



ENHANCEMENT IN MICROSTRUCTURAL CHARACTERISTICS AND MECHANICAL PROPERTIES OF A6082-BASED MMCS VIA YTTRIA STABILIZED ZIRCONIA INCORPORATION AND T6 HEAT TREATMENT

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Abstract

This study investigates the impact of Yttria Stabilized Zirconia (YSZ) and T6 heat treatment on the mechanical and microstructural properties of A6082-based Metal Matrix Composites (MMCs) prepared using the conventional stir casting route. A6082 is widely used in various industrial applications due to its excellent physical, structural and functional properties with good corrosion resistance. YSZ with superior mechanical and thermal properties that can enhance the strength and wear resistance of aluminium-based composites. T6 heat treatment is a widely adopted technique to improve the material properties of aluminium alloys. The results of this study indicate that the T6 heat treatment significantly refines the grain structure of the composites, resulting in improved hardness and tensile values compared to untreated samples. Mechanical properties are also enhanced by YSZ's integration into the composites, with a 9wt.% YSZ addition to the matrix alloy leading to a noticeable improvement in hardness for both the untreated and treated composites. The tensile parameters show a similar trend, with a gain in Ultimate Tensile Strength and Yield Strength up to 6wt.% YSZ addition, after which the findings follow a downtrend (at 9wt.%). The results of this research show that A6082/YSZ composites subjected to T6 heat treatment have great promise as a high-performance material.

Keywords: Yttria stabilized Zirconia (YSZ), Magnesium Silicide (Mg₂Si), T6 heat treatment.

1. Introduction

The automotive industry is continuously seeking ways to reduce vehicle weight to improve fuel efficiency, reduce energy costs, and minimize vehicle emissions. Aluminium (Al) alloys are becoming increasingly popular in this industry due to their high specific strength and excellent

corrosion resistance [1], [2], [3]. Among the various Al alloys, the weldable 6xxx series Al-Mg-Si sheet alloys are already the preferred material for outer panels and hang-on parts, and are now being increasingly used for structural parts where strength is a crucial factor. These alloys are heat-treatable by age-hardening, which

involves a precipitation sequence starting with a supersaturated solid solution (SSSS) followed by solute clusters, GP zones (spherical), β'' (needle), β' (rod), and finally β (Mg₂Si) [4], [5]. Peak age hardening leads to the most effective β'' phase, resulting in maximum strength, while natural ageing leads to intermediate strength produced by GP zones.

Compared to 5xxx series alloys, 6xxx series alloys offer better mechanical properties, although their corrosion resistance is relatively low. Among the 6xxx series alloys, alloy 6082 exhibits the highest strength, which can be further enhanced by T6 tempering [6]. Additionally, its corrosion resistance is improved due to the low copper content, which can be further increased by precipitation hardening heat treatment. The strengthening during precipitation hardening is a result of the precipitation of various metastable phases in the aluminium solid solution. Super Saturated Solid Solution (SSSS), to be strengthened requires formation of secondary phase particles, which are small and uniformly dispersed in the main phase [7]. In 6xxx series aluminium alloys, it is believed that the precipitation sequence during ageing follows the general scheme for ternary Al-Mg-Si alloys: supersaturated solid solution α (Al) \rightarrow GP zones \rightarrow β'' \rightarrow β' \rightarrow β (Mg₂Si).

After the solution heat treatment of Al alloys, the formation of supersaturated solid solution α (Al) takes place upon rapid cooling to room temperature. Subsequently, clusters of Mg and Si atoms form as coherent matrix in GP zones. Metastable β'' phase in the form of partially coherent, fine needles and β' phase in the form of rod shape is then formed. The final stage of decomposition is the equilibrium β (Mg₂Si) phase [8]. By varying the temperature and duration of solution heat and artificial aging, modifications in precipitate phases can be achieved.

