Research Article GU J Sci 32(1): 286-298 (2019)

Gazi University

Journal of Science

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Evaluation of Multi-Walled CNT Particulate Reinforced Ti6Al4V Alloy Based Composites Creep Behavior of Materials Under Static Loads

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Article Info

Received: 19/10/2017
Accepted: 09/10/2018

Keywords

Creep
Sintering
Ti-6Al-4V
Multi-Walled Carbon Nanotube

Abstract

This study examines the effects of additions of 0.5-5 v/v percentage multi-walled carbon nanotubes into the Ti-6Al-4V matrix by mechanical alloying at low rates and investigates the results of the different sintering conditions on the density, microstructure, and Creep behavior. Mechanical properties, microstructural and density of composite materials (Ti64 / CNT) produced by cold isostatic press molding method have been investigated. MWCNTs reinforced metal matrix composite powders were molded by cold isostatic pressing method using polyacrylonitrile (PAN) based binder. The binder decomposition was carried out by heat treatment. After molding, the specimens have been sintered at high temperature in high vacuum (10-2 bar). Metallographic experiments were carried out to examine density and microstructure. Experimental results indicate Ti–6Al–4V particulate can be sintered to up to 98,5% of calculated density. Maximum hardness was obtained 538 HV at 1300 °C for 3 hours and creep life inverse. By using SEM and X-ray diffractometer the characteristics of produced composite samples were investigated.
Although Ti–6Al–4V alloys are used as biomaterial, this study aimed at using MWCNTs containing Ti-6Al-4V composites at high temperature applications. Because MWCNTs reinforced Ti-6Al-4V composites are cheaper and has lower weight than the other materials used in this kind of applications.

1. INTRODUCTION

The aerospace, automotive and biomedical industries are some of viably used fields where titanium alloys have been used. Because of its heat treatment capability, good mechanical strength, high corrosion resistance and biocompatibility, Ti–6Al–4V alloy containing 6 wt.% Al and 4 wt.% V, has been used in airplane turbines and medical implants [1, 2].

The requirement of high strength, lightweight and fuel-efficient materials in automotive and aerospace industries provides a big research interest [3, 4]. Mechanical and corrosion resistance characteristics of Titanium (Ti) and alloys, which have high specific strength and low specific gravity, makes them highly relevant to these industries [5,6]. Because of its superior mechanical performance in service, Ti-6Al-4V alloy has been widely preferred in the aerospace industry and constitutes 45 % of the total Ti production [7-9]. Although Ti-6Al-4V has important characteristics that make it a good candidate for the aerospace industry, the production of the alloy is a major drawback due to the high affinity to the nitrogen and oxygen during the process. So, it can endanger the ductility property [10]. Therefore, processing of Ti-6Al-4V under vacuum atmospheres at high temperatures is a key requirement unless the properties of the bulk material are put at risk by interstitial elements [11]. This necessity results in very expensive process and limits the application of Ti-6Al-4V products.
Composite materials reinforced with Multi-walled carbon nanotubes (MWCNTs) have made great progress and experience a few distinctive applications by the advantages of mechanical, electrical and thermal

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properties. [8-12]. Due to their extremely high strength, MWCNTs are used as reinforcement element for the composite materials. In addition, the multi walled carbon nanotubes maintain good dispersion strengthening of composite materials [13-16]. The reinforcement of metal matrix composites can alter the mechanical properties of the composites as a result their applications can broaden. Since titanium carbide (TiC) was compatible with the titanium alloys, it has been used for the reinforcement to date [17-20]. But, the studies on MWCNTs reinforced MMC and CMC materials are restricted. Hot pressing and high sintering temperature methods have been used to produced MWCNTs reinforced Ceramic Matrix Composites materials [21-23].

Recent studies have proposed that MWCNTs is an ideal candidate for the ultra-high strength reinforcements [24]. MWCNTs, which has a low density, is used as an exquisite reinforcement against friction for metal matrices due to extraordinary physical properties. Nevertheless, number of reports in this field are limited due to challenges during the processing of MWCNTs [25-27].

