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DEEP CRYOGENIC TREATMENT AND TEMPERING AT DIFFERENT TEMPERATURES
OF HS6-5-2 HIGH SPEED STEEL

The paper presents properties of HS6-5-2 high speed steel subjected to deep cryogenic treatment (DCT) and subsequent tempering at different temperatures. DCT process of HS6-5-2 steel leads to shifting of maximum hardness peak to the lower temperature and the reduction of the obtained maximum hardness by about 1 HRC.

These changes in hardness may be due to the shifting of the stage of nucleation and growth of carbide phases to lower temperatures or the changes taking place in the matrix, connected with the additional transformation of the martensite proceeding during the isothermal martensitic transformation occurring at cryogenic temperatures and more extensively occurring precipitation processes, lowering the content of the carbon in the martensite, determining thereby its lower hardness.

Keywords: deep cryogenic treatment, high speed steel, tempering, hardness, microstructure

1. Introduction

The literature findings show that the improvement of the properties of high-speed steel can be made through the use of the additional heat treatment operation – deep cryogenic treatment (DCT). One of the first studies of tool steels subjected to DCT showed an increase in wear resistance, in comparison with steel subjected to conventional heat treatment. Reasons for the improvement of the performance of tools subjected to DCT have not been explained clearly so far. The DCT process and its effects on the mechanical properties of the tool steels are related to the following phenomena:

- Lowering the content of the retained austenite due to its transformation to martensite.
- Forming of the fine carbide phase precipitates in tempered martensite.

The first phenomenon is related to the proceeding of martensitic transformation when it is followed at low temperatures. This is important for the tool steels whose *martensite finish M_f* temperature is much lower than room temperature. Temperature used in a conventional sub-zero treatment (approx. –80°C) is insufficient to complete transformation of the austenite into martensite, and such treatment leaves considerable amount of retained austenite. The use of lower temperatures (so called cryogenic temperatures, about –180°C) is justified by almost complete reduction of the amount of retained austenite and is used in order to induce other phenomena that occur during the process which improve the properties of the steel. Considering the impact of martensitic transformation on the properties of sub-

zero treated steel. It should also be noted that a retained austenite content may favorably affect the strength of steel.

The second phenomenon significantly affecting the properties of the steel is proceeding of the precipitation processes of η-type carbides during the decomposition of martensite [1] at cryogenic temperatures and a low-temperature heating to the room temperature [2]. η-carbides are formed as a result of changes of the hexagonal crystal lattice network of ε-carbide into orthorhombic one, caused by rearrangement of distribution of carbon atoms. Earlier studies on the phenomenon of the above carbides precipitation have been discussed in the paper [3].

Precipitation of η-carbides preceded by the formation of atoms clusters have been widely accepted explanation of the wear resistance improvement of tool steel [1,2]. In this explanation, however, there are some inconsistencies in relation to the generally accepted precipitation sequence occurring during tempering of quenched steel [4]. In most cases tool steels are used in the heat treated for secondary hardness state and after tempering at a temperature of above 400°C (in DCT treated steel or in steel without that treatment) the ε and η type carbides, as transition carbides, are absent. In the precipitation processes sequence, however, these carbides are „in situ" and „independent" nucleation sites of cementite and therefore they are responsible for increasing the properties of the steel after heat treatment for secondary hardness.

According to current literature reports, in addition to the athermal martensitic transformation, very important role appears to play a martensitic transformation with the isothermal kinetics, occurring at temperatures range of –100 ÷ –196°C [5,6].

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The processes taking place during the isothermal martensitic transformation and subsequent processes of nucleation and growth of carbides, occurring during the heating to the tempering temperature and soaking at this temperature, seem to play a key role in explaining the effect of DCT on the properties of the steel.

