Jurnal Tribologi 38 (2023) 82-99

MYTRIBOS Malaysian Tribology Society

Jurnal Tribologi

Website : jurnaltribologi.mytribos.org e-issn: 2289-7232

Nano sized SiC - Gr particulates reinforced Al7075 hybrid composite: Experimental studies and analysis of quenching agents

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KEYWORDS

Al7075
Microstructure analysis
Mechanical behavior
Wear behavior
SEM

ABSTRACT

The cooling agent, cooling rate and temperature of cooling are expected to influence the hardness, tensile, and wear behavior of the Metal Matrix Composites (Al MMCs). This research shows the outcomes of a sequence of experiments conducted to study the mechanical and wear behavior of the Al7075 reinforced by nano sized SiC-Gr hybrid composites that are quenched with two different quenching agents like water and ice cubes. Heat treated hybrid composites were subjected to evaluate the hardness, tensile, and wear behavior. The outcomes for the ice quenched samples with 3% SiC & 1% Gr reinforced samples reveals that the heat treatment significantly enhances 64.10% of hardness, 44% of tensile and compression strength and 77.7% of wear resistance were observed in hybrid composites. Highest hardness 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 and micro fissures were observed in the wornout surface of the wear test samples of developed hybrid composite.

Received 27 October 2022; received in revised form 22 December 2022; accepted 3 March 2023.
To cite this article: Ravikumar et al. (2023). Nano sized SiC - Gr particulates reinforced Al7075 hybrid composite: experimental studies and analysis of quenching agents. Jurnal Tribologi 38, pp.82-99.

1.0 INTRODUCTION

Environmental 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. The most advanced light-weight materials are Al composites. The SiC and Gr particulates reinforced hybrid Al composite is widely used in brake pads, brake rotors, piston head, drum brakes, engine cylinders, engine pushrod, light weight structures and so on. 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. One of the techniques used to enhance the material strength is the heat treatment process (Ravikumar et al., 2022, Narayan et al., 2020, Gangadharappa Et. al., 2021). Heat treatment process can increase the grain refinement and also improve bonding between reinforcement and the matrix. Al7075-Al_2O_3-Gr is an Al composite frequently used in automotive components. In automotive parts, the Al composites are usually heat treated under the T6 condition. Heat-treatment is a sequence of heating and cooling to change mechanical and physical characteristics of the developed composite (Ravikumar et al., 2018, Benjunior et al., 2017).

In the heat treatment process, heating is done to a particular temperature followed by cooling in various cooling media. The heat-treatment of Al MMCs is generally done by T6 condition followed in the various stages: solutionizing, quenching and finally, artificial ageing (Ravikumar et al., 2021, Aytekin et al., 2015). Earlier studies (Marialaura et al., 2017) 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. 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 (Narayan et al., 2019, Ravikumar & Rudra naik, 2022). Normally, this precipitation enhances the properties of a developed material, i.e. mainly, tensile and hardness. Researcher (Murlidhar Patel et al., 2010) investigated the tribological properties of stir cast AA5052/B_4C MMC under different loads. The results show that at all applied load, AA5052/B_4C AMMC having improved wear resistance and reduction in wear rate with lower density. 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. The quenching rate should be high enough to achieve a better strength in the developed composite material. Previous research study of showed that faster cooling created high residual stresses which initiated distortion and cracks (Ravikumar et al., 2018, Milkereit et al., 2014). Quenching consistency is a process of quick cooling which leads to a reduction in the fracture and also residual stress. The cooling rates significantly influence the micro-structure and material properties of composites (Campos & Totten, 2005). 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 6XXX 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 (Buczek & Telejko, 2013). When the temperature is less than 200°C, the rate of cooling is normally slowed, resulting in the best mechanical behaviour (B.M. Viswanatha et al., 2021, Li et al., 2013, Chobaut et al., 2015). 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 SiC and Gr reinforced Al7075 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 Al7075-SiC-Gr hybrid MMCs to attain better mechanical properties and high wear resistance.

2.0 MATERIALS, FABRICATION, AND HEAT TREATMENT PROCESS

Al7075 commercial grade material was used as a base material (chemical composition of the Al7075 alloy is as shown in Table 1). The two different reinforcing particulates such as nano sized SiC - Gr with wt. % of 1, 2, 3 were used for the preparation of hybrid composites. The nano sized SiC reinforcement is significantly improves the ultimate tensile strength, hardness and tribological behavior. Nano sized Gr 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, SiC and Gr with 80 nm size of particulates were used as reinforcements. A stircasting process technique was implemented for producing the hybrid metal matrix composites. The SiC and Gr were pre-heated in a separate crucible till 350° C. The base material was melted (640°C) using electric furnace and hard reinforcing SiC and Gr pre-heated particulates were mixed into molten melt. The stirring process was maintained continuously at 100 rpm. The molten melt was poured continuously in to the metallic mold which was pre-heated. 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 standard used for tensile test specimen is ASTM-E8, for hardness test specimen is ASTME384-11-1 and for wear test specimens' preparation is ASTMG99-05. The developed composites and prepared samples as per ASTM standards are shown in Figure 1.

