



# Optimization of milling parameters for the synthesis of nano-structured duplex and ferritic stainless steel powders by high energy planetary milling



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## ABSTRACT

Nano-structured duplex and ferritic stainless steel powders were prepared by high energy planetary milling of elemental Fe, Cr and Ni powders. Here, we have studied the effect of process control agent (PCA) such as stearic acid (SA), effect of ball to powder weight ratio (BPR 6:1 and 12:1) and milling speed (64% and 75% critical speed) during planetary milling of elemental Fe–18Cr–13Ni (duplex) and Fe–17Cr–1Ni (ferritic) powders for 10 h in a dual drive planetary mill (DDPM). We have found that all these milling parameters have great influence in tuning the final particle morphology, size and phase evolution during milling. It was found that addition of PCA, a BPR of 12:1 and 75% critical speed is more effective in reducing particle size and formation of duplex and ferritic stainless steel after 10 h milling of elemental powder compositions than their counterparts. The particle size of duplex and ferritic stainless steel milled in the presence of SA for 10 h is found to be 13 and 14  $\mu\text{m}$ , whereas the particle size is 20 and 16  $\mu\text{m}$  without SA respectively. The particle size of powder milled at 12:1 BPR is lower than powder milled at 6:1 BPR because of more impact energy induced by balls on powders and it is found to be 10 and 12  $\mu\text{m}$  respectively for duplex and ferrite in presence of SA. The median particle size of duplex and ferritic stainless steel milled at 75% critical speed is found to be 3.5 and 2.4  $\mu\text{m}$  respectively.

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## 1. Introduction

The use of stainless steel has been increased extensively in various fields from the past few decades. Nowadays stainless steels are in great demand due to the properties of high corrosion resistance, good toughness, low thermal expansion, high energy absorption, weldability and high strength [1]. Ferritic stainless steel has body centered cubic (BCC) lattice structure and contains very less weight percentage of expensive Ni and 10 to 20 wt.% of Cr [2]. Today ferritic stainless steel is one of the important grades of stainless steel due to its high thermal conductivity, low thermal expansion, creep resistance, wear resistance, higher yield strength, excellent high temperature oxidation resistance and less stress corrosion properties [3]. These stainless steels are mainly used in refrigeration cabinets, bench work, cold water tanks, chemical and food processing, water treatment plant, street furniture, electrical cabinets etc. [4]. Nano-oxide dispersed ferritic stainless steel (ODS) is also a candidate material in nuclear industry due to its superior creep properties.

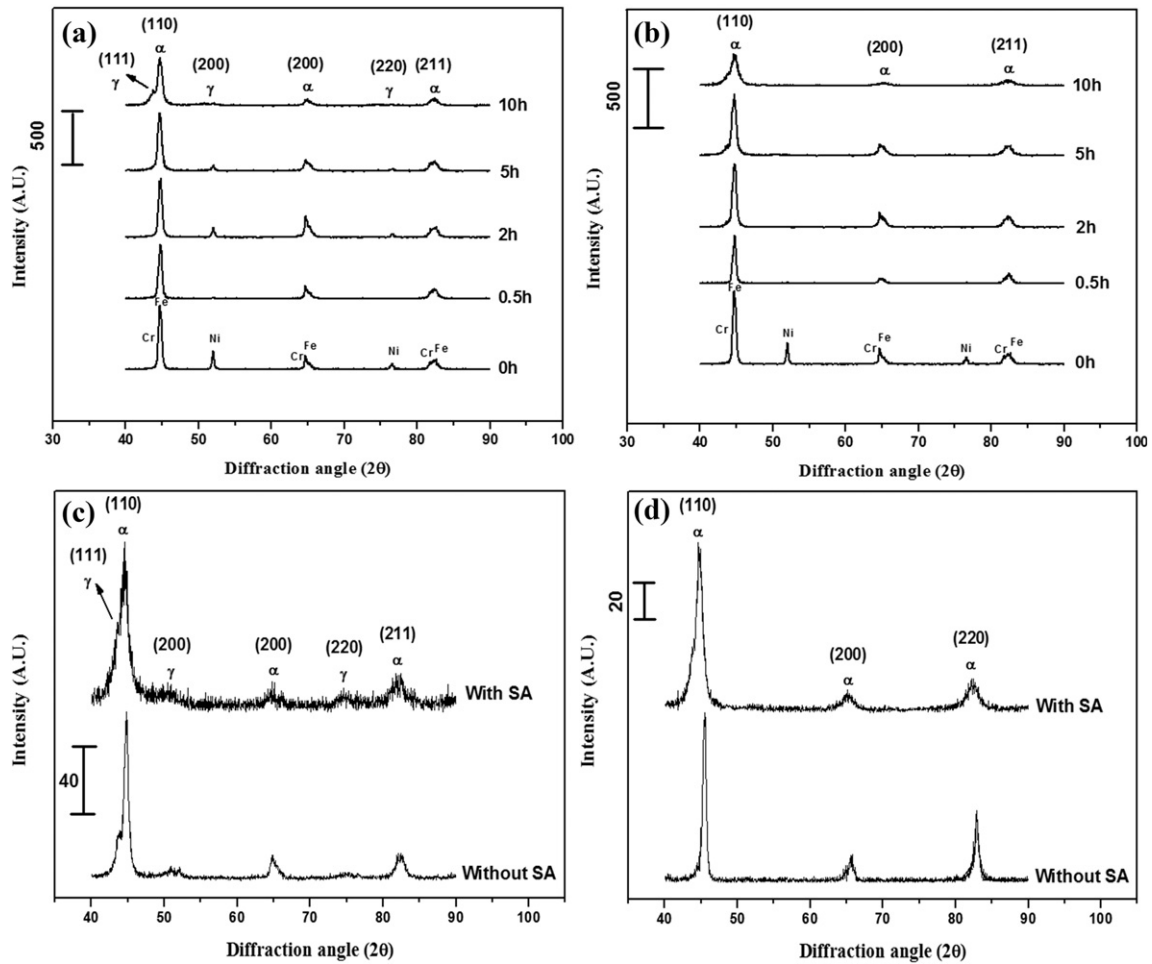
On the other hand, duplex stainless steel which meets all the above properties and hence it is one of the popular and widely applicable stainless steels. Duplex stainless steel is a combination of austenitic and ferritic stainless steel grades in almost equal proportions in which ferrite phase imparts high strength while austenite guarantees the toughness and high corrosion resistance [5]. Hence, duplex stainless steels have a wide range of applications starting from chemical, oil, petrochemical, marine, nuclear power to paper and pulp industries [6–8].

