Microstructure Evaluation of Si$_3$N$_4$ reinforced Al6082 Composites subjected to Severe Plastic Deformation

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Abstract: - In this research, work is carried out on Al6082 alloy because of its thermal stability and stiffness which can be used for strong structural application, here the hard ceramic particle of Si$_3$N$_4$ are reinforced with Al6082 for further enhancing its strength, stiffness. Stir casting technique is used to fabricate aluminum metal matrix composition with varying wt. % of Si$_3$N$_4$, this technique involves cost effective process and also results in homogenous mixture between the matrix and the reinforcement. The percentage of reinforcement varies from 0 and 9wt%. When subjected to severe plastic deformation it tends to change the grain size from micron to submicron level size in the grain structure by the application of load. The scanning electron microscope reveals the distribution of Si$_3$N$_4$ on the surface of the sample. The hardness value increased as compared to as- cast Al6082 alloy.

Keywords: Al6082, Si$_3$N$_4$, Severe plastic deformation, Hardness, Stir casting;

1. Introduction

Metal grid composites (MMCs) are developing consistently. Metal is used as a matrix in this, whereas ceramic or other organic compounds are used as reinforcements. Aluminum, magnesium and titanium are light weight and are also commonly used as a matrix material [1- 3]. Ceramic reinforced composites are discovered to be quite popular materials with the end goal of basic applications because of its phenomenal properties such as high quality, good malleability, durability and high modulus; which is the product of the interaction of its constituent process, i.e., ceramic reinforcement and matrix metal [4–5]. Form of the composite strengthening and manufacturing process plays a significant role in affecting the composites mechanical and physical properties [6]. Reinforcements may be in the form of fibre, particulate or whiskers. An appropriate collection of reinforcement and matrix and the strengthening form is extremely valuable for improving the property of the composites [7–9]. Due to its high strength-to-weight ratio, ease to produce, and low cost of production, aluminum and its alloys have become the most commonly used non-ferrous metals in recent years and have become more common [10,11,12]. Composites of aluminum metal matrix (AMMCs) strengthened with ceramic particles are quick and effortless to process and have the ability to provide customized combinations of properties [13]. These unique combinations of properties make composites more encouraging for aviations and vehicle applications [14-16]. High hardness and low thermal expansion coefficient developments are some of the basic properties of ceramic reinforcements that offer ascent to immediate and indirect reinforcement of the ultimate composite of aluminum [17, 18, and 19]. Components of automobiles that operate under friction and temperature conditions are produced by hard ceramic composites and are commonly used to produce AMMCs for this SiC, B$_4$C, Al$_2$O$_3$, Si$_3$N$_4$ [20-27].

From the literature, the nitride ceramic reinforcement (AIN, Si$_3$N$_4$) are favorable materials for the production of AMMCs because they have good heat conductivity, specific modulus, low density and high temperature stability. Researchers also led to many methods of fabricating AMMCs, such as
stirring, mechanical alloying and pinch casting. However, in an effort to yield products that are less cost effective and mass produced, stirring is the furthermost popular procedure used by numerous researchers. Many researchers recognized the introduction of ceramic particles to develop mechanical characteristics. Ceramic reinforced with matrix material is the most common method of generating MMC.

Through the last few years, studies have been based on the efforts on enhancing the thickness of the grain material using different SPD methods. Such as (i) Equal Channel Angular Pressing (ECAP), (ii) Accumulative Roll Bonding (ARB), (iii) Large Strain Hot Rolling (LSHR), (iv) High-Pressure Torsion (HPT), (v) Extrusion and (vi) Constrain groove pressing (CGP) and others [31, 32]. Among these Constrain groove pressing is one of the effective severe plastic deformation process which is mainly used for producing an ultra fine grin structure throughout shape and size of the specimen. The whole method of Constrain groove pressing (CGP) the material is mounted in a symmetrically formed groove and then smoothed by the load operation. It is necessary to replicate the cycle in order to achieve the broad effective strain essential to refine the rough grain structure. By contrast to the commonly employed ECAP method for structural grinding, the benefit of the CGP procedure is that immense plastic deformation can be applied to metal by shape of sheet or plate. The Hall Petch effect is measured to critically enhance the properties of metallic materials by abating grain size [28, 29]. Besides, the reduction in grain size it produces a super plastic resistance at elevated temperatures [30]. Usually, SPD activates at higher compressive load to obtain ultra grain pre arrangement with a high-angle grain size. The variation of grain size can be applied to produce products with the vital properties. Physical and mechanical properties will expressively help from a reduction in grain size. The evolution and character of the new interfaces appears as a very significant property when studying microstructure in SPD materials as regards evaluating their effect on the mechanical properties. Given the deformation processing state, the heterogeneity in the formation of microstructures was often observed across the bulk specimen depending on the strain introduced [33, 34]. This dissertation examines the processing problems in anticipating commercialization attempts. Next, a set of major SPD processes will be presented (i.e. constrain groove pressing). Later on in the article, the contribution of highly deformed aluminum to microstructural evolution and hardness improvement is addressed in short.

