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To cite this article: K V Almaeva *et al* 2021 *J. Phys.: Conf. Ser.* **1989** 012016

Regularities of the deformed microstructure of ferritic-martensitic steel EP-823 after high-temperature thermomechanical treatment

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Abstract. The features of the microstructure of 12% chromium ferritic-martensitic steel EP-823 near the neck region of samples deformed by tension at T=-70, -40 °C, 20 °C and in the temperature range close to the reactor core operating temperatures of T = 650, 720 °C after high-temperature thermomechanical treatment (HTMT) are investigated. It is shown that at negative and room temperatures plastic deformation leads to curvature and fragmentation of martensitic lamellae, formation of new low-angle boundaries and a significant increase in the dislocations density. Deformation at high temperature contributes to the dynamic recovery, dynamic polygonization and increased sizes of carbide and carbonitride particles ($M_{23}C_6$ and MX type).

1. Introduction

The 9-12% chromium ferritic-martensitic steels are intended for use as structural materials for the fuel-element cladding of new nuclear and thermonuclear reactors [1-4]. Due to the requirements for heat and radiation resistance, these structural materials must provide high values of long-term high-temperature strength and have a low tendency to low-temperature (including radiation) embrittlement. The investigation of mechanical properties, features of plastic deformation and fracture near the range of operating temperatures (T=650-720 °C) and near the interval of the viscous-brittle transition is a necessary stage of pre-reactor testing of structural steels for use in nuclear power applications. A visco-brittle transition is observed in ferritic-martensitic steels at negative temperatures [5]. Under irradiation it can shift to the region of positive temperatures, which determines the need to study the low-temperature properties of ferritic-martensitic steels.

High-temperature thermomechanical treatment (HTMT) is one of the ways to modify the microstructure of steels in order to improve their mechanical properties [1, 6, 7]. The prospects of using this treatment were shown for Russian 12% chromium ferritic-martensitic steels EK-181 and EP-823 [8, 9]. The influence of HTMT on the features of plastic deformation and fracture of ferritic-martensitic steels, including those near the interval of the visco-brittle transition, is currently poorly investigated.

This paper presents the results of studying the influence of the deformation temperature (near the visco-brittle transition temperatures and operating temperatures) in the nuclear reactor core under mechanical tensile tests on the features of the deformed microstructure and the mechanisms of plastic deformation of ferritic-martensitic steel EP-823.

2. Material and methods

Heat-resistant 12% chromium ferritic-martensitic steel EP-823 after HTMT was investigated. The elemental composition of the steel EP-823 is: Fe is base, 0.14 wt % of C, 11.56 wt % of Cr, 0.58 wt % of Mn, 0.74 wt % of Mo, 0.40 wt % of Nb, 0.34 wt % of V, 0.68 wt % of Ni, 0.03 wt % of N, 1.09 wt % of Si, 0.10 wt % of Ce, 0.01 wt % of Ti, 0.006 wt % of B, 0.02 wt % of Al. HTMT consisted of heating T = 1100 °C, holding for 1 h, hot plastic deformation by rolling to a value of $\varepsilon \approx 50\%$ and subsequent quenching in water. After the deformation the tempering was carried out at T = 720 °C for 1 h. Mechanical tensile tests at negative temperatures T = (-70, -40 °C) were performed in a mixture of liquid nitrogen and ethyl alcohol at room temperature – in air, at temperatures T = (650, 720 °C) - in a vacuum of $\approx 2.7\times10^{-3}$ Pa. High-temperature tests were performed on a NIKIMT 1246R-2/2300 high-temperature vacuum testing machine (Russia). We used dog-bone samples with a gage length of 13 mm and a gage section of 2 mm.