Creep measurements of a titanium component is not easy because of its entire creep life which can reach up to 120000 h [28]. On the other hand, short-term creep test method is widely preferred for the precursory investigation on creep characteristics of Ti-6Al-4V alloy [29, 30].

2. EXPERIMENTAL

In this study, Ti–6Al–4V and MWCNTs powders, were used as the starting materials. The raw materials were characterized in terms of physical properties and was tested for identification of some values. In this research, gas atomized Ti–6Al–4V powders provided by Phelly Materials Company (USA) was used. It has a particle size distribution of D10= 58,27 μ, D50 =110,65 μ, D90=171,39 μ with a density of 4.43 g/cm3 as raw material. MWCNTs reinforcement material were purchased by Cheep Tubes Company (USA), with a density of 2.31 g/cm3. The average particle size of MWCNTs powders were about 10-30 nanometers. Different volume percentage of MWCNTs (0.5 to 5 v/v%) were dispersed into Ti-6Al-4V powders inside glass containers (inner diameter: 40 mm, capacity: 100 ml) and mixed with the help of a turbula ball mill (Turbula PM 400 MA, Switzerland) using alumina balls of same sizes (diameter 7 mm, weight ratio: 1:6:1). Using same size of milling balls during the process makes sure that sufficient collision energy is provided to the powder particles [3,31,32].

A binder system consisting of poliacrilonitril (PAN) was used. This process was prepared at first and then powder blend added incrementally. The powder loading in this mixture was 98.5 vol. percentage. Creep specimens were uniaxial pressed using a 300 MPa molding machine. The sintering cycle applied to the samples was as follows: samples first were heated to the 1300 °C at a rate of 10 °C/ min, then sintered at this temperature for 3 h in vacuum atmosphere between ten to twentyfour Torr) [33]. The Archimedes water immersion method was applied in order to determine the densities of the sintered samples were measured by means of the Archimedes water immersion method. The specimens were cut by abrasive cutter from their centers before metallographic examination, then ground and polishing. As etchant, the Kroll reagent (1 or 2 part (s) of hydrofluoric acid, 2 or 3 parts of nitric acid in about 50 parts of distilled water) was selected for optical metallographic observation. All Creep tests were carried out using cylindrical creep specimens with 30 mm gauge length and 10 mm diameter, on an Instron Model 8802 Creep test machine with under constant load in the 100 MPa at 300 °C. The hardness tests were performed using a Future-Tech (FM-110) micro hardness test machine at HV scale. At least fifteen measurements were tested on the same specimen under the same conditions to guarantee the reliability of the results. XRD analysis was performed using CuKα radiation source and a graphite filter where the range was between 20° and 85° with a step of 0.05° and 1 s of counting time. The powder morphologies and fracture surfaces of molding and sintering samples were examined using a scanning electron microscope (Jeol- JSM 6335F).

3. RESULTS AND DISCUSSION

3.1. Characterization of Powders Produced

SEM was used in order to observe the morphology of the starting powders (Ti-6Al-4V and MWCNTs) and the images are given in Figure 1.

