**ANNOTATION**

of the PhD degree «6D071000 – Materials science and technology of new materials» dissertation of

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# LOCALIZED PLASTIC DEFORMATION IN METALS (CU, FE) AND AUSTENITIC STAINLESS STEELS (12CR18NI10TI, AISI 304), IRRADIATED WITH NEUTRONS

**General characteristics of the work.** The dissertation work presents experimental results of materials science studies of localization of plastic deformation in samples of model metallic materials (Fe, Cu) and reactor austenitic steels (12Cr18N10T, AISI 304) irradiated with neutrons to various damaging doses.

**Relevance of the topic.**

Scientifically based extension of the service life of nuclear reactors to maintain their generating capacity without investing in new capital construction is an important stage in the development of the global and Kazakhstani nuclear industry. The service life of nuclear power reactors depends on the performance of the core components, namely, the reactor vessel and internals. The service life of internal devices made of austenitic steels, type 12X18H10T and AISI 304, is limited by the manifestation of radiation-stimulated effects such as swelling, stress corrosion and low-temperature radiation embrittlement.

Low-temperature radiation embrittlement (LTRE) of austenitic steels is caused by a decrease in the uniform plasticity of the material with an increase in the damaging dose of neutron irradiation. In non-irradiated metals, deformation mechanisms localized at the micro level, such as dislocation defect structures, large stacking faults and twins, lead to strengthening of the material during deformation and prevent localization of deformation at the macro level (formation of a geometric “neck”). As the dose of neutron irradiation increases, the number of possible types of dislocation structures decreases, the ability of the material to strain hardening is suppressed, which leads to a decrease in uniform plasticity and premature formation of a “neck.” There is a dose of neutron irradiation, after reaching which a neck in austenitic steels forms immediately after the yield point, bypassing the stage of uniform deformation, 15–40 displacements per atom (dpa), which is comparable to the calculated irradiation dose of the internals of nuclear reactors (for example, fragments of the baffle of the VVER-1000 reactor) after 30–40 years of operation (up to 50 dpa).

One of the promising directions for solving the problem of LTRE is the use of deformation mechanisms localized at the microlevel, the stimulation of which can cause additional strengthening during the deformation process and prevent localization at the macrolevel. Such processes include the martensitic $\gamma \rightarrow \alpha'$-transformation. During plastic deformation at relatively low temperatures (<100°C), α'-martensite grains are formed in the austenitic matrix of metastable stainless steels, which are characterized by significantly greater strength compared to austenite.

Over the past 10 years, cases have been reported in which the low-temperature plasticity of metastable austenitic steels of type AISI 304 and 12Cr18Ni10Ti after high-dose (>40 dpa) neutron irradiation significantly exceeded the expected one. Currently, there is a scientific consensus on the decisive role of the martensitic $\gamma \rightarrow \alpha'$ transformation in the formation of plasticity in these cases. It is noteworthy that this effect is observed in an extremely wide range of damaging doses from 7 to 150 dpa.

Dynamic strain aging is of particular interest. During plastic flow at elevated temperatures in a certain range of strain rates, periodic blocking of moving dislocations by impurity atoms occurs. This process can be considered as a source of additional strengthening of the material.

Despite numerous studies devoted to the study of the processes of embrittlement and destruction of irradiated structural reactor materials, issues related to establishing the causes and patterns of development of concentrated deformation at different scale levels are clearly insufficiently covered. Systematized knowledge of the patterns and features of the manifestation of deformation localization in irradiated structural materials is of great scientific and practical interest in the framework of solving the problem of LTRE and safely extending the service life of nuclear reactors.

**The purpose of the dissertation work was** to establish the features of localized plastic flow in samples of model metallic materials (Fe, Cu) and reactor austenitic steels (12Cr18N10Ti, AISI 304) irradiated with neutrons in the reactors of Kazakhstan WWR-K and BN- 350.

