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Thermo-mechanical responses of nanocrystalline Al–Fe alloy processed using mechanical alloying and high frequency heat induction sintering

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ABSTRACT

In the current investigation, a nanocrystalline alloy Al-10 wt.% Fe was synthesized from metallic powders using the mechanical alloying (MA) technique, for various milling hours. The consolidation and sintering of the alloyed powders was performed in a high frequency induction heat sintering (HFIHS) machine. The minimum crystallite size and the maximum hardness of the sintered sample was found to be 30 nm and 2.05 GPa, respectively. The maximum compressive yield strength of the alloy was observed to be 660 MPa at room temperature. The bulk nanocrystalline alloy produced from 150 h milled powder showed significant enhancement in the thermal stability, this specific alloy displayed a compressive yield strength of 570 MPa at 573 K. The compression experimental results of sintered samples revealed high strength coupled with large deformation to failure.

1. Introduction

The demand for aluminum and its alloys has been ever increasing due to their high strength to weight ratio. It is well known that through precipitation hardening mechanisms the strength of certain aluminum alloys at room temperature varies between 550–600 MPa [1]. However, a significant decrease in their strength values with an increase in temperature ($>$ 423 K) has been reported in an earlier investigation [2]. The instantaneous drop in the strength level of the aluminum alloy from room to high temperature has resulted in the limited use of the alloys, especially in high temperature applications. To address this limitation, significant number of investigations were performed, of which few suggested modifications in the manufacturing processes to be implemented in the traditional/or existing processes. A few others suggested alloying of aluminum with transition metals (TM). This suggestion was based to the observations that aluminum when alloyed with TM results in the formation of intermetallics /or secondary phases. These phases tend to provide additional stability to the evolved microstructure and inherently increase the resistance of the alloy against fracture, at higher temperatures [1].

Based on this observation, few investigations have been performed wherein; aluminum was alloyed with TMs for various applications [3–5]. Of the various Al-TMs alloys, Al–Fe alloys is of significant interest owing to the low diffusivity of Fe in aluminum [1,6–9]. The presence of Fe in aluminum matrix results in the formation of stable microstructure of the processed alloy. Fe forms secondary phases with aluminum including their super saturated solution. The microstructural stability of Al–Fe alloys even at elevated temperatures is mainly attributed to the formation of the secondary phases. Thus, it is highly desirable to increase the Fe content in aluminum matrix. However, there exists a solubility limit of Fe in Al matrix if traditional processing methods are employed. The maximum solubility limit of Fe in Al matrix is observed to be less than 0.03 at.% even at elevated temperatures [1] using traditional processing techniques. To enhance the mechanical and physical properties of the Al alloys at elevated temperatures, it is imperative to increase the alloying content of Fe in Al beyond its solubility limit of 0.03 at.%. Due to the limitation of the traditional processing methods, new techniques such as mechanical alloying/milling (MA) [10–13] and rapid solidification (RS) [14,15] can be used. These techniques if carefully implemented tend to increase the solubility limit of Fe in Al matrix. The MA technique is also referred to as non-equilibrium processing technique wherein the alloying process takes place in non-equilibrium condition. Processing of alloys using MA technique not only result in extending the solid solubility limit of Fe in Al matrix but also result in refining the microstructure to ultrafine /or nanometer level with homogeneous dispersion of oxides and intermetallics. Since the alloys produced using these technique consists of higher

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content of Fe and refined microstructure, it is expected that the processed alloy shows improved mechanical and physical properties compared to its traditional counterparts [16]. There exists several parameters that define the morphology of final processed powders using MA technique. These parameters include the time and type of mill, milling atmosphere, ball to powder weight ratio, grinding medium and process control agents [13]. MA takes place in a high energy ball mill wherein the powder particles are subjected repetitive welding, fracturing, and re-welding of powder particles resulting in fine particles within a fine grained matrix due to high energy impact [16].

The current study was performed to investigate two aspects. Firstly, to increase the solid solubility limit of TM (Fe) in Al matrix and secondly, to investigate the mechanical properties of the processed alloy at elevated temperatures. The current investigation further explores the synergistic effect of increasing the alloying time and the reduction in the crystallite size of the processed alloy, on the thermo-mechanical properties of the sintered alloy which has not been fully explored in earlier investigations.

