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Investigation of the possibility of introducing yttrium oxide by vacuum induction melting to produce nickel-free stainless steel

Abstract: The possibility of smelting an oxide-dispersed strengthening steel alloy using five different approaches involving the introduction of finely dispersed Y2O3 particles into liquid Fe-13Cr steel alloy under vacuum conditions has been investigated. To achieve the objective, five series of experiments were carried out, each differing in the conditions and process of the experiments. Two series of melting, were performed to evaluate the possibility of mechanical introduction of yttrium oxide into the melt under different conditions of introduction. The subsequent three series, were carried out to study the possibility of oxidation of metallic yttrium in the melt with the formation of yttrium oxide particles. In these experiments such parameters as duration of melt holding time and residual pressure in the furnace chamber were varied. When the obtained ingots were analysed by X-ray fluorescence (XRF), inductively coupled plasma atomic emission spectroscopy (ICP-AES) and energy dispersive X-ray spectroscopy (EDS), it was found that a significant amount of yttrium oxide was not successfully incorporated into the steel volume. A method using oxidation of metallic yttrium in the melt by reduction of added iron oxide was found to be the most promising.

Keywords: ODS steel, yttrium oxide, induction furnace, nickel-free steel.

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Introduction

The study of new materials for application in various fields of science and technology is an important task for scientists. One of the most important materials contributing to the technological progress of mankind is steel, which is widely used in various spheres of human life (Kenzhaliyev, 2019; Chukmanova et al., 2022; Ozili & Ozen, 2023). Nuclear power represents one of the solutions to the energy crisis and is becoming widespread in developed countries such as USA, China, France, Japan, etc. (Valone, 2020). This method of energy production is not only environmentally friendly but also highly efficient.

The study of structural materials for use in nuclear power generation is an important task for scientists. Steels used in this field must fulfil various requirements such as high resistance to high pressures, reactor neutron irradiation, aggressive corrosion, etc. (Wang et al., 2021). Nickel steels, which are the basic materials for all generations of nuclear plants, cannot cope as well with new generation and future reactors. One of the main problems with nickel steels is due to the fact that nickel under the influence of neutron irradiation changes to the radioactive isotope nickel-63, which creates difficulties with subsequent utilization. The solution to this problem can be oxide dispersion hardened steels (ODS), which are ceramic particles embedded in the metal matrix of steel.

The advantages of such steels over nickel steels are their resistance to the accumulation of induced radiation on the material. The most common method of production of steels with ODS is powder metallurgy, based on the large number of scientific studies devoted to this topic. The least common methods are additive lachser melting and hybrid processes (Ermakov et al., 2013). However, the use of liquid metallurgy to create ODS steels remains a less explored area, which can be judged from the limited number of scientific publications on this topic.

In most cases, the microstructure of ODS steels is characterised by submicron grains, low defect density, presence of nano-oxides with diameter <d> ≈ 1-5 nm, density N ≈ 10^23-24 m^{-3} and content of about 0.5-1%. These structures possess coherent and semi-coherent interfaces as well as dislocations between the nanooxides and the matrix (Hirata et al., 2011; Zinkle, 2013; Stan, 2020; Zhang, et al., 2015).

In this study, a series of experiments were carried out to investigate the possibility of introducing yttrium oxide into steel using liquid metallurgy. Five series of melts were carried out, of which two studied the possibility of mechanical introduction of yttrium oxide into the melt, and in three - the possibility of oxidation of metallic yttrium with subsequent formation of yttrium oxide in the melt.

Research Methods

For the experimental part the induction vacuum furnace UIPV-0,001 was used. This furnace is designed for melting in vacuum or inert gas atmosphere at temperature up to 2200 C and residual vacuum pressure not less than 10 Pa. The materials used were AISI 410 steel, yttrium (III) oxide nanopowder sintered <50 nm (Sigma-Aldrich), metallic yttrium, pure iron powder and iron oxide powder. In the experiment, 5 series of melts were carried out under 5 different conditions each (Figure 1).

