EFFECT OF THERMOMECHANICAL TREATMENT WITH MULTIPLE PLASTIC DEFORMATION ON THE NANOPHASE HARDENING OF DIE STEEL WITH REGULATED AUSTENITIC TRANSFORMATION

ВЛИЯНИЕ ТЕРМОМЕХАНИЧЕСКОЙ ОБРАБОТКИ С МНОГОКРАТНОЙ ПЛАСТИЧЕСКОЙ ДЕФОРМАЦИЕЙ НА НАНОФАЗНОЕ УПРОЧНЕНИЕ ШТАМПОВОЙ СТАЛИ С РЕГУЛИРУЕМЫМ АУСТЕНИТНЫМ ПРЕВРАЩЕНИЕМ

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Abstract: *A new class of die steels for hot deformation, namely, steels with regulated austenitic transformation realized during exploitation (RATE) have been developed in Russia. These steels are characterized by the technological advantages typical of traditional α-solid solution-based steels and, when working in the austenite state, assure the enhanced tool life. The structure, phase composition and hardening of RATE steel have been analyzed by an example of steel containing 0.41 % C, 1.04 % Si, 3.59 % Mn, 2.41 % Cr, 5.07 % Ni, 2.40 % Mo, 0.69 % V, 0.51 % Ti, 0.25 % Co subjected to thermomechanical treatment, which includes the austenization and multiple plastic deformation. The nanophase hardening occurs in RATE steels being in the austenitic state, during multiple plastic deformation at temperatures of 450-750 °C. The precipitation of nano-sized heat-resistant excess phases ensures the inhibition of recrystallization processes at operating temperatures, inhibits the grain growth, and increases the stability of the austenite.*

KEY WORDS: HOT-WORK TOOL STEEL, DISPERSION HARDENING, NANOPHASE HARDENING, RECRYSTALLIZATION, AUSTENITE, $\alpha \rightarrow \gamma$ TRANSFORMATION

1. Introduction

The durability of die tools largely determines the profitability of hot-forming processes of precision workpieces. When work surfaces are heated to temperatures of to 600-650 °C, traditional die ferrite steels hardened with carbide or carbide and intermetallics assure the adequate stability [1]. However, at the higher temperatures, even the most heat-resistant of the steels undergo intense softening, which is the main reason for the rapid failure of tools. The application of heat-resistant austenitic alloys is also limited owing to their tendency to cracking, poor machinability and high costs of their components.

A new class of die steels for hot deformation, namely, steels with regulated austenitic transformation realized during exploitation (RATE) have been developed in Russia in 1970-1980. These steels are characterized by the technological advantages typical of traditional α-solid solution-based steels and, when working in the austenite state, assure the enhanced tool life.

RATE steels undergo the $\alpha \rightarrow \gamma$ transformation at operating temperatures and demonstrate the high stability of the supercooled austenite in the pearlite region. This allows one to eliminate the austenite decomposition during possible cooling of the tool to 500-350 °C and to use the low-temperature thermomechanical treatment for hardening the surface layers of the tool. The feature of the steels consists in the fact that they use the stresses and high temperatures to increase the strength of the tool (Ozerskiy-Krugljakow effect) [2]. This is a fundamentally new concept in choosing alloying systems and hardening treatment conditions for die steels for their operation at temperatures of 600-800 °C.

It is known that, when being in the austenitic state, steels of this class, tend to either deformation, or dispersion, or complex hardening under conditions of temperature and force loads applied to the tool directly during its operation [2, 3]. The use of multiple plastic deformation at 450 °C assures the significant improvement of strength properties of the steel at test temperatures up to 800 °C without a substantial decrease in its ductility [4]. In this case, the optimization of the thermomechanical treatment diagrams of RATE steels, which assures the additional nanophase strengthening of work-hardened austenite is of interest because the nature of the hardening of the steels has not been studied exhaustively. This fact may also be of particular interest in the study of the nature of hot work hardening.