**Research objectives:**

– To conduct mechanical tests for uniaxial stretching of samples of copper, ARMCO iron and austenitic steels exposed to neutron irradiation in the WWR-K and BN-350 reactors, to determine their physical and mechanical characteristics and parameters of the formation of a geometric neck.

– To reveal the effect of neutron irradiation on the dynamic deformation aging of ARMCO-iron deformed at elevated temperatures.

– To determine the features of martensitic $\gamma \rightarrow \alpha'$-transformation in steels 12Cr18N10Ti and AISI 304 and to identify its effect on the localization of deformation.

– To establish a connection between the microstructure formed as a result of prolonged high-dose irradiation in the BN-350 reactor and the features of localized plastic flow in 12Cr18N10Ti austenitic steel samples.

**The object of the study.** Modular metal materials with FCC and BCC crystal lattices (Fe, Cu) and reactor austenitic steels (12Cr18N10Ti, AISI 304) irradiated with neutrons in the WWR-K and BN-350 reactors.

**The subject of the study.** Localized plastic flow in neutron-irradiated metallic materials.

**Provisions submitted for defense:**

1. Neutron irradiation in the WWR-K reactor to a dose of 0,05 dpa leads to a decrease in the value of “true” local deformation corresponding to the beginning of the formation of a stable “neck” in austenitic steels 12Cr18N10Ti and AISI 304 by 10% due to the suppression of strengthening deformation mechanisms localized at micro level; while the magnitude of the “true” critical stress remains constant.

2. Defects of a radiation nature that arise in the structure of ARMCO iron irradiated with neutrons in the WWR-K reactor in the range of 0,006–0,06 dpa lead to the suppression of strain-induced dynamic aging in the process of uniaxial stretching at temperatures of 100–300°C, thereby contributing to the manifestation of more complex types of dislocation structure.

3. Additional strengthening of AISI 304 steel due to a more intense deformation-induced martensitic transformation increases the uniform deformation by 2 times compared to 12Cr18N10Ti steel both in unirradiated and in irradiated states up to 0,05 dpa in the WWR-K reactor, which makes it possible to consider deformation- induced martensitic transformation as an effective way to combat premature strain localization at the macro level in austenitic steels.

4. In metastable austenitic steel 12Cr18N10Ti, irradiated to high damaging doses of 45–57 dpa, an increase in the irradiation temperature from 305 to 405°C leads to an increase in the plasticity of the material by 2 and a change in the mechanism of localized deformation: from the development of a stationary geometric “neck” to the formation a movable “neck” and its movement from one edge of the sample’s working area to the other.

**Experimental and theoretical methods.** To implement the task, modern methods of studying the structure and properties of modular metals and austenitic stainless steels were applied, including low-temperature mechanical tensile tests with simultaneous control of the phase composition in local micro-volumes of the sample and dynamic image correlation, optical and electron microscopy, microhardness, isochronous annealing.

**Scientific novelty and main results of the work:**

– For the first time, a systematic and comprehensive materials science study of the localization of deformation in polycrystalline metals with BCC (Fe) and FCC (Cu and austenitic steels) crystal lattices, irradiated with neutrons to low (<1 dpa) and high (>40 dpa) damaging doses, was carried out.

– For the first time, the effect of increasing uniform plasticity and suppressing dynamic strain aging in neutron-irradiated ARMCO iron was discovered.

– Using the digital image correlation technique, two different deformation mechanisms were identified in highly irradiated (45–57 dpa) austenitic steel 12Cr18N10Ti depending on the irradiation temperature (300–400°C) in the BN-350 reactor. As a result of microstructure studies, it was found that the determining factor in the formation of plasticity in this case is the martensitic $\gamma \rightarrow \alpha'$ transformation and the formation in the structure of the material of large defects such as voids and secondary phases and the creation of areas free from small defects, such as black-dots or small loops.

– For the first time, curves of the deformation-induced martensitic $\gamma \rightarrow \alpha'$ transformation in highly irradiated (>40 dpa) austenitic steel 12Cr18N10Ti were obtained, and the kinetic parameters of the process were determined.

