ISSN: 2277-3754
ISO 9001:2008 Certified
International Journal of Engineering and Innovative Technology (IJEIT)
Volume 8, Issue 7, January 2019

Investigation of lower weight % addition of B_4C as reinforcement on the wear response of magnesium based MMC using indentation scratch test technique

Shishir Keerti[1], Aditya Gokhale[1], Jayant Jain[1] , S. K. Sinha[2], Gururaj Parande[3], Manoj Gupta[3]
[1] Materials Characterization Laboratory, Department of Materials Science and Engineering, Indian Institute of Technology, Delhi, 110016, India
[2] Department of Mechanical Engineering, Indian Institute of Technology, Delhi, 110016, India
[3] Department of Mechanical Engineering, National University of Singapore, Singapore

Abstract: ***Micro scale wear response of magnesium matrix composite with boron carbide as reinforcement was investigated in this study. Effect of leaner addition of B_4C on the wear behaviour of the material was studied for 0.5 and 1.5wt. % nano-sized reinforced composites. Further, effects of load and multiple pass scratching on the wear rates of the composites was found out using indentation scratch test technique. Unlike the reinforcement content, grain size seems to play a dominant role in explaining the wear rates observed. Scanning electron microscopy and optical profilometry were used for quantification of the wear and wear mechanisms involved for each test condition***
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Keywords – **Scratch, composite, hardness, twins, slip.**

I. INTRODUCTION

Magnesium composites are gaining interest in various industrial applications including aerospace and automobile industry [1]due to their desirable mechanical properties. Magnesium is lighter than aluminium and steel making it a suitable candidate for aerospace and automobile applications [1].Its high specific strength along with good damping and machinability [2] makes them a better option for manufacturing purpose. However, the use of magnesium is not yet significant due its limited number of slip systems at room temperature leading to poor ductility, high anisotropy and poor wear resistance [3]. Metal matrix composites (MMCs) are showing a promising future in automobile, space research and many more industrial sectors. Researchers have shown promising results in improving the strength and wear characteristics of magnesium based composites by adding SiC, TiC particulates and ZnO [4-7]. Boron carbide (B_4C) is a ceramic with very high hardness and low density.

Manuscript received: 25 December 2018
Manuscript received in revised form: 20 January 2019
Manuscript accepted: 05 February 2019
Manuscript Available online: 10 February 2019

Using it as reinforcement in MMCs for better mechanical properties has been tried by several researchers [8-11]. Jiang and co-workers [9] has shown a significant improvement in hardness of the composites with B_4C weight compositions of 10 and 20%. They further investigated the wear response of the materials using pin on disc set up for dry sliding at 5 and 35N normal loads for a constant speed and found a significant decrease in the wear rates of composites than pure magnesium. The wear rate reduced to half for 10wt.% composition and one third for 20wt.% composition. Same trend was followed for 35N load. Guleryuz and co-workers [10] investigated the improvement in hardness and compressive strength of 3, 6 and 9wt. % of B_4C and found significant rise in hardness. Researchers have shown promising improvement in mechanical and wear properties of material with high B_4C wt.% but not much has been investigated at leaner addition and micro level wear response. To investigate the micro mechanics and nano-characterization of the materials, scratch testing shows reliable outcomes. Studies dealing with the nano-tribological behavior of magnesium composites are limited. Nautiyal and co-workers [11] studied the effect of microstructure on the wear response of AZ80 alloys using nano-scratch test technique. They found significant improvement in wear resistance of aged alloys than the solution treated. Aged alloys showed less twinning than the solution treated alloys along with cracking on the scratch surface. Cracks were also noticed in our previous study[12] on the solution treated Mg-xZn for single pass scratch on Mg-6Zn alloy and suppressed twinning with increasing zinc concentration.

Present study focuses on the effects of small weight percent addition of nano-sized boron carbide particulates on the nanotribological behavior of the composites. Effect of load and multi pass scratches on the wear mechanics of the material is also investigated in this study.