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 (Li et al., 2013, Gangadharappa et al., 2018). Heat-treatment process was selected on the basis of functions and the nature of material as defined by the kind of cooling agent. Generally, the quenching plays a major role in the heat-treatment process (Reddappa et al., 2011). Developed hybrid composite samples were subjected to solutionizing at 510ºC of temperature for 2 hours and separately quenched. Here, two types of quenching agents such as ice cubes and water were used. The water quenching of samples were carried out at temperature of 30°C for the duration of 60 minutes. Similarly ice quenched samples were processed through ice cubes at the temperature of at 0°C for the duration of 60 minutes. After quenching, the same samples were subjected to age-hardening process at a constant temperature of 160ºC for 4 hours and finally cooled at room temperature. The surface of the water and ice quenched samples shows the case hardening upto the 0.3 - 0.5 mm depth from the surface. Further, it is going to affecting the processing like forming and machinability of materials. So, for the normalization of entire structure the age hardened processes were carried out.

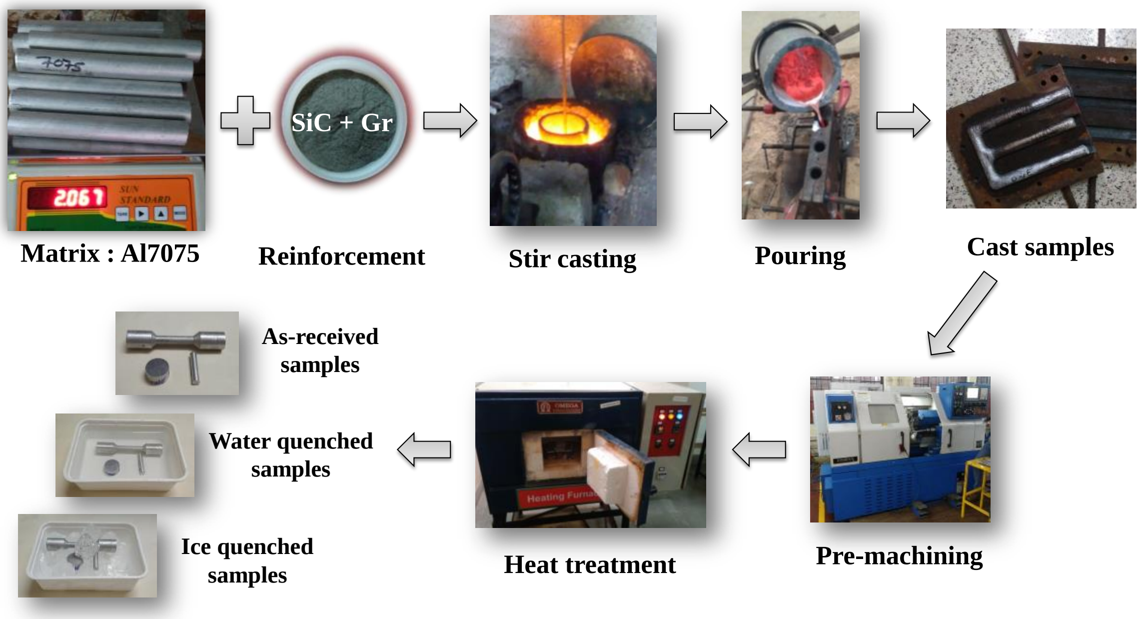

Figure 1: Developed composites and prepared samples as per ASTM standards.

Table 1: Chemical Composition of Al7075 alloy with wt. %.