The incorporation of ceramic nanoparticles

in the aluminium matrix has been found to significantly enhance the properties of the resulting material, leading to the development of aluminium matrix composites [9], [10]. The strength of these composites depends on the type of reinforcement used, which must have a high strength-to-weight ratio and a high modulus ratio in order to effectively withstand applied loads [11]. Various types of ceramic nanoparticles, such as ZnO, ZrO₂, MgO, B₄C, Al₂O₃, TiB₂ and SiC, are commonly used as reinforcements due to their excellent properties, including high hardness, refractoriness, wear-resistance, and high compressive strength [12], [13], [14], [15], [16], [17]. To produce aluminium nano metal matrix composites, effective casting techniques are crucial. Ensuring an even distribution of reinforcements, promoting good wettability, and preventing oxidation between the matrix and reinforcements are critical obstacles to overcome. The characteristics of the resulting nano composites are significantly influenced by the choice of both reinforcements and the base matrix. By selecting an appropriate combination of materials, the resulting composite can exhibit improved properties that are well-suited for use in engineering applications [18].

Hence a novel approach has been adopted to fabricate A6082-based composites which is reinforced with different quantity of Yttria stabilized Zirconia. The work involves the microstructural and mechanical analysis of the fabricated composites with and without T6 heat treatment. From the literature, it can be clearly seen that, although the researchers have concentrated on reinforcing A6082 with different ceramic particles, but no attempt has been made to analyse the effect of YSZ on the microstructural and mechanical behaviour of the A6082-alloy. Therefore, this work will spread light on the new MMC and its widespread application.

2. Materials and Method

To fill the void from the literature review made. The A6082 alloy was chosen as the base matrix, while Yttria stabilized Zirconia (YSZ) as the reinforcement. These are procured from MINCOMETSAL, Bengaluru and been analysed for composition using EDX.

Stir casting route, remains most economical and extensively used for fabricating the MMCs. Initially, the desired amount of A6082 alloy is melted in an electrical resistance furnace at $700\pm 5^{\circ}\text{C}$ and pre-heated ($400\pm 5^{\circ}\text{C}$) YSZ reinforcement then added (with an increment of 1.5 wt.% until 9 wt.%) to the molten alloy followed by three stages of mechanical stirring (300, 600 and 900 rpm for 2-3 mins) in the presence of degassing agent (KCl - NaCl). Finally, the reinforcement treated stirred mixture is then poured into preheated mould, allowed to solidify and cool under

ambient atmosphere. The reinforcement treatment with molten alloy is varied from 0 - 9wt.% in increments of 1.5.

The ascast-A6082 and developed MMCs are subjected to T6 heat treatment process after cleaning them thoroughly with the acetone to remove the surface contaminations, if any. At first, the specimens are heated at 480°C for 4 hrs. Once the heating is completed the specimens are immediately quenched in to water and then again heated to 185°C for 8 hrs, followed by furnace cooling. Once the specimens complete the heat treatment cycle, they are subjected to machining to obtain the samples for experimentation according to ASTM standards. Casted specimens are designated as, as-cast A6082-UT (Untreated), as-cast A6082-T6 (T6-Treated), MMCs-UT (Untreated) and MMCs-T6 (T6-Treated). Complete process is shown in figure 1(a)-(f).