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II. EXPERIMENTAL PROCEDURES

A. Initial material

Samples used for this study are extruded rods of pure magnesium and it's composite with two different wt. %(0.5 and 1.5) composition of boron carbide. A disintegrated melt deposition technique was used to fabricate these composites. The detail of fabrication process is given elsewhere [13]. To further validate the result obtained from these samples, more composites with different wt. % (0.5,3 and 5) were prepared using bottom pour type vacuum stir casting technique. For this, 99.9% pure nano-boron carbide powder (particle size =500nm) was used along with 99.9% commercially pure magnesium. Pure magnesium was melted in the furnace at 600°C, nano powder of boron carbide was pre heated at 400°C for 1hr in another furnace. Nano powder was then added into the melt and stirred simultaneously with vertical movement of impeller at 400rpm for 10 minutes to ensure homogeneity of the reinforcement in the matrix. The melt was poured in a flat die of dimensions 15 mm × 100 mm × 300 mm.

B. Sample preparation

All samples were heat treated at 300°C for 10 hours before testing to remove residual stress and to homogenize the microstructure. For microstructure and scratch study, samples were polished with SiC papers upto 4000 grit size and then cloth polished in colloidal silica solution until mirror finished was obtained. For etching, 10% ortho-phosphoric acid solution in ethanol was used. Etching time was 10-15 seconds. Microstructures were viewed under optical microscope. Linear intercept method was used to determine grain size.

C. Micro hardness test

Micro hardness tests were conducted on HMV-2 Shimadzu micro hardness tester. Vickers diamond tip indenter was used for hardness measurement at a normal load of 1.9 N with 10 s holding time. Tests were conducted 10 times at random location on each sample and then the average value was reported.

D. Scratch Tests

Scratch tests were carried out on samples using ASMEC Universal Nanomechanical Tester (Bautzner Landstra ße 45, Germany). Two load conditions were used to make the scratches i.e. 100mN and 500mN.Indenter used for these tests was a diamond cono-spherical tip with a 10 µm tip radius. Scratches of length 200 µm, at a constant scratch speed of 5µm /s, were made for single and multiple passes.

E. Characterization of Scratches

To measure the cross-sectional area of the scratch, MACWIN Zeta-20 optical tomography microscope was used. Three scratches were made at each load for single, triple and five passes. Cross section area was measured at three different locations for each scratch giving 9 cross section areas for each test condition. The area was then multiplied with the scratch length to obtain the wear volume. To get the wear rate per pass, wear volume was divided by the total scratch length i.e. 200µm for single, 600µm for triple and 1000µm for five pass scratches. The scratches were examined with the Hitachi back-scattered electrons-based tabletop scanning electron microscope TM3000 (Tokyo, Japan) to understand the wear mechanisms involved in each test condition.

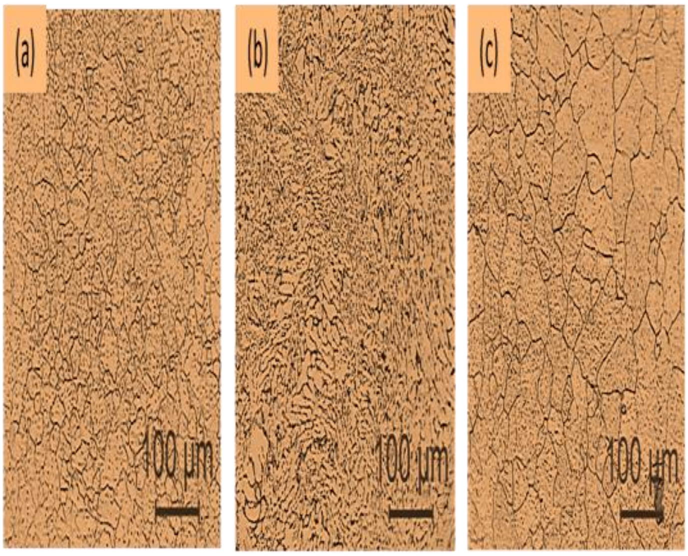

Fig 1. Optical micrographs of the samples after heat treatment at 300°C for 10 hours (a) pure Mg (b) Mg-0.5B_4C (c) Mg-1.5B_4C.

III. RESULTS AND DISCUSSION

A. Microstructure

Figure 1 shows the initial microstructure of samples after 10 hours of heat treatment at 300°C. The average grain size of pure magnesium, Mg-0.5B_4C and Mg-1.5B_4C are found to be 20µm, 9 µm and 24 µm, respectively. It should be noted that the presence of B_4C cannot be conclusively indicated in these micrographs. This might be due to leaner additions and small size particles as reinforcement.