There are only few foreign publications related to tool steels with the close chemical composition in the literature, which are focused mainly on the features of the microstructure of these steels [5].

In present study, we analyze the structure, phase composition and hardening of RATE steel (by an example of steel containing 0.41 % C, 1.04 % Si, 3.59 % Mn, 2.41 % Cr, 5.07 % Ni, 2.40 % Mo, 0.69 % V, 0.51 % Ti, 0.25 % Co) subjected to thermomechanical treatment comprising the austenization and multiple plastic deformation.

2. Experimental procedure

We studied RATE steel (containing 0.41 % C, 1.04 % Si, 3.59 % Mn, 2.41 % Cr, 5.07 % Ni, 2.40 % Mo, 0.69 % V, 0.51 % Ti, 0.25 % Co, less than 0.03 % S, less than 0.03 % P).

Steel was subjected to oil hardening from a temperature of 1020 °C followed by double tempering at 610-620 °C and 560-580 °C for 2 h (the hardness is 42 HRC) that is typical heat treatment of the steel. This treatment simulates the process of operation and prevents the tool from the pre-out of the tolerance before the $\alpha \rightarrow \gamma$ transformation.

Cylindrical specimens of the steel 10.00 ± 0.03 mm in height and 7.00 ± 0.03 mm in diameter were cut from an initial billet 13.32 ± 0.02 mm in diameter were used to carry out the thermomechanical treatment (TMT). The axis of the cylindrical specimen was coincides with the axis of the initial billret. TMT was performed using an installation Gleeble System 3800. After testing, transverse specimens for subsequent investigations were cut from both the edge and middle parts of the cylindrical specimen (Figure 1).

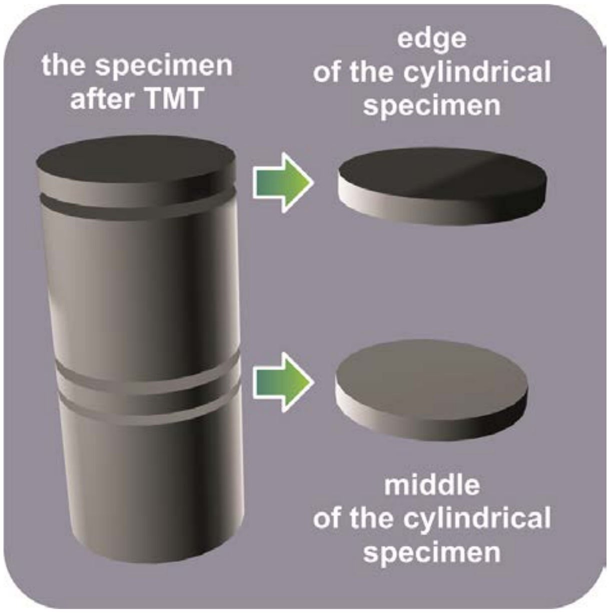

Fig. 1. *Scheme of cutting samples for investigation*

Figure 2 shows the TMT diagram of TMT. The TMT includes the austenization at 1150 °C, cooling to a temperature of 450 °C, holding for 15 min and subsequent compressive deformation (5 cycles with 2 % deformation in each cycle; the rate of deformation is 0.1 c^{-1}). Further heating was performed at a temperature of 750 °C, which corresponds to the operating temperature of die; the subsequent compressive deformation was realized in accordance with the same mode. After the TMT, the specimens were cooled for 5 min. The structure of the steel in the initial and treated states was studied by optical and transmission electron microscopy.

Metallographic analysis of the structure of samples was performed at magnifications x100 and x500 using the Buehler optical microscope and sections etched in a 5 % nitric acid solution.

Electron microscopic studies of the structure were performed using thin foils, a JEM-2100 (JEOL) transmission electron microscope and an accelerating voltage of 200 kV. The foils were prepared from plates cut from the specimens, which were thinned to a thickness of ~100 μm by mechanical grinding. After that, the foils were thinned by jet electrolytic polishing at room temperature and a voltage of 20 V using an electrolyte containing 100 ml $HClO_4$ and 900 ml CH_3COOH.

