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Effect of Alloying Oxides Y_2O_3 and ZrO_2 on the Microstructure of Austenitic Steel $18Cr_{10}NiTi$

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Nanomaterials are materials of new generation with unique characteristics which are not characteristic for other materials. Nanofilms, nanoprocessors, nanorobots – the more well known technologies, but demands of modern industry, electronic industry, power industry dictate the development of nanomaterials in higher scales. The use of nanomaterials is very promising direction in nuclear power. Conditions of operation of structural materials in nuclear reactors, namely, high temperature (higher 300°C for thermal reactors and higher 600°C for fast reactors) which together with high density of neutrons (~10^{21}-10^{22} neutrons/cm^{-2}) cause the degradation of initial physical-chemical characteristics and the significant dimension changes. These factors influence considerably on safe and long-term operation period of reactor.

Keywords: Nanomaterials**,** ODS steel, austenitic, TEM, microstructure, oxide.

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1. INTRODUCTION

Austenitic steels are used in nuclear power as material for pressure vessel internals and fuel claddings in fast reactors, and in thermal reactors as claddings and other internals. In comparison with ferritic-martensitic steels they are characterized by higher high-temperature strength, but have lower radiation resistance [1]. The problem solution of the improvement of austenitic steels radiation resistance with simultaneous increasing of high-temperature resistance is possible by the nanostructural state production in these alloys, that characterized by the presence of nanosized particles (~2-10 nm) with high density (~10^{15}-10^{16}cm^{-3}) and uniform distribution into the matrix. Increase of radiation resistance is caused by the large extent of boundaries "matrix-nanoparticles", which are the effective sinks for radiation defects [2-4]. Production of oxide precipitates of extremely low dimensions (~some nanometers) and with high density is one of possible way of improvement of steels characteristics. Production of such material by traditional methods, such as melting, doesn't permit to obtain satisfactory quality and resistance of austenitic steels. In the case of traditional way of steels production is not possible to obtain high densities of precipitates and their nanodimension. Unique technology of steel production was developed. This technology was named "mechanical alloying". The proposed method allows obtain steels with strengthening nano precipitates which outperform steels obtained by traditional methods. Now steels dispersion-strengthened by oxides are very topical. Thermodynamically stable nanooxides may serve as such nanoparticles and steels, strengthened by such particles are named oxide dispersion-strengthened steels (ODS steels).

2. MATERIALS AND METHODS

Commercial austenitic steel 08Cr18Ni10Ti was used as basic matrix. Material was prepared with mechanical alloying [5-8]. Ribbon fragments with dimensions 3x3 mm^2 or the initial steel powder were mixed with 0,5%wt. of oxide nanopowder and mechanically alloyed in argon in high-energy ball mill (rotation rate was 480 rpm) during 1-10 hours. The balls made of steel and with of different diameters were used. Obtained powder consisted of agglomerated particles with multi modal distribution and substantial size distribution within the range of some μm to 500 μm and more. Further the fraction with the size less than 300 μm was used in our work. All kinds of mechanical treatment from powder compacting to rolling of compacted blank were carried out at room temperature. In addition, mechanical treatments were alternated with short-time annealing in vacuum at temperature 1200°C. In the result the bands of ODS-steel 18Cr10NiTi with thickness 200 μm were obtained.Initial steel microanalysis showed on Fig.1, that its composition by main elements complies with State standard. Characteristics of used oxide nanopowders are presented in table 1.

Table 1 – Parameters of nanopowders of system Y_2O_3-ZrO_2

Y_2O_3 Content, % mol.	Particle size* (crystallite size), nm
80	16.5
20	29.0
8	14.3

3. RESULTS AND DISCUSSION

Microstructure investigation showed that particles were represented as quite dense formations without visible boundaries and with a certain quantity of micropores with size from several fractions of micron to few microns.

