Effect of consolidation and sintering parameters on the mechanical responses of nanocrystalline Al-Fe alloy processed by Mechanical Alloying

Muneer Baig[a], Hany Rizk Ammar[b], Asiful Hossain Seikh[a],

Mohammad Asif Alam[a], Jabair Ali Mohammed[a]

[a]Advanced Manufacturing Institute - CEREM, King Saud University, Riyadh, Saudi Arabia
[b]Metallurgical & Materials Engineering Department, Suez University, Suez, Egypt

E-mail : bmuneer@ksu.edu.sa; Hany_Ammar@uqac.ca, aseikh@ksu.edu.sa, moalam@ksu.edu.sa, jmohammed@ksu.edu.sa

Abstract: In this investigation, bulk ultra-fine grained and nanocrystalline Al-2 wt.% Fe alloy was produced by mechanical alloying (MA). The powder was mechanically milled in an attritor for 3 hours and yielded an average crystal size of ~63 nm. The consolidation and sintering was performed using a high frequency induction sintering (HFIS) machine at a constant pressure of 50 MPa. The prepared bulk samples were subjected to uniaxial compressive loading over wide range of strain rates for large deformation. To evaluate the effect of sintering conditions and testing temperature on the strain rate sensitivity, strain rate jump experiments were performed at high temperature. The strain rate sensitivity of the processed alloy increased with an increase in temperature. The density of the bulk samples were found to be between 95 to 97%. The average Vickers micro hardness was found to be 132 $Hv_{0.1}$.

Introduction

The effect of grain refinement on the mechanical properties of the metallic materials has been an area of significant interest over the last decade. Independent investigations carried by Hall [1] and Petch [2] formed the basis ($\sigma \, \alpha \, d^{-1/2}$) for further studies on reducing the grain size of a polycrystalline materials. Since then, several investigations [3-6] were performed on polycrystalline materials wherein a significant increase in strength with grain refinement was observed. The use of MA for the refinement of coarse-grained powders is based on synthesizing the powders in a state that is far from equilibrium, which is highly desirable for producing nanocrystalline powders. Using MA, alloys can be produced from insoluble elements, which are difficult to process using other traditional methods due to the high possibility of heavy segregation of the alloying elements during solidification from the liquid phase. MA uses a high-energy ball mill in which the metallic powders of different metals are processed by the repeated processes of cohesion and breaking of the coarse-grained powder particles [7, 8]. Mechanical alloying, however, is a complicated process that requires adequate control of several parameters to obtain the desired properties of the final products. These parameters significantly influence the properties of the products: type of mill used, milling speed, milling duration, grinding medium, ratio of the weight of the balls to the weight of the powder used, amount of power present in the container, milling temperature and use of various process control agents. The optimization of the previously mentioned variables is crucial to achieve the desired properties and microstructure of the produced alloys. Several aluminum alloys, such as Al-Mg, Al-Ti and Al-Zr, have already been developed using mechanical alloying. This technique has also been used to develop high-strength Al-Ti alloys for elevated temperature applications by dispersing nano/submicron scale Al_3Ti particles into an AL matrix [8].

The objective of this investigation is to study the effect of consolidation and sintering conditions on the mechanical behavior of Al+2%Fe. The sintering of powders was conducted in a HFIS, wherein; the sintering attributes were the temperatures of sintering 773 and 823K and holding times of 3 and 6 minutes.