B. Micro hardness

An increase in hardness with the increase in boron carbide addition is reported in various studies [13, 14], but limited study is available dealing with the leaner concentration of boron carbide. Sankaranarayanan and co-workers [14] showed in their study, the texture modification due to boron carbide addition enhancing the hardness and other mechanical properties of the magnesium based composite. In the present study, a decrease in hardness was observed with increasing wt. % of boron carbide. To further validate this claim, composites with different boron carbide additions

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(0.5,3 and 5wt. %) within the range of the previous samples were prepared and micro hardness tests were again conducted. Figure 2 shows the variation of micro hardness with B4C addition. From the hardness graph, it is clear that for leaner additions, the hardness decreases initially and then increases when the wt. % is increased from 1.5 to 3%. The hardness value is found to be maximum at 5%.

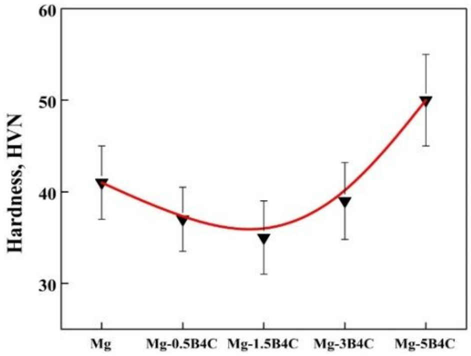

Fig 2. Micro hardness for composites with different weight % of boron carbide.

C. Single/Multipass Scratch Test

1. Effect of single and multiple pass scratch at constant load on the wear response of the composites.

Figure 3(a) shows the wear response of the samples at 100mN load for single, three and five pass scratches. It is clear that the wear rate doesn’t correspond well with the hardness measurements presented above. The hardest among all the samples is the pure magnesium, but wear resistance of Mg-0.5B_4C is very less as compared to magnesium as well as Mg-1.5B_4C composite. It is noteworthy that grain size of Mg-0.5B_4C sample is minimum as compared to other two cases. For Mg-1.5B_4C, the grain size is highest and so is the wear rate. It is quite evident that effect of grain size on the wear response of material is playing a bigger role in restricting the wear. Same trend was found for repeated scratching as well. Though, the difference in wear rates for repeated scratching is not much but sudden drop in wear rates of Mg and Mg-1.5B_4C is clearly visible. For repeated scratching, material is subjected to repeat loading which results in strain hardening.

From the wear rates, it can be concluded that Mg-0.5B_4C shows the same wear behavior, for repeated scratches, as that for pure Mg (Figure 3b). The wear resistance for Mg-0.5B_4C for single pass is dramatic as when compared to pure Mg. However, at higher load the effect starts to diminsh and this can be attributed to the length scale effect(Figure3(b)). For higher load, the change in wear rates for single and triple scratches makes it clear that Mg-0.5B_4C shows prominent strain hardening which was not in case with lower load.

With increasing number of scratch passes, the wear rate starts to decrease at all load conditions. Wear rate for single scratch is highest for every test condition and decrease in wear rate fromsingle to triple scratch is very much as compared to triple and five pass scratches. Due to repeated scratching, the material starts to show increased hardness as the result of strain hardening.Depth of penetration in depends on the hardness of material and lower scratch depth with number of scratch passes lower down the wear rate. Figure 4 shows the effect of number of scratch passes on the scratch depths of the materials for different load conditions.

S. Mezlini and co-workers [15] showed the effect of grain size on wear rates of aluminium alloys using a conical indenter with tip radius 10μm. They found that for finer grain alloy, the depth of penetration was lesser than that of coarser grain material. The depth of penetration remains lesser for finer grain alloy which lets indenter to come into contact with the superficial layer of the grains only and subsequently decreasing the wear rate even though the hardness values of the alloys are more or less the same.

