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# Influence of Gr, $MoS_2$ and BN on the Hardness and Wear resistance of AA2014 Hybrid Composite after Artificial Aging

**Sachin K Murali, A. Shanmugasundaram**

***Abstract*: The main objective of this research work is to study the effect of tribological behaviors and mechanical properties of Aluminium alloy 2014 (AA2014) hybrid composite reinforced with Boron Nitride (BN), Molybdenum disulfide ($MoS_2$) and graphite (Gr). Stir casting technique is used for the fabrication of this composite material. The composite is subjected to three step ageing process which consists of solution heat treatment, quenching and aging. The base alloy was reinforced with 4 wt.% of BN, 4wt.% $MoS_2$ and 4, 6, 8 wt.% of Gr. The hardness of the composite steadily increased up to 6 wt. % of graphite and further increase in graphite decreases the hardness. The sample with Graphite of 4 wt. % resulted in maximum hardness of 192 HV with respect to AA2014 base hardness. Addition of graphite up to 6% resulted in lower wear rate when compared to the base metal. The wear rate of sample with Graphite of 4 wt. % is reduced by 49 % from that of the base metal at maximum wear condition. Scanning Electron Microscope (SEM), Energy-dispersive X-ray Spectroscopy (EDX) and X-Ray Diffraction (XRD) were used for the characterization of composite.***

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***Index Terms*: Stir Casting, AA2014, Boron Nitride, Graphite.**

## I. INTRODUCTION

AA2014 belongs to the category of wrought aluminium alloy and mostly used in aerospace and automotive sectors [1]. The blend of high strength, corrosion resistance and useful mechanical properties has made AA2014 appropriate for use as a base matrix material while fabricating composite. To enhance the properties of aluminium alloys, various ceramic and metal particles are added to the base matrix through liquid metallurgy route and aluminium metal matrix composite (AMMC) were fabricated [2]. AMMC are fabricated by modifying the bulk through many methods. Spray forming, powder metallurgy (PM), liquid metal infiltration, stir casting, compo casting, friction stir welding etc. are the techniques used to fabricate MMC. Out of all the bulk modification techniques stir casting is one of widely used one because of its simplicity and ease of control [3].

AA2014 reinforced with $Al_2O_3$ using stir casting technique and the results shows that yield strength, young's modulus, tensile strength and hardness is improved considerably when compared to the base metal [4]. SiC is reinforced with AA 2014 using stir casting technique then the molten metal is compressed through squeeze casting and AA 2014 + SiC AMMC is fabricated. The result reveals that addition of more particulate content tends to increase the porosity level and when the particle size is increased, porosity level decreases [5]. $TiB_2$ is produced using exothermic reaction during melting of AA2014. The resulting AA2014 +$TiB_2$ composite shows a significant wear resistance [6]. The effect of aluminium alloys with the addition of BN through PM technique is studied and the BN contributes better wear resistance [7].

Hybrid surface composite using friction stir processing with the combinations of BN and $TiB_2$ on the surface of AA 6082 is fabricated and compared the wear result with the AMMC of AA 6082 with BN and $TiB_2$ separately. When compared to the individual particle MMC, the hybrid composite shows a better wear resistance [8]. BN coatings are widely used in the aluminium casting industry due to its lubrication properties. Also BN has high temperature and thermal shock resistance [9]. Molybdenum Di-Sulphide ($MoS_2$) belongs to Transition Metal Dichalcogenides (TMD) family and is used as solid lubricants in vacuum and inert atmospheres [10]. $MoS_2$/Al composite is fabricated using stir casting process. The result shows that at 4% of $MoS_2$ showed better results in tensile and hardness properties comparing with the base metal [11]. A comparative study of Al-Si10Mg alloy and Al-So10Mg-$MoS_2$ composite through stir casting is attempted through stir casting. The addition of $MoS_2$ created a solid lubricating layer results 65% reduction in wear rate [12]. The effect of graphite on the mechanical properties of Al-4 wt% Mg alloy fabricated using compo casting method is studied. The result reveals that the increase in wt. % of Gr reduces the hardness [13]. Study on the mechanical and tribological properties of ZA 27 zinc-aluminium alloy reinforced with Gr and SiC is conducted. The combination of SiC and graphite increases the wear resistance, hardness and tensile strength [14]. Examination of abrasive wear resistance and microstructure characteristics of AA2014/SiC after age hardening is studied. Presence of coherent precipitates is observed after continuous aging process. Coherent precipitates having resistance to plastic deformation hence it provided low wear resistance [15]. Heat treatment process and age hardening process for improving fatigue behavior of AA 2014 is investigated. The samples were solution heat treated at 510°C and further aged for 7 hours at 190°C resulted in 43% increase in fatigue strength [16].

