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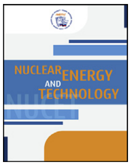

X-ray radiography investigation of structural conditions of Fe-15Cr-35Ni-11 W steel irradiated by ion-plasma fluxes

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Abstract

It was found that structural-phase transformations induced by radiation in the highly doped heat resistant Fe-15Cr-35Ni-11 W alloy under the effects of treatment with ion-plasma beams differ from the transformations in steels of types 0 × 18H10T and 0 × 16N15M3B widely used in nuclear power engineering. Presence of these differences was established by performing X-ray radiography analysis, which demonstrated that additional reflections on the X-ray patterns of irradiated samples of Fe-15Cr-35Ni-11 W alloy appear from the side of large angles relative to the reflections for the initial solid solution. Detailed X-ray diffraction studies carried out by the authors showed that additional peaks appeared from the side of smaller angles in the X-ray diffraction patterns of iron-chromium alloys of type 0 × 18 (10–30) H additionally doped with Ti, Mo, Nb, Al to the amount of 1–3% and irradiated with ion-plasma beams.

In both cases the phase thus formed is of isomorphic matrix type and is thermally metastable and, in contrast to 0 × 18H10T steel, Fe-15Cr-35Ni-11 W alloy undergoes softening. The analysis of published data on the possible causes inflicting similar structural-phase transformations in materials subjected to intensive ion-plasma treatment was performed. Concentrations of crystalline lattice stacking faults in Fe-15Cr-35Ni-11 W alloy and in 0 × 18H10T steel in the deformed state were determined by X-ray diffraction analysis. It was found that concentration of structural stacking faults in this state is 4 times higher for 0 × 18H10T steel, which indicates the lower stacking fault energy in this steel. Conclusion was made that the observed effects are associated with the mechanism of radiation-induced plastic deformation. Structural-phase changes in Fe-15Cr-35Ni-11 W alloy are associated with deformation by twinning, in contrast to 0 × 18H10T steel, where the observed transformations are due to slip deformation.

Keywords: Ion-plasma treatment; Austenitic stainless steel; X-ray diffraction-pattern; Stacking faults; Slip deformation; Twinning deformation.

Introduction

Pronounced structural-phase faults take place in structural materials of commercial nuclear reactors in polycrystalline and monocrystalline states under the effects of continuous irradiation with ion-plasma fluxes with energies ranging from tens of kiloelectron-volts to 1 MeV [1–24]. Typical variations of X-ray diffraction pattern in irradiated alloys are shown in Fig. 1. In Fe-18Cr-10Ni and Fe-16Cr-15Ni alloys doped with small additions of Ti, Nb or Mo having face-centric cubic lattice (FCC) these changes are manifested in the form of appearance of additional X-ray reflections from the side of small angles relative to the principal lines of the matrix solid solution. For materials with Fe-12Cr-Mo-V-Nb, Fe-(18–24Cr) composition having body-centered cubic lattice (BCC) the initial X-ray line underwent broadening.

The above described changes were accompanied by increased microhardness, which is by three- four times higher for materials with BCC lattice compared with those with FCC lattice.

Assumption was made in [25,26] that radiation effects inflicted by ion beams are mainly associated with plastic deformations within the near-surface volume of materials. Such

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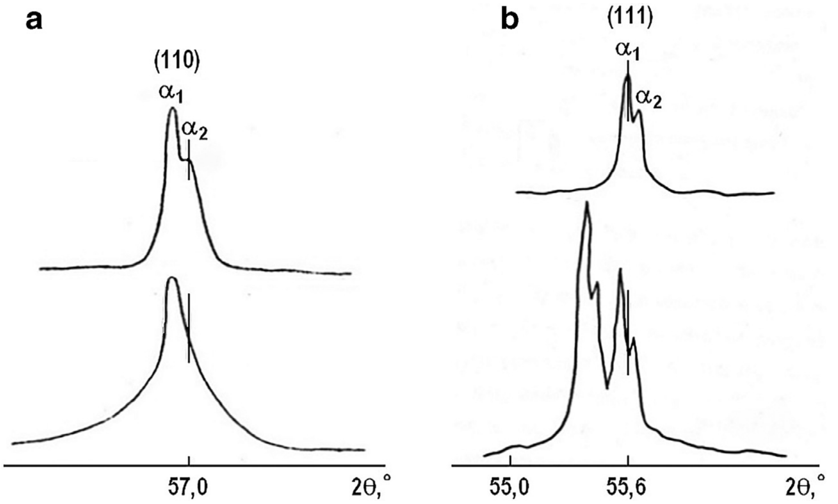

