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Influence of Nano-Reinforcements on the Mechanical Properties and Microstructure of Titanium Matrix Composites.

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Abstract:

The goal of this work is the evaluation of nano-scaled reinforcements; in particular nano diamonds (NDs) and carbon nanotubes (CNTs) on properties of Titanium Matrix Composites (TiMMCs). By using nanosized materials as reinforcement in TiMMCs, superior mechanical and physical properties can be expected. Additionally, titanium powder metallurgy (P/M) offers the possibility of changing the reinforcement content in the matrix within a very wide range. In this work, TiMMCs have been produced from titanium powder (grade 4). The manufacturing of the composites was done by hot pressing, followed by the characterisation of the TiMMCs. The Archimedes density, hardness and oxygen content of the specimens in addition to the mechanical properties were compared and reported in this work. Moreover, XRD analysis and SEM

observations revealed in situ formed titanium carbide (TiC) phase after hot pressing in TiMMCs reinforced with NDs and CNTs, at 900ºC and 1100ºC respectively. The strengthening effect of NDs was more significant since its distribution was more homogeneous in the matrix.

Keywords: A. Metal Matrix Composite, A. Nano-particles, B. Mechanical properties, E. Powder processing

1. Introduction

Nowadays, the interest in the development of new materials with attractive properties is increasing. In particular the manufacturing of composite materials is a promising way to achieve materials with outstanding properties. The well known properties of titanium and its alloys [1-3] as matrix metal, in combination with the advantages of powder metallurgy techniques [4], offer an interesting possibility to the development of metal matrix composites [5, 6]. The use of innovative reinforcement materials plays an important role for the production of composite materials with outstanding properties. Several studies present nano-scaled reinforcements as interesting materials for the production of metal matrix composites [7, 8]. Few works present Titanium Metal Matrix Composites (TiMMCs) strengthened with nano-carbon reinforcements [9, 10]. The main objective of this work is the study of the behaviour of TiMMCs reinforced with nano carbon materials, carbon nanotubes (CNTs) and nano diamonds (NDs), and produced via powder metallurgy at different processing conditions. The manufacturing of the titanium composites was usually carried out via hot pressing at different consolidation temperatures. Additionally, heat treatments at different temperatures were performed to evaluate a possible reaction between titanium matrices and the carbon

nano-reinforcements. Another important focus of research was the distribution of nanodiamond particles and carbon nanotubes in the titanium matrix.

2. Starting materials

In this work one type of titanium powders, commercial pure titanium (CPTi) grade 4, was employed as starting matrix material. The theoretical density of the matrix powders was measured by means of the pycnometer AccuPy 1330V3.3. Chemical analysis, i.e. oxygen and nitrogen contents, were realized by LECO TC500. Additionally, the mean particle size was measured by Mastersizer2000. Table 1 shows the characteristic of the titanium matrix powder. The carbon nano-reinforcements were nano-diamond particles (NDs) and carbon nanotubes (CNTs). Their characteristics according to the supplier are listed in tables 2 and 3.

Concluding the characterization of the starting materials, SEM micrographs were obtained by high resolution scanning electron microscopy (HRSEM Zeiss Gemini Supra VP40). The irregular morphology of the titanium powder can be observed in figure 1. Particles from 1 to 30 μm are detected in this CPTi powder, as also confirmed by its particle size measurements. The mean particle size of the titanium matrix powder was 13.75 μm (table 1). Following, SEM images of the NDs and CNTs reinforcements were taken. Due to the nano-size and geometry of NDs and CNTs, a preliminary dispersion of the agglomerates in ethanol using ultrasound was done. However, the individual particles and nanotubes are not easily recognised in figure 2.A and figure2.B.

3. Experimental procedure

Powder treatment

The first task when working with nanosized reinforcing phases is to achieve an optimal distribution of the carbon nano-reinforcements, NDs and CNTs, in titanium matrices. Therefore the preparation of powder mixtures was performed according to the results obtained from previous tests [11]. Mixing was done in a Sintris mixer; the mixing time was 16 hours. Ceramic balls (ZrO_2) with 3 mm of diameter were usually used. The weight ratio of ceramic balls and powder was 10:1.Additionally, hexane was used to distribute the nano-reinforcement materials in the metallic matrix [12]. The powder mixture was dried and then blended again for a few minutes without the ceramic balls. The same volume percent of nanotubes and NDs was employed (1.8 % vol.). For both materials the same blending procedure was used. As it is well known, nanotubes are difficult to disperse due to their special geometry. By applying the above mixing procedure it was not possible to achieve a perfect distribution of the CNTs. However, since one objective of this work was the evaluation and comparison between these twocarbon-based reinforcements in titanium matrices processed using the same route, both reinforcements were used under same manufacturing conditions.

Hot consolidation

The technique employed for hot consolidation of the composites was hot pressing (HP). All the hot pressing processes were carried out in a press HPW 315/400-2200-1000-PS (FCT, Rauenstein, Germany). The mechanical pressure applied was 30 MPa, and the heating rate was 10 K/min. These conditions were fixed for all the experiments. Also the vacuum was close to 10^{-1} mbar for all the experiments. The vacuum used was the maximum that can be achieved by this hot pressing equipment. Furthermore, the holding time of the hot pressing was one hour. Only the temperature was varied and the tested values were 900°, 1100° and 1300°C.

