JMEPEG (2010) 19:231–236
DOI: 10.1007/s11665-009-9458-y

1059-9495/$19.00

Synthesis of TiN Reinforced Aluminium Metal Matrix Composites Through Microwave Sintering

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(Submitted December 12, 2008; in revised form March 12, 2009)

Al-TiN (10, 20, 30 wt.%) composites were fabricated by using microwave radiation. Al and TiN powders were selected as starting materials, mixed in a ball mill for ~10 min and sintered for various times. Results indicate that an optimum microwave sintering time of 2 min was essential and responsible for the improved densification and mechanical properties. The presence of TiN particles at grain boundaries plays a significant role in improving the densification and hardness values. Dry sliding wear results show the improved wear resistance of the composite (Al-TiN) due to the presence of TiN particles and the wear results are superior to the Al-TiN samples made by hot pressing technique.

Keywords mechanical properties, metal matrix composite, microstructure, microwave sintering, powder metallurgy

1. Introduction

Composite materials are well known for their tailored properties that are achieved by combining two or more materials to obtain the required spectrum of properties. The most distinctive feature of composites is that, while the individual constituents retain their properties, they synergize to give properties which may not be found in any one of them alone. Recently, extensive research study has been carried out, and it was shown that there is tremendous promise of ceramic-reinforced metal-matrix composites (MMCs) (Ref 1-3). Various processing techniques have been developed to engineer composites for diverse fields of applications (Ref 1, 2) such as aerospace, defense, automobile, and sports sectors. In MMCs, the addition of a small amount of second-phase materials with high shear strength imparts unique properties to the base materials (Ref 4-6). The strength of MMCs is strongly influenced by the shape, orientation, and volume fraction of the second phase. Thus, the different possibilities of topological combinations of the different materials in space have led to the development of different types of composites such as laminated, continuous fiber, reinforced, interpenetrating, discontinuously reinforced etc.

Aluminum-based MMCs have received much attention due to their light weight, high strength-to-weight ratio, ease in melting and casting, and abundantly available raw materials. Most of the research on the Al-based composites is to improve their mechanical properties by the incorporation of hard ceramic oxides/carbides/nitrides into the metal matrix. Among various processing techniques, the powder metallurgy route has been extensively studied. The process includes cold-isostatic pressing (CIP) followed by sintering (Ref 7), hot-pressing (Ref 8), and hot-isostatic-pressing (HIP) (Ref 9) techniques. Another route of synthesis is via a liquid metallurgy route where the reinforcing phases are either infiltrated with (Ref 10-12) or injected into the liquid metal. Mixing, in situ growth, and spray-forming techniques are also used to prepare the MMCs. The spray forming of composites involves the forming of composites by spraying liquid metal and reinforcing particulates simultaneously (Ref 13-15). Among various ceramic additions to Al, SiC has been given much greater attention by many researchers but the Al-SiC still has some limitations such as the formation of Al_4C_3 during high temperatures which impairs the mechanical properties of the Al MMCs (Ref 1). Thus, efforts are made to overcome the problems by incorporating other reinforcing materials such as TiN (Ref 16) and also through innovative synthesizing techniques like microwave sintering (Ref 17-20).

Microwave sintering (Ref 21, 22) has been recognized as a time-saving and cost-effective process as compared to the conventional powder metallurgy processes for synthesizing composite materials. In the normal mode of resistance heating, the direction of heat flow is from outside to inside of the powder compact. This leads to inferior microstructural properties of the core. In contrast, for microwaves, the direction of heating or heat flow is from the inside of the powdered compact to the outside. However, this does not yield very good microstructural properties of the surface. Thus, both these methods are not perfect to yield a good overall property of the sample. A combined action of microwaves and microwave-coupled external heating has therefore been proposed to overcome the drawbacks of both these sintering methods, to realize rapid sintering from both outside and inside of the powdered compact. A microwave susceptor material such as SiC is, therefore, used to assist sintering from outside (Ref 16, 22).