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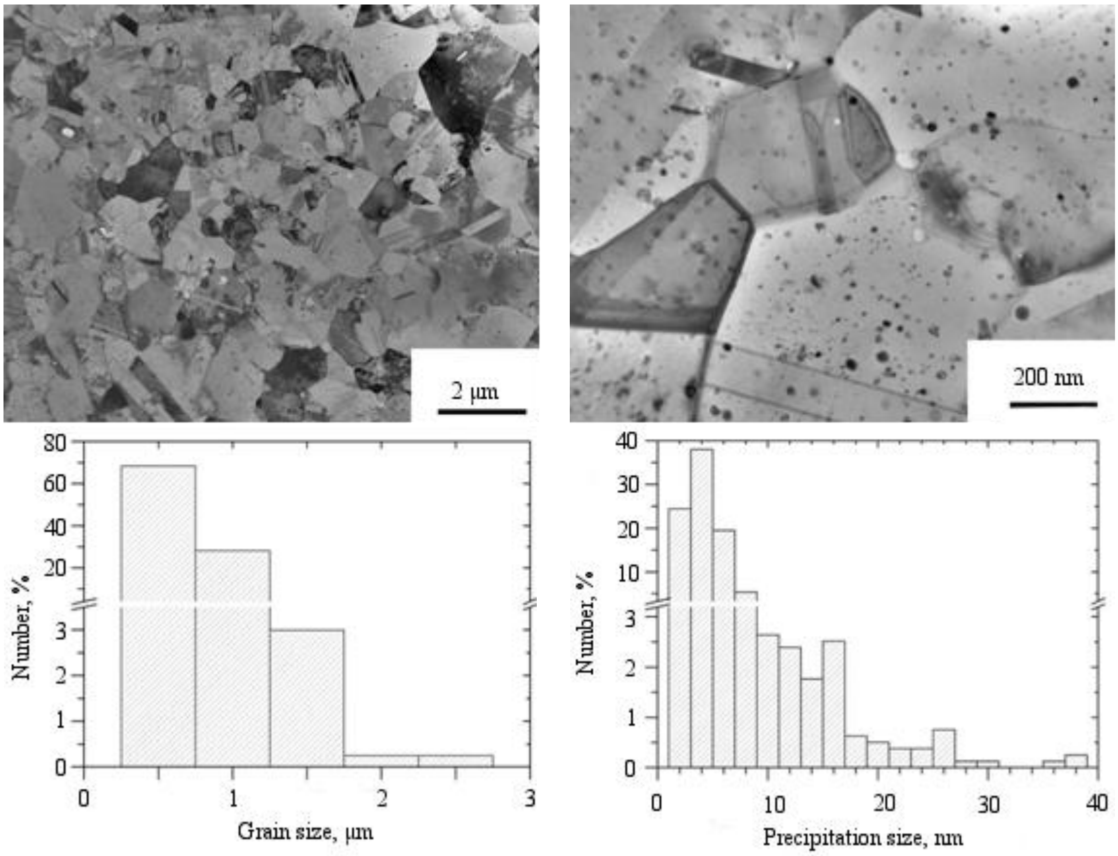
As the result of characteristic radiation data collection it has been found, that all basic elements (Fe, Cr, Ni, Zr, Y, Mn and Si) had been distributed uniformly by cross section of particles. Spherical particles ("balls") diameter size of 2-3 mm, were selected for electron-microscopic study. These particles were obtained by mechanical alloying of fast quenched steel strips and oxide powder (with composition 80 mol% Y_2O_3-20 mol% ZrO_2) during 10 hours. These particles were annealed at 1200°C during 1 hour, squashed and annealed again. Electron microscopy images of the structure and treatment results are shown in Fig.1.