The phase composition of the specimens was determined using a DRON 3M diffractometer and monochromatized Co*Kα* radiation.

The Vickers microhardness measurements was measured at a load of 1 N (the holding time is 10 s) using a Micromet 5101 (Buehler) tester equipped with Mitron MTV-62W1P digital camera and «ImageExpert MicroHardness 2» sofware.

3. Results and discussion

Figure 3 shows micrographs the structure of the steel subjected to TMT, which were taken with an optical microscope. The average grain size in samples cut from the edge and the middle areas of the cylindrical specimen was 14 and 23 μm, respectively. The light areas are probably areas of residual austenite (Figure 3 d).

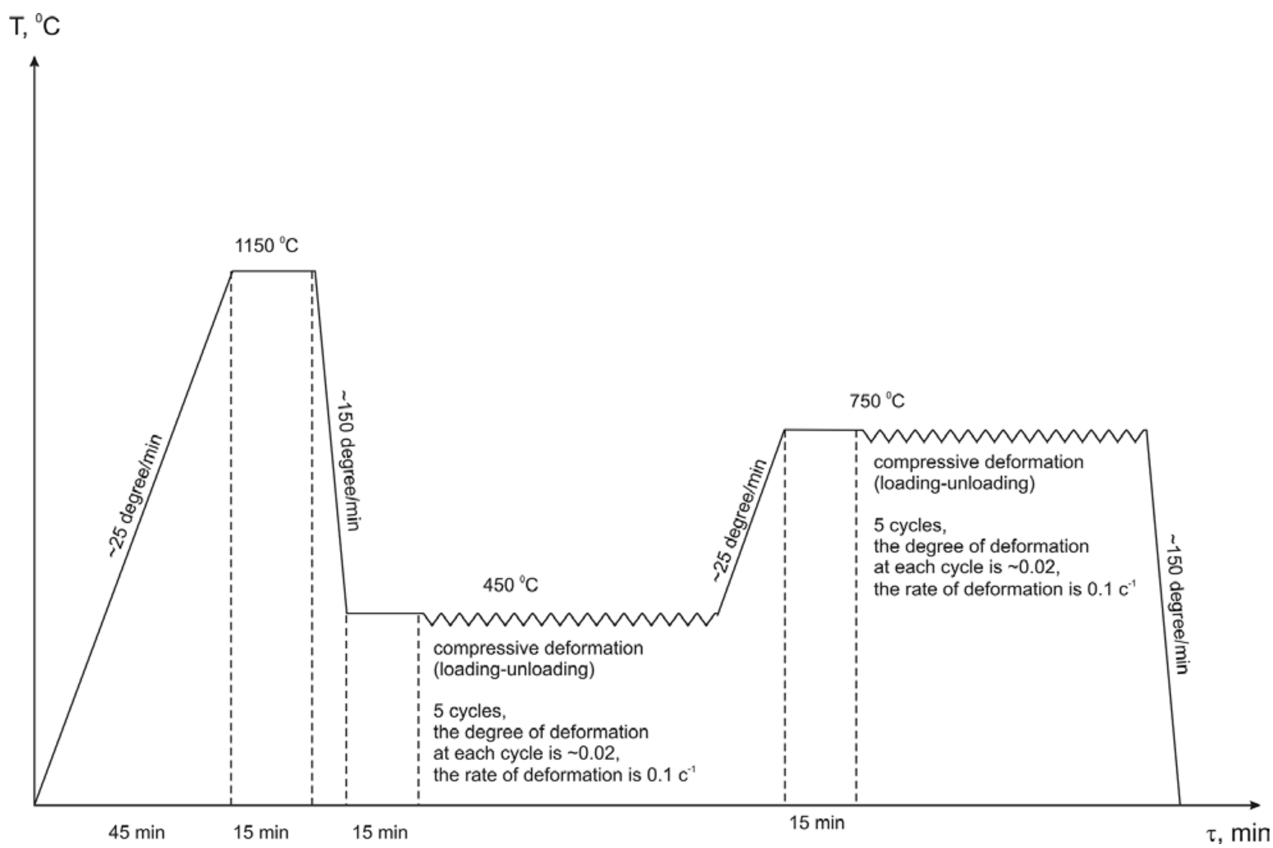

Fig. 2. *Diagram of TMT of die steel*

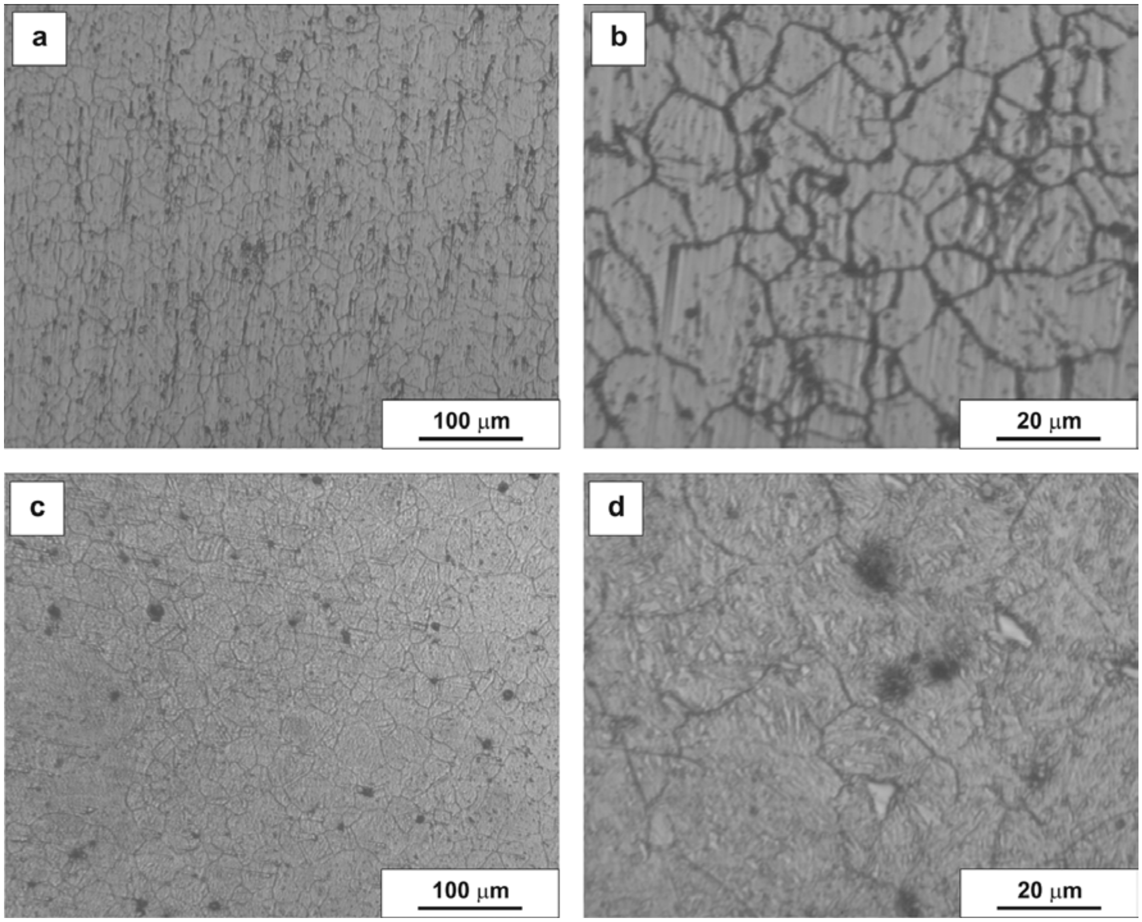

Fig. 3. *The microstructure of the samples of the steel subjected to TMT (optical microscopy): a, b – edge of the cylindrical specimen; c, d – middle of the cylindrical specimen.*

According to transmission electron microscopy (TEM) and electron microprobe analysis (EMA) data, numerous nano-sized (50-200 nm long) Laves phase- (Fe_2Mo), σ-phase-like (FeCr) particles and particles of complex compounds are observed in the structure of the steel in the initial state (Figure 4). The structure also contains isolated large titanium carbide inclusions (more than 1 μm in diameter).