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SS316L reinforced with BN and $MoS_2$. Comparing the fabricated composites, SS316L/BN/$MoS_2$ hybrid composite provided superior improvement in properties [17]. AA 2219 is reinforced with $MoS_2$/B4C. The study reveals that addition of $MoS_2$ and B4C improves wear resistance property [18]. The literature study reveals that very little work has been done on the improvement of mechanical and tribological properties of AA2014 hybrid composites. The aim of this work is to investigate the effect of Gr, $MoS_2$ and BN on tensile strength, wear resistance and hardness of AA2014 hybrid AMMC.

## II EXPERIMENTAL PROCEDURE

Aluminium alloy 2014 is used as the base material. The average sizes of Gr, $MoS_2$ and BN particles reported by the particle analyser equipment are 279.8, 187 and 269.4 nm respectively. Optical Emission Spectrometer (METAVISION - 1008 I) is used to test the elemental chemical composition of the base material. The result is reported in **Table 1**

**Table 1 Elemental composition of AA2014**

| Element | Mg | Fe | Si | Mn | Ni | Cu | Ti | Al |
|---|---|---|---|---|---|---|---|---|
| Composition | 0.549 | 0.214 | 0.718 | 0.423 | 0.105 | 4.427 | 0.067 | Bal |

### A. *Fabrication of Hybrid Composite by Stir Casting:*

The AA2014-$MoS_2$-BN-Gr-MMC is fabricated using stir casting technique. AA2014 is heated above its liquidous temperature (800°C). Reinforcement particles are pre-heated to a temperature of 450°C and it was dispensed into the molten base matrix. Then stirring was done by using an automatic stirrer for 10 minutes at an average speed of 450 rpm. The molten metal along with reinforcement particles are then poured into a preheated cast iron permanent mould.

The stir casting process parameters are listed in **Table 2**. The reinforcement weight percentage for three variants is listed in **Table 3**.

**Table 2: Composition of AA2014 Aluminium MMC**

| S. No | Material | Sample 1 (wt%) | Sample 2 (wt%) | Sample 3 (wt%) |
|---|---|---|---|---|
| 1 | BN | 4 | 4 | 4 |
| 2 | $MoS_2$ | 4 | 4 | 4 |
| 3 | Gr | 4 | 6 | 8 |
| 4 | AA2014 | 88 | 86 | 84 |

**Table 3: Stir casting process parameters.**

| S. No | Parameters | Values | Units |
|---|---|---|---|
| 1 | Stirring temperature | 800 | $^0$C |
| 2 | Reinforcement preheat temperature | 450 | $^0$C |
| 3 | Mould Preheat temperature | 300 | $^0$C |
| 4 | Stirring Time | 10 | Mins |
| 5 | Stirring speed | 450 | Rpm |

### B. *Heat Treatment:*

Fabricated composite are subjected to three step ageing process after stir casting. In the first step the samples are solution heat treated for 2 hours at 510°C. After solution heat treatment process water quenching is done for about 15-20 minutes. The ageing temperature is maintained at 160 °C. Samples are cut into 12 pieces and kept at the heating furnace and after every two hour one sample is taken and measured for hardness. The optimum ageing time is arrived depends on the maximum hardness of the sample at particular hours.