Content	Al	Cu	Mg	Si	Fe	Mn	Ni	Pb	Sn	Ti	Zn	Cr
Wt. %	Rem	1.480	2.306	0.059	0.256	0.052	0.052	0.023	0.012	0.052	5.424	0.280

3.0 RESULTS AND DISCUSSION

3.1 Microstructure Analysis

Uniform distributions of reinforcements show an improvement wear and mechanical behavior of developed composites (Ravikumar et al., 2018). Figure 2(a) depicts the micro-structure of the water quenched C4 sample (3% SiC + 1% Gr). Figure 2(b) shows the micro-structure of ice quenched C4 sample (3% SiC + 1% Gr). 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 (Ravikumar et al., 2018). 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 (Ravikumar et al., 2022). Generally, it is known that reinforcing of hard particulates in the Al base matrix causes increase in grain refinement. 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. From the Figure 2 (a & b), it is also observed that, the water quenched samples shows the minimum dendritic structure growth. Similarly for ice quenched samples with dendritic needle like structures were observed. And also sharp increase in hardness and tensile strength can be observed. Similar results were observed by other researchers (Ravikumar et al., 2021, Ravikumar et al., 2021) and it is concluded that, the Al grain generally solidifies near the hard particulates which leads to compromising of the resistance to grain growth.

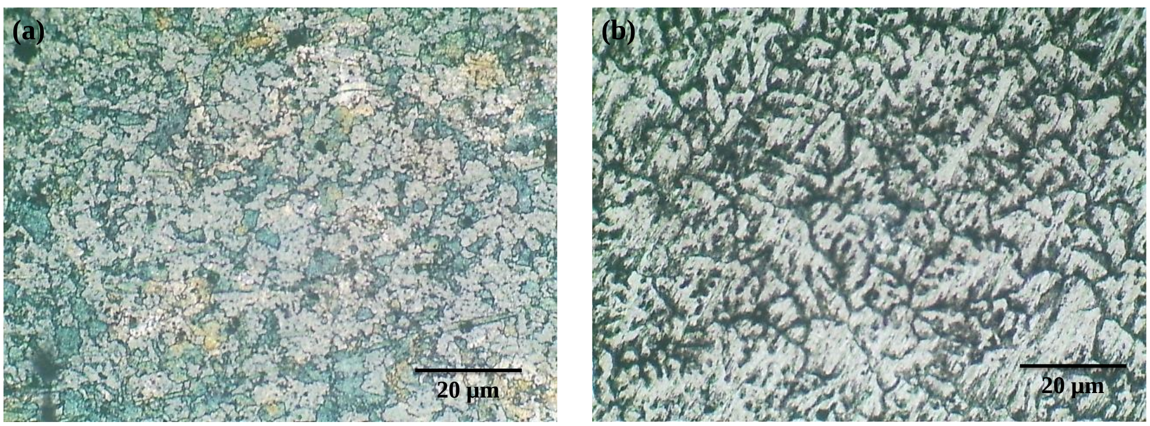

Figure 2: Microstructure of (a) water quenched sample (Al7075 + 3% SiC + 1% Gr), (b) Ice quenched sample (Al7075 + 3% SiC + 1% Gr).

3.2 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 diamond-shaped indenter 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 Figur 3. Addition of hard particulates (SiC) to the Al matrix enhanced the hardness of composites (Vasanth kumar et al., 2022). In Figure 3, higher hardness values are observed due to increasing of SiC content which proves that the adding of SiC particulates effects 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 (Ravikumar et al., 2017) 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 Gr content. The reduction in the hardness by increasing in Gr particulates is because of high lubrication characteristics of the Gr particles that cause movement of grains (Pradeep et al., 2017. From Figure 3, 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. The treatment of solutionizing indicates the development of intermetallic phases which leads to higher hardness.

In heat treated MMCs, thermal mismatching of the base material and reinforcement leads in density enhancements in the dislocation. The water quenching and ice quenching process were enhancing the properties of composite materials. During these processes, the grain growth takes place and it leds to the density enhancement. 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. Other researchers (Reddappa et al., 2011, Vencl & Mijskovi, 2008) 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.