For hot pressing, the loose powder mixture was put into a die made of graphite (Ø 65 mm). The die was usually lined with a thin graphite foil coated with boron nitride (BN). The die assembly was then loaded into the hot pressing equipment.

Heat treatment

Heat treatments (HTs) were applied only to specimens produced via HP at 900°C. The objective of this heat treatment is to study the reactivity of carbon reinforcements and titanium matrix, Ti being a strongly carbide-forming element. The temperatures selected to carry out the heat treatment were 1100°, 1200°, 1300° and 1400°C. The holding time for each run was one hour. The heat treatment was carried out in high vacuum using a Thermal Technology Inc. furnace model Astro. It can reach a vacuum of 10^{-6} mbar. Several specimens cut from the consolidated plate at 900°C were used for the heat treatments.

Characterisation of samples

First the graphite foils were completely separated from the sample surfaces using sand blasting. The final dimensions of the cylindrical plates were approximately 65 mm diameter and 4 mm in height. The characterisation of the final samples involved measurements of density, hardness, oxygen content, XDR and microstructural study. The density was determined at room temperature using Archimedes's method in water. Due to the closed porosity, the density measurements could be done without impregnation. In this present work, the values of the relative density were calculated and reported considering the total reaction between the carbon nano-reinforcements and the titanium from the matrix, being the TiC phase the product of such reaction. Within this framework, the theoretical density values are shown in the table 4.

Hardness (HV10) was measured in a tester model "Shimadzu HSV-30". Subsequently, chemical analysis of several samples was carried out in an analyzer LECO TC 500. The microstructural study was realised by high resolution scanning electron microscopy. Furthermore, reaction between carbon nano-reinforcement and the titanium based matrix was evaluated by XRD analysis. The method used to measure the flexural properties of the specimens was the three-point flexural test at room temperature. MPIF Standard 41 was the standard sample used for the bending tests [13].

The equipment employed was the uni-axial hydraulic press "MICROTEST". The cross-head speed was 0.4 mm/min. The plates were perfectly cut to standard dimension for the flexural tests. The dimensions of the cut samples were 6.4 x 3.5 x 24mm^3.

4. Results and discussions

After the characterisation of all the samples, the obtained results indicated there were several differences between the titanium pure matrix and the composites reinforced with NDs and CNTs.

Archimedes density and hardness

These two properties, Archimedes density and hardness (HV10) values were first measured for the TiMMCs hot pressed at 900º, 1100º and 1300ºC. Furthermore, they were compared to Archimedes density and hardness (HV10) values obtained for TiMMCs hot pressed at 900ºC and HT at four different temperatures: 1100°, 1200°, 1300° and 1400°C. These results are listed in tables 5 and 6.

When observing the composites density at different temperatures, an excellent densification of TiMMCs with NDs resulted after hot pressing at 1100° and 1300°C (relative density up to 99%).

The relative density relating to pure titanium matrix, produced via HP at 1100º and 1300ºC in previous works, were close to 99.5% (up to 4.519g/cm^3 at 1100ºC and up to 4.518 g/cm^3 at 1300ºC). Additionally, for pure titanium matrix hot consolidated at 900ºC, its relative density was close to 98% (mean Arch. density 4.42g/cm^3) [14-15]. In this present work for both types of composites, the lower HP temperature used, the less densification was obtained (after HP at 900ºC relative density up to 97.6% and mean Arch. density 4.415g/cm^3). The highest relative density values were achieved in composites heat treated at 1300ºC, the two types of TiMMCs presented values up to 99.1%.

Mainly, the reinforced titanium matrices presented higher hardness values than the pure titanium matrix (theoretical value of 270 HV10 for CPTi grade 4) [1-3, 16]. After HP at 900ºC, there was a slight difference among the hardness values of the two types of TiMMCs, being the hardness of composites with NDs higher than TiMMCs with CNTs. After HP at the highest temperature (1300ºC), the hardness difference between the two types of TiMMCs was more significant than at low HP temperatures (900º and 1100º), showing the composites with NDs higher hardness values than composites with CNTs.

It was observed in specimens hot pressed at 900ºC and HT at 1300º and 1400ºC, how their hardness increased considerably in comparison to composites only hot pressed at 900ºC (without the HT) (table 6). In particular for TiMMCs with CNTs, the higher HT temperature used, the higher composites hardness values were measured.

Comparing the hardness of composites hot pressed (at 1100º and 1300ºC) and heat treated at the same temperature (1100º and 1300ºC), several differences were observed between the two types of composites. There was a hardness variation between composites reinforced with CNTs after HP at 1300º and composites with CNTs hot

pressed at 900ºC and HT at 1300ºC. However in TiMMCs with NDs such difference was not significant (at 1300ºC).

