Effect of scandium additions on pressure less sintering of Al-TiN metal matrix composites

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Synthesis Al-TiN (10, 20, 30 wt%) composites without and with Scandium (Sc) (0.2 and 0.6 wt%) have been prepared using pressure less sintering. An amount of 4 wt% Cu is added in all the samples for the better wettability purpose. Powders of Al, Cu and TiN are mixed thoroughly and compacted to pellets of 12 mm diameter and 4 mm thickness. Samples of Al-TiN are sintered at 450, 550 and 620 °C in a controlled atmosphere tubular furnace purged with Ar gas. The hardness results suggest that samples sintered at 620 °C and higher TiN contained samples exhibited higher hardness values. The samples sintered at other temperatures showed abrupt hardness values which suggest that the sintering temperatures are not sufficient for complete sintering. Further sintering was carried out at 620 °C for scandium added Al-TiN composites. The results suggest that the 0.2% Sc additions did not show significant improvement in microstructure and hardness. But the samples with 0.6% scandium showed significant improvement in hardness and very fine grain structure with very good interfacial bonding between the matrix and the TiN particles. The Al-0.6Sc-30TiN composite showed the best hardness and microstructure of all the tested samples. The wear behavior of the developed composites indicated that the scandium additions not only showed significant improvement in microstructure and improved hardness results but also exhibited superior wear resistance properties.

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1. Introduction

In the recent past, extensive research work has been carried out and it is shown that there is tremendous promise of ceramic reinforced metal-matrix composites (MMCs) [1–3]. Processing techniques have been developed to synthesize the MMCs for diverse field of applications [1, 2] in aerospace, defense, automobile and sports sectors. In MMCs, addition of a small amount of second phase materials with high shear strength was added to the base materials to obtain unique properties [4–6]. Among the various MMCs, Al based MMCs have received much attention due to their light weight, strength to weight ratio, ease in melting and casting and most abundantly available raw materials. Most of the research on the Al based composites is to improve its mechanical properties by the incorporation of hard ceramic oxides (carbides) nitrides into the metal matrix. Powder metallurgy route has been extensively studied. Another route of synthesizing of MMCs is by liquid metallurgy route, where the reinforcing phases are either infiltrated [7–9] 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 composite involves spraying of both liquid metal and reinforcing particulates simultaneously [10]. Among various ceramic additions to Al, SiC has been given much attention by many researchers and the synthesizing of Al-SiC MMCs is still has some limitations [1] and hence efforts are made to overcome the problems by incorporating other reinforcing materials such as TiN [11] and also to study the sintering behavior in pressure less conditions. In the recent past, the present authors had studied the sintering behavior of these composites at hot pressing conditions [12].

2. Experimental procedure

Aluminum powder of 99.7% purity, copper of 99.5% purity and TiN powders were selected as the starting materials. Hereafter, % is always reported as wt% only. The preparation of TiN powder is by selecting Ti sponge (>98% pure) and nitriding the same at 1823 K for 45 min and 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 complete nitridation of Ti and the formation of a single-phase TiN powder [12]. Al powder (commercial purity) was prepared by atomization technique in our laboratory using a graphite nozzle of 3 mm diameter and at a gas pressure of 50 psi and sieved to -300 mesh (<53μm). Copper powder (-300 mesh and 99.5% pure) was used to improve the wettability of Al and TiN. 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 by hand using agate motor and cylindrical pellets of diameter 22 mm were made by applying load of 200 MPa. Subsequently, the pellets were transferred to a tubular furnace where temperatures of 450, 550 and 620 °C and Ar atmosphere were maintained. The sintered samples were polished and prepared for hardness determination and metallographic examination. Bulk hardness of Al-TiN composite was determined using Vickers hardness tester at 5 kg load. The samples were etched with Kellers reagent (2.5 ml HCl, 1.5 ml HNO_3 and 1 ml HF and rest is H_2O) and examined under SEM (JEOL 840A, Japan make). Dry sliding wear test was carried out on a pin-on-disc wear test machine (TR 20 LE, DUCOM make, Bangalore, India) with