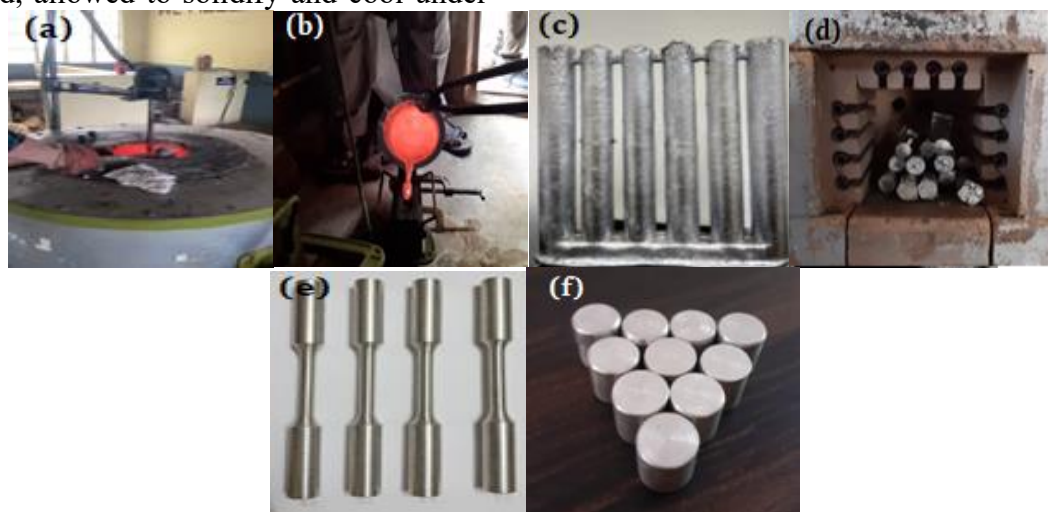


Fig 1 (a) Stir casting setup (b) Pouring of mixture in to moulds (c) Fabricated composite specimens (d) Heat treatment (e) Tensile test specimens (f) Hardness & Density test specimens

3. Experimentation

3.1 Microstructural analysis: Microstructural analysis is carried using optical microscope. SEM with EDS is used to analyse the composition in the samples. The specimen preparation for microstructure analysis is carried out following standard metallographic procedures (Fig 2). Keller's etchant is used to etch the specimen for analysis.



Fig 2: Specimen for microstructural analysis

3.2 Density measurement: The density of the as-cast A6082 & developed MMCs are determined using water displacement method based on Archimedes' principle and using following formula.

Density = Weight of sample in air / (Weight of the sample in Air – Weight of sample in water) x ρ_{water} (gm/cm³)

3.3 Hardness and Tensile test: To determine the hardness of the fabricated samples (fig 3), a Micro-Vickers hardness

tester is employed, utilizing a diamond indenter. The specimens are prepared in accordance with the ASTM E384 standard. All the test samples polished to a mirror-like surface finish. The indenter is applied to the surface of the specimen with a predetermined load of 1kgf is applied on specimen surface for 10 sec of and dwell time and the resulting hardness values are recorded in Vickers Hardness Number (VHN).



Fig 3: Specimens for density and hardness measurement

Tensile test evaluation is performed using computerized UTM on the specimens (fig 4) prepared using composites as per ASTM E8 standard.



Fig 4: Tensile test specimens

4. Results and Discussion

4.1 Microstructural analysis

The microstructure analysis of the Al6082 alloy prior to treatment (Fig. 5a) reveals a configuration of equiaxed grains, with a noticeable occurrence of pores and grain boundaries. The pores can emerge during the casting process. The grain size in the untreated alloy typically ranges from 50 to 200 microns. Conversely, subjecting the A6082 alloy to T6 heat treatment (Fig. 5b)

resulting in a substantially modified microstructure compared to the untreated A6082 alloy. The heat treatment produces smaller and more uniform grains, with a size of approximately 20 microns. The heat treatment also contributes to the formation of equally spaced grain boundaries and a higher density of dislocations, enhancing the mechanical properties of the treated alloy.

The heat treatment also promotes the precipitation of Mg₂Si particles, which

contribute to the strengthening of the alloy. These particles are formed by the reaction of magnesium with silicon, which are common alloying element in A6082. During the solution treatment stage, the magnesium, silicon and other solute atoms dissolve in the aluminium matrix to form single phase homogeneous solid solution. This homogeneous solid solution with

dissolved atoms will freeze upon quenching in water at atmospheric temperature. The artificial aging process allows precipitation of magnesium and silicon atoms, later start to cluster together, forming minute particles of Mg_2Si . These particles to grow and become more numerous, resulting in a significant increase in strength and hardness of the alloy.