Experimental Procedures

The mechanical alloying was conducted in a 1 gallon 304 stainless steel tank in an attritor procured from Union Process, USA. A mixture of Al (5 µm particle size) and Fe (13 µm particle size) powder was degassed at 373K for 24 hours in vacuum before milling. The ball to powder ratio during the alloying was set to 30:1. In addition, 1wt.% stearic acid was added to the powders in the vial at the beginning, as a process control agent to minimize cold welding of particles. The initial alloying speed was set to 100 rpm during which time the stainless steel balls were charged in the attritor followed by the powders and stearic acid, then the valve for argon flow is opened for maintaining an inert atmosphere in the milling vial during the whole attrition period. The rotation of the shaft is then increased to 250 rpm. After 3 hours of milling time, the powder was collected and checked for crystal size by the X ray diffraction using X-ray line broadening techniques proposed by Scherrer [9]. The processed powders were then charged into the graphite molds inside a 2G glove box in an argon atmosphere. Since the sintering process is carried out at high temperature, the powders were confined by a layer of graphite powder at the top and bottom. The powders were consolidated and sintered in a HFIS machine inside the graphite dies under a constant pressure of 50 MPa in a vacuum chamber of 10^{-3} torr. The sintering conditions were varied among sintering temperatures (773- 823K) and sintering times (3 and 6 minutes) with a constant heating rate of 773K/min for all samples. To charactrize the strain rate sensitivity with temperature, quasi-static strain rate jump (from 10^{-4} s^{-1} to $10^{-2}s^{-1}$) high temperature (400K and 500K) experiments we performed on the sintered samples.

Results and Discussion

Fig. 1 shows the true stress – strain response from room temperature simple compression experiment performed on consolidated samples at two different strain rates. These samples were obtained after consolidation and sintering at different sintering conditions. It is observed that sintering conditions of Figs. 1c and 1d resulted in higher yield strength however, Figs. 1a and 1b showed higher elongation to failure. It is observed that the samples exhibited positive strain rate sensiti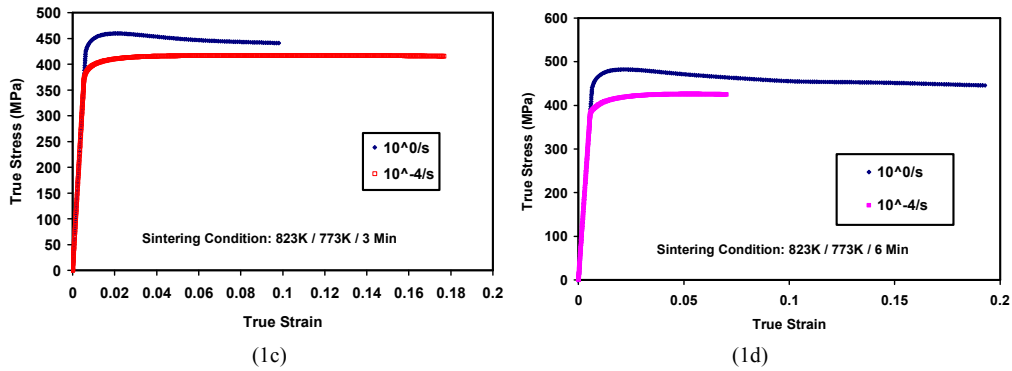vity in all the experimented conditions. Also, it is observed that sintering conditions of Figs. 1c and 1d resulted in higher yield strength however, Figs. 1c and 1d showed higher elongation to failure. It is observed that the samples exhibited positive strain rate sensitivity in all the experimented conditions.

(1a)

(1b)

(1c)   (1d)
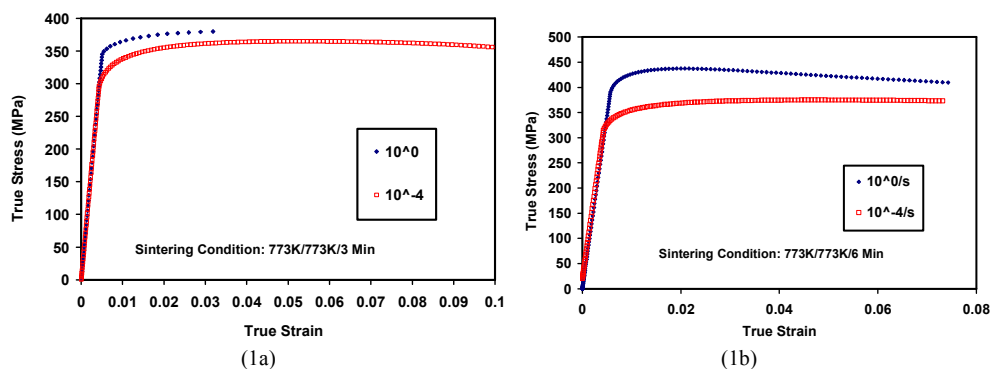
Fig. 1. The room temperature true stress – strain responses of samples obtained using various sintering conditions.
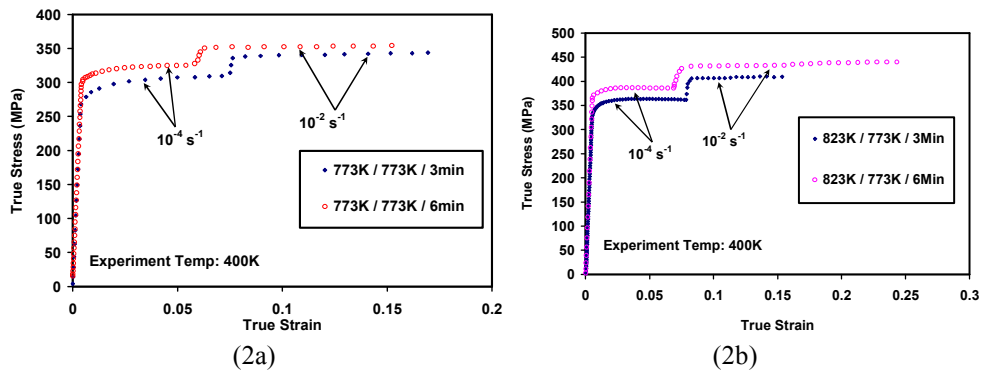

