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Phase transformation and microstructure study of nano-structured austenitic and ferritic stainless steel powders prepared by planetary milling

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ABSTRACT

In the present work, nano-structured austenitic and ferritic stainless steel powders were prepared in bulk by milling elemental powders in a specially designed dual-drive planetary mill (DDPM) and Fritsch pulverisette planetary mill (P5 mill) for 10 and 40 h respectively. The progress of milling and phase transition of stainless steel have been studied by means of X-ray diffraction. The crystallite size and the lattice strain of the austenitic stainless steel after 10 h of milling are 9 nm and 5.59×10^{-3} respectively. Similarly, the crystallite size and the lattice strain of the ferritic stainless steel after 10 h of milling are 8 nm and 9.05×10^{-3} respectively. Annealing of milled powder at 750 °C promotes ferritic to austenitic transformation in both argon and nitrogen atmospheres as limited transformation takes place after milling. However, nitrogen favors the transformation to a greater extent than argon. Lattice parameters calculated from both high resolution transmission electron micrographs (HRTEM) and Nelson–Riley method match with austenitic and ferritic stainless steels. It has been found that initially particles are flattened and finally become almost spherical of size around 10–15 μm in both cases.

1. Introduction

Stainless steel prepared by powder metallurgy route has attracted scientist attention due to its capability of producing components with near-net shape, superior corrosion resistance and a wide range of mechanical properties through various alloy selections and processing options. Although different grades of stainless steels are produced by powder metallurgy but among them austenitic and ferritic stainless steels are extensively studied due to their versatile applications. Austenitic stainless steel is conventionally used in orthopedic applications as surgical cutting tools or as medical implants and low temperature applications where high toughness levels are compatible with cryogenic applications. However, nanosize oxide dispersed ferritic stainless steels with 12–14 wt.% Cr have attracted significant interest for potential high temperature structural, automotive industry and fuel cladding applications in advanced nuclear reactors due to their excellent high temperature mechanical properties and creep resistance [1].

Many processing methods have been adopted to improve the structure and properties of stainless steel. It is expected that further improvement of stainless steels can be achieved by refinement of their structure down to a nanocrystalline range. In recent years a number of methods like equi-channel angular pressing, hydrostatic extrusion and high pressure torsion are used for refining the structure of metals and alloys [2]. Among the different processes able to produce nanocrystalline powders in bulk quantities, high-energy milling is one of the most interesting from an industrial point of view. High-energy planetary milling of nanophase powder is one of the less sophisticated and inexpensive technologies to produce nanostructured powders; in fact, it manipulates devices and processes that have many aspects in common with mixing, fine grinding, and comminution of materials. However, it does have an interest for industrial applications due to its very low cost process. During planetary milling, material transfer takes place by diffusion and this makes it possible to achieve true atomic level alloying which is often accompanied by transformation to metastable phases with a nanosized structure [3–5]. The creation of a high density of dislocations and grain boundaries in powders and solute microsegregation at these defects can lead to the extended solid solutions.

Several researchers have prepared stainless powder by planetary milling of elemental powders. Kaloshkin et al. [6] studied the evolution of alloy formation in Fe–Ni alloy system using a AGO-2U planetary mill with water-cooled vials, where the elemental powder weight was only 15 g. They studied the martensitic transformation behavior and concluded that, low temperature treatment and deformation result in phase transformation from FCC to BCC [6]. Enayati and Bafandeh studied the phase transition of stainless steel powders prepared from a planetary ball mill under argon atmosphere after 60 h milling [7]. Oleszak et al. [8] prepared austenitic stainless steel powder using a Fritsch planetary P5 mill using 10 g of powder after milling for 100 h. They

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concluded that the formation of both austenitic and martensitic phases in stainless steel structure depends upon milling time [8]. Similarly, Haghir et al. synthesized high-nitrogen Fe–18Cr–11Mn austenitic stainless steel powder in a high energy planetary ball mill (Retsch, PM100) after 120 h of milling and studied phase transformation of α to γ [9]. Salahinejad et al. prepared austenitic stainless steel powder in a high energy shaker mill in nitrogen atmosphere for 120 h of milling. They concluded that increase in nitrogen concentration decreases grain size of austenitic stainless steel [10]. Amini et al. synthesized austenitic stainless steel powder in a Fritsch planetary ball mill in nitrogen atmosphere after 144 h of milling. They noticed that milling atmospheres such as argon and nitrogen can result in the transformation of phases [11].

From the available literature it is found that large milling time (>100 h) is required to prepare stainless steel powder and very small amount of powder can be prepared (<50 g) in pulverisette type planetary mills. But the specially designed dual-drive planetary mill (DDPM) is one of the highly efficient mills to prepare nanostructured stainless steel powder compared with other traditional milling techniques as it has a very high acceleration field (73 g). The DDPM consists of a gyratory shaft and cylindrical jars and both rotate simultaneously in opposite directions at high speed and we can control their speed of rotation by a frequency regulator. This allows ball to move strongly and rigorously, which led to large impact energy between the balls and the materials. Our investigation reveals that the DDPM requires only 10 h to prepare nanostructured stainless steel powder but the pulverisette planetary mill requires more milling time. Single run of the DDPM can easily synthesize more than 200 g of powder but the range is limited to less than 50 g in pulverisette milling. Moreover, none of the literatures have studied microstructure evolution and phase transformation in details during synthesis of stainless steel powder by planetary milling. Our main intention is to study the feasibility of bulk synthesized nanostructured stainless steel powder prepared from the DDPM and carry out a detailed study of the phase transformation as well as microstructure during milling and after annealing.

