ANNOTATION

of the dissertation thesis for the degree of Doctor of Philosophy (PhD) in the educational program «8D07202 - Metallurgy of ferrous and non-ferrous metals»

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DEVELOPMENT AND STUDY OF THE TECHNOLOGY FOR OBTAINING OXIDE-DISPERSION HARDENED STEEL FOR USE IN NUCLEAR POWER ENGINEERING

Relevance of the research topic.

The development of nuclear energy is associated with the continuous increase in operating temperatures and pressures in reactor cores. Structural materials for new-generation nuclear reactors (Gen-IV) and prospective thermonuclear reactors must operate under more extreme conditions involving higher temperatures, pressures, and intense fluxes of ionizing radiation. Simultaneously, there is a pressing need for small-scale nuclear reactors of traditional design with exceptionally long autonomous operation to ensure stable power supply in extreme environments without the need for maintenance.

The main challenge in enhancing the power and longevity of nuclear systems—and thus their safety—lies in the degradation of structural materials under prolonged exposure to radiation in aggressive environments and at high temperatures. Traditionally, austenitic stainless steels are used as the primary structural materials. However, they present significant drawbacks related to the generation of long-lived radionuclides, especially nickel isotopes under neutron irradiation, complicating radioactive waste management.

Improving and stabilizing the properties of stainless steels without using nickel can be achieved by introducing dispersed hard oxides, typically yttrium oxide (Y_2O_3), resulting in a nanocomposite material known as oxide dispersion-strengthened (ODS) steel. Nanosized oxides, densely distributed within the ferritic-martensitic matrix, can stabilize dislocations and grain boundaries and serve as effective traps for radiation-induced point defects, thereby enhancing the radiation resistance of the material. As such, the oxide inclusions not only strengthen the material but also ensure structural stability under extreme service conditions.

However, the production of such materials faces technological limitations, particularly with conventional powder metallurgy techniques, including mechanical alloying. These methods involve complex processes, high costs, and limited scalability for industrial production. Likewise, traditional metallurgy fails to meet the required quality due to the inability to effectively incorporate yttrium oxide particles into the steel matrix.

Thus, the development of new technologies for producing ODS steels capable of overcoming these limitations is highly relevant. One promising direction is the use of liquid metallurgy methods, such as vacuum induction melting, using exogenous, endogenous techniques, or yttrium-containing master alloys. This approach enables the creation of steels with uniformly distributed yttrium oxide particles and enhanced operational performance.

To achieve even higher ODS steel properties, additional methods must be considered, particularly those involving severe plastic deformation (SPD). SPD enables the formation of ultrafine-grained (UFG) structures, significantly improving the mechanical and radiation properties of steel. Combining liquid metallurgy and SPD processes offers a viable solution to existing technological challenges and paves the way for the production of high-quality ODS steels for use in extreme environments.

This research involved a comprehensive analysis and validation of various exogenous and endogenous technologies for producing ODS steel ingots using vacuum induction melting. Based on the experimental results, the most promising melting technique was selected, providing optimal yttrium oxide content. A detailed study of the rheological properties of the obtained steel was conducted to build a unique property database necessary for accurate modeling of SPD processes.

The application of SPD aimed to significantly enhance the mechanical properties of the material, including strength and ductility, as well as its radiation resistance at elevated temperatures. This approach creates the potential for a high-quality final product suitable for critical applications in demanding industries.

The study of the new alloy's rheological properties across a temperature range of 600–1200 °C and strain rates of 0.5–15 s^{-1} enabled the creation of a data set for finite element modeling of deformation processes. Based on this modeling, the optimal SPD technique—radial-shear rolling—was selected, ensuring effective grain refinement and improved mechanical properties.

Experimental validation of radial-shear rolling at temperatures above 800 °C and a drawing ratio of 6.05 demonstrated the feasibility of achieving a UFG structure with grain sizes of 400–800 nm and enhanced mechanical properties. For the first time, the influence of this technique on cast structure transformation and defect evolution in ODS steel was thoroughly investigated.

The justification for applying the developed material and production technology in nuclear energy is essential for improving the safety and efficiency of nuclear reactors. The results contribute to advancing the scientific and technical field of developing new high-strength materials, aligning with the priorities of modern materials science and the needs of the nuclear industry.

Thus, the topic of this dissertation is highly relevant and focused on solving key challenges in nuclear energy through the development of innovative materials and technologies.

The purpose and objectives of the study.

The aim of the work is to research and develop a technology for producing dispersion-reinforced steel ingots and improving their structure to an ultrafine-grained state for promising use as a material for nuclear energy.

