THE VARIOUS WAYS INJECTION OF Y_2O_3 PARTICLES INTO THE NICKEL-FREE STAINLESS-STEEL MELT BY EXPERIMENTAL VACCUM INDUCTION MELTING

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Abstract

In this work, was studied the possibility of producing nickel-free stainless ODS steel using liquid metallurgy. In order to investigate the injection of dispersed yttrium oxide into steel, 5 different methods under induction vacuum melting conditions were tested. The aim of the study was to approbate the possibility of mechanical introduction of yttrium oxide powder into the martensitic-ferritic matrix of AISI 410 steel by two methods, or the possibility of oxidation of yttrium metal by 3 methods (from iron oxide, from residual oxygen in the furnace chamber, from injected oxygen into the furnace chamber). As a result of the performed work, the highest yttrium concentration of 0.0552 ppm from the oxidation of metallic yttrium during the reduction of iron oxide was recorded.

Keywords: Vacuum induction furnace, steel, ODS, metallurgy

1. INTRODUCTION

The study of obtaining new types of materials for use in promising fields of science and technology is one of the main research tasks of scientists. The most important material for human progress is steel, which is used in all areas of human life. One of the problems of economy and industry can be considered the energy crisis, resources for coal and gas plants are running out, while obtaining energy from the environment will not meet all the needs of businesses and cities [1]. One of the solutions to the energy crisis used by the advanced countries of the world USA, China, France, Japan, etc. is nuclear power. This method of energy production is not only environmentally friendly but also highly efficient [2].

The study of structural materials for the nuclear power industry is one of the main tasks scientists are pursuing. Steels used in this field must have a list of characteristics such as resistance to high pressures, reactor neutron irradiation, corrosive effects, etc. [3]. Nickel steels, used as the basic material in the operation of past generations of plants, do not have sufficient characteristics for long-term operation. But the main problem is the transition of nickel to the radioactive isotope nickel-63 under the influence of neutron irradiation, which complicates further disposal. A solution to this problem could be ODS steels, which are reinforcing ceramic particles in a ferritic steel matrix.

The advantage of these steels over nickel steels, used everywhere in the nuclear power industry, is the absence of induced radiation after their failure. The main and most common method of producing ODS steels is powder metallurgy, as evidenced by the large number of articles on producing steels by this method. The least common processes are additive forging and hybrid processes [3]. The topic of obtaining ODS steels by liquid metallurgy is the least studied, based on the availability of scientific articles on the subject of ODS. The main problem with liquid metallurgy for producing ODS steels is an edge wetting angle of 140□, which indicates an almost complete lack of wettability of yttrium oxide melt [4].

In most cases, the microstructure of ODS steel has the following characteristics: submicron grain size, defect density of about 10^{16} m^{-2}, nanooxides with average diameter <d> ≈ 1-5nm, N density ≈ $10^{23\text{-}24}m^{-3}$ and volume fraction f ≈ 0.5-1%, coherent and semi-coherent interface bonds and dislocations between nanooxides and matrix [5-8].

In this work, based on a small study of this subject in liquid metallurgy, it was decided to conduct a series of experiments on steel melting to investigate the possibility of introducing yttrium oxide into the melt. Five series of melts were carried out in 2 of which we tested the possibility of introducing yttrium oxide mechanically to the melt and 3 of which we tested the possibility of oxidation of metallic yttrium to form yttrium oxide in the melt.

2. MATERIALS AND EQUIPMENT.

For the experimental part an induction vacuum furnace UIPV-0,001 was used. This furnace is used for smelting in a vacuum or inert gas atmosphere, at temperatures up to 2200 C and a residual vacuum pressure of at least 10 Pa. The materials used were AISI 410 steel, Yttrium (III) oxide nanopowder, <50 nm sintered (Sigma-Aldrich), metallic yttrium, pure iron powder and iron oxide powder. In the experiment 5 series of melts were carried out at 5 different conditions each (**Figure 1**).