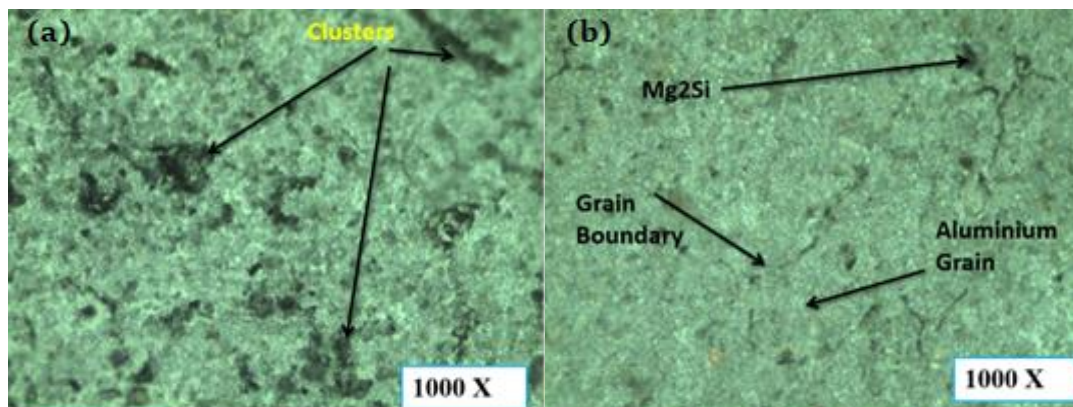


Fig 5: Optical microstructures of (a) untreated as cast A6082 alloy and (b) T6 heat treated (480°C for 4Hrs, and 185°C for 8 Hrs) as-cast A6082 alloy

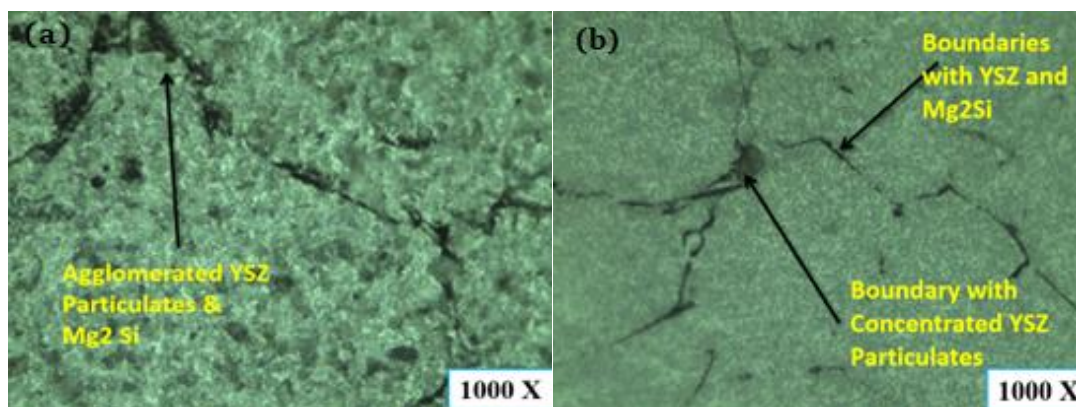


Fig 6: Optical microstructures of (a) untreated MMCs with 3% YSZ and (b) T6 heat treated (480°C for 4Hrs, and 185°C for 8 Hrs) MMCs with 3% YSZ

Untreated MMCs with 3wt.% YSZ typically have a heterogeneous microstructure (fig 6 a) with the YSZ particles dispersed throughout the metal matrix. The optical microstructure of the untreated MMCs shows that the YSZ particles tend to cluster and agglomerate in certain regions, leading to the formation of local hotspots. These hotspots can result in increased wear, erosion and failure of the material under high stress or strain conditions. In contrast, the optical

microstructure of T6 heat treated MMCs with 3% YSZ (fig 6 b) shows more even distribution of the YSZ particles throughout the matrix and at grain boundaries, with minimal agglomerations. The artificial aging process allows the formation of Mg_2Si particles, which tend to concentrate at the boundaries between the YSZ particles and the metal matrix. These Mg_2Si particles act as pinning sites for dislocations and help to strengthen the material.

Untreated MMCs with 6% YSZ (fig 7 a) often exhibit larger agglomerations of the YSZ particles, which gives way to the formation of pores and voids during processing. T6 heat treated MMCs with 6% YSZ (fig 7 b) have a more homogeneous microstructure, with Mg_2Si particles

concentrated at the boundaries between the YSZ particles and the metal matrix. However, care must be taken during polishing, as the agglomerated YSZ particles can easily chip off and leave behind surface pores.