This article mainly deals with three aspects of the microwave synthesis of composites: (1) incorporating TiN in Al matrix in place of SiC, (2) overcoming the problems from melting, spray-forming and powder metallurgy routes by opting for the microwave-sintering technique, and (3) comparing the improvement in mechanical properties with our earlier studies (Ref 23).

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2. Experimental Details

Air-atomized aluminum powder of 99.7% purity, globular copper powder of 99.5% purity, and TiN powders were selected as the starting materials. Hereafter, % is always reported as wt.%. The TiN powder was prepared by nitriding the Ti sponge (>98% pure) at 1823 K for 45 min. After nitridation, the lumpy mass was manually ground to yield powder by using an agate mortar. The powder was again nitrided and reground to ensure the complete nitridation of Ti and the formation of a single-phase TiN powder. The process of nitridation and grinding was repeated three times (Ref 23). Al powder (99.7% purity) was prepared by air-atomization technique in our laboratory using a graphite nozzle of 3-mm diameter and gas pressure of 50 psi and sieved to ~50 μm. Copper powder (99.5% purity) was used to improve the wettability of Al and TiN (Ref 23). The required amounts of Al, TiN, and Cu powders were taken in three separate batches with the compositions given in Table 1. The powder mixture was thoroughly mixed using agate motor, and cylindrical pellets of diameter 22 mm were prepared by applying a pressure of 200 MPa. Subsequently, the pellets were transferred to a specially designed double-walled reactor consisting of two chambers, inner and outer and placed at the center of a microwave oven (Fig. 1). The samples were kept in the inner chamber and enclosed by the outer chamber filled with SiC susceptor since it is a good microwave absorber (ε 10.0). Since the SiC susceptor is larger as compared to the sample size, and the microstructures after sintering at edge, center, and another edge are uniform, it is believed that sintering occurred uniformly. The reaction chamber also had gas purging facility. Argon gas of LR1 grade supplied by BOC, Jamshedpur, India was passed through the inlet tube. The whole reactor set up was maintained in an argon atmosphere and sintering was carried out several times. The samples were sintered in a continuous supply of argon for 1, 2, 3, and 4 min, respectively, and allowed to cool for 10 min in the microwave oven.

