



# Influence of quenching agents on mechanical, wear, and fracture characteristics of $\text{Al}_2\text{O}_3$ / $\text{MoS}_2$ reinforced Al-6061 hybrid Metal Matrix Composites (MMCs)

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**ABSTRACT.** Aluminium (Al) based composites enhance the mechanical and wear behavior by heat treatment. The quenching factors like cooling agent, cooling rate and temperature of cooling are expected to influence the hardness, tensile, and wear behavior of the Al MMCs (Metal Matrix Composites). This research shows the outcomes of a sequence of experiments conducted to study the mechanical and wear behavior of the Al6061-Al<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> hybrid composites that are quenched with different quenching agents. Heat treated hybrid composites were subjected to evaluate the hardness, tensile, and wear behavior. The outcomes reveal that the heat treatment significantly enhances the mechanical and wear behavior of hybrid composites. Highest hardness of 138.1 VHN was achieved under the ice quenched samples. The mechanical and wear behavior improved under the ice quenched samples. Voids, particle pullouts and dimples were observed in the tensile fractured surface. Micro pits, delamination layers, ploughing and micro fissures were observed in the wornout surface of the wear test samples.

**KEYWORDS.** Al-6061, Stir casting, Microstructural study, Mechanical behavior, Wear behavior, SEM analysis.



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## INTRODUCTION

Globl warming of our environment has attracted lot of attention since last 10 years. Ecological problem has drawn much attention towards manufacture of automotive and structural components. Demand for better strength in light-weight materials is still increasing year by year. A strong light weight material offers the benefit of energy savings in marine and automotive fields [1]. The most advanced light-weight materials are Al composites. Al composites are generally produced by stircasting method. Fabrication of composites by the stircasting method poses problems such as wettability of reinforcements. The low wettability leads to non-uniform dispersal of reinforcements.



Non uniform distribution of reinforcements can lead to reduced strength in material [2, 3]. One of the techniques used to enhance the material strength is the heat treatment process. Heat treatment process can increase the grain refinement and also improve bonding between reinforcement and the matrix. Al6061-Al<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> is an Al composite frequently used in automotive components. In automotive parts, the Al composites are usually heat treated under the T6 condition [4]. Heat-treatment is a sequence of heating and cooling to change mechanical and physical characteristics of the developed composite. In the heat treatment process, heating is done to a particular temperature followed by cooling in various cooling media [5, 6]. The heat-treatment of Al MMCs is generally done by T6 condition followed in the various stages: solutionizing, quenching and finally, artificial ageing [7]. Earlier studies [8] show that heat treatment causes significant effects on the properties of Al alloy. Outcomes show that the heat treatment plays a significant role in inter-metallic bonding effect among the reinforcements and base material. Al composites show significant enhancement of mechanical properties after heat treatment as compared to an as-cast condition [9]. Under rapid cooling conditions, magnesium compounds formation occurs in the grains, but under slower cooling, magnesium compounds are strongly formed at the grain boundaries. Generally, an effect in Mg precipitation takes time for convergence at grain boundaries [10]. Normally, this precipitation enhances the properties of a developed material, i.e. mainly, tensile and hardness. Prabhu et al. [11] evaluated the mechanical and wear properties of heat-treated Al composites. The mechanical properties of developed composite were enhanced with increasing SiC content. Heat treatments showed enhanced tensile strength and higher wear resistance of developed composites. Previous literature surveys indicated that heat-treatment with different quenching media affected the material characteristics of the composite [12]. The quenching rate should be high enough to achieve a better strength in the developed composite material. Previous research study of [13] showed that faster cooling created high residual stresses which initiated distortion and cracks. Quenching consistency is a process of quick cooling which leads to a reduction in the fracture and also residual stress [14, 15]. The cooling rates significantly influence the micro-structure and material properties of composites. The rate of cooling generally changes the precipitant leading to change in material properties of the alloy. However, on the further addition of reinforcements, the development of extreme precipitant leads to reduction in the mechanical properties of the developed composite materials. For Al "6" series, the cooling must be carried out at a slow rate, and then the developed material requires quenching at sensitive temperatures to avoid the precipitation being heterogeneous. When the temperature is less than 200°C, the rate of cooling is normally slowed, resulting in the best mechanical behaviour [16-18]. Meanwhile, the values of different quenching agents have been investigated in less number. The present research work is undertaken to evaluate the influence of different quenching agents on tensile, hardness, and wear behavior of Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub> reinforced Al6061 composites. The main purpose of the present research work is intended to prove the forecast that it is essential to identify the influence rates of cooling under different quenching media on Al6061-Al<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> hybrid MMCs to attain better mechanical properties and high wear resistance.

