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Effect of temperature on morphology and wear of a Cu-Ti-TiC MMC sintered by abnormal glow discharge

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Abstract

Microstructure and tribological properties had been studied in a Cu-Ti-1%TiC MMC with titanium concentration of 10%, 15%, and 20%. The composite was manufactured following the pulvimetallurgical route that included: mechanical and ultrasonic mixing in 2-propanol, compaction at 400 MPa and sintering assisted by abnormal glow discharge in an atmosphere of 10% nitrogen and 90% argon. The discharge was established in the direct current regime and the voltage values were adjusted according to the sintering temperatures of 750 °C and 850 °C. The sintering process was carried out for 30 min and then cooled in the same atmosphere. As a result, a differentiated in morphology and wear properties were obtained in the sintered parts. At 750 °C a microstructure characterized by the stability of Ti grains with intermetallic precipitates such as CuTi2 and CuTi were observed at the interface with the matrix. On the other hand, the intermetallic phases as Cu3Ti, Cu4Ti and CuTi2 had been detected in the sintered at 850 °C. These phases were related to diffusive processes that occurred during the sintering, enhanced by the energy provided by the process. It had been observed that with increase of titanium content an improvement of the MMC tribological properties. In titanium contents of 20% at 750 °C was estimated a wear coefficient of $2.7 \times 10^{-8} \text{ mm}^3\cdot \text{N}^{-1}\cdot \text{m}^{-1}$ with a track width of 312 μm. Despite the pores originated during the sintering of MMC at 850 °C was found a wear coefficient value of $2.4 \times 10^{-8} \text{ mm}^3\cdot \text{N}^{-1}\cdot \text{m}^{-1}$ and a track size of 778 μm, related to the plastic deformation exerted on the porous structures during the wear process. Results of the tribological analysis lead that this compound may be applied in fields where the balance between adequate tribological properties and lightness are highly required such as the automotive, aeronautical and biomedical industries.

1. Introduction

Manufacture of components in powder metallurgy has gained interest in recent years due to the variety of shapes and applications that have been obtained through this technique. Currently, the powders of Cu-Ti-TiC system is matter of the exploration of the multiple uses and possibilities that this combination offers to the industry. For instance, in the improvement of the mechanical properties of copper based materials such as sound absorbers [1], heat sinks and resistance welding electrodes as pointed out by Ding et al [2]. Besides, the future uses of this system include implant components due to the high biocompatibility [3]. The sintering process pulvimetallurgical parts had been researched through unconventional methods means, originating techniques such as Spark Plasma Sintering (SPS) [4–8], but the cost of equipment limits its application in start-up industries. Techniques such as Abnormal Glow Discharge (AGD) [9–12] due to similar energy processes of SPS is presented as an alternative in the synthesis of powder metallurgical pieces. Therefore, AGD had been successful due to the low costs in the operation, as well as the results achieved through this technique in metallic and composite materials, promoting the densification and diffusive reactions.

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The Cu-Ti-TiC system has been studied due to expected excellent final properties in the mechanical and tribological performance [13–17] on reason of the challenge to understand the combination of the fragile nature between copper and titanium carbide. The incorporation of a third phase (metallic type) will provide an improvement of properties such as compatibility, wear resistance and hardness. In reason of that titanium is ideal candidate due to it had been observed hard phases in the Cu-Ti system in the conventional alloy [18] through the micrometric control of components. Laik et al [19] had reported that the control of the growth of the intermetallics as CuTi, CuTi4, Cu3Ti and Cu2Ti3 promote to obtain different hard layers that could provide an adequate mechanical response and interaction of the components [20]. Hence, in the synthesis of materials Cu-Ti system had been observed the formation of intermetallic phases in that enhance of the final properties such as wear and mechanical response [1, 21, 22]. Furthermore, it had been established that these types of components favor compatibility between metallic and ceramic aggregates.

The improvement of the morphological structure of the Cu-Ti-TiC system is of recent interest, in reason of the knowledge about this system is largely unknown in the literature and depends on the conditions of synthesis. In virtue of majority of studies has been focused on the development of copper-based compounds with interest in the TiC precipitations. As a consequence, disparate results had been found among researchers from precipitations with heterogeneous segregations to problems of embrittlement [2]. A starting point in the study of the Cu-Ti-TiC system had been supported by researches around the Cu-Ti-C system [13, 16, 17, 23], this had evidenced the predominance of morphologies with intermetallics-Cu-TiC phases. These studies had been focused on the analysis of the effect of the presence of the ceramic phase, leaving aside the examination of the influence that intermetallic reactions in the sintering of pure elements Cu-Ti-TiC in mechanical and tribological performance.

By virtue of this, the study of the improvement of the microstructure as well as the understanding of the morphology in materials constituted by a copper matrix with titanium additions is an essential element in the current development of this type of composites. However, there is no consensus on studies that relate the type of morphological behavior in compounds with a combinations of dispersed species of pure TiC and Ti as a raw material within composites. In accordance with the previous studies is necessary to understand the resulting microstructure and the improvement of the structure compounds through the improvement of the necessary conditions to the control of grow, synthesis, and reactions of intermetallics in this type of materials. The synthesis of composites Cu-Ti had been mainly explored through reactive milling or highly energetic process such as laser welding or reactions of explosive type. Nonetheless, the influence on the reactivity of components contribute by plasma-based processes such as AGD could influence of reactions (diffusive, precipitate formations and sinteribility) such as it had been observed similar characteristics of diffusion and high wettability of components [24] in other metallic systems and combinations.