2. Experimental

The high speed steel HS6-5-2 steel was austenitized at a temperature of 1200°C and quenched with nitrogen in a vacuum furnace with high pressure gas quenching system. Deep cryogenic treatment was carried out in a cryogenic processor at −180°C for 24 hours. After quenching or quenching and DCT treatment the steel was subjected to double tempering at different temperatures in the range of 490÷570°C. Parameters of heat treatment processes of steel samples are presented in table 1.

TABLE 1

Parameters of heat treatment of HS6-5-2 steel

	Austenitizing	Quenching	DCT	Double tempering
1	Austenitize (1200°C)	Quench (nitrogen, 4 bar)	———	490°C
2				510°C
3				530°C
4				550°C
5				570°C
6			DCT (−180°C, 24h)	490°C
7				510°C
8				530°C
9				550°C
10				570°C

Rockwell hardness tests were carried out using a laboratory hardness meter. Observations of the metallographic specimens (etched with 3-% Nital or Murakami reagent) were performed with aid of metallographic microscope KEYENCE VHX 5000 with magnification up to ×2500. Electron microscopy imaging was carried out with aid of HITACHI HD2700 STEM microscope using foils made of rolls with a diameter of 3 mm.

## 3. Results of tests

3.1. Microstructure

Observations of microstructure of steel subjected to Nital etching allowed to determine the size of the primary austenite grain size and observe primary carbides and secondary carbides in the material's matrix. The average size of the primary austenite grains for steel subjected to quenching (without tempering, Fig. 1a) was approx. 8-10 mm, which corresponds to 10-11 on the ASTM scale. This parameter remained unchanged in case of deep cryogenic treatment process (Fig. 1b). At the same time, it should be noted that the specified grain size is of an estimated nature and is burdened with a rather large measurement error. In [7], one can find a statement that in the case of deep cryogenic treatment, this value may change by approx. 4 to 6%. The size of the primary austenite grains also did not change after tempering processes in the range of 490 to 570°C.

In the samples tempered at 550°C martensite plates are visible (Fig. 2a, 2b), while in case of untempered samples the marensite morphology remains undisclosed (Fig. 1a, 1b). In the case of the variant involving the quenching and DCT (Fig. 1b) pay attention also strongly outlined the primary austenite grains. Strongly outlined primary austenite grains and undisclosed morphology of martensitic matrix can be related to the state of internal stress.

In order to reveal the presence of carbide precipitates and facilitate the evaluation of their quantity, observations of metallographic specimens subjected to etching with Murakami reagent were carried out. Observations were made at 2500× magnification (Fig. 3). Etching with Murakami reagent allowed to notice the differences between microstructures of samples subjected to
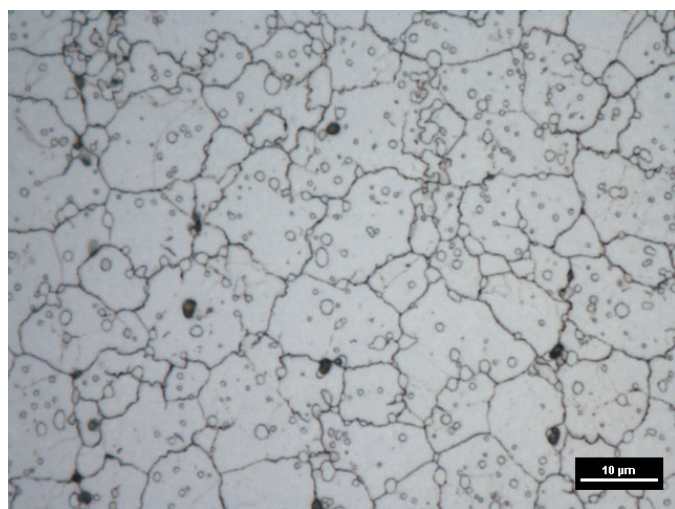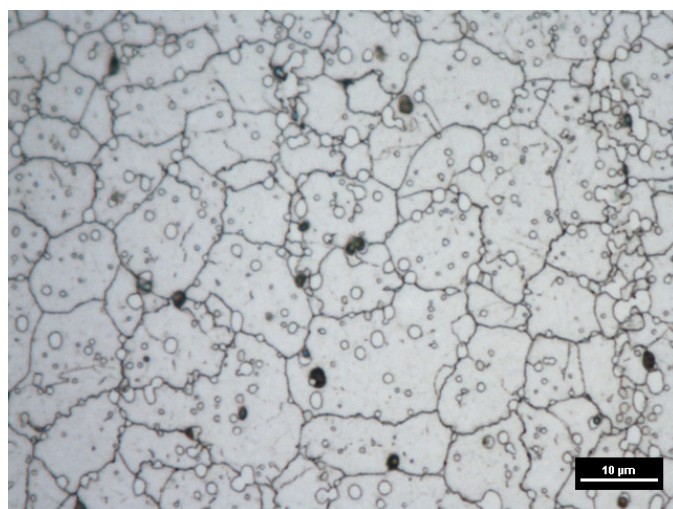
a)   b)