### C. *Hardness Measurement:*

Mitutoyo microhardness tester (MVL-H11) is used to measure microhardness. ASTM E3-11 standard was followed for polishing the specimens. The specimens with dimensions 10 mm ×10 mm × 10 mm were used for the microhardness test. The specimens were then polished with 400, 600, 1000 and 2000 grit abrasive paper. Vickers microhardness tester with a diamond intender was used with a load of 100 gm force for 15 seconds for hardness measurement. Average microhardness was obtained from 10 readings taken at different points for each specimen.

### D. *Porosity:*

Porosity is considered as the measurement of void spaces in the material. Formation of porosity can affect the mechanical properties of a material. Archimedean method is used to find the actual density. Small pieces with sample size of 1.5 mm x 1.5 mm x 1.5 mm were machined and weighed using an electronic balance with an accuracy of 1 mg. For immersion test 10 ml graduated test tube was taken and partially filled with distilled water. Initial volume in the test tube is recorded. Then weighed sample was immersed in the liquid and the final volume is also recorded. The actual densities, theoretical densities and Porosity percentage were calculated using the following formulae.

**Theoretical Density** $$\delta_t = \frac{W_c}{\frac{W_m}{\delta_m} + \frac{W_{FA}}{\delta_{FA}} + \frac{W_{Si3N4}}{\delta_{Si3N4}}} \quad (1)$$

**Actual Density** $$\delta_c = \frac{M}{V} \quad (2)$$

$$Porosity\ percentage = \left(1 - \frac{\delta_c}{\delta_t}\right) * 100 \quad (3)$$

### E. *Tensile Test:*

The tensile test (UTS) of the Aluminium hybrid composite is carried out using Tinus Olsen machine (H25KT). The measurements were taken on the basis of ASTM E8M-04 standards.

### F. *Wear:*

Dry sliding wear test were carried out using Pin-on-Disc wear tester (Ducom : TR 20) attached with wear & friction monitor (TR - 2OLE - PHM 200). Tests were conducted according to ASTM standard.(ASTM, 2000) The specimens were mounted in the machine in stationary position. Wear track diameter is then set to 80 mm. The counterpart is a rotating disc with a diameter of 100 mm made up of EN-31 steel with hardness of 60 HRC. Cantilever mechanism in the equipment is used to apply load on the specimen.

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The disc and test specimens were cleaned and polished prior and after the test. The initial and final mass of each specimen was measured prior and after the test by an electronic weighing scale with an accuracy of 0.0001 g. Wear rate is calculated by mass loss method. After each run, the disk is cleaned using emery paper. The applied normal force is varied by 15, 25 and 35 N. The sliding speed is varied by 1,1.5 and 2 m/s. The sliding distance is fixed at 1000 m.

$$Wear\ rate = \frac{(Mass\ loss\ /density)}{Sliding\ distance} \quad (4)$$

Where, mass loss is in grams, density is in gm/mm$^3$, velocity is in m/sec, and sliding distance is in metre. Respective time and rpm value for each sample are then fed into the wear monitor and experiments were performed.

### *G. Characterization technique:*

The polishing of specimen was carried out according to standard metallographic procedure and etching was done with Keller's reagent (150 ml $H_2O$, 3 ml $HNO_3$ and 6 ml HF) [14]. The etched samples were subjected to microstructural analysis by using Carl Zeiss metallurgical microscope. To study the microstructure of hybrid composite SEM analysis was done. To confirm the presence of reinforcement particles line EDX and XRD were carried.

## III RESULTS AND DISCUSSION

### *A. Energy Dispersive X-Ray Analysis (EDX):*

Energy Dispersive X-ray Analysis (EDX) was used to confirm the elemental composition of the material. A line EDX is performed and found that the presence of Molybdenum, Sulphur, Boron, Nitrogen, Carbon which constitutes the compounds $MoS_2$, BN and Gr. The elemental composition analysis also shows that the major elements of AA 2014 such as Copper (Cu), Silicon (Si), Magnesium (Mg). These constituents of reinforcements and base alloy are depicted in the enlarged view of the line EDX shown in the Figure 1.