(2a)   (2b)

Fig. 2. The high temperature (400K) true stress – strain responses of samples obtained using various sintering conditions.

Also, it is observed that samples experimented at a low strain rate exhibited higher strain to failure with an exception of Fig. 1d. Fig. 2 shows the true stress – strain response from a strain rate jump performed on consolidated sample at two different strain rates and at a high temperature of 400K. The material exhibited significant positive strain rate sensitivity. Fig. 3a shows the variation of strain rate sensitivity parameter for the four sintering conditions for both testing temperatures of 298 and 400K. It was observed that the sensitivity increased with temperature. Also, the sample sintered at 823K/773K/3 Min exhibited the highest strain rate sensitivity of 0.025. Fig. 3b shows the variation of yield strength with temperature. It is observed that the decrease in yield strength is severe on sample with processing condition 773K/773K/3 minutes and less severe on sample with processing condition 823K/773K/6 minutes.
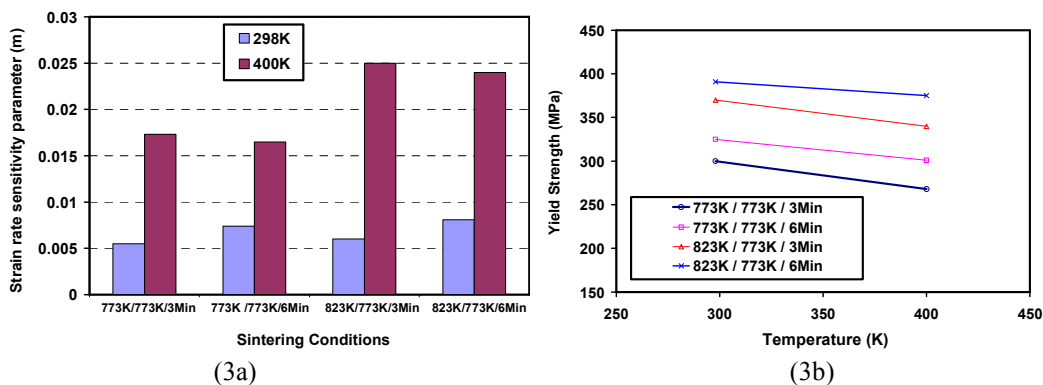

(3a)   (3b)

Fig. 3. Effect of (a) strain rate sensitivity and (b) yield strength with temperature for different sintering conditions.

Table 1

Hardness (HV)	**132 Hv$_{0.1}$**
Crystal Size (nm) after sintering	**102 nm**
Percentage of full density %	**95 to 97 %**

Conclusions

In this investigation, Al-2% Fe alloy was successfully alloyed using MA. The alloyed powder was consolidated using different sintering conditions to achieve significant strength and enhanced ductility. The strain rate sensitivity of the processed alloy increased with an increase in temperature.

Acknowledgement

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