



# A new method for preparing 9Cr-ODS steel using elemental yttrium and Fe<sub>2</sub>O<sub>3</sub> oxygen carrier



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## ABSTRACT

Casting oxide dispersion strengthened (ODS) steel is difficult due to the coarsening and agglomeration of oxide particles during melting and solidification. In the present paper, a new method was developed to prepare 9Cr-ODS steel via the in situ reaction between elemental yttrium in molten Fe and activated oxygen from Fe<sub>2</sub>O<sub>3</sub> oxygen carrier. To identify whether coarsening and aggregation of yttrium oxides can be suppressed, the cast steels did not suffer plastic deformation. Finally, tempered martensite matrix was obtained accompanied with about 0.2–1.1 μm yttrium oxides. Some yttrium oxides suffered coarsening and agglomeration due to the high oxygen content. 9Cr-ODS steel exhibited higher ultimate tensile strength, yield strength and slight lower elongation compared to 9Cr steel. The results indicated that yttrium oxides could be effectively introduced into the steel matrix via this casting method, which provided an alternative method for ODS steel fabrication.

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## 1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic (F/M) steels containing a high number of fine oxide particles are being considered as the primary candidate materials for fuel claddings in fission reactors and blankets in fusion reactors because of their excellent resistance to irradiation and high-temperature creep as well as extraordinary structural and chemical stability in extremely harsh environments [1–4]. The typical strengthening oxide is Y<sub>2</sub>O<sub>3</sub>, which is one of the most stable oxide and more stable than classical nitrides and carbides [5]. Usually, the ODS F/M steels were manufactured via complicated powder metallurgy (PM), which consisted of mechanical alloying (MA), degassing & canning, hot powder consolidation and heat treatment [6–9]. However, PM has some inherent shortcomings involved with the ODS F/M steel production, i.e., inducing anisotropy to microstructure and mechanical properties [10], introducing more impurities [11], rendering batch production complicated [12], economically unfeasible for the large-

scale industrial components production [13]. Furthermore, pores on the prior particle boundaries (PPB) and precipitates survived from PM degraded the creep resistance [10]. Besides, argon nanocavities formed during PM may reduce the fracture toughness and welding performance [14].

Taking these drawbacks of PM into considering, some alternative methods were explored recently to manufacture ODS steels. F. Han et al. directly added 2 μm Y<sub>2</sub>O<sub>3</sub> powders into molten Fe alloy for producing 9Cr ODS steels via vacuum induction melting, magnetic stirring casting, forging and heat treatment [12,15]. The results indicated that some micro-scale Y<sub>2</sub>O<sub>3</sub> particles suffered coarsening, agglomeration and large yttrium oxides of about 5 μm were formed. The primary reason for oxide accumulation and coarsening during casting of steels is the high thermal stability of rare earth oxides such as Y<sub>2</sub>O<sub>3</sub> at casting temperature and thus high interface energy and poor wettability between these particles and molten Fe [16]. Specifically, the melting point of Y<sub>2</sub>O<sub>3</sub> is 2683 K, and the high temperature contact angle of pure electrolytic Fe drops in contact with Y<sub>2</sub>O<sub>3</sub> was approximately 90° [17]. This prevents the physical bonding between Y<sub>2</sub>O<sub>3</sub> and molten Fe.

To prevent the coarsening and agglomeration of oxide particles during melting and solidification, the oxygen carrier concept was developed to prepare ODS alloy in Ref. [18]. ODS Fe-10Ni-7Mn alloy

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was produced through vacuum casting using  $\text{TiO}_2$  and dissolved yttrium in the liquid metal as the sources of oxygen and yttrium, respectively. Firstly, FeNiMn alloy and Fe-20Y (wt.%) interalloy were prepared in a vacuum arc melting furnace, respectively. Fe-10 $\text{TiO}_2$  (wt.%) oxygen carrier discs were prepared by PM using Fe and  $\text{TiO}_2$  powders. Afterwards, FeNiMn master alloy and the Y-rich Fe-20Y (wt.%) interalloy were remelted in a vacuum arc remelting furnace to produce the FeNiMn-Y alloy. Finally, FeNiMn-Y ingot was remelted together with Fe-10 $\text{TiO}_2$  (wt.%) oxygen carrier discs in the vacuum arc remelting furnace to prepare the ODS Fe-10Ni-7Mn alloy. The results indicated that the addition of  $\text{TiO}_2$  as an oxygen carrier succeeded in the size control and distribution of  $\text{Y}_2\text{O}_3$  as well as the prevention of agglomeration and coarsening of oxides during casting. However, this method consists of several times of casting together with PM process, which is very complicated.

In the present work, a new method was developed to prepare 9Cr ODS F/M steel via in situ reaction between elemental yttrium in molten Fe and activated oxygen from  $\text{Fe}_2\text{O}_3$  oxygen carrier during the solidification. The aim of this study was to explore the feasibility of this casting method for ODS steel preparation and to identify whether the coarsening, aggregation of yttrium oxides can be suppressed effectively. Finally, tempered martensite matrix accompanied with yttrium oxides of about 0.2–1.2  $\mu\text{m}$  were achieved via the vacuum induction melting and casting followed by heat treatment without plastic deformation. Furthermore, 9Cr ODS F/M steel exhibited higher tensile strength and yield strength as well as slight lower elongation and Charpy impact properties compared to 9Cr F/M steel without yttrium oxides.