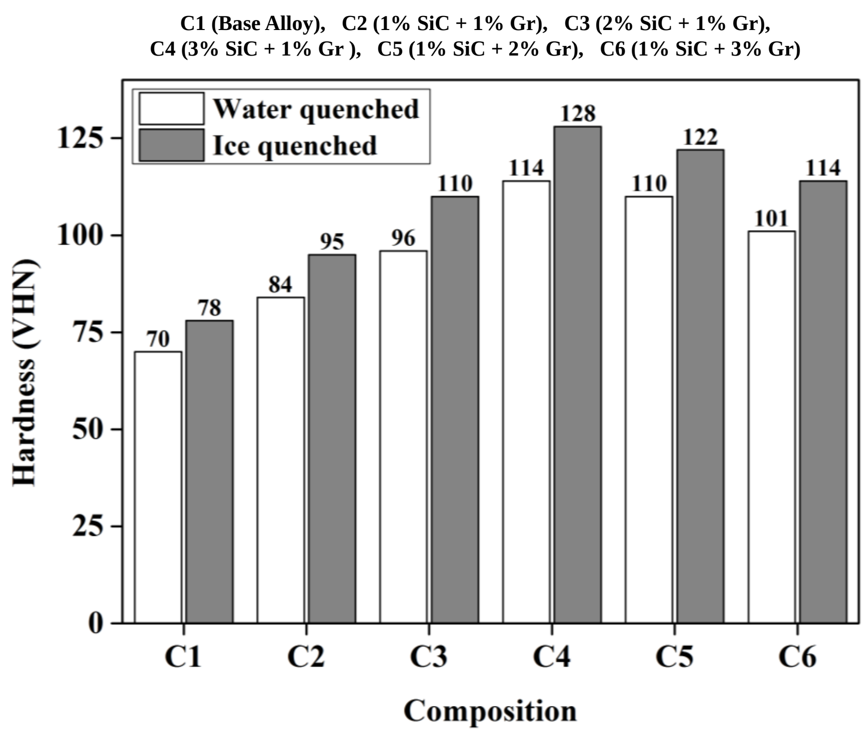

Figure 3: Vickers micro hardness with varying content of SiC and Gr.

3.3 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. Figure 4 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 SiC. This is generally, because of the presence of hard particles in developed composites. 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 (Subramanya et al., 2017, Palanisamy et al., 2019). The hard particulate correlates with the dislocation, resulting in enhancement of strength in developed composites. Similar results have been witnessed by other researchers (Gangadharappa et al., 2018). Further, the tensile strength was reduced by increasing the Gr content and a similar outcome was found by the other researcher (Juliyana et al., 2022).

Due to the presence of Gr particulates, there may be possibility of crack propagation and 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.

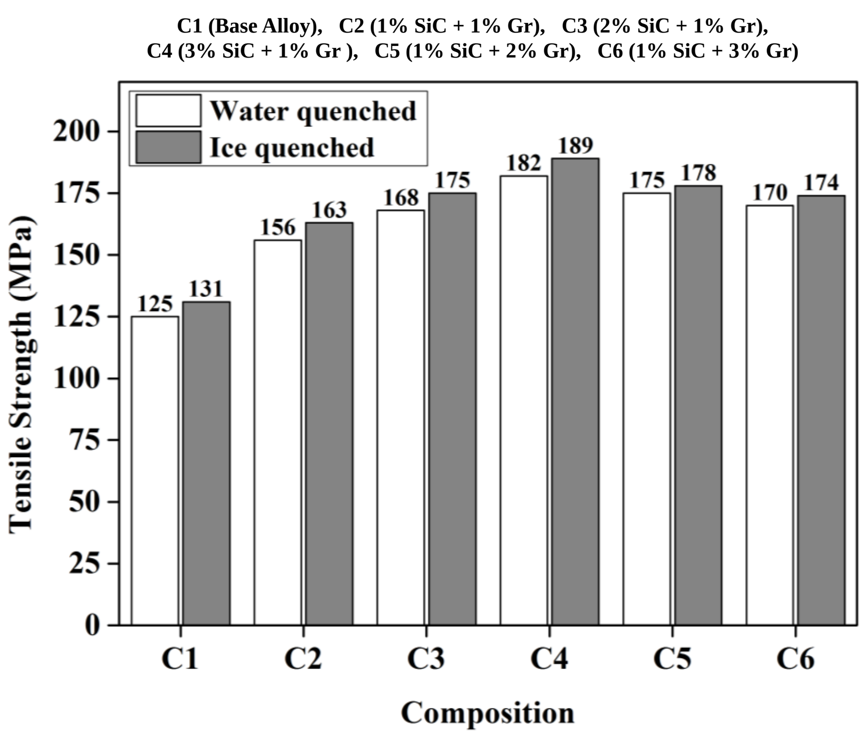

Figure 4: Tensile strength with varying content of SiC and Gr.

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 Figure 4, 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 inter-metallic 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 (Uvaraja et al., 2015).

The tensile stress-strain curves of the composite samples fabricated by the stir casting method are shown in Figure 5. Out of all the developed composite specimens, the tensile strength is higher for ice quenched specimen. The stress-strain curve indicates the improved toughness apart from high tensile strength. This is significant. Meanwhile, most strength improvement methods cause decreasing ductility.

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. During the fracture, the SiC particles will detachment from the surface and it creates the deep voids. The fractography of sample quenched in water and sample quenched in ice cubes are depicted in the Figure 6.

