

MICROSTRUCTURAL CHANGES IN HEAT TREATMENT OF PM HIGH-SPEED STEELS

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Abstract: Properly performed heat treatment can significantly affect final properties of high-speed steels, so the heat treatment parameters are chosen depending on the specific properties of the particular tool. Deep cryogenic treatment is carried out at a temperature of $-196\text{ }^{\circ}\text{C}$ (liquid nitrogen). By combining deep cryogenic treatments and various austenitization temperatures, which affects the volume of retained austenite, it is possible to affect the hardness and fracture toughness of tools and other steel parts. However, it may also affect the increase in wear resistance regardless of the increase in hardness. In addition, dimensional stability is achieved by transformation of residual austenite into martensite, eliminating the change in volume by 4%. This paper investigated the effect of deep cryogenic treatment on microstructures in high-speed steel produced by powder metallurgy in comparison to vacuum heat treatment.

Keywords: deep cryogenic treatment; high-speed steel; microstructure

1 INTRODUCTION

Properly performed heat treatment can significantly influence the finite properties of high-speed steel, so the heat treatment parameters are chosen depending on the specific properties of the particular tool. The high-speed steel heat treatment consists of austenitization in a protective atmosphere, quenching and three high temperature tempering treatments. High temperature tempering treatments are conducted for the purpose of the elimination of residual austenite, which is present in the microstructure after quenching up to 40% (depending on the height of austenitization temperature). The austenitization temperature, which is usually around $50\text{ }^{\circ}\text{C}$ below the solidus temperature, is a very important factor in the heat treatment of high-speed steels.

In an annealed condition, the ferrite matrix of high-speed steel is almost carbon-free and consists predominantly of iron and chromium. Carbon is bound to eutectic, eutectoid, and secondary carbides. Although, in high-speed steels the austenitic area starts at a temperature somewhat less than $900\text{ }^{\circ}\text{C}$, the carbon content in the matrix is low, and almost all the carbides are out of the solid solution. Very high austenitization temperatures allow for the dissolution of sufficient amount of carbide to obtain a sufficient amount of carbon and alloying elements in the austenite. At temperatures from $900\text{ }^{\circ}\text{C}$ to $1100\text{ }^{\circ}\text{C}$, M_{23}C_6 carbide dissolves. M_6C carbides dissolve at an interval of $1150\text{ }^{\circ}\text{C}$ to the solidus temperature, and the minor part will melt only at liquidus temperature. MC carbides dissolve very poorly and are still present in large amount at liquidus temperature. Partial dissolution of the M_6C carbides results in a partial entry of the vanadium into a solid solution resulting in stronger pronounced secondary hardening with tempering [1-3]. Kinetics of carbide dissolution depends on the duration and height of the austenitization temperature and on the quenching technology, i.e. the duration of heating to the austenitization temperature.

The usual holding time at austenitization temperature is from 80 to 150 s [3], which is enough to dissolve sufficient amount of carbide and not affect the dimensions and mass of the tool. The austenitization temperature is also selected according to the properties of the tool. Higher austenitization temperatures are chosen for tools that will work at high temperatures, requiring primarily high hardness; lower austenitization temperatures are selected for cutting tools that will work in cold conditions and where the toughness of the tool is very important.

After quenching, in the microstructure of high-speed steel beside martensite there is 20% to 40% of the retained austenite present. Secondary and eutectic carbides are also present. The retained austenite may be transformed into martensite by deep cryogenic treatment at $-196\text{ }^{\circ}\text{C}$ or by destabilizing with tempering followed by rapid cooling to room temperature. The tempering is performed in two or three steps in the temperature range between $500\div 600\text{ }^{\circ}\text{C}$. At the tempering temperature, carbon and alloying elements from martensite and the retained austenite form tempering carbides, which reduces the tetragonality of martensite and thus hardness. Partially decreased amount of carbon in the retained austenite leads to the increase of the martensite start temperature, and the austenite with the lower carbon content becomes less stable and starts to transform into martensite when cooling to room temperature.