Microstructural study

In the study of the TiMMCs microstructures the reinforcements distribution in the matrix and the reactions between the matrix and the reinforcements were examined. Comparing the TiMMCs microstructures, several differences between the two types of composites were detected. Since the reinforcement geometries and their reactive surface are different, in addition to their dispersion behaviours, these differences between them were expected [17]. The distribution and size of the reinforcing phases in the titanium matrix were these significant differences. The presence of larger grey micro-areas in titanium matrices fabricated at 900°C with CNTs than in TiMMCs with NDs, is widely observed in figures 3.A, 3.B, 4.A and 4.B. It could be understood as a consequence of the mixing problems well known for CNTs. Then, these large micro-areas observed in figure 3.B could be CNTs [10, 18]. Furthermore, a possible explanation to the microstructure showed in figure 3.A, is subtle reactions between the Ti matrix and the NDs, being these small grey micro-regions the TiC formed. In these TiMMCs with NDs the higher HP temperature, the larger size of these grey micro-areas is observed (figures 3.A, 4.A and 5.A). However, it was not possible to distinguish the TiC phase formed from NDs and CNTs agglomerations in these SEM images. The higher HP temperature (1300ºC) used, the less size differences between the formed phase (grey micro-areas) in both TiMMCs were virtually detected (figures 5.A and 5.B).

Moreover, the microstructure study of the TiMMCs HT at 1100º, 1200º, 1300º and 1400ºC was carried out (figures 6 to 10). After the heat treatment above the consolidation temperature, some porosity consisting of rounded pores was detected.

This fact is very significant in TiMMCs with NDs and after a heat treatment at 1100ºC. It was in agreement with obtained values of the density measurements (table 5). To explain such porosity, two hypotheses were considered. One possible hypothesis could be the removal of some clusters of the reinforcements during the cutting of the plates, i.e. the porosity would be an artefact caused by sample preparation of the micrographs. This hypothesis was discarded since this high porosity did not appear for composites without HT. Therefore, the most probable reason is attributed to entrapped porosity at the consolidation temperature that expands at temperatures above the hot consolidation temperature [18].

Furthermore, the increase of HT temperatures contributed to growth of the TiC phase area formed. It was confirmed by XRD analysis (figure 11). Additional examinations were carried out by EDS analysis (table 7). In figures 7.A and 7.B, the measurement points are marked. Impurities after polishing were also detected (figure 7.A, i.e. in spot 2 and in figure 7.B spot 3). The morphology of the porosity in both titanium matrices is very similar.

Comparing the microstructures showed in figures 8 to 10, the composites reinforced with NDs presented a homogeneous distribution of the TiC phase, since the reinforcement was better distributed in the matrix.

Moreover, there are appreciable differences comparing microstructures of composites after hot pressing at 1100° and 1300°C (figures 4 and 5) and composites heat treated at the same mentioned temperatures (figures 6 and 9). The composites consolidated via HP at 1100° and 1300°C did not present so high porosity, which was widely observed in composites heat treated. Furthermore, the TiC formed in the TiMMCs HT presented slightly large phase area, independently of the type of the carbon reinforcement used.

Observing the microstructure of TiMMCs reinforced with NDs, the TiC phase formed achieved a size up to 10 µm for TiMMCs hot pressed at 1300ºC (figure 5.A) and up to 15 µm for TiMMCs heat treated at the same temperature (1300ºC) (figure 9).

XRD analysis

Due to an effect of the less quantity of TiC phase formed in TiMMCs with CNTs than in composites with NDs, in several X-Ray measurements this phase for TiMMCs with CNTs was undetected. This fact took place in the composites hot pressed or heat treated below 1200ºC (figure 11). However, the results of the XRD analysis show for TiMMCs with NDs, the presence of TiC phase formed even after HP at 900ºC.

Comparing the integrated area of the TiC peaks, the mass content of the formed TiC was calculated (relative intensity ratio method). After HP at 900ºC, there is a 2.5 mass% of TiC phase formed in TiMMCs reinforced with NDs in comparison to a 0 mass% of TiC calculated for TiMMCs reinforced with CNTs. However, at 1100° and 1300ºC (HP) there are appreciable variations in TiC phases formed in TiMMCs with CNTs. After HP at 1100ºC the TiC content was 1.1 mass% and after HP 1300ºC the TiC formed increased slightly up to 1.3 mass%. In TiMMCs with NDs, 2.5 wt% TiC phase was detected after HP independently of the consolidation temperatures.

Following, reactions during a subsequent heat treatment of TiMMCs produced at 900°C via HP have been evaluated. The results of TiMMCs reinforced with NDs and CNTs are shown in figures 12.A and 12.B, respectively. The composites with NDs presented reactions between the matrix and NDs, independently of the heat treatment temperature (figure 12). In contrast composites reinforced with CNTs did not manifest clearly such reaction up to 1200ºC (figure 12.B). There was an undetectable weak signal of TiC phase in the X-Ray measurements.

