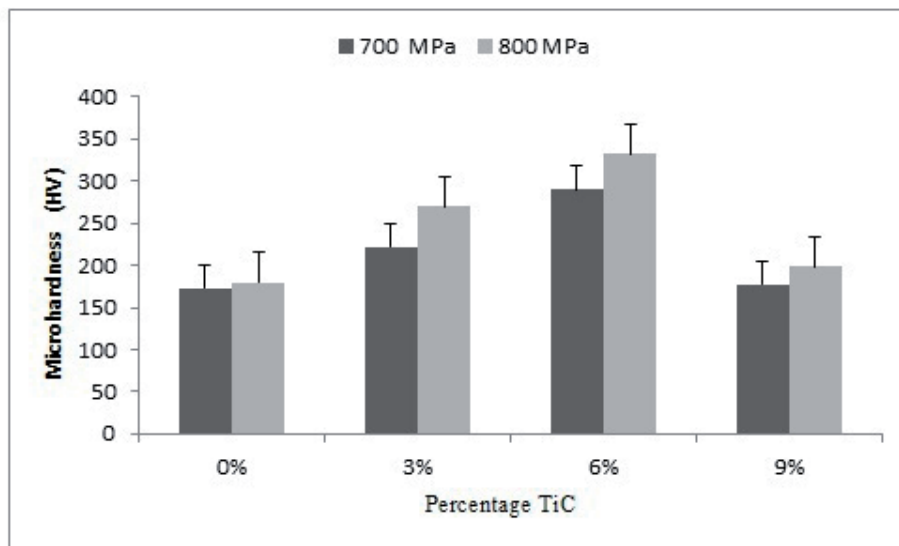


Figure 7. Microhardness in compacted sintered at 700 and 800 MPa.

This figure shows that the microhardness of austenitic steel that is used as matrix increases when titanium carbide particles are incorporated as reinforcement (Xinhong et al., 2006; Velasco et al., 2001). However, the addition of hard particles introduces failure initiation points that often reduce the mechanical properties of composite materials (Velasco et al., 2001). It is evidenced that when increasing the fraction of volume up to 6% TiC, the microhardness of the compound is increased, which probably can obtain less wear. It is evidenced that when increasing the fraction of volume up to 6% TiC, the microhardness of the compound is increased, therefore obtaining less wear can be probable. However, by increasing volume fraction of particles to 9% TiC, these can limit the plastic deformation that can be absorbed by the matrix, leading to fracture and detachment of particles. (Doğan et al., 1999). Also, with this it is difficult to achieve a good interaction between the matrix and reinforcement, caused by the formation of particle agglomerates (Sheikhzadeh & Sanjabi, 2012) and

the low density shown by these compounds with the largest percentage of porosity. This contributes to the detriment of the sintered properties, generating decrease in its microhardness. However, it is clear that this property increases with an increase in compaction pressure, which is attributed to these compounds that have higher density with the lowest percentage of porosity. Considering that the wear of the materials is one of the main problems in the industry, which largely affects the production sectors, the behaviour of MMC is evaluated by subjecting them to wear tests using the pin-on-disk test, searching for mechanical components and conditions that extend the life of the equipment, through the control and reduction of wear.

Figure 8 shows the wear coefficient achieved by sintered when subjected to the wear test by the pin-on-disk testing and compacted at 700 and 800 MPa. In this test the friction coefficient is obtained simultaneously. It is presented in figure 9.

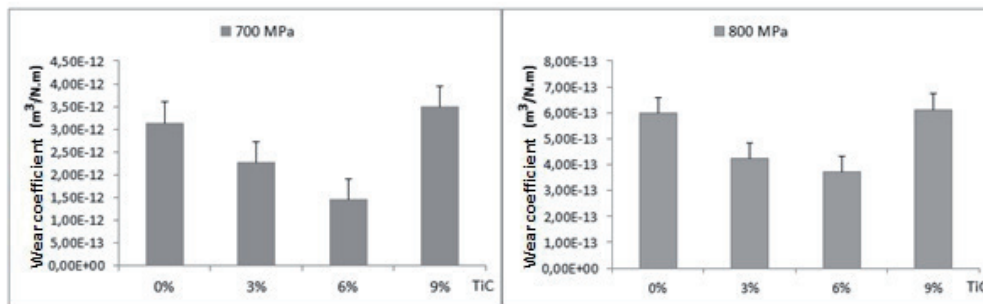


Figure 8. Wear coefficient of sintered compacted at 700 and 800 MPa.

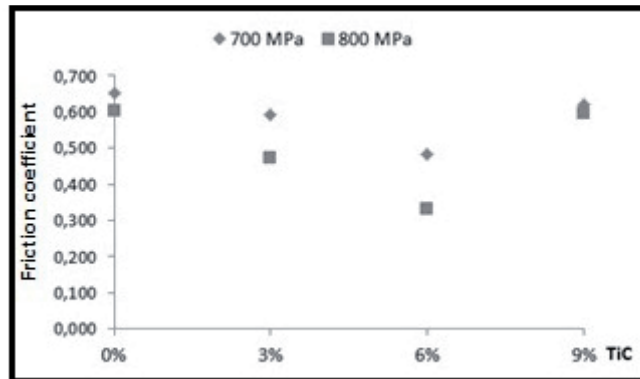


Figure 9. Friction coefficient in sintered compacted at 700 and 800 MPa.

