ICOTRIME 2020
IOP Conf. Series: Materials Science and Engineering
1104 (2021) 012023 doi:10.1088/1757-899X/1104/1/012023

Mechanical Characterization & Phase Evaluation of ODS Aluminum Alloy

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Abstract. Nano-oxide dispersed aluminum alloys are widely used as structural components in different fields in aerospace, defence, transport and automobile industries. It is observed that the hardness of these Aluminium alloys (65-100 HV) could be significantly enhanced by uniform dispersion of nanometric Y2O3 and TiO2 in high-Al matrix in presence of other alloying elements like Fe and Ti. In the present work, synthesis of Al (70%), 22 Fe (22 %), Ti (8 %), Al (69 %), Fe (22 %), Ti (8 %) with 1% nano-Y2O3 and Al (69%) , Fe (22%), Ti(8%) with 1% nano-TiO2 dispersed particles through mechanical alloying has been carried out. These alloys have been named as alloy A, B & C respectively on the basis of their weight percentages. Subsequent consolidation of this alloy has been carried out through conventional sintering at 1000°C for 1 hour. X-ray diffraction study (XRD), field emission scanning electron microscopy (FESEM) has been used to analyse the phases evolved during various stages of milling. The crystallite size and lattice strain were analyzed by Williamson-Hall method and finally, the structures and Phases are correlated. Sharp peaks with high intensity reveal the presence of amorphous and crystalline phases. For alloy B, intermetallic peaks like Al3Fe, and Al3Ti, Y2O3 and TiO2 for alloy C has been observed after sintering.

Keywords: XRD, FESEM, Phase evolution, intermetallic, sintering, crystallite size and lattice strain

1. Introduction
Al based alloys find its application in many areas like defence, aerospace, transport and automobile sector. The desirable properties including reduced density, low cost, and low elastic modulus makes Al alloys better to use. Al alloys also find its application on the basis of some other important properties like elastic limit, strength to weight ratio, corrosion & wear [1].

As compared to structural material (steel), Aluminum is sixty percent lighter and has superior strength to weight ratio. Both impact toughness and corrosion resistance behaviour can be possessed by Al in normal atmospheric condition. They can be used because of their better thermal and electrical conductivity. The method of fabrication also makes aluminium superior to use. In order to fulfil the requirements and challenges like greater efficiency, reduced emissions and environmental impact of the aerospace industries, the performance & characteristics of these materials must be improved [2].
Alloying of elements is one of the most common methods to enhance the strength of aluminium properties of materials. Factors such as formability, cost, reusable-ability, increased toughness, and easy availability are considered while selection of alloying element especially for dynamic structure. In Today’s world, Both Al and Mg alloys are widely used for structural materials in surface transportation and aviation. Mechanical alloying assumes further significance owing to the scope of developing heterogeneous microstructures comprising amorphous and nano crystalline phases either by controlled amorphization (during milling) or by subsequent partial crystallization of the amorphous precursor (by annealing), and thereby, attaining a novel composite microstructure with superior mechanical properties[3,4]. The Mechanical strength of Al-alloys can be enhanced by a heat treatment process called aged hardening. Yield strength of structural Al alloys can reach up to 600 MPa [3] by age hardening heat treatment process[5]. The general procedures to produce Al-based alloys are rapid quenching such as melt spinning[6,7] or with the help of mechanical alloying [8] but these processes restrict the sample size to slightly thin robins or in the shape of powder. Hence it is required to synthesize amorphous based bulk samples. It is very difficult for alloying elements to combine through traditional melting method . Hence it can be done with the help of mechanical alloying [9]. Intermetallics possess outstanding elevated temperature properties such as high strength with increased stiffness, better corrosion /oxidation resistance. These properties can be observed because of its systematic & ordered nature of existence. Reduced dislocation motion and low diffusivities are the two important attributes of intermetallics. Intermetallic compounds generally show brittle characteristics and having high melting point.

Oxide dispersion strengthening (ODS) alloys are produced by solid state and other powder processing techniques. One such example is Mechanical alloying (MA). The constituents of powder can be in the primary, intermetallic or pre alloyed state. Both the metallic and oxide (Y_2O_3 or TiO_2) powders are mixed together and mechanically alloyed by using a high energy ball milling.

The repeated impacts during the process like cold welding, fracture, and re-welding of mixed powder resulting in a very fine and homogenized mixture of constituents. The shearing action leads to the growth of new fragmented alloyed particles.