In the present work had been investigated the effect of temperature sintering in the microstructure and tribological behavior of a Cu-XTi-1%TiC MMC sintered by abnormal glow discharge at 750 °C and 850 °C, with variable titanium contents of 10%, 15% and 20% wt%.

2. Methods

Copper (99% purity), titanium (99.5% purity), titanium carbide (99.95% purity) powders supplied by Sigma-Aldrich were selected to synthetized the composite. The average particle sizes of the components were 75 μm for copper, 60 μm for titanium and 4 μm for titanium carbide. Three mixtures were elaborated with different contents in titanium (mass fraction): 10%, 15% and 20%, maintained in each one of them 1% of titanium carbide. In order to obtain a homogeneous mixture and avoid agglomeration of the components were made liquid suspensions calibrated to 150 ml of 2-propanol. The mixtures were submitted to ultrasonic bathing (40W) for 10 min and magnetic stir at 1250 rpm for 30 min with subsequent drying at 50 °C for 1 h.

The mixed powders were consolidated in uniaxial pressure at 400 MPa, obtaining cylindrical specimens with a diameter of 10 mm and an average thickness of 5 mm. The green samples were sintered by AGD. The operating parameters were: direct current, pressure 2 Torr, inert atmosphere with a constant flow of 90 ml.min⁻¹ of argon and 10 ml.min⁻² of nitrogen, heating rate of 100 °C.min⁻¹, sintering temperatures of 750 °C and 850 °C for 30 min. The following factors were taken into account in the selection of the temperatures: on the one hand, the phase diagram of the Cu-Ti system [25] was considered at temperatures between 750 °C and 850 °C due to the possibility of the precipitations of hard intermetallic phases [26] in this zone of the diagram. Notwithstanding, the type of interactions between the metallic particles is largely unknown under the study conditions. Furthermore, this temperature range was used in reason of the interest found to evaluate these points below the high transition temperatures of titanium (890 °C). On the other hand, the studies [19, 27] indicate these temperature ranges of interest due to the possible to promote high modulus intermetallic phases with strong interaction with copper matrix. Finally, another factor of interest in the selection was linked to the study of the
morphological and mechanical influence of the use of low temperatures in the sintering of parts by AGD, using lower temperatures in comparison to previous studies in metallic systems [9, 11, 28], which had required higher energy contributions in the consolidation of metallic and ceramic particles. The voltage and current parameters to obtain these temperatures are summarized in table 1. The sintered MMC was cooled to room temperature in the sintering chamber in order to avoid oxidation.

MMC was observed through scanning electron microscopy on a LEO 430 operated at 30 kV coupled to EDS (Oxford instruments model 7089). X-ray diffraction (XRD) was used to determine the phases with a Pertpro PANalytical equipment was used with cobalt characteristic line wavelength. The diffractograms were taken using Bragg-Brentano geometry by sweeping the angle 2θ between 20° and 90°. The MMC were tested for microhardness Vickers on a Qualitest QV-1000AAT under a 100 gf load with a dwell time of 15 s with twenty measurements per sample for statistical purposes. Tribological tests were performed using the pin-on-disk technique on a Microtest MT/10/Ni machine with a hardened steel pin. The parameters of the test were: 300 m of wear distance, speed of 0.1 m.s⁻¹, normal load of 10 N and an environmental atmosphere at 20 °C with relative humidity of 52%. The coefficient of friction for the different synthesis conditions was determined in the machine. The characteristic wear coefficient of the different study variables was estimated using the equation (1):

\[
k = \frac{\text{loss of material volume [mm}^3\text{]}}{\text{applied load [N] } \times \text{sliding distance [m]}} \tag{1}
\]

3. Results

3.1. Microstructure and chemical composition

Figures 1 and 2 show SEM - backscattering electrons microstructure images of the sintered MMC sintered during 30 min at 750 °C and 850 °C, respectively. A market differentiation was observed between the sintered MMC, depending of the Ti contents.

| Temperature (°C) | Current (mA) | Voltage (V) |
|------------------|--------------|-------------|
| 750              | 135 ± 3,92   | 468,13 ± 7,64 |
| 850              | 197,75 ± 9,54| 592,21 ± 4,36 |

Figure 1. SEM-BSE images of the MMC Microstructure sintered at 750 °C.
between the titanium grains and the matrix. It was observed a homogeneous distribution in all cases in boundaries of Ti grains with different contrasts. The metallic aggregate in the grain edges was characterized by the presence of intermetallic phases as observed through the contrast differences.