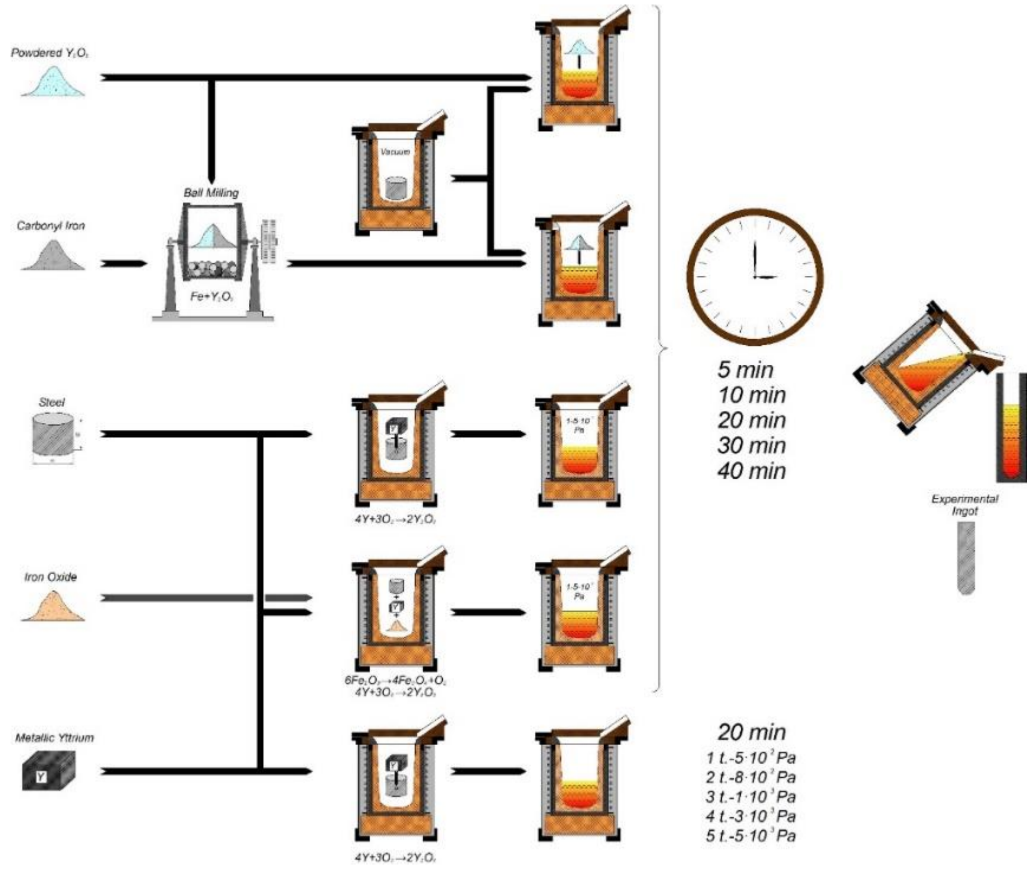

Fig. 1. Schematic diagram of the steelmaking experiment

Three techniques were used to investigate the molten steel: inductively coupled plasma mass spectrometry (ICP-MS) (Thermo scientific iCAP RQ ICP-MS), inductively coupled plasma atomic emission spectroscopy (ICP-AES) (Thermo Fisher Scientific iCAP 6300 DUO) and scanning microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) (Jeol JSM IT-200LA). For the first two analyses, chips from different locations of the ingot were used to average the results; for the EDS analysis, the steel was sawn into 5 mm thick discs and pressed into epoxy resin (Figure 2).