2.0 Materials and Method

2.1Raw Material

Elemental powders of aluminium (Al), iron (Fe), titanium (Ti), oxides of yttrium and titanium oxide (Y_2O_3 & TiO_2) are used to prepare the desired alloys. These can be tabulated as shown in Table1.

| Alloy  | Al (wt. %) | Fe (wt. %) | Ti (wt. %) | Y_2O_3 (wt. %) | TiO_2 (wt. %) |
|--------|------------|------------|------------|----------------|---------------|
| Alloy A | 70         | 22         | 8          | -              | -             |
| Alloy B | 69         | 22         | 8          | -              | 1             |
| Alloy C | 69         | 22         | 8          | 1              | -             |

Table 1: Composition (initial) of the powder mix for mechanical alloying/milling

2.2 Mechanical Alloying and Process variables:

In the present study, alloy A, B and C were subjected to high energy ball milling in a multivial Fritsch Pulverisette planetary ball mill .The ball mill consists of a hardened steel container of maximum capacity (500 ml) & steel balls having diameter 10 mm. For proper mixing, the ball to powder ratio was kept 10:1 in terms of weight. Milling operation was carried out at 300 rpm with subsequent time interval of 30 minutes. Prior to milling, Samples were taken out after a time interval of 0h, 10h, 20h, 30h, 40 and 50 h for characterization (FESEM and XRD). The various milling parameters considered in study are mentioned below as shown in table 2.
| Parameter                  | operational condition |
|----------------------------|-----------------------|
| Types of mill             | Planetary mill        |
| Milling container         | Chrome steel          |
| Milling speed             | 300 rpm               |
| Grinding media            | Toluene               |
| Ball diameter             | 10mm                  |
| Ball to powder weight ratio | 10:1                |
| Time of milling           | 50hr                  |

Table 2: Milling process parameter

2.3 Compaction and Sintering

The compaction experiments were executed to make a cylindrical sample. In order to make Al based alloy specimen, cylindrical punch and die made of stainless steel was used. Compacts of 15 mm diameter were made with the punch and die. Maximum of 450MPa pressure of load was applied on it very slowly. Compacts were cast out from the die in the same direction as the compression.

The process of sintering is carried out at high temperature (1000°C) but below the melting of the materials being sintered. The actual value of sintering temperature of most of the materials ranges between 70-80% of their melting temperature. In order to produce a pore free body densification of the material is done which eliminates the pores because of neck formation.

2.4 X-Ray Diffraction (XRD) study

In order to find the phases present in Aluminium alloy X-pert MPD X-ray diffractometer is used. The purpose of XRD study is to identify the different phases and its evolution associated with different milling hours. Phase evaluation can also be analysed after sintering processes. The variation in peaks and correlation with the standard specimen were analyzed with the help of (JCPDS) data file. Additional software was used to identify the crystal structure constituent phases. X-ray diffraction were recorded in the scanning range from 30° to 90° with an accelerating voltage of 40kV using Cu Ka (λ=1.542Å) and current of 35 mA. Estimation of crystallite size and lattice strain was approximated by using Williamson-Hall Plot.

2.5 Field emission scanning electron microscope (FESEM):

In order to investigate the polished surface of Al-based alloys pellets specimen were carried out field emission scanning electron microscope

3.0 Result and Discussion

3.1 Phase Evolution of Alloys during Mechanical Alloying:

Milling of Al (70%), 22 Fe (22 %), Ti (8 %), alloy A powders at different stages of milling time in hrs (0- 50hrs) can be seen through the series of XRD pattern (figure-1). During manual blending for 0 h, only the peaks of Aluminium, iron and Titanium were observed. Furthermore, as the milling time progresses, presence of high intensity peaks or sharp peaks can be observed. Broadening in XRD peak reveals that the powders are disjointed, cold weld and rewelded. This leads to the formation of amorphous/ nanocrystalline phases. Shifting & Formation of new intermetallic (Al₃Fe, andAl₃Ti)
peaks can be seen after 30 h of milling. This is due to the reaction of stoichiometric ratio of all the metallic elements (Fe, Al & Ti) in course of milling.

3.2 Phase Evolution of Alloys after Sintering:

Fig. 2 shows the XRD pattern of alloy A, B and C respectively. Sintered samples at 1000°C for 1 hour in an inert (Ar) atmosphere were investigated. It reveals that the presence of FCC-Al peak is predominant peak and also some intermetallic peaks like Iron aluminide (Al<sub>3</sub>Fe) and Al<sub>3</sub>Ti, Yttrium oxide (Y<sub>2</sub>O<sub>3</sub>) for alloy B and Titanium oxide (TiO<sub>2</sub>) for alloy C respectively.