In samples sintered at 850 °C (figure 2), the interaction between the titanium particles and the copper matrix is evident the influence of the sintering process. In Ti contents of 10%, a grain formation continues was exhibited surrounding the pores with microstructural precipitations close to the pore limits. At 15% and 20% Ti contents had a similar distribution around the pores, as well as a formation of similar structures. There was a strong connection between the concentration and the final configuration of the microstructure of the system.

In figure 3 are shown the results of chemical variation in regions inside the material in the sintered MMC at 750 °C. The metallic aggregate in the particle edges were characterized by the presence of intermetallic phases with contrast differences. Towards the matrix with increases of titanium were observed Cu(Ti), CuTi, CuTi2, CuTi3 and Cu4Ti3.

In figure 4 are shown the results obtained in regions of interest inside titanium particles. It had been evidenced that these results are in accordance with the profiles of the figure 3. Intermetallic phases such as CuTi, CuTi2, CuTi3 due to the contrast differences were recorded in the 10% and 20% mixtures, and in minority Cu4Ti3 was observed in concentrations of 20%.

In figure 5 are shown the compositional profiles of the samples subjected to sintering at 850 °C. The regions with solubilized titanium within the material had been observed through of different interactions.

The following results had been recorded: At 10% Ti intermetallics towards the matrix were evidenced in form of Cu4Ti phase. The solubilizing mechanism of titanium promoted the formation of the CuTi in interfacial regions in the bordering regions between the matrix and titanium. There was the presence of CuTi2 and formations of the CuTi2 + Ti, this type of precipitates was due to decompositions of CuTi3 in simpler phases. The results with contents of 15% were observed the appearance of demarcated intermetallics formations such as CuTi, Cu4Ti and Cu3Ti, Cu4Ti3. In the results were observed the presence of the predominant CuTi with CuTi2 intercalations. Lamellar microstructure was observed in figure 5 and the compositional profiles. These phases had provided the stability of the titanium diffused from the areas where it was before sintering (pores), this can be explained according to the diffusive characteristics described by Uzunov et al [29] in relation to CuTi and CuTi2 during their formation in order to promote the stabilization of phases with higher titanium content such as Cu3Ti. The Ti-Ni system in the early work of Igharo et al [30] are a starting point to explain this type of pore configuration due to diffusion processes and intermetallic formation in sintered parts.

In figure 6 are shown results of chemical details of interest within the material under the different compositions.

Figure 6 presents additional details about the type of interactions that occur in the material during sintering: in concentrations of 10%, Cu4Ti phase was established as a solubilization component of titanium in the matrix. The intermetallics such as CuTi2, CuTi and Cu4Ti3 were supported on the Cu3Ti phase. At 15% the presence of the Cu4Ti and Cu3Ti phases near the pore boundary and alpha titanium indicate the reaction βTi(Cu) = αTi + CuTi2 during cooling [31]. The regions in compositions of 20% had the same behavior as in the other concentrations with intermetallic aggregates of Cu3Ti and Cu4Ti. The characteristics of the structures...
detected in this composition was related to the CuTi$_2$ and CuTi, which were located in groups surrounded by intermetallic phases with high copper contents. The origin of many of the porosities was related to the interdifusive reaction, a cause of the dominance of intermetallic creation consumes the energy to continue a mass transport to the spaces left by titanium during the reaction with copper.

3.2. DRX results

DRX are presented in figure 7. A significant difference was established between the phases obtained at 750 °C and 850 °C.

The diffractograms confirmed the same results found in the chemical microanalysis by EDS. In the samples at 750 °C are shown a marked signals corresponding to copper and titanium. The micrographs and microanalyses were detailed stability of the titanium particles within the matrix, but it was manifested a diversity of phases close to the grain edges. However, these phases due to their low volumetric content do not represent a strong signal in the diffractograms under this sintering temperature.

On the other hand, at 850 °C a variation was detected between the different concentrations of titanium: at 10% was observed CuTi$_2$, Cu$_3$Ti due to the low concentration of titanium. In the 15% were determinated CuTi$_2$ and Cu$_3$Ti. The signal associated to the Cu$_4$Ti$_3$ and α-Ti were shown in a minority way due to their low volumetric concentration. Cu phase presents a low indexation, which was responded by a greater amount of titanium responsible for reacting with the matrix in order to generate intermetallics. At 20% Ti, signals of Cu$_3$Ti and CuTi$_2$ had been mainly characterized in the composite, suggesting the high solubilization Cu-Ti under the synthesis conditions of the present study.

3.3. Microhardness of the MMC

It had been evidenced that the concentration of titanium increased the microhardness of material, compared to target the values increase gradually. The results under the different synthesis conditions are shown in figure 8.
According to the data in figure 8 the addition of titanium had positive effects on the microhardness of the compounds compared with values of 40.34 HV at 750 °C and 45.92 HV at 850 °C in the targets. The results of hardness at 750 °C with the titanium variation from 10% to 20% presented a microhardness value of 140.4 HV, 234.85 HV and 321.28 HV. The synthesis condition at 850 °C exhibited the highest hardness values with respect to the other synthesis conditions, despite the porous at 10%, 15% and 20% had been estimated a microhardness close to 291.36 HV, 403.17 HV, and 457.62 HV, respectively.