Chemical analysis

Additional chemical analysis was carried out in order to determine the effect of the addition of oxygen and nitrogen bulk content of the TiMMCs. In table 8, oxygen and nitrogen mass percentages are listed for the different TiMMCs hot pressed at 900°, 1100° and 1300°C. There was a slight increase of the oxygen content for TiMMC produced at 900ºC compared to the oxygen content of the Ti powder (table 1) and the oxygen content of pure titanium matrix produced under the same manufacturing conditions [14]. At 1100º and 1300ºC HP temperature, the oxygen content is increasing up to 0.65 mass% in both types of composites. Moreover, the nitrogen content shows an increase from 0.01 mass% to 0.02 mass%. Even thought the titanium grade 4 powder can admit up to 0.05 mass%, it could involve a decrease of the mechanical properties of the specimens [2, 16, 19, 20]. High nitrogen content, in a titanium sample above 0.05 % mass, could cause a significant embrittle of the sample. In the same way, high oxygen content above 0.5 % mass could imply a diminution of the theoretical ductility of the titanium matrix (i.e CPTi grade 4). This is widely reported in previous works [19, 20]. Furthermore, chemical analyses were also performed in specimens hot treated at 1200º and 1300ºC in order to observe the effect of the temperature on the interstitial content. The results are reported in table 9. TiMMCs reinforced with CNTs present slight higher values of oxygen than TiMMCs with NDs, being processed at the same manufacturing and heat treatment conditions.

Flexural properties

The flexural test results are listed in tables 10 and 11, showing several differences between composites reinforced with NDs and CNTs. Three parallel measurements were done per TiMMC.

In general, by increasing the manufacturing temperature, the flexural properties of all TiMMCs decreased. In particular, titanium matrices reinforced with NDs exhibited better flexural properties than composites reinforced with CNTs. It was very significant after HP at 900ºC, being the high flexural strength for TiMMCs with NDs up to 1473MPa compare to 947MPa for TiMMCs with NDs. However, after HT the differences between the flexural strength of both types of TiMMCs are insignificant (table 11). The higher HT temperature used, the lower flexural strength measured in both types of TiMMCs. In particular, the flexural strength of TiMMCs with NDs decreased from 1473MPa after HP at 900º to 512MPa after HT at 1400ºC (tables 10 and 11). Despite their low flexural strength obtained after 1400ºC HT, these composites exhibited deformation properties up to 1.5%. In case of TiMMCs with CNTs, the deformation values could not be calculated due to measurement problems with the equipment.

5. Conclusions

The characterisation results showed several differences between two types TiMMCs manufactured and heat treated under the same conditions. Their properties obtained and their microstructures are related to the behaviour of each type of reinforcing material used. Additionally, the processing parameters affected considerably the properties of the composites hot pressed and heat treated.

Considering the manufacturing process, the application simultaneously of pressure and high temperature (above 900ºC) contributed to produce composites with excellent densification (up to 99.1%).

Through the microstructure analysis, it can be seen the formation of the TiC phase and its distribution, which was very homogenously distributed in case of TiMMCs with NDs

compare to the composites with CNTs. Furthermore, the higher hot pressing temperature used, the larger phase areas of TiC formed were particularly appreciated in SEM images of TiMMCs reinforced with NDs. However, this fact can not be clearly differentiated in case of TiMMCs with CNTs. For TiMMCs with NDs, the formed TiCs could be measured after HT at 1100ºC. Contrary to expectations, the reaction between the titanium matrix and the CNTs was not clearly detected by X-Ray measurements after HT at 1100º and 1200ºC.

It was shown that TiMMCs with well distributed and small reinforcing phase presented the best flexural strength (1473MPa for TiMMCs with NDs hot pressed at 900ºC compare to 400MPa for titanium pure matrix [10, 16]). In addition to, the results of the lowest flexural strength obtained for each one of the TiMMCs are in agreement with high hardness and low density results.

In general the flexural properties of TiMMCs reinforced with NDs are higher than the properties of TiMMCs with CNTs, under the same operational conditions. The in situ formed TiC particles were distributed more uniformly in the matrix for TiMMCs reinforced with NDs than in composites with CNTs. It suggests that such particles could cause a pinning effect which is effective for improving the mechanical properties response [23]. Therefore, the presence of large reinforcing phases affected as disadvantage to the strengthening effect as it was observed in TiMMCs with CNTs.

Finally, the heat treatment meaning is regarding to a possible route to evaluate indirectly the distribution of the carbon reinforcements. In this way, indirect information of the homogeneous distribution in the titanium matrix of the in situ formed TiC particles was shown. Additionally, after the heat treatment above the consolidation temperature some porosity conformed by round pores was observed. This phenomenon

was attributed to the entrapped porosity at the consolidation process that expands at temperatures above the HP temperature, i.e. the heating temperature up to 1200ºC. At highest heating temperatures (1300º and 1400ºC) a slight "re-sintering" effect can be observed.

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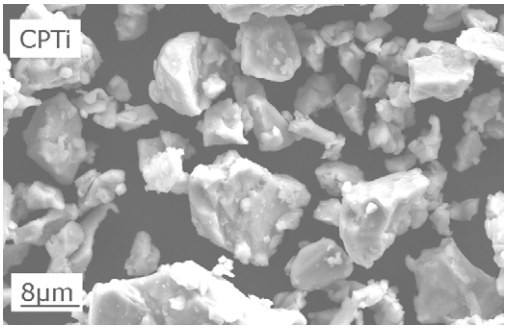

Figure 1. SEM micrograph of CPTi matrix powder.