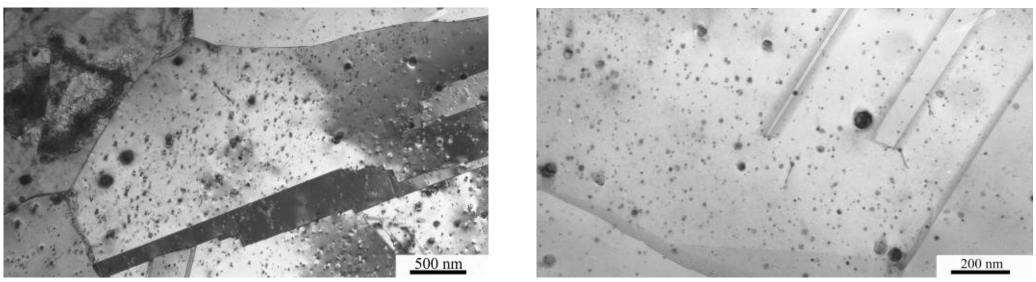


a b

Fig. 1 – Structure of steel spherical particle ("ball"), obtained by mechanical synthesis, pressed and annealed at 1200°C: a – grains structure and grain size distribution, b – precipitates appearance and size distribution

As can be seen, there are a lot of fine precipitates inside of grains. Electron microprobe analysis had showed that precipitates contain titanium, yttrium, zirconium, oxygen and some other elements. Supposedly they have composition $Y_2(Ti, Zr)_2O_7$ but this result must be clarified. Besides the fine precipitates there is also a low quantity of larger particles size of up to 100 nm. According to microanalysis data these particles are titanium oxycarbonitrides and titanium oxides. Nano-sized powder 80mol% Y_2O_3-20mol% ZrO_2 was used as oxide on mechanical alloying and fast quenched ribbons were used as steel.

Microstructure images of 18Cr10NiTi steel, alloyed by Y_2O_3 - ZrO_2 nanooxides, and oxide precipitates size distribution histograms are shown on Fig. 2(a,b). Grain structure was the same approximately for all samples, average grain size was 1.2-2.0 µm. Significant concentration of precipitates and its near-uniform distribution are observed for all samples. This is a dominant condition on ODS steel production.



a b

Fig. 2 – a) typical microstructure for 18Cr10NiTi-ODS sample; b) typical image of oxide precipitates in 18Cr10NiTi-ODS sample.

Precipitations size varied from some of nanometers to hundreds of nanometers, but the last were a few orders less, thus, its contribution to concentration and average size was negligible. Note, that calculations were performed on large data array (more than 1000 precipitates per composition). Table 2 contains generalized data on precipitates characteristics of specimens with different composition.

Table 2 – Microstructure characteristics of ribbons, alloyed by oxides of different composition

Alloying oxide composition, mol%	Average grain size, µm	Average oxide precipitates size, nm	Density of precipitates, cm^{-3}
$8Y_2O_3$ - $92ZrO_2$	2	13	$1,7 \cdot 10^{15}$
$20Y_2O_3$ - $80ZrO_2$	1,5	13	$4,5 \cdot 10^{15}$
$80Y_2O_3$ - $20ZrO_2$	1,2	10	$7,3 \cdot 10^{15}$

As it is seen from table the best characteristics are observed in ODS steel specimen, obtained using $80Y_2O_3$-$20ZrO_2$ nanopowder. In this specimen minimal average grain and precipitates size, maximal precipitations density and most narrow its size distribution are observed. Large precipitations (size of more than 50 nm) are practically absent. Determination of formed oxides composition is a goal of the following investigations.

4. CONCLUSION

Features of austenitic 18Cr10NiTi ODS steel mechanical alloying by Y2O3 - ZrO2 nanooxides were studied. It is shown that powders contain austenitic and ferritic phases after mechanical alloying. Moreover, the quantity of mechanically induced ferrite mainly depends on quality of the initial steel before alloying. In the case of equiaxial powders, obtained by mechanical abrasion, the quantity of ferrite is considerably higher than on using fast-quenched ribbons. Austenitic and ferritic phases of steel powders, mechanically alloyed by nanooxides, have approximately the same crystallite size. But it is observed the tendency of crystallite size decreasing and increasing of the microstrains level with the yttria content increasing in basic oxide nanopowders. Annealing at temperatures above 800oC leads to austenization, and, at the same time, crystallite size increases and microstrains level decreases.

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