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 (Suvarna & Kumar, 2014). Dimple sizes on the fractured surfaces which were quenched in ice cube is much smaller when compared with the fractured surfaces of the composite samples quenched in water, size of the dimples has a direct proportional relationship with the strength of the developed composite (Ravikumar et al., 2017).

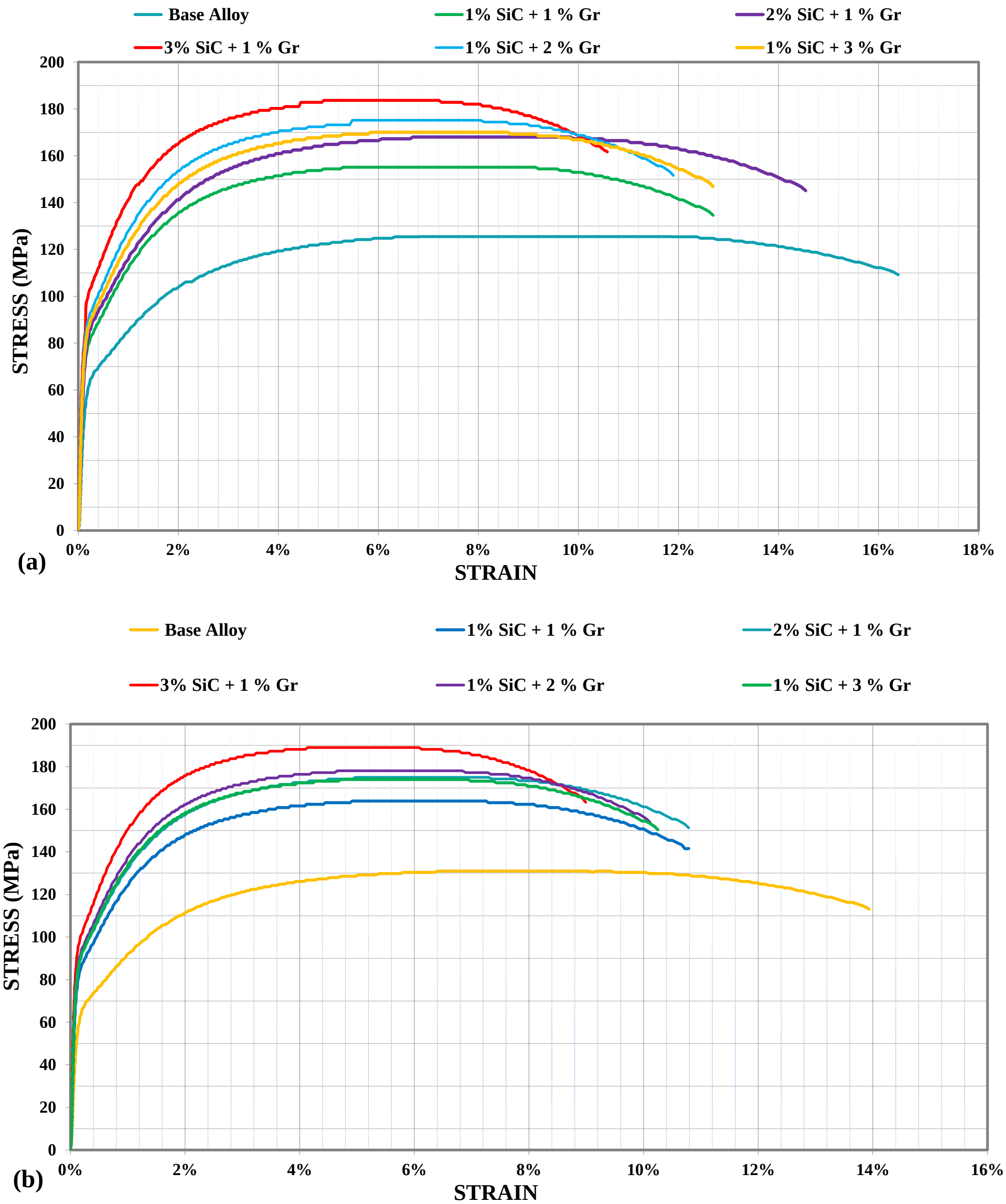

Figure 5: (a) Stress-strain curve for water quenched samples (b) Stress-strain curve for ice quenched samples.