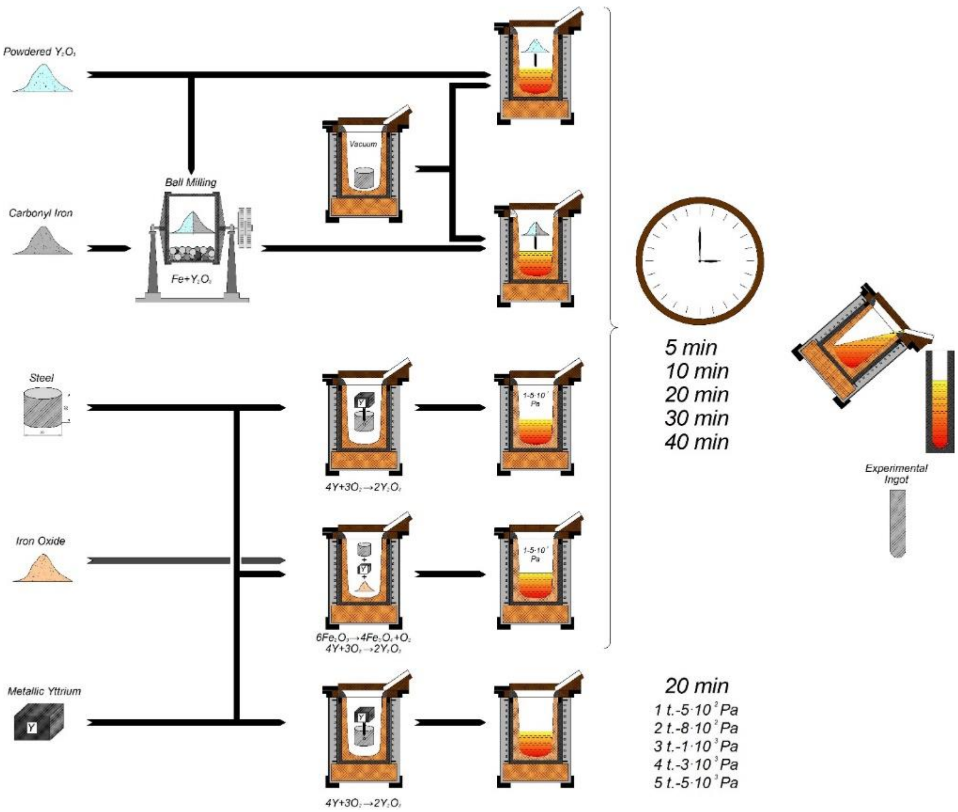

Figure 1 Scheme of melting steel experiment

Three methods were used to examine the melted 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 in 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**).

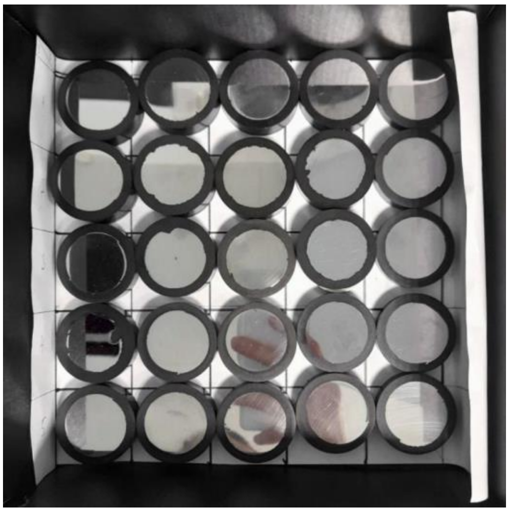

Figure 2 Steel prepared for EDS analyses

3. EXPERIMENTAL PART

In the course of the experiment 5 series of experiments were carried out (hereinafter the series of experiments will be marked with a Roman numeral and the mode of experiment with an Arabic numeral):

In I the possibility of introducing yttrium oxide nanopowder into the melt during melting by mechanical means and further curing is investigated. The melting was carried out at ~1600 ˚C, vacuum atmosphere ≥ 100 Pa. During the experiment melt residence time in the crucible after full melting was varied from 5, 10, 20, 30 and 40 minutes. After holding time, the steel is poured into an ingot of diameter 30±1.

In II the possibility of introducing yttrium oxide nanopowder into the melt on a carrier of pure iron powder is investigated. A planetary mill with tungsten carbide balls is used to mix the two powders. The powder mixture is poured into the steel melt at a temperature of ~1600 °C and a vacuum atmosphere of ≥100 Pa. The experiment varies the holding times of the melt in the crucible after steel melting for 5, 10, 20, 30 and 40 minutes. After holding time the steel is poured into the mold with a diameter of 30±1.

In III the possibility of oxidation of metallic yttrium from residual oxygen in the furnace is investigated. It is assumed that metallic yttrium will be oxidized from residual oxygen in the furnace chamber and converted to an oxide form, subsequently remaining in the melt volume. The metallic yttrium was placed in a drilled hole in the steel. The melting took place at a temperature of ~1600 ˚C and a residual pressure in the chamber of 1-5·10^2 Pa. During the experiment the holding time of the melt in the crucible after full melting of steel was varied 5, 10, 20, 30, 40 minutes. After soaking, steel is merged into the crucible of 30±1 diameter.

In IV investigated the possibility of oxidation of metallic yttrium during the reduction of iron oxide during melting. The metallic yttrium is placed in a drilled hole in the steel and sealed with a plug. Powder of iron oxide was placed at the bottom of the crucible and steel with metallic yttrium was placed on top. Smelting took place at ~1600 ˚C and a residual pressure in the chamber of 1-5·10^2 Pa. The experiment varied the holding time of the melt in the crucible after complete melting of steel 5, 10, 20, 30, 40 minutes. After holding time, the steel is poured into an ingot of diameter 30±1.