Fig. 1. Microstructure of HS6-5-2 steel subjected to a) quenching or b) quenching and DCT (Nital etched, LM)
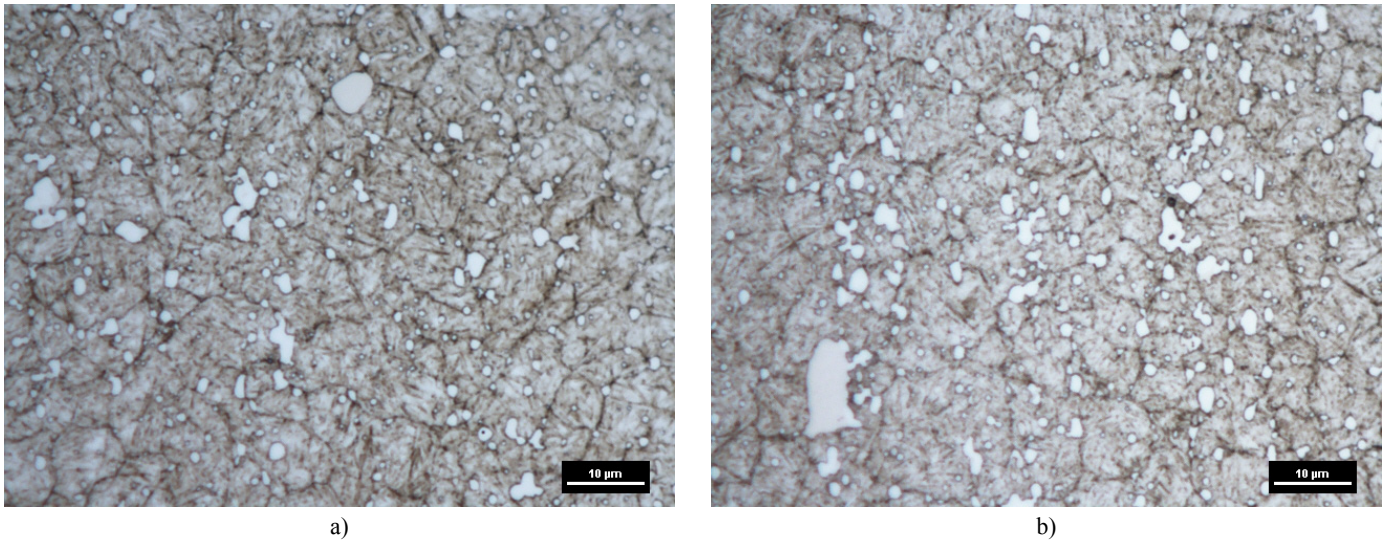
Fig. 2. Microstructure of HS6-5-2 steel subjected to a) quenching and tempering at 550°C or b) quenching, DCT and tempering at 550°C (Nital etched, LM)
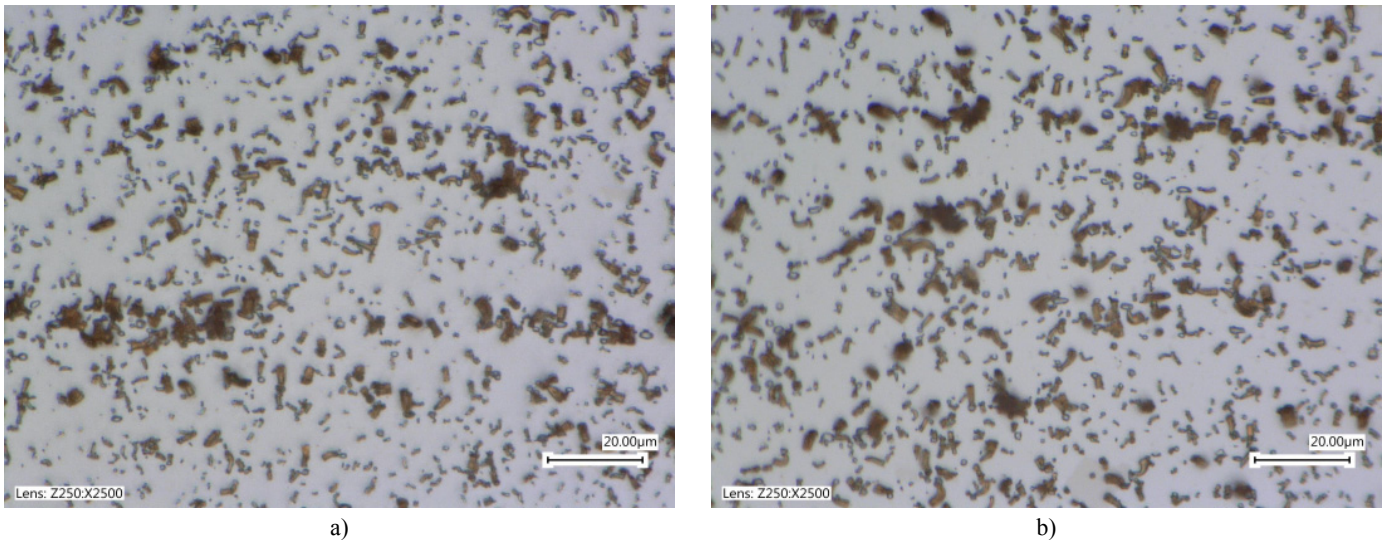