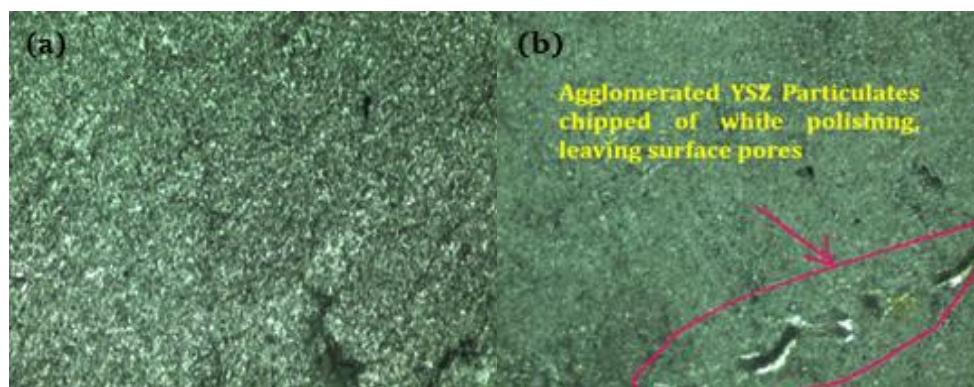


Fig 7: Optical microstructures of (a) untreated MMCs with 6% YSZ and (b) T6 heat treated (480⁰C for 4Hrs, and 185⁰C for 8 Hrs) MMCs with 6% YSZ

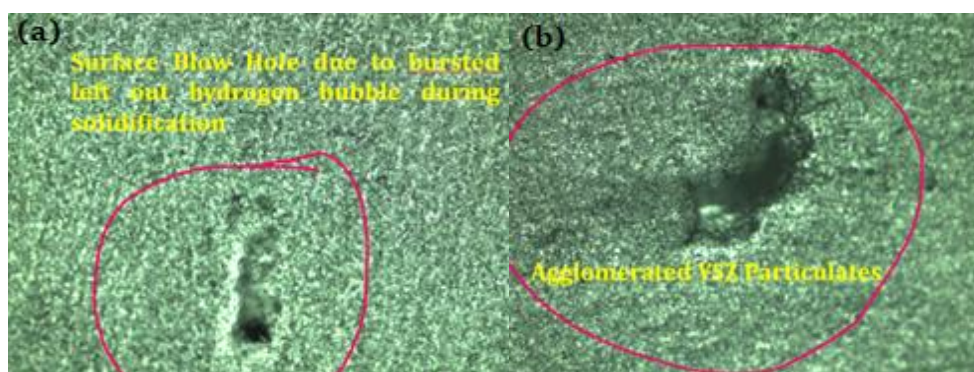


Fig 8: Optical microstructures of (a) untreated MMCs with 9% YSZ and (b) T6 heat treated (480⁰C for 4Hrs, and 185⁰C for 8 Hrs) MMCs with 9% YSZ

Untreated MMCs with 9% YSZ often exhibit a heterogeneous microstructure (fig 8 a) with larger agglomerations of the YSZ particles. The surface blow hole appeared in one of the specimen may be due to left-out hydrogen bubbles during solidification. Hydrogen gas, during solidification, can become trapped in the molten metal matrix. During the cooling and solidification of the metal, hydrogen gas may become trapped and create bubbles. These bubbles can eventually burst on the surface of the material, resulting in surface blow holes. The presence of YSZ particles within the metal matrix can intensify this issue, as the particles can serve as locations for the

initiation of gas bubble formation. This can lead to the formation of larger and more frequent blow holes in the MMCs. As per the observation it is evident that, many specimens are free from these blow holes due to the effective de-gassing of mixture.

T6 heat treated MMCs with 9% YSZ have a more homogeneous microstructure (fig 8 b) with Mg_2Si particles concentrated at the boundaries between the YSZ particles and the metal matrix, leading to improved strength and wear resistance. However, agglomerated YSZ particulates can still chip off during polishing, leaving behind surface pores.

