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In-situ formation of complex oxide precipitates during processing of oxide dispersion strengthened ferritic steels

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HIGHLIGHTS

- Use of dual drive planetary ball mill for Bench scale (>1 kg) production.
- X-ray diffraction and TEM were used to study transformations during sintering.
- HIPped and rolled samples with nearly 99% density successfully produced.

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ABSTRACT

In fusion and fission reactor material development, ODS alloys are the most suitable candidate materials due to its high temperature creep properties and irradiation resistance properties. This paper describes the preparation of oxide dispersion strengthened alloy powder in large quantity (>1 kg batch) in dual drive planetary ball mill using pre-alloyed ferrtic steel powder with nano sized Y_2O_3. The consolidation of the powders was carried out in hot isostatic press (HIP) followed by hot rolling. 99% of the theoretical density was achieved by this method. The vickers hardness values of pressed and rolled samples were in the range of 380 ± 2HV and 719 ± 2HV, respectively. Samples were further investigated using X-ray diffraction particle size analyzer and electron microscope. Initial increase in particle size with milling was observed showing flattening of the particle. It was found that 5 h of milling time is sufficient to reduce the particle size to achieve the desired size. Transmission electron microscopy analysis of milled ODS steel powder revealed a uniform distribution of combustion synthesized nano-Y_2O_3 in ferritic steel matrix after a milling time of 5 h. Preliminary results demonstrated suitability of dual drive planetary ball mill for mass production of alloy within a short time due to various kinds of forces acting at a time during milling process. Fine monoclinic $Y_2Si_2O_7$ precipitates were also observed in the steel. This study explains the particle characteristics of nano Y_2O_3 dispersed ODS powder and formation of nano clusters in ODS ferritic alloy.

1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic steel has already established itself as the most promising material as a construction material for fission and fusion reactors, but, most suitable composition is yet to be achieved. Most of the compositions had already been investigated but little variations and new techniques are still being tried with the hope to get success to achieve suitable alloys with optimum time and energy consumption. Excellent swelling resistance and thermal properties of ferritic/martensitic steels make it very much suitable for nuclear reactors [1]. In spite of all this, under neutron irradiation its impact toughness decreases, and ultimately it reduces its upper operating temperature maximum up to 600 °C [2]. Oxide dispersion strengthening is one of the most suitable techniques reported till now for improving creep strength needed in ferritic/martensitic steels to increase its higher operating temperature. Different types of high energy ball mills already being used for the dispersion of oxide particles in the steel matrix. A recently added new device to this list is duel drive planetary ball mill. Use of this mill has already been introduced and reported elsewhere [3,4]. Base composition of the alloy system also plays important role in designing the alloy. Therefore special care need to be taken before selecting the alloy system

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Table 1
Chemical composition and powder properties.

Chemical analysis		Physical properties	
Element	%	Apparent density	2.82 g/cm^3
Carbon	0.024	Flow rate	29.8 s/50 g
Molybdenum	1.00	Sieve analysis	
Manganese	0.1	+150 µm	1.29%
Chromium	16.2		
Silicon	0.8	−45 µm	35.37%
Iron	Balance		

Table 2
Parameters of ball milling.

Starting material	Fe-16Cr-1Mo (434L grade) steel powder + 0.35 wt% Y_2O_3 nano powder
Medium	Wet (Toluene)
Ball to powder ratio	20:1
Gyro speed (disk)	150 rpm
Ball mill jar speed (vial)	300 rpm
Critical speed	67%
Ball weight	3.7 kg
Sample weight	185 g
Time for milling	1 h, 3 h, 5 h

according to its working environment. For example corrosion resistance in high temperature waters decreases with decreasing the chromium content (<13%) [5–9]. These low chromium steels are targeted for sodium cooled fast breeder reactors and they cannot be used for water cooling solid breeder fusion blanket. Taking this into consideration, in the present paper, pre-alloyed ferritic steel powder with higher chromium content (Fe-16Cr: 434L grade) was used as matrix material for the ODS alloy preparation. Use of nano-Y_2O_3 as a dispersoid is already very popular due to its high temperature phase stability and low neutron absorption cross section than any of the rare earth [10].