Fig. 3. Microstructure of HS6-5-2 steel subjected to a) quenching and tempering at 550°C or b) quenching, DCT and tempering at 550°C (Murakami reagent etched, LM)

DCT or without this treatment for all used tempering temperatures. Carbides with unrecognized size with dimensions smaller than 1 μm were disclosed in higher amount in microstructure of deep cryogenically treated samples, which may be associated with the changes taking place in the matrix of material, affecting the increase in the quantity of nucleation sites. More detailed analysis of differences in the amount of precipitated carbides requires in-depth studies involving quantitative image analysis. Exemplary microstructures of steel subjected to tempering at 550°C are presented in Figure 3.

Increased quantity of precipitated carbide phases was also observed by the authors of several works [8]. It should be noted that authors frequently use much lower tempering temperature after DCT, which allows the forming and observation of the transition η-type carbides, precipitating at temperatures below 200°C [2,9]. Above this temperature these phases undergo dis-

solution and subsequent changes associated with the precipitation of cementite and other carbide phases. The observed in this work carbides are probably MC and M_2C type carbides. Their higher amount may be associated with changes taking place in the material's matrix, affecting the increased quantity of their nucleation sites.

Steel microstructure observed using SEM of samples subjected to heat treatment processes with or without DCT and subsequent tempering at a temperature of 550°C are shown in Figure 4. SEM observations of steel samples allowed observing of two types of primary carbides in the metal matrix. The smaller carbides of spherical shape and a diameter of approx. 1 μm (probably VC carbides) were located usually inside the martensite grains, while oval-shaped carbides with a larger diameter of approx. 2 μm (probably M_6C carbides), at the grain boundaries [10].
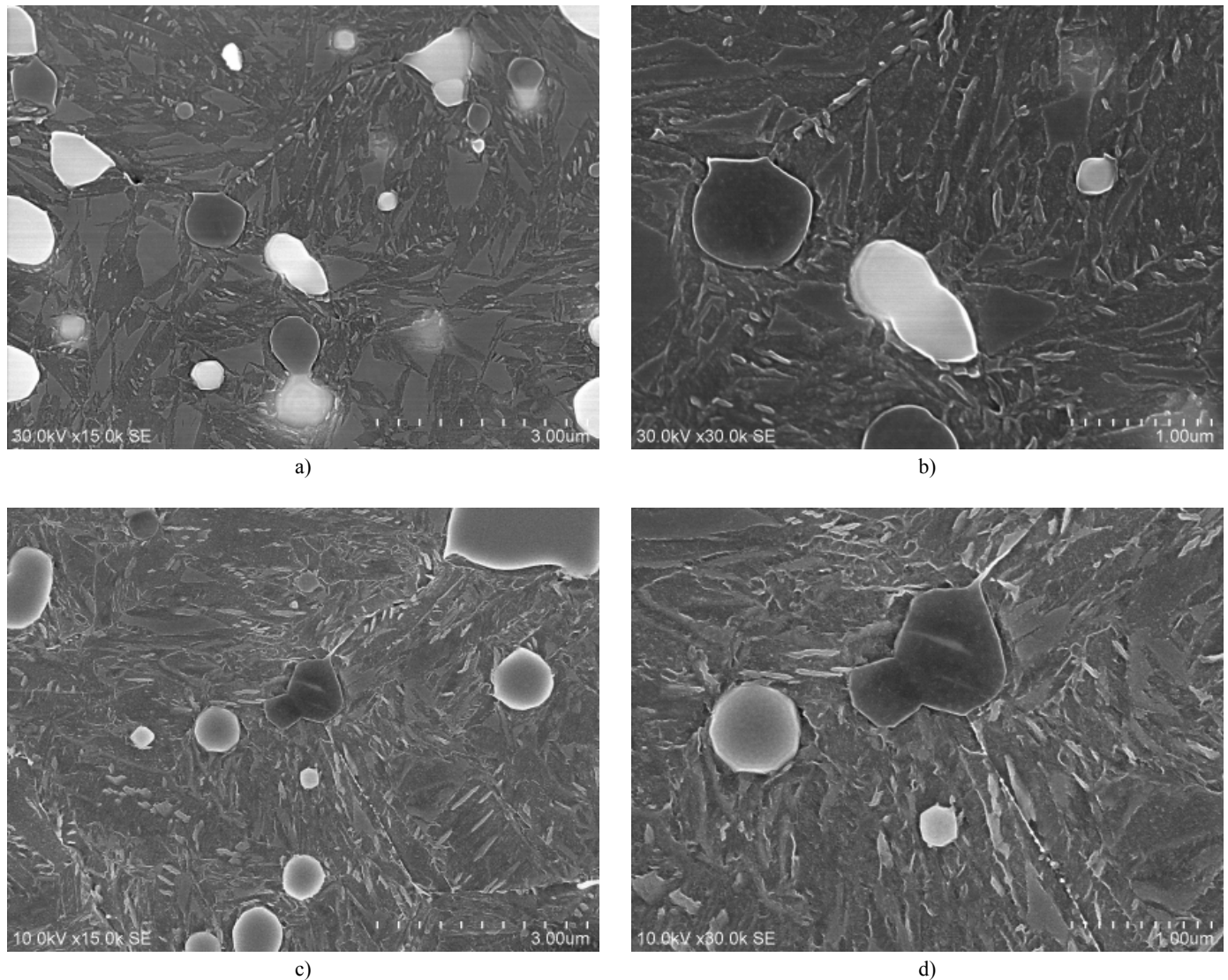
Fig. 4. Microstructure of HS6-5-2 steel subjected to a, b) quenching and tempering at 550°C or c, d) quenching, DCT and tempering at 550°C (SEM)

