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# Optimization of milling parameters, processing and characterization of nano-crystalline oxide dispersion strengthened ferritic steel

Abhishek Pandey [b,c], K. Jayasankar [a,*], P. Parida [d], M. Debata [a], B.K. Mishra [a], S. Saroja [d]

[a] CSIR-Institute of Minerals and Materials Technology, Bhubaneswar 751013, India
[b] CSIR-Advanced Materials and Processes Research Institute, Bhopal 462024, India
[c] Academy of Scientific and Innovative Research, New Delhi, India
[d] Microscopy and Thermo-Physical Property Division, Physical Metallurgy Group, Indira Gandhi Centre for Atomic Research, Kalpakkam 603102, India

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ABSTRACT

Ferritic steel powder was mechanically milled in a dual drive planetary ball mill, under different milling conditions to optimize the milling parameters. The resulted powder was characterized, using particle size analyzer, X-rays and electron microscope. X-ray peak broadenings were investigated to estimate crystallite size, lattice strain and deformation stress. Better Pearson's coefficient was observed for uniform stress deformation model (USDM) (0.988) in comparison to uniform deformation model (UDM) (0.64) which shows better estimation of lattice parameter. An increase in fineness was observed with an increase in ball to powder ratio as well as for an increase in rotational speed. At the optimized condition, ferritic steel powder, together with $Y_2O_3$, was milled in the dual drive mill to produce oxide dispersion strengthened ferritic steel powder, suitable to be used in nuclear applications. Convoluted morphology, desired for better alloying, was confirmed using an electron microscope. A significant increase in per unit surface area was noticed due to milling using BET surface area analysis. Negligible contamination was observed due to milling atmosphere and mill container. The steel powder produced, was sintered using spark plasma sintering and its density and hardness were measured. High hardness and lower crystallite size were recorded using spark plasma sintering. Addition of $Y_2O_3$ shows decreases in the thermal expansion coefficient. Effect of added titanium was studied and an adverse effect on oxide dispersion strengthened steel was noticed.



## 1. Introduction

Selection of suitable structural material for fast breeder reactors is dependent on conventional properties such as thermo-physical, mechanical, corrosion and compatibility and ability to withstand neutron induced atomic displacement which causes void swelling and materials hardening/embrittlement. Ferritic steels have good radiation resistance inherently but its creep resistance is poor at high temperature of the breeder reactor. With dispersion of nano-ceramic oxide particles ($Y_2O_3$) in the matrix of the ferritic steels, its creep resistance shows a significant improvement. These $Y_2O_3$ particles together with Ti which forms complex stable nano-clusters of Y–Ti–O particles inside the matrix obstruct the motion of dislocation thus giving creep strength. The stability of these nano-clusters is mostly due to the high lattice coherency with the bcc steel matrix [1]. Also, the tensile and creep behavior of ODS steel varies with inter-particle distance and highest resistance to deformation is obtained for smallest inter-particle distance which is directly proportional to the particle size [2]. With the improved creep strength and superior swelling resistance it is assumed that ODS steels would be the most suitable materials for cladding in the next generation fission reactors.

Mechanical alloying (MA) is one of the popular techniques which has been proved to be effective as well as simple. The variables in milling, which affect the properties of nanocrystalline powders are, BPR taken by weight, rotational speed of the jar i.e. revolution per minute (RPM), milling time and ball diameter. There are different types of ball mills commercially available such as attritor mills, SPEX shaker mill, planetary ball mill, the Simoloyer (Zoz mill) and dual drive planetary ball (DDPB) mill. In the present investigation, DDPB mill was used and milling conditions were optimized for the production of nanocrystalline ferritic steels.

