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The effect of particle size of iron powder on α to γ transformation in the nanostructured high nitrogen Fe–Cr–Mn–Mo stainless steel produced by mechanical alloying

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ABSTRACT

In this study, the effect of particle size of iron powder on α to γ transformation in the nanostructured high nitrogen Fe–18Cr–10Mn–4Mo stainless steel, produced by mechanical alloying (MA) was investigated. For this purpose iron powders with two different particle sizes were used. MA was performed under nitrogen atmosphere, using a high-energy planetary ball mill. X-ray diffraction (XRD) patterns and nitrogen analysis revealed that by decreasing the iron mean particle size, a higher transformation rate is obtained due to increase in the rate of nitrogen absorption. Moreover, nitrogen solubility in both milled samples was increased noticeably by increasing the milling time. This is believed to be due to the increase of lattice defects and development of nanostructure through MA. Variations of grain size and internal lattice strain versus milling time, for both iron particle sizes, showed that the critical ferrite grain size for austenite nucleation is less than 10 nm.

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1. Introduction

Nitrogen is a gamma stabilizer and an excellent solid solution strengthening element in stainless steels. In addition, it improves many properties such as strength, ductility, toughness (especially at low temperatures), creep strength as well as resistance to crevice corrosion, pitting corrosion, and stress corrosion cracking [1–4]. Due to the unique properties, high nitrogen stainless steels (HNSS) have wide range of applications. In the field of biomaterials, nickel free high nitrogen stainless steels are highly attractive. This is because the nickel ions are the most common contact allergen in the human body. A nickel allergy triggers visible inflammatory reactions on the skin and mucus membranes in the form of red patches or, in the worst case, eczema [5–8].

Commercial grades of HNSS are produced by high-pressure techniques such as pressurized induction furnace melting, pressurized plasma furnace melting, and pressurized electroslag remelting (PESR). However, these processes are very expensive. Alternatively, powder metallurgy such as hot-isostatic pressure nitriding, steel nitriding under mechanical fluidized vacuum, and mechanical alloying (MA) can be used in order to increase the nitrogen content of steels [8–10]. Recently, it has been shown that MA has proved to be a suitable technique for producing HNSS powders. Apart from simple equipment used in this method, the amount of nitrogen in the steel powder could be increased well above the equilibrium solubility limit. Apart from that a nanostructured powder could also be easily produced by MA process. This, in turn, leads to higher mechanical properties such as strength, fracture toughness as well as resistance to grain growth [4,11–13].

Rawers and Maurice [14] reported that α to γ transformation could occur by milling pure iron powders under the nitrogen atmosphere. Cisneros et al. [15] investigated the MA of Fe–18Cr–11Mn powder mixtures using two MA processing units, an attritor and a high energy Spex mill. They reported the presence of amorphous ferrite after 72 h of milling in the attritor mill under nitrogen atmosphere. Their results showed complete transformation of ferrite to austenite after 1 h annealing at 1000–1100 °C. However, in the case of Spex mill, 100% austenite was produced after 120 h of milling. They concluded that formation of fully austenitic structure depends on the milling time, the nitrogen content, annealing temperature, and the energy of the mill. Also, Mendez et al. [16] reported that a fully austenitic structure can be obtained by milling Fe–18Cr–11Mn–5Mo powders under nitrogen atmosphere after 192 h of milling and subsequent annealing at 1200 °C. Haghir et al. [17] investigated the effect of MA on α to γ phase transformation in Fe–18Cr–11Mn powder mixtures under N_2 and Ar atmospheres. They depicted that nucleation of austenite phase occurred after 120 h of milling under Ar atmosphere. While, under N_2 atmosphere, phase transformation of α to γ was completed after 100 h of milling. They concluded that the amount of nitrogen absorbed in the milled

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Table 1
Properties of the starting powders used.

Powder	Fe		Cr	Mn	Mo
	I	II			
Particle size (μm)	3–14	20–75	100–150	70	>20
Purity (%)	99.9	99.9	>99	>99	99.7

powder can provide the thermodynamic and kinetic conditions necessary for α to γ transformation. The purpose of this research is to investigate the effect of particle size of iron powder on α to γ transformation during the production of nanostructured Ni-free high nitrogen Fe–18Cr–10Mn–4Mo stainless steel by MA.

2. Materials and methods

The Fe, 18 wt% Cr, 10 wt% Mn and 4 wt% Mo powder mixtures were prepared using high purity elemental powders. Particle size and purity of the starting powders used in this work are given in Table 1. In addition, high purity N_2 gas (99.99%) was used as the milling atmosphere. In order to investigate the effect of particle size of iron powder on α to γ transformation, two different particle sizes of iron powder were used. Particle size of iron powder was measured by laser particle size analyser (LPSA). Fig. 1 shows particle size distribution and morphology of both iron powder particles. In this work, samples prepared with smaller particle size of iron were named samples (I) and other samples with larger particle size of iron were named samples (II).

MA was performed in a planetary high-energy ball mill (Retsch, PM100) with a ball-to-powder weight ratio of 25:1. Stainless steel balls with 20 mm diameter, rotating speed of 300 rpm and nitrogen atmosphere were used. The milling was performed at ambient temperature.

