Synthesis and Characterization of Ultrasonically Treated Al/Al$_2$O$_3$ Nanocomposites

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Abstract. Aluminium-Alumina (Al/ Al$_2$O$_3$) nanocomposites are synthesized for different amounts of reinforcement through liquid route. Ultrasonic treatment (UST) is carried out during the solidification of the melt. It is expected that the addition of nanoparticles to the melt and UST provides grain refinement for the specimens due to increased number of nucleation sites and cavitation effects respectively. The effects of UST on these nanocomposites are studied using the results obtained from optical microscopy, hardness and XRD. Reduction of porosity and grain refinement is observed in the synthesized nanocomposites.

Keywords: Ultrasonic treatment, grain refinement, nanocomposites

1. Introduction
Aluminium and aluminium alloys owing to their low density and high strength to weight ratio finds numerous applications in automobile and aerospace industries. Mechanical properties of these alloys can be further improved by reinforcing them with nanoparticles, called nanocomposites. Various methods have been developed for synthesizing aluminium matrix nanocomposites out of which the liquid route using stir casting is considered economical [1]. Ultrasonic treatment (UST) in aluminium alloy melts is known to yield degassing effect and grain refinement. These effects are due to cavitation generated by the high frequency ultrasound wave [2].

Ultrasound treatment in the synthesis of nanocomposites can disperse the particulate reinforcements uniformly. Introduction of nanoparticles to the melt increases heterogeneous nucleation sites leading to grain refinement [3][4]. The objective of this study is to investigate the effect of alumina nanoparticle addition in pure aluminium for ultrasonically treated melts.

2. Experimental Methods
Specimens of Al/Al$_2$O$_3$ nanocomposites are synthesised with aluminium as the matrix and alumina nanoparticle as the reinforcement. Alumina nanoparticle used for this study is purchased from Sigma Aldrich, Bangalore and is of 50 nm average size. Specimens prepared are i) aluminium, ii) aluminium with 2% alumina nanoparticle and iii) aluminium with 3% alumina nanoparticle, with ultrasonic treatment for all the three specimens during solidification of the melt. These are then characterized
using optical microscopy, Vicker’s hardness and X-ray diffraction. Details of the specimens used for characterization are given in Table 1.

**Table 1.** Details of specimens used for characterization

| Specimen No | Details                               |
|-------------|---------------------------------------|
| S1          | As received                           |
| S2          | With UST and no reinforcement         |
| S3          | With UST and 2% reinforcement         |
| S4          | With UST and 3% reinforcement         |

To synthesize the nanocomposite specimens following method is used. Pure aluminium is melted on a table top furnace in a clay bonded graphite crucible. Melt is heated up to 740°C and alumina nanoparticle is added. The mixture is mechanically stirred for 10 minutes and is poured into a steel mould. The steel mould is then placed on the ultrasonic agitator and ultrasonic wave is propagated through the mould till the composite melt solidifies. Experiments are conducted with ultrasound frequency of 40 KHz. The schematic diagram of the experimental setup is shown in Figure 1.

**Figure 1.** Experimental Setup for the synthesis of nanocomposite specimens

### 3. Results and Discussion

#### 3.1 Optical Microscopy Analysis

The microstructures of the specimens prepared are given in figures 2 and 3. As seen from the microstructures it is evident that significant grain refinement has happened for specimens which are ultrasonically treated.

The average grain sizes of the specimens are determined using the line intercept method and is given in figure 4. The average grain size of the as-received specimen is greater than 70 µm. For specimen S2 the average grain size is found to be < 50 µm. For specimen S3 also the average grain size is found to be < 50 µm. And for specimen S4 the average grain size is around 20 µm. It is also observed that in comparison with the as received specimen there is significant reduction of porosity. This can be attributed to the degassing effect that the UST has [1].
Figures 2. Microstructure of the a) as-received specimen, b) specimen with UST and no reinforcement

Figures 3. Microstructure of the specimen a) with UST and 2% alumina b) with UST and 3% alumina

All ultrasonically treated specimens are found to have non-dendritic equiaxed grains, which is consistent with the results of earlier studies [4]. It is also observed that a bimodal distribution of grain sizes exists in the UST specimens, which improves the ductility of the material [5].

Figure 4. Comparison of grain size for specimens
A significant reduction in grain size is observed in the specimen S4. This is due to the fact that adding 3% by weight alumina nanoparticle to the melt and ultrasonic treatment has increased the number of nucleation sites there by decreasing the grain size of the material.

### 3.3 X-ray Diffraction Studies

It has been observed from our XRD studies that the crystallographic texture strongly depends on the percentage of $\text{Al}_2\text{O}_3$ in the composite. For specimen S4 where the percentage of $\text{Al}_2\text{O}_3$ is 3%, a double texture of (111) and (311) is observed whereas the other samples with no alumina and 2% alumina shows a strong (111) texture [6] as shown in figure 5.

![XRD Plots for the specimens](image)

**Figure 5.** XRD Plots for the specimens

### 3.3 Hardness Testing

The hardness values of the ultrasonically treated specimens are given in figure 6. It is observed that the hardness values increase as the percentage of reinforcement in the composite increases. The increase in hardness values can be attributed to two main factors; one is the strengthening effect due to
reinforcements and the second is the decrease in grain size. Both act as hindrances to dislocation motion there by strengthening the composite [7].

![Figure 6](image)

**Figure 6.** Comparison of hardness for specimens

4. Conclusions
UST aluminium and Al/Al₂O₃ nanocomposites were successfully synthesised. It was found that UST decreases the grain size and gives a equiaxed non-dendritic structure. Significant grain refinement was observed in Al /3% Al₂O₃ nanocomposite with ultrasonic treatment during solidification. Another observation was the reduction in porosity of the ultrasonically treated specimen. The crystallographic texture was found to be dependent on the percentage of alumina nanoparticles in the composite. It is also observed that the addition of nanoparticles and ultrasonic treatment resulted in the increase of hardness values.

References
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