This chart shows that there is improvement against wear when the austenitic steel 316 is reinforced with TiC, achieving a better performance when MMC are compacted at 800 MPa. It is noteworthy that an increase in pressure leads to obtain higher density and microhardness of the compounds with lower percentages of porosity; these variables affect the wear behaviour. It is evidenced that the MMC containing the highest percentage of porosity are adversely affected, showing the higher

friction coefficient and therefore lower wear resistance, this is behaviour exhibited by reinforced composites 9% TiC. This is a condition under which a good interaction between the matrix and reinforcement is not possible to obtain.

By scanning electron microscopy, the track product is analysed with the pin-on-disk test to find out the type of wear present in the sinter, the analysis is shown in Figure 10.

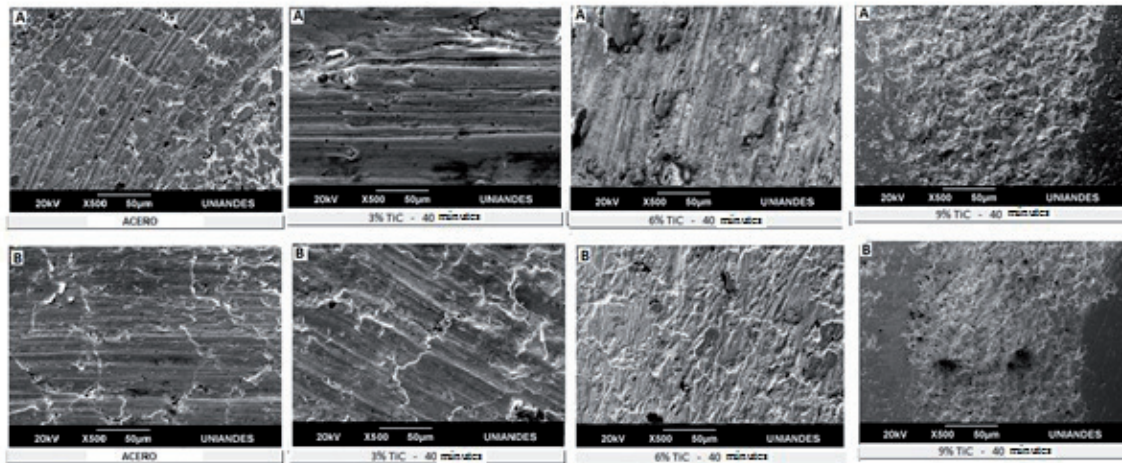


Figure 10. Wear track of sintered compacted at 700 MPa (A) and 800 MPa (B).

The type of wear present is abrasive. The ASTM G40-13 standart (Conshohocken, 2015) defines wear such as the loss of mass resulting from the interaction between particles or hard asperities that are forced against a surface and move along it.

The steel without reinforcement exhibits the track; this is typical of abrasive wear. Although this material has the highest densification this is not a condition that contributes to obtain ideal service behaviours where its resistance to wear is as expected. This demonstrates that the incorporation of particles acting as reinforcement in the austenitic steel matrix is necessary in order to obtain a better response to wear. Moreover, it is evident that the MMC that show a more irregular track are those containing the highest amount of reinforcement (9% TiC). With this mixture the densification and microhardness achieved do not allow good interaction between the matrix and reinforcement,

since the carbide tends to agglomerate, which causes great detachment generating wear with presence of three bodies.

It is worth mentioning that the compounds compacted at 800 MPa have a more homogeneous track, also, the compound containing 6% TiC shows the best production conditions reaching the highest microhardness, the lowest percentage of porosity and the lowest coefficients of friction, having good resistance to wear.

4. Conclusions

In the sintering stage of the samples, by using the plasma of the abnormal glow discharge, allow to reach the sintering temperature in a short time, reducing energy consumption significantly.

Adding TiC to AISI 316 steel, under the conditions studied, generates less densification of the composite

material, based on 316 steel alone, and increases its porosity. It is worth mentioning that with 9% of TiC an appropriate interaction between the matrix and the reinforcement is difficult, which produces detachments of TiC during the pin on disk test that is influenced by the tendency of carbide to agglomerate. As a result this composition of TiC in AISI 316 steel causes the lowest microhardness of MMC, leading to obtain higher coefficients of friction, changing the wear behaviour, in an unfavourable way for the compound.

The best condition for manufacturing the shaped MMC is obtained when compaction pressure of 800 MPa and reinforcement content of 6% are used. Under these parameters the highest density and microhardness were obtained with lower porosity and lower coefficient of friction, therefore, the addition of ceramic particles (TiC) in the austenitic steel matrix (316) represent improvements in wear resistance of AISI 316 steel.

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