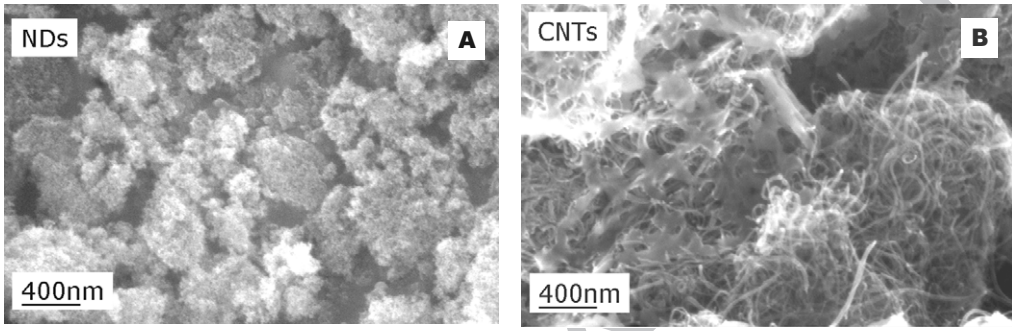

Figure 2. SEM micrograph of nanodiamonds (NDs) (A) and carbon nanotubes (CNTs) (B).

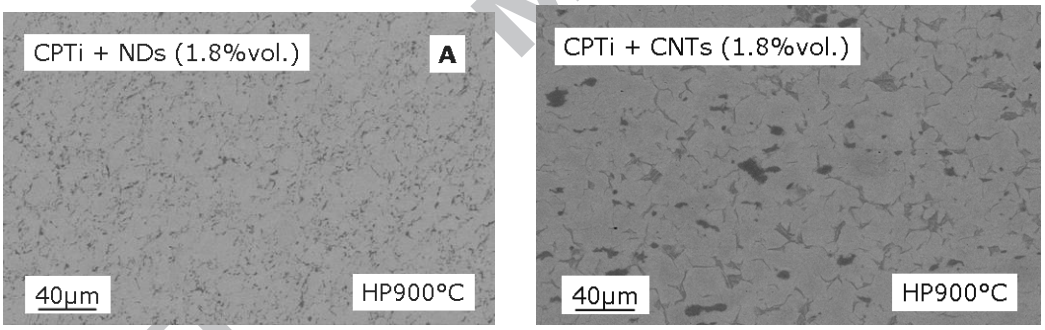

Figure 3. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) (hot pressing at 900°C and 30 MPa for 1 hour in vacuum).

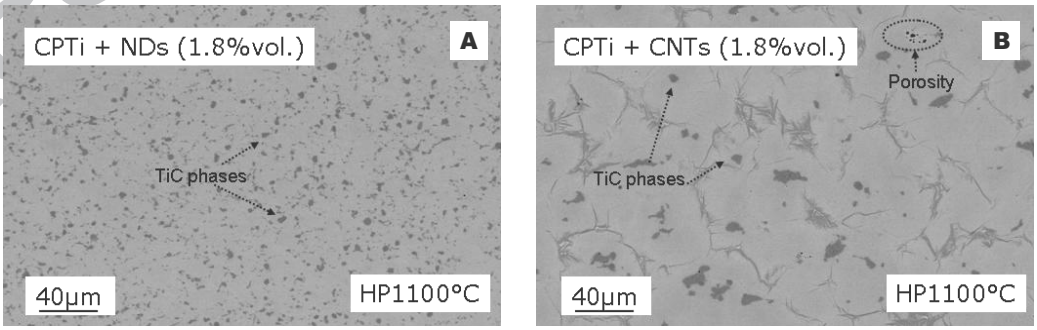

Figure 4. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) (hot pressing at 1100°C and 30 MPa for 1 hour in vacuum).

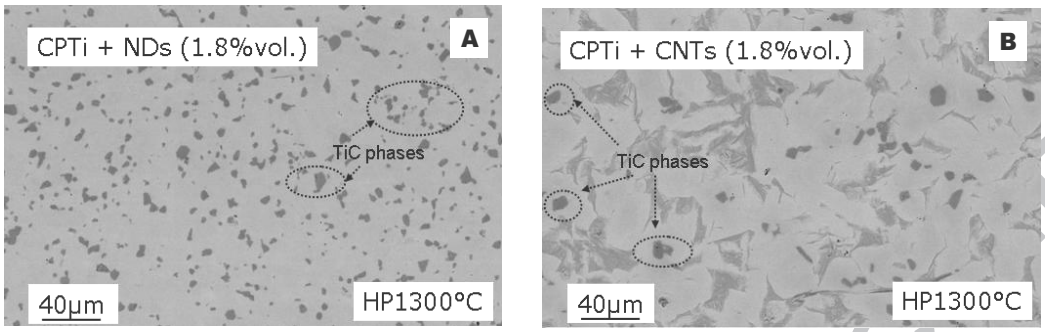

Figure 5. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) (hot pressing at 1300°C and 30 MPa for 1 hour in vacuum).