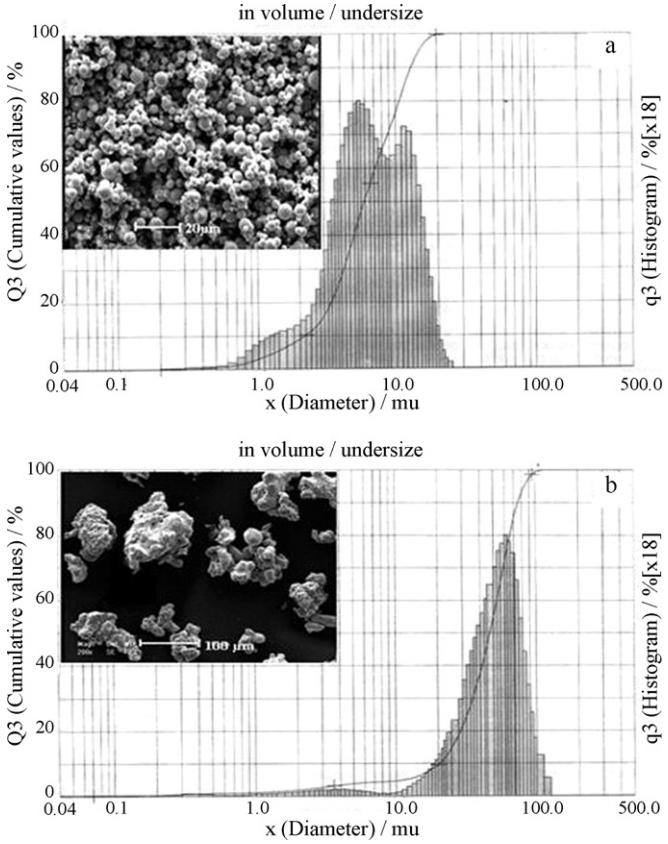

Fig. 1. Particle size distribution and scanning electron micrographs of the iron powders before milling (a) iron (I) and (b) iron (II).

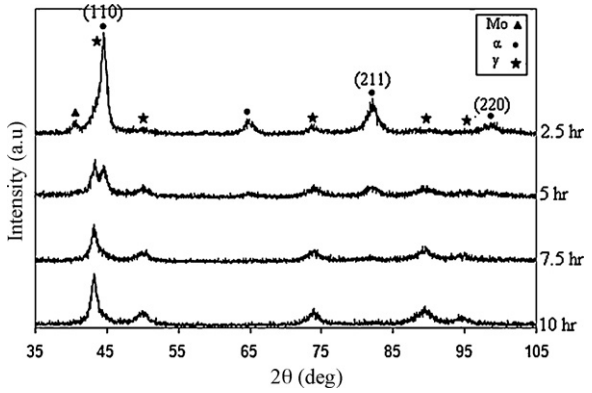

Fig. 2. XRD patterns of samples (I) at various milling times.

Microstructural and phase analysis of the milled powders were carried out employing SEM (Philips XL30 at an accelerating voltage of 30 kV) and XRD (Philips X'PERT MPD, with Cu Kα radiation), respectively. Nitrogen contents were determined using nitrogen analyser equipment (ELTRA, ON900). The grain size and lattice strain were calculated from XRD patterns, using the Williamson–Hall equation:

$$\beta \cos\theta = \frac{0.89\lambda}{d} + 2\varepsilon \sin\theta \quad (1)$$

where β is the peak width at half the maximum intensity, θ is Bragg's angle, λ is wavelength of the X-ray used, d is grain size, and ε is internal lattice strain.

3. Results and discussion

3.1. Phase analysis

Figs. 2 and 3 show the XRD patterns of the two milled powders for different time periods. As seen, the primary peaks of the starting materials tend to broaden or disappear by increasing the milling time. This behavior could be affected by parameters such as: (1) dissolution of alloying elements in the Fe lattice, (2) increase of lattice strain due to the increase in structural defects and (3) grain size reduction during milling. As shown in Figs. 2 and 3, peaks of ferrite phase gradually disappear in both samples, and ultimately a fully austenitic structure is achieved. In samples (I), α to γ transformation is completed after 10 h of milling (Fig. 2). Conversely, in samples (II), a fully austenitic structure is obtained after 100 h

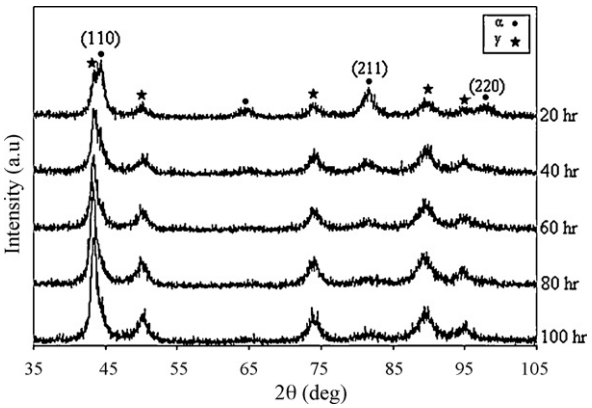

Fig. 3. XRD patterns of samples (II) at various milling times.

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