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Effect of chromium and aluminum addition on anisotropic and microstructural characteristics of ball milled nanocrystalline iron

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ABSTRACT

Prior studies on synthesis of nanocrystalline elements have discussed the effect of ball milling on lattice parameter, crystallite size, and micro-strain. For elemental milled powders, the anisotropic peak broadening does not change with increasing milling time. However, the effect of alloying addition on the anisotropic behavior of ball milled nanocrystalline powders remains an unexplored area. Here we report the effect of chromium and aluminum addition on the anisotropic behavior of iron in nanocrystalline Fe −20Cr−5Al (wt%) alloy powders synthesized by ball milling. The experimental results show that the anisotropic behavior of iron changes towards isotropic with milling. This change was also correlated to the theoretically calculated anisotropic factor from the change in elastic constant of iron due to milling. Addition of alloying elements exhibited a monotonic rise in the lattice parameter with crystallite size, which was attributed to the excess grain boundary interfacial energy and excess free volume at grain boundaries. Transmission electron microscopy image confirmed the crystallite size and nature of dislocation obtained using modified Williamson-Hall method.

1. Introduction

In recent years, nanocrystalline materials are being widely investigated due to unique properties like mechanical, physical, and corrosion resistance [1–6]. These materials have been developed by electro-deposition [7], severe plastic deformation [8], alumino–thermic reaction [9], chemical vapor deposition [10], and high-energy ball milling [4,11] process. Among all these techniques, high-energy ball milling is widely employed to synthesize artifact-free bulk nanocrystalline materials [1,2,12–14]. During high-energy ball milling, high impact force is imposed into the powder which induces change in crystallite size and lattice parameter, development of micro-strain, dislocation density, excess free volume and excess grain boundary interfacial energy [1,13,14]. All these structural changes play a significant role during consolidation and also alter physical [15], mechanical [16] and corrosion [4] properties of materials after consolidation.

Nanocrystalline materials, produced by severe plastic deformation such as high-energy ball milling, have shown non-monotonous changes in lattice parameter with crystallite size, an initial lattice contraction followed by a lattice expansion [1,13,14,17]. Qin et al. [13] have proposed that the non-monotonous change in lattice parameter depends on the equilibrium between interfacial stresses developed due to excess grain boundary (GB) interfacial energy and the excess free volume at grain boundaries. The increase in lattice parameter is manifested as a shift in x-ray diffraction (XRD) peak to lower angle, while the reduction in crystallite size and the development of micro-strain during milling are reflected by peak broadening. XRD peak broadening of iron during milling is a non-monotonous function of diffraction angle which is demonstration of anisotropic behavior of iron [2]. Such anisotropic behavior is correlated to the unequal elastic modulus of iron in different crystallographic directions. Accordingly, Williamson-Hall (WH) plot for milled iron powder exhibits

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anisotropic broadening of iron, which does not change with increase of milling time [2]. Synthesis of nanocrystalline elemental iron, nickel, copper, and tungsten has discussed the effect of ball milling on lattice parameter, crystallite size and micro-strain [1,2,13,14,18–20]. However, the effect of alloying addition on anisotropic behavior of ball milled nanocrystalline ferrous system remains an unexplored area.

The effect of chromium and aluminum addition on anisotropic behavior of iron in nanocrystalline Fe–20Cr–5Al (wt%) alloy powder synthesized by ball milling is reported in the present study. The WH technique was used to verify the anisotropic behavior of the alloy whereas the crystallite size was determined by modified Williamson-Hall (MWH) method. The change in lattice parameter of alloy with its crystallite size was calculated and this change was correlated with the excess GB interfacial energy and the excess free volume developed in the alloy at grain boundaries owing to nanocrystallization.

2. Experimental procedure

2.1. Materials and methods

The nanocrystalline Fe–20Cr–5Al alloy powder was prepared using high-energy planetary ball mill (Pulverisette P-5; Fritsch GmbH, Germany). The starting materials, iron powder (99.9% purity and average particle size < 37 μm) and chromium powder (99.9% purity and average particle size < 28 μm) were obtained from Alfa Aesar while aluminum powder (99.9% purity and average particle size < 1.3 μm) was procured from Hunan Jinhao Aluminum Industrial Co. Ltd, China. As-received powders were milled for composition Fe–20Cr–5Al at 250 rpm for durations up to 20 h, using toluene as the inert medium. The ball-to-powder weight ratio was maintained at 10:1. Mill was periodically stopped for 15 min after every 1 h of milling to avoid excessive temperature of the tungsten carbide vessel. Powder samples were collected at 2, 5, 10, 15, and 20 h of milling to characterize the microstructural changes during milling. As received iron powder was annealed at 900 °C for 10 h into a convention tubular furnace (Model No. HCS/HTF/1000/90-900, Supplier: Heat & Control Systems, Mumbai, India) of capacity 1000 °C and the forming gas (95% Ar + 5% H_2) was purged throughout the annealing process. The XRD line broadening of the fully annealed powder was considered as an instrumental broadening. The whole XRD analysis of Fe–20Cr–5Al alloy powder was done after considering this as an instrumental broadening.