**The reliability and validity** of the results obtained is due to the use of up-to-date literary data published in peer-reviewed thematic journals; the use of modern and relevant research methods, maintaining constant contacts with foreign collaborators. The results of the dissertation were published in highly rated scientific journals.

**Practical significance of the study.** The experimental results obtained can be used to predict the performance of internals of nuclear power plants made of stainless austenitic steels, in order to justify the safe extension of the service life of existing industrial nuclear reactors; as well as in the development of the fundamental principles of the physics of plastic deformation of highly irradiated metals and alloys, which will certainly be useful in the nuclear energy industry of Kazakhstan.

**Connection of the dissertation topic with plans for scientific works.** The dissertation work was carried out within the framework of research under the grant funding program of the Ministry of Education and Science of the Republic of Kazakhstan on the topics: 0380GF/4 «Development of physical foundations for solving the problem of low–temperature (20-300°C) radiation embrittlement of structural materials for nuclear reactors», AP08052488 «Control of plasticity of reactor materials after high-dose neutron irradiation».

**The work was presented** at seminars of the Department of Radiation Solid State Physics of the RSE INP RK, as well as at 14 international scientific conferences, including: TMS 2021 Annual Meeting & Exhibition, 2021 (Online, USA); 20th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 2022 (Aspen, USA); NuMat2022: The Nuclear Materials Conference, 2022 (Ghent, Belgium).

Based on the dissertation materials, 13 articles were published, including 5 articles in journals recommended by the CCSON, 4 works were published in the proceedings of international conferences, indexed in the Scopus and Thomson Reuters databases. 4 articles were published in foreign scientific journals:

1. ***Merezhko, M.S.*** Merezhko, D.A., Rofman, O.V., Dikov, A.S., Maksimkin, O.P., Short, M.P. Macro-Scale strain localization in highly irradiated stainless steel investigated using digital image correlation // Acta Materialia. – 2022. – Vol. 231 – P. 117858 (IF=9,4; Q1. CiteScore=15,1; 96$^{th}$ percentile ).

2. Merezhko, D.A. Gussev, M.N., ***Merezhko, M.S.***, Rofman, O.V., Rosseel, T.M., Garner, F.A. Morphology and elemental composition of a new iron-rich ferrite phase in

highly irradiated austenitic steel // Scripta Materialia. – 2022. – Vol. 215 – P. 114690. (IF=6; Q1. CiteScore=10,7; 94th percentile ).

3. ***Merezhko, M.S.***, Merezhko, D.A., Tsai, K.V. Mechanical Properties of Neutron-Irradiated Armco Iron upon Plastic Deformation at Elevated Temperatures // Physics of Metals and Metallography. – 2022. – Vol. 123, № 2. – P. 193-199 (IF=1,2; Q3. CiteScore=2,0; 30th percentile ).

4. ***Merezhko, M.S.,*** Maksimkin, O.P., Merezhko, D.A., Shaimerdenov, A.A., Short, M.P. Parameters of Necking Onset during Deformation of Chromium–Nickel Steel Irradiated by Neutrons // Physics of Metals and Metallography. – 2019. – Vol. 120, № 7. – P. 716-721 (IF=1,2; Q3. CiteScore=2,0; 30th percentile ).

In all publications, the dissertation author took part in writing the final text (if the dissertation author is listed as the 1st author - the main part of the manuscript), and determining the conclusions. In some cases he acted as a corresponding author.

**Personal contribution of the dissertation**

During the research, the author conducted comprehensive materials science studies of the localization of deformation in polycrystalline metals, based on a literature review, analyzed the results obtained and formulated conclusions.

**Scope and structure of the dissertation.**

The dissertation work contains a list of notations and abbreviations used, an introduction, the main part consisting of three sections, a conclusion, application and a list of literary sources used. The volume of the dissertation is 125 printed pages, 66 figures, 27 tables and 160 literary sources.