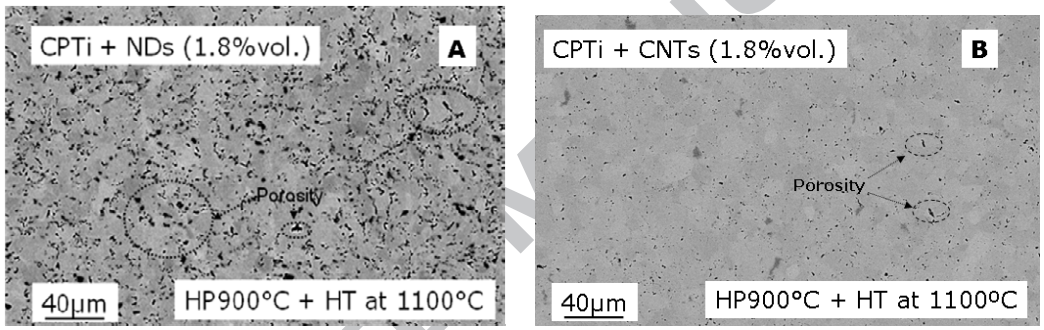

Figure 6. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) fabricated via hot pressing at 900°C (30 MPa for 1 hour in vacuum) and heat treated at 1100°C for 1h in vacuum.

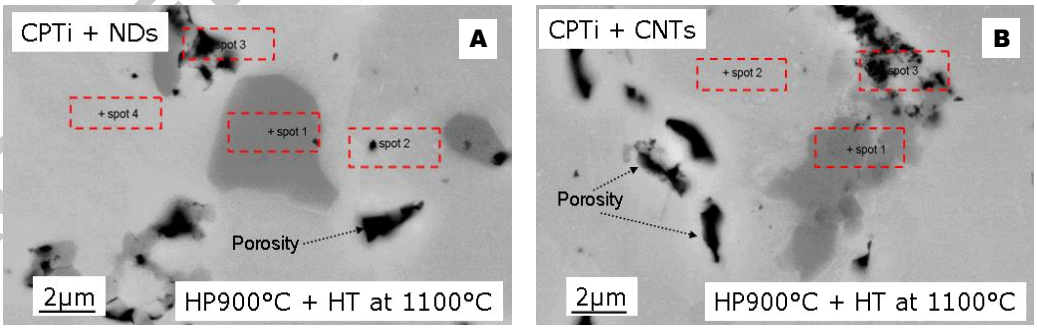

Figure 7. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) fabricated via hot pressing at 900°C (30 MPa for 1 hour in vacuum) and heat treated at 1100°C for 1h in vacuum.

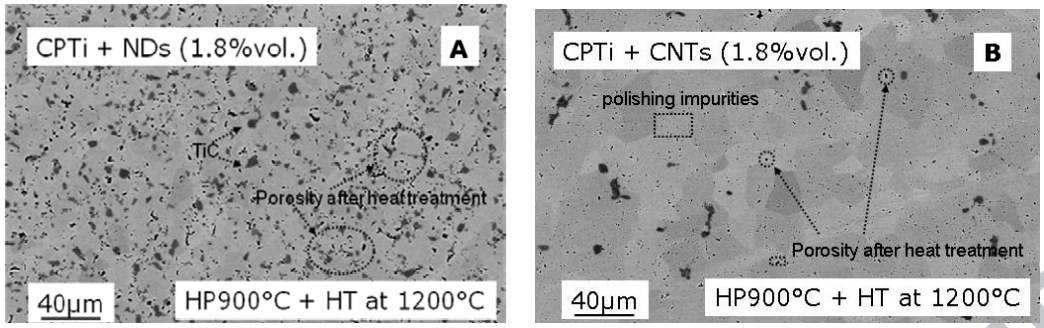

Figure 8. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) fabricated via hot pressing at 900°C (30 MPa for 1 hour in vacuum) and heat treated at 1200°C for 1h in vacuum.

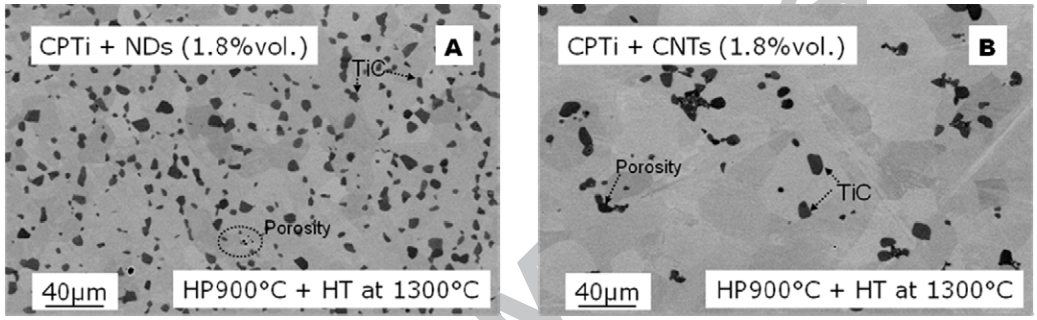

Figure 9. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) fabricated via hot pressing at 900°C (30 MPa for 1 hour in vacuum) and heat treated at 1300°C for 1h in vacuum.