To achieve the stated goal, the following research objectives were defined:

1. Conduct a comprehensive literature and patent analysis of existing methods for producing dispersion-reinforced steel (ODS steel) and methods for improving the structure and properties of ingots;

2. Study methods for forming yttrium oxide inclusions in a steel matrix through a sequential experimental study of various exogenous and endogenous methods of introducing yttrium oxide into the melt, identifying and determining the most effective conditions for the stable formation of oxide inclusions in the steel matrix;

3. Study the rheological properties of the resulting ingot material to create a database and conduct computer modeling of ingot pressure treatment.

4. Study the effect of radial shear rolling on the evolution of casting defects in an ingot produced using the new technology, conducting a detailed analysis of defect evolution at each stage of deformation.

5. To study the effect of intense plastic deformation by radial-shear rolling on the evolution of the cast structure of steel ingots containing strengthening particles of yttrium oxide, with the aim of forming an ultrafine-grained structure of the material with improved properties.

Research Object. The research focuses on the technology of producing dispersion-reinforced steel ingots and subsequent processing using severe plastic deformation methods to improve their structure and properties.

The subject of this dissertation is the production of an ingot with reinforcing yttrium oxide inclusions by casting and subsequent processing of this ingot using severe plastic deformation methods to produce high-quality blanks intended for the production of nuclear power components.

Research methods.

The research methods included a literature review to review current approaches to producing ODS steel, melting in an induction vacuum furnace to produce ingots, and the use of optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) to study the microstructure and morphology of the material. Energy-dispersive spectroscopy (EDS) on SEM and TEM was used to analyze the elemental composition and distribution of yttria in the metal matrix, while SAED (Selected Area Electron Diffraction) and EBSD (Electron Backscatter Diffraction) analysis were used to determine the material's texture and crystallographic grain orientation. The rheological properties of the resulting material were studied using a plastometer to create a property database, which was then used in finite element modeling (FEM) of the rolling processes to optimize deformation parameters. Radial shear rolling (RSR) was used to improve the ingot microstructure. 3D evolution analysis using volume reconstruction based on defect cross-sections in CAD defect models was used to visualize structural changes at each rolling stage. This comprehensive approach enabled an in-depth study of ODS steel production methods and their characteristics.

The level of scientific development of the problem. Despite the significant volume of research conducted in this area, including both theoretical and experimental work, existing technologies for producing ODS steels often do not provide the required level of structure homogeneity and the resulting mechanical properties on a large scale. And they cannot provide the volume of production for industrial use. Difficulties in the uniform distribution of oxide inclusions and ensuring their stability during subsequent processing remain urgent tasks. In the context of increasing requirements for the operational characteristics of reactor structural materials, further in-depth study of the methods of forming the structure and dispersion of reinforcing particles seems necessary. This requires additional theoretical and experimental research to improve technologies and develop approaches that ensure the stability of properties and the possibility of scaling them for industrial production.

The main provisions submitted for defense.

Based on the conducted research, the following scientific propositions of the dissertation were formulated:

1. The impossibility of directly introducing dispersed yttrium oxide particles into the melt exogenously was established due to the weak interaction of yttrium oxide particles with the steel melt.

2. A new technology for the endogenous formation of strengthening dispersed yttrium oxide particles in steel was developed. This technology is based on the oxidation of metallic yttrium directly during smelting and subsequent intensive deformation treatment of the ingot using radial-shear rolling.

3. The evolution of transverse through defects in a steel ingot was studied for the first time using radial-shear rolling.

4. The effectiveness of radial-shear rolling of steel ingots with strengthening Y_2O_3 particles in achieving an ultrafine-grained state and improving material properties was experimentally confirmed.

Scientific novelty of the study:

1. It was experimentally established that the direct introduction of exogenous yttrium oxide particles into the steel melt is inefficient due to the high wetting angle of more than 120° and the significant difference in densities (a factor of 1.58) between the introduced particles (5.01 g/cm³) and the steel melt (7.9 g/cm³).

2. The technological parameters of melting that ensure the endogenous formation of yttrium oxide particles in the steel melt were determined as follows: temperature – 1550±10 °C, holding time – 1 minute, and yttrium content – 2%.

3. For the first time, a database of flow curves for steel containing reinforcing yttrium oxide particles was obtained in the deformation rate range of 0.5 s^{-1} to 15 s^{-1} and the temperature range of 600 °C to 1200 °C.

4. The effectiveness of radial-shear rolling on the evolution of the cast structure of steel ingots containing strengthening yttrium oxide particles was established, which leads to the formation of an ultrafine-grained structure with a grain size of 400–800 nm.