Objective of this work was to give an effort to produce ODS ferritic steel powder using DDPB mill and its characterization. Pre-alloyed ferritic steel powder with composition of Fe–9Cr–2.25W–0.2Ti and externally added $Y_2O_3$ and Ti, is used to synthesize ODS ferritic alloy. There are many methods to produce a bulk or finished product after MA as hot extrusion [3], cold compaction followed by sintering [4–6], equi-channel angular pressing [7,8], isostatic pressing [9] and SPS. The last one mentioned was used here as it has many advantages over the

* Corresponding author at: Advance Materials Technology Department, CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, 751013, India. Tel.: +91 674 237 9377; fax: +91 674 256 7637.
*E-mail address:* jayasankar@immt.res.in (K. Jayasankar).

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others. By conventional methods, densification takes place at higher temperatures which also accompanies grain growth and loss of nano or ultra fine structure [10–12]. By SPS route original microstructure can be preserved and it takes much less process time in comparison to other processes. SPS is most promising pressure assisted process with a high heating rate, low sintering temperature and holding time [13,14]. Powder is heated by the direct pulse current which flows through the punches dies and the materials to be sintered. A high current density used and localized micro-plasmas into the gap or contact point between the conductive powders is generated if the powders are conducting [15].

## 2. Experimental

### 2.1. DDPB mill

DDPB mill has horizontal axis of rotation and there are two drive system, as the name suggest (dual drive), one for the jar and other for the disk holding the jar. Therefore it is easy to control the speed of the jar and disk separately [16]. Both are rotated at high speed simultaneously and separately. High speed rotation causes balls to move strongly and violently, which causes large impact energy. With precise control, the attainable multiple transmission ratios possible are (1/4:1 to 4:1). It is the transmission ratio of the mill which plays a critical role in determining critical speed of rotation.

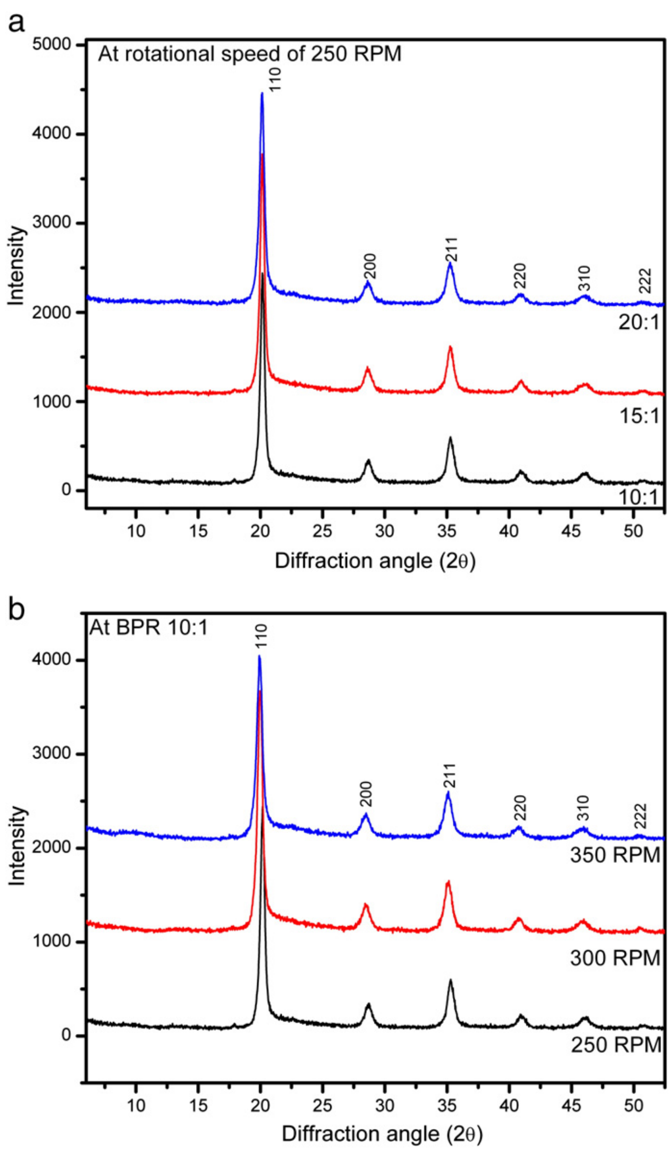


**Fig. 1.** (a) X-ray diffraction patterns for ferritic steel powder showing the effect of ball to powder ratio. (b) X-ray diffraction patterns for ferritic steel powder showing the effect of rotational speed.

