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Development of nano-structured duplex and ferritic stainless steels by pulverisette planetary milling followed by pressureless sintering

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Nano-structured duplex and ferritic stainless steel powders are prepared by planetary milling of elemental Fe, Cr and Ni powder for 40 h and then consolidated by conventional pressureless sintering. The progress of milling and the continuous refinement of stainless steel powders have been confirmed by means of X-ray diffraction and scanning electron microscopy. Activation energy for the formation of duplex and ferritic stainless steels is calculated by Kissinger method using differential scanning calorimetry and is found to be 159.24 and 90.17 KJ/mol respectively. Both duplex and ferritic stainless steel powders are consolidated at 1000, 1200 and 1400 °C in argon atmosphere to study microstructure, density and hardness. Maximum sintered density of 90% and Vickers microhardness of 550 HV are achieved for duplex stainless steel sintered at 1400 °C for 1 h. Similarly, 92% sintered density and 263 HV microhardness are achieved for ferritic stainless steel sintered at 1400 °C.

1. Introduction

Duplex stainless steel contains almost equal proportions of ferrite and austenite phases in its structure [1]. Ferrite phase imparts more strength while austenite phase assures the toughness and better corrosion resistance. Duplex stainless steel has the combining features of two major classes of stainless steel, austenite and ferrite and thus made it very attractive for numerous applications [2]. Duplex stainless steels are having very good toughness, high corrosion resistance, low thermal expansion, high energy absorption, weldability and high strength compared to single phase austenitic and ferritic stainless steel hence used in chemical, oil, petrochemical, marine, nuclear power, paper and pulp industries [3–6]. The extent of heat treatment and re-crystallization decides the amount of ferrite and austenite ratio in duplex stainless steel along with the formation of sigma phase (δ), inter-metallic phases like primary alpha (α), various carbides and chrome nitrides. High yield strength, tensile strength, toughness and low percentage elongation of duplex are due to the presence of interstitial atoms and inter-metallic phases which act as obstacles for dislocation motion [7,8].

Ferritic stainless steel is one of the important types of stainless steel having body centered cubic (BCC) lattice structure. It contains Cr with very less percentage of expensive Ni, hence it is known as cost saving material whereas more weight percentage of Ni is required to stabilize austenite structure. Ferritic stainless steel is magnetic in nature, hence used as sticking memos on the fridge, storing knives and other metallic implements. It is also used as pans in induction cooker which involves the generation of heat by transfer of magnetic energy. Some of the properties such as low thermal expansion, excellent oxidation resistance at high temperature, high thermal conductivity, creep resistance, high yield strength and less stress corrosion properties make ferritic stainless steel an important type of stainless steels [9]. Both duplex and ferritic stainless steels are having a wide range of applications, hence continuous research work is going to improve their structures and properties. It is expected that nano-structured stainless steel can improve the properties exceedingly, so we made an attempt to bring down the stainless steel microstructure to nano-level. Nowadays, a decent number of methods are available to refine the structure of metals and alloys by serious plastic deformation methods such as equal channel angular processing, hydrostatic extrusion, high pressure torsion [10], ultrasonic shot peening [11], and hydraulic pressings [12]. But mechanical alloying (MA) [13] is one of the most widely used plastic deformation methods to achieve the extreme refinement of structure. The advantage of using MA to synthesize nano-crystalline materials lies in its ability to produce bulk quantities of materials in solid state using simple equipment at room temperature. Additionally, mechanically alloyed powder material reduces the oxidation of the constituent powders due to the short time of processing [14]. The creation of high density dislocations, grain boundaries and micro-segregation of solute at these defects can lead to an extended solid solution.

Dobrzanski et al. synthesized duplex stainless steel by mixing austenite base powder of composition Fe–17Cr–13Ni–2.2Mo along with alloying elemental powders such as Si, Mn and Cu in a turbula mixer. They consolidate the powder sample at 800 MPa load and at 2300 °F sintering temperature in argon atmosphere for 1 h [2]. Shashanka and

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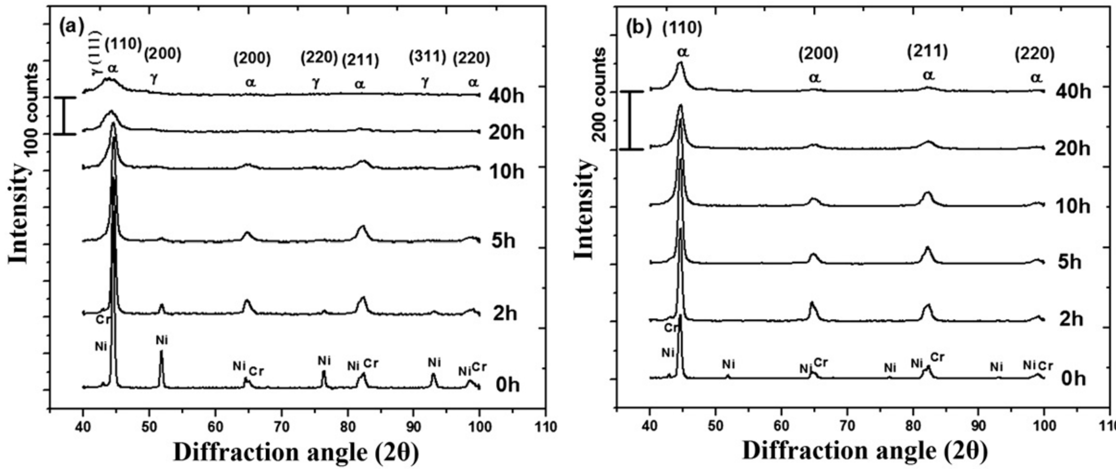

Fig. 1. XRD spectra of (a) duplex stainless steel and (b) ferritic stainless steel powder samples milled for 40 h.

