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Thermal Diffusivity Behaviour of Multi-walled Carbon Nanotube Reinforced Ti6Al4V Metal Matrix Composites

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Abstract. This study investigated the thermal diffusivity behaviours of spark plasma sintered (SPS) multi-walled carbon nanotubes (MWCNTs) reinforced Ti6Al4V composites containing 0, 1, 2 and 3 wt. % of the reinforcement respectively, over a range of 50 – 300 °C. The MWCNTs were dispersed into the Ti6Al4V matrices by high-energy ball milling (HEBM) technique and the milled composite powders were consolidated by SPS under a vacuum atmosphere. The sintering conditions employed were heating rate (100 °C/min), holding time at temperature (5 min), sintering temperature (850 °C) and applied pressure of 50 MPa. The relative densities of the composite discs were measured according to Archimedes' principle while the thermal diffusivities of as-sectioned composite samples were measured using the Laser Flash equipment. Relative densities of the synthesized nanocomposites deteriorated with increase in the weight fraction of MWCNTs added to Ti6Al4V. The thermal diffusivities of the composites containing 1 and 2 wt. % MWCNTs improved with increase in temperature and weight fraction of MWCNTs contents. A reverse trend was observed in the composite containing 3 wt. % MWCNTs, as the measured thermal diffusivities continued to drop with increase in temperature. Although this composite exhibited the best thermal diffusivities compared to the other composites up to 200 °C, a significant drop in thermal diffusivity was recorded between 250 and 300 °C respectively with the values lower than that of the unreinforced Ti6Al4V alloy. However, the thermal diffusivities of MWCNTs/Ti6Al4V composites were generally not dependent on their densification as the composites with higher weight fractions of the reinforcement had higher thermal diffusivities in spite of their lower relative densities.

1. Introduction

In spite of the attractiveness of titanium (Ti) and its alloys in high demanding light weight and high strength engineering service environments, their poor thermal performances both at room and high temperatures have continued to limit the applications of these group of materials [1, 2]. For instance, the thermal conductivity of pure Ti is substantially lower than that expected ordinarily for a metal and Ti alloys (with specific reference to Ti6Al4V), commonly exhibit even lower thermal conductivities than the commercially pure grade material [3]. The excellent thermal characteristics of carbon nanotubes

(CNTs) as suggested by several researchers, will affect positively the thermal characteristics of the resulting metal matrix composite (MMC) when used as reinforcement in the latter [4, 5]. Despite this knowledge, the development and characterization of MWCNTs reinforced Ti matrix composites (TMC) have received little attention from researchers till date. In addition, there has not been any reported work on the thermal characteristics of MWCNTs reinforced Ti6Al4V MMC up to now.

Hence, this present study was designed to bridge this identified gap in knowledge by investigating the influence of varied weight fractions of MWCNTs on the thermal diffusivity behaviour of novel MWCNTs/Ti6Al4V nanocomposites synthesized by the SPS technique. We anticipated that the results of this study would open new frontiers for the applications of these high performance new materials as possible replacements for the unreinforced Ti6Al4V alloys in the harsh and unstable service environments where they commonly used.

2. Methodology

2.1. Raw Materials

The starting raw materials used in this study were spherical shaped Ti6Al4V (APS 25 μm, TLS Technik GmbH & Co., Germany) and MWCNTs (outside diameter = 20-30 nm, length = 10-30 μm, ash <1.5 wt %, purity > 95 wt %) powders respectively.

2.2. Dispersion of MWCNTs into Ti6Al4V Metal Matrix

Different weight fractions of MWCNTs (0, 1, 2 and 3 wt %) were dispersed into Ti6Al4V matrices via the HEBM technique. The Retsch PM 400 MA, Germany was employed at a rotational speed of 50 rpm for 6 h using a ball-to-powder ratio (BPR) of 10:1. The milling machine was set to stop for 10 min after every 10 min of milling. This was to control cold welding and excessive frictional heat, which may occur due to ball-powder-vial wall interactions during milling.