## MATERIALS, FABRICATION, AND HEAT TREATMENT PROCESS

**A**l6061 commercial grade material was used as a base material and two different reinforcing particulates such as Al<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> and wt. % of 3, 6, 9 were used for the preparation of hybrid composites. The Al<sub>2</sub>O<sub>3</sub> reinforcement is significantly improves the ultimate tensile strength, hardness and tribological behavior. MoS<sub>2</sub> is used to increase the machinability of composite materials. It acts as a self-lubricating material and improves the wear resistance of the aluminium metal matrix composites. In the present study, Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub> with 100 mesh size of particulates were used as reinforcements. A stircasting process technique was implemented for producing the hybrid metal matrix composites. The Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub> were pre-heated in a separate crucible till 450° C. The base material was melted using electric furnace and hard reinforcing Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub> pre-heated particulates were mixed into molten melt. The stirring process was maintained continuously at 250 rpm. The melt was poured continuously into the metallic mold which was pre-heated [19]. The casted composite samples were taken out from the mold box. Further, the tensile, hardness and wear test samples were machined according to ASTM standards. The hybrid metal matrix composites produced from the stircasting process need to be enhance the grain refinement. The grain refinement and mechanical and wear properties can be improved through heat treatment process. Heat-treatment process was selected on the basis of functions and the nature of material as defined by the kind of cooling agent [20]. Generally, the quenching plays a major role in the heat-treatment process. Developed hybrid composite samples were subjected to solutioning at 510°C of temperature for 2 hours and separately quenched. Here, two types of quenching agents such as ice cubes and water were used. After quenching, the same samples were subjected to age-hardening process at a constant temperature of 180°C for 4 hours and finally cooled at room temperature.



## RESULT AND DISCUSSION

### *Microstructure analysis*

**U**niform distributions of reinforcements show an improvement wear and mechanical behavior of developed composites [21]. Fig 1(a) depicts the micro-structure of the base alloy without any reinforcement content. Fig 1(b) shows the micro-structure of Al6061 reinforced with 9%  $\text{Al}_2\text{O}_3$  and 3%  $\text{MoS}_2$  with uniform distribution. The reinforced particulates in the developed MMCs are clearly resolute near the grain boundaries. It is observed that the particulates are free from the agglomeration which is generally due to stircasting technique used in manufacture of hybrid MMCs. The distribution of hard particulates in the matrix is a significant requirement for achieving improvement of the wear and mechanical behavior of the developed hybrid MMCs [22]. Generally, it is known that reinforcing of hard particulates in the Al base matrix causes increase in grain refinement [23]. Microstructure study reveals that the grains around the hard reinforcement particulates are much finer when compared to the grains around the reinforcement's free base alloy. So, hard particles can cause the recrystallization of an Al alloy by accelerating the particles nucleation between the reinforcements and base matrix phase. The metal matrix composites with heterogeneous structures have established the mechanical properties with high hardness, strength and plasticity. Some of the researchers have demonstrated the reinforcing agent of metal matrix composites with disperse structures with fine and coarse grain structure. The fine structure of the composites and the interactions with the matrix material develop the better bonding when compared to the coarse grain structure. And also sharp increase in hardness and tensile strength can be observed. Similar results were observed by other researchers [24, 25] and it is concluded that, the Al grain generally solidifies near the hard particulates which leads to compromising of the resistance to grain growth.

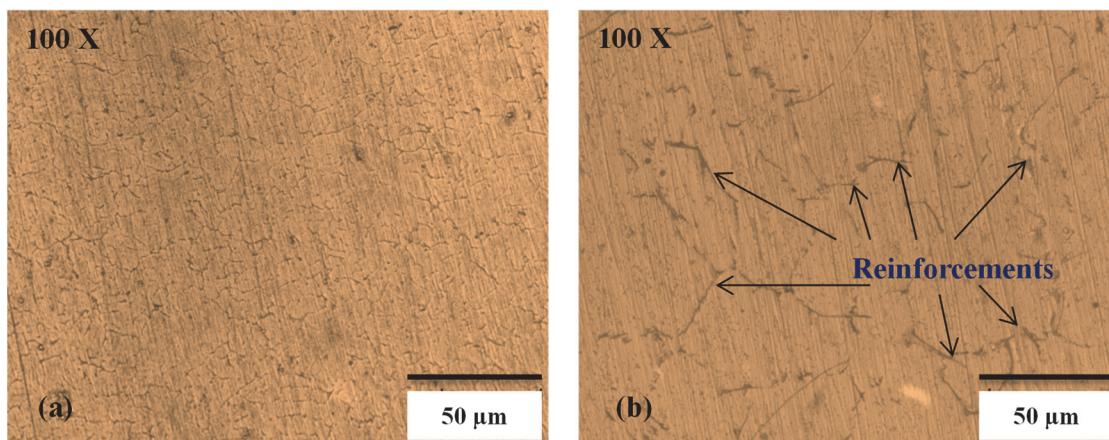


Figure 1: Microstructure of (a) Al6061 (b) Al6061 + 9%  $\text{Al}_2\text{O}_3$  + 3%  $\text{MoS}_2$  with uniform distribution.

### *Hardness*

Assessment of hardness was performed by using the sample size of 20 mm diameter \* 20 mm thickness based on ASTM standards. By adopting the Vicker's micro hardness testing equipment, using ball indenter (10 mm) at the constant load of 0.5 kg for 30 seconds of duration the hardness was measured. Hardness of developed composites was studied at 3 different zones on the test specimen's surface for the determination of average values of the composite hardness. The hardness of developed hybrid composites is depicted in Fig. 2. Addition of hard particulates ( $\text{Al}_2\text{O}_3$ ) to the Al matrix enhanced the hardness of composites [26]. In Fig. 2, higher hardness values are observed due to increasing of  $\text{Al}_2\text{O}_3$  content which proves that the adding of  $\text{Al}_2\text{O}_3$  particulates effects to improvement in the hardness value of the developed hybrid composites. In composites, hardness values of the test samples were significantly influenced by the homogeneous dispersal of hard particulates in the base matrix. Similar results have been observed by other researchers [27] and it was concluded that the enhancement in hardness values could be due to the presence of reinforced particulates, which makes the movement of dislocation more difficult within the base matrix. Further, the hardness of developed hybrid composites gradually decreased with increase in the  $\text{MoS}_2$  content. The reduction in the hardness by increasing in  $\text{MoS}_2$  particulates is because of high lubrication characteristics of the  $\text{MoS}_2$  particles that cause movement of grains [28]. From Fig. 2, it can be found that the hardness values increased due to the heat treatment process. The improvement in hardness after the heat-treatment is because of the development of hard phase caused by precipitation age hardening. Composite samples quenched in ice cubes show improved hardness when compared with water-quenched samples and samples cooled at