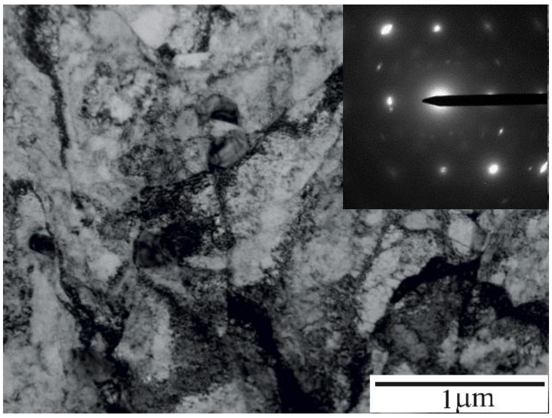
Structural investigations were performed in a Philips CM12 transmission electron microscope at an accelerating voltage of 120 kV. Thin foils for transmission electron microscopy were prepared using a Hitachi FB-2100 focused ion beam system near the neck area of the sample deformed by tension. The change in the elemental composition was investigated on thin foils in a JEM-2100 transmission electron microscope with the INCA Energy X-ray microanalysis system.

3. Results and discussion

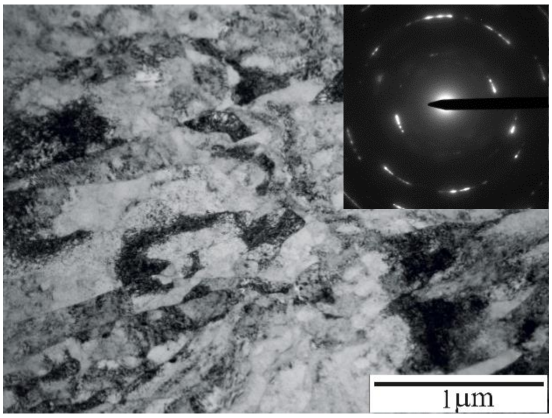
Investigation of the microstructure of steel near the neck region after tensile test at room and negative temperatures (-70 °C, -40 °C) have shown that plastic deformation leads to curvature of the sub-boundaries of martensitic lamellae, fragmentation of lamellae and martensitic packets (Figure 1). The width of the martensitic lamellae is from 50 to 500 nm. The dislocation density near the neck increases significantly relative to the volume of the material and in some areas reaches 10^{16} m^{-2}. Many extinction contours are observed, including strongly curved contours, which indicates high local internal stresses. In the diffraction patterns (Figure 1), low-angle azimuthal misorientation are detected. In some areas (Figure 1 b), there are regions of localized deformation with a highly fragmented fine structure with quasi-ring diffraction patterns. This indicates the formation of new submicron grains and subgrains during the deformation process. Coarse particles of $M_{23}C_6$ (M-Cr, Fe, Mn) are observed, mainly of a rounded shape with dimensions of less than 100 nm.

In the temperature range from -70 °C to 20°C in the course of plastic deformation, the cross slip of dislocations is difficult. This leads to the formation of dislocation pileups, an increase in the angle of misorientation between lamellae and martensitic packets. The dislocation density inside the martensitic lamellae increases and dislocation substructures are formed. A low temperature of deformation prevents the processes of dislocation annihilation and relaxation of local internal stresses.

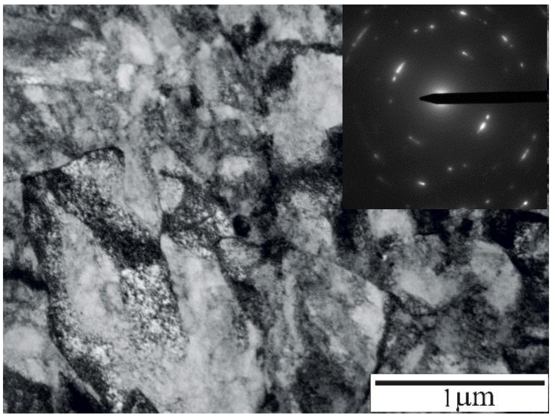
As shown earlier [8], after HTMT the dislocations in EP-823 steel are pinnied by fine particles of the MX type (where M is V, Nb, X is C, N) with dimensions of 5-10 nm. This determines the high strength properties of this steel at the test temperatures. The yield strength at T= 20 °C reaches 793 MPa. When the deformation temperature decreases to -70 °C the yield strength increases to 1005 MPa. At the same time, the relative elongation remains at a fairly high level (12 %). The relative elongation value does not significantly change in the temperature region of the visco-brittle transition observed in impact tests of ferritic-martensitic steels. Accordingly, the deformed microstructure of steel at low (-70 °C, -40 °C) temperatures is qualitatively similar.