With multiple tempering in quenched martensite multiple tempering carbides form followed by secondary martensite forming from the retained austenite. This significantly reduces the amount of the retained austenite. The result is an increase in tool hardness and increased wear resistance.

Tempering carbides form in martensite during the tempering at the temperatures around $500\text{ }^{\circ}\text{C}$ and are M_3C type in HS 6-5-2 high-speed steel or M_{23}C_6 type in HS 18-0-1 high-speed steel [4]. Up to $400\text{ }^{\circ}\text{C}$ only M_3C carbides form. Also, cementite formation is established at the same temperature interval. At $600\text{ }^{\circ}\text{C}$ carbon is almost entirely bonded within the carbides [2].

At higher tempering temperatures there is no new carbide formation, but only transformation of one to another type of carbide, or the dissolution of one carbide type and the formation of the other. In case of the present vanadium in steel, in secondary hardening at 550 °C, the dominant carbide type is MC (VC, V₄C₃). It has higher crystal lattice parameter, and in the microstructure, it can be observed on the boundaries of martensitic needles [3].

1.1 Deep - cryogenic treatment

For the purpose of eliminating the retained austenite, experiments with cryogenic treatment started at the beginning of the 20th century. However, due to poor equipment and the inability to achieve temperatures below -80 °C, the results were not satisfactory. With the development of equipment and low-temperature techniques in the 70s of the 20th century, it was possible to achieve very low temperatures – up to -196 °C – enabling the intensification of the research on the influence of deep cryogenic treatment to the various steels properties.

Cold treatment at about -80 °C ("dry ice") to -120 °C is sufficient to transform the retained austenite from quenched steel to martensite (Fig. 1) and it is in wide commercial application, particularly for removal of the retained austenite in carburized and surface quenched parts. For high alloyed tool steels, deep cryogenic treatment improves dimensional stability because of the retained austenite transformation to martensite thus eliminating 4 % volume change [6÷11].

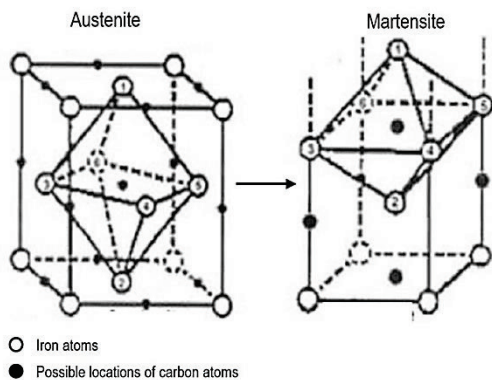


Figure 1 Transformation of the retained austenite into martensite [6]

Deep cryogenic treatment (DCT) is carried out at a temperature of -196 °C (liquid nitrogen). By combining DCT and various austenitization temperatures, affecting the volume of retained austenite, it is possible to affect the hardness and fracture toughness [12, 13] of certain components, but it can also affect the increase in wear resistance regardless of hardness. [11, 14]

In DCT two metallurgical phenomena occur: transformation of retained austenite into martensite with the start of nucleation for subsequent precipitation of very fine carbides from the martensite. It has been shown that the effect of DCT takes place in several stages. In the first stage, up to -130 °C, a transformation of the retained

austenite to martensite occurs, resulting in increased hardness. Meng et al. [10] state that the martensite formed during DCT is not equivalent to the martensite formed by quenching and tempering. This transformation, as in classical heat treatment, is not time-dependent.