5. The possibility of accelerated processing of a dispersion-strengthened steel ingot by radial-shear rolling was identified, which ensured a reduction of the volume of the through model defect from the initial 480 mm^3 to the final 155 mm^3.

Theoretical and practical significance.

This dissertation has both theoretical and practical significance. A new technology for producing steel with strengthening yttrium oxide particles has been developed. This technology involves introducing metallic yttrium into the melt, followed by its endogenous oxidation, casting, and processing the resulting ingot by radial shear rolling. This technology has potential for scalability for industrial use as a structural material in nuclear power engineering. New approaches to introducing yttrium oxide into the steel matrix of grade 12X13 steel were studied. A database of the rheological properties of molten steel with Y_2O_3 inclusions was obtained and used to model plastic deformation processes and determine optimal radial shear rolling parameters. Based on the created database, plastic deformation processes in steel were simulated. The effect of RSR on the microstructure and mechanical properties of steel, as well as fragmentation processes in the cast structure, were studied. From a practical perspective, a technology for producing ODS steels has been developed, ensuring their suitability for high-temperature and radiation-exposed applications. The feasibility of using radial shear rolling to form an ultrafine-grained structure, which improves strength and mechanical properties, has been confirmed. The technology for oxidizing metallic yttrium during the smelting process has industrial potential due to its scalability and compatibility with existing equipment. The results obtained can be applied in the development of new structural materials for nuclear power, aerospace, and energy, as well as for improving operational reliability in extreme conditions.

The main findings of the dissertation were presented at scientific and practical conferences and published in journals indexed in Scopus and in national peer-reviewed publications.

Publications: Eight papers have been published on the topic of the dissertation research. Of these, five are in the Scopus and Web of Science databases, two are in the proceedings of international scientific and practical conferences, and one is in publications recommended by the Committee for Quality Assurance in Education and Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan.

List of publications in Scopus and Web of Science databases:

1. Arbuz A., Kawalek A., Panichkin A., Ozhmegov K., Popov F., Lutchenko N. Using the radial shear rolling method for fast and deep processing technology of a steel ingot cast structure // Materials. 2023. Vol. 16. No. 24. Article No. 7547.(Q1-Web of Science)

2. Arbuz A., Panichkin A., Popov F., Kawalek A., Ozhmegov K., Lutchenko N. Modeling the evolution of casting defect closure in ingots through radial shear rolling processing // Metals. 2024. Vol. 14. No. 1. Article No. 53(Q2-Web of Science).

3. Arbuz A. S., Panichkin A. V., Popov F. E., Lutchenko N. A., Volokitina I. E. Using the radial shear rolling method for deep development of the cast structure of ingots of special materials // Metallurgist. 2024. Vol. 67. No. 11-12. P. 1826–1836(Q4-Web of Science).

4. Popov F., Lutchenko N., Panichkin A., Lezhnev S., Panin E., Vinogradov L., Arbuz A. Modelling the evolution of casting defect closure by radial shear rolling // Journal of Chemical Technology and Metallurgy. 2024. Vol. 59. No. 1. P. 197–206. (34 Percentile – Scopus)

5. Panichkin A., Popov F., Lutchenko N., Beldeubayev A., Samokhvalov I., Arbuz A. Research of injection methods for Y_2O_3 nanoparticles into nickel-free stainless steel during induction vacuum remelting // Journal of Chemical Technology and Metallurgy. 2024. Vol. 59. No. 1. P. 173–182.(34 Percentile – Scopus)

List of Publications in Journals Recommended by the Committee for Quality Assurance in the Field of Education and Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan:

1. Найзабеков А.Б., Попов Ф.Е., Паничкин А.В., Лежнев С.Н., Арбуз А.С. Моделирование применимости метода радиально-сдвиговой прокатки для закрытия дефектов литой структуры стального слитка // Труды университета. – 2023. – №4(93). – С. 67-71.

List of Presentations at International Scientific Conferences:

1. Popov F., Panichkin A., Lutchenko N., Beldeubayev A., Samokhvalov I., Arbuz A. The Various Ways Injection of Y2O3 Particles into the Nickel-Free Stainless-Steel Melt by Experimental Vacuum Induction Melting // METAL Conference Proceedings. 2023.

2. Попов Ф., Паничкин А., Лученко Н., Белдеубаев А., Самохвалов И., Арбуз А. Введение частиц Y_2O_3 в расплав безникелевой нержавеющей стали методом вакуумной индукционной плавки // Proceedings 32nd International Conference on Metallurgy and Materials. – 2024. – С. 59–63.

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Structure of the dissertation. The dissertation consists of an introduction, four sections, and a conclusion, presented on 149 pages. It contains 85 figures, one table, 202 references, and four appendices.