a sample of 8 mm diameter and 40 mm length. The diameter and height of the sintered sample was 8 and 5 mm respectively. The sample size requirement for conducting the sliding wear test is 40 mm height. Therefore, the sintered pellet was made to 40 mm height by attaching a steel sample of similar diameter. The specimens were allowed to slide against a rotating EN 32 steel disc (counter face) of hardness 65 Rc, where co-efficient of friction, and wear loss in microns were monitored as a function of load (1.5 and 2.5 kg) up to 3200 m sliding distance.

3. Results and discussion

Initially, samples A, B, C are sintered at 450, 550 and 620 °C for 20 min holding time. Since the proper sintering was not observed for samples that are sintered at 450 and 550 °C, all the remaining sintering tests are carried out at 620 °C only for all the alloys. Fig. 1. shows the XRD pattern of Al-10TiN, Al-20TiN and Al-30TiN, and marked as A, B and C respectively. XRD studies suggest that intensity of Al peak decreased and TiN intensity is increased with the increase in TiN content. In addition, Al_2Cu peaks also have been observed since 4% of Cu is added to all the samples. Further, the results suggest that only Al and Cu are reacted and there is no product of Cu with TiN.

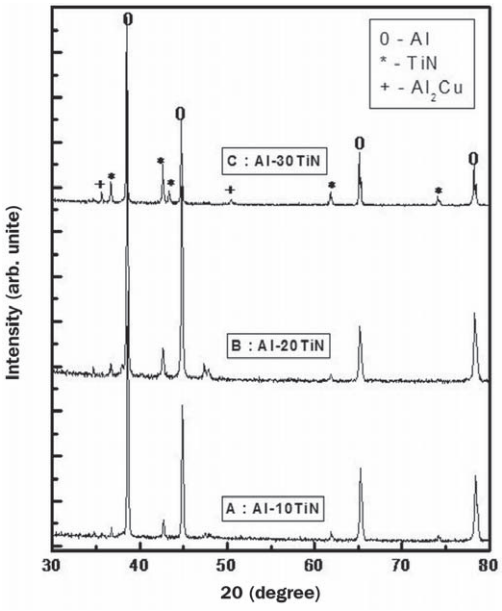

Fig.1. XRD pattern of samples A-C
1. ábra Az A-C minták röntgen diffraktogramjai

Table 1. shows the chemical composition and various identifications for each sample of all the experimental alloys. Similarly Table 2. shows the density and hardness values for the Al-TiN MMCs with and without Sc contents (samples A–I). It is seen that there is no significant change in the density of samples D, E, F as compared to A, B, C samples. A nominal improvement in hardness measurements is noticed for samples D, E and F. However, significant change in the density and hardness values are observed for samples G, H and I when the Sc content is enhanced to 0.6% (Table 2.). From our earlier studies also, the grain refinement of Sc addition of CPAl suggest that there is no affect of Sc additions below 0.5% [15]. Hence the present results are also in conformity with our earlier studies.

Sample designation	Composition, wt%		
	Al	TiN	Cu
A	86	10	4
B	76	20	4
C	66	30	4
	Al-0.25Sc	**TiN**	**Cu**
D	86	10	4
E	76	20	4
F	66	30	4
	Al-0.6Sc	**TiN**	**Cu**
G	86	10	4
H	76	20	4
I	66	30	4

Table 1. Sample designation and composition for all the experimental alloys
1. táblázat A kísérleti minták jelölése és összetétele

Sample designation	Density, g/cc	Hardness (H_v5)
A	2.59	57
B	2.67	61
C	2.78	72
D	2.58	59
E	2.70	64
F	2.87	75
G	2.65	68
H	2.77	77
I	2.86	90

Table 2. Density and hardness values for all experimental alloys
2. táblázat A kísérleti minták sűrűsége és keménysége