Fig. 1. X-ray diffraction pattern for Fe-12Cr-Mo-V-Nb (a) and FCC Fe-16Cr-15Ni-3Mo-Nb (b) BCC steels: initial pattern is shown in the graphs above, that after ion-plasma treatment is presented in the graphs.

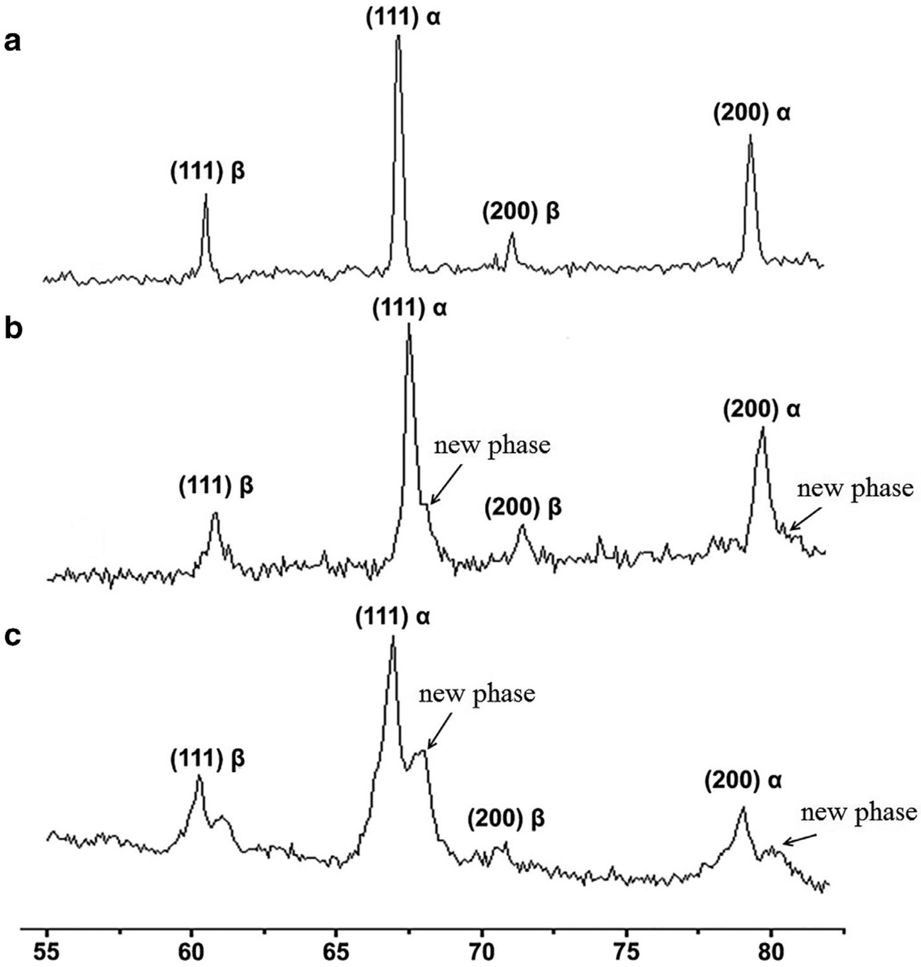

Fig. 2. X-ray radiograms for Fe-15Cr-35Ni-11 W in the initial conditions (a); in the conditions of argon irradiation exposure reaching $D = 6.7 \cdot 10^{20}$ ions/cm^2 (b), and in the conditions of argon irradiation exposure reaching $D = 9 \cdot 10^{20}$ ions/cm^2 (c).