2. Experimental details

2.1 Material

The material used was Aluminum AA6082 alloy of purity 96.55% with composition as shown in Table II. Silicon nitride (Si₃N₄) reinforcement involves 50nm size of powder particles; the purity of silicon nitride particles is 99.7%. The silicon nitride powders were pre heated to obtain homogenous mixing before adding it into the molten mixture. Chemical composition and physical properties of Al6082 is shown in table I and table II respectively. In the present study, the addition level of Si₃N₄ is selected as 0 and 9 wt% to know the effect of higher weight fraction of the Si₃N₄ on microstructural characterization and mechanical properties of produced composites (Al6082-Si₃N₄).

| Table 1. Chemical composition of Al6082 |
|----------------------------------------|
| Element | Si | Fe | Cu | Mn | Mg | Zn | Ti | Cr | Al |
|% present | 0.7-1.3 | 0.0-0.5 | 0.0-0.1 | 0.4-1.0 | 0.6-1.2 | 0.0-0.2 | 0.0-0.1 | 0.0-0.25 | Balance |

| Table 2. Physical properties of Al6082 |
|----------------------------------------|
| Properties | Density | Melting point | Thermal Expansion | Modulus of Elasticity |
| Value | 2.71 g/cm³ | 555°C | 24 x10⁻⁶ /K | 70 GPa |

2.2 Method

Aluminum 6082 metal block is first sliced into small parts and carefully washed to eliminate salt, dirt, etc. so that the slag can be minimized. Such aluminum strips are placed inside a crucible and placed in a furnace. A 350g 6082 aluminum alloy sample was melted using a 6kW electric furnace. The ceramic materials were then preheated to 250°C to increase the wettability and remove the gases deposits on
the surface of the particles. Pre-heating of the reinforcement improves the wettability of the heated aluminum alloy and the reinforced substance while retaining the temperature of the mix, reducing the surface impurities and gasses involved with the agglomeration of the metal. The casting obtain by stirring process is one of the cost effective process. To prevent the mixing of the molten metal with iron, the stirrer used must be a stainless steel stirrer covered with zirconium. The AA6082 was charged to a graphite crucible, furnace is heated at 750°C. The preheated Si$_3$N$_4$ was supplied to the molten metal and stirred with a mechanical stirrer rotating at a speed of 300 rpm to generate a complete vortex. Si$_3$N$_4$ is now slowly added in to the fine vortex created by mechanical stirrer. The duration of the stirring was 5min and the surface of the furnace is again tightly closed and waited till the temperature reaches to 750°C and then the fluid is permitted to pass around the edge of the die. Finally, the melt is poured into a rectangular cavity (70mm x 35mm x 50 mm) container. Table III demonstrates the process parameters used in the present studies to produce the composites.

