A comparative assessment of crystallite size and lattice strain in differently cast A356 aluminium alloy

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Abstract. In this investigation, A356 aluminium alloy has been prepared by different routes viz. gravity casting, rheo pressure die casting (RPDC) and RPDC with T6 heat treatment. X-ray diffraction studies of these samples have been done in the scanning range of 20 – 90°. X-ray peak broadening analysis has been used to estimate the crystallite size and lattice strain, in all the samples. The sample prepared by RPDC with T6 treatment has comparatively smaller crystallite size and lesser lattice strain than gravity cast and RPDC samples.

1. Introduction
A356 aluminium alloys (Al–7Si–Mg) are finding increased use in variety of structural, automotive, aerospace, and engineering applications due to its excellent cast-ability, corrosion resistance, mechanical properties and especially high strength to weight ratio [1-3]. However, the new technology for fabrication of this alloy and the methods of enhancing their properties are the subject of many investigations. Particle size and crystal morphology play important roles in various applications of this alloy, which have driven researchers to focus on the fabrication techniques, type of modification done and heat treatments etc. Analysis of X-ray diffraction peaks has become a very powerful tool for characterization of microstructure of materials either in bulk or in powder form. For the calculation of the crystallite size and the lattice strain form the broadening of X-ray peaks, the Williamson–Hall method was most commonly used.

No material has a perfect crystal structure due to their finite size causing a deviation from perfect crystallinity which leads to broadening of the X-ray diffraction peaks [4]. Broadening of x-ray diffraction peaks is easily apparent in patterns obtained with a diffractometer, and this information can be directly quantified. However, it is important to realize that broadening of diffraction peaks arises mainly due to three factors viz. instrumental effects, crystallite size and lattice strain. The broadening...
is evaluated by measuring the angular width “B”, in radians, at intensity equal to half the maximum intensity (FWHM). Subtracting the instrumental effect from the obtained peak broadening, two main properties; crystallite size and lattice strain are extracted from peak width analysis. Crystallite size and lattice strain affect the Bragg peak by increasing the peak width and intensity and shift the 2θ peak position accordingly [4]. The crystallite size varies as $1/\cos \theta$ and stain varies as $\tan \theta$ from the peak width. The size and strain effects on peak broadening are known from the above difference of 2θ [5]. W-H analysis is an integral breadth method. Size-induced and strain-induced broadenings are known by considering the peak width as a function of 2θ [6].
In this study, the crystallite size and lattice strain are estimated by using a well known Williamson and Hall method for A356 aluminium alloy fabricated by gravity cast and rheo pressure die cast technique. Also rheo pressure die cast samples with T6 heat treatment was taken into consideration in this analysis.

2. Experimental details

2.1. Material and its fabrication technique
The material selected for this investigation is A356 aluminium alloy. The alloy is prepared by two different casting techniques i.e. gravity casting and rheo pressure die casting (RPDC). The dimension of each cast was 15×15×150 mm$^3$. The method of gravity casting is similar to popularly used techniques available for it; a method is described by Arunkumar et al. [7]. Rheo pressure die casting is one of the semi solid casting techniques. In this process, aluminium alloy is melted in a melting furnace which is then allowed to pass through a cooling slope to obtain semi-solid slurry (solids fraction varies from 12-20% ) of the alloy. This semi-solid slurry is collected in a holding furnace which stores the slurry iso-thermally (slurry temperature at the exit of the cooling slope). Then, some quantity of slurry is taken from holding furnace by the ladling unit and poured in the sleeve of the pressure die casting machine (350T - model DC 350J-MS). Temperature of about 300°C was maintained in mould halves and sleeve through circulating hot oil to avoid premature solidification. By applying a pressure the molten alloy is forced into the mould halves and allowed for solidification. A part of the RPDC samples are subjected to T6 heat treatment process. In this, the samples are solution treated at 540°C for 30 minute followed by a water quench (25°C); then artificially aged at 180°C for 6 hrs followed by water quenching.

2.2. Microstructure and X-ray diffraction
The optical micrographs of mechanically polished samples are examined using ZEISS optical microscope at different magnification levels. To estimate the crystallite size, lattice strain etc. X-ray diffraction studies of the sample are done using an X-Ray Diffraction system (Make/Model: RIGAKU JAPAN/ULTIMA-IV) with scanning rate of 5° per minute and step size of 0.05 in the scanning range of 20-90°. Cu Kα radiation is used for this.

3. Results and discussion

3.1. Microstructural analysis
The optical microstructures of the samples of gravity cast, RPDC and RPDC with T6 treatment are illustrated in figure 1(a) to 1(c) respectively. In all the images, primary alpha aluminium is present which is surrounded by eutectic silicon. In case of gravity cast sample, typical elongated dendritic shape of primary alpha phases are distributed randomly throughout the sample, whereas in case of RPDC sample, nearly equi-axed primary alpha phase was observed which are distributed uniformly throughout the matrix. Such variation in the microstructure by varying casting technique was also reported by Tahamtan et al. [8]. The microstructure of the RPDC with T6 is indeed different from RPDC as the eutectic silicon is getting rounded of due to T6 treatment. As there is an irregular shape
of primary alpha phase is present in the gravity cast sample, the aspect ratio of the phase was calculated and presented in Table 1 for all the samples. It can be noted from the table that aspect ratio of primary alpha phase decreases from gravity cast to rheo cast sample. After heat treatment of RPDC samples, no much variation in aspect ratio was found.

