Mechanical and Microestructural Properties of Titanium Matrix Composites Reinforced by TiN Particles

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Abstract

Particulate reinforced titanium composites were produced by PM rout. Differents volumetric percentages of TiN reinforcements were used, 5,10,15 vol%. Samples were uniaxial pressed and vacuum sintered at differents temperatures between 1200-1300ºC. Density, porosity, shrinkage, mechanical properties and microstructure were studied. Elastic properties and strength resistance were analysed by flexural strength and tension tests, and after the test, fractured samples were analysed too, obtaining a correlation between the fracture, interparticulated or intraparticulated, and the reinforcement addition.. Hardness and microhardness test were applied too, in order to complete the study about mechanical properties . In order to study wear resistance pin-on-disc test were used. In addition, the temperature influence, the reactivity between matrix and reinforcement, and the microstructures developed were observed by optical and electron microscopy.

Keywords : titanium matrix composites, powder metallurgy, TiN reinforcement, wear resistance

1. Introduction

Titanium and its alloys stand out primarily due to their specific strength and excellent corrosion resistance, this explains their early success in the aerospace and chemical industries, but other markets such as architecture, chemical processing, medicine, power generation, marine and offshore, sports and leisure, and transportation are seeing increased application of titanium [1]. Only at temperatures below 300ºC do carbon fibre reinforced plastics have a higher specific strength than titanium alloys. At higher temperatures the specific strength of titanium alloys is particularly attractive.

Titanium nitride is a member of the refractory transition metal nitrides family which exhibit characteristics of both covalent and metallic compounds. The most remarkable properties of TiN materials are its high hardness and its very good resistance to wear corrosion [2].

One of the effective approaches has involved the development of the metal matrix composites utilizing particulate additions as the reinforcing phase. Remelt casting and powder metallurgy (PM) processing are the two main procedures for fabricating particle-reinforced metal matrix composites (PMMMCs). As compared with casting procedure, in addition to inherent economic benefits, PM procedure has the advantage of greatly reduced particle/matrix interfacial reaction and more homogeneous particle distribution in the matrix.

Titanium products fabricated by powder metallurgy techniques can be divided into two general categories: blended elemental PM [3] or prealloyed PM. In this work blended elemental products are used, in which a blend of elemental powders, along with master alloy or other desired additions, is cold pressed into shape and subsequently sintered to higher density and uniform chemistry.

In this work we intend to put together the benefits of Titanium as matrix and the TiN compound as reinforcements to develop materials with improved properties.

2. Experimental procedure

Materials. In this work two different materials in powder state are used. The matrix material is titanium powder, supplied by Atlantic Equipment Engineers (AEE), a Division of Micron Metals Inc., Ti quality 3 powder, with average particulate size of -325 mesh and with polygonal shape, obtained by Kroll process. This matrix powder has been mixed with a titanium compounds, TiN powder, supplied by Alfa-Aesar, 99.8% purity, with average particulate size of 5 microns and polygonal shape too. Different titanium matrix composites are obtained by mixing titanium powder with different percentages of reinforcement particles. The mixtures obtained are titanium with 5, 10 and 15 vol.% of TiN.

Powder metallurgy route. The mixtures are homogenised in a ball mill, the milling produced by 30 alumina balls with 15 mm diameter size in a alumina jar, and the process had a duration about 2 hours turning about 1.5 cicles/s. After milling process the powders has been consolidated in a uniaxial press, Instron 1343, obtaining samples in a corresponding matrix. Compaction has been carried out with a velocity about 5 MPa/s, to a maximum pressure of 680 MPa. After the compaction process, the

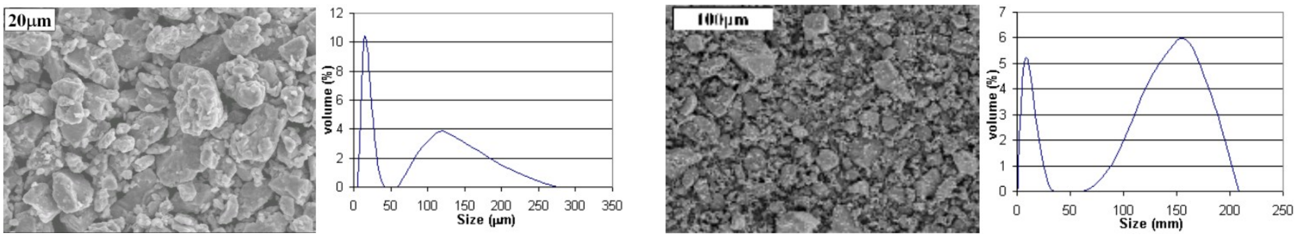


Fig. 1. a) Ti powder SEM image and particle size distribution. b) TiN powder SEM image and particle size distribution

green samples have been sintered in a tubular vacuum furnace, Carbolite model HVT/15/75/450. Samples are sintered at different temperatures in a range from 1200ºC to 1300ºC. The sintering cycle is defined by a heating rate of 15ºC/min from 800ºC and a permanence of 30 minutes, a second heating rate of 10ºC/min from final temperature and a permanence of 1 hour, and cooling in the furnace to room temperature. The sintering process has been realised in vacuum of about 10-4 bar.