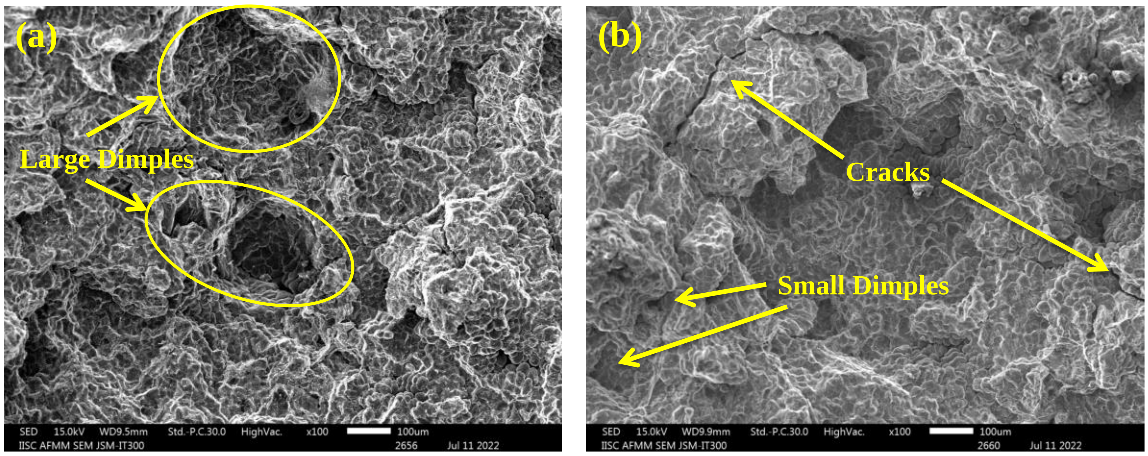

Figure 6: Tensile fracture imageries of hybrid composites quenched in different media,
(a) Sample C4 (3% SiC +1% Gr) quenched in water b) Sample C4 (3% SiC +1% Gr) quenched in ice cubes.

3.4 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 Figure 7. The developed MMCs revealed considerably high compression strength with higher SiC 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. Uniform dispersal of reinforcement in MMCs is the common cause for alloy strength. In this process, SiC 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 (Saheed et al., 2021, Amit et al., 2018). Generally, the hardness as well as compressive strength of the materials was directly proportional to each other. Therefore, high compression strength in MMCs could be attributed to the enhanced hardness. Figure 7 shows that the compression strength of developed hybrid MMCs were reduced when there was an increase in Gr content. Researchers (Nikhil et al., 2018) 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 Gr particulates (Swamy et al., 2011). 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 (Ravikumar, et al., 2018).

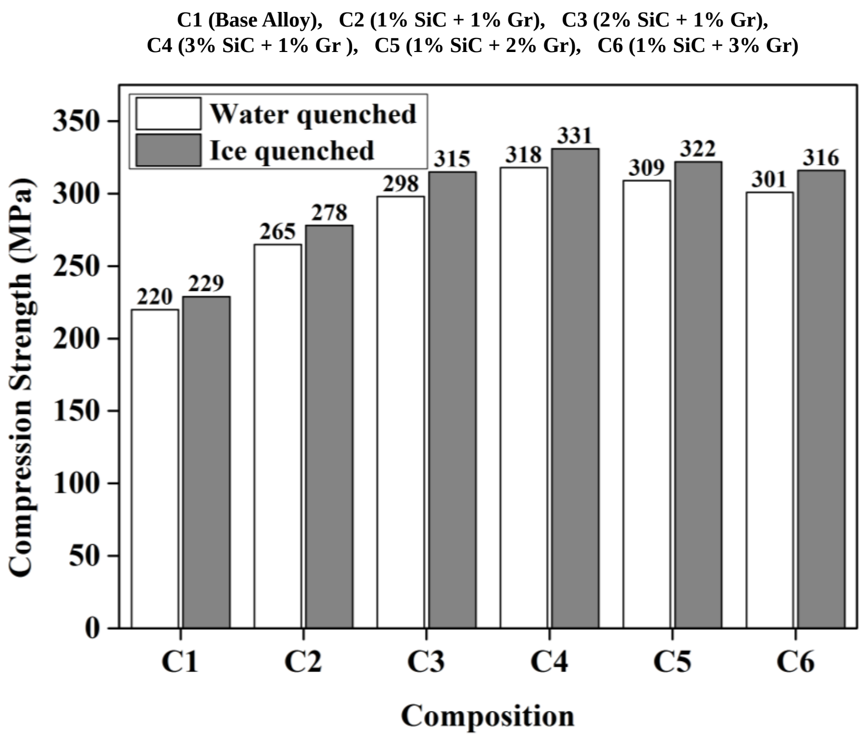

Figure 7: Compression strength with varying content of SiC and Gr.