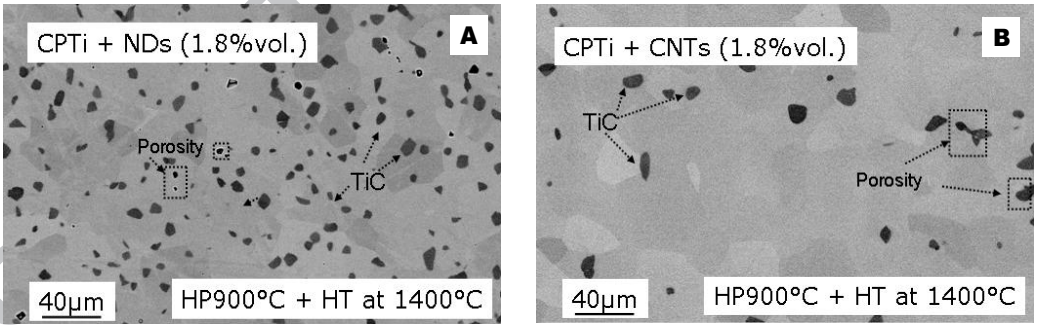

Figure 10. SEM micrographs of the titanium composite reinforced by nanodiamond particles (A) and carbon nanotubes (B) fabricated via hot pressing at 900°C (30 MPa for 1 hour in vacuum) and heat treated at 1400°C for 1h in vacuum.

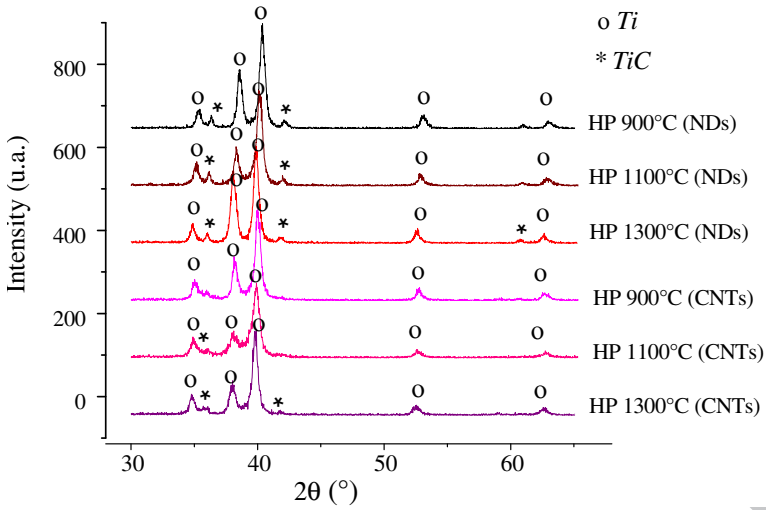

Figure 11. Comparison of XRD patterns of all composites produced via Hot Pressing at three different temperatures.

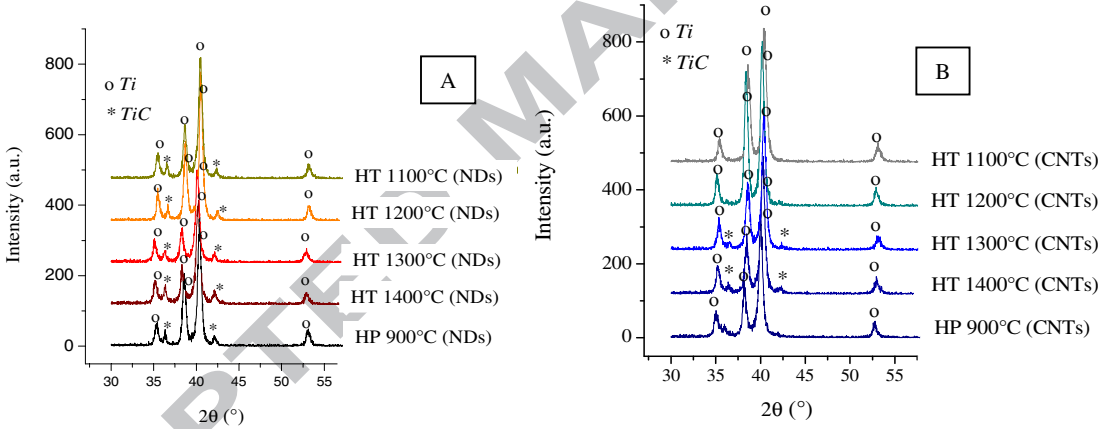

Figure 12. Comparison of XRD patterns of all composites produced via Hot Pressing (HP) at 900°C and heat treated (HT) at four different temperatures.

Table 1. Characteristics of the matrix powders: Density by pycnometer, oxygen and nitrogen contents by LECO TC 500 and particles size distribution by Mastersizer 2000.

Type of powder	Density by pycnometer (g/cm^3)	Impurity content		Particle size distribution		
		O (mass %)	N (mass %)	d(0.1) (µm)	d(0.5) (µm)	d(0.9) (µm)
CPTi	4.54	0.45	0.01	6.65	13.75	25.63

Table 2. Characteristics of the nanodiamonds (NDs).

Purity(%)	Average particle size (nm)	Density (g/cm^3)	Specific surface area(m^2/g)	Oxygen (mass %)
98-99	4-6	3.05-3.3	282.83	5.5

Table 3. Characteristics of the carbon nanotubes (CNTs).