## 2. Theoretical considerations and approaches

Dissolved elemental yttrium in the liquid Fe alloy and  $\text{Fe}_2\text{O}_3$  were selected as the sources of yttrium and oxygen, respectively. Fig. 1(a) shows the Y-Fe binary alloy phase diagram [19]. Elemental yttrium will be completely dissolved in molten Fe at temperature above 1811 K when yttrium content is very low. Thus firstly, elemental yttrium powders were added into the molten Fe alloy in a vacuum melting furnace at 1873 K under argon atmosphere to prepare molten Fe alloy dissolved with elemental yttrium. Fig. 1(b) shows the O-Fe binary alloy phase diagram [19]. FCC\_B1 is the Wüstite. Actually, there are three kinds of Fe oxides, i.e.,  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$  and Wüstite with oxygen content of 30.06%, 27.64% and 23.16%–25.60%, respectively. It should be noted that the theoretical oxygen content in  $\text{Fe}_2\text{O}_3$  is the highest. Furthermore,  $\text{Fe}_2\text{O}_3$  can be reduced and formed  $\text{Fe}_3\text{O}_4$ , Wüstite and Fe accompanied with activated oxygen in sequence at high temperature. Thus  $\text{Fe}_2\text{O}_3$  was selected as the oxygen carrier compound, which was placed at the

bottom of the mould before casting.

Fig. 2 shows the schematics of introducing yttrium oxides into the steel matrix via casting method using elemental yttrium and  $\text{Fe}_2\text{O}_3$  oxygen carrier. Step 01: metallic Y powders were added and then dissolved into the liquid steel. Step 02:  $\text{Fe}_2\text{O}_3$  powders were placed at the bottom of the mould. Step 03: liquid steel with dissolved Y was poured into the mould. During the casting process, elemental yttrium would in situ react with the activated oxygen from  $\text{Fe}_2\text{O}_3$  immediately and then yttrium oxides were generated. The elapsed time between pouring of the liquid alloy and solidification was several minutes, which meant that the yttrium oxides would not undergo heat preservation and suffer coarsening and aggregation.

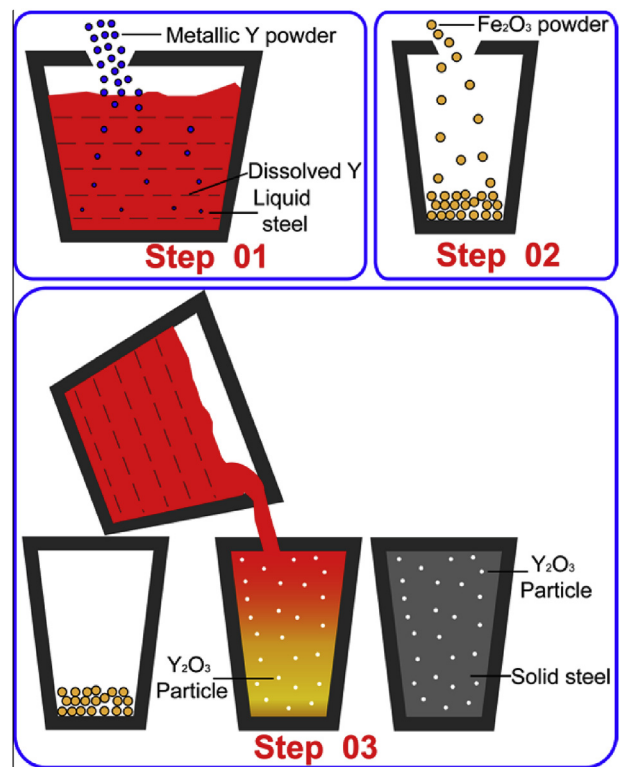


Fig. 2. Schematics of introducing yttrium oxides into the steel matrix via casting method using elemental yttrium and  $\text{Fe}_2\text{O}_3$  oxygen carrier.

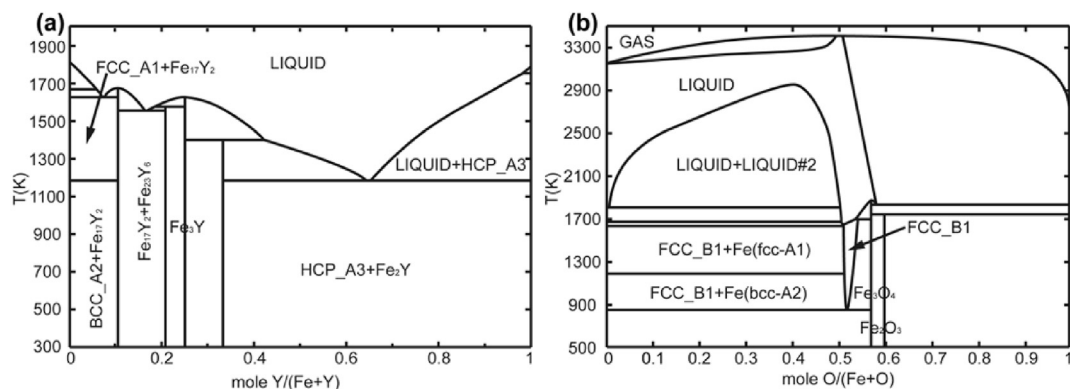


Fig. 1. (a) Fe-Y phase diagram [19]; (b) Fe-O phase diagram [19].

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