2.3. Consolidation of composite powders

All the as-milled composite powders were synthesized into bulk cylindrical composite discs with dimensions Ø40 mm × 5 mm thickness via SPS (model HHPD-25, FCT Germany) technique. The sintering was carried out in vacuum at a sintering temperature of 850 °C, holding time 5 min, heating rate 100 °C/min and applied pressure of 50 MPa.

2.4. Density Measurements

The densities of the consolidated composite discs were measured following the Archimedes' principle at room temperature according to ASTM B962. The recorded density was an arithmetic mean of five consecutive measurements taken from the same sample while the relative densities were calculated as percentages of the composites' theoretical densities.

2.5. Thermal Diffusivity Measurements

Test specimens (10 mm × 10 mm × 10 mm) for diffusivity measurements were sectioned from the as-sintered bulk composite samples. The thermal diffusivities of these samples were then measured following the Laser Flash technique (NETZSCH LFA 427) in the temperature range 50 – 300 °C, and according to ASTM E1461. The recorded thermal diffusivity value was an arithmetic mean of three consecutive measurements taken on the same sample at the same temperature under investigation. This was done to ensure repeatability, accuracy and reliability of the diffusivity data collected.

2.6. Microstructural Analysis

Samples for microstructural analyses were prepared following the conventional metallographic procedures. Cross-sectioned sample surfaces were ground with successive silicon carbide papers from 200 to 1200 grade and polished to a 1 μm finish. The samples were subsequently cleaned with acetone, washed in distilled water and then dried in air. The mirror-polished samples were etched with Kroll's reagent before microstructural examination using a high resolution, field emission scanning electron microscope (JSM-7600F, JEOL, Japan).

3. Results and Discussion

3.1. Densification pattern in sintered MWCNTs/Ti6Al4V composites

Table 1 shows the densification trend in the MWCNTs/Ti6Al4V composites sintered at 850 °C. The densification pattern showed relative densities of the composites declined with increase in the weight fraction of the MWCNTs reinforcement in Ti6Al4V metal matrix. This observation was attributed to the increased difficulty in achieving a homogeneous dispersion of MWCNTs within the matrix alloy with increased content of the reinforcement arising from the agglomeration and clustering of the MWCNTs. The other possible explanation for this could be the anticipated poor interfacial bonding between the matrix and the reinforcement due to inadequate diffusion between Ti6Al4V and MWCNTs during sintering. Hence, a situation of thermal mismatch occurs between the matrix and the reinforcement [2, 4]

Table 1: Densification characteristics of MWCNTs/Ti6Al4V composite compacts consolidated by SPS at 850 °C

MWCNTs content (wt %)	Theoretical density* (g/cm^3)	Measured density** (g/cm^3)	Relative density (%)
0	4.43	4.38 ± 0.0001	98.80 ± 0.01
1	4.41	4.32 ± 0.0021	97.99 ± 0.05
2	4.38	4.29 ± 0.0010	97.98 ± 0.23
3	4.36	4.20 ± 0.0015	96.34 ± 0.03

* Rule of mixture (ρ Ti6Al4V = 4.43 g/cm^3 and ρ MWCNT = 2.1 g/cm^3),
** Archimedes principle

3.2. Microstructure

The results of the microstructural analyses performed on the unreinforced Ti6Al4V and MWCNTs/Ti6Al4V composites containing varied weight fractions of MWCNTs is as shown in Figure 1. The secondary electron image of the sintered unreinforced matrix alloy (Figure 1a) exhibited some features of a near fully lamellar Widmanstätten structure, typical of Ti6Al4V. However, with the addition of MWCNTs, the observed microstructure in the composites (Figure 1b-d) changed from lamellar to equi-axed structures consisting of distinct α and β phases. The β phase (white) being on the grain boundaries while the α phase is the matrix.

It was also observed from the composites that the amount of pores in their microstructures increased with increased weight fractions of MWCNTs in Ti6Al4V matrix. This is consistent with the earlier stated deterioration in relative density as seen in Table 1. These pores were thought to be predominantly responsible for the declining relative densities with increased weight fraction of MWCNTs reinforcement. Retained and Reagglomerated MWCNTs were also seen in the microstructures of the composites containing 2 and 3 wt. % MWCNTs respectively. This again affirms the inefficient dispersion of MWCNTs occurring with increased weight fractions of the reinforcement with the matrix metal alloy.