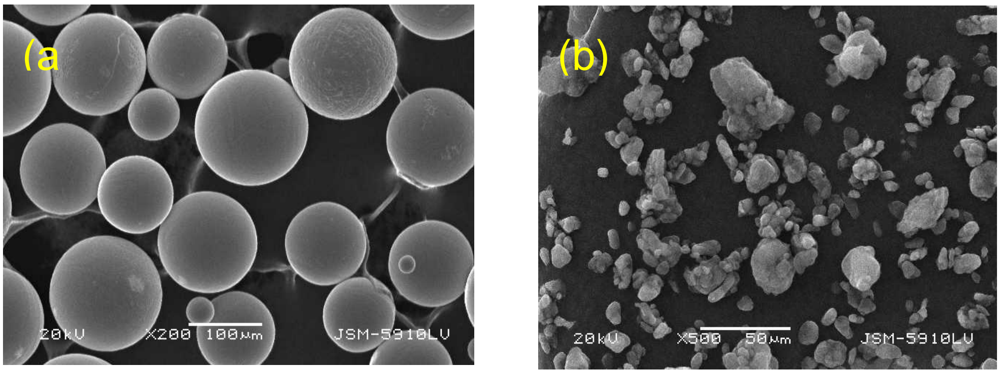

***Figure 1**. SEM images of Ti-6Al-4V and MWCNT powders a) Ti-6Al-4V and b) MWCNT*

As a characteristic of gas-atomization process Ti-6Al-4V powders have spherical shape which is shown in Fig. 1a and there was no agglomeration. Different than Ti-6Al-4V powders, MWCNTs were highly agglomerated and entangled as presented in Fig. 1b. The reason behind the agglomeration is attributed not only to the large surface area and high aspect ratio of MWCNTs powder but also as well as the collective and cooperative strong intermolecular bonding induced from the singular nanotubes [3,34]. Backscatter image of the Ti-6Al-4V / MWCNTs powders after 5h of grinding by ball milling process is given in Figure 2.

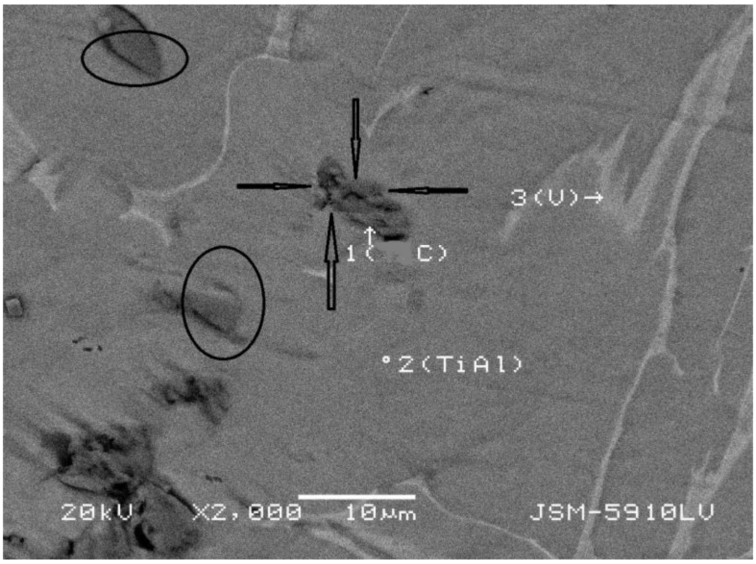

***Figure 2.** Backscattering image of the composite material (TiC (1), (TiAl(2), V (3).*

Different volume ratios of MWCNTs were dispersed as reinforcement in the matrix alloy and the homogenous dispersion of the reinforcement was dependent on the volume ratio: the possibility of achieving homogenous dispersion was less when the volume ratio of MWCNTs is more. This is ascribed to the large surface area, high aspect ratio (with a length many times that of their width), powerful intermolecular bonding in the singular tubes and tubular morphology/nano size dimensions of the MWCNTs which are giving rise to clustering and agglomeration [3,35]. Therefore, a relatively homogenous distribution of the MWCNTs was obtained in the mixture having 1% MWCNTs. On the other hand, clusters and agglomeration of MWCNTs, which is depicted by ellipses in Figure 2, was noticed when the volume ratio is high. The dissipation of the MWCNTs in Ti-6Al-4V matrix was adequate with 4% volume ratio. This was considered due to the trouble in distributing MWCNTs within the metal main phase when their volume ratio is high which supports the explanation aforementioned above.

The XRD analysis of the pulverized mixtures for different volume ratio of MWCNTs are given in Figure 3.