D. Wear mechanisms : SEM observation

1. Effect of load on wear mechanism for Single scratch

To examine wear mechanisms at every load condition, SEM images of scratches were taken. Figure 5(a,b,e,f) shows the scratch grooves of single pass scratches at100mN load. As the wt. % of B4C has increased, the size of chip has decreased and grains have started to show intergranular rupture. Mg-1.5B4C (Fig5.e) shows more grain rupturing than the other two cases with small amount of twinning and chipping. Piling up of the material is more for composites and length of chip for composites is much smaller than the pure magnesium which clearly shows that ploughing of material is dominant for composites and micro-cutting is the mechanism formagnesium. Though no drastic effect on twinning is visible, all materials show small amount of twinning at lower load. Figure 5(c,d,g,h) shows SEM images of samples at 500mN load for single scratch. Difference in wear mechanisms between the composites and pure magnesium is clearly visible. Twins are visible at the vicinity of the scratch for pure magnesium along with small chips, broken chip debris and wear debris at the end of scratch shows ductile ploughing to be dominant for magnesium. For composites, very small amount of pile up can be seen for both the compositions; interestingly the length of chip at the end of scratch is very large as compared to pure magnesium which clearly shows micro cutting to be the dominant phenomenon of wear in composites.

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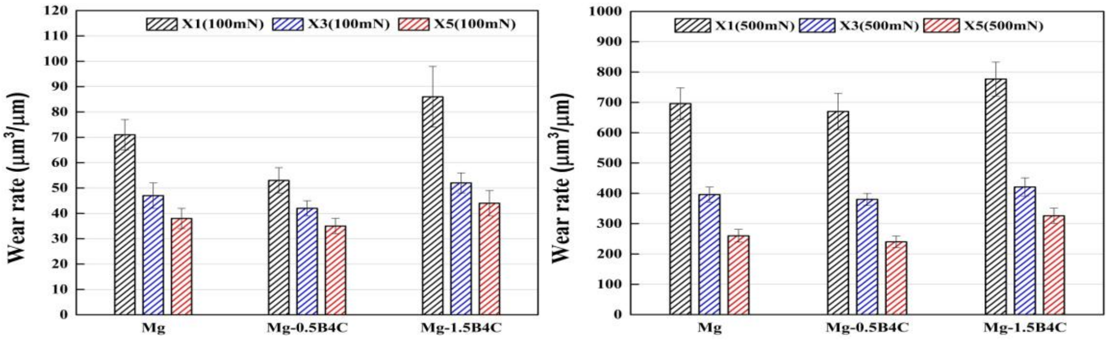
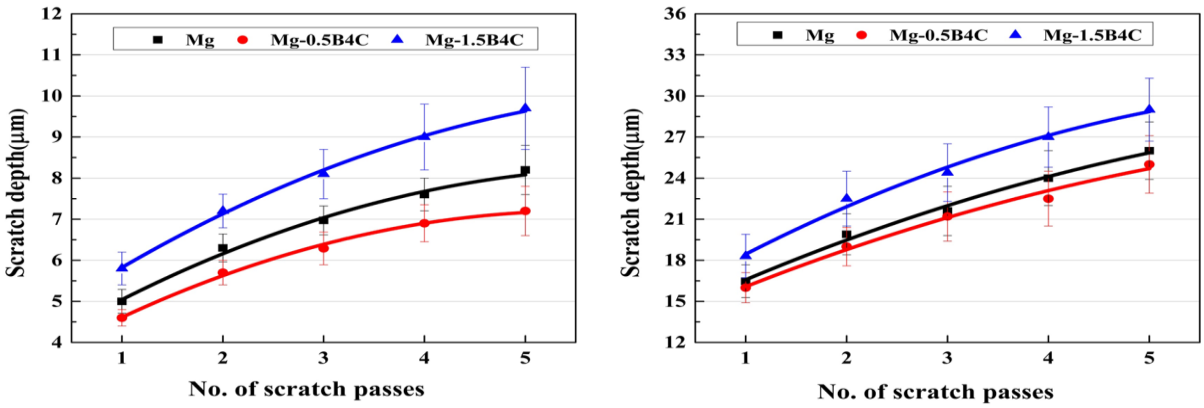


(a) **(b)**

Fig.3. Effect of B_4C addition and multiple pass scratches on the wear rate of composites at (a)100mN and(b) 500mN loads.



(a) (b)

Fig 4. Variation of scratch depths for pure Mg and its composites with the number of scratchesat (a) 100mN load and (b) 500mN load.