2. Experimental procedure

2.1. Production of ultrafine/nanocrystalline Al -10%Fe alloys

The initial powders used in this investigation include; 99.95% pure Al powders (average particle size equal to 2 μm) and 99.95% pure Fe powders (average particle size equal to 3 μm). In order to remove any traces of moisture present in the initial powders, the mixture (consisting of Al-10 wt.% Fe powder) was degassed in vacuum oven at 373 K for 24 h. The degassed mixture was charged into the milling containers along with steel balls of 15 mm diameter in a glove box under inert atmosphere. The number of balls in each container was selected so that the ball-to-powder weight ratio (BPR) of 10:1 was maintained. The MA of charged powders was performed in a pulverisette-P5 planetary ball mill with a milling speed of 120 rpm. To understand the effect of milling time on the severity of alloying, the milling times performed in this investigation include 10, 30, 70, 100 and 150 h. To avoid excessive heating of powders each milling cycle consisted of 15 min of milling alternated with 15 min of pause time. Processing of powders using MA could lead to a significant agglomeration of powder particles due to the repeated cold welding, fracture and re-welding [13]. Thus, to inhibit the agglomeration of the powder particles during alloying, various process control agents (PCA) are normally used [12,17,18]. In the current investigation, 1 wt.% stearic acid was used as a process control agent (PCA). After milling, the milled mixture was charged into a graphite mold in a glove box maintained under inert atmosphere. To ensure inert atmosphere while transferring the mold from glove box to sintering machine, the top and bottom surface of the charged powder was covered with a thin layer of graphite powder. The powders in the mold were then consolidated and sintered in a HFIHS machine under vacuum to form bulk nanocrystalline alloy. The rate of heating and the sintering temperature were set to 823 K/minute and 823 K, respectively. Once the desired temperature of 823 K was reached, the sintering was performed for an additional 6 minutes, while maintaining a constant pressure of 50 MPa. The density of the sintered bulk nanocrystalline alloy was measured using Sartorius density measurement kit and the bulk density of the sintered alloy was found to be 2.84 g/cm^3.

2.2. Vickers microhardness

The Vickers microhardness measurements were performed on the sintered Al-10 wt.% Fe alloy obtained from milled powders for various milling times, using Buehler microhardness tester. The load for the indentation was set to 100 g. The surface of the samples were polished using sand papers of different grits. The different grit sizes used range from 220 to 4000. The final polishing was performed using a colloidal silica solution. The measurements were performed at an interval of 0.25 mm along the sample diameter.

2.3. Compression experiments at room temperature

The quasi-static uniaxial room temperature compression experiments were performed on the sintered Al-10 wt.% Fe alloy samples, at a constant engineering strain-rate of $10^{-2}\,s^{-1}$ on an Instron material testing system. The typical specimen dimensions include 13 mm length and 9 mm diameter. To maintain a constant engineering strain-rate during the experiment, the samples were experimented in the displacement-controlled mode. A uniaxial high elongation strain gage manufactured by Kyowa was bonded on the sample surface. The strain gage was connected to P3500 strain indicator manufactured by Vishay micro measurements. The reading obtained from strain indicator was later corrected using calibration factor to obtain strain. The stress was calculated from the load values obtained from load transducer. It is well known that the friction plays a significant role in the compression experiments. To negate the friction effects, the interface between the surface's of the test specimen and machine grips was lubricated with a combination of a Teflon sheet of 0.3 mm thickness and Molycote grease.

2.4. Quasi static compression experiments at different temperatures

To investigate the effect of temperature on the mechanical responses of sintered Al-10 wt.% Fe alloy, the samples were subjected to compressive loading at a strain-rate of $10^{-2}\,s^{-1}$ and at temperatures of 373 K, 473 K and 573 K. The displacement from the machine transducer was corrected for the machine compliance. The corrected displacement data was used to calculate the strain. High temperature grease manufactured by Dow Corning was used as a lubricant. The thermocouple (Type J) mounted on the surface of the specimen provided the temperature reading. Before performing the experiment, the specimen was heated to the desired temperature and held at that temperature for additional 15 minutes to obtain the uniformity of the temperature throughout the specimen.

2.5. X-Ray diffraction

The surfaces of newly fabricated nanocrystalline Al-10 wt.% Fe bulk sample was mirror polished using various grit sand papers starting with a coarse paper and ending with fine polishing using colloidal silica solution. The polished surface of the sample was characterized on a Discover D8 diffractometer operating in the θ–θ geometry using a standard Cu-K_α (λ=0.154 nm) radiation. The sample surface was scanned at a rate of 5° per minute for a 2θ value between 30 and 50 degree. This 2θ range was sufficient enough to record the first two peaks of Al. The maximum intensity peak obtained from (1 1 1) diffraction plane of Al was used to calculate the average grain (crystallite) size using Debye-Scherrer's [19] equation given by

$$D = \frac{K\lambda}{B\,\mathrm{Cos}\,\theta} \tag{1}$$

where, D, K, λ, B and θ represents the crystallite size, shape factor (assumed to be 0.9), wavelength (0.154 nm) of Cu-K_α radiation, full width at half maximum (in radians) and the peak position, respectively.