TEM images of the microstructure of the steel subjected to TMT are shown in Figures 5 and 6.

Previously, high-temperature method of X-ray phase analysis showed that 100 % of the austenite is present in the steel structure when heated to a temperature of 1100-1150 °C [3]. The structure formed in the steel during TMT is similar to the lath (needle) austenite. The structure also is characterized by the nano-sized precipitates 10 - 400 nm in size. The average size of the particles and their volume fraction are lower substantially than those for the initial state of the steel. It is likely that, among the found particles, particles, which were present in the initial steel and were not completely dissoled in solid solution during austenization, and particles precipitated during multiple plastic deformations in the course of TMT are observed.

The structure of the steel subjected to TMT (sample cut from the edge of the cylindrical specimen) is characterized by nano- and submicron-sized particles (50-400 nm in length) of complex compounds; particles of (Ti, V)C carbides are also observed (Figure 5). The structure also contains numerous small spherical particles 10-20 nm in diameter, whose chemical composition could not be determined.

The structure of the steel subjected to TMT (sample cut from the middle of the cylindrical specimen) is characterized by nano- and submicron-sized particles (50-300 nm in length) of complex compounds and particles of FeCr intermetallic compounds as well (Figure 6). The amount of particles of the secondary phases in the specimen is less than that in the specimen cut from the edge of the cylindrical specimen; this fact can explain the slightly higher grain size for the specimen cut from the middle of the cylindrical specimen.

Optimum temperatures of austenization of RATE steels usually are within a range of 950-1050 °C [3]. In present study, the austenization temperature was increased to 1150 °C in order to realize the complete dissolution of particles present in the initial structure of the steel; this is likely to ensure the precipitation of smaller particles during subsequent multiple plastic deformation in the course TMT.

According to X-ray phase analysis data, the structure of the steel in the initial state consists mainly of α-Fe and a small amount of γ-residual. The structure of the steel subjected to TMT also consists of α-Fe and γ-residual; however, the volume fraction of the austenite in the specimen cut from the edge of the cylindrical specimen reaches ~40 %.

The phase composition of particles present in the steel structure and observed by TEM, is not defined by X-ray diffraction analysis owing to their extremely small sizes.