room temperature. The treatment of solutionizing indicates the development of intermetallic phases which leads to higher hardness.

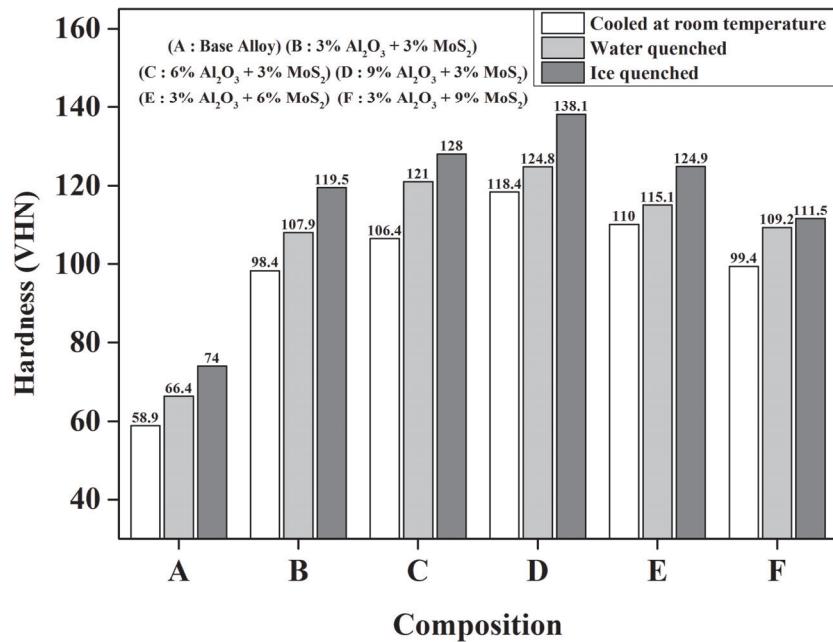


Figure 2: Vickers micro hardness with varying content of Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>.

In heat treated MMCs, thermal-mismatching of the base material and reinforcement leads in density enhancements in the dislocation. This eventually leads to progressive resistance to plastic deformation generally which results in improvement of hardness. The ice quenched composites exhibit an improved hardness due to the enhanced bonding among the base matrix and reinforcements. The improvement in hardness is also due to the stabilization of the inter-metallic phases within the base material. High cooling rates produced distortion which led to the hardness of hybrid composites. This phenomenon affects distortion, generally which will be formed by dislocation of slip and has a high influence on hardness of developed hybrid MMCs [4]. Other researchers [29] concluded that the heat-treatment did not drastically change the morphology, whereas hardening of the base matrix due to precipitation of hardening took place resulting in enhanced hardness in the composites.

#### Tensile Strength

Tests were performed on an Electronic Tensometer whose maximum load capacity is of 20 Kilonewtons. Tensile test specimens were prepared as per the ASTM E8 standards with gauge length of 16 mm and 4 mm of gauge diameter. Fig. 3 depicts the graphical representations of tensile strength for developed hybrid MMCs. The outcome shows, noticeably, that the tensile strength of the hybrid composites is enhanced by enhancement of Al<sub>2</sub>O<sub>3</sub>. This is generally, because of the presence of hard particles in developed composites [30]. Also it is seen that, the tensile strength of developed hybrid composites reinforced by hard ceramic particulates increases due to resistance of dislocation. Generally, the nature of hard particles is the main reason for improvement of material strength [31]. The hard particulate correlates with the dislocation, resulting in enhancement of strength in developed composites. Similar results have been witnessed by other researchers [19]. Further, the tensile strength was reduced by increasing the MoS<sub>2</sub> content and a similar outcome was found by Siddesh Kumar et al. [32]. Due to the presence of MoS<sub>2</sub> particulates there may be possibility of crack propagation and also particulates pull out, which may lead to the decrease in tensile strength in the developed hybrid composites. This can also form due to the solid lubricant particulates that do not carry any load effectively. It is found that there is a possibility in improvement of coherent precipitates due to heat-treatment process. Lattice coherence between precipitates and the host matrix occurs up to certain temperature, after which lattice vibrations generate incoherent precipitates in the host matrix. It is well known that ageing treatment causes development of fine precipitates on soft matrix (Al) which leads to improvement in the characteristics of composite. The improvement in the ductility of developed MMCs is because of the effect of a several small hard particles and thermal modification during heat treatment process. In the Fig. 3, high strength is observed for the developed hybrid MMCs when it was quenched in the ice cubes. This remarkable improvement in



strength of the developed hybrid composites subjected to the heat-treatment can be attributed to development of intermetallic precipitates, which, usually, act like obstacles for pinning down of the dislocation. This phenomenon bounds the mobility of the dislocation, reducing the level of plastic deformations and thus, results in major enhancement in tensile strength of the developed hybrid MMCs [33].