Table 1 Various compositions of Al-TiN MMCs with 4% Cu

	Composition, wt.%	
Sample	**Al**	**TiN**
A	86	10
B	76	20
C	66	30

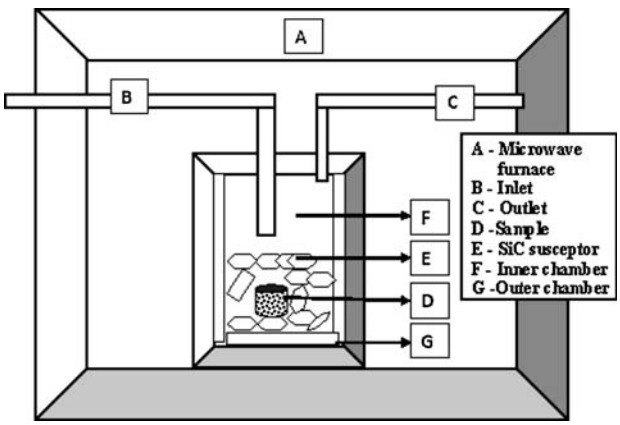

Fig. 1 Schematic diagram of double-walled setup in the microwave oven

The sintered samples were taken out of the reactor after closing the gas supply. It was observed that the shrinkage of pellets increases with increase in sintering time resulted in higher densification. The sintered samples were polished and prepared for hardness determination and metallographic examination. Both the bulk hardness of Al-TiN composite and micro hardness of individual phases including the interface was determined using Vickers hardness tester and Leitz micro hardness tester, respectively. The samples were etched with standard Keller's reagent and examined under both optical microscope and SEM (JEOL 840A). The chemical compositions of the individual phases were confirmed using energy dispersive x-ray spectroscopy (EDS). Dry sliding wear test was carried out on a pin-on-disc wear test machine (TR 20 LE, DUCOM, Bangalore, India) with a sample of 8-mm diameter and 40-mm length. The sintered pellet has a height of 5 mm which has been raised to 40 mm by adhering the samples to a steel sample of 8-mm diameter and 35-mm height. A thermocouple was inserted at a height of 2.5 mm from the bottom of sample for recording the variation in temperature during sliding test. The specimens were allowed to slide against a rotating EN 32 steel disc (counter face) of hardness 65 Rc, where coefficient of friction, temperature, and wear were monitored as a function of sliding distance up to 3200 m at 1 and 2 kg loads.

3. Results and Discussion

Figure 2 shows the XRD pattern for the experimental alloys A, B, and C. Samples A, B, and C denote Al-10%TiN, Al-20%TiN, and Al-30%TiN, respectively. All the samples showed the presence of TiN peaks along with Al peaks. It is