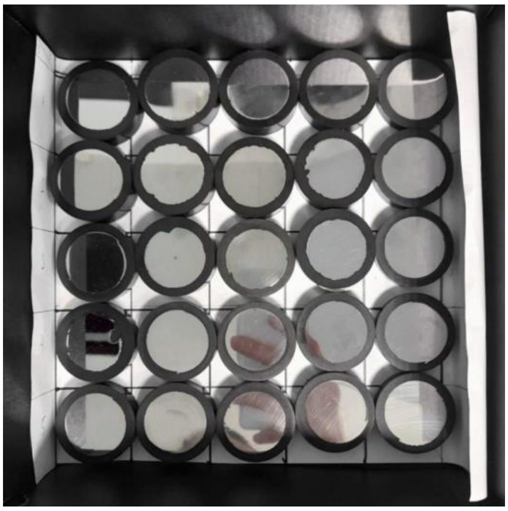

Fig. 2. Steel prepared for EDS analysis

Research experiment

During the experiment five series of experiments were carried out, and here is a brief description of each of them:

In the first series of experiments, the possibility of introducing yttrium oxide nanopowder into the melt of steel by mechanical method followed by solidification was investigated. The melting was carried out at a temperature of about 1600 °C in a vacuum atmosphere with a pressure of ≥ 100 Pa. During the experiment, the holding time of the melt in the crucible after complete melting was varied between 5, 10, 20, 30 and 40 minutes. After holding time, the steel was poured into an ingot with a diameter of 30±1 mm.

In the second series of experiments, the possibility of introducing yttrium oxide nanopowder on a carrier of pure iron powder into the steel melt was investigated. A planetary mill with tungsten carbide balls was used to mix the two powders. The powder mixture was poured into the steel melt at a temperature of about 1600 °C and under vacuum with a pressure of ≥ 100 Pa. In the experiment, the holding time of the melt in the crucible after melting the steel was varied as 5, 10, 20, 30 and 40 minutes. After holding time, the steel was poured into a crystalliser with a diameter of 30±1 mm.

In the third series of experiments the possibility of oxidation of metallic yttrium in the furnace from residual oxygen was investigated. Metallic yttrium was placed in a drilled hole in steel. Melting took place at a temperature of about 1600 °C and residual pressure in the furnace chamber in the range of 1-5·102 Pa. In the experiment, the holding time of the melt in the crucible after complete melting of the steel varied from 5 to 40 minutes. After holding time, the steel was poured into a crucible with a diameter of 30±1 mm.

In the fourth series of experiments the possibility of oxidation of metallic yttrium during reduction of iron oxide in the process of melting was investigated. Metallic yttrium was placed in a drilled hole in steel and closed with a stopper. Iron oxide powder was placed at the bottom of the crucible and steel with metallic yttrium was placed on top. Melting took place at a temperature of about 1600 °C and residual pressure in the furnace chamber in the range of 1-5·102 Pa. The experiment varied the holding time of the melt in the crucible after complete melting of the steel from 5 to 40 minutes. After holding time, the steel was poured into an ingot with a diameter of 30±1 mm.

In the fifth series of experiments the oxidation of metallic yttrium from oxygen supplied to the furnace chamber was investigated. Metallic yttrium was placed in a drilled hole in the steel and closed with a plug. The melting was carried out at a temperature of about 1600 °C and a melt residence time of 20 minutes in the furnace. During the melting process, the residual pressure in the furnace chamber varied in the range of $5\cdot10^2$ Pa, $8\cdot10^2$ Pa, $1\cdot10^3$ Pa, $3\cdot10^3$ Pa, $5\cdot10^3$ Pa. After tempering, the steel was poured into a mould with a diameter of 30±1 mm.

Results and discussions

The obtained steel samples were investigated by ICP-AES, ICP-MS, SEM/EDS methods. Steel melts of series I-II, in which the possibility of introducing yttrium oxide by mechanical means was investigated, did not show positive results for yttrium content in steel. But positive results were shown by melts of series III-V, where the possibility of oxidation of metallic yttrium was investigated. The results are given in Table 1. Data for melts of series I and II are not given due to lack of sufficient yttrium content. Since yttrium was detected in different fractions in different analyses, it was decided to use EDS analysis to detect yttrium oxide particles in the steel.