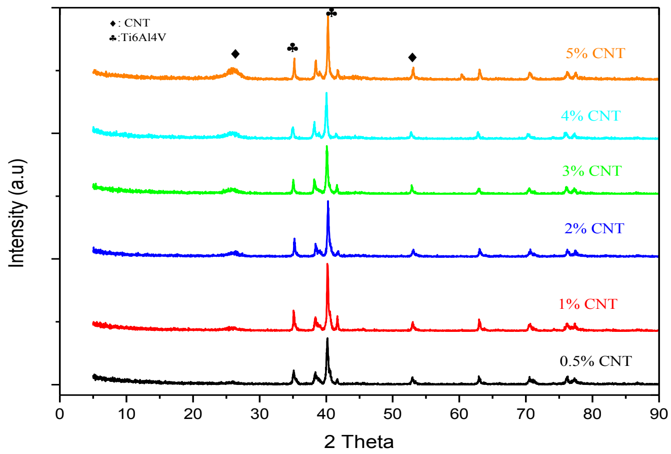

Figure 3. *XRD pattern of 5 % content MWCNTs particles in Ti–6Al–4V Matrix.*

The XRD analysis of the pulverized mixtures indicated the effect of broadening at the peaks. Some indications of MWCNT's presence were significant due to the observation of some MWCNT's in the mixture as a proof of uniform distribution of MWCNT's in the metal matrix, also implied by Cai et al. [36].

The X-Ray diffractions of the samples having various MWCNTs contents are shown in Figure 3 which shows the peaks at 2° of 38.44° and 44.7° belongs to the Ti6Al4V, and, MWCNTs peaks are observed at 25° and 53°. In addition, additional MWCNTs peak formed at 63.5° for the sixth sample that has 5% MWCNTs. Moreover, the intensity of the peaks showed an obvious enhancement depending on the MWCNTs ratio. In other words, that increasing MWCNTs contents cause an increase in the area of the main peaks of MWCNTs.

3.2 Characterization of the Produced Specimen Materials

Microstructures of the plain Ti-6Al-4V and Ti-6Al-4V/MWCNTs reinforced composites after sintering at 1300 °C were investigated using SEM as shown in Fig. 4.

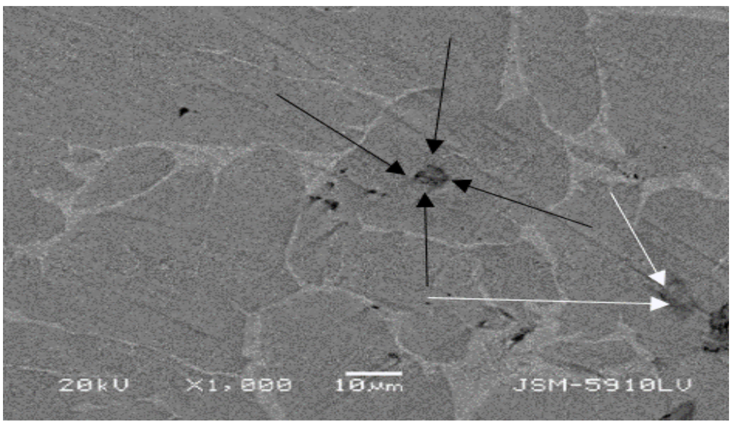

Figure 4. *SEM image of Ti-6Al-4V/MWCNT composite.*

Equiaxed structures were obtained with the addition of MWCNTs to the Ti-6Al-4V as shown in Figure 4. There was not any significant porosity in the microstructure which shows a good sintering behavior. Samples with 4 and 5% volume of MWCNTs indicated re-agglomeration, depicted by white arrows in Figure 4, and interfacial product of titanium carbide (TiC) was observed in the microstructure, indicated by black arrows in Figure 4. Inadequate dispersion of the MWCNTs resulted in this re-agglomeration and it is promoted the interfacial reaction between MWCNTs and Ti-6Al-4V giving rise to formation of TiC. This situation is more noticeable for the samples having 5% volume MWCNTs where there are more clustered particles of TiC as MWCNTs in this sample were highly agglomerated into large clumps. It is believed that these clustered TiC particles were produced around the around the lump of MWCNTs which is also reported in other studies [3,37].