3.4. Tribological performance

The characteristic tribograms under the different synthesis conditions are shown in figure 9:

At 750 °C in general was shown a zone of stability over 100 meters of sliding wear and with the titanium increases a decrease in coefficient of friction (CoF). The lowest coefficient of friction was found with contents greater than 15%. In all cases samples with contents of 10% showed a similar behavior compared to the target. The experimental results of the CoF in the samples synthesized at 750 °C and 850 °C are reported in table 2.

In the sintered composites at 850 °C the behavior the CoF was reduced with the increase of titanium, generating in the tribological pairs with concentrations higher than 15% suitable for applications where it is required low values of friction between the components.

The high hardness of the samples under sintering at 850 °C provided the higher wear resistance (table 2). The difference observed between the two temperatures was due to the presence of porosities. In spite of the porosity that had been observed in this condition (850 °C), values of coefficient of friction were no superior to the target. This could be established by the influence of the porosities that did not degenerate strongly the final wear properties.

In concentrations of 10%, a decrease in wear resistance was observed in comparison with target, but this behavior was stood with the increase of titanium. Despite the results showing a low wear values (in proportion of $10^{-6}$) compared to those experienced by the target ($10^{-5}$).
The behavior of the macroporosities observed in the samples at 850 °C against wear was related to factors such as: composition of various hard phases that lodge the porosities. These pores created a resistance to wear by becoming sinks of the materials detached during wear in view of the nature of the debris, that agglomerated by cause of compressive phenomena during the test, originating regions that improved the resistance against wear.

The micrographs in figure 10 had shown a smaller track extension with increments of titanium. This phenomenon was remarked in concentration superior to 15%. In the levels of 20% the morphology was typical of a deformed material. This effect was explained by the intergranular reactivity in the titanium and copper grains due to the intermetallic phases, which to establish contact between the parts that inhibited the displacement of the matrix in a pronounced way.

In figure 11 are presented the wear profiles of the samples at 750 °C. There was an influence on the final deformation of the wear track as the titanium increments. It was revealed a decrease in the maximum depth of the track of approximately half in contrasts to the target. The observation of more pronounced peaks and valleys with the incorporation of titanium suggest a degradation of the material in a heterogeneous form, this effect was increased by limit zones (peaks) that inhibited the rapid material detachment. In these regions an accumulation of material was generated as a result of compressive phenomena in the micro porosities of the MMC. In figure 12 are presented the results of EDS of the wear track.

It had been observed a fine distribution of iron on the tracks, especially in concentrations higher than 15%. The mechanical characteristic stability of titanium had been noticed without detecting diffusive effects or abrupt detachments of particles. The wear tracks of the sintered at 850 °C are presented in the figure 13. The
morphology in wear track distribution had the presence of a mark in the direction of the test. There was a gradual reduction in the final track with titanium increments, this reduction was significant under compositions of 20%.

In figure 14 are shown the profiles of the wear tracks of the composites at 850 °C.

In concentrations greater at 15% Ti samples were analyzed some regions with accumulations of material on the tracks as a response to the pin-sample wear system. The size of the track of the composites sintered at 850 °C did not increase abruptly, which suggested that porosity did not deteriorate this characteristic. The maximum depth (figure 14) by the composite against wear had a significant variation with the titanium increasing, in concentrations of 10% decreased with respect to the target. In contents superior to 15% had a significant

**Figure 6.** Dots by EDS in areas of interest of the MMC at 10%, 15% and 20% Ti with compaction at T = 850 °C.

**Figure 7.** DRX diffractograms under 750 °C and 850 °C.

| Point | Concentration (at.%) | Phase |
|-------|----------------------|-------|
| Cu    | Ti                   |       |
| 1     | 11.08                | βTi(Cu) |
| 2     | 50.45                | CuTi  |
| 3     | 80.55                | Cu₂Ti |
| 4     | 56.41                | Cu₁Ti₂ |
| 5     | 34.79                | Cu₁Ti₂ |
| 6     | 79.86                | Cu₂Ti |

| Point | Concentration (at.%) | Phase |
|-------|----------------------|-------|
| Cu    | Ti                   |       |
| 1     | 47.17                | Cu₄Ti |
| 2     | 8.88                 | αTi(Cu) |
| 3     | 74.07                | Cu₄Ti |
| 4     | 78.54                | Cu₁Cu₄Ti |
| 5     | 49.84                | Cu₁Ti₂ |

| Point | Concentration (at.%) | Phase |
|-------|----------------------|-------|
| Cu    | Ti                   |       |
| 1     | 76.83                | Cu₄Ti |
| 2     | 51.70                | Cu₄Ti |
| 3     | 79.76                | Cu₁Cu₄Ti |
| 4     | 61.08                | Cu₁Ti₂ |
| 5     | 34.11                | Cu₁Ti₂ |
| 6     | 48.90                | Cu₄Ti |
reduction of the deformation in comparison with the other conditions of synthesis. The wear tracks in figure 15 had exhibited a demarcation of the iron on the track in relation to the titanium increments. The results by the EDS spectrum had shown the highest iron concentration on the track compared to peers at lower temperature. The distribution of iron in this case had been presented in a marked way on the tracks of wear, observing homogeneity on the path of the test. The EDS spectrum had shown that the presence of iron was related to a process of abrasion-adhesion between the MMC and pin, particularly it was presented that the iron contents greater than observed in the tracks of the samples at 750 °C in titanium concentrations superior to 15%.