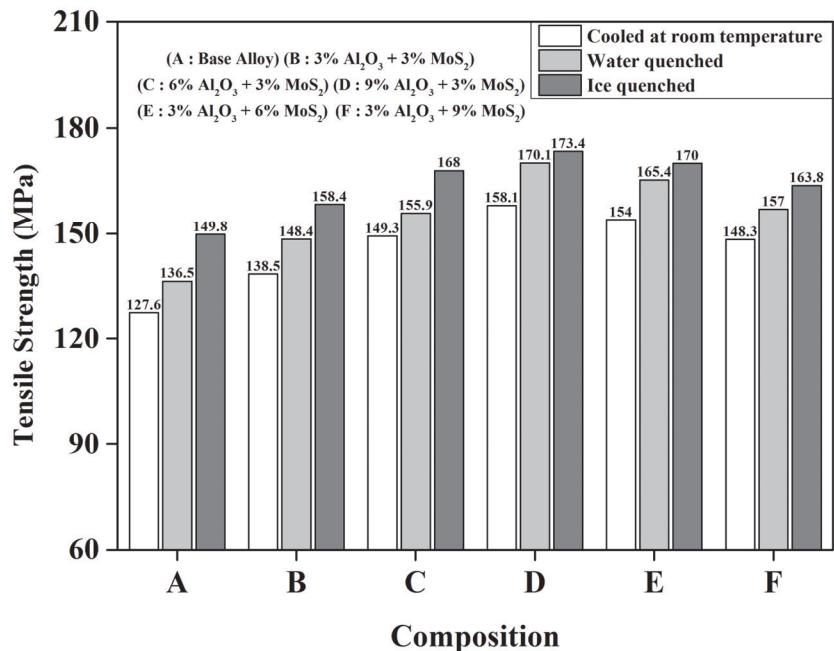


Figure 3: Tensile strength with varying content of  $\text{Al}_2\text{O}_3$  and  $\text{MoS}_2$ .

The tensile stress-strain curves of the composite samples fabricated by the stir casting method are shown in Fig. 4. Out of all the developed composite specimens, the tensile strength is higher for ice quenched specimen. So, a stress-strain curve is plotted for ice quenched samples and all the points are indicated. The stress-strain curve indicates the improved toughness apart from high tensile strength. This is significant. Meanwhile, most strength improvement methods cause decreasing ductility.

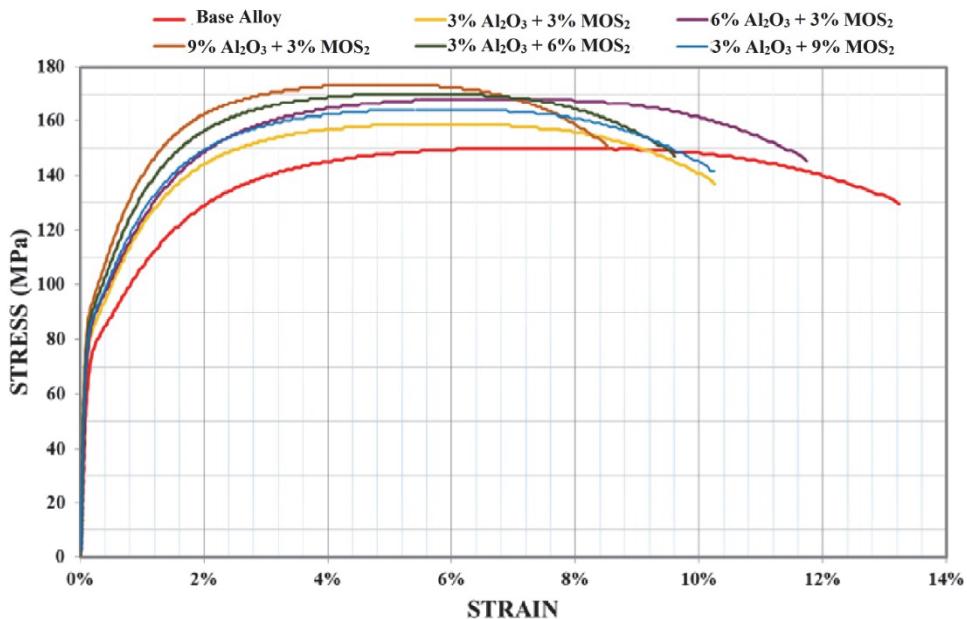


Figure 4: Stress-strain curve for ice quenched samples.

After the evaluation of tensile strength, surface fracture tests were carried out to study the fracture behavior and relationship of the interface among the reinforcements and the base matrix. The SEM image of the fractured surface of heat treated samples was captured at a uniform magnification. This study shows the analysis of microstructural effect on tensile properties of hybrid composites. In case of hybrid composites, it is always brittle in nature. Subsequent growth of micro voids causes a dimple rupture which is related to the progression of fracture. Since the ceramic particles are present as a reinforcing material, the fracture process changes markedly. The fractography of all three different samples ((a) sample cooled at room temperature, (b) sample quenched in water and (c) sample quenched in ice cubes) are depicted in the Fig. 5.