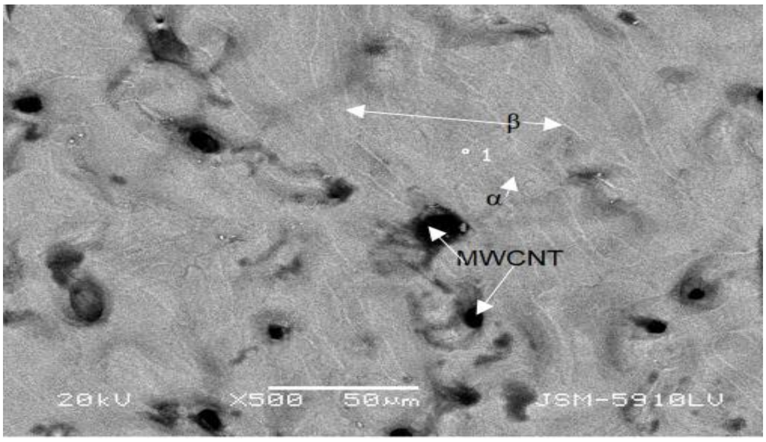

Figure 5. *SEM image of Ti-6Al-4V/MWCNT composite*

Sample with 1% volume of MWCNT had a completely compact microstructure, also homogeneously dispersed α and β phases without any re-agglomeration and interfacial products which supports the adequate distribution of MWCNTs. The matrix phase (grey-colored) is α and the white strips like phase located on the grain boundaries is β as given in Figure 5. The final evaluation of the microstructure (Alpha and beta) phases is given in the SEM micrograph of the sintered samples in Figure 5. The reinforced Ti–6Al–4V alloy had only two phases of titanium (alpha and beta). Carbon structure were detected in the specimens with 4 and 5 volume % MWCNTs, respectively. These outcomes are congruent with the microstructure of the specimen shown in Figure 5. Crystalline TiC phases were formed in samples with higher volume rate of the MWCNTs reinforcement. [3,38].

3.3. Effect of MWCNTs Reinforced on the Density of Ti-6Al-4V/MWCNTs Composites

Archimedes density measurement results of the samples which were sintered at 1300 °C are given in Fig. 6 for different volume ratio of MWCNTs.

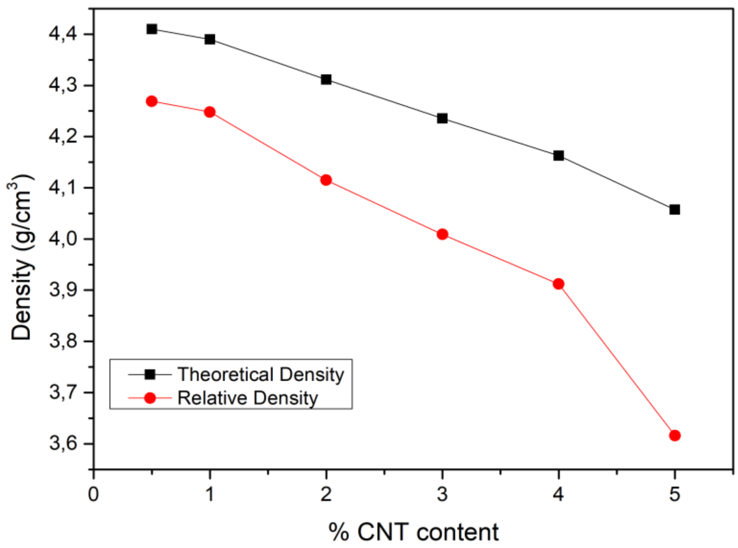

Figure 6. *CNT percentage reinforced by the density of different exchange rates.*

Sintered density of Ti-6Al-4V/MWCNTs composited had a tendency of decreasing density by an increase in the MWCNTs ratio. This is most likely due to the clustering and agglomeration of the MWCNTs reinforcement in the matrix. Another reason for the decrease in the density might be insufficient diffusion between Ti-6Al-4V and MWCNTs which results in a weak interfacial bonding leading to thermal mismatch between the MWCNTs reinforcement and Ti-6Al-4V matrix. Better relative density obtained for the low MWCNTs content such as 1% volume was considered as a result of better homogenous dispersion of the reinforcement within the matrix [39].