Figure 8. Microhardness of MCC under the conditions of study.

Figure 9. Tribograms under the different conditions of synthesis.

| Sample | Coefficient of friction (u.a) 750 °C | Coefficient of friction (u.a) 850 °C | Wear coefficient (x10^{-5} mm³.N⁻¹.m⁻¹) 750 °C | Wear coefficient (x10^{-5} mm³.N⁻¹.m⁻¹) 850 °C |
|--------|-----------------------------------|-----------------------------------|--------------------------------|--------------------------------|
| Target | 0.7947 ± 0.0162                   | 0.7164 ± 0.0203                   | 1.1893                        | 1.0629                        |
| 10% Ti | 0.7559 ± 0.0258                   | 0.2077 ± 0.0124                   | 0.0958                         | 0.2499                        |
| 15% Ti | 0.3635 ± 0.0141                   | 0.1889 ± 0.0022                   | 0.0135                         | 0.0135                        |
| 20% Ti | 0.1268 ± 0.0014                   | 0.1287 ± 0.0154                   | 0.0027                         | 0.0024                        |
4. Discussion

4.1. Microstructure of the composites

At 750 °C was presented CuTi3, its formation was propitiated in the initial stages of the genesis of other intermetallics. The existence of this component inside the titanium grains suggest the following reaction: 

\[ \text{CuTi} + 2\text{Ti} = \text{CuTi}_3 \]

The fact that CuTi component was the first to appear due to its stability promoted the reaction with the surrounding titanium. Moreover, CuTi2 is a component with a rich percentage of titanium close to 66.6% and its presence was favored by less stable intermetallics such as CuTi3 that tend to decompose (\[ \text{CuTi}_3 = \text{CuTi}_2 + \text{Ti} \]), this cycle of decomposition and recomposition of this intermetallics explains the low presence [31]. In figures 5 and 6 were shown the results in regions of interest within the titanium grains and it had been evidenced that these results in accordance with those recorded in the profiles. In the 10% and 20% titanium contents was evidenced CuTi2 phase inside the titanium grains. Likewise, intermetallic phases such as the CuTi due to the contrast difference in the 15% case were recorded in the 10% and 20% mixtures. In the compositional profiles were presented in the majority dilution of titanium in copper through the conformation of intermetallic titanium as the case of the Cu4Ti3. These phases were presented as a reaction product between copper and titanium in reason to the strong precipitation of other products as CuTi. Additionally, the solubilization of titanium was evidenced in the form of Cu(Ti) and Cu4Ti in the profiles under the same level of mixture.

The stability of intermetallic sintered at 750 °C had been reported in this temperature range (750 °C–790 °C) by [16], favoring the formation of intermetallic in the following way: CuTi > Cu4Ti3 > CuTi2. However, the CuTi and CuTi2 in the present study were recurring in comparison to Cu4Ti3 that require a longer time to promote their growth. Another important element was the low formation enthalpy of CuTi and CuTi2 with titanium increments [32, 33], and the Gibbs energy associated to the precipitations of these phases at 750 °C [6, 33]: \( \Delta G_{\text{CuTi}} = -15.71 \text{ kJ.mol}^{-1} \), \( \Delta G_{\text{CuTi2}} = -22.0 \text{ kJ.mol}^{-1} \), \( \Delta G_{\text{Cu4Ti3}} = -7.41 \text{ kJ.mol}^{-1} \). These values had demonstrated the stability of the intermetallics in the grain contours under the sintering temperature. The low presence of these phases is linked to the long incubation time that require in order to form homogeneous layers in this temperature range.

In contrast, at 850 °C had a microstructure characterized by the presence of macroporosities and an absence of titanium delimited zones, as in the lower temperature cases. The phases indexed in the samples at 850 °C had presented an evolution of the intermetallic appearance; the Cu4Ti appeared gradually with the increase of titanium, being more demarcated its presence with contents higher than 15%, besides CuTi2 with minority phases (\( \alpha \text{Ti} \) and Cu4Ti3) were presented in the diffractograms. The appearance of the Cu4Ti and CuTi phases in some study regions, and the non-indexation of this intermetallics in the diffractograms was associated with a low volumetric concentration.

Figure 10. Morphology of wear marks for compact samples sintered at 750 °C.
The high diffusion of copper towards titanium by virtue of the transformation of titanium to its β-form with cubic structure (BCC) facilitated the diffusion of titanium towards copper. This effect is related to that titanium within the copper matrix increases the tendency to allotropic transformation, especially in the presence of beta-stabilizing agents such as copper or nickel [34]. At temperatures above 805 °C had been observed the allotropic transformation up to 905 °C [35, 36]. In addition, the high difference between the thermal conductivity of titanium and copper [37] (Ti: 15.7 W.K⁻¹.m⁻¹, Cu: 393.6 W.K⁻¹.m⁻¹) produced the formation of heat.