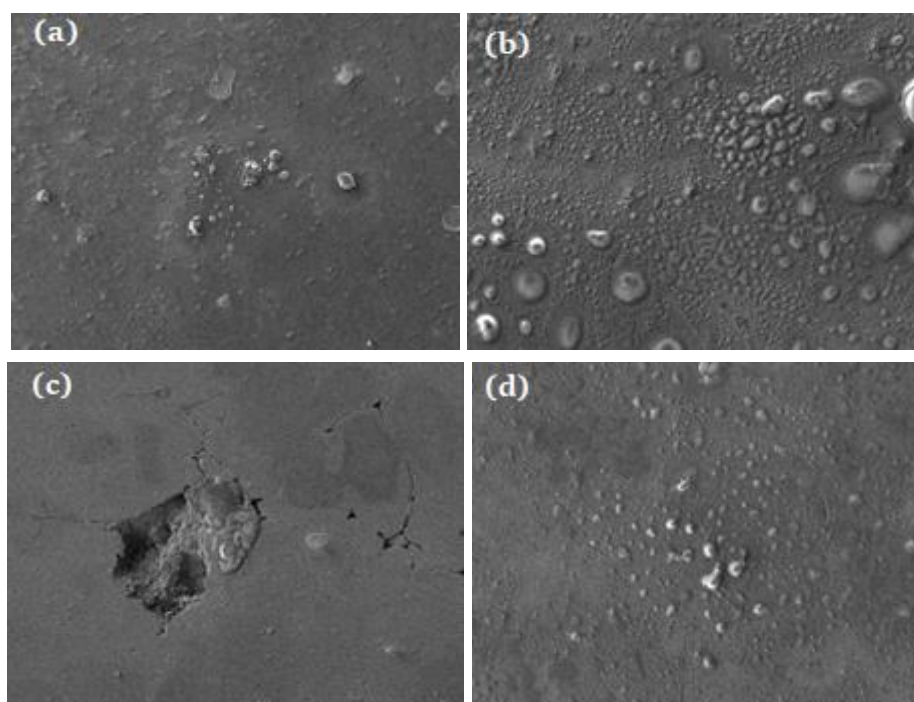


Fig 9: SEM images of (a) 0%, (b) 3%, (c) 6% and (d) 9% YSZ reinforced MMCs

Microstructural variations are visible in SEM pictures of A6082 MMCs reinforced with 0%, 3%, 6%, and 9% YSZ. The A6082 aluminium matrix dominates the microstructure of the 0% YSZ sample, and the aluminium grains are distributed uniformly throughout. There donot seem to be any secondary phases or particles floating around on the surface. There are discernible shifts in microstructure as YSZ content rises. Tiny YSZ particles may be seen floating around in the MMC with 3% YSZ since it is mixed with aluminium. These particles stand out from the background the same way the aluminium granules do, although their contrast is somewhat different. Although some agglomeration may exist, YSZ particles appear to be spread out very evenly. Greater YSZ particle concentrations are seen in the 6% YSZ MMC. The SEM picture shows a greater concentration of YSZ particles, and the particles appear to be clustered or agglomerated in certain places. There appears to be strong interfacial adhesion between the YSZ particles and the aluminium matrix. There is a dramatic increase in the density and size of YSZ

particles in the microstructure of the 9% YSZ MMC. The SEM picture shows that there are more YSZ particles present than in earlier mixtures. At this greater YSZ level, attaining a perfectly homogenous distribution of particulates becomes a bit problematic, since some clogging or agglomeration of YSZ particles may be more noticeable. The YSZ particles are still very evenly distributed in the aluminium matrix and show strong interfacial adhesion.

The SEM pictures show that the YSZ particles were successfully incorporated into the A6082 MMCs. The composite's mechanical and physical characteristics can be drastically altered when the density of the particles increases with increasing YSZ content. Microstructural evidence suggests that YSZ particles added to an A6082 matrix can significantly alter the matrix's microstructure, potentially improving the material's hardness, wear resistance, and thermal resilience.

4.2 Density and Hardness measurements

Figure 10 shows that when the fraction of YSZ added to the A6082 alloy increases,

the density values are seen to raise marginally. Several things have contributed to this. To begin, YSZ particles are denser than the A6082 alloy matrix in which they are embedded. As a result, a higher YSZ concentration results in a denser composite material. Second, when the percentage of YSZ particles in the composite material rises, the material's average density rises with it. This is because the YSZ particles' extra mass adds to the total mass of the material, making it denser as a result. In addition, YSZ particles can have a beneficial effect on packing density. Due to their lower size, YSZ particles are able to fill the voids between the bigger A6082 alloy matrix grains. As a result, the material's density increases as the packing density improves.

