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The Effects of Quenching and Tempering Treatment on the Hardness and Microstructures of a Cold Work Steel

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Abstract. The X153CrMoV12 ledeburitic chromium steel characteristically has high abrasive wear resistance, due to their high carbon and high chromium contents with a large volume of carbides in the microstructure. This steel quality has high compression strength, excellent deep hardenability and toughness properties, dimensional stability during heat treatment, high resistance to softening at elevated temperatures. The higher hardness of cryogenic treated samples in comparison with conventional quenched samples mean lower quantity of retained austenite as at samples quenched to room temperature and tempered in similar condition. In the microstructure of samples were observed that the primary carbide did not dissolve at 1070°C and their net structure have not been changed during to heat treatment. During to tempering at high temperature the primary carbides have become more and more rounded. After low tempering temperature in martensite were observed some small rounded carbides also, increasing the tempering temperature the quantity of finely dispersed carbides increased, which result higher hardness. The important issues in heat treatment of this steels are the reduction or elimination of retained austenite due to cryogenic treatment.

Introduction

Tool steels are divided into different category as: cold work, hot work, plastic mould, high speed tool steels [1, 2]. In many industrial applications tool steels are subjected to extremely high and variable loads. Cold work tool steels are used for fabricating stamping or forming dies, punches, forming rolls, knives, slitters, shear blades or any other component for shaping a material into a part or component adapted to a definite use. The mechanical properties of these steels can be improved by modifying of microstructure. The microstructure can be modified by using different heat treatments technologies.

Tool steel properties and its wear resistance can be enhanced by optimizing heat treatment parameters [3]. This type of steels is generally used in a quenched and tempered state and, therefore, the precipitation of secondary carbides and the evolution of the matrix in the steel during tempering determine the properties of the steel. The tempering process at unalloyed and low alloyed steels usually leads to a decrease in strength due to the precipitation of carbides from carbon that is originally in solid solution in the martensite.

Increasing the carbon content and alloy content of the steel such as by increasing the austenitising temperature and holding time, will produce a harder martensite, but simultaneously lower the M_s and M_f temperatures. After Hollomon and Jaffe [4] this temperature can be calculated

$$M_s = 550\text{-}350C\%\text{-}40Mn\%\text{-}35V\%\text{-}20Cr\%\text{-}17Ni\%\text{-}10Cu\%\text{-}10Mo\%\text{-}\ 8W\% + 15Co\% + 30Al\% \qquad (1).$$

Hougardy proposed a correction to this formula [5]:

$$M_s= 0.495M_{sjh}+0.00095M^2_{sjh} +40 \qquad (2)$$

M_s- Corrected martensite start temperature [°C]

M_{sjh} - Martensite temperature start according to Jaffe & Hollomon [°C]

Alloy amount-[weight %]

After Hougardy the volume fraction of martensite can be calculated by

$$V_M=1-\exp[-k(M_s-T)^q] \qquad (3)$$

$$k= 0.36x10^{-3}+0.10x10^{-4}\ M_s-0.34x10^{-6}\ M_s^2+0.32x10^{-8}\ M_s^3-0.52x10^{-11}\ M_s^4 \qquad (4)$$

$$q=2.088-0.76x10^{-2}\ M_s+0.16x10^{-4}\ M_s^2-0.90x10^{-8}\ M_s^3 \qquad (5)$$

Notation:
V_M -Volume fraction of martensite
T -temperature [°C]
M_s - Temperature at which 1% martensite forms [°C]

The presence of high carbon and high alloy content in tool steels lower their characteristic temperatures of martensite start and martensite finish [6]. Therefore, conventional hardening treatment of these steels fails to convert considerable amount of austenite into martensite often leading to unacceptable level of retained austenite in the as quenched structure of these steels. The retained austenite is soft and thus adversely affects the desirable properties such as hardness and wear resistance [7]. Moreover, the retained austenite is unstable and transforms into martensite at the service conditions of tool steels. The freshly formed martensite being untampered is very brittle and hence undesirable. Furthermore, transformation of austenite to martensite is associated with approximately 4% volume expansion [8], which leads to dimensional changes and distortion of the components, even failure in extreme cases [9].