Characterization. Powdered materials have been characterized by scanning electron microscopy, Jeol 6300, and particle shape, size and surfaces are studied.

After compaction, the green samples have been measured to compare green dimension with dimensions after sintering, to analyse densification. Porosity in green samples has been studied by Archimedes technique and mechanical properties has been studied by flexion test. Densification of samples is analysed both by measuring the samples prior and after sintering and by means of the study of evolution of porosity during sintering.

Flexural strength was evaluated by flexion test followed by a fractographic study by means of scanning electron microscopy. Dry sliding wear experiments were carried out with a pin-on-disc equipment, the pin was a chrome steel ball of 4 mm diameter, and normal load of 10 N was applied to the pin. The electronic balance which is able to weigh up to 10^{-4} g was used to measure the weight loss of the specimens. And for a better understanding of mechanical behaviour, hardness and microhardness measurements were also carried out in addition. Finally, analysis of the microstructure evolution and modification due to the different sintering conditions has been carried out by means of optical and scanning electron microscopy.

3. Results

In order to know the physical behavior of different composite materials Figures 2 and 3 show the shrinkage happened in the sintering process and geometrical density measured after sintering process respectively. Figure 1 shows in all cases a tendency to increase the shrinkage with the temperature, and the greater shrinkage was happened in pure titanium material, decreasing with the addition of TiN particles reinforcement.

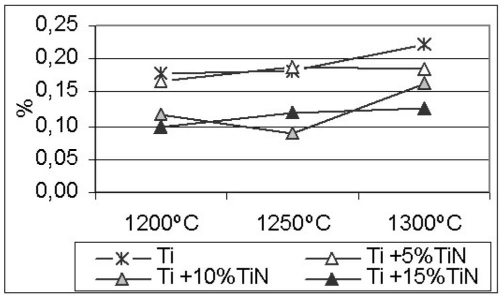


Fig. 2. Shrinkage in titanium and titanium matrix composites with TiN

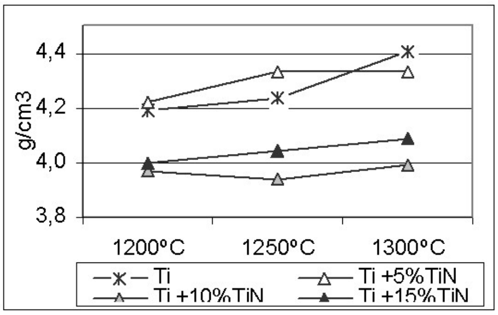


Fig. 3. Density versus sintering temperature.

In case of the geometrical density study, Figure 2 shows the results obtained. In general density increases if sintering temperature increases. Greater densities were observed in samples with low contents of reinforcement, the greater density was observed for 5%TiN reinforced samples, followed by pure titanium ones, this phenomena is due to the respectively materials density have influence in the final geometrical density, and TiN density is 5.29 g/cm^3 respect to Ti density, 4.507 g/cm^3. In the other hand greater addition of reinforcement induce greater porosities in the samples producing the minor geometric density in samples with high contents of TiN particles.

Mechanical properties was analysed with different mechanical test. Materials hardness was studied and results are show in Figure 4. According to Figure 3, a greater hardness was obtained adding TiN particles to the titanium matrix, these phenomena is due to the good dispersion of TiN particles in the matrix. TiN particles had less size than titanium powder and had a grain size reduction effect. TiN particles had a very high hardness but high percentage of reinforcement additions produces porosity increases too, for these reason the best result are not for 15% addition. Figure 5 shows the materials behaviour in flexural strength test. Flexural strength in pure titanium decreases at high temperatures, probably due to de grain growth. In the other hand TiN addition decreases dramatically de flexural strength, specially for high content of reinforcement. Complementing the flexural strength study with the fractures electron microscopy analysis of the fractures, a fragile typology of fracture was observed, Figure 6. Concluding, titanium matrix composites had an increase in the hardness and in the fragility with the addition of TiN particles reinforcement.

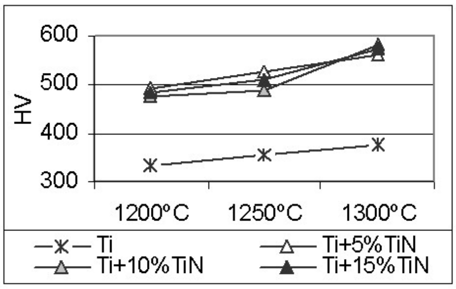


Fig. 4. Hardness versus sintering temperature and TiN addition.

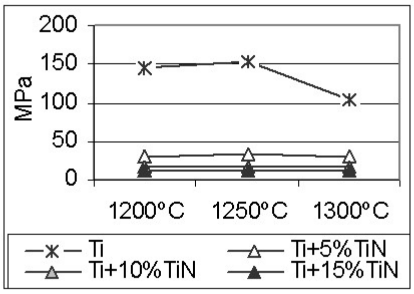


Fig. 5. Flexural strength versus sintering temperature and TiN addition.