3.3. Thermal Diffusivity Behaviour of MWCNTs/Ti6Al4V Composites

The thermal diffusivity behaviour of SPS Ti6Al4V and MWCNTs/Ti6Al4V composites following a series of Laser Flash experiments is as illustrated in Figure 2. The recorded thermal diffusivities of the sintered composites containing 1 and 2 wt. % MWCNTs respectively were generally improved over that of the unreinforced Ti6Al4V matrix alloy at the temperatures under investigation. It was also found that diffusivities in these samples increased with increase in temperature and weight fractions of MWCNTs in Ti6Al4V.

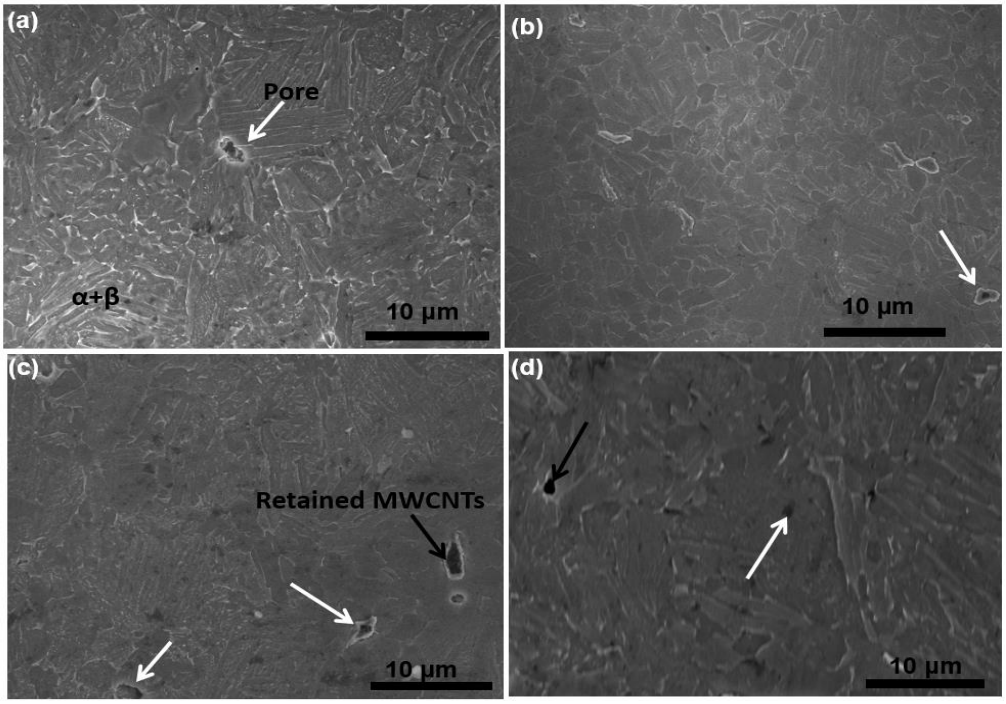

Figure 1: SEM images of MWCNTs/Ti6Al4V composites sintered at 850 °C, containing varied weight fractions of MWCNTs (a) 0 wt. %, (b) 1 wt. %, (c) 2 wt. % and (d) 3 wt. %