![Figure 1. (a) Molten metal poured into the mould cavity. (b) Specimen obtains from die.](image)

**Table 3. Process parameter of Stir casting**

| Process parameter | Stirring temperature | Stirring speed | Stirring time | Preheat temperature of reinforced particles | Preheat temperature of Permanent method |
|-------------------|----------------------|----------------|---------------|---------------------------------------------|----------------------------------------|
| Value             | 750°C                | 200rpm         | 5min          | 250°C                                       | 250°C                                  |

2.3 Steps involved in SPD (Constrain groove pressing)

The composite samples has prepared by using stir casting technique and the same samples is subjected to severe plastic deformation using flat and corrugated as shown in fig. 2(a) with the application of the load (50KN and 60KN) for which the material plastically deform and again the plastically deform specimen is placed between the flat die and the same amount of load is applied (50KN and 60KN) to obtain fine grain structure throughout the volume of the material. Fig 2(b) demonstrates deformed specimen with the help of corrugated die and fig 2(c) demonstrates deformed specimen under flat die.
Figure 2. (a) Severe plastic deformation die (i.e., flat and corrugated die). (b) Sample deformed under corrugated die. (c) Sample deformed under flat die.

3. Results and Discussions

3.1 Microstructural studies
Olympus microscope is used to examine the microstructure. To get the microstructure of the material, the material should be polished by using different size of grit papers (example 200, 400, 600, 800, 1000, 1200 and 2000). It is then polished with velvet cloth to get a mirror finish. Before placing the samples in the Olympus microscope the sample should be thermally etched. It is then placed in between the lenses to get the micrograph of the required samples by using elevating screw while focusing on the sample.

Figure 3. (a) As-cast aluminium (b) Al6082 reinforced with 9wt % Si₃N₄.

3.2 SEM analysis
Scanning electron microscope (SEM) reveals the distribution of silicon nitride powder in a metal matrix composition. The microscope reveals the microstructure of each polished specimen at different location on the surface of the specimen and the image shows proper distribution of Si₃N₄ on the surface of the metal. The particles are clearly visible on every increasing magnification in the SEM (example 100X, 500X 1000X, 1500X).
SEM image shows the microstructure on the surface of the specimen. Figure 4 (a) shows the as-cast aluminum alloy without severe plastic deformation, the grain size remains itself because the material doesn’t undergo any deformation so the length of the grains size will be standard. In figure (b) the same as-cast aluminum alloy under goes a sever plastic deformation, here the grain size increased as compared to as-cast aluminum alloy, the high compressive load (50KN) rubs the grains on the surface of the material and tends to increase grain length. Figure (c) reveals micrograph of the 6082Al alloy reinforced with 9wt% of Si$_3$N$_4$. The material will not undergo any deformation, while the white particles represent the Si$_3$N$_4$ particles on the surface of the specimen. In figure (d) the reinforced material undergoes a severe plastic deformation, which increases the length of the grain size under the load of 50KN.

3.3 Evaluation of Microvickers hardness
The Microvickers hardness was measured on a flat surface of the specimen. Variation of hardness values were observed by increasing Si$_3$N$_4$ in Al6082 alloy, it clearly shows that increasing wt. % of reinforcement in matrix material tends to increase the hardness value. The test was carried out with a specific load of 0.2HV for every specimen. The homogeneous distribution of Si$_3$N$_4$ led to increasing hardness values in the composites materials. Many factors are affected by the hardness value like particle size, percentage reinforcement, and the method of preparation of composites.
Figure 5. Microvickers hardness value of Al6082 with 9w.t% Si₃N₄

Table 4. Microvickers hardness values

|                | Without SPD | With SPD |
|----------------|-------------|----------|
| As-cast        | 76.65       | 87       |
| 9wt%           | 92.6        | 98.5     |

4. Conclusions
Microstructure evaluation of Al6082 alloy reinforced with Si₃N₄ particles subjected to severe plastic deformation led to the following conclusion:

1. Aluminum MMC with 0 and 9w.t% of Si₃N₄ is successfully developed by automated stir casting technique.
2. Olympus microscope revealed the microstructure of Al6082 as-cast alloy and 9w.t% Si₃N₄ reinforced Al6082 composite with and without severe plastic deformation.
3. SEM micrographs revealed the distribution of Si₃N₄ in the Al6082 matrix. The micrographs revealed the grain refinement process formed due to severe plastic deformation.
4. The hardness of as-cast Al6082 alloy without carrying out SPD is 79.65VHN while the as-cast Al6082 alloy that was subjected to SPD is 87VHN. The composite with 9w.t% Si₃N₄ reinforcement without being subjected to SPD has a hardness value of 92.5VHN while the same composite that under went through SPD could achieve a hardness of 98.5VHN.

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