2. Mill fabrication

A high energy dual drive planetary mill (DDPM) was developed particularly to synthesize bulk nanostructured stainless steel powder. The specially designed DDPM has a main shaft of 640 mm (L) in length and two steel jars of 100 mm (2R) diameter (volume—1000 ml each) which rotates about their own axes around the common axis of the main shaft. The planetary mill was attached with two 5HP motors used to drive the main rotating shaft and the vials. The rotating speeds of both motors can be varied independently by two different frequency controllers. Fig. 1(a) shows the photograph of the dual-drive planetary mill that was fabricated to synthesize nanostructured stainless steel powder. The details of mill design are available elsewhere [12,13]. The mill mechanics of the fabricated DDPM can be calculated as follows:Critical speed constant,

$$K_c = \frac{\varpi_2}{\varpi_1} = -1 \pm \sqrt{L/_{2R}} = -1 \pm \sqrt{\frac{640}{100}} = -1-2.52 = -3.52. \quad (1)$$

Taking negative sign, as main shaft and jars are rotating in opposite direction.

The percentage critical speed is calculated by considering the shaft speed (ϖ_1) and jar speed (ϖ_2) as 275 and 620 rpm, respectively. Therefore

$$\%CS = \frac{\varpi_2}{K_c \times \varpi_1} \times 100$$
$$\%CS = \frac{620}{3.52 \times 275} \times 100 = 64. \quad (2)$$

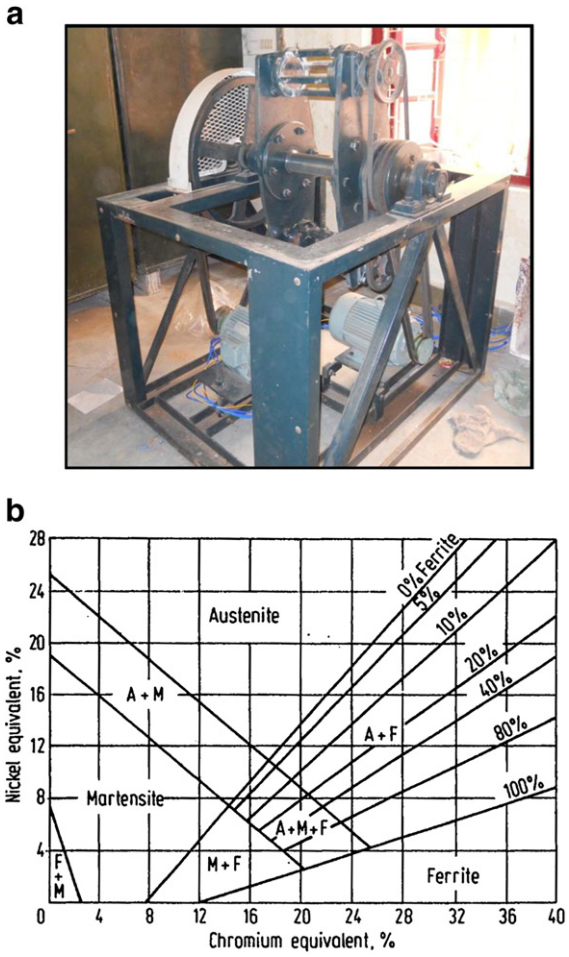

Fig. 1. (a) Photograph of dual-drive planetary mill, (b) Schaeffler–de Long diagram.

Here, all the milling experiments in the DDPM mill were carried out at 64% of critical speed.

3. Experimental

Elemental powder mixture of Fe (99.5% pure), Cr (99.8% pure) and Ni (99.5% pure) powders were used as starting materials. Elemental compositions of Fe–18Cr–13Ni (austenite) and Fe–17Cr–1Ni (ferrite) were selected from Schaeffler diagram (Fig. 1(b)). Milling of the above compositions was carried out in the specially designed DDPM and Fritsch pulverisette planetary (P5) mill for 10 h and 40 h respectively under toluene atmosphere to prevent oxidation. The milling media of the DDPM consist of 1 kg stainless steel balls of 8 mm diameter. The milling was conducted at room temperature and 6:1 ball-to-powder weight ratio was maintained in both types of milling. In the DDPM, the angular velocity of the vials and the supporting main shaft were 620 and 275 rpm respectively but the milling speed was 300 rpm in the P5 mill. Powders from both mills were characterized by X-ray diffraction (XRD) in a Philips PANalytical diffractometer using filtered Cu Kα-radiation (λ = 0.1542 nm). Powder samples prepared from only the DDPM were used for further characterizations as well as to study the phase transformation. Crystal size and lattice strain of the milled powders were calculated using Williamson–Hall and Scherrer methods. Nelson–Relay (N–R) method was used to calculate lattice parameter. Powder morphology was investigated by scanning electron microscopy (SEM) using JEOL JSM-6480LV and particle size was measured by a Malvern Mastersizer. Thermal behavior of final powder samples