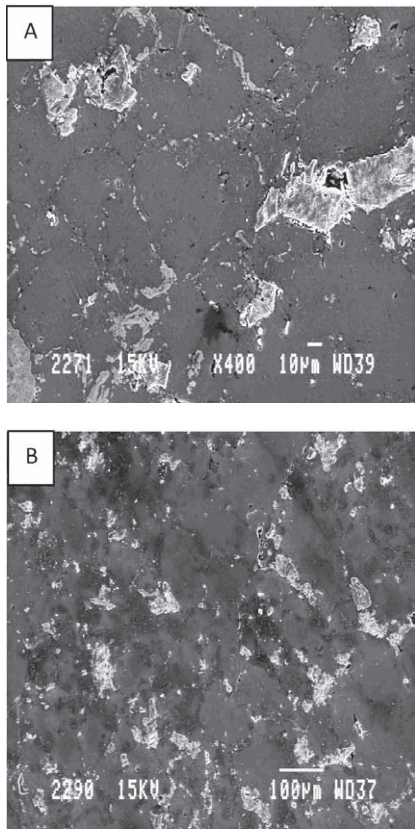

Fig. 2. SEM images of (A) Al-10TiN-4Cu and (B) Al-0.25Sc-10TiN samples
2. ábra Az Al-10TiN-4Cu (A) és az Al-0,25Sc-10TiN (B) minták pásztázó elektronmikroszkópos felvételei

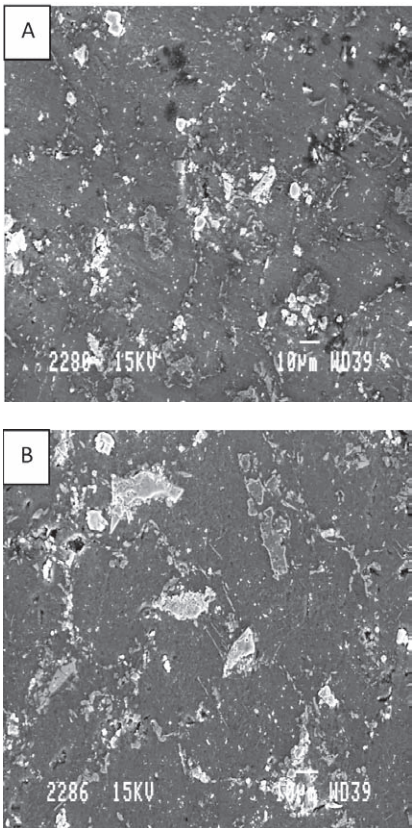

Fig. 3. SEM images of (A) Al-0.6Sc-20TiN-4Cu and (B) Al-0.6Sc-30TiN-4Cu samples
3. ábra Az Al-0,6Sc-20TiN-4Cu (A) és az Al-0,6Sc-30TiN-4Cu (B) minták pásztázó elektronmikroszkópos felvételei