Therefore, one of the major challenges in the heat treatment of tool steels is to minimize the amount of retained austenite or eliminate it.

Retained austenite content in tool steels can be reduced substantially by cryogenic treatment [10]. Multiple tempering and cryogenic treatments improves the strength and hardness of tool steels.

Studies on cryogenically treated tool steels show microstructural changes in the material that can influence tool service lives, material properties and productivity significantly. In the last years cryogenic treatment is getting increased attention in many tool applications. It is defined as an add-on process to conventional heat treatment if added after the conventional quenching and tempering process or as supplemental process between quenching and tempering [11]. In order to simultaneously increase strength and toughness properties, cold work steel grades may be alloyed to improve the morphology, size and distribution of the strengthening carbides [12].

Cryogenic treatments were also already used in order to improve the wear resistance and toughness of these steels [13-16]. Recent research work of Tyshchenko et al. [17] and Gavriljuk et al. [18] clearly shows that the full austenite-to-martensite transformation does not occur in high-carbon steels.

The purpose of this study was to investigate the influence of conventional and of cryogenically treatments effects on hardness and microstructure. There were studied the effects of multiple tempering on different tempering temperatures.

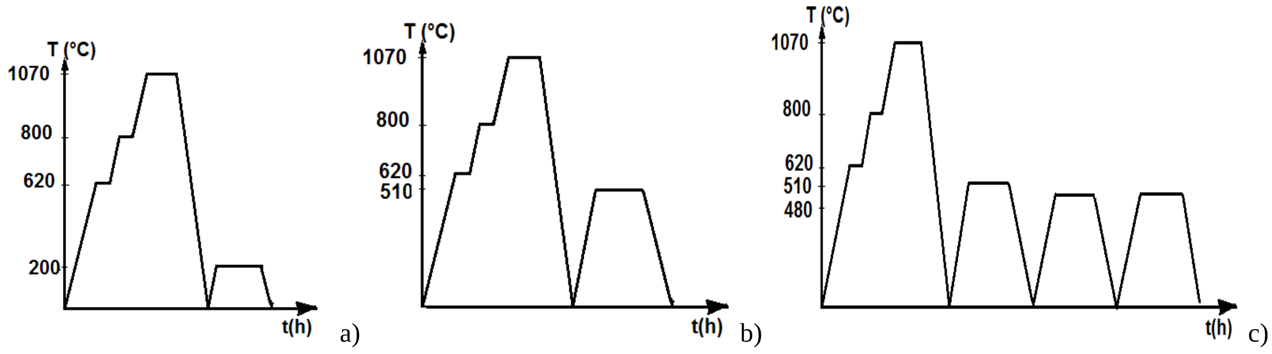
Experiments

The chemical composition of studied specimens is given in Table 1. Each specimen was cut and machined from rolled and soft annealed bars, subsequently heat treated in a horizontal vacuum furnace with uniform high pressure gas quenching using N_2 at a pressure of 5 bar.

1.2379	C(%)	Si(%)	Mn(%)	P(%)	S(%)	Cr(%)	Mo(%)	V(%)
	1,6	0,37	0,41	0,02	0,02	11,3	0,84	1,2

Table 1. Chemical composition of the steel in this investigation

The samples were preheated at the rate of 30°C/min up to 650°C and kept at this temperature for 15 minutes, after than were preheated with the same heating rate up to 900°C and kept it for 15 minutes. Then for austenitizing, these samples were heated at the rate of 15 minutes and kept at austenitizing temperature for 50 minutes. After it the sample were quenched in inert gas to 80°C respectively. Three of the samples were treated conventionally and the rest were cryogenically quenched (Figure 1).