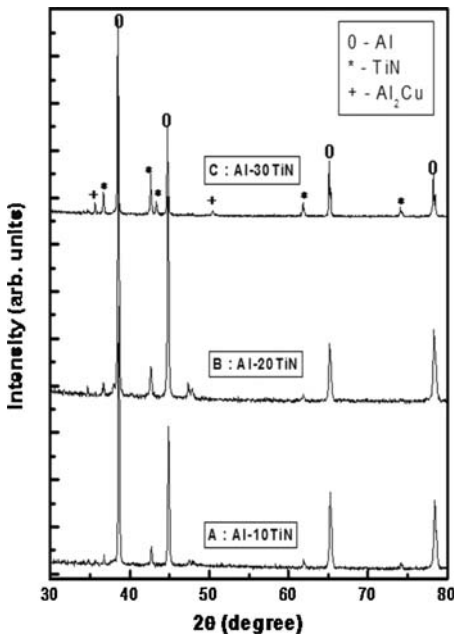

Fig. 2 The XRD pattern for samples A, B, and C

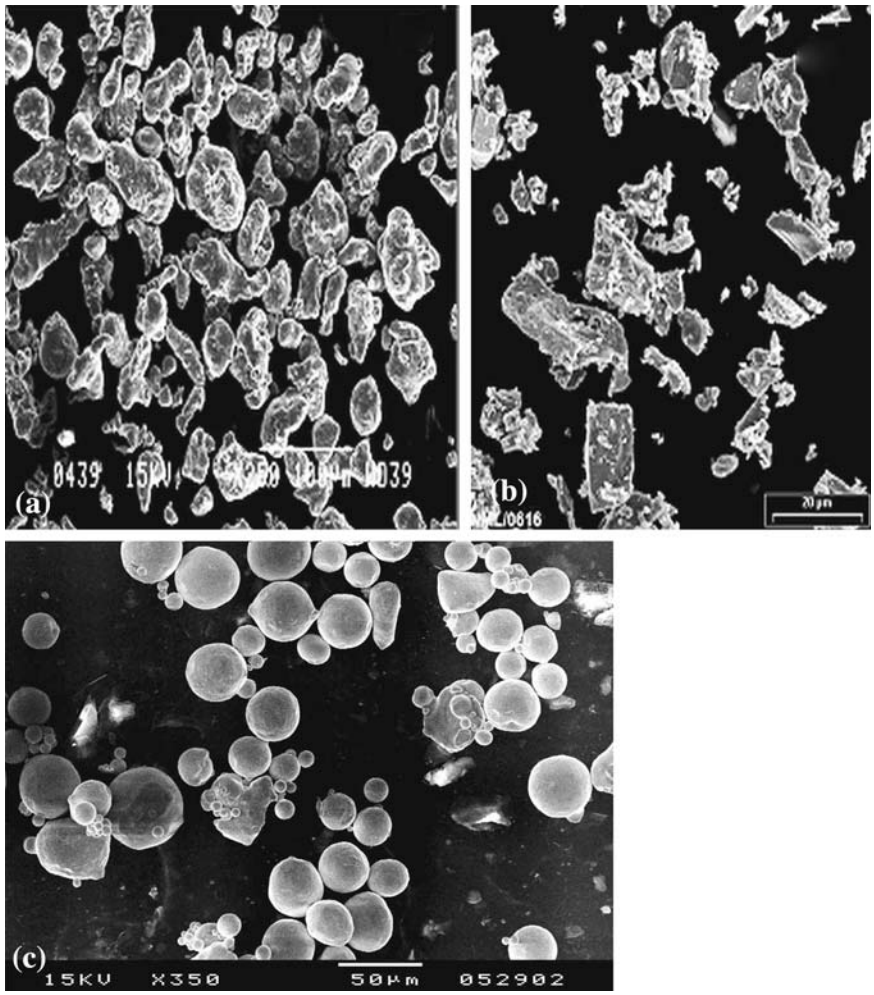

Fig. 3 (a) SEM image of atomized Al powder, (b) SEM image of TiN powder, and (c) SEM image of Copper powder

clearly observed that the intensity of TiN peaks has increased with increase in TiN content from 10% to 30%. The formation of Al_2Cu is also observed along with the TiN and Al peaks in the XRD pattern. There are some peaks for sample B (Al-20TiN) at 2θ values between 46° and 48° and are unidentified. Figure 3(a), (b), and (c) shows the SEM images of Al, TiN, and copper powders, respectively. The average particles size of copper powder is ~25 µm and most of the particles are globular in shape. The size distribution of Al particles mostly lies in the range of 40-45 µm with some fine particles in the range of 5-8 µm. As the TiN powder was prepared by nitriding Ti sponge, it showed porous structure as observed from Fig. 3(b). It is known that such porous structure is more favorable for sintering (Ref 24) and also expected to have better bonding with Al. The particle size distribution of TiN powder exhibited that most (~75%) of the particles are in the range of 4-6 µm. Some TiN particles are agglomerated due to their fineness and are in the range of 1-2 µm. Similarly, the average particle size for copper and Al is in the range of 35-40 µm.

TiN is a highly stable covalent compound, and hence its wettability with metals and ceramics is very poor. Copper, which is known to increase the wettability of TiN (Ref 23), was added to the Al-TiN mixture. Figure 4(a) and (b) shows the SEM images of samples A and C, respectively. Most of the TiN particles observed are in the range of 4-6 µm and marked as TiN (Fig. 4a). A good interfacial metallurgical bonding is observed between a large TiN particle and Al matrix taken at higher magnification (Fig. 4c). A close observation also revealed the presence of interfacial phase formation having the composition of Al_2Cu in the Al matrix which is responsible for improved wettability. The presence of TiN and Al_2Cu phases is confirmed by EDS. There results suggest that some percentage of Cu is reacting with Al to form Al_2Cu during sintering, which is consistent with the results obtained from the XRD studies. No attempt is made to determine the Al_2Cu content, and therefore, it is difficult to predict how much Cu reacted with Al to form Al_2Cu. Moreover, the effective volume fraction of copper that is not reacted with any other elements is less than ~1% and not detected in XRD studies. The XRD results obtained for the Al-TiN samples are similar to the samples prepared by powder metallurgy route that are observed in our earlier studies (Ref 23).