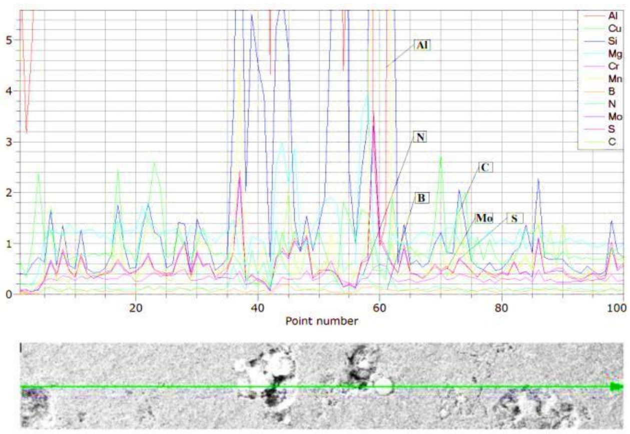



**Fig. 1 Line EDAX spectrum of AA2014 composite.**

### *B. X Ray Diffraction (XRD):*

XRD results of AA2014 composite is shown in the Figure 2. The diffraction peak of Boron Nitride, Carbon (Graphite), Aluminium, Manganese, Copper Silicon, Chromium, Molybdenum Nitride and Magnesium Boron can clearly be seen in the XRD spectrum. The reference code of each compound is listed below.

- Boron Nitride -01-080-5311
- Carbon -01-049-1720
- Aluminium Manganese -00-040-1150
- Copper Silicon -01-071-3786
- Chromium -98-015-0832
- Molybdenum Nitride -01-075-1150
- Magnesium Boron -01-089-1534

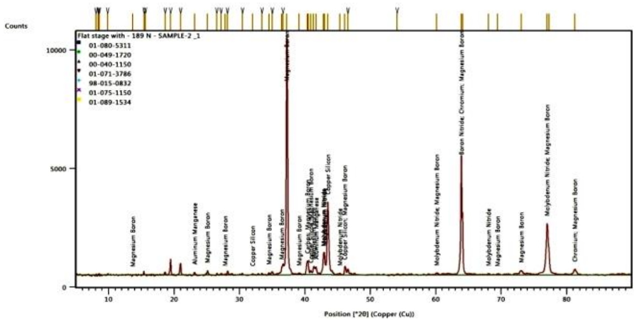



**Fig. 2 XRD spectrum of AA2014 composite.**

### *C. Microstructure Analysis using FE-SEM:*

FESEM image is used to observe the reinforcement particles used for the fabrication of hybrid composite. Figure 3 (a) and (b) shows the microstructure of hybrid matrix. FESEM image displays the graphite grains in black color, $MoS_2$ particle in irregular shape and BN particle in spherical shape. Figure 3 (b) displays the porosity in the hybrid matrix. Also the FESEM micrographs reveal that there is a strong tendency for reinforcement particles to embed in the base AA2014.

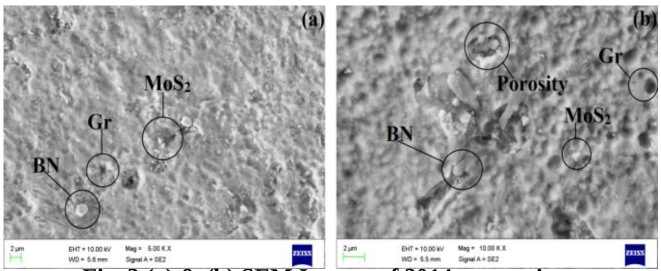
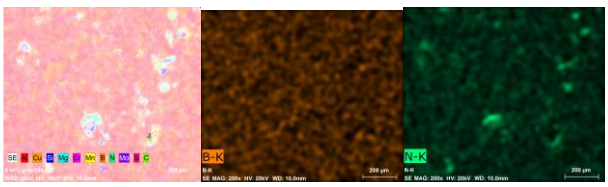



**Fig. 3 (a) & (b) SEM Images of 2014 composite.**

### *D. Colour Mapping:*

Colour Mapping is used for visual understanding of the presence and distribution of the elements in the Aluminium 2014 matrix and is shown in Fig. 4. The figure 4 shows the reinforcement elemental composition of BN, $MoS_2$ and Gr i.e. B, N, Mo, S and C.