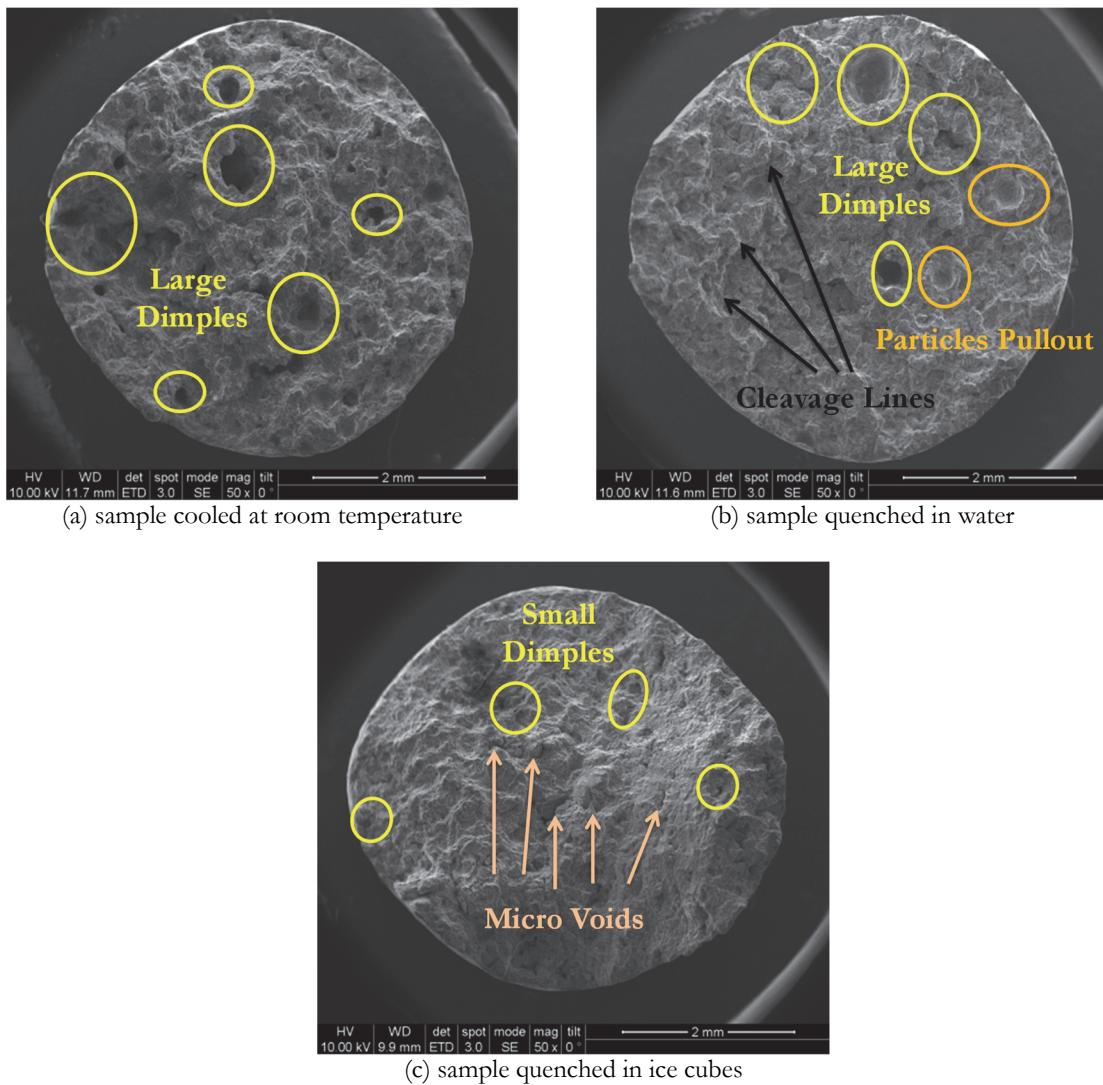


Figure 5: Tensile fracture imageries of hybrid composites quenched in different media.

A study on structures of fractured surface was carried out to examine the fractured region. This study helps to identify the initiation of micro-cracks and excessively loaded regions to find acceptable degree / level of features of fracture [34]. Dimple sizes on the fractured surfaces which were quenched in water is much smaller when compared with the fractured surfaces of the composite samples which were cooled under room temperature. Similarly, the ice quenched samples show smaller size of dimples when compared with the samples quenched in water. Normally, size of the dimples has a direct proportional relationship with the strength of the developed composite [27].

#### *Compression strength*

Compressive strength experiments were studied on the developed hybrid MMCs. The compression test specimens were prepared as per the ASTM E9 standards with specimen size of dia 10 mm \* 25 mm thickness. Outcomes are depicted in Fig. 6. The developed MMCs revealed considerably high compression strength with higher Al<sub>2</sub>O<sub>3</sub> content. This outcome



can be mainly due to the stircasting process and homogeneous distribution of reinforcement. The bonding among the reinforcement and the base matrix generally led to reduction in the porosity [35]. Uniform dispersal of reinforcement in MMCs is the common cause for alloy strength. In this process, alumina as fine particulates throughout the base matrix can act like a barrier to the dislocation, generally, which leads to improvement in the mechanical characteristics of AMMCs [36]. Generally, the hardness as well as compressive strength of the materials was directly proportional to the each other. Therefore, high compression strength in MMCs could be attributed to the enhanced hardness. Fig. 6 shows that the compression strength of developed hybrid MMCs were reduced when there was an increase in MoS<sub>2</sub> content. Researchers [37] stated that the solid lubricant particulates effectively affect the compression stability. However, the negative outcomes impact the robustness. The reduction of compressive strength in the present research work may be due to particulates pull-out and crack propagations, which are instigated by the presence of MoS<sub>2</sub> particulates [38]. From the outcomes, it is also observed that the test samples quenched in ice cubes exhibit high compression strength. This improvement is due to the developments of the intermetallic precipitations, which, usually, prevent pinning down of the dislocations, and thus reduces the extent of plastic deformations and results in significant enhancement in compression strength of developed hybrid composites [39].