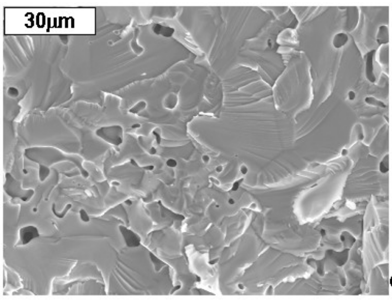
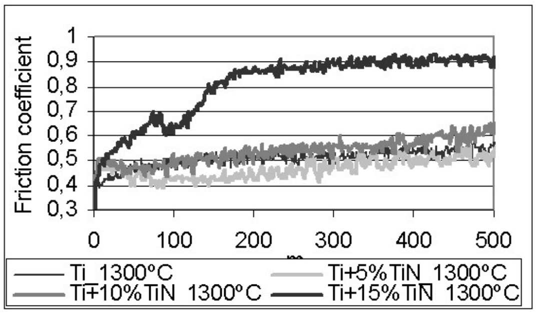
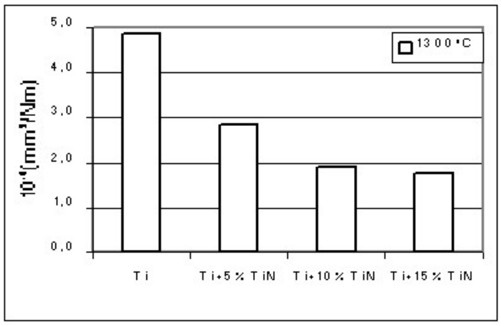


Fig. 6. Fracture of Ti+5%TiN sample sintered at 1200ºC.

Dry sliding wear experiments results can be shown in Figures 7 and 8. Figure 7.a) show slight increases in friction coefficient with low contents of reinforcement, 5, 10 vo%, but with high content of reinforcement, 15 vol%, friction coefficient growth dramatically. In the other hand wear rate decreases significantly in all cases respect pure titanium without reinforcement, decreasing with the TiN reinforcement increasing, 5 vol% TiN addition produces a 57% decrease in wear rate, while 10 and 15 vol% addition, produce 38% and 35% respectively.



a)



b)

Fig. 7. Dry sliding wear in samples sintered at 1300ºC a) Friction coefficient, b) Wear rate.

Microstructural characterization has been made by means of optical and electron microscopy. Figure 8.a) shows an optical microscopy image of the microstructure of titanium with 5 vol.% TiN particles sintered at 1200ºC. Homogeneous dispersion of TiN particles was observed. Porosity was situated inside the grains and in the grain boundaries, and grain size was about 23 microns. Figure 8.b) shows a SEM image with EDX analysis of a titanium matrix composites reinforced with 10 vol.% TiN sample sintered at 1200ºC. TiN particles was positioned initially in the cavities showed and occupied now by titanium and silicon rests. The impurities of the matrix material appears in the grain boundary forming the white phase showed in the figure.

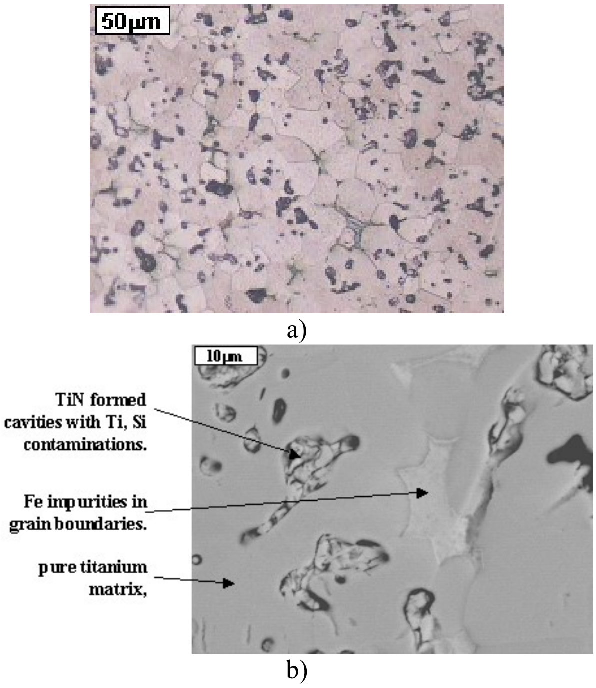


Fig. 8. Microstructure of titanium matrix composites reinforced with TiN particles a) Optical microscope image Ti+5 vol.% TiN sintered at 1200ºC, b) SEM image Ti+10 vol.% .

4. Conclusions

- Powder metallurgy is a good route to obtain composites titanium matrix reinforced with TiN particles.
- Increases in the TiN addition decreases the samples shrinkage, and increases the porosity.
- Addition of TiN particles produce increases in the hardness but induce fragility in the composite material.
- Wear resistance properties increases dramatically with the TiN contents of reinforcements, increasing the friction coefficient and decreasing the wear rate.

5. References

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