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USE OF PHYSICAL METHODS AND ELECTRON MICROSCOPY IN ANALYZING STRUCTURAL CHANGES IN Ni_3Fe ALLOY

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Results of studies of structural and mechanical properties of Ni_3Fe *alloy are presented. The densities and types of dislocations in the alloy, the parameters of dislocation interaction, and the energy expended in deformation of* Ni_3Fe *specimens in various initial states are estimated. The strain hardening behavior of ordered* Ni_3Fe *alloy is shown to be influenced by a number of external factors, such as deformation or* γ*-irradiation.*

Introduction. Strain hardening of metals and ordered alloys depends strongly on the structure of these materials. In such alloys, the sliding dislocation structure of such alloys undergoes substantial transformations upon transition from a disordered to an ordered state. This allows one to obtain a broad spectrum of dislocation substructures [1–3] in the same alloy and, hence, study the evolution of the substructure during plastic deformation, irradiation by particles, or heating [1–4].

In this connection, it is of interest to elucidate the strain hardening behavior of alloys that undergo order–disorder phase transitions. In this study, self-ordering alloy Ni_3Fe [5] was used as an example since fairly extensive data are available on the long-range atomic ordering in this material after various thermal treatments [6]. In this alloy, an order–disorder phase transition occurs at $T = 802$ K (the $L1_2$ superstructure is a disordered solid solution with the A1 structure). This paper reports results of studies of the strain hardening behavior of Ni_3Fe alloy in various structural states after plastic deformation, annealing, or irradiation.

Materials and Experimental Procedure. Alloy Ni_3Fe in the ordered and disordered states was studied. The state of long-range atomic order was attained by annealing at 810 K followed by cooling to room temperature at a cooling rate of 5 K/24 h. The degree of long-range atomic order η was about 0.9, and the dimensions of the antiphase domains were 25–35 nm [9]. The state with short-range order was obtained by quenching specimens in water from a temperature of 1000 K. Both polycrystals and single crystals were studied.

The specimens were irradiated with γ-quanta from a ^{60}Co isotope source with radiation intensity of 278 R/sec for 120 h, which corresponded to a total irradiation dose of 10^7 R. The specimen temperature was higher than 40°C. To study the dislocation structures resulting from deformation of both the initial and irradiated materials, we compressed single crystal specimens along the ⟨111⟩ axis at a rate of $10^{-3}sec^{-1}$ at room temperature. This orientation was chosen since it is the one of multiple sliding (six sliding systems were equally loaded). Polycrystalline specimens with a mean grain size of about 40 μm were deformed by rolling at room temperature. Electrical resistance was measured by the standard four-point method [7].

The structures of the specimens after different treatments were studied by transmission diffraction electron microscopy and x-ray diffractometry. The foil was examined on an ÉMB-100AK electron microscope, and x-ray patterns were recorded by a DRON-3 diffractometer. The change in the defect structure of the specimens after irradiation and slight deformation was studied by Rutherford backscattering (RBS) using the "channeling effect" along the ⟨111⟩ direction.

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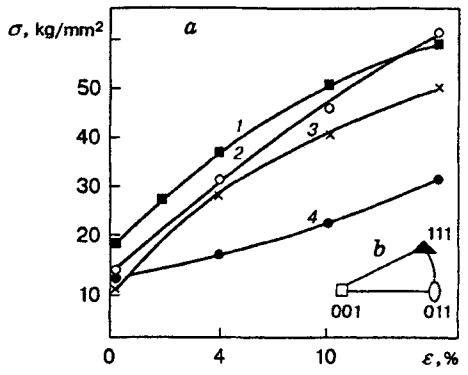

Fig. 1

Results and Discussion. Figure 1a shows curves of the strain stress applied along the ⟨111⟩ direction versus strain ε for Ni_3Fe single crystals after various treatments: long-range order annealing, annealing with subsequent irradiation (irradiation dose $2 \cdot 10^6$ R), quenching followed by irradiation, and short-range order quenching (curves 1–4, respectively, and points are experimental data), and Fig. 1b shows the standard stereographic triangle.