**Figure 11.** Wear profiles for compositions: target, 10%, 15%, 20% Ti compact at 400 MPa under sintering at 750 °C.
concentrations inside the titanium grains by a lower heat dissipation. As a result, this concentration of energy in reason of the Joule effect and cathodic emission of the discharge due to predominantly metallic particles promote in the external zone of the titanium particle in contact with the matrix a greater possibility of dissipating the heat through the interface with the copper, which promoted a strong interaction with the matrix (Cu-TiC).

Thus, areas with heat concentrations permitted a greater diffusion towards the outside which with time increased the pore size.

The appearance of intermetallics at 850 °C was favored in the following way: CuTi$_2$ > CuTi > Cu$_3$Ti. It had been shown CuTi$_2$, Cu$_3$Ti and CuTi with recurrence inside the material in the different concentrations due to the Gibbs energy in this range of temperature [6, 33]: $\Delta G_{\text{CuTi}_2} = -20.59 \text{kJ.mol}^{-1}$, $\Delta G_{\text{CuTi}} = -15.06 \text{kJ.mol}^{-1}$, $\Delta G_{\text{Cu}_3\text{Ti}} = -14.24 \text{kJ.mol}^{-1}$. The presence of CuTi was observed in several of the microanalyses and the stability analysis shown that its existence was possible due to the low concentration. This low concentration may be due to interaction with other phases, as had been observed with Cu$_4$Ti$_3$ in the reaction: Cu$_4$Ti$_3$ + 8CuTi = 3Cu$_4$Ti$_3$. This reaction had been reported in metallic powders of the Cu-Ti [19] system, the appearance of Cu$_4$Ti at temperatures higher than 790 °C had a shorter incubation time due to the greater mobility of the atoms, but its metastable character affects the extensive presence. The production of Cu$_4$Ti$_3$ was limited to the appearance of Cu$_4$Ti, which tends to follow mainly the partial decomposition reaction [33] Cu$_4$Ti = Cu$_3$Ti $+$ Cu. The Cu$_4$Ti was observed in reason of reaction of the Cu$_4$Ti in copper-saturated regions [29, 38]. This reaction stabilized Cu$_4$Ti in more stable phases like Cu$_3$Ti. However, the Cu$_4$Ti phase was

Figure 12. Mapping and spectrum by EDS of the wear track of target, 10%, 15% and 20% sintered MMC at 750 °C.
observed in some regions of chemical microanalysis with unreacted titanium that delays the decomposition reaction.

According to the results at 750 °C in the diffusive and reactive process CuTi and CuTi₂ were the first to form precipitations in the composite. The relations with other regions was increased with the temperature at 850 °C, as a result the higher concentrations of titanium and temperature promoted the evolution towards the formation of intermetallic with a greater number of Ti atoms that stimulated the evolution of the lattice. In reason to this was observed the evolution since FCC (Cu) and HCP (Ti) to phases such as: Cu₃Ti and βCu₄Ti. These are structures of orthorhombic type with crystal class: Pmmn and Pmma [26], respectively. In contrast with the tetragonal system of the class P4/nmm and I4/mmm in the CuTi and CuTi₂ phases [32], originated in the first stages of the precipitations during the sintering process. Consequently, Li et al [39] had pointed out that the phases with the highest ductility tendency in the Cu-Ti system are those of a tetragonal nature in contrast with orthorhombic phases. This suggests a point of compression about the influence of these phases on the tribological and mechanical behavior as per the composition and sintering temperature in the manufacture of this composites.

The precipitations were in the following order: Ti in combination with CuTi in reason of the high reactive and diffusive character tended to form intermetals through the reaction [29]: CuTi + βTi = CuTi₂, this reaction had been reported at temperatures above 790 °C [19]. The CuTi₂ was reported in diffractograms and chemical microanalysis by the stability of this. An additional phase was observed at 15% and 20%, the presence of peaks associated with the presence of αTi and CuTi₃ in sintered composite. In reason that βTi(Cu) were not
saturated with copper in the diffusion, undergo a transformation of eutectoid type at 790 °C during cooling, which followed the reaction: \( \beta \text{Ti(Cu)} = \alpha \text{Ti} + \text{CuTi}_3 \) \((\Delta G_f = -5.42 \text{ kJ mol}^{-1})\) \cite{31}, by cause of that \( \beta \text{Ti} \) (Cu) was a minority phase due to decomposition reaction. This was explanation for the low indexation of the \( \alpha \text{Ti} \) and \( \text{CuTi}_3 \) phases and the presence in the chemical microanalyses of \( \text{CuTi}_3 \) in form of \( \text{CuTi}_2 + \text{Ti} \) in contours of regions in the microstructures.