3.5 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. Figure 8 shows the wear loss of hybrid MMCs. The presence of ceramic hard SiC particulates within the Al matrix leads to high wear resistance (Uvaraja et al., 2015). Generally, the SiC particulates lead to an improvement in the transition load due to the strengthening mechanism. The SiC particulate decreases the inter-particulate spacing and also acts like a good barrier dislocation for the movement. The hard particles have an ability to carry a high load mainly due to the improved hardness. And, also, the fractured SiC particles rub against the hard disc which generally leads to better wear resistance of developed hybrid MMCs. In Al matrix, presence of SiC particles grips the applied load and Gr creates a lubricating film, leading to a reduction in plastic deformations. The presence of Gr is basically sustainable particulates leads to better wear resistance (Saikrupa et al., 2021).

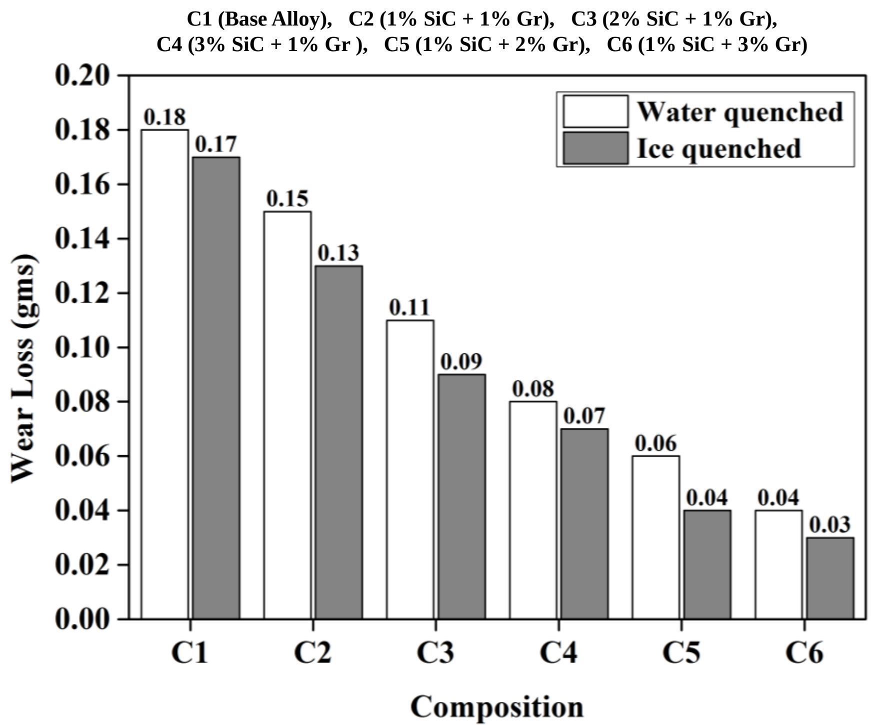

Figure 8: Wear loss with varying content of SiC and Gr.

The reinforcements possess lubricating properties, which favours them using them within the MMCs. The solid lubricating particulates like Gr 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 Gr 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 (Amar et al., 2021). 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 (Pradeep et al., 2017). 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 (Gomez & Barrena, 2014). When increase in the SiC particles, it leds to decreases the wear rate. It is due to the SiC particles resist for the wear of the developed composites and acts as a barrier for the dislocation of movements in matrix material. Whereas, Gr particles is one of the solid lubricant which leds to form a lubricating layer on the contact surface and slides smoothly which helps to reduce the wear rate.

SEM images of the wornout surface of the (a) sample quenched in water and (b) sample quenched in ice are depicted in Figure 9. The study of SEM analysis on the wear tracks has 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.