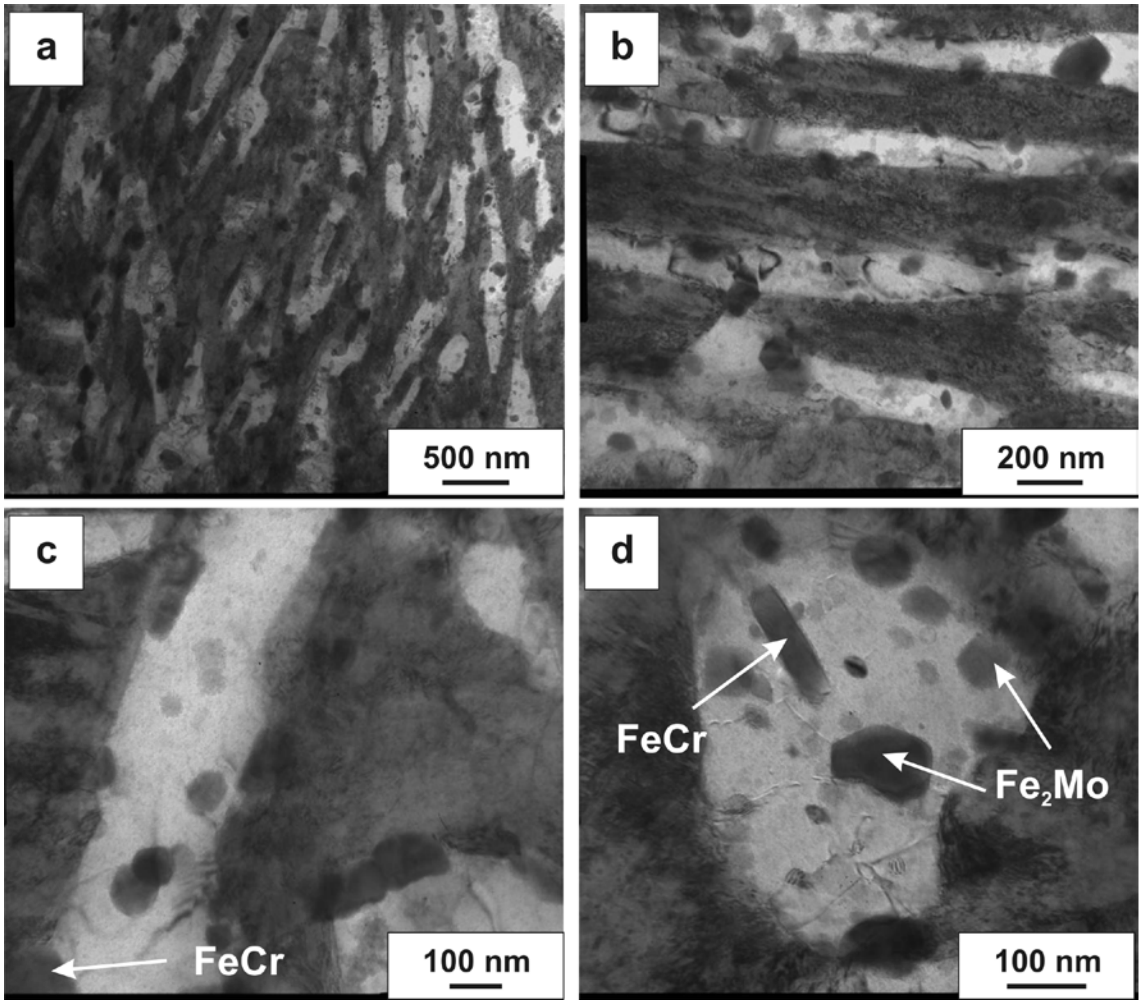

Fig. 4. The microstructure of the samples of the steel in the initial state

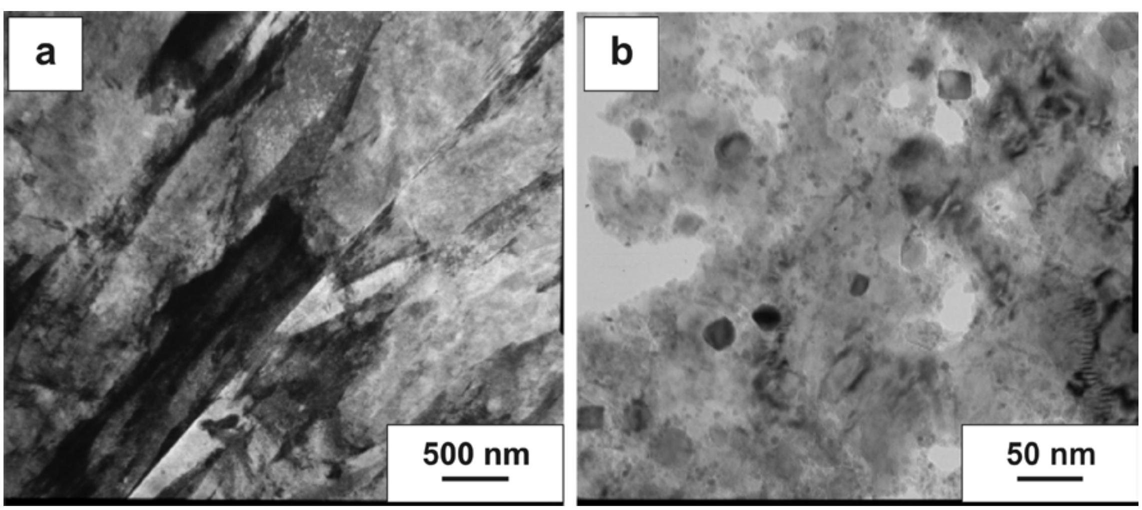

Fig. 5. *The microstructure of the samples of the steel subjected to TMT (the edge the cylindrical specimen)*