Additionally, the behavior of the precipitations of the intermetallic phases was related to the interfacial energy (IE) between the components. Based on calculations provided by Kundu \textit{et al} \cite{40}; it had been observed values of IE in \( \text{CuTi}, \text{CuTi}_2 \) of 233.56 mJ.m\(^{-2}\) and 217.36 mJ.m\(^{-2}\), respectively at 750 °C. These values in this temperature range suggest that not only the stability of the phases was according to Gibbs’ energy. But rather the low interfacial energy associated with \( \text{CuTi}_2 \) played an important role, causing the formation of this intermetallic layers in the contours of the titanium particles. On the other hand, taking into account the results of interfacial energy at 850 °C \cite{40} had been observed that the presence of \( \text{CuTi}_2, \text{CuTi}, \text{Cu}_4\text{Ti}_3 \) phases was related to the low interfacial energy that these intermetallics with values close to 208.25 mJ.m\(^{-2}\), 224.33 mJ.m\(^{-2}\) and 229.56 mJ.m\(^{-2}\), respectively. These phases were reported due to the low interfacial energy, but the low presence is related to the reaction with other components to form other intermetallics. The formation of \( \text{Cu}_4\text{Ti}_3 \) is linked to reduction of interfacial energy greater than provided for \( \text{CuTi}, \text{CuTi}_2, \text{Cu}_4\text{Ti}_3 \) in the reactions between Cu-Ti and first intermetallics of the sintering process, in combination with TiC particles that modified the nucleation of complex phases as reported by Zhang \textit{et al} \cite{41}.

The considered system has not been widely studied under the developed synthesis conditions, thereby some interesting investigations can serve as a basis for the comparison and understanding of the observed microstructure. Previous works such as Karakulak \textit{et al} \cite{42} had evidenced the formation of intermetallics in the contours of the titanium particles in spite of employing short sintering times. This research had suggested that the use of longer sintering times and higher temperatures could increase the production of intermetallic layers. As a consequence, the present results validated the work of Ružič \textit{et al} \cite{43}, this proved that the possibility in formation of huge intermetallic layers at low temperatures of about 550 °C and times of up to 20 h with pure copper-titanium powder combinations. Thus, Hao \textit{et al} \cite{44} had shown that the production of these intermetallics at low temperatures plays an important role in facilitating diffusion from hard particles, as well as in the configuration of microstructures similar to those observed in the present work. As a result, this allows us to understand an additional factor in the explanation of the high diffusion that is obtained in the composite at 850 °C.

Finally, porosity was related to a difference in the diffusion mechanisms of copper and titanium; primarily due to the stabilization of the \( \beta \text{Ti} \), favored by the presence of a strong beta-stabilizer such as copper. BCC structures characteristic of \( \beta \text{Ti} \) is known as a crystalline system that promotes the diffusion in solid state, especially among other structures such as the FCC of copper. In this condition, copper diffused more rapidly towards titanium than titanium towards copper. The titanium particles were covered by the diffusive reactions
on their surface, generating a progressive net loss of atoms that are displaced towards the areas richer in copper, creating porosities due to the diffusion in the regions were located the titanium particles. Additionally, the difference between the thermal expansion values between the copper-titanium and the intermetallics generated the expansion of the material, leaving the pores behind in the regions of titanium particles. This type of porosity had been observed in powder metallurgical materials of the Ti-Ni system, which presented similar mechanisms of intermetallic creation and high diffusion Ni towards Ti with the subsequent formation of porosities with

Figure 14. Wear profiles for compositions: target, 10%, 15%, 20% Ti compact at 400 MPa under sintering at 830 °C.
similar crystalline behavior (Ni: FCC; Ti: BCC) as well as the influence of the beta stabilization of titanium [30, 45, 46].

4.2. Microhardness of the material.
At 750 °C, it was found intermetallics in the grain boundaries CuTi₂ and CuTi. These intermetallics have a high hardness of approximately 800.20 HV and 866.46 HV [32, 39], respectively. These hard formations distributed in the ductile matrix, generated zones in which the deformation of the matrix was interrupted by the interaction with these regions of high hardness; in comparison to the matrix (~40 HV) that contained the mixed phase Cu-Cu(Ti)minority and the grains of Ti (304 HV) [47], that lodge these regions in the limits.

The results at 850 °C shown the presence of a rich phase Cu₃Ti accompanied by CuTi₂, CuTi, Cu₄Ti₃ in minority. These phases produced a MMC with moderately ductile precipitates within the intermetallic group of the Cu-Ti system with an average hardness of 544.34 HV for Cu₃Ti [39], which together with the intermetallic CuTi₂, CuTi (533 HV) [37], and Cu₄Ti₃ (587.15 HV) provided high hardness values. At 20%, a microhardness of 457.62 HV was presented as the highest value compared to the other study conditions.

The majority presence of Cu₃Ti and CuTi₂ phases was detected in this concentration, in contrast with the theoretical values by this type of phases, it would be expected that the MMC had higher value (figure 8). This effect was due to the porosity exhibited by the MMC, but this did not deteriorate the properties dramatically to values lower than the target. As a result, porosity is a factor in the deviation of the values observed in the literature for this type of precipitations and these porous formations were not involved by ductile phases, in particular its formation was surrounded by intermetallic regions of high hardness as CuTi₂ and CuTi. The interfaces that
constituted the pores were of high hardness, which generated regions to contain the efforts in comparison to materials without titanium and intermetallics (high ductility).