In the case of MMCs, the little boost in density can have various benefits. To begin with, it aids in the enhancement of the composite's mechanical characteristics. A greater load-carrying capacity and resistance to deformation may result from a material with a higher density. This is especially useful in contexts where dimensions and structural stability are paramount. The composite's thermal conductivity may also rise as a result of the density boost. The addition of YSZ particles can increase the MMC's thermal conductivity since YSZ has a greater thermal conductivity than the aluminium alloy matrix. This has potential use in thermal management systems and heat exchangers, where effective heat dissipation is essential. In addition, the MMCs' increased density can improve their resistance to wear and prolong their useful life. In addition to increasing the material's resistance to wear, fatigue, and other types of mechanical deterioration, a higher density can also contribute to a denser and more compact microstructure.

Hardness levels are raised when YSZ particles are mixed into the as-cast A6082 alloy (figure 11). Several things have contributed to this. To begin, the

incorporation of YSZ particles into the aluminium matrix results in a hardening effect by reducing the mobility of dislocations. The density of strengthening particles in the matrix increases as the fraction of YSZ particulates rises from 3% to 9%, leading to a comparable rise in hardness. This explains the 15.39%, 58.78%, and 60.18% increases in hardness found for the MMCs with 3%, 6%, and 9% YSZ additions. Figure 11 shows how the hardness values of as-cast A6082 alloy and MMCs rise after T6 heat treatment compared to their untreated form. There is an increase of 70.57% in the as cast alloy compared to its untreated counterpart. Heat treated MMCs had a hardness increase of 4.61%, 6.42%, and 15.58%, respectively, compared to the heat treated as cast alloy. After being subjected to a solution heat treatment, the YSZ particles and any other solute atoms are incorporated into the aluminium matrix to create a uniform material. This method is useful for alleviating any residual stresses or flaws.

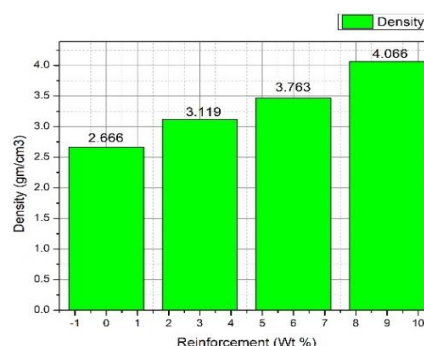


Fig 10: Variation of density values with the variation of wt.% of YSZ

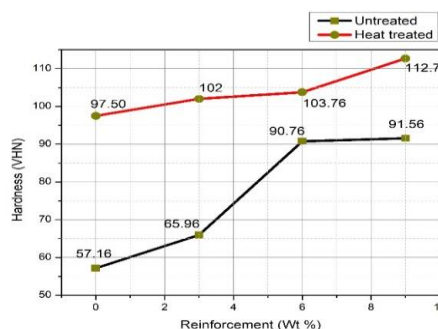


Fig 11: Variation of hardness with respect to the variation of wt. % of YSZ

Fine precipitates (θ' , theta prime - Al-Mg-Si phase) develop during artificial ageing inside the aluminium matrix, particularly at the locations of YSZ particles. The particles in this phase are quite small and are scattered everywhere over the aluminium matrix. The material is strengthened and hardened due to the creation of the θ' phase. Coherence with the aluminium matrix means that the θ' precipitates are well aligned with the aluminium atoms around them. By making it more challenging for dislocations to propagate in the material, this coherent structure aids in the strengthening action and adds to the rise in hardness. T6 heat treatment also helps disperse YSZ particles evenly throughout the matrix, which reduces the likelihood of particle clustering and boosts the material's overall hardness. The hardness levels of both A6082 alloy and MMCs seen to be increased after being subjected to both YSZ addition and T6 heat treatment.

4.3 Tensile Measurements

The ultimate tensile strength (UTS) for the composites with 3wt.% and 6wt.% addition of YSZ shows gradual increase by 112.68% as the percentage of YSZ increases (ref fig 12). This strengthening effect can be attributed to the presence of YSZ particles, which hinder dislocation movement and increase the strength of the material. The observed increase in UTS up to 6wt.% YSZ is in line with previous studies on the effect of ceramic particles on the mechanical properties of metal matrix composites. However, the decrease in UTS at 9wt.% addition of YSZ may be due to the presence of agglomerated particles or other microstructural defects that can act as stress concentrators and lead to premature failure of the material. This decrease in UTS at 9wt.% YSZ is consistent with findings from other studies on the effect of excessive particle addition on the mechanical properties of metal matrix composites.