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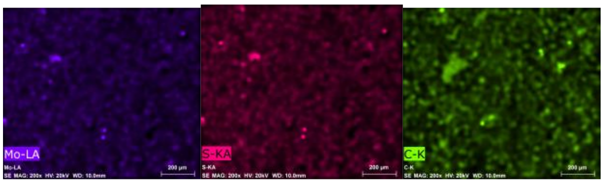


**Fig. 4 Colour Mapping Images of AA2014 composite.**

### *E. Hardness:*

The microhardness value of the base metal is 150 HV. Optimized ageing hours are arrived with respect to the highest microhardness of S1, S2 and S3. The maximum hardness of S1 is 192 HV at 6 hours, S2 is 157 HV at 16 hours and S3 is 126 HV at 12 hours of ageing. The S1 sample reported an increase of hardness by 28 % and S2 sample by 4 % when compared to base metal hardness. However S3 sample hardness is decreased by 16 % when compared to base metal hardness. With respect to sample S1 (Gr wt. % 4), S2(Gr wt. % 6) and S3 (Gr wt. % 8) the hardness result reveals that when the wt. % of graphite increasing then the hardness decreases. The hardness change in S1, S2 and S3 samples are presented in the Figure 5. Previous studies also reported that addition of graphite particles resulted in reduction of hardness [13].

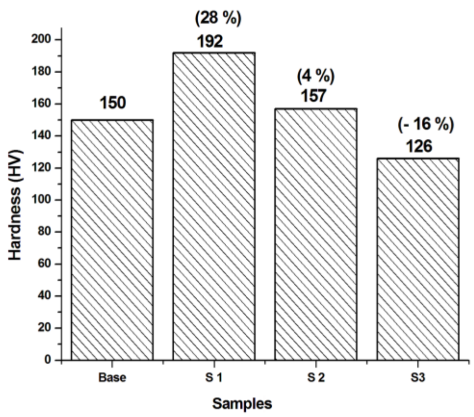


**Fig. 5 Hardness comparison between base and composite.**

### *F. Porosity:*

Porosity, actual density and theoretical density are shown in the Table 4. The porosity percentage shown in the table reveals that when the graphite percentage is increased from 4 % (S1) to 6 % (S2) the porosity level decreases very little. But when the graphite percentage is increase from 6 % (S2) to 8 % (S3) then the porosity level increases marginally. In general the porosity of the AA2014 AMMC is increased from the base metal value of 3.4 % to 22.26 % in the case of S3. The addition of graphite increase the porosity is already reported by the previous researcher [13].

**Table 4: Porosity and Density Values of base and composite**

| Material | Density (gm/cm$^3$) | Porosity (%) |
|---|---|---|
| AA2014 | 2.8 | 3.4 |
| 4 wt% Gr | 2.218 | 20.9 |
| 6 wt% Gr | 2.258 | 18.58 |
| 8 wt% Gr | 2.18 | 22.26 |

### *G. Tensile:*

The tensile strength of the base alloy and hybrid AA2014 are shown in the Figure 6. The tensile strength of S1, S2 and S3 samples are less than that of the base metal. In all the samples $MoS_2$ and BN are reinforced with 4 % as constant. The tensile strength of hybrid composite AA2219 + $MoS_2$/$B_4C$ decreased [18]. BN is reinforced with AA 6061 and found that the shear stress is high at the interface leading to interfacial de-bonding in AA 6061 + BN composite. It is observed that a cleavage is seen in the matrix due to lack of load transfer from the matrix to the particle [19]. The combined effect of self-lubrication property of $MoS_2$ & Gr, interfacial de-bonding of BN and high porosity of AA 2014/$MoS_2$/BN / Gr AMMC resulting in the reduction of tensile strength with respect to the base alloy of AA 2014.