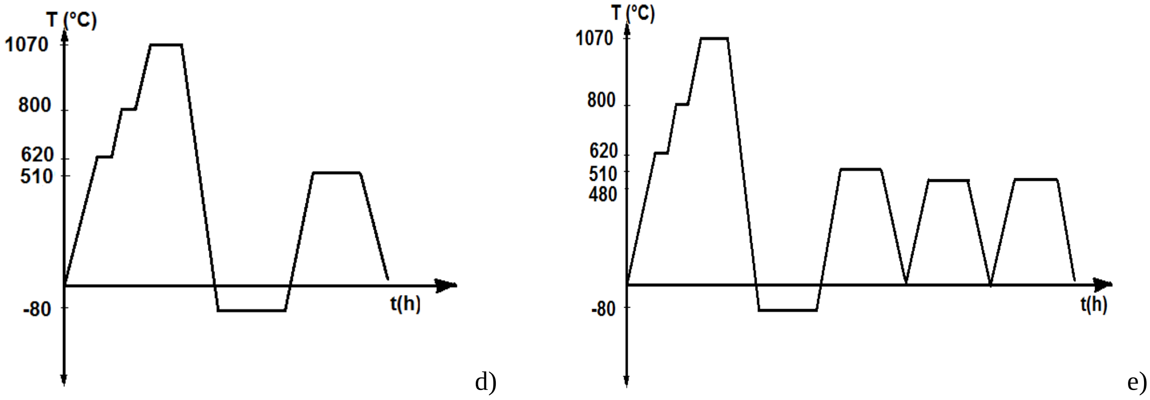


Figure 1. Heat treatments diagrams of specimens a) nr.1 b) nr. 2 c) nr.3 d) nr.4 e) nr.5

The microhardness testing were executed with Vickers testing machine type Buhler 1105. The microstructures of samples were studied by Olympus PMG3 microscope after conventional metallographic preparation. For etching were used nital 2%.

The microstructure of samples in delivered condition consist primary carbides on borders of the primary grains in the matrix with spheroid structure as it is showed in *Figure 2*. The hardness of samples in this conditions were 240HV$_1$.

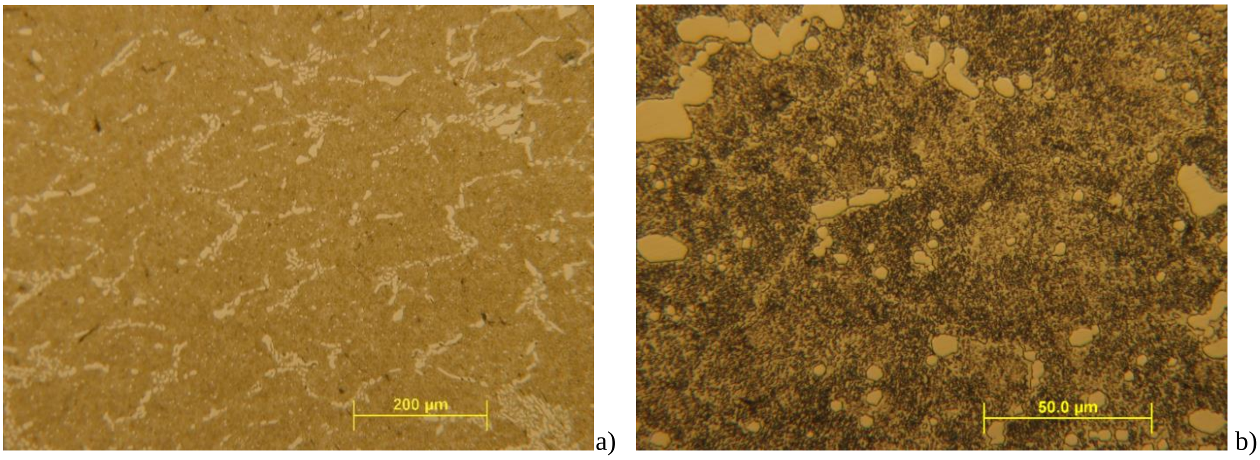


Figure 2. Micrograph of the samples in delivery condition: annealed a) N $_{original}$=100x b) N $_{original}$=500x

Results

In the microstructure of the samples after quenching were observed that the primary carbide did not dissolve at 1070°C during to austenitization and their net structure have not been changed during to heat treatment. The primary austenite grain sizes were ASTM 8. The primary carbides sizes are between 10-50 µm. In etched region small rounded carbides are visible as it showed in *Figures 3*.