3.2. Estimation of crystallite size and lattice strain

In order to determine crystallite size and lattice strain of the casts, X-ray diffraction profile analyses was done for all the casts. Intensity vs. $2\theta$ plots of the investigated samples are presented in figure 2. This figure illustrates all the detectable peaks with their respective hkl values for the investigated samples. All these detectable peaks could be indexed as cubic structure found in the standard reference data (for Al: JCPDS 01–1180 and for Si: JCPDS 03–0544). Similar peaks for Al and Si at approximately same $2\theta$ values are also reported by Chen and Alpas [9]. One can note from these figures that there is little shifting of peaks in RPDC sample towards lower $2\theta$ value from the gravity cast sample. In contrast, the T6 heat treated RPDC sample shows peak shifting towards higher $2\theta$ value as those of the RPDC sample. The obtained peaks of Mg were of very low intensities; thus not presented in the given X–ray diffraction plots for all three conditions; significantly low wt.% of Mg in all the casts may be the reason for not getting these peaks.

| Sample                        | Aspect ratio |
|-------------------------------|--------------|
| A356 Al alloy: Gravity cast   | 1.38 ± 0.50  |
| A356 Al alloy: RPDC           | 0.98 ± 0.28  |
| A356 Al alloy: RPDC + T6      | 1.08 ± 0.18  |

The crystallite size and lattice strain can be calculated from the broadening of X-ray diffraction peaks by considering the full width at half maximum (FWHM) of the all individual peaks. If the observed X-ray peak has broadening of width $B_o$ and the width due to instrumental effect is $B_i$, then the remaining broadening of the peak due to
crystallite size and lattice strain is $B_r$, which can be expressed considering Gaussian profile as [6]:

$$B_r^2 = B_o^2 - B_i^2$$ (1)

Many methods are available in the literature to estimate crystallite size and lattice strain. Out of those methods, Williamson and Hall [10] is one that is most commonly used. According to this, the particle size and strain contribution to the line broadening are independent to each other and both have a Cauchy-like profile; the observed line breadth is simply the sum of the breadth due to crystallite size and lattice strain [11].

$$B_{hkl} = B_{\text{crystallite}} + B_{\text{strain}}$$ (2)

The well known Scherrer equation [12] for determination of crystallite size from broadening of X-ray diffraction peaks is expressed as $B_{\text{crystalline}} = (k \lambda / L \cos \theta)$ where $\lambda$ is the wavelength of the X-rays used, $\theta$ is the Bragg angle, $L$ is the average crystallite size measured in a direction perpendicular to the surface of the specimen, and $k$ is a constant ($k = 0.94$ for small cubic crystal). The broadening due to lattice strain in the material can be represented by the relationship $B_{\text{strain}} = 4 \eta \tan \theta$, where $\eta$ is the strain in the material. Hence we can write that

$$B_{hkl} = \frac{k \lambda}{L \cos \theta} + 4 \eta \tan \theta$$ (3)

Rearranging equation (3) we get

$$B_{hkl} \cos \theta = \frac{k \lambda}{L} + 4 \eta \sin \theta$$ (4)

Williamson and Hall plots for different samples are shown in figure 3 (a) to (c). A linear fit of the scattered results is taken into consideration. From this linear fit, the crystallite size was estimated from the y-intercept, and the strain, from the slope of the fit.

The crystallite size and lattice stain is calculated by comparing the linear fit equations with equation (6) for three different samples are presented in Table 2. One can note from this table that the lattice strain developed in A356 alloy prepared by RPDC technique is more than the gravity cast technique. This is attributed to the application of pressure in RPDC samples which may cause some lattice structure variation; resulting increased lattice strain in the material. Lattice strain is a measure of the distribution of lattice constants, such as lattice dislocations, which arise from crystal imperfections [4]. In order to minimize this strain, the rheo cast samples are subjected to T6 heat treatment. The heat treated samples has comparatively smaller crystallite size and lesser lattice strain than gravity cast and rheo cast samples.
Figure 3. Williamson and Hall plots for A356 aluminium alloy: (a) gravity cast, (b) rheo pressure die cast and (c) rheo pressure die cast with T6 heat treatment.

Table 2: Crystallite size and lattice strain of different samples of A356 aluminium alloy.

| Samples                | Williamson and Hall method |
|------------------------|-----------------------------|
|                        | Crystallite size (L) in nm  | Lattice strain ( X 10^-3) |
| A356: Gravity cast     | 50.88                       | 0.33                      |
| A356: RPDC             | 71.27                       | 0.80                      |
| A356: RPDC + T6        | 41.38                       | 0.15                      |

4. Conclusions

Microstructure and X-ray diffraction have been studied for the samples of A356 aluminium alloy fabricated by gravity cast, rheo cast and rheo cast with T6 treatment. The following conclusions can be resolved from this investigation:

1. The micrographs show that, in gravity cast samples, the primary alpha phase have elongated shapes with some dendrite shapes found randomly. The microstructure of RPDC with T6 is indeed different from RPDC; as the eutectic silicon is getting rounded of due to T6 treatment. However, the aspect ratio of the primary alpha in RPDC and RPDC with T6 don’t have significant variation.
2. The broadening due to crystallite size and lattice strain was analyzed by Williamson and Hall method. Both crystallite size and lattice strain developed in the sample prepared by RPDC are more compared to the sample prepared by gravity cast. However, with application of T6 heat treatment after RPDC, samples have comparatively smaller crystallite size and lesser lattice strain than gravity cast and RPDC samples.

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