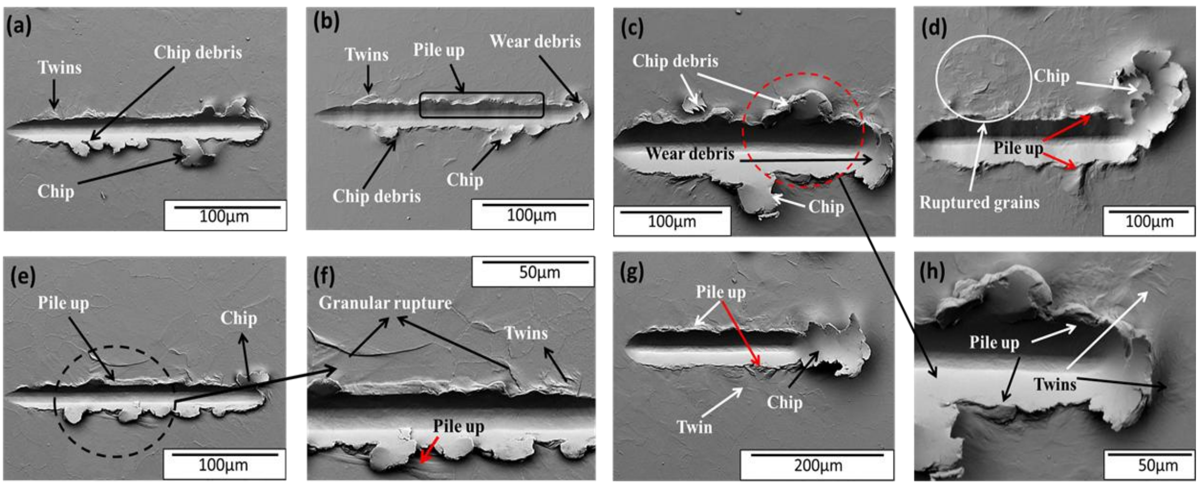


Fig 5. SEM images of samples after single pass scratch at 100mN and 500mN loads(a) Mg,100mN, (b) Mg-0.5B_4C,100mN,(c) Mg,500mN, (d) Mg-0.5B_4C,500mN,(e) Mg-1.5B_4C,100mN, (f) Enlarged view of the circled region in 5(e) to show ruptured grains and twins, (g) Mg-1.5B_4C,500mN and (h) enlarged view of the circled region in 5(c)to show twins and pile up.

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Twinning also seems to be suppressed for composites, same was reported byG. Garcés and co-workers [16], where the presence of the ceramic reinforcement hindered both twin nucleation and growth. An enlarged view of scratch groove for magnesium shows high twinning at the neighboring grains of scratched grains but, only small twinning can be seen for composites along with ruptured grains which have failed under compression load induced by the indenter.