The mechanical properties (hardness values) of microwave-sintered samples are much superior to the earlier reported values (Table 2). It is inferred from the table that the bulk hardness of the samples A, B, and C are 58, 73, and 90 Hv, respectively. This suggests that microwave sintering is responsible for the enhancement of bulk hardness. Furthermore, microwave sintering is a cost-effective and less time-consuming process. A visual examination followed by hardness testing was carried out for samples sintered for 1, 2, 3, and 4 min. The

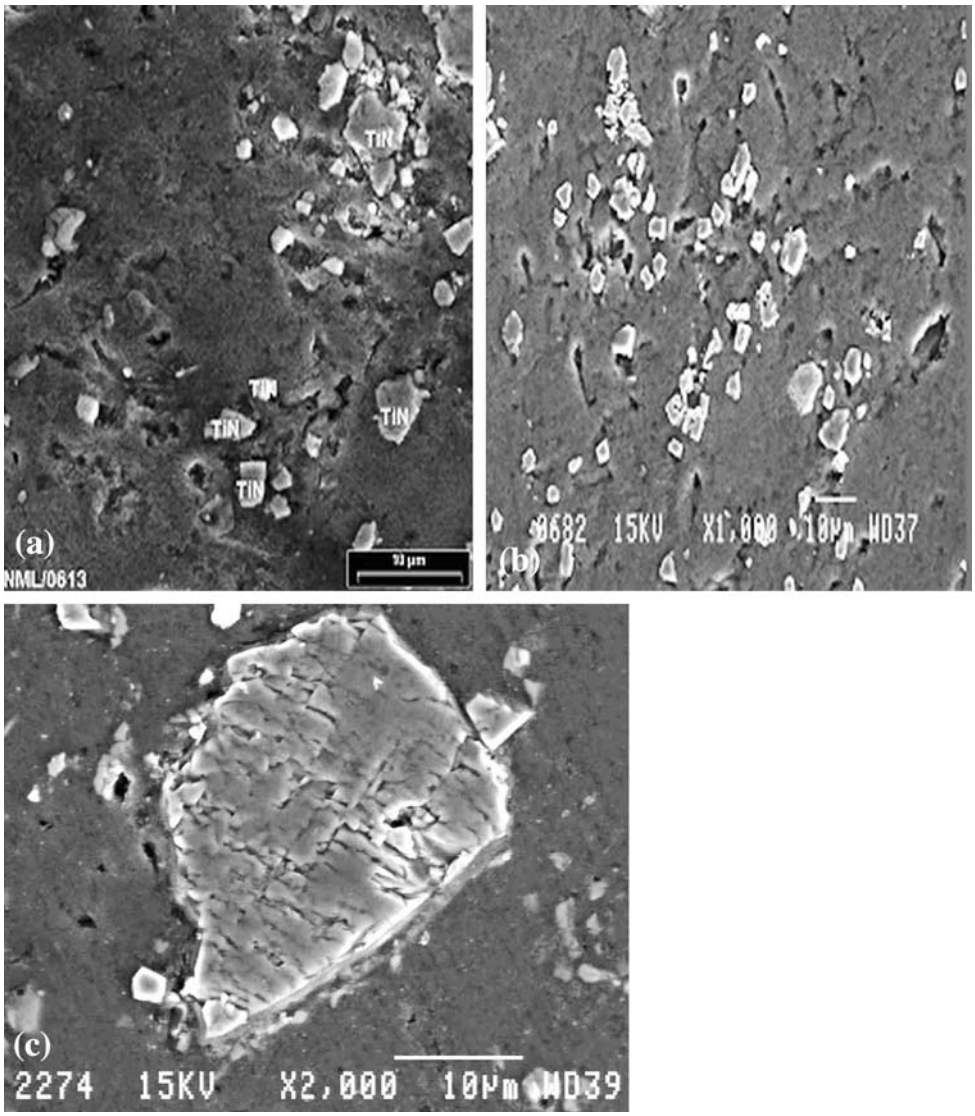

Fig. 4 (a) SEM images of sample A, (b) SEM images of sample C, and (c) shows better bonding of TiN with Al matrix for sample B