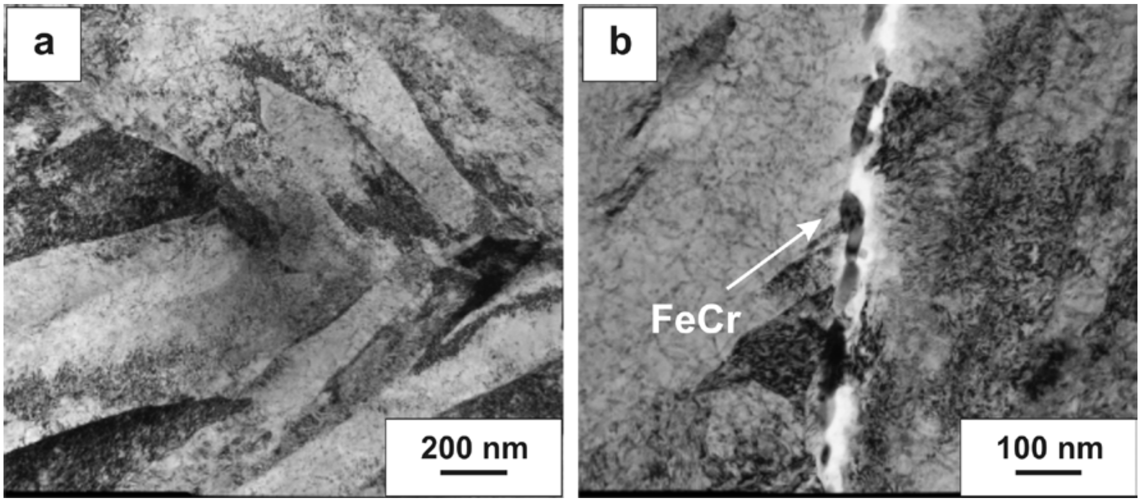

Fig. 6. *The microstructure of the samples of the steel subjected to TMT (the middle of the cylindrical specimen)*

Figure 7 shows the "loading-unloading" curves measured for the steel specimen in the course of TMT. It is seen that the multiple plastic deformation under cyclic loading ensures the significant hardening of austenite at a temperature of 450 °C. At a temperature of 750 °C, the tendency to deformation hardening appears much weaker, while the high level of the hardened state, ~ 540 MPa, takes place.

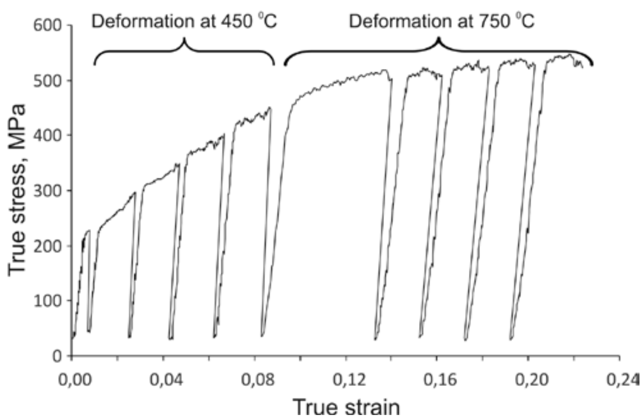

Fig. 7. *The "loading-unloading" curves measured for the steel specimen during TMT*

It should be noted that two competitive processes usually occur simultaneously in RATE steels during operation at 750 °C; these are the hardening (as a result of work hardening of austenite) and softening (due to the polygonization and recrystallization). However, the onset of recrystallization process of supersaturated solutions are not always accompanied by a significant loss of strength, when the growth of nuclei inhibited by the second phase particles, which cause the considerable phase work hardening [6]. In the present study nano- and submicron-size particles of complex compounds and particles of intermetallic compounds as well are responsible for the holding of the hardened state of the steel at a temperature of 750 °C.