It was previously discussed that [36] MWCNTs reinforced Al alloys and Ti matrices can have a deterioration in the relative density of sintered bulk composite material with an increase in the MWCNTs ratio which supports the observation of this study. This deterioration in the density can be expected due to the higher possibility of pre-agglomeration and re-agglomeration which increases by an increment in the volume ratios of MWCNTs as mentioned previously.

3.4. Influence of MWCNTs Reinforced on the Microhardness of Ti-6Al-4V/MWCNTs Composites

Micro hardness measurement of the sintered plain Ti–6Al–4V and Ti–6Al–4V /MWCNTs reinforced samples were performed and the results are given in Figure 7. It was shown that the hardness increases by the ratio of MWCNTs which is different than the density trend. The enhancement in micro hardness with augmented high sintering temperature that was detected in the Ti–6Al–4V /MWCNTs composites was partially credited to the development of decreasing in pore volumes and intensification in density associated with elevated sintering temperature.

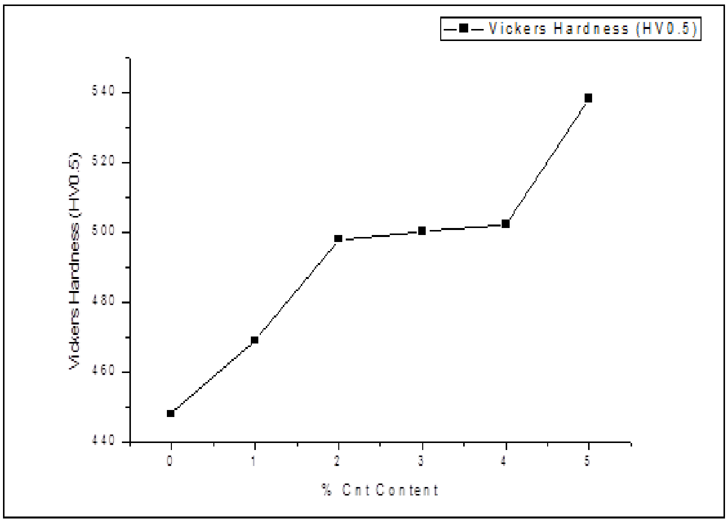

Figure 7. *The hardness versus different MWCNTs content (0.5- 5 %)*

The reason for increase in hardness with MWCNTs content might be related to the hard interfacial product TiC which is more pronounced at higher volume % of MWCNTs. Additionally, another reason for the increase in hardness by adding more MWCNTs reinforcement can be the existence of retained MWCNTs as well as crystalline TiC hard phases [3,38, 40].

3.5. Creep Test Results

Creep tests were performed at 300 ºC and the humidity condition was controlled in the laboratory. These tests took 3000 second and 4800 cycle [41]. The strain that was determined during the creep which is a time dependent deformation process showing the overall effect of stress and temperature.

Creep displacements decreased with increasing MWCNTs content. Although at 5% MWCNTs content, the creep displacement value is near to the 4% MWCNTs content composite. The life of the 5% MWCNTs reinforced composite is lower than the 4% MWCNTs. In contrast, at the creep life is diminished, as is obvious from the graph 5% MWCNTs reinforced samples. Increasing the MWCNTs decreases the creep displacement. In general, the deformation ends to increase as a function of the applied stress. Due to the hardening induced by carbon diffusion, the “Ti–6Al–4V/CNT” composites shows lower values of instantaneous deformation. The steady-state creep rate increases with the applied stress for both conditions studied. Relative to those of the pureTi6al4v alloy, the steady-state creep rate values of the “Ti–6Al–4V / MWCNTs” alloy are inferior [3,42].