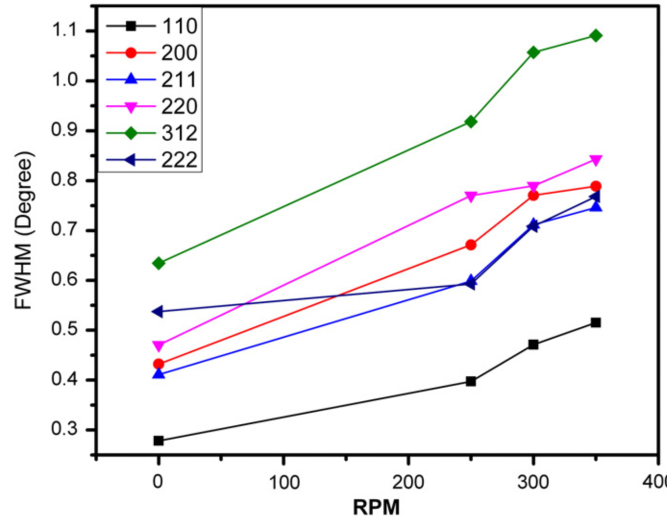


**Fig. 2.** Change in FWHM of the samples milled at different rotational speeds.

### 2.2. Sample preparation

Pre-alloyed gas atomized ferritic steel powder was used with composition Fe–9Cr–2W–0.1C–0.2Ti (in wt.%) with a mean particle size of 75 μm for mill optimization. The jar and the balls (10 mm diameter) were made up of stainless steel to avoid any contamination. The transmission ratio taken was 1:2 throughout the experiment. Optimization was followed by processing of ODS steel powder, with two different compositions (a) Fe–9Cr–2W–0.1C–0.2Ti + 0.5%$Y_2O_3$ (SMP1) and (b) Fe–9Cr–2W–0.1C–0.2Ti + 0.5%$Y_2O_3$ + 0.1%Ti (SMP2) under optimized condition. Further, they were loaded in to graphite die with diameter 20 mm, lined with graphite foil and were sintered using SPS Syntex Inc. SPS furnace with varying heating rates: initial

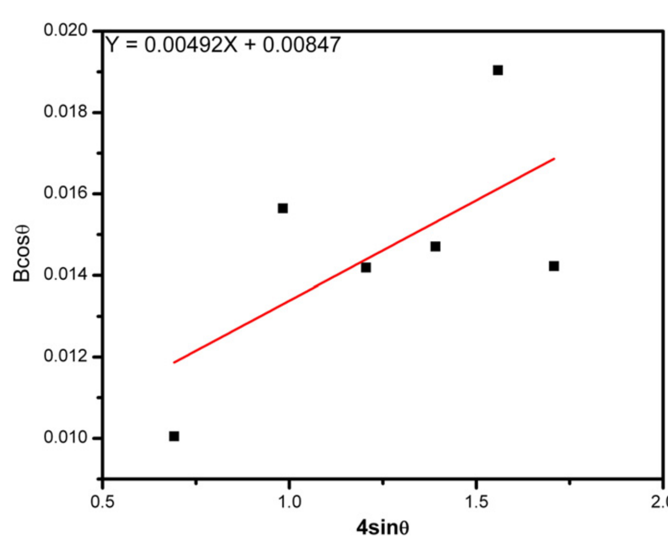


**Fig. 3.** W–H–UDM plot for ferritic steel powder with 350 RPM & BPR 20:1.