At the second stage, at about -196 °C, time-dependent decomposition of the primary martensite begins. This decomposition causes decrease of steel hardness, but there is a nucleation of a large number of fine-dispersed nanometer carbides. During the subsequent tempering, formation and precipitation of very fine nanometric η -carbides occurs (Fig. 2) [10, 11]. According to Hirotsu and Nagakura (1972) [15, 16] η -carbides are type M₂C with orthorhombic crystal lattice with the parameters $a = 4.525$ Å, $b = 4.318$ Å, $c = 2.830$ Å, with the following gaps between plane $d_{101} = 0.242$ nm, $d_{020} = 0.215$ nm, $d_{121} = 0.161$ nm. The number of these carbides has been shown to increase with the duration of the treatment (minimum 24 h) [15], a lower temperature (optimally -196 °C), and lower austenitization temperature (with the aim of formation as much primary martensite as possible). Meng et al. [10] have found that the dominant mechanism of increasing wear resistance in tool steels is the formation of these very small nanometric η -carbides.

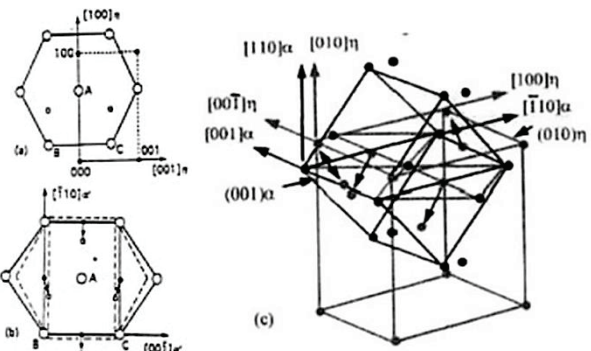


Figure 2 Formation of the η -carbides from martensite [10]

The purpose of this research was to examine the influence of deep cryogenic treatment on the microstructure in high-speed steel produced by powder metallurgy compared to classical heat treatment.

2 EXPERIMENTAL PROCEDURE

The material used in this study was a high-speed steel grade PM S390 MC produced by powder metallurgy. The chemical composition of steel is shown in Tab. 1.

Table 1 Chemical composition of the PM S390 MC high-speed steel

%C	%Si	%Mn	%Cr	%Mo	%V	%W	%Co	%Fe
1.64	0.60	0.30	4.80	2.00	4.80	10.40	8.00	rest

Heat treatment was conducted in an Ipsen vacuum furnace. Samples were preheated at the 650 °C / 30 min, 850 °C / 20 min, 1050 °C / 15 min before the heating to the austenitization temperature. Heating to the austenitization temperature of 1130 °C was 5 °C/min. Duration of the austenitization was 6 min. After that, the samples were

quenched in the nitrogen flow, $p = 1050$ mbar. The heat treatment parameters are shown in Tab. 2.

Table 2 Heat treatment parameters

Batch	Austenitization, °C/min	Deep cryogenic treatment, °C/h	Tempering, °C/h
1.	1130 / 6 min	-	520 / 520 / 490 / 2 h
2.	1130 / 6 min	-196 °C / 24 h	520 / 2 h

After quenching, the test samples were immersed in liquid nitrogen at controlled speed and left for 24 h. Other samples were tempered three times. After tempering and deep cryogenic treatment, the Rockwell hardness and HV 1 were measured. On each sample hardness was measured ten times. The results represent the mean value of ten measurements. The test samples were excluded after each of the individual heat treatment phases and analyzed on the field-emission scanning electron microscope. For microstructure testing, the samples were prepared by a standard metallographic preparation procedure. The microstructure was tested on the FE SEM JEOL JSM6500F electron microscope. For microstructure testing, the samples were etched with 3% Nital solution.

3 RESULTS AND DISCUSSION

Tab. 3 shows hardness test results. For this research, the austenitization temperature and the tempering temperature were chosen for both batches of the heat treatment to obtain high hardness and wear resistance. Since it is recommended for deep cryogenic treatment to choose the lowest austenitization temperature to obtain as many primary martensite as possible and less retained austenite after quenching, the hardness test results show that in the classical heat treatment the highest hardness was achieved after quenching.