a



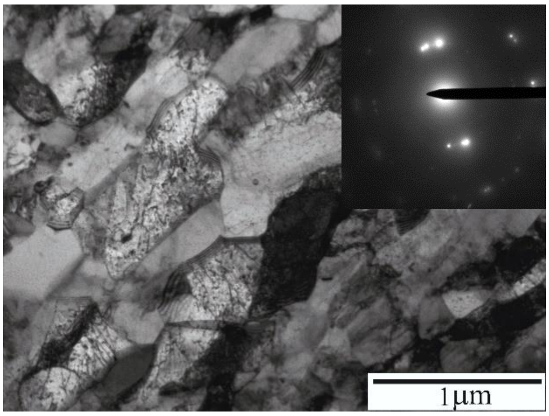
b



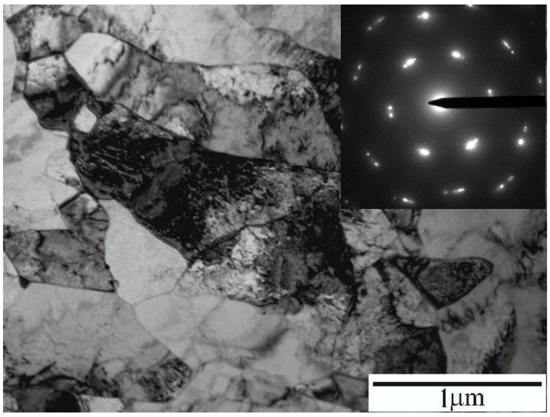
c

Figure 1. Microstructure of EP-823 steel near the neck area, bright-field images and corresponding micro-diffraction patterns: a – 20 °C; b – -40 °C; c – -70 °C.

At tensile test temperatures of 650 °C and 720 °C, both elongated martensitic lamellae and equiaxed fragments with low-angle misorientation boundaries (Figure 2) were found near the neck region. In some fragments an increased dislocation density of 10^{14}-10^{15} m^{-2} is observed, in other areas it is much lower. The size of fine particles of the MX type increases up to 15-20 nm at these temperatures.



a



b

Figure 2. Microstructure of EP-823 steel near the neck area, bright -field images and corresponding micro-diffraction patterns: a – 650 °C; b – 720 °C.

 1989 (2021) 012016 doi:10.1088/1742-6596/1989/1/012016

These features of the deformed microstructure indicate the processes of dynamic recovery and dynamic polygonization. At 720 °C, these processes are more intensive. An increase in the deformation temperature leads to a decrease in the strength properties of the steel. The yield strength at T= 650 °C reaches ≈ 350 MPa, at 720 °C it decreases to ≈ 265 MPa. In the course of tensile testing near the operating temperatures (650-720 °C), the thermally activated processes of the dislocation climb over the particles and dislocation pileups are intensified. The efficiency of the mechanisms of precipitation and substructural strengthening is significantly reduced.

The high temperature of the tensile tests increases the size of the carbide and carbonitride particles relative to the initial state. In the deformed microstructure of steel, both inside the martensitic lamellae and at the boundaries of grains and subgrains, several types of particles were found – coarse (tens to hundreds of nm) and nanoscale. To identify these particles, their elemental analysis was carried out (Figure 3). It showed that the coarse particles belong to two types of carbides (carbonitrides) – $M_{23}C_6$ (M – Cr, Fe, Mn) and MX (M – Nb, V, X-C, N). Coarse particles are mainly Cr- and Mn-based carbides – of the $M_{23}C_6$ type (Figure 3 b, e). There are also coarse particles of the MX type based on niobium with a size of 50 nm. However, their density is much lower than that of $M_{23}C_6$ carbides. The fine particles belong to a vanadium-based MX-type phase.