Moreover, SEM observations allowed observing the martensitic matrix morphology. Martensite has the lamellar-lenticular structure, internally twinned, characterized with irregular surfaces limiting the plates. SEM imaging showed a significant difference between the substructure of the martensite plates, whose length and width were several times smaller for the steel subjected to DCT (Fig. 4a,b and 4c,d). There was also a significant difference in the number and size of secondary carbides present in the form of rods arranged parallel to one another, which may suggest their nucleation and growth at the same network planes. Carbides in the form of rods in the deep cryogenically treated steel had several times larger dimensions, reaching a length up to approx. 6 µm and a width of approx. 0.5 µm. Larger sizes of carbides may be due to the phenomenon of displacement to the lower temperatures of processes of their nucleation and growth, shown also during DSC (Differential Scanning Calorimetry) studies in previous work of the author of the paper [10]. Shifting to lower temperatures of steps of carbides nucleation and growth in the process of tempering was all suggested by the results of TEM diffraction studies [10]. During the tests of deep cryogenically treated HS6-5-2 high speed steel, the higher intensity of the reflections derived from fine carbides was observed, which may suggest their presence of a higher quantity.

The use of transmission electron microscopy TEM allowed observing of differences in the substructure of steel treated with or without DCT. Dimensions of single martensite crystals after the DCT were significantly smaller (Fig. 5a, 5b).

3.2. Hardness

The hardness obtained after quenching or quenching and DCT have been marked in Figure 6 by a dashed line. In this figure has also been marked measuring points and trend lines showing the change in hardness of the steel occurring during tempering in the temperature range from 490 to 570°C. Measurement of HRC hardness showed that the hardened high speed steel has a hardness of 65,1 HRC. Process of deep cryogenic treatment enabled obtaining of higher hardness (66,1 HRC), which is certainly connected with continuing of martensitic transformation
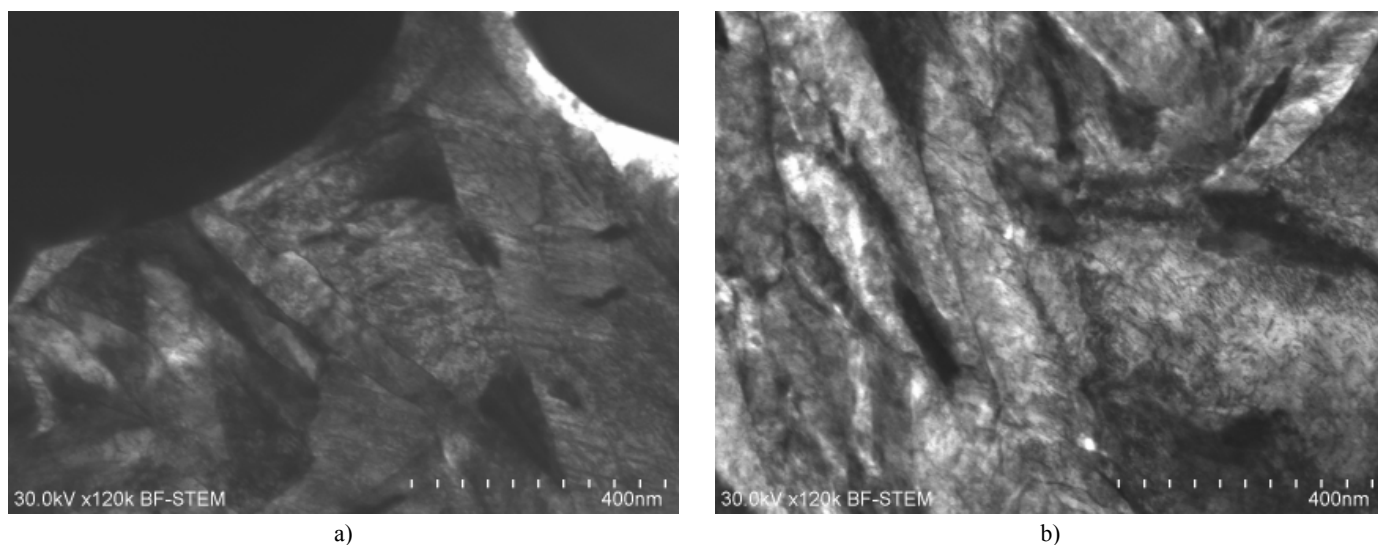
Fig. 5. Microstructure of HS6-5-2 steel subjected to a) quenching and tempering at 550°C or b) quenching, DCT and tempering at 550°C (TEM)