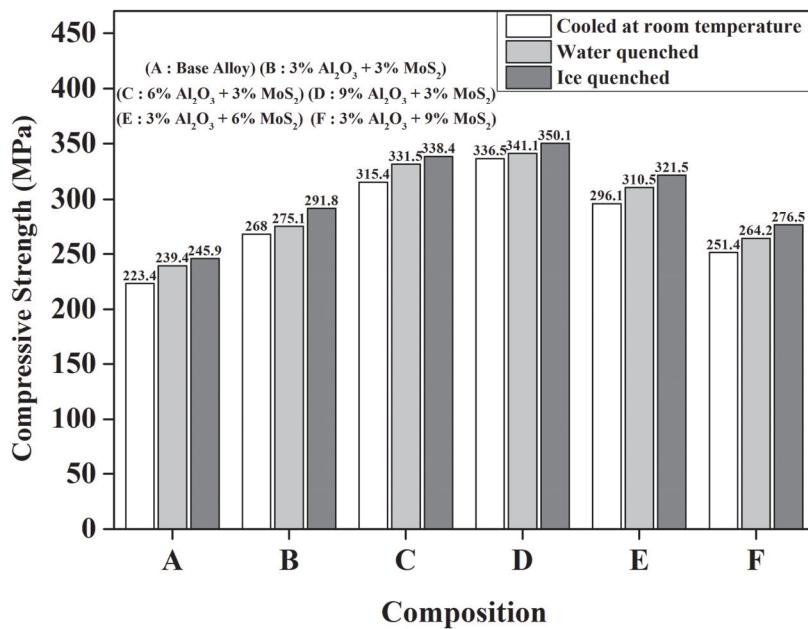


Figure 6: Compression strength with varying content of Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>.

#### *Wear loss*

Wear behavior was studied by conducting tests as per ASTM standards at a constant sliding-speed of 1.66 m/s and a load of 30 N against a steel disc (grade: EN-32). Test samples of 32 mm length and dia of 8 mm were manufactured by CNC machining. The amount of wear out of the developed hybrid MMCs was determined by the loss of weight method. Fig. 7 shows the wear loss of hybrid MMCs. The presence of ceramic hard Al<sub>2</sub>O<sub>3</sub> particulates within the Al matrix leads to high wear resistance [33]. Generally, the Al<sub>2</sub>O<sub>3</sub> particulates lead to an improvement in the transition load due to the strengthening mechanism. The Al<sub>2</sub>O<sub>3</sub> particulate decreases the inter-particulate spacing and also acts like a good barrier dislocations for the movement. The hard particles have an ability to carry a high load mainly due to the improved hardness. And, also, the fractured Al<sub>2</sub>O<sub>3</sub> particles rub against the hard disc which generally leads to better wear resistance of developed hybrid MMCs. In Al matrix, presence of Al<sub>2</sub>O<sub>3</sub> particles grips the applied load and MoS<sub>2</sub> creates a lubricating film, leading to a reduction in plastic deformations. The presence of MoS<sub>2</sub> is basically sustainable particulates leads to better wear resistance [40]. The reinforcements possess lubricating properties, which favours them using them within the MMCs. The solid lubricating particulates like MoS<sub>2</sub> show enhanced wear resistance of the developed MMCs. The enhancement of the wear resistance in the MMCs is due to development of rich film layers on wear surface because of dry sliding behaviour generally which restricts the plastic deformations in developed composite. Tribo layers are formed on these MMCs, and the development of these layers is due to the existence of MoS<sub>2</sub> content. Generally, the layers which form on the pin will avoid direct contact between the Al and the steel disc. Moreover, better wear rate is achieved because of development of strain fields nearby the reinforcements. The fine dispersal of the reinforcements within the base matrix

is the main factor for improving the wear behavior. Uniform distribution of the reinforcements prevents dislocations, which occurs in the material structures [41]. The wear study revealed that the high hardening of the base matrix was achieved when the MMCs were heat treated at a temperature of 510°C and quenched in ice cube. It is revealed that heat treatment under T6 condition provided better hardness and thus it led to higher wear resistance in MMCs [42]. The higher hardness of the developed MMCs by T6 heat treatment condition would have the benefit of avoiding the development of Al debris and also decreasing its transfer towards the surface of steel [28]. The wear loss equation is as shown in Eqn. (1).

$$\text{Wear loss (g)} = (\text{Initial Weight} - \text{Final Weight}) \quad (1)$$