Table 2 Hardness values of samples A-C

Sample	Bulk hardness, Hv		Microhardness, Hv
	Sintered (Ref 23)	Microwave sintered	
Al-10TiN-4Cu	50	58	Matrix ~105
Al-20TiN-4Cu	…	73	Interface ~180
Al-30TiN-4Cu	54	90	On Particle ~2500

samples microwave processed for 1 min did not sinter whereas the samples processed for 2, 3, and 4 min showed excellent sintering behavior and good hardness. However, a sintering time of more than 2 min (3 and 4 min) caused sweating of Al from the pellets. Since the trend is similar for all Al-TiN (10, 20, 30 wt.%) samples, we have reported the hardness values only for 2-min-sintered samples. These results further suggest that a reaction time of 2 min is sufficient for good sintering, and that the higher holding time is responsible for probably exceeding the temperature of ~700 °C due to which Al melts out from the pellet. The increased hardness could be attributed to increase in the amount of hard phases (TiN) in the Al matrix. Furthermore, it is observed that Al matrix exhibited higher micro hardness values compared to pure Al sample. This may be due to the distribution of fine TiN particles in the Al matrix. In addition to this, a higher micro hardness value (~180 Hv) was observed at the interface of a large TiN particle as compared to the Al matrix. The possible reason for the higher hardness value could be that the applied load was shared between Al matrix and TiN particle. No separate micro hardness measurement was made for Al_2Cu compound as their concentration is very low. The micro hardness value for TiN particles was ~2500 Hv and is close to the reported value (Ref 25).

Figure 5 shows the wear performance for a continuous sliding distance for samples A-C at two different applied loads, 1 and 2 kg. The wear loss is plotted against the sliding distance. It is clear from Fig. 6 that samples at 2-kg load showed higher wear loss as compared to samples at 1-kg load. Sample C exhibited higher wear resistance as compared to samples A and B. This is expected as higher content of TiN and good