Fig. 2. A, B shows the SEM images of sample A and D. Some black spots are clearly observed between the two TiN particles and are confirmed to be pores (Fig. 1. A). The bright particles are mostly in the range of 40–45µm are confirmed as TiN particles and the grey color small particles ~5 µm that are along the grain boundaries are observed as Al_2Cu particles. Some amount of agglomeration of TiN particles is observed and this is possibly occurred during sintering process. The volumetric concentration of Al_2Cu particles are lower as compared to TiN particles and the presence has been already confirmed from our XRD studies. The microstructural studies further confirm that the TiN particulates are uniformly distributed in the Al matrix. The volume concentrations of TiN particles are increased as the TiN content is increased from 10 to 30% (not shown here). Fig. 2. B shows the presence of few small particles and are possibly Al_3Sc precipitates. In addition to the Al_2Cu particles at grain boundaries, TiN particles also have been precipitated along the grain boundaries. This has a prolonging effect of interlocking of Al grains which, along with the difference in thermal expansion co-efficient of Al and TiN [14], strength of composite is boosted and it becomes slightly brittle. In view of understanding the effect of higher Sc additions, 0.6% is added to Al-4Cu-TiN samples and Fig. 3. A, B shows the SEM images of G and H samples respectively. As expected, more TiN particles are in sample-H (Fig. 3. B) as compared to sample-G (Fig. 3 A). However, more refined microstructure is observed in these cases as compared to samples D–F. This directly suggests that Sc has significantly refined the microstructure and this has directly influence on the hardness values also. Sample A had the density of 2.59 g/cc where as the sample I has 2.86 g/cc. This higher density is due to the Sc additions and refined structure that ahs led to less porosity levels and ahs direct influence on the density and hardness values. Sample A has the hardness values of 57 and sample C has 76 HV. This is due to higher concentration of TiN content whereas sample I gave 90 HV as compared to sample C (76 HV). This is due to the grain refinement affect due to Sc additions. In order to confirm the presence of Sc, Cu and Ti, EDX has been carried out on the sample H and the pattern is shown in Fig. 4. It can be clearly seen from this pattern that Al, Ti, Cu and Sc elements are present and are in appropriate quantities. When scanning is done on the grey particles, only Cu and Al peaks are observed and from the concentration levels, the particles can be confirmed as Al_2Cu. Similarly, TiN and Al_3Sc also confirmed from the EDX studies (respective patterns are not shown here). Fig. 5. shows the SEM image of sample I where one TiN particle at higher magnification. A close observation of this image suggests that there is absolutely no interfacial deboning between the matrix and the particle. When compared to sample C, the porosity levels also very low for sample I.

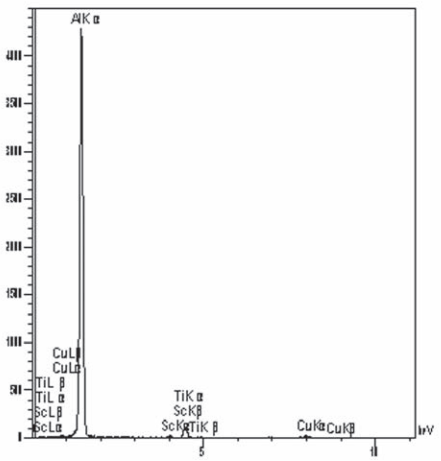

Fig. 4. EDX pattern on the sample I
4. ábra Az I minta elektrondiffrakciós képe

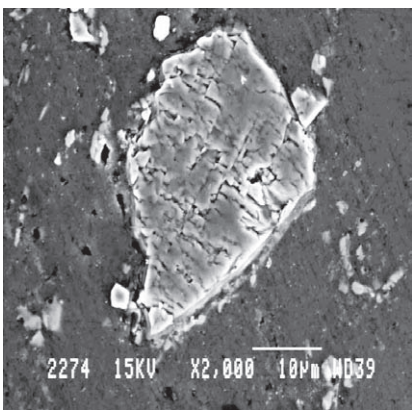

Fig. 5. SEM image of Al-0.6Sc-30TiN-4Cu at higher magniifcation
5. ábra Az Al-0,6Sc-30TiN-4Cu minta nagyobb nagyítású pásztázó elektronmikroszkópos felvétele

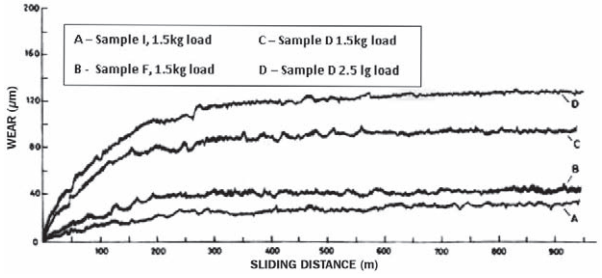

Fig. 6. Sliding wear performance of Al-TiN MMCs under different condiitons
6. ábra Az Al-TiN fémmátrixú társított anyagok csúszási kopása különböző körülmények között