at lower temperatures and forming of a structure with a much lower retained austenite content [2]. Deep cryogenic treatment process led to shifting of the maximum hardness peak to the lower temperature and the reduction of the obtained maximum hardness. The resulting maximum hardness after tempering was lower by approx. 1 HRC. Shifting of the secondary hardness peak to lower values at the tempering curve of high speed steels was also observed by authors in the paper [11]. In addition, the reduction of the maximum hardness was observed also in works [8,12-14].

Observed changes in hardness may be due to the shifting of the stage of nucleation and growth of carbide phases to lower temperatures or the changes taking place in the matrix, connected with the additional transformation of the martensite at sub-zero temperatures and more extensively occurring precipitation processes, lowering the content of the carbon in the martensite, determining thereby its lower hardness. During tempering the processes connected with lowering of hardness (tempering of martensite and precipitation of carbides lowering the hardness of the matrix) and increasing of hardness (transformation of austenite and carbide precipitation processes) are superimposed on each other and determine the obtained final hardness [15]. In the steel subjected to DCT process the lower retained austenite content will determine a smaller share of its transformation on the obtained after tempering the hardness of material. Obtained after tempering lower hardness may be therefore the result of the impact of the process of DCT on the retained austenite content and proceeding in an altered manner precipitation processes.
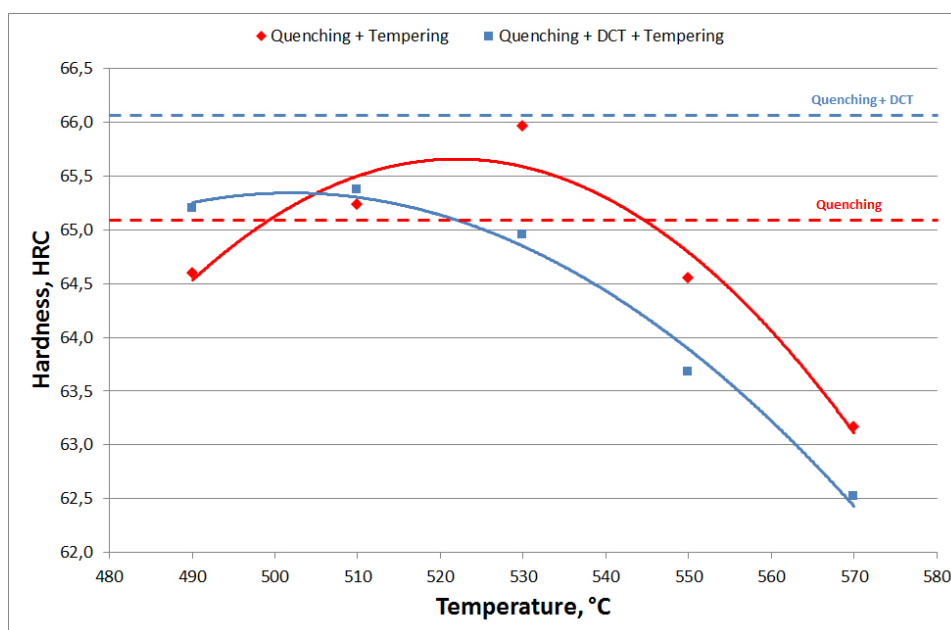