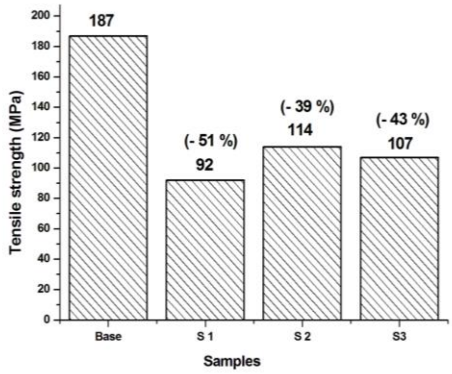


**Fig. 6 Comparison of UTS values for base and composites.**

### *H. Wear:*

Figure 7 shows the wear rate of the samples with respect to constant velocity and varying loads. Figure 8 shows the wear rate of the samples with respect to constant load and varying velocity. From the Fig. 7 it can be observed that the wear rate increases with respect to increase of load. Initially with the load of 15 N the asperities of hybrid composite contacted with the asperities of rotating disc. The hardness of rotating disc is much higher (60 HRC) than the hybrid composite so asperities of hybrid material is get ruptured and break into small particles and when the load increases all these broken particles act as an abrasive slurry and the hybrid material is worn out quickly. Similar results are already revealed by many researchers [20].

The wear rate of the base metal and composite samples according to the sliding velocity and the load are listed in the Table 5.

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**Table 5: Wear rate of Base, S1, S2 and S3**

| Sl. No. | Load (N) | Slid. Vel. m/s | Base | Wear rate (mm$^3$/m * 10$^{-3}$) V - I | Wear rate (mm$^3$/m * 10$^{-3}$) V - II | Wear rate (mm$^3$/m * 10$^{-3}$) V - III |
|---|---|---|---|---|---|---|
| 1 | 1 | 15 | 2.6606 | 1.2400 | 2.2978 | 3.0646 |
| 2 | 1.5 | 15 | 1.9266 | 0.9654 | 1.7021 | 2.1494 |
| 3 | 2 | 15 | 1.6100 | 0.5757 | 1.2765 | 1.7632 |
| 4 | 1 | 25 | 2.9541 | 2.1523 | 2.7659 | 3.4006 |
| 5 | 1.5 | 25 | 2.5688 | 1.1957 | 2.0340 | 2.6448 |
| 6 | 2 | 25 | 1.9724 | 0.8414 | 1.3617 | 2.4769 |
| 7 | 1 | 35 | 3.6238 | 2.4357 | 3.2340 | 3.7783 |
| 8 | 1.5 | 35 | 2.8440 | 1.6829 | 2.5277 | 3.1066 |
| 9 | 2 | 35 | 2.2477 | 1.1957 | 2.0851 | 2.8212 |