Fig. 6. shows the sliding wear performance of samples D and I. In case of sample D the response of load is clearly shown in the Fig. 6. C, D. It can be seen from this graph; the wear is linearly increased up to the sliding distance of 150 m and followed continuously up to 950 m. The sample D experienced a total wear loss of ~75 μm at 1.5 kg load (Fig. 6. C) and further increase in load to 2.5 kg, the total wear loss is ~115 μm (Fig. 6. D). However when the TiN concentration is high, i.e. in sample I, the sliding wear response is different. In fact, the effect of Sc additions is mainly studied in this case. Fig. 6. A, B shows the wear performance of sample F and sample I. It can be seen from the graph that lower Sc content exhibited higher wear loss suggesting that 0.25 % Sc are not sufficient for improving the both hardness or wear resistance properties. However, 0.6 Sc additions certainly exhibited better wear resistance when compared to all other alloys. The presence of Al_3Sc precipitates are responsible for this better wear resistance property since these precipitates are able to absorb the load and load transformation to the matrix gets minimized. Thus this alloy exhibited better wear resistance. In addition, the TiN particles also play significant role in change the wear performance of MMCs. Sample I experienced a wear loss of 25 μm which is 3 times lower than the sample D (Fig. 6. A), whereas the sample F experienced a wear loss of ~32 μm which are very close to sample I wear loss (Fig. 6. B). These results are compared to each other since the TiN concentration is same and only the sc content is changed.

4. Conclusions

Al-4Cu-TiN (10, 20, 30 wt%) MMCs with and without Sc were successfully sintered (pressure less) at 620 °C for 20 minutes in the controlled (Ar) atmosphere furnace. Microstructural results suggest that TiN particulates are uniformly distributed in the Al matrix and no debonding is observed between the particulates and matrix. Increase in the TiN content is not only responsible for improvement in hardness values but also exhibited superior wear resistance properties. In addition, higher Sc content (0.6 wt%) is responsible for further improvement in mechanical and tribological properties. Even though the coefficient of friction and temperature changes due to additions of Sc to Al-TiN MMCs and varying load have been made, it is not discussed here.

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A szkandium adalékolás hatása Al-TiN fémmátrixú társított anyagok nyomásmentes szinterelésére

Al-TiN (10, 20, 30 m/m%) társított anyagokat állítottunk elő nyomásmentes szintereléssel Sc adalékolás nélkül, illetve 0,2 és 0,6 m/m% Sc adalékolásával. A jobb nedvesíthetőség érdekében valamennyi mintába 4 m/m% Cu-t is bekevertünk. A kísérletek során az Al, Cu és TiN porokat alaposan összekevertük, majd a porelegyből 12 mm átmérőjű, 4 mm vastag pasztillákat készítettünk. Az így kapott mintákat csőkemencében, argon áramban 450, 550 és 620 °C-on szintereltük. Azt tapasztaltuk, hogy a nagyobb TiN tartalmú, illetve a 620 °C-on szinterelt minták keménysége jobb volt a többi mintáénál. Az alacsonyabb hőmérsékleteken szinterelt pasztillák kis keménysége nem teljes zsugorodásra utal. A Sc-mal adalékolt mintákat ezért 620 °C-on szintereltük. A 0,2 m/m% Sc-t tartalmazó mintáknál sem a mikroszerkezet, sem a keménység nem javult. Ugyanakkor a 0,6 m/m% Sc-t tartalmazó mintáknál a keménység jelentősen megnövekedett. Ezek a minták nagyon finom szemcseszerkezetűek, és bennük erős felületi kötések alakultak ki az Al mátrix és a TiN részecskék között. A legnagyobb keménységet és a legjobb mikroszerkezetet az Al-0,6Sc-30TiN mintánál tapasztaltuk. A társított rendszerek kopási viselkedésének vizsgálata során megállapítottuk, hogy a Sc adalékolás nemcsak a mikroszerkezetet, hanem a kopási tulajdonságokat is javítja.

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