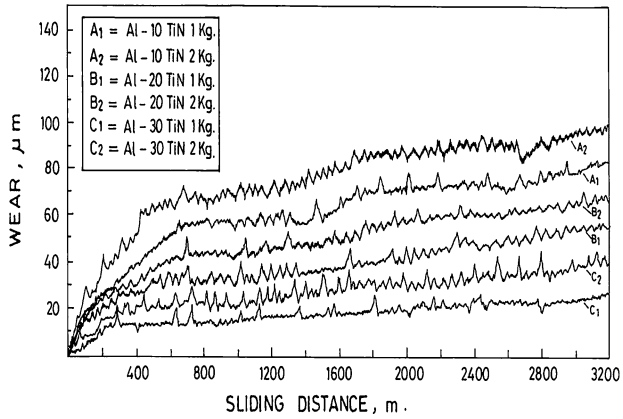

Fig. 5 Sliding Wear behavior of Al-TiN composites

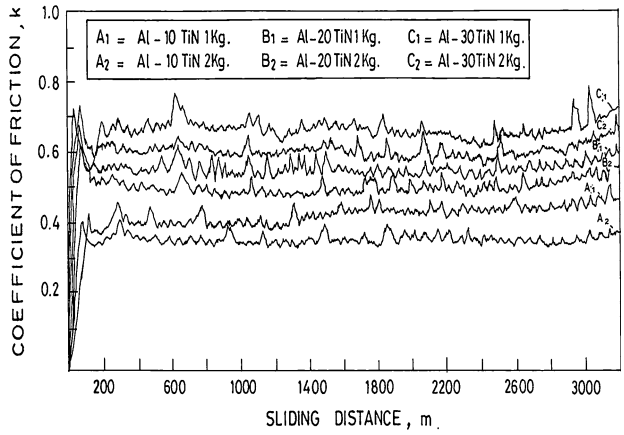

Fig. 6 Coefficient of friction variation with sliding distance

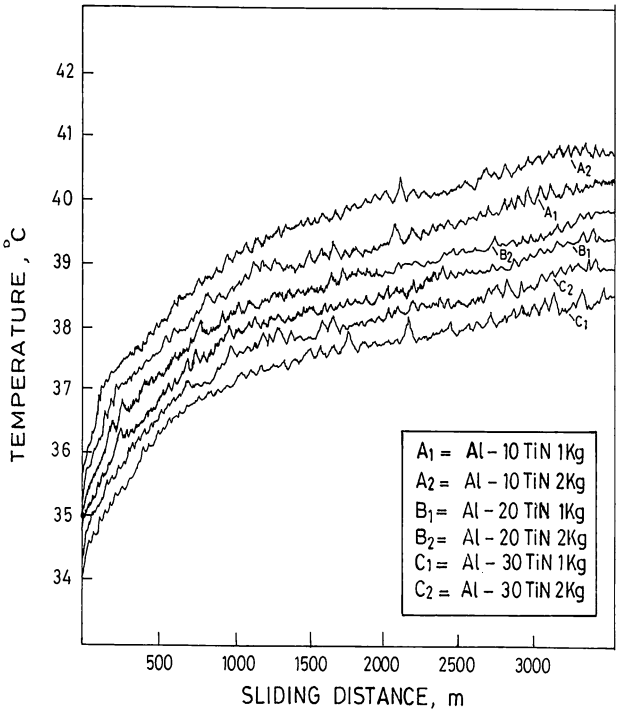

Fig. 7 Variation of temperature with sliding distance

metallurgical bonding with Al matrix resulted in better wear resistance. Figure 6 shows the values of coefficient of friction for a continuous sliding distance of samples A, B, and C. It is observed that initially the coefficient of friction between the sample and steel disc increases steeply up to a maximum value and remains stable until the end of sliding distance of 3200 m. In some cases, a marginal increase in the friction values is also observed. The coefficients of friction (k) for the samples A1 and A2 are ranging from 0.40 to 0.30, which and are comparatively lower than the samples C1 and C2 (0.70 to 0.60). The samples at higher loads showed low coefficient of friction values and is in agreement with the observations made by Grzesik et al. (Ref 26). At higher loads, lower coefficient values are observed for TiAlN coated samples. The temperature variation during sliding wear test is also observed and shown in Fig. 7. The results suggest that initially there is a sudden rise in the temperature up to a sliding distance of 200 m due to adhesion mechanism between the sample and the disc and beyond that there is a marginal increase in the temperature.

4. Conclusions

1. Al-TiN MMCs containing 10, 20, and 30 wt.% TiN have been successfully synthesized by using microwave radiation technique. The process of microwave sintering has been considered as the most convenient and economical as compared to other conventional sintering processes.
2. The presence of SiC susceptor during microwave sintering is responsible for an efficient way of supply of heat to the samples in the microwave oven.
3. Our experimental results suggest that the microwave sintering of 2 min is sufficient and further increase in sintering time led to melting out of Al from Al-TiN pellet, suggesting that the larger reaction timings are responsible for increase in the reaction time and probably when the temperature reaches more than ~700 °C. The other conventional sintering processes, in general, take few hours for proper sintering.
4. Increasing TiN from 10 to 30 wt.% showed superior hardness and wear resistance properties as compared to Al-TiN composites prepared by hot pressing.

Acknowledgments

The authors wish to express their sincere thanks to Prof. S.P. Mehrotra, Director National Metallurgical Laboratory (NML) for his kind permission to publish this article. We sincerely thank Mr. M.K. Gunjan and Mr. B. Mahato for SEM and XRD studies, respectively. We also thank Mr. K. Seetharaman and Mr. Y.P.K. Rao, technical officers, NML, Jamshedpur for fabricating the quartz reaction chamber used in the microwave oven and providing engineering drawing support, respectively. We sincerely thank Prof T.G. Langdon, University of Southern California, USA for his invaluable help in correcting this manuscript.

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