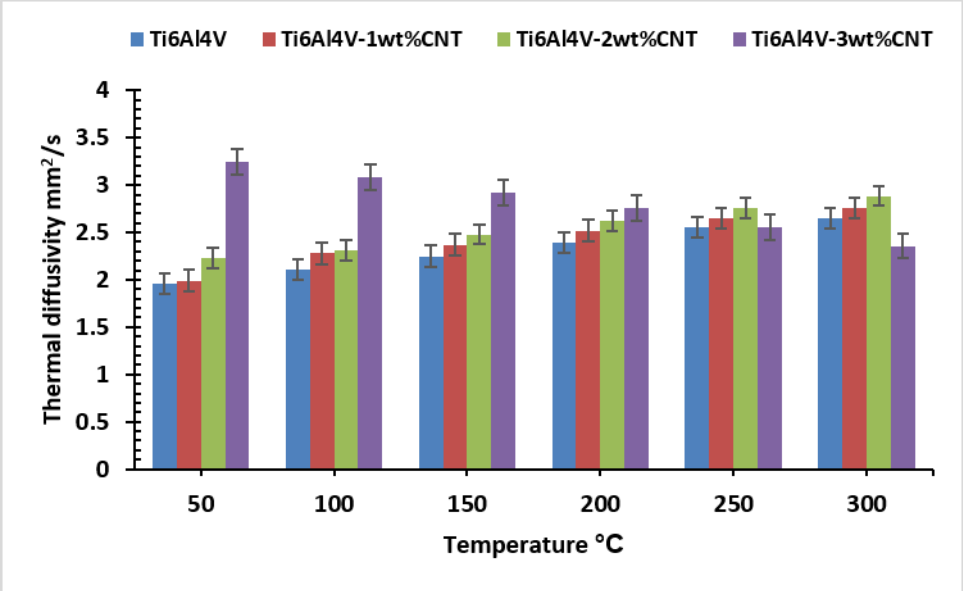

Figure 2: Thermal diffusivity characteristics in Ti6Al4V and MWCNTs/Ti6Al4V composites with varying wt. % MWCNTs contents sintered at 850 °C

Nevertheless, a reverse trend was observed in the composite containing 3 wt. % MWCNTs reinforcement. The measured thermal diffusivity values in this sample declined as the temperature under which the parameter was investigated increased. Although this composite exhibited the best diffusivity values at temperatures 50 and 200 °C, a significant drop in the measured diffusivities was noticed between 250 and 300 °C to the extent of dropping below that of the unreinforced Ti6Al4V alloy.

This observed trend in the thermal diffusivity behaviour of MWCNTs/Ti6Al4V composites suggests that the improvement in the thermal diffusivity of Ti6Al4V alloy with MWCNTs addition is dependent on the extent of dispersion of the reinforcement within the metal matrix. The results from this present study is consistent with that obtained from an earlier study on the thermal diffusivity characteristics of pure Ti matrices reinforced with MWCNTs [2]. It was reported that the thermal diffusivities of the MWCNTs/Ti composites improved continuously with increase in the weight fraction of MWCNTs in a declining growth rate manner.

This present study also shows that the thermal diffusivity characteristics of the composites is independent of the relative densities of the samples as the composites with higher weight fractions of the MWCNTs reinforcement had higher thermal diffusivities in spite of their lower relative densities. In addition, the results of this study showed that thermal diffusivity values in the composites were enhanced with increased temperatures. This suggests that the measured parameter was dependent on thermal transport mechanisms. However, this must have been hampered with increased weight fractions of the MWCNTs reinforcement leading to the observed diminishing thermal diffusivities in the 3 wt. % composite at higher temperatures. This could be attributed to the presence of pores arising from increased tendency of MWCNTs to agglomerate and be clustered together as seen in Figure 1c and d. There pores are capable of acting as thermal barriers to the inherent heat flow within the composites and thereby reduce their thermal diffusivities [6, 7].

4. Conclusion

The thermal diffusivity behaviour of spark plasma sintered unreinforced Ti6Al4V and MWCNTs/Ti6Al4V composites containing varied weight fractions of MWCNTs were investigated between 50 – 300 °C in this present study. The results showed that the relative densities of the synthesized composites deteriorated with increase in the weight fractions of the MWCNTs reinforcement within the Ti6Al4V matrix. Although, the composites exhibited improved thermal diffusivities compared to the unreinforced Ti6Al4V alloy, the extent of enhancement was found to be dependent on the dispersion of MWCNTs within the metal matrix. Thermal diffusivities in the 3 wt. % composite showed a substantial deterioration between 250 and 300 °C. This was attributed to the presence of pores as a result of the re-agglomeration and clustering of MWCNTs in this sample. These pores acted as barriers to thermal transport mechanisms. Hence, the observed decline in thermal diffusivities. Thermal diffusivities were also found to be intrinsic to the samples as the composites that had lower relative densities exhibited higher thermal diffusivity values.

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