The hardness results are in accordance with the behavior observed by previous investigations where the titanium increase had a positive effect on the hardness value, especially in values higher than 5% [8, 42, 48]. The studies carried out by Akbarpour et al [20] present a close reference to the hardness values found in the composites, despite the need for an aging process in order to promote intermetallic growth and the elimination of the thermal stress of the sintering to the extent that ideal values proposed by Shon et al [49]. Finally, despite the observations of pores at 850 °C had been observed that their presence does not significantly deteriorate the hardness of the study system as pointed out by Zhao et al [50]. Besides by virtue of the presence of intermetallics of the Cu-Ti system that confer a high modulus characteristic and an engineering potential due to the lightness and high wear resistance.

4.3. Tribological behavior
It was assessing at 750 °C with the increases of titanium the presence of iron on the wear tracks. This fact was related to the presence of a discontinuous phase of greater hardness as the titanium and the intermetallics in the grain boundaries in comparison with the matrix. It had been created a multiple wear process with the abrasion of the soft regions, leaving exposed areas of high hardness with angular nature with less tendency to deformation. Therefore, the abrasion of the pin was deposited in part of its material on the track. In general, at 750 °C and 850 °C the materials were released to debris by the counterpart, that compressive factors and the heat in the...
friction surrounding atmosphere promoted the adhesion of these materials on the wear track. These materials are mixed oxides of Fe-Ti-Cu in reason of the stability generated a decrease in the friction between the sphere and the MMC. These layers adhered to the track in reason of their nature tended to detach with less difficulty in contrast to materials subjected to total abrasion and the formation of this condition contributed positively to the final properties against the wear. This fact can be seen with increases greater than 15%, there was a greater presence of iron and oxygen in this process. Likewise, this layers provided another explanation for the low coefficients of friction and wear reported for higher titanium contents, especially when the intermetallics increase in the composite that promoted regions of the high hardness.

MMC’s at 850 °C despite the porosity of the compounds had exhibited a higher wear resistance compared at a lower temperature. It may be controversial to observe porous structures in which no deterioration of properties at levels below target or those expressed by materials of the same nature. This behavior responds to a topic of recent interest; the influence of the final properties against the wear of the material is not only determined by the porosities. The morphology, distribution and chemical composition of the pore walls influenced the behavior between pore-surfaces and the wear counterpart [51, 52]. On account this, pores acted as sinks of the materials released with less cohesive energy [53] in reason of the compaction and agglomeration phenomena in the wear, prolonging the usefulness of the composite. Another factor of the behavior exhibited by the compounds at 850 °C was related to wall-surfaces in pores, by virtue of high tribological properties in phases such as Cu3Ti, CuTi2, Cu4Ti3 with high resistance to wear [1, 50].

Similar tribological systems of interaction between parts with intermetallics of the laminar type in the titanium particles and the friction generated by a steel pin had been observed in previous studies [42], evidencing that the increase of titanium has a positive effect on the decrease of wear rates and wear coefficient. In addition, the increase of intermetallic layers as described by Yan et al [33] has a positive effect under wear conditions in cause of the compacted oxide films formation by the wear processes on the surface and greatly reduce the wear rate between the friction counterparts. In the case of the composites at 850 °C due to the high hardness of the phases at this temperature; adhesive effects can be observed in a similar tribological pair as described in previous works [54], in view of the mechanical differences between the intermetallic propagation and the pin, generating an adhesion of pin materials on the surface of study due to the decrease of plastic deformation and the smaller contact area.

5. Conclusions

It had been established an influence on the microstructure, hardness and tribological properties of a Cu-Ti-1% TiC MMC according to the titanium concentration and sintering temperature in the Abnormal Glow Discharge. It had been observed with temperatures of 750 °C the precipitation of intermetallic phases (CuTi, CuTi2, CuTi3) on the grain boundaries of the titanium, improving the hardness and the resistance against the wear. This effect was increased with the concentration of titanium in the composite, achieving hardness of 321 HV and a wear rate of $2.7 \times 10^{-8}$ mm$^3$.N$^{-1}$.m$^{-1}$ in concentrations of 20%. It had been observed lower deformation in the wear track in the composites sintered at this temperature, by cause of the stability in titanium particles and layers of intermetallics. On the other hand, the generation of a porous microstructure with a distribution of different intermetallic formations (Cu3Ti, Cu4Ti, CuTi2) had been observed at 850 °C. It had been estimated high hardness up to 457 HV with concentrations of the 20% at 850 °C. In spite of pore morphology was assessed with titanium contents higher than 20% a wear coefficient of $2.4 \times 10^{-8}$ mm$^3$.N$^{-1}$.m$^{-1}$. This results were attributed to the influence of the intermetallic phases and redeposit of materials in the pores, favoring the performance of the composite. This material is recommended according to the morphology (lightness and porosity), tribological and mechanical needs to obtain in the design of devices and machines in the automotive, aeronautical and biomechanical world. It is recommended in the case of sintered composites at 850 °C a subsequent thermo-mechanical treatment in order to seal pores and increase the homogeneity of the hard-phases.

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