Fig. 6. Hardness of HS6-5-2 steel after tempering at different temperatures

4. Discussion

The key issue in the explaining of the influence of DCT on properties of high speed steel is a description of phenomena initiated by sub-zero treatment, taking place in the materials structure. Attempts to make the description of these phenomena should take into consideration the structure and morphology of phases subjected to heat treatment, as well the impact of their changes on the altered precipitation processes occurring during tempering process of steel. The mechanical properties of deep cryogenically treated high speed steels are closely related to the phenomena occurring during the heating-up of the material from liquid nitrogen temperature to the room temperature and during the subsequent tempering. Occurring phenomena are associated with the processes of decomposition of martensite, the change of crystal's morphology, refinement of martensite substructure and processes of precipitation from the altered matrix of significant amounts of homogenously distributed fine carbides.

Analysis of the current literature reports leads to the conclusion that the process of DCT should be considered as a process that allows proceeding of the isothermal martensitic transformation occurring in high carbon steels at temperatures $-100 \div -196°C$ [5,6]. The change in size of the martensite crystals observed in deep cryogenically treated HS6-5-2 steel (Fig. 5) may be related with such transformation. According to accepted explanation the freshly formed in this way orthorhombic κ martensite is relatively soft and, due to the differences in specific volumes of martensite and retained austenite, is subjected to considerable deformation leading to an increase in dislocations density. The consequence of the plastic deformation of the martensite is increased density of dislocations, movement of dislocations and capturing by them of the carbon atoms which form clusters that are sites of nucleation of carbide phases precipitates. During the tempering, clusters present at the dislocations evolve do rod-like precipitations at densely packed with atoms network planes $\{100\}\alpha$. In DCT-treated steel, these precipitations seem to appear in much higher quantity than in case of conventional heat treatment (Fig. 4, 5).

Processes taking place during the isothermal martensitic transformation and subsequent processes of nucleation and growth of carbides, occurring during the heating up to the tempering temperature and soaking at this temperature, seem to play a key role in explaining the effect of DCT on the properties of the steel. Altered precipitation processes occurring during tempering of the deep cryogenically treated HS6-5-2 steel led to shifting of maximum hardness peak to the lower temperature and the reduction of the obtained maximum hardness. This effect of DCT is associated most likely with:

1. a reduced content of retained austenite, and therefore lower hardening of steel caused by transformation of austenite during tempering,
2. altered precipitation processes, leading to a higher quantity of fine secondary carbides, due to changes made in the matrix and creating of additional nucleation sites by movement of the mobile dislocations.

5. Conclusions

The use of deep cryogenic treatment process in the heat treatment cycle of tool steel requires performing of tempering process at a specific temperature adequately selected to precipitation processes occurring in a different manner. Deep cryogenic treatment of HS6-5-2 high speed steel leads to shifting of maximum hardness peak to the lower temperature and the reduction of the obtained maximum secondary hardness. These changes in hardness may be due to the shifting of the stage of nucleation and growth of carbide phases to lower temperatures or the changes taking place in the matrix, connected with the additional transformation of the martensite at sub-zero temperatures and more extensively occurring precipitation processes, lowering the content of the carbon in the martensite, determining thereby its lower hardness. In order to achieve the maximum hardness of this steel it is recommended to carry out tempering at a temperature of about 40°C lower than maximum hardness peak from the tempering curve designated for steel without sub-zero process.

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