The work done in deformation depends on the degree of imperfection and the evolution of the defect substructure of a single crystal under loading, other conditions being equal. This is confirmed by the strain hardening curves of Ni_3Fe single crystal specimens subjected to different treatments. This circumstance was used in analyzing the dependences obtained. The deformation work $A \sim \sigma\Delta\varepsilon$ [8], i.e., the total work done to deform a specimen by $\varepsilon = 15\%$ at $T = 293$ K, is equal to the area under the σ–ε curve. The areas determined from the experimental strain hardening dependences of Ni_3Fe single crystals lead to the following chain of inequalities: $A_1 > A_{1+\gamma} > A_{2+\gamma} > A_2$. Here A_1, A_2, $A_{1+\gamma}$, and $A_{2+\gamma}$ is the work done to deform the following specimens: an annealed single crystal with a high value of the long-range order parameter ($\eta \to 1$); a disordered single crystal quenched from the temperatures exceeding the T_K of the order–disorder transition ($\eta \to 0$), a single crystal annealed and irradiated with γ-quanta, and a disordered and irradiated single crystal.

For the material states considered, the deformation energies are related as follows:

$$E_1 > E_{1+\gamma} > E_{2+\gamma} > E_2$$

(the subscripts are the same as in formula for A).

After active plastic deformation of Ni_3Fe, the dislocation density increases appreciably (from 10^8 to 10^{10} cm^{-2}). It is well known [1–3] that the most prominent increase in the dislocation density is usually observed at the second stage of hardening (3–20%). At the third stage (20–60%), the rate of dislocation accumulation decreases, and at the fourth stage ($\varepsilon > 60\%$), the dislocation density increases at constant velocity. The physical nature of the deformation mechanism differs from stage to stage. At the same time, the order–disorder transition does not affect the nature of the stages but strongly influences their duration [1], i.e., the duration of the stages depends on the structural state of the alloy and on the orientation of the single crystal during deformation. In this paper, the strain range studied is limited by the second stage. In self-ordering alloys, an increase in the degree of strain leads to an increase in the dislocation density and accumulation of antiphase boundaries on the sliding surface [5]; distortion of the long-range atomic order in the ordered state is also possible. Note that the latter assumption is still a widely debated topic.

The dislocation density ρ in Ni_3Fe specimens subjected to various treatments was determined by transmission electron microscopy on foils (see Table 1). In a nonirradiated, quenched specimen and an annealed nonderformed specimen, the dislocation densities are shown to be identical, and, being deformed by 25%, the alloy has a network-cellular substructure with a dislocation density of $2.5 \cdot 10^{10}$ cm^{-2}. Analysis of transmission electron microscopy images showed that the irradiation only results in an insignificant decrease in the dislocation density. The data obtained suggest that the plastic deformation of the irradiated specimens

TABLE 1

Deformation, %	ρ, cm^{-2}			
	Annealing	Annealing + irradiation	Quenching	Quenching + irradiation
0	$(1{,}0 \pm 0.2) \cdot 10^8$	$(0.7 \pm 0.2) \cdot 10^8$	$(1.0 \pm 0.2) \cdot 10^8$	$(1.0 \pm 0.2) \cdot 10^8$
10	$(1.0 \pm 0.1) \cdot 10^{10}$	$(0.8 \pm 0.1) \cdot 10^{10}$	$(1.1 \pm 0.1) \cdot 10^{10}$	$(1.8 \pm 0.1) \cdot 10^{10}$
15	$(2.0 \pm 0.2) \cdot 10^{10}$	$(2.1 \pm 0.1) \cdot 10^{10}$	$(1.6 \pm 0.2) \cdot 10^{10}$	$(1.9 \pm 0.1) \cdot 10^{10}$

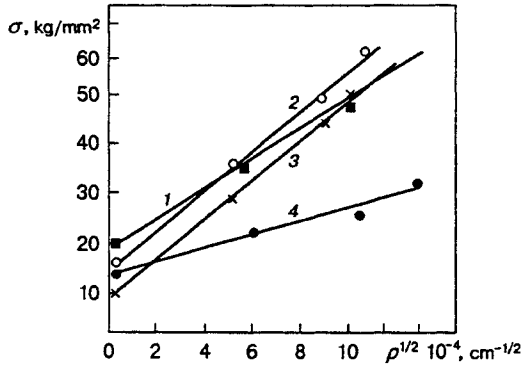

Fig. 2

is accompanied by generation of a smaller number of dislocations and dislocation loops. This effect becomes more pronounced as the degree of strain increases. Deformed irradiated specimens that were in the disordered state prior to irradiation have superdislocations. This indicates partial ordering of the disordered solid solution during the irradiation, i.e., increase in the long-range atomic order in it.