2. Triple pass scratch

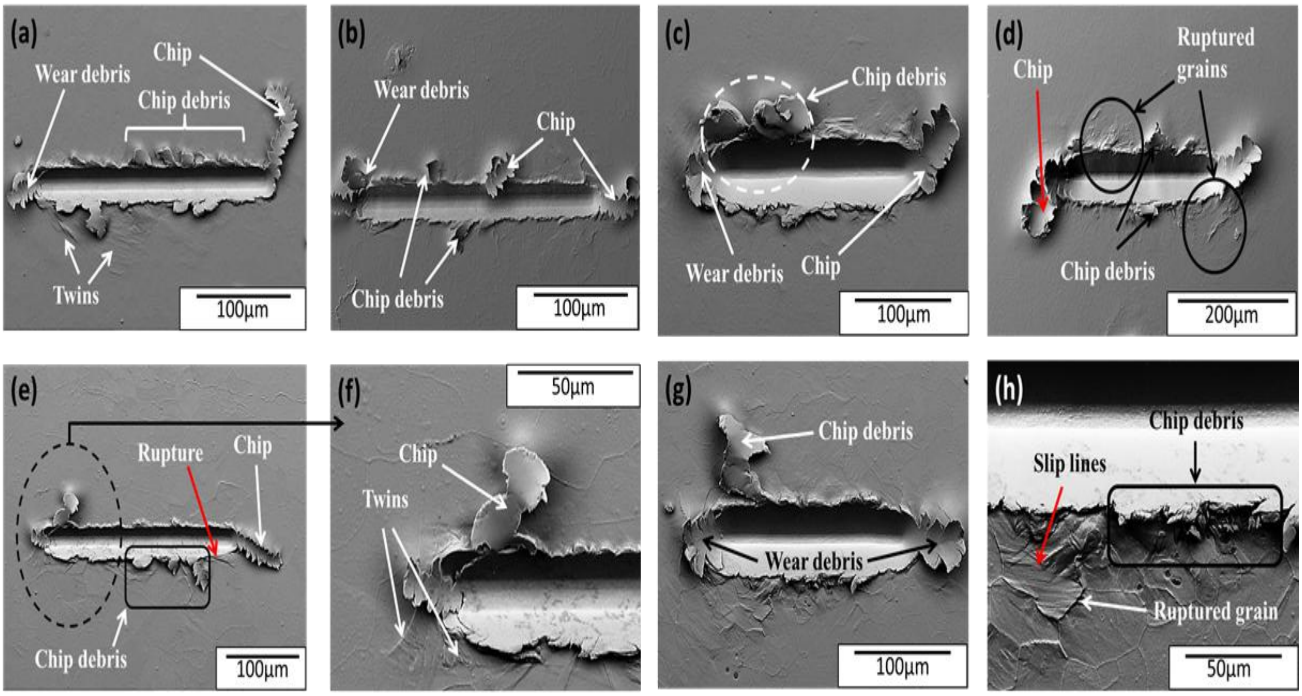

Fig 6.SEM images of samples after triple pass scratch at 100mN and 500mN loads(a) Mg,100mN, (b) Mg-0.5B$_4$C,100mN,(c) Mg,500mN, (d) Mg-0.5B$_4$C,500mN (e) Mg-1.5B$_4$C,100mN (f) enlarged view of the circled region in 6(e) to identify twins, (g) Mg-1.5B$_4$C,500mN and (h) enlarged view of the circled region in 6(g) to illustrate slip lines and ruptured grains.

Figure 6(a,b,e,f) shows the SEM images of triple pass scratches for 100mN load. Again, twinning seems to be supressed for composites as compared to pure magnesium. Small sized wear debris isseen in magnesium with large twinning and long chips. No traces of ploughing is visible for triple scratching. Though small traces of twins can be noticed for Mg-1.5B$_4$C composite but no significant twinning was observed for this sample. Longer chips were noticed for all samples without any piling up of the material showing micro-cutting to be the only wear phenomenon working as the number of passes increased. Microcutting leading to rupturing of grains as the composition increased is noticed from SEM observation.Figure 6(c,d,g,h) shows SEM of samples at 500mN for triple pass scratch. For higher load condition, composites show longer chips as compared to pure magnesium, transition from ploughing to micro-cutting with increase in reinforcement wt. % is clearly visible in SEM images. Another interesting observation is the tendency of grains to rupture as the reinforcement concentration increases. Mg-0.5B$_4$C shows ruptured grains under compressive load which seems to get more effective for Mg-1.5B$_4$C sample. Slip lines are also visible for Mg-1.5B$_4$C sample although no traces of twinning were found for composites. Figure6(h) shows slip lines and rupturing of grains at the vicinity of the scratch. No significant signs of pile up were noticed for both composites as compared to pure magnesium, not showing any evidence of micro-ploughing.

3. Five pass scratch

Fig 7(a,b,e,f) shows SEM images of samples at 100mN load for five pass scratches. Again for low load and multi pass scratching, microcutting seems to be the dominant phenomenon for material removal. Long chips at the end of scratches prove the same for all samples, chip debris were noticed mostly in pure magnesium with small amount of twinning. Chipping seems to decrease with increased reinforcement wt. % where ploughing can be noticed throughout the scratch length. Twinning again was supressed for composites but for Mg-1.5B$_4$C sample, slipmechanism was activated which certainly depends on the grain orientation. Transition from microcutting to ductile

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ISO 9001:2008 Certified
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ploughing seems to be the mechanism active as the number of passes were increased.