Purity (%)	Number of walls	Outer mean D (nm)	Outer (nm)	Inner D (nm)	Length (µm)	Bulk density (g/cm^3)
99	3 - 15	13 - 16	5 - 20	4	>1	1.40-2.30

Table 4. Theoretical density values.

Material	Th. Density (g/cm3)	
Ti (Grade 4)	4.51	
TiC	4.90	
Ti with NDs (1.8%vol. or 1.4 mass %)	4.49 (C reaction 0% with Ti)	4.53 (C reaction 100% with Ti)
Ti with CNTs (1.8%vol. or 0.6 mass %)	4.48 (C reaction 0% with Ti)	4.52 (C reaction 100% with Ti)

*Table 5. Archimedes density of TiMMCs reinforced with NDs and CNTs. (HP: Hot pressing; HT: Heat treatment after HP at 900°C). *Relative density considering total reaction between Ti and the carbon nano-reinforcements.*

HP temperature (ºC)	HT temperature (ºC)	TiMMC with NDs		TiMMCs with CNTs	
		Arch. Density (g/cm^3)	Relative density (%)*	Arch. Density (g/cm^3)	Relative density (%)*
900		4.42±0.01	97.6±0.2	4.41±0.01	97.6±0.2
900	1100	4.43±0.03	97.8±0.7	4.46±0.03	98.7±0.7
900	1200	4.45±0.04	98.2±0.9	4.48±0.01	99.1±0.2
900	1300	4.49±0.03	99.1±0.7	4.48±0.05	99.1±1.1
900	1400	4.44±0.01	98.1±0.2	4.47±0.04	98.9±0.9
1100		4.47±0.04	98.7±0.9	4.40±0.04	97.4±0.9
1300		4.49±0.01	99.1±0.2	4.43±0.01	98.1±0.2

Table 6. Hardness (HV10) of TiMMCs reinforced with NDs and CNTs. TiMMCs heat treated have been produced via HP at 900°C.

HP temperature (ºC)	HT temperature (ºC)	TiMMC with NDs (HV10)	TiMMCs with CNTs (HV10)
900		378±1	351±16
900	1100	373±4	356±5
900	1200	350±2	358±4
900	1300	410±3	384±2
900	1400	392±8	425±23
1100		391±3	355±6
1300		393±3	332±2

Table 7. Results of EDAX analysis in several spots in TiMMCs produced via HP at 900°C for 1 h and heat treated at 1100°C for 1h.

Specimen	Spot	Ti (mass %)	C (mass %)	Ti (at %)	C (at %)
TiMMCs+NDs	1	87.23	12.77	63.13	36.88
TiMMCs+NDs	3	86.58	13.42	61.81	38.19
TiMMCs+NDs	4	100	0	100	0
TiMMCs+CNTs	1	89.01	10.99	67.01	32.99
TiMMCs+CNTs	2	100	0	100	0

Table 8. Oxygen and nitrogen content in TiMMCs fabricated at the three different hot pressing temperatures. (HP: Hot pressing)

HP Temperature (°C)	TiMMCs with NDs (1.8%vol.)		TiMMCs with CNTs (1.8%vol.)	
	Oxygen mass%	Nitrogen mass%	Oxygen mass%	Nitrogen mass%
900	0.48	0.01	0.53	0.01
1100	0.57	0.01	0.58	0.01
1300	0.65	0.02	0.65	0.02

Table 9. Oxygen and Nitrogen content of TiMMCs produced by hot pressing at 900°C and heat treated at different temperatures. (HT: Heat treatment.)

HT Temperature (°C)	TiMMCs with NDs (1.8%vol.)		TiMMCs with CNTs (1.8%vol.)	
	Oxygen mass%	Nitrogen mass%	Oxygen mass%	Nitrogen mass%
1200	0.54	0.01	0.67	0.01
1300	0.59	0.01	0.68	0.01

Table 10. Flexural strength and deformation (%) of TiMMCs produced via hot pressing at three different temperatures.

HP Temperature (°C)	TiMMCs with NDs (1.8%vol.)		TiMMCs with CNTs (1.8%vol.)	
	Flexural strength (MPa)	Deformation (%)	Flexural strength (MPa)	Deformation (%)
900	1473±125	2.4±1.2	947±71	1.7±0.8
1100	990±110	1.9±0.7	558±68	1.2±0.9
1300	851±185	1.7±1.1	765±70	1.3±0.5

Table 11. Flexural strength and deformation (%) of heat treated TiMMCs at different temperatures (HT). All of the composites are reinforced with NDs and CNTs fabricated via HP at 900°C. (n.d.: no determined)

HT Temperature (°C)	TiMMCs with NDs (1.8%vol.)		TiMMCs with CNTs (1.8%vol.)	
	Flexural strength (MPa)	Deformation (%)	Flexural strength (MPa)	Deformation (%)
1100	628±47	1.2±0.3	627±40	1.2±0.7
1200	620±60	0.8±0.2	556±30	0.9±0.3
1300	n.d.	n.d.	504±38	n.d.
1400	512±77	1.5±0.6	454±25	n.d.