![XRD peaks of sintered compacts of alloy A, alloy B and alloy C.](image1)

![Figure 1: XRD peaks of alloyed powders of Al (70%), 22 Fe (22 %), Ti (8 %) (alloy A) at various milling time (0- 50hrs).](image2)

![Figure 2: XRD patterns of sintered products of alloy A, alloy B and alloy C.](image3)
Fig. 3: FESEM micrographs of sintered pellets of alloy B and alloy C respectively.

Fig. 4 shows the dispersion of nano-Titanium oxide ($\text{TiO}_2$) in aluminum matrix of alloy C. The dispersoids and intermetallic phases improve the creep resistance property of the aluminum alloy.

3.4 Determination of Crystallite Size and Lattice strain

Crystallite size and lattice strain of milled powders (0 h, 10 h, 20 h, 30h, 40h and 50 h) were determined with the help of William-Hall equation. As per earlier discussion, the XRD peaks get broaden (Fig. 1) during milling this is due to the fact that various factors such as instrumental error, crystallite size and micro strain have been influenced during high energy ball milling. The broadening of peaks was used to determine the particle size and residual strain of the material. This broadening is because of the small crystal size and was evaluated through Scherrer’s formula (Eq. .1).

The crystallite size is calculated by Scherrer’s formula as follows:

$$d = \frac{0.9 \lambda}{\beta_g \cos \theta} \Rightarrow \beta_g = \frac{0.9 \lambda}{d \cos \theta} \quad (1)$$

Where, $d =$ crystallite size; $\lambda =$ the wavelength of the X-ray (0.1541nm); $\beta_g =$ Full width half maximum intensity (FWHM and $\theta =$ angle of diffraction.)
The lattice strain is calculated by the given formula:

$$\eta = \frac{\beta_s}{4\tan\theta}$$  \hspace{1cm} (2)

$$\Rightarrow \beta_s = 4\eta\tan\theta$$  \hspace{1cm} (3)

Where, $\eta$ = Lattice strain; $\beta_s$ = Full width half maximum intensity (FWHM) due to micro strain and $\theta$ = angle of Diffraction

$$\beta\cos\theta = \frac{6\lambda}{d} + 4\eta\sin\theta$$  \hspace{1cm} (4)

We can calculate the crystallite size and residual strain with the help of Equation (4) called Williamson-Hall equation.

| Milling Time | Crystallite Size (nm) | Lattice strain(%) |
|--------------|-----------------------|-------------------|
| 0 h          | 247                   | 0.15              |
| 10 h         | 230                   | 0.18              |
| 20 h         | 182                   | 0.23              |
| 30 h         | 78                    | 0.89              |
| 40 h         | 62                    | 1.23              |
| 50 h         | 32                    | 1.32              |

Table 3: Summary of crystallite size and lattice strain of alloy A: calculated by William –Hall equation at various stages of milling (0- 50 hrs).

It is found (Table-3) that there is a gradual decrement in crystallite size from 247 to 30 nm as milling time increases and residual strain increases from 0.15 to 1.32 (%) as milling time increases. A plot between crystallite size and lattice strain vs milling time has been shown in figure.5. It can be observed from the graph that with an increase in milling time the crystallite size decreases. Also there is an increase in lattice strain with increase in milling time.
The present study investigates the characterization of ball-milled Aluminium alloy powder under various process parameters. Evolution of phases through XRD techniques and effect of milling on various Al based alloys (A, B and C) have been examined. The subsequent key findings obtained from the experimental results are mentioned. XRD pattern of different alloy with successive time of milling were investigated. FESEM micrographs of sintered pellets confirm that the average grain size of alloy B and alloy C was 249.67 nm and 510.84 nm respectively. For alloy C, dispersion of nano-TiO$_2$ in aluminum matrix is observed. These dispersoids and intermetallic phases improve the creep resistance property of the aluminum alloy. The analysis of particle size revealed that there is a decrement in particle ranges from 55 μm to 5 μm during 50 h of milling. The variation of crystallite size with milling time is linearly independent. Also there is an increase in lattice strain with increase in milling time. During manual mixing of elemental powders, XRD peaks of only Al, Fe & Ti were observed. With an increase in milling time from 0hr to 50 hrs, pointed and elevated XRD pattern were observed. The lengthening and sharpening of XRD pattern confers that the elemental powders are disjointed, cold weld and rewelded which leads to the formation of amorphous and nano crystalline phases. Shifting and formation of new XRD pattern as intermetallics phases (Al$_3$Fe and Al$_3$Ti) can be observed after 30 hrs of milling.

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