Table 3 Hardness of the test samples after conducted heat treatments

Heat treatment		HRC	HV 1
1.	Austenitization (1130 °C / 6 min) + quenching	67.5	-
	1. tempering (520 °C / 2 h)	67	-
	2. tempering (520 °C / 2 h)	66	-
	3. tempering (490 °C / 2 h)	66	917
2.	Austenitization (1130 °C / 6 min) + quenching	67.5	-
	Deep cryogenic treatment (-196 °C / 24 h)	68	-
	1. tempering (520 °C / 2 h)	66.5	945

The microstructure of the tested HSS PM S390 MC samples in annealed condition and after quenching is shown in Fig. 3.

Figs. 4 and 5 show a comparison of the microstructural changes with the quenching and tempering and with the deep cryogenic treatment.

The microstructure analysis showed a characteristic microstructure of high-speed steel produced with powder metallurgy. Eutectic carbides of nodular shape with a diameter of less than 1 μm are evenly distributed over the martensitic matrix. EBSD and EDS analysis [1] identified two types of carbides, M_6C ($\text{Fe}_3\text{W}_3\text{C}$) and MC (VC) carbides. On the larger magnification of the microstructure shown in Figs. 4 and 5, secondary carbides of the M_3C type

are also visible. MnS and complex sulfides $(\text{Mn}, \text{Cr}, \text{Fe})_x\text{S}_y$ were also present in the microstructure.

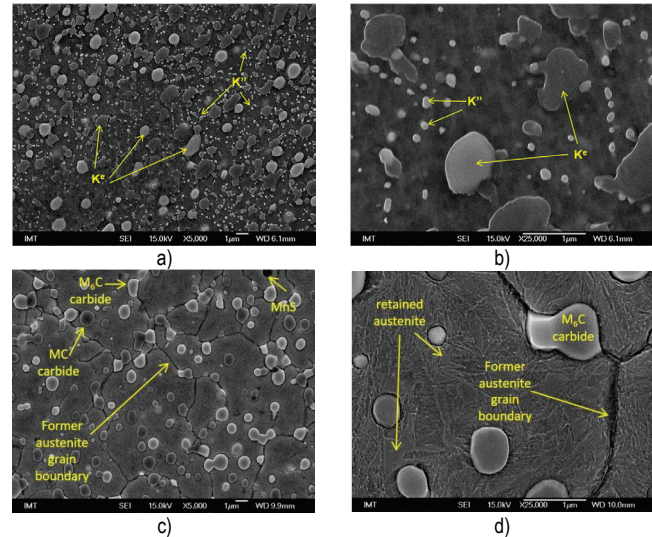
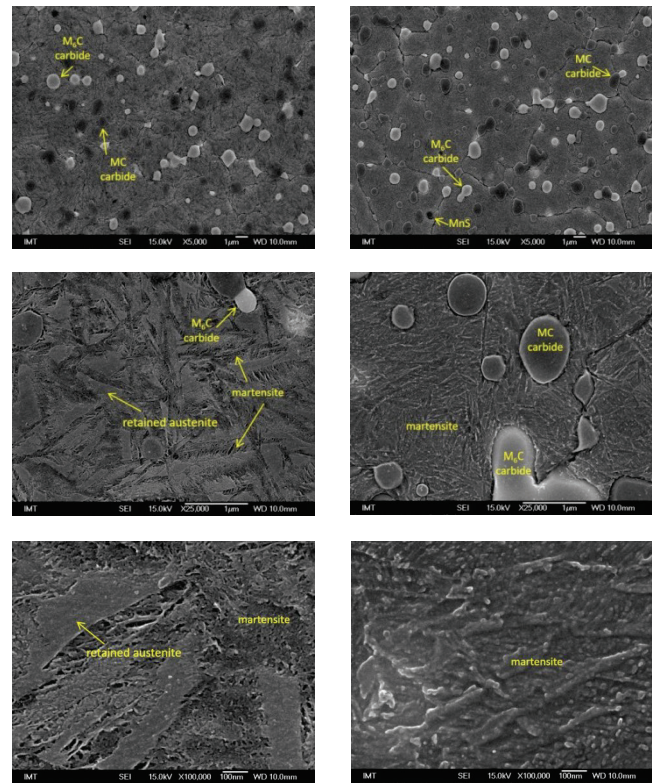