In addition, although creep life generally increases with MWCNTs content after 4% of MWCNTs creep life decreases. At 5% of MWCNTs significantly lower creep life was obtained when compared with 3 and 4% of MWCNTs.

Figure 8 presents the creep displacement in creep test against MWCNTs content in Ti–6Al–4V matrix reinforced with MWCNTs.

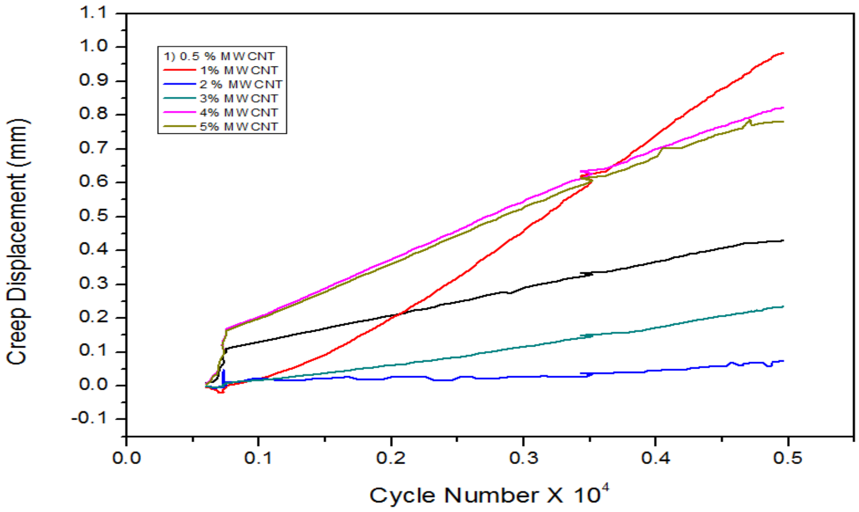

Figure 8. *Static Creep test of the Ti–6Al–4V matrix composites containing different v % MWCNTs*

Figure 9 shows that creep life generally increases with MWCNTs content, creep displacement decreases with MWCNTs content. Moreover, creep life decreases with MWCNTs content above 4% of MWCNTs.

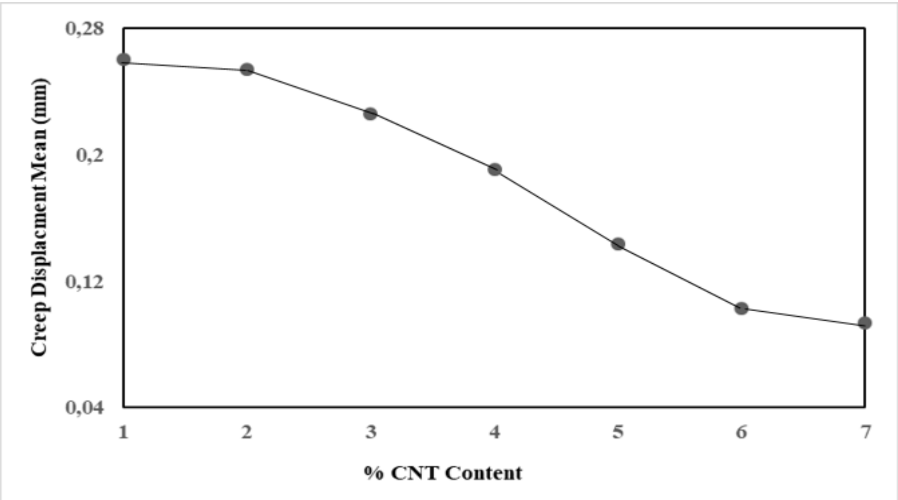

Figure 9. *Static Creep displacement of the Ti–6Al–4V matrix composites containing different v % MWCNTs*

Figure 10 presents that with MWCNTs content, creep life. Moreover, creep life decreases with MWCNTs content above 4% of MWCNTs.