deformations occur in constrained conditions and are associated with formation and evolution of not one-dimensional (dislocations) but, instead, two-dimensional faults of the crystalline structure (stacking faults, twins, etc.). The mechanism of deformation depends on the type of faults – plasticity carriers – which, in turn, is determined by the energy of stacking faults. Thus, changes of line (111) in the X-ray diffractogram for FCC alloy (see Fig. 1) must be associated with high concentration of stacking faults generated in the process of plastic deformation. Comparison of structural changes taking place under the effects of ion-plasma fluxes for alloys belonging to the same structural class but differing to a significant extent as pertains to the composition and stacking fault energy.

Materials and methodology of the experiment

Both heat-resistant Fe-15Cr-35Ni-11 W alloy and 0X18H10T steel refer to austenitic class characterized, however, with probability of formation of stacking faults in the process of mechanical deformation. It was established as the result of processing of radiograms for steels after mechanical deformation using the well-known methodology [27,28] that probability of formation of stacking faults in Fe-15Cr-35Ni-11 W steel amounts to $\alpha = 8.3 \cdot 10^{-3}$, while that for 0X18H10T steel is equal to $\alpha = 2.5$. It can be assumed that in Fe-15Cr-35Ni-11 W alloy having lower probability of formation of stacking faults slip deformation is to a considerable extent suppressed and deformation mechanism by twinning is the most probable one. Therefore, attempt was made to compare the behavior under the effects of ion-plasma treatment of Fe-15Cr-35Ni-11 W alloy and 0X18H10T steel obtained by the method of vacuum-arc re-melting.

Prior to irradiation cold-deformed samples were annealed in oil-free vacuum of 10^{-6} Pa at 1000 °C during one hour in order to remove distortions caused by the mechanical processing. X-ray radiography analysis was performed after austenization which demonstrated that in its initial conditions steel represented homogenous solid solution with FCC lattice. Samples of Fe-15Cr-35Ni-11 W alloy were treated by irradiation with argon ions of ion-plasma beam with density reaching 10^{19} ions/cm^2 s and with energy up to 5 keV under the conditions of pressure in the chamber equal to 10^{-1} Pa at temperatures within 450–600 °C. X-ray structural analysis was performed using Dron-2.0 diffractometer irradiated