This paper was targeted to produce ODS steel powder using duel drive planetary ball mill. Pre-alloyed ferritic steel powder together with nano-Y_2O_3 was milled in duel drive mill with the aim to produce a structural material to be used in nuclear reactors. The powders produced were compacted using hot isostatic press (HIP). Further, it was subjected to secondary processing and characterization.

2. Experimental

2.1. Material preparation and milling

An efficient dispersion of nano-oxides in ODS ferritic steels was achieved by employing high energy ball milling technique, dual drive planetary ball (DDPB) mill. The effect of milling on the crystallite size, morphological and micro structural properties of powder particles were examined systematically. During milling, samples were collected at different time intervals to study significant changes if there is any (>1 kg scale). The starting material used in this study was 434L-grade ferritic steel powder supplied by M/s Hoganas, Belgium. The chemical composition along with relevant powder properties are given in Table 1.

The nano-Y_2O_3 dispersoids were prepared by combustion synthesis route in the form of particles between ∼30 and 50 nm in size. The complete design of the mill is shown in Fig. 1. The rotation of jar and the shaft holding the jar is independent here so it is very easy to control the motion of both separately. High speed rotation causes balls to move strongly and violently, which in turn causes a large impact energy. With a precise control, the attainable multiple transmission ratios possible in this mill varies from 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 [11,12]. Milling parameters of mechanical alloying (MA) of ODS ferritic steel are given in Table 2.

2.2. Consolidation of ODS alloys

The milled powders were consolidated using HIP and hot rolling was carried out. The milled powders were filled in stainless steel can, evacuated, and then sealed tightly. The canned specimens were transferred to the HIP and consolidated at 1150 °C, under 120 MPa pressure for 3 h holding time under argon atmosphere. Samples were heated at 1100 °C for 2 h holding and then subjected to hot rolling at a condition of 50% reduction by 0.5 mm per pass. The thickness of the hot rolled strip is 10 mm. Fig. 2(a) shows the HIPed ODS steel specimen and Fig. 2(b) shows the hot rolled ODS steel specimen.

2.3. Characterization techniques

XRD data were recorded with the help of a Philips diffractometer (PAnalytical X'Pert PRO) equipped with Mo-$K\alpha$ radiation (λ = 0.7094 Å). Broadening in the diffraction peak due to instrumental error was determined and corrected using a polycrystalline Si standard sample. Changes in particle size and shape of milled powder have been evaluated by particle size analyzer (CILAS 1064). Samples were also examined using field-emission electron microscope (FESEM) and transmission electron microscope (TEM). For FESEM, powders were mounted over a carbon tape and in some cases it was coated with a thin layer (∼50 nm) of gold applied by sputter deposition before examination with the FESEM to make the sample conducting. FESEM was done using a Zeiss, Carl Zeiss SMT

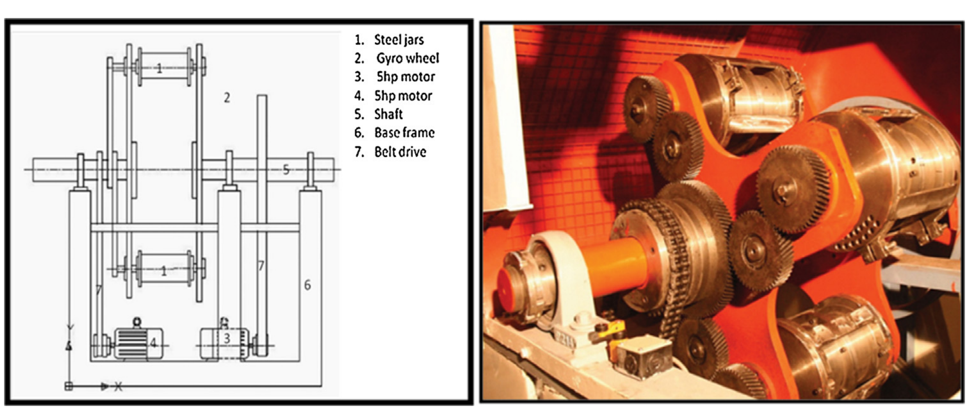

Fig. 1. Schematic diagram and photograph of dual drive planetary ball mill.