2.2. Characterization of milled powder

The microstructural changes in iron at different milling times were determined by XRD, using Rigaku–Smart Lab system (Rigaku Corp, Japan) with Cu Kα radiation (λ = 0.154056 nm), with a step size of 0.01° in 2θ range of 20–120°. HighScore Plus (version 3.0d) [21] was used for multiple peak fitting of XRD patterns of the milled powder using the pseudo-Voigt function [22] and corresponding peak broadening was calculated after considering the instrumental broadening. The extent of peak broadening and change in peak position were analyzed to estimate crystallite size, dislocation density, lattice parameter, change in excess GB interfacial energy, and excess free volume at grain boundaries. Nanocrystalline Fe–20Cr–5Al alloy powder was formed after 20 h of milling that was confirmed using transmission electron microscopy, TEM (JOEL JEM-2100F), and selected area electron diffraction (SAED).

3. Results and discussion

3.1. X-ray diffraction of milled powder

Fig. 1a shows XRD patterns of ball milled Fe–20Cr–5Al powder blends at different stages of milling. It is evident from Fig. 1a that the intensity of major aluminum peak (2θ = 38.47) shows a gradual drop with progress of milling and disappearance by end of 5 h of milling. Such an observation is attributed to the formation of a solid solution of chromium and aluminum with iron. Iron and aluminum have a tendency to form intermetallic phases like FeAl and Fe_3Al during ball milling [23]. However, such intermetallic peaks were not detected in the XRD pattern, plausibly because the aluminum content (~5 wt %) was too low to enable their formation. Careful analysis of the XRD patterns in Fig. 1a also reveals a gradual shift of peak positions toward lower diffraction angle with increase in milling time. The observed peak shift indicates an increase in lattice parameter. The lattice expansion occurs due to dissolution of aluminum and chromium into iron to form an alloy, shown in Fig. 1b. The lattice parameter for Fe–20Cr–5Al alloy after 20 h milling is 0.28851 nm which is comparable with the lattice parameter reported by Capdevila et al. [24].

3.2. Analysis of anisotropic behavior of iron

The grain refinement and development of micro-strain/lattice distortion, due to defects, leads to XRD peak broadening as visible in Fig. 1a. The micro-strain/lattice distortion developed along any crystallographic direction depends on the elastic modulus along that direction [25]. The lattice distortion is favorable along the crystallographic direction with lower elastic modulus as it requires less energy to develop defects [26]. Consequently, in an anisotropic material like Fe, more broadening is expected along the crystallographic direction with lowest elastic modulus.

The elastic modulus of pure iron has identical value of 221 GPa in [100], [112] and [220] crystallographic directions, whereas the elastic moduli in [200] and [310] crystallographic directions are 132 GPa and 154 GPa, respectively [25]. Many authors [2,5,27,28] have reported significant broadening along [200] and [310] directions in iron powder after ball milling. This indicates that the peak broadening in milled iron powder is not a monotonous function of diffraction angle, possibly due to its distinct elastic modulus in different crystallographic directions. The elastic modulus of iron in the crystallographic direction can be reduced after addition of chromium and aluminum, since the elastic constant of iron [25] is higher that of chromium [29] and aluminum [25], shown in Table 1. The elastic modulus (E) of BCC system depends on either stiffness (C_{11}, C_{12}, C_{44}) or compliance (S_{11}, S_{12}, S_{44}) constant of crystal and direction cosines (l, m, n) of reflection, and can be calculated using following formula [25].

$$\frac{1}{E} = S_{11} - 2\left[(S_{11} - S_{12}) - \frac{1}{2}S_{44}\right]\left(l^2m^2 + m^2n^2 + n^2l^2\right) \quad (1)$$

The change in elastic modulus of iron due to alloying addition can be ascertained using WH technique. Considering classical WH method, the total peak broadening due to crystallite size (d_V) and micro-strain (ε) are related by following expression [30,31]:

$$\Delta K = \frac{0.9}{d_V} + 2\varepsilon\left(\frac{2\sin\theta}{\lambda}\right) \quad (2)$$

where $K = 2\sin\theta/\lambda$, and $\Delta K = (2\Delta\theta)\cos\theta/\lambda$ is caused by peak broadening. WH plots for Fe–20Cr–5Al alloy powder for different milling time are shown in Fig. 2. It is evident from Fig. 2 that the