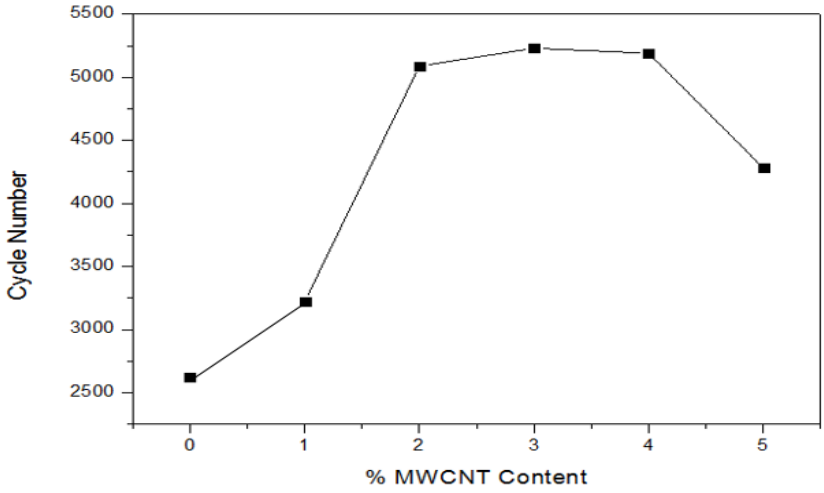

Figure 10. *Creep cycle rate of the Ti–6Al–4V matrix composites containing different v % MWCNTs*

Seen in figures, a steady state creep manifesting itself by an approximatively linear zone after a deflection with a prompt nonlinear deformation following the application of the stress. It is clear that creep strain is decreased with increasing MWCNTs content. The specimens reach the steady-state condition within two hours [43,44].

4. CONCLUSION

This work was carried out in order to analyze the effect of MWCNTs addition on the hardness, densification and creep properties of Ti-6Al-4V/MWCNTs composites to shed light on its applicability in several industries where the combination of higher elastic modulus with low density are advantageous.

It has been shown that production of fully dense lightweight Ti-6Al-4V/MWCNTs composites is possible by means of powder metallurgy technique. At a constant temperature, the density of the sintered composites decreased by an increase in the volume ratio of MWNCTs and this was ascribed to the difficulty to obtain a homogenous distribution of MWCNTs within the matrix. However, micro hardness of the sintered samples was enhanced with more MWCNTs addition. This increase in hardness was considered as a result of hard interfacial product TiC.

Ti-6Al-4V/MWCNTs composites can draw attention to creep applications in several industries where the higher temperatures, temperature differences and loading at the mean time are involved during applications. Therefore, this research introduced new prospects for the application of Ti-6Al-4V/MWCNTs composites. All of the sintered samples had $\alpha + \beta$ microstructure. Formation of TiC increased the hardness value (538 HV) and the short-term creep behavior of the samples was improved with the help of those carburs which give rise to reduction of immediate deformation and steady-state creep rates. Nevertheless, the effect of carburs on the fracture time was not clear which can ascribe to the possible increase in the surface roughness. Dislocation climbing creep process was concluded as the creep mechanism takes place in the present conditions. All samples revealed an αlpha and beta microstructure. A carburized layer, is formed by TiC carburizing increased the hardness value (538 HV). Immediate deformation was decreased and creep rates became steady as the carbide structure enhanced the short-term creep behavior. Nonetheless, the effect of carbide microstructure to the fracture mechanism was ambiguous, might be also associated with the increment of the surface roughness. In the current creep case, dislocation climbing creep processes might be included in creep mechanisms.

Good mechanical properties of Ti-6Al-4V having TiC confirms the possible potential of this microstructure in order to improve the high-temperature performance of Ti alloys. The results of this study based on very short creep durations.

ACKNOWLEDGEMENTS

This work was supported by the Scientific Research Project Program of Marmara University (FEN-K-070317-0107).

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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