Fig7(c,d,g,h) shows SEM images for 500mN load for five pass scratches. Slip mode seems to be acive for all samples at this load condition. Slip lines were noticed in all samples, mostly in Mg-1.5B_4C sample. Piling up of material in pure magnesium was noticed along with chipping showing a transition from microcutting to ploughing. For composites, Mg-0.5B_4C sample showed no traces of pile up rather broken chip debris at the side of scratch was noticed along with the ruptured grains, the localized stress induced by the indenter tip has made the material to yield and ultimately break off while microcutting. Microcutting was also noticed for Mg-1.5B_4C sample as the chipping for this sample seems to be most as compared to other samples.

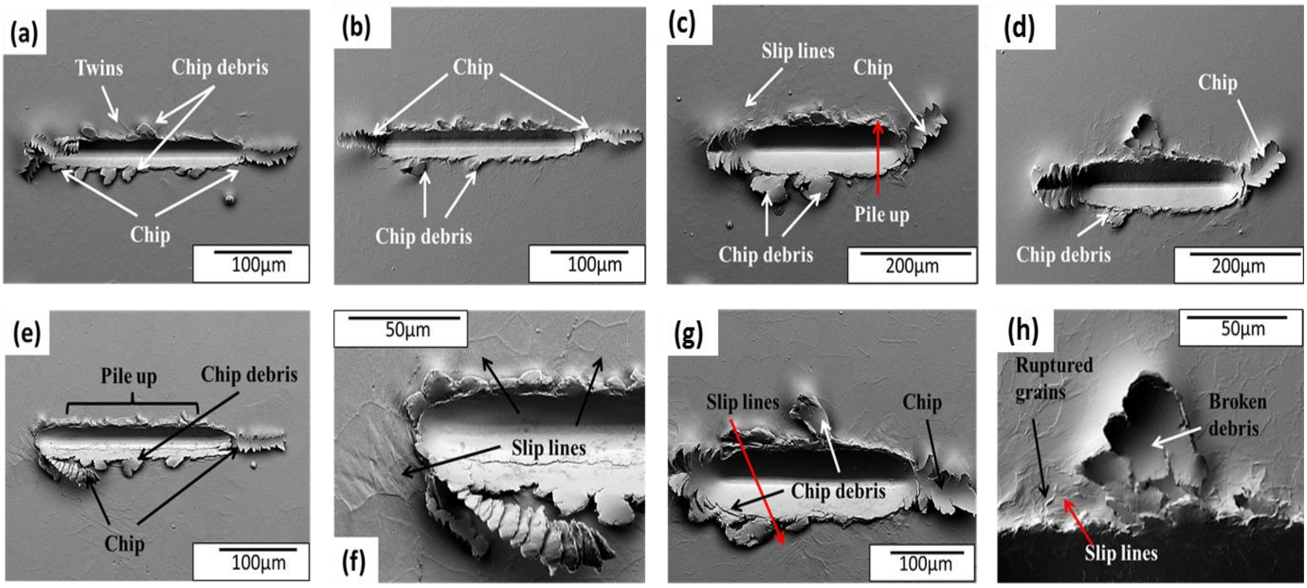

Fig 7. SEM images of samples after five pass scratch at 100mN and 500mN loads (a) Mg,100mN (b) Mg-0.5B_4C,100mN, (e) Mg-1.5B_4C,100mN, (f) Slip lines at the edge of scratch for Mg-1.5B_4C, (c) Mg,500mN, (d) Mg-0.5B_4C,500mN, (g) Mg-1.5B_4C,500mN and (h) Slip lines at the edge of scratch for Mg-0.5B_4C composite along with ruptured grains and broken debris.

IV. CONCLUSION

Effect of leaner addition of boron carbide as reinforcement on the hardness and wear response of the magnesium has been investigated using indentation scratch test technique for single and multiple pass scratch condition for different loads. Following are the conclusion that can be drawn from this study:

1. Boron carbide has a softening effect on magnesium matrix up to 1.5wt. %.
2. Effect of grain size plays the role in restricting the wear, even though the hardness value of Mg-0.5B_4C composite was lower than the pure magnesium.
3. Twinning was suppressed with boron carbide addition which was more noticeable at higher loads. Composites showed higher rupturing of grains at higher load condition.
4. Micro-cutting was the dominant phenomenon for composites along with the micro-ploughing at low load. Pure magnesium shows transition from micro-ploughing to micro-cutting as the number of scratches was increased.
5.

ACKNOWLEDGEMENT

The authors would like to thank Nanoscale Research Facility (NRF) of IIT Delhi.

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