Because of excellent properties and wide range applications, duplex and ferritic stainless steels attracted researchers' attention to improve their structure and properties. Properties of the materials improve tremendously when bringing down their size to nano level [9]. Hence, we synthesized nano-structured duplex and ferritic stainless steel by high energy planetary milling. Many synthesis routes like equal channel angular processing, hydrostatic extrusion, high pressure torsion [10], ultrasonic shot peening [11], and hydraulic pressings [12] are used to refine the structure of metals and alloys by plastic deformation and solid solution mechanism. But planetary milling [13,14] is one of the most simple and widely used plastic deformation methods to achieve extreme refinement in structure of materials. The rate of particle refinement during milling depends on several milling parameters like PCA, ball to powder weight ratio, milling time, milling speed, size of the balls used and many more.

Abbreviations: DDPM, dual drive planetary mill; BPR, ball to powder weight ratio; PCA, process control agent; SA, stearic acid; CS, critical speed; PSA, particle size analysis.

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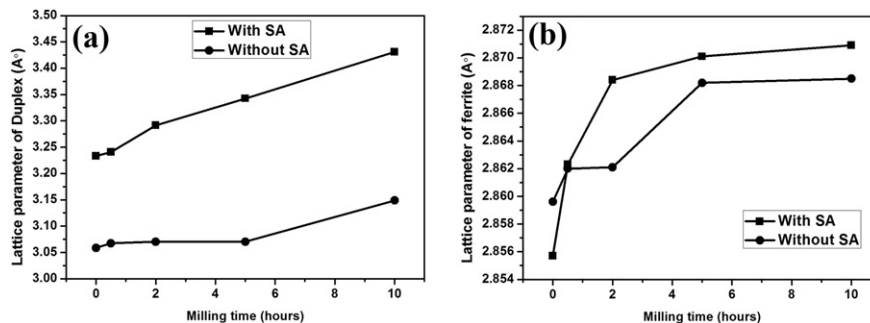
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**Fig. 1.** XRD patterns of 0 to 10 h milled. (a) Duplex stainless steel. (b) Ferritic stainless steel in the presence of 1 wt% SA. XRD patterns of only 10 h milled. (c) Duplex stainless steel. (d) Ferritic stainless steel in the presence and absence of SA.

Pandey et al. prepared ferritic stainless steel by DDPM with different BPR (10:1, 15:1 and 20:1) and milling speeds (250, 300 and 350 rpm). They reported that crystallite size and particle size of ferritic stainless steel decrease with increase in BPR and milling speed. Similarly, BET surface area and strain also increase with BPR and milling speed [15]. Rahmanifard et al. studied the effect of BPR (10:1 and 15:1) and milling speed (300 and 420 rpm) during milling of the ODS ferritic stainless steel. They observed that crystallite size and particle size of ferritic stainless steel powder reduce whereas lattice parameter increases with increase in BPR and milling speeds. They concluded that milling by using 8 mm diameter ball, 420 rpm milling speed and ball-to-powder

weight ratio of 10:1 can reduce the milling time to 30 h [16]. Ismail et al. prepared  $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$  alloy by milling in SPEX 8000D shaker mill at different BPR (4:1, 6:1, 8:1, 10:1, 12:1, 14:1, 16:1, 18:1 and 20:1) and studied the morphology of the prepared alloy. They reported that crystallite size decreases up to 16:1 and then start increasing with increase in BPR due to cold welding. Similarly, strain goes on increasing up to 16:1 BPR and after that it will start to decrease [17]. Liu et al. prepared  $\text{SnS}_2$  anode material by mechanical alloying route with different process control agents (ammonium chloride and SA) along with different BPR (20:1 and 25:1) to improve the particle morphology. They concluded that use of ammonium chloride as PCA not only reduces



**Fig. 2.** Graphical representation showing the effect of milling time on the lattice parameter (calculated from the Nelson–Riley extrapolation method) of (a) duplex stainless steel and (b) ferritic stainless steel in the presence and absence of SA.

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