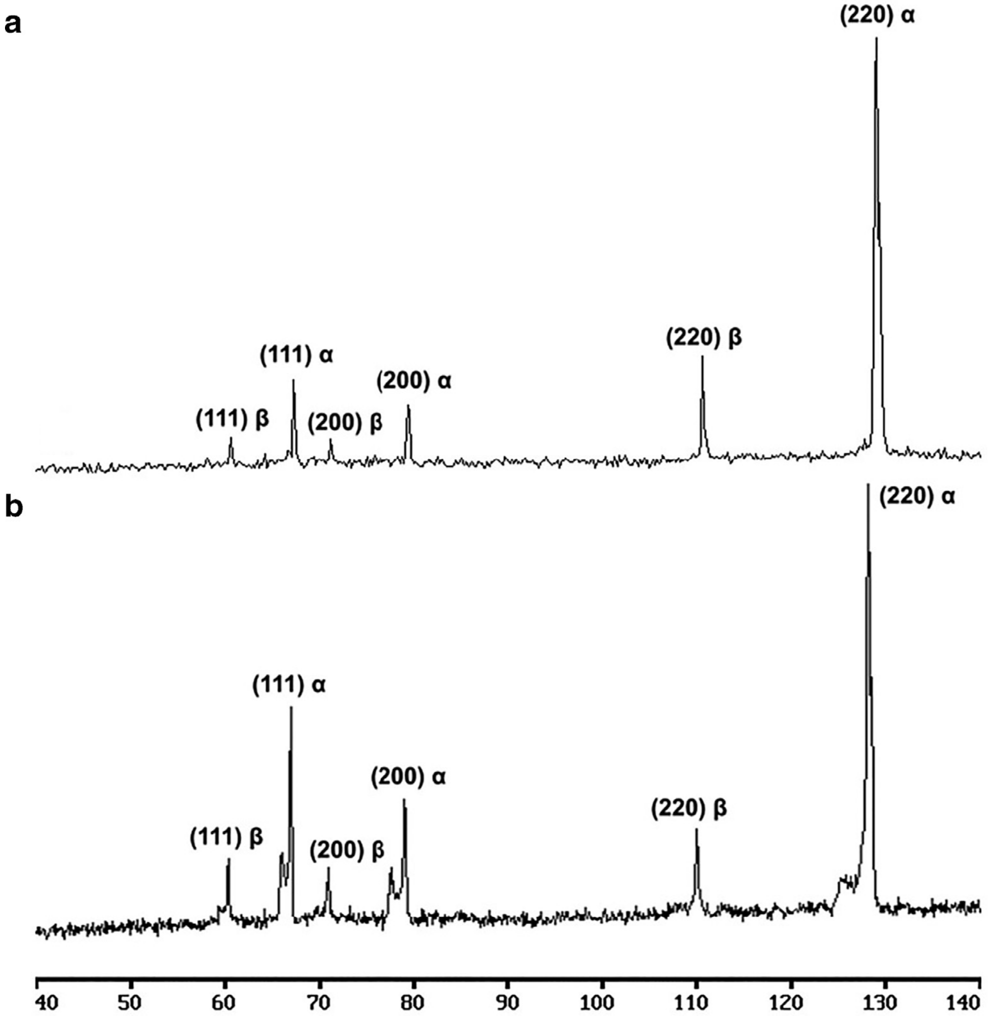

Fig. 3. X-ray radiogram for initial sample of 0X18H10T steel (a) and sample irradiated to reach argon exposure dose $D = 9 \cdot 10^{20}$ ions/cm^2 (b).

by monochromatic Cr–K_α radiation. The obtained radiograms were processed using the OUTSET code.

X-ray diffractograms are shown in Fig. 2 for Fe-15Cr-35Ni-11 W in initial conditions and after irradiation by ion-plasma fluxes. Additional X-ray radiography peaks from the side of large angles appeared after irradiation. The additional peaks are manifested only in the vicinity of lines for the initial solid solution, which proves unequivocally that the new phase has the same FCC structure as the initial alloy with, however, smaller crystalline lattice spacing. Crystalline lattice spacing for the new phase of Fe-15Cr-35Ni-11 W alloy differs from the initial value $a = 3.5843$ Å and decreases to $a = 3{,}5547$ Å (exposure dose for argon $D = 6.7 \bullet 10^{20}$ ions/cm^2) and $a = 3{,}5619$ Å (exposure dose for argon $D = 9 \bullet 10^{20}$ ions/cm^2).

Analysis of intensities of initial and new lines indicates that volume fractions of the new phase amount to approximately 20–30% of the initial values. It can be assumed that in heat-resistant Fe-15Cr-35Ni-11 W steel having high yield stress and higher stacking fault energy constrained radiation-induced plastic deformation takes place according to the twin formation mechanism with development of isomorphous phase. In this case, in accordance with [24], isomorphous phase is formed as the result of deformation under high shearing stresses.

Typical measurements of X-ray diffraction picture are shown in Fig. 3 for 0X18H10T austenitic steel with low value of stacking fault energy in the initial conditions and after ion-plasma treatment with argon ions.

It is evident from the X-ray radiograms that additional peaks are formed from the side of small angles relative to the principal reflexes in the initial conditions. New phase is isomorphous with the initial one but, however, it has larger crystalline lattice spacing. Structural transformations in austenitic steel with low energy of stacking faults are also associated with plastic deformation. Structural transformations in austenitic steel with low energy of stacking faults are also associated with plastic deformation but, according to the mechanism of formation of stacking faults, they are associated with appearance of isomorphous phases with larger crystalline lattice spacing.