In IV the oxidation of metallic yttrium from the oxygen fed into the furnace chamber is investigated. It is assumed that the metallic yttrium dissolved in the steel will be oxidised by the oxygen fed into the furnace chamber. The metallic yttrium is placed in a drilled hole in the steel and sealed with a plug. Smelting took place at a temperature of ~1600 ˚C and melt residence time of 20 minutes. During melting the residual pressure in the furnace chamber was varied 5-10^2 Pa, 8-10^2, 1-10^3 Pa, 3-10^3 Pa, 5-10^3 Pa. After tempering the steel is poured into the mold of diameter 30±1.

4. DISCUSSION

The obtained steel samples were examined by ICP-AES, ICP-MS, SEM/EDS. The steel melted in series I-II, where the possibility of introducing yttrium oxide by mechanical means was investigated, did not show any results for yttrium content. But positive result showed melts of series III-V where the possibility of oxidation of metallic yttrium was investigated. The results are shown in table 1. Data for series I and II melts are not shown due to lack of yttrium content. As yttrium was found in different fractions in the different analyses, it was decided to use the EDS analysis to detect yttrium oxide particles in the steel.

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

	ICP-AES	**ICP-MS**
№ of 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

The SEM images in the sample with large amounts of fixed yttrium(IV,1) showed non-metallic particles in the steel volume (**Figure 3**). In which yttrium concentrations were suspected, as further proved by EDS analysis (**Figure 4**).

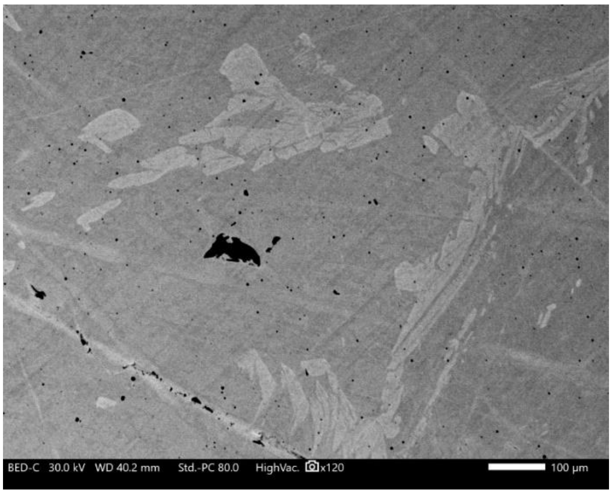

Figure 3 SEM image of steel with non-metal inclusions

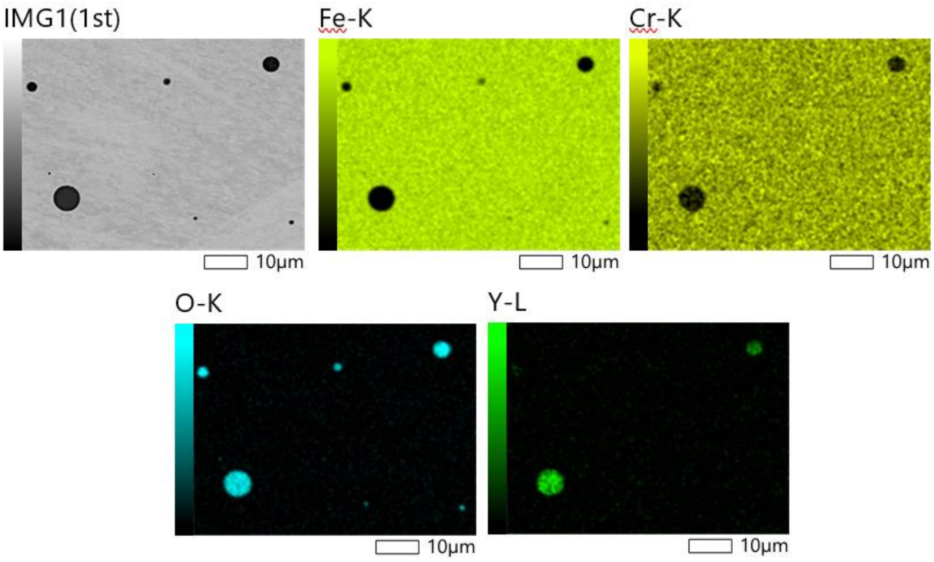

Figure 4 EDS analyses of non-metal inclusions

5. CONCLUSIONS

The analysis of the melted steel revealed non-metallic inclusions in which yttrium and oxygen are concentrated, which may indicate a concentration of yttrium oxide compounds in this area. However, this is not the way to produce ODS steel as yttrium oxide does not disperse into the ferrite matrix of the steel and therefore does not act as a reinforcing agent. The objective of the study was to investigate the possibility of introducing yttrium oxide into steel dispersively, the following conclusions can be drawn from the results:

- Obtaining ODS steel by mechanical introduction of yttrium, or oxidation of metallic yttrium is not possible.
- When melting steel to oxidize metallic yttrium, all the yttrium is concentrated in non-metallic clusters that do not interact with the steel matrix.
- This study provides information for further research on the production of ODS steels by liquid metallurgy.

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