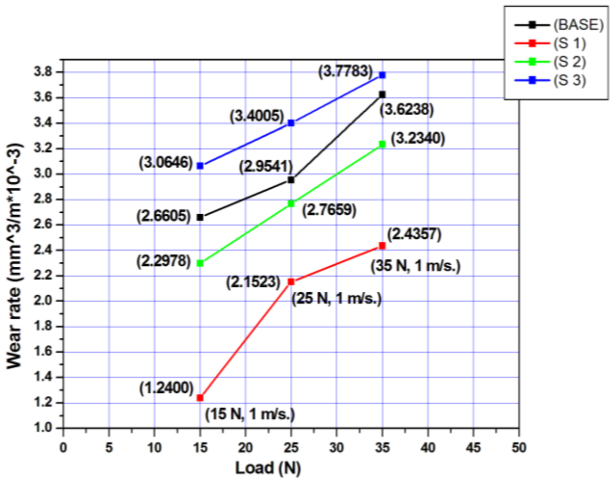


**Fig. 7 Effect of load on wear rate at constant sliding velocity of 1 m/s.**

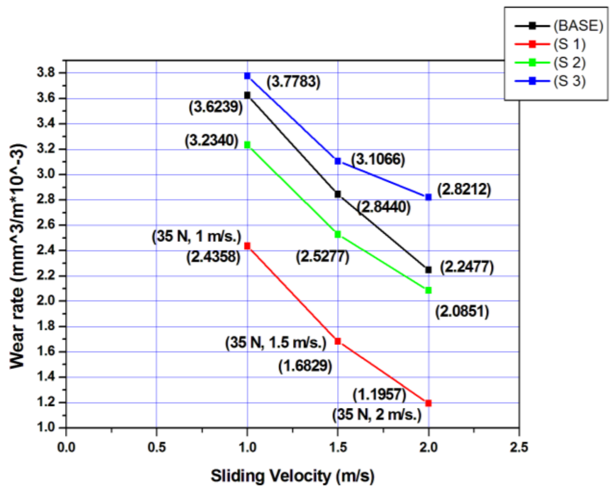


**Fig. 8 Effect of sliding velocity on wear rate at constant load of 35 N.**

From the Figure 8 it can be observed that the wear rate decreases with the increase of sliding velocity. The hybrid sample pin is getting contacted with the rotating disc and getting worn out with the maximum load of 35 N and with the sliding velocity of 1 m/s. The friction between the rotating disc and the hybrid sample pin increases and it leads to raise in temperature of the tip of the hybrid pin when the sliding velocity increases. Formation of an oxide layer occurs on the tip of the hybrid pin and it facilitates the pin to slide over the counterpart freely which in turn reduces the wear rate. This is a reason wear rate decreases when the sliding velocity increases with the same load. Similar results are reported by many researchers [21].Also from the Figures 8 and Figure 7 it can be concluded that the maximum wear takes place with the sliding velocity of 1 m/s and at the maximum load of 35 N. Out of 9 combinations of wear test parameters minimum wear takes place with the sliding velocity of 2 m/s and at the load of 15 N.

The S1 and S2 samples wear rate is less than that of AA2014. When the Gr wt.% is increased from 4 to 6 the wear resistance of the hybrid composite increases. The wear resistance of S3 sample is less than that of the base metal. The reason can be of very high porosity percentage of S3 samples. The S3 sample has 22.26 % of porosity, whereas the porosity of base metal is only 3.4 %. Literature study reveals that when there is lack of interfacial bonding between the reinforcements and the base aluminium then it leads to high amount of porosity and non-uniform dispersion of reinforcement matrix phase. This resulted in the reduction in mechanical and tribological properties. Stir casting process variable such as stirring time, speed, size and position of the impeller and holding time are also plays an important role in determining the mechanical and tribological properties of AMMC [22]. So, it can be concluded that optimization of stir casting process parameters will help in solving these kind of issues in the hybrid AMMC.In overall the S1 sample results in very less wear rate. The wear resistance of S1 sample is improved by 49 % from base metal at maximum wear condition. The wear resistance of S2 sample is increased by 12 % only from base metal at maximum wear condition.

## IV CONCLUSION:

In the present work, AA 2014-BN-$MoS_2$-Graphite composite is fabricated by using Stir casting technique. Solution heat treatment, water quenching and artificial aging were conducted after stir casting process. The effect of reinforcement particles on the hardness, tensile strength and wear resistance of AA2014 were studied and summarized as follows.

- The maximum hardness of 192 HV is obtained in S1 sample. The increase is about 28 % with respect to AA2014 base hardness.
- Hardness increased up to 6 wt. % of graphite and further it reduces.
- The tensile strength of all samples is found to be lower than the base metal.
- The reduction in tensile strength of fabricated composite samples is due to high porosity.
- Addition of graphite up to 6% resulted in lower wear rate when compared to the base metal.
- S1 sample wear rate was reduced by 49 % from that of the base metal at maximum wear condition.

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