An important parameter of various theories of strain hardening is the parameter α of the dislocation interaction [5], which relates the flow stress σ and the dislocation density ρ:

$$\sigma = \sigma_f + m\alpha G|\boldsymbol{b}|\rho^{-1/2}.$$

Here σ_f is the strength to deformation of a nondislocation nature, m is the Schmidt factor, $|\boldsymbol{b}|$ is the absolute value of the Burgers vector of a sliding dislocation, and G is the shear modulus. The parameter α can be determined from the dependence $\sigma = f(\rho^{1/2})$, which for Ni_3Fe alloy in various initial states is shown in Fig. 2 (the notation of the curves is the same as in Fig. 1). In the strain range studied, the indicated dependences are approximated by straight lines. The values of α determined from the slope of these straight lines are as follows: 0.9 after annealing (the ordered state), 1.2 after annealing and irradiation, 0.4 after quenching (disordered state), and 1.2 after quenching and irradiation. As can be seen, the minimum value of α is observed in the quenched material and the maximum value is in the material irradiated after annealing and quenching. Thus, the irradiation enhances dislocation interaction, and deformation in the irradiated material is ensured by a lower dislocation density compared to the nonirradiated material. It should be noted that these processes take place in a material in which there is no marked increase in the concentration of radiation-induced defects (the number of radiation-induced vacancies is comparable with that of quenched-in vacancies [10]).

The results of transmission electron microscopy indicate the formation of long-range atomic order in quenched specimens subjected to γ-irradiation. This is confirmed by the x-ray diffraction studies performed. Analysis of the diffraction intensity profiles obtained by x-ray diffractometry in the vicinity of the superstructural reflection (100) indicates that Ni_3Fe alloy with a short-range atomic order (quenched state) becomes partially ordered after γ-irradiation. The long-range atomic order parameter estimated from the ratio of the total intensities of the superstructural and main (200) reflections is characterized by $\eta = 0.2 \pm 0.1$. The apparent dimensions of the coherent scattering regions calculated from the half-width of the superstructural

reflection via approximation was found to equal (25 ± 5) nm. The insignificant quantitative change in the intensities of the main and superstructural reflections for the specimen with a long-range order after irradiation does not make it possible to estimate the long-range order parameter in it. The assumption that irradiation of disordered specimens leads to the formation of a long-range order in them is indirectly confirmed by deformation studies of the irradiated and nonirradiated materials. Results of Mossbauer studies [11] provide evidence for redistribution of atoms in the solid solution under γ-irradiation.

This conclusion is also confirmed by energy spectra obtained in Rutherford backscattering studies. Analysis of these spectra suggests that deformation of pre-irradiated single crystals is accompanied by generation of a smaller number of displaced atoms compared to the number of such atoms in deformed but nonirradiated samples.

Comparison of the dislocation densities and types, parameters of dislocation interaction, and works done to deform Ni_3Fe specimens in various initial states allows the following conclusion to be drawn. An increase in the dislocation interaction in the irradiated material leads to local structural changes near defects and to partial atomic ordering. Small-angle x-ray scattering studies of the material structure [4] provide an indication of both a change in the mean imperfection of the medium and redistribution of the electron density in the material. Indeed, the data obtained by this method for initially ordered, irradiated and nonirradiated Ni_3Fe alloy are indicative of the formation of a special structural state [4]. A possible cause of changes in the strain hardening curves in irradiated specimens is redistribution of the electron density compared to the initial state, which leads to local structural changes near defects and partial atomic ordering [12].

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