Figure 3 Microstructure of the PM S390 MC HSS [1]

a) annealed condition, b) annealed condition, higher magnification
c) quenched condition, d) quenched condition higher magnification



Quenched + 1 tempering (520 °C/2 h)

Quenched + DCT (-196 °C/24 h)

Figure 4 Microstructure of the PM S 390 MC HSS [1]

In Fig. 5, at a large magnification, the martensitic matrix of a deep cryogenic treated sample appears to be finer, i.e. martensitic needles seem to be smaller than those in quenched and tempered samples.

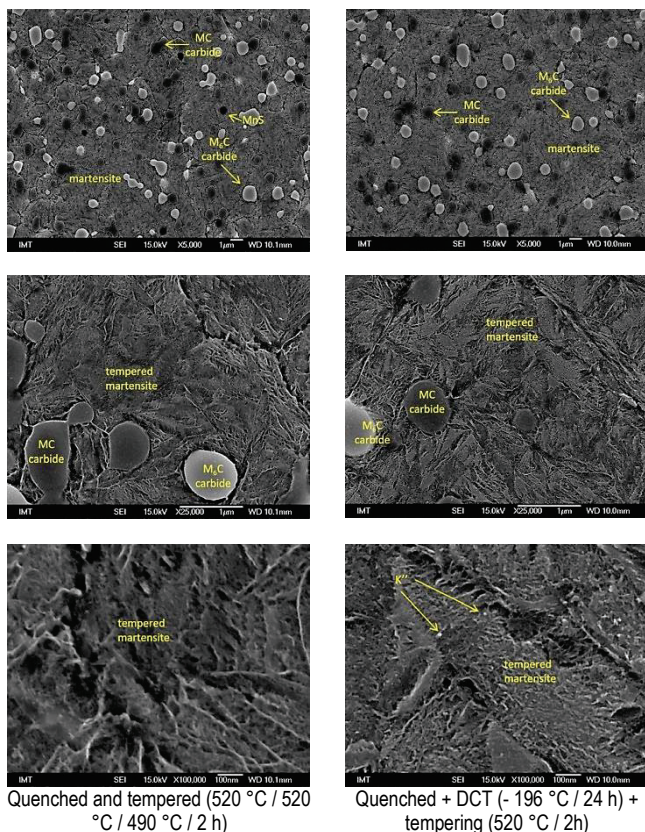


Figure 5 Microstructure of the PM S 390 MC HSS after heat treatments [1]

4 CONCLUSION

Properly conducted heat treatment can significantly influence the finite properties of high-speed steels, so the heat treatment parameters are chosen depending on the specific properties of the particular tool. By combining deep cryogenic treatment and various austenitization temperatures, affecting the volume of the retained austenite, it is possible to affect the hardness and fracture toughness of certain components. Moreover, it can increase wear resistance regardless of changes in hardness values. In this paper, the effect of deep cryogenic treatment on the microstructure changes in high-speed steel produced by powder metallurgy compared to classical heat treatment was investigated. Metallographic analysis of samples showed a characteristic microstructure of high-speed steel manufactured with powder metallurgy. The higher magnifications of the microstructure showed differences in morphology of the martensitic matrix between classically heat treated and deep-cryogenically treated samples. Based on the obtained results, it is possible to conclude that the deep cryogenic treatment mechanism has effect on the high-speed steel microstructure which cannot be characterized by conventional metallographic methods but requires more thorough and precise procedures.

Note: This research was presented at the International Conference MATRIB 2017 (29 June - 2 July 2017, Vela Luka, Croatia).

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