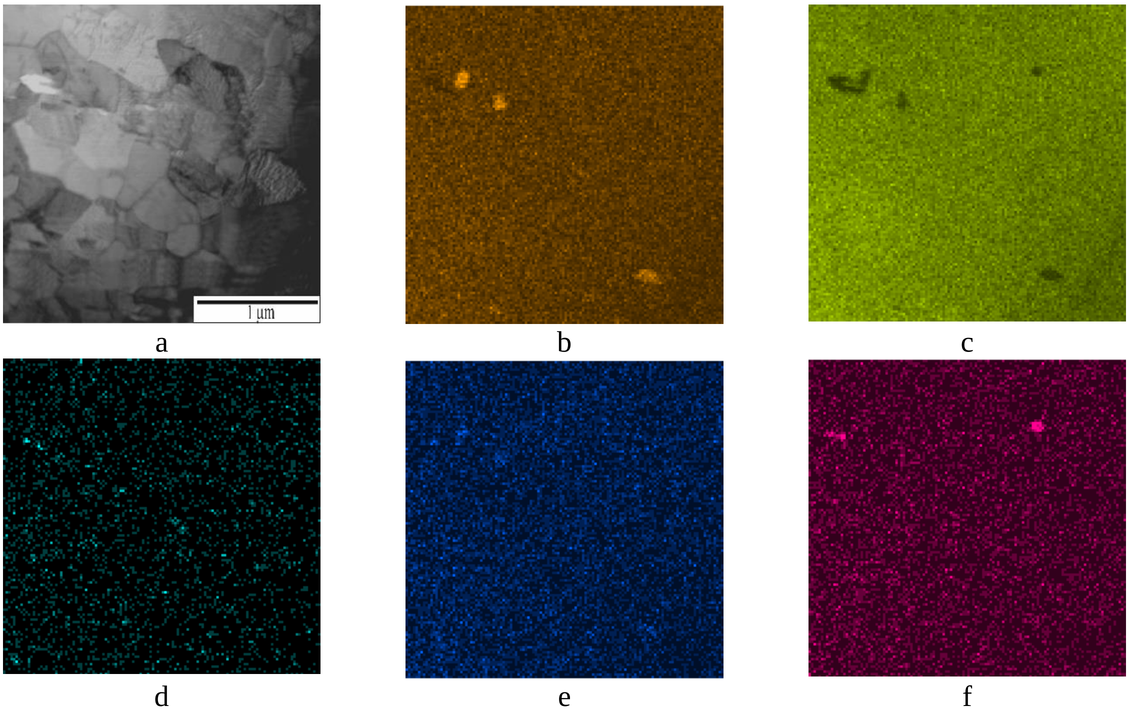


Figure 3. Microstructure of steel EP-823 at the test temperature T = 720 °C after HTMT: (a) image in the reflected electrons; (b)-(f) – maps of the distribution of elements: b – Cr, c – Fe; d – V; e – Mn; f – Nb.

4. Conclusions

It has been shown that plastic deformation of EP-823 steel at the temperatures of -70 to +20 °C leads to the curvature and fragmentation of martensitic lamellae, the formation of new low-angle misorientation boundaries, and a significant increase in the dislocation density inside the martensitic lamellae and ferritic grains. The increased dislocation density and their pinning by fine particles of the MX type contribute to the high efficiency of the mechanisms of precipitation and substructural strengthening at these temperatures, which determines the high strength properties of steel after HTMT.

Plastic deformation near the nuclear reactor operating temperature range (T= 650-720 °C) develops with the participation of the processes of dynamic recovery and dynamic polygonization. At the same time, thermally activated processes of dislocations climbing over nanoscale particles and dislocation pileups are enhanced. The efficiency of the mechanisms of mechanisms of precipitation and substructural strengthening is significantly reduced, which leads to a decrease in the strength properties of steel.

The high temperature of the tensile tests leads to an increase in the size of the carbide and carbonitride phases. An elemental analysis of coarse particles has shown that they are carbides of the $M_{23}C_6$ type and carbonitrides of the MX type.

Acknowledgments

This study was funded by RFBR, project No 19-38-90139 and performed according to the Government Research Assignment for the Institute of Strength Physics and Materials Science of the Siberian Branch of the Russian Academy of Sciences (ISPMS SB RAS), project No. FWRW-2021-0008.

The authors are grateful to Prof. V. M. Chernov and Dr. M. V. Leontieva-Smirnova, JSC "VNIINM", Moscow, for providing the samples of EP-823 steel.

The investigations were carried out using the equipment of the Tomsk Materials Research Share Use Centre of Tomsk State University and Share Use Centre "Nanotech" of the Institute of Strength Physics and Materials Science SB RAS.

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