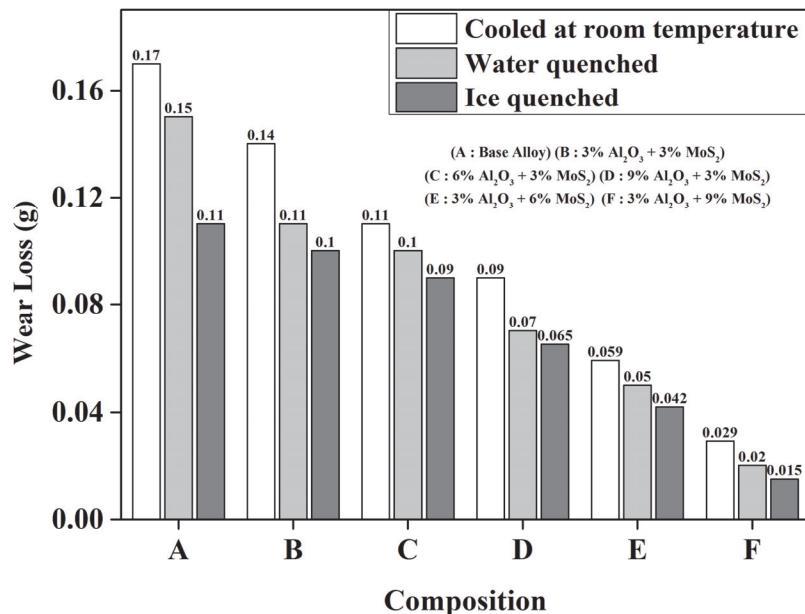
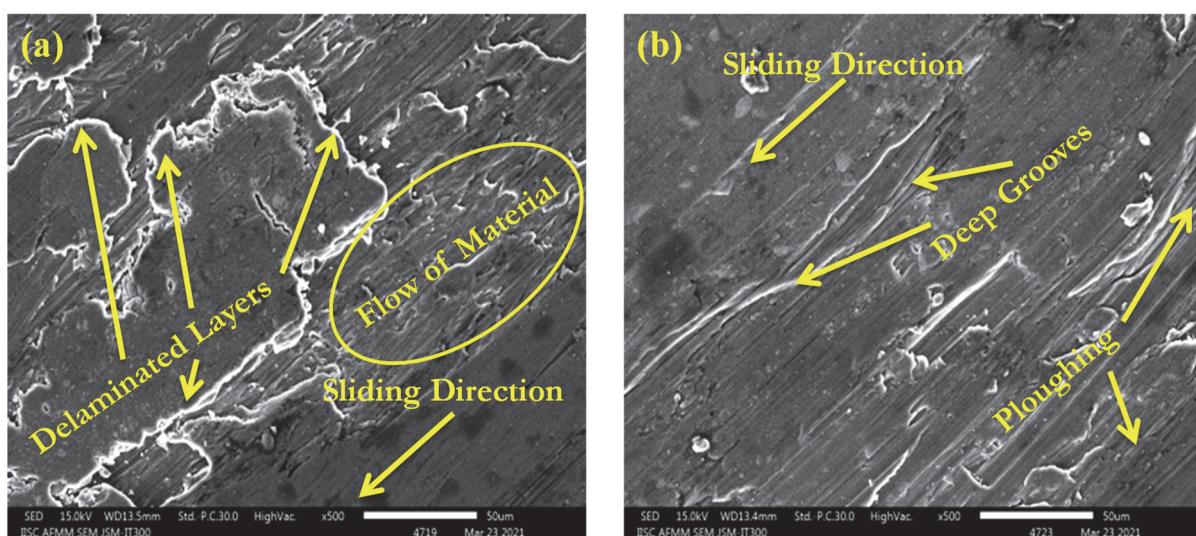


Figure 7: Wear loss with varying content of Al<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>.

SEM images of the wornout surface of the (a) sample cooled at room temperature (b) sample quenched in water and (c) sample quenched in ice are depicted in Fig. 8. The study of SEM analysis on the wear tracks have made a possible to analyse that, the path was not homogenous, but a non-uniform one, showing wear surfaces areas with deep grooves along the direction of sliding and also plastic deformations.



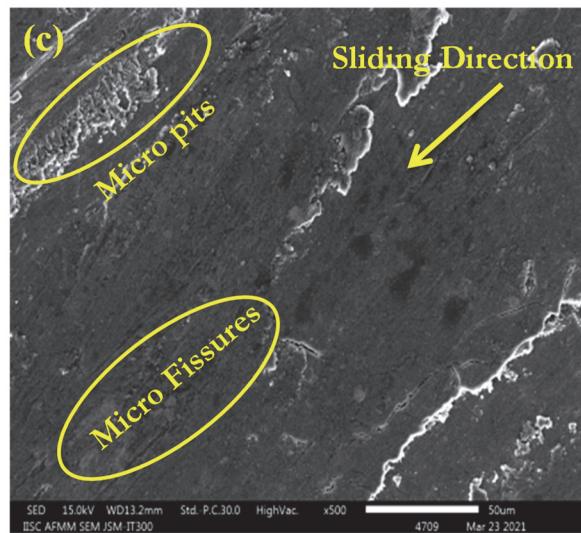


Figure 8: Wornout SEM images of (a) sample cooled at room temperature, (b) sample quenched in water (c) sample quenched in ice cubes.