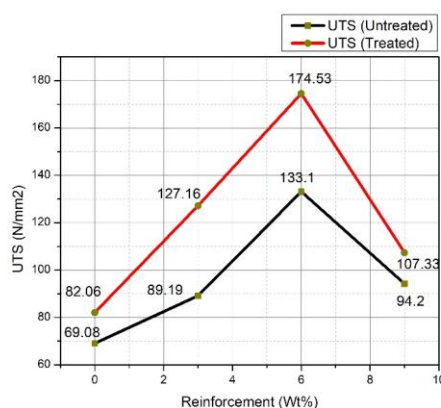


Fig 12: Ultimate strength for different compositions for both untreated and heat treated MMCs

The trend observed for yield strength of the composites with different YSZ compositions is shown in Figure 13. The yield strength increases up to 6wt.% YSZ addition by 116.5% in the untreated MMCs. However, for the 9wt.% addition, the yield strength decreases. Similar results were observed for the heat-treated MMCs also, where the yield strength increases up to 6wt.% addition by 95.46% and then decreases. The decrease in yield strength with excessive particle addition is a common phenomenon in metal matrix composites. The movement of dislocations and the presence of defects like porosity and voids influence the yield strength of the material. The addition of ceramic particles can lead to an increase in these defects, causing a decrease in the yield strength. The heat-treated MMCs show a greater UTS compared to untreated MMCs due to the formation of a fine and evenly distributed precipitate phase during the controlled heating and cooling cycles. This phase acts as a strengthening agent and hinders dislocation movement, leading to an increase in the material's strength. Moreover, the heat treatment process promotes the recrystallization and grain growth of the material, resulting in a more uniform and finer grain structure, reducing the likelihood of stress concentrations and crack initiation sites. The addition of YSZ particles further reinforces the matrix,

acting as barriers to dislocation movement and enhancing the material's strength.

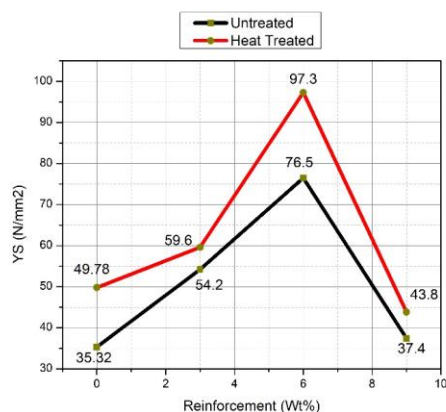


Fig 13: Yield strength for different compositions for both untreated and heat treated MMCs

It can be observed that the values of 0.2%YS for 0%, 3%, 6% and 9% addition of YSZ show a non-linear trend with increasing YSZ content. This non-linear behaviour is expected because the strengthening effect of the YSZ particles is not proportional to their concentration in the material. Instead, there is an optimum concentration of YSZ particles that provides the maximum strengthening effect, beyond which the development of agglomerates can lead to a decrease in yield strength.

5. Conclusion

Incorporating YSZ in A6082 alloy has proven to be advantageous in several aspects. Firstly, it results in a refined microstructure with YSZ particles dispersed uniformly throughout the matrix, leading to substantial increase in hardness in both untreated and heat-treated composites. The rise in density and hardness is proportional to the amount of YSZ added to the alloy, up to a certain limit beyond which it leads to a decrease in strength due to the agglomeration of YSZ particles.

Additionally, adding YSZ has been observed to enhance the ultimate tensile

strength (UTS) of both untreated and heat-treated composites, up to a certain concentration of YSZ (up to 6wt.%). This improvement in UTS is attributed to YSZ particles acting as reinforcing agents, thereby enhancing the load-bearing capacity of the composite material. However, beyond a certain concentration (9wt.% in this case), the UTS decreases due to the clogging of YSZ particulates, leading to a reduction in strength.

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