Figure 8 demonstrates the microhardness distribution along the diameter of cylindrical specimen of the steel before and after TMT. After TMT, the maximum microhardness reaches ~640 HV (as compared to that for the initial state ~475 HV). For the specimen cut from the edge of the cylindrical specimen, the microhardness of the edge is higher than that in the center (640 and 540 HV, respectively). For specimen cut from the middle of the cylindrical specimen, the microhardness of the edge is lower than that observed for the center (580 and 640 HV, respectively).

Higher microhardness indicates an additional strain hardening of a thin surface layer of the edge of the cylindrical specimen of the steel, which was in contact with the punch of Gleeble System 3800.

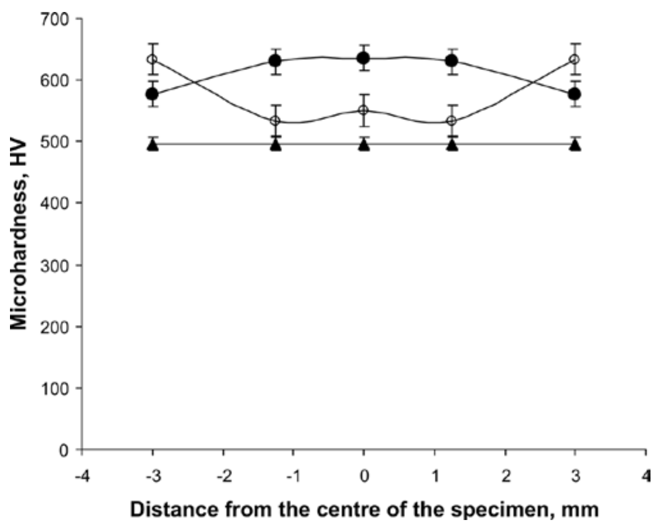

Fig. 8. *Distribution of the microhardness along the diameter of the specimen of the steel before and after the TMT*
▲ *- specimen in the initial state,* ○ *- the edge the cylindrical specimen after TMT;* ● *- the middle of the cylindrical specimen after TMT*

The similar hardening occurs within the thin surface layer of tool, which is directly sensing the cyclic thermal and mechanical loads under the contact with a deformable metal during the operation, and enhances its stability [7]. Herewith the life of the tool itself is known to depend substantially on the properties of the thin surface layer.

4. Conclusion

1. It is shown that, along with the deformation, dispersion and complex hardening processes, the nanophase hardening characterized by the high thermal stability of "hardening state" occurs in RATE steels being in the austenitic state, during multiple plastic deformation at temperatures of 450-750 °C.

2 The precipitation of nano-sized heat-resistant excess phases ensures the inhibition of recrystallization processes at operating temperatures, inhibits the grain growth, and increases the stability of the austenite, which is one of the main factors of the steel hardening during TMT.

3 The higher stability of the austenite creates a reserve for the accumulation of hardening during the operation, provides its stabilization and, thereby, improves the stability of die tools.

4 The additional hardening of the thin surface layer by nano-sized particles, which, when contacting with the deformable metal during operation, receives directly the cyclic thermal and mechanical stresses, also increases the tool life.

5 The use of steels with RATE and methods of hardening treatment in the austenitic state can improve the tool life when pressing hard-copper alloys such as Л63, БрАЖН10-4-4 (Russia); CuZn37, CuAl10Ni5Fe4 (EU); C27200, C63000 (USA) by 6-10 times compared to conventional die 3Х2В8Ф (Russia); X30WCrV9-3 (EU); H21 (USA) steel.

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