Sample of Fe-15Cr-35Ni-11 W alloy after ion-plasma treatment with Ar$^+$ ($E = 3.5$ keV, $T_{\text{irr.}} = 500\,^\circ$C) was subjected to post-irradiation annealing at temperatures of 500, 600 and 700 °C. As it is evident from Fig. 4, the isomorphous phase developed is metastable and there exists the temperature of phase relaxation at which the formed phase disappears.

Measurements of microhardness demonstrated that after ion-plasma treatment hardness of Fe-15Cr-35Ni-11 W sample decreased from 2160 to 1090 MPa. It can be assumed that with formation of the new phase with decreased crystalline lattice spacing tensile areas are formed in local volumes of the initial matrix, and this is what specifically leads to the decrease of microhardness. In contrast, microhardness of 0X18H10T steel increases after ion-plasma treatment by two–three times. This is associated with appearance of the new phase with larger crystalline lattice spacing.

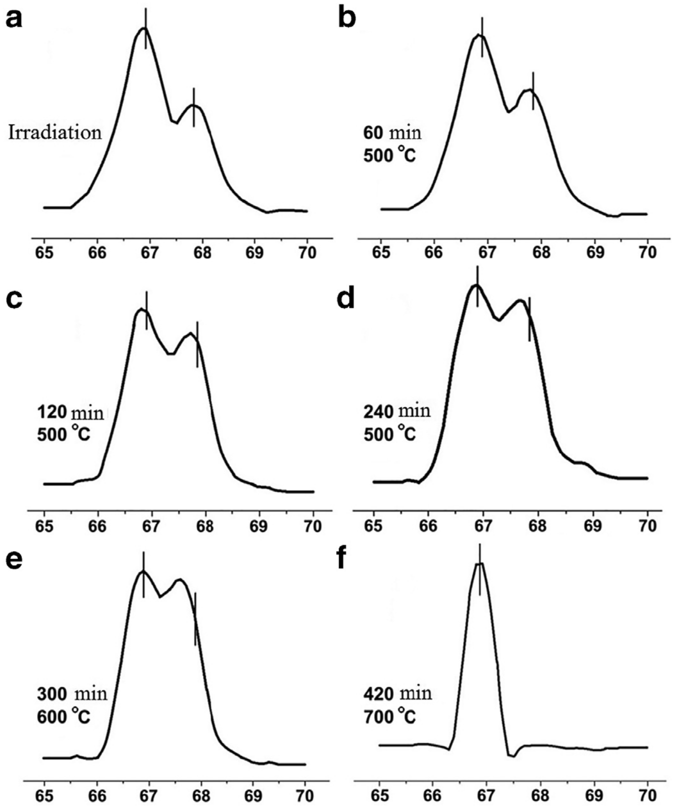

Fig. 4. Variation of shape of X-ray diffraction line (111) for sample of Fe-15Cr-35Ni-11 W subjected to ion-plasma treatment with Ar$^+$ with energy of 3.5 keV at temperature of 500 °C (a) and sample annealed at 500 °C (b, c, d), 600 °C (e), and 700 °C (f).

Conclusion

It was demonstrated that as the result of ion-plasma treatment the character of structural transformations in austenitic steels depends on the stacking fault energy. In austenitic Fe-15Cr-35Ni-11 W alloy with larger stacking fault energy radiation-induced plastic deformation proceeds according to the twin formation mechanism. This is manifested in the form of appearance in the X-ray radiograms of additional reflections from the side of larger angles. Softening of the alloy takes place simultaneously with the above process. Such behavior differs from the behavior of previously investigated steels of austenitic class (0X18H10T and 0X16H15M3Б) with low energy of stacking faults where structural transformations induced by ion-plasma treatment are also associated with plastic deformation but, however, they occur according to the mechanism of formation of stacking faults. This is manifested in the appearance of additional reflections in X-ray radiographs from the side of smaller angles. Increase of microhardness of alloys takes place as well.

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