Chaira prepared ferritic and austenitic stainless steel powders by elemental milling of Fe, Cr and Ni powders in a dual drive planetary mill for 10 h as compared to 40 h in pulverisette mill. They studied the phase transformation of austenitic and ferritic stainless steel powders both in argon and nitrogen atmospheres and concluded that nitrogen acts as austenitic stabilizer [15]. Brytan et al. prepared duplex stainless steel by mixing base ferritic stainless steel powder (16.86%Cr, 1.15%Si, 0.18%Mn, 0.02%C) with controlled addition of elemental alloying powders and was sintered at 1250 °C in a vacuum furnace with argon atmosphere for different time periods [16]. Enayati and Bafandeh studied the effect of annealing on milled powders of composition Fe–18Cr–8Ni and pointed out the presence of dual structure consisting of austenite and martensite phases [17]. Oleszak et al. prepared austenitic stainless steel powder using Fritsch planetary P5 mill. They concluded that the formation of both austenitic and martensitic phases in stainless steel structure depends upon milling time [18]. Similarly, Haghir et al. synthesized high-nitrogen Fe–18Cr–11Mn austenitic stainless steel powder in a high energy planetary ball mill (Retsch, PM100) milled for 120 h with ball to powder weight ratio of 25:1 and mill speed of 300 rpm. They carried out milling in Ar and N_2 atmospheres respectively to study the phase transformation of α-Fe to γ-Fe. Finally they concluded that particle size of austenite milled in Ar atmosphere is very less compared with the austenite milled at N_2 atmosphere where agglomeration occurred [19]. Amini et al. synthesized austenitic stainless steel powder in Fritsch planetary ball mill with ball to powder weight ratio 20:1 and mill speed of 250 rpm milled in nitrogen atmosphere for 144 h. They noticed that milling atmospheres like argon and nitrogen can also play an important role in phase transformation [20].

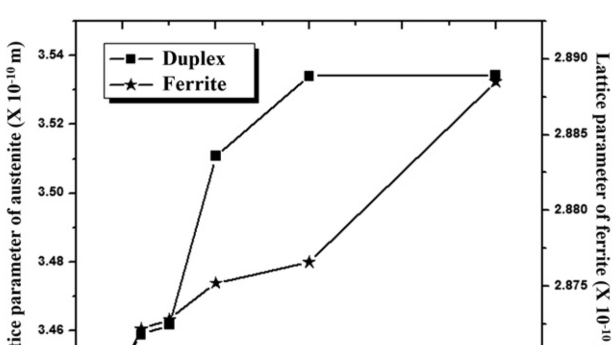

Fig. 2. Variation of lattice parameter (calculated by Nelson–Riley extrapolation method) with milling time.

Usually materials at nano-range behave entirely different from their bulk form [21] and hence we prepared nanostructured duplex (Fe–18Cr–13Ni) and ferritic (Fe–17Cr–1Ni) stainless steel powders using pulverisette-5 (P5) mill. From the above literatures it is clear that although research papers are available on the synthesis of duplex and ferritic stainless steel powders by MA but less research proofs are available regarding the study of thermal analysis of both duplex and ferritic stainless steels and consolidation of such stainless steels. None of them has given proper attention on the study of thermodynamic parameters during MA and mechanical strength of such stainless steel. Hence, we put a successful effort to study them in detail by computing activation energy, change in enthalpy, crystallization temperature, Curie temperature and mechanical strength. We also studied the effect of milling time on crystallite size, particle size and lattice strain. Milled powder samples were compacted in a hydraulic press at a load of 700 MPa and sintered in a tubular furnace at 1000, 1200 and 1400 °C to study the effect of temperature on microstructure, density and hardness.

2. Experimental

2.1. Synthesis of nano-structured stainless steel powder

Elemental powder mixture of Fe (99.5% pure), Cr (99.8% pure) and Ni (99.5% pure) was used as starting materials. Elemental compositions of Fe–18Cr–13Ni (Duplex) and Fe–17Cr–1Ni (Ferrite) were selected from Schaeffler diagram [22] to obtain duplex and ferritic stainless steel alloys during milling. Milling of the above compositions was carried out in Fritsch planetary mill (P5 mill) for 40 h under toluene atmosphere to prevent oxidation. Mill speed of 300 rpm and ball to powder weight ratio of 6:1 were maintained. The milling media consist of 500 ml milling jar and 300 g of chrome steel balls of 10 mm diameter each and it was filled around 40% by volume. Milled powders were characterized by X-ray diffraction (XRD) in a Philips PANalytical diffractometer using filtered Cu Kα-radiation (λ = 0.1542 nm). Crystal size and lattice strain of the milled powders were calculated using Williamson–Hall method and Nelson–Riley (N–R) method was used to calculate lattice parameter. Powder morphology is investigated by scanning electron microscopy (SEM) using JEOL JSM-6480LV and particle size by Malvern Mastersizer. Thermal behavior of final powder samples was studied using differential scanning calorimetry (DSC) Netzsch, Germany. Differential scanning calorimeter (DSC) was performed by continuous heating of 40 h milled duplex and ferritic stainless steel