Table 1. ICP-AES and ICP-MS analyses of the obtained steel in III-V series of the experiment

	ICP-AES	**ICP-MS**
№ ingot	**Y, ppm**	**Y, %**
III,4	0,0127	0,28
IV,1	0,0552	0,97
IV,4	0,0044	0,17
V,1	0,0013	0,03

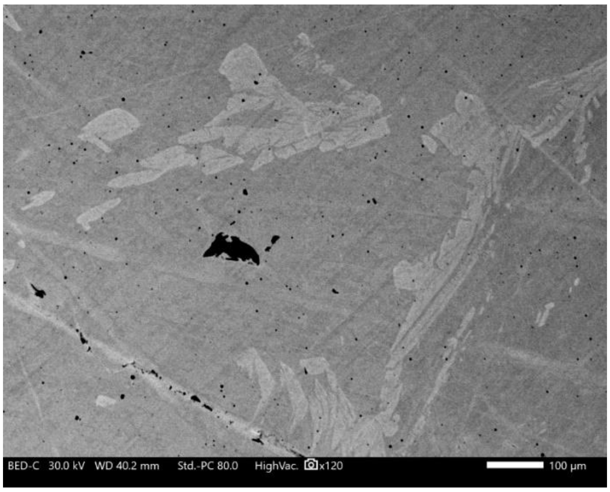

Fig. 3. SEM images of steel with non-metallic inclusions

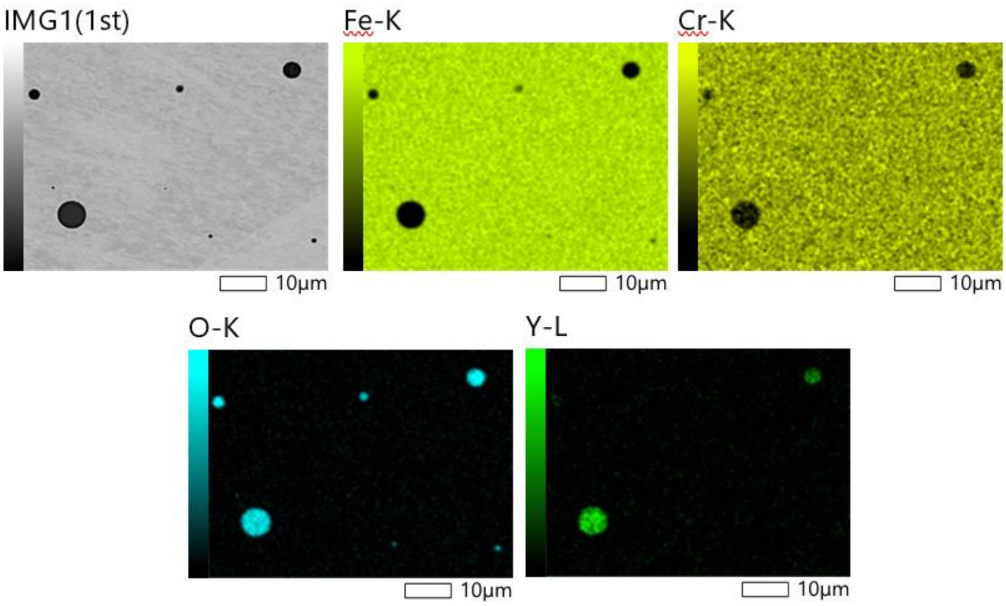

Fig. 4. EDS analysis of non-metallic inclusions

On SEM-images in the sample with a large amount of fixed yttrium(IV,1) non-metallic particles in the steel volume were visible (Fig. 3). In which yttrium concentration could be suspected, which was additionally confirmed by EDS-analysis (Fig. 4).

Conclusions

Analysis of the melted steel revealed non-metallic inclusions where yttrium and oxygen are concentrated. This observation may indicate the presence of yttrium oxide compounds in these inclusions. However, this method of introducing yttrium into the steel was not effective because yttrium oxide is not dispersed in the ferritic matrix of the steel and therefore does not fulfil the function of strengthening the material.

The study showed the following conclusions:

Obtaining ODS steel by mechanical introduction of yttrium or oxidation of metallic yttrium cannot be used as a structural material.

When the steel was melted to oxidise metallic yttrium, all the yttrium concentrated in non-metallic inclusions that do not interact with the steel matrix.

The results obtained from this work provide information for further research aimed at developing methods for producing steels with oxide-dispersed structures (ODS) using liquid metallurgy.

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