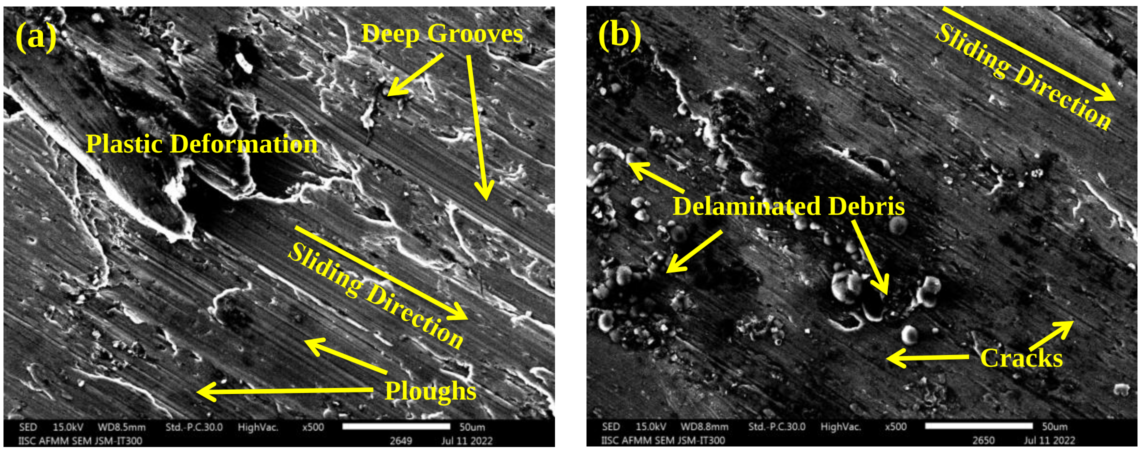

Figure 9: Wornout SEM images of (a) Sample C4 (3% SiC +1% Gr) quenched in water b) Sample C4 (3% SiC +1% Gr) quenched in ice cubes.

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 (Mahagundappa et al., 2007). 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 result leads to enhancement of wear resistance. In Figure 10, EDS study indicates the main elements of SiC/Gr reinforced hybrid AMMCs such as Fe, Mg, Zn, O and C are detected. The observation / presence of the "Si" may be due to the existence of the silicon carbide (SiC) content (Ravikumar et al., 2021) and presence of "C" confirms the existence of Gr content in developed hybrid composite (Banerjee & Sahoo, 2022). The outcomes reveal that the composition of the SiC/Gr reinforced Al hybrid MMCs are reliable.

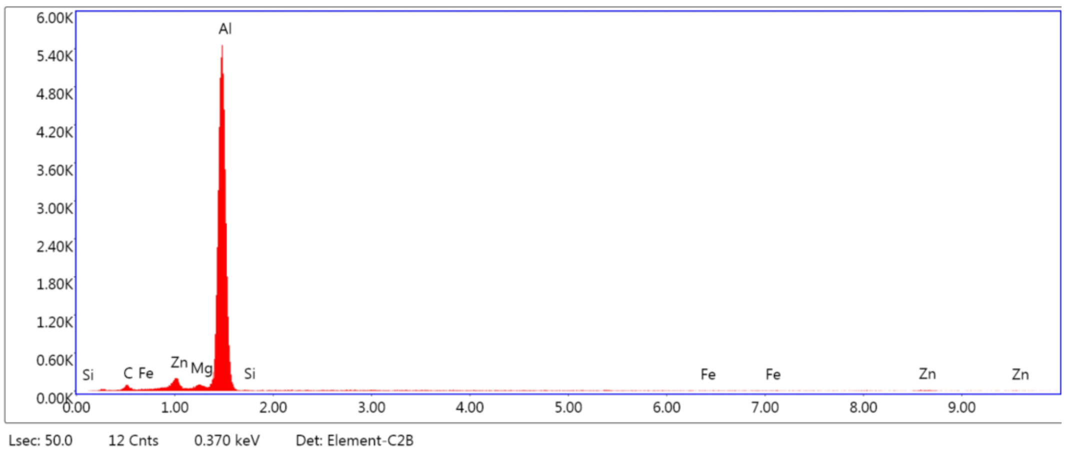

Figure 10: EDS of hybrid composites (Al + 3% SiC + 1% Gr).

CONCLUSIONS

In the present research work, the Al7075 reinforced by nano sized SiC +Gr 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 nano sized SiC content. Further, the hybrid MMCs became deformable by addition of nano sized Gr particulates which led to a decrease in micro hardness of hybrid MMCs. Tensile and compression strength of hybrid composite was improved by addition of nano sized SiC particulates. However, further increase in wt. % of nano sized Gr 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 the dislocation of density. The outcome also reveals that increase in the dislocation of density; strength of developed hybrid composites can be improved.

Along with the SiC particles the quenching agents supported for the grain growth,and it will leds to enhance the mechanical and wear properties of the developed composite materials. From the outcomes it is found that, the composite samples quenched in ice cubes show better material properties compared to water quenched samples. For ice quenched samples (3 % SiC +1 % Gr), 64.10 % of hardness, 44 % of tensile and compression strength and 77.77 % of wear resistance were observed in hybrid composites when compared to the water quenched samples.

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 nano sized SiC and Gr 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, nano sized SiC and Gr particles are revealed to have significant effects on wear behavior of hybrid MMCs. From the EDS analysis, the presence of oxygen “Si” is identified due to existence of the SiC content and “C” confirms the existence of Gr content in developed hybrid composite.

ACKNOWLEDGMENT
The authors gratefully acknowledge the technical support of Sri Vivekananda Research Laboratories, Bangalore.

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