Generally, the grooves are formed by the reinforcing particulates. It is observed that the layers of material have been removed like debris from the sample surfaces and that debris is in form of a thin sheet. From the SEM analysis of wornout surfaces, it was found that wear damages were caused due to plastic flow of the alloy with an accumulation of materials [43]. From the SEM images of wornout surfaces, it was observed that sample quenched in ice cubes has fewer grooves and scratches on the surface. Generally, this is due to the particulates stuck between the sliding surfaces at the time of contact between the samples surface and hard steel disc. The chances of de-bonding of the particles is due to continuous sliding which causes the particles to get detached from the base matrix and stick on the sliding surfaces. And this acts like an abrader leading to the short duration of abrasive wear in samples. This results leads to enhancement of wear resistance. In Fig. 9, EDS study indicates the main elements of  $\text{Al}_2\text{O}_3/\text{MoS}_2$  reinforced hybrid AMMCs such as Fe, Mg, Zn, O and Mo are detected. The observation / presence of the (O) oxygen may be due to the existence of the alumina ( $\text{Al}_2\text{O}_3$ ) content and presence of "Mo" confirms the existence of  $\text{MoS}_2$  content in developed hybrid composite [44]. The outcomes reveal that the composition of the  $\text{Al}_2\text{O}_3/\text{MoS}_2$  reinforced Al hybrid MMCs are reliable.

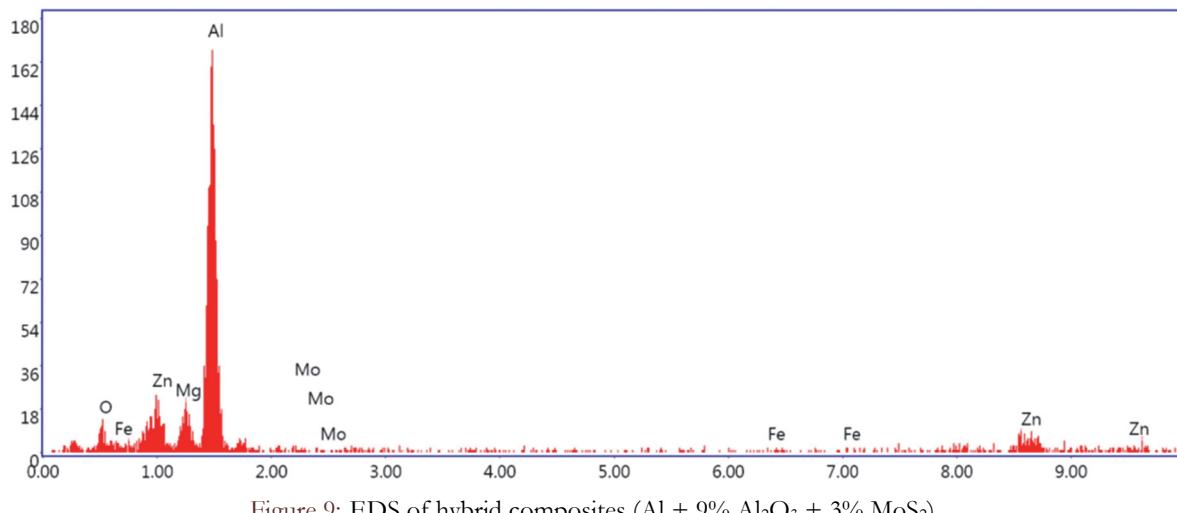


Figure 9: EDS of hybrid composites (Al + 9%  $\text{Al}_2\text{O}_3$  + 3%  $\text{MoS}_2$ ).

## CONCLUSION

In the present research work, the Al6061-Al<sub>2</sub>O<sub>3</sub>+MoS<sub>2</sub> hybrid composites were effectively fabricated using stircasting method. The microstructural study, mechanical, and wear behavior of developed hybrid MMCs were evaluated. The outcomes are as given below:



- A uniform distribution of reinforced particles is seen in the developed hybrid MMCs.
- Hardness of the developed hybrid composites increased with increase in  $\text{Al}_2\text{O}_3$  content. Further, the hybrid MMCs became deformable by addition of  $\text{MoS}_2$  particulates which led to a decrease in micro hardness of hybrid MMCs.
- Tensile and compression strength of hybrid composite was improved by addition of  $\text{Al}_2\text{O}_3$  particulates. However, further increase in wt. % of  $\text{MoS}_2$  particulates, led to a decrease in tensile strength. The strength of developed hybrid composite enhanced due to the change in CTE. Generally, this led to an increase in the dislocation of density. The outcome also reveals that increase in the dislocation of density, strength of developed hybrid composites can be improved.
- From the outcomes it is also found that, the composite samples quenched in ice cubes show better material properties compared to water quenched samples and samples cooled at room temperature.
- From the fractography study, it is concluded that ice quenched samples show smaller size of dimples when compared to the samples quenched in water and samples cooled at room temperature. Generally, the sizes of dimples are directly proportional to the relationship with the strength of composites.
- The presence of  $\text{Al}_2\text{O}_3$  and  $\text{MoS}_2$  particulates among the contact surfaces reduces amount of wear in the developed hybrid composites. It is evident from the study that the developed hybrid MMCs showed self-lubricating behavior which makes resource effective materials.
- From the SEM images of wornout surface,  $\text{Al}_2\text{O}_3$  and  $\text{MoS}_2$  particles are revealed to have significant effects on wear behavior of hybrid MMCs.
- From the EDS analysis, the presence of oxygen (O) are identified due to existence of the alumina ( $\text{Al}_2\text{O}_3$ ) content and "Mo" confirms the existence of  $\text{MoS}_2$  content in developed hybrid composite.

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