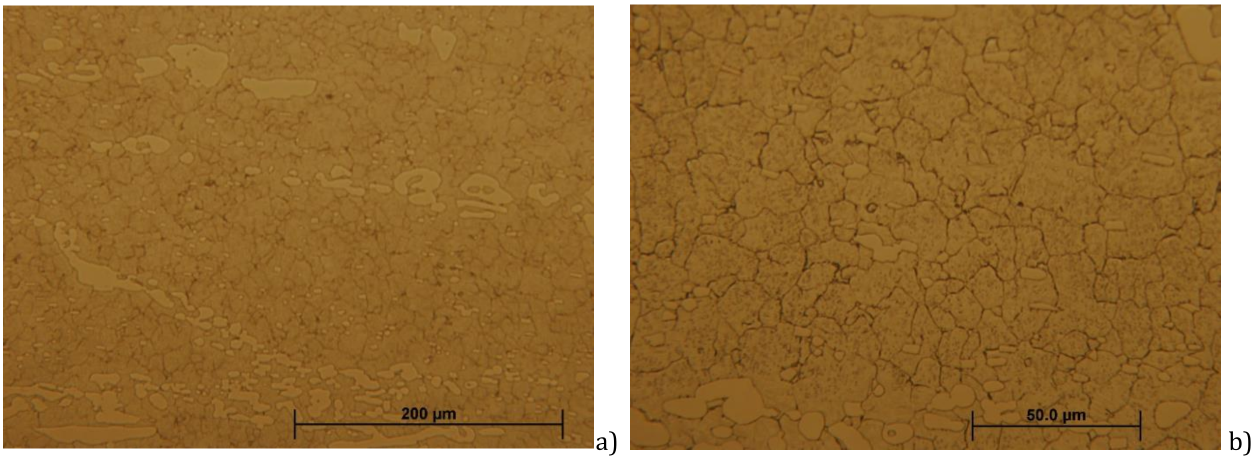


Figure 3. Micrograph of the quenched samples N $_{original}$=100x b) N $_{original}$=500x

The *Figure 4* show the microstructure of the samples heat treated conform Figure 1 a). The primary carbide shape and distribution is similar to quenched samples. In the etched structure became visible small compounds precipitations. The hardness of the matrix is 641 HV_1.

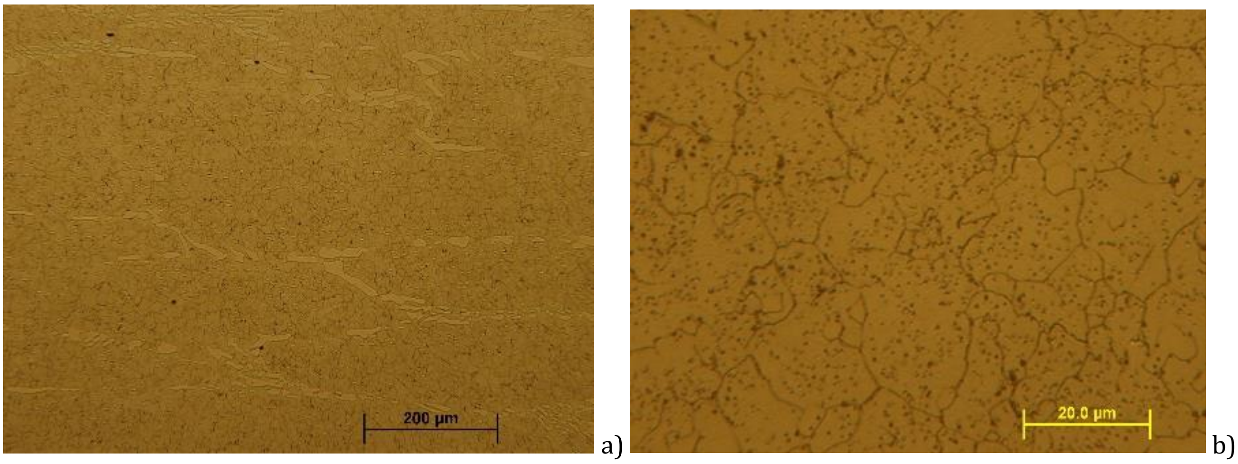


Figure 4. Micrograph of samples quenched and tempered at 200°C. a) N $_{original}$=100x b) N $_{original}$=1000x

After tempering at 510°C the matrix of the samples became homogenous as it presented in Figure 5.a). Studying with higher resolution in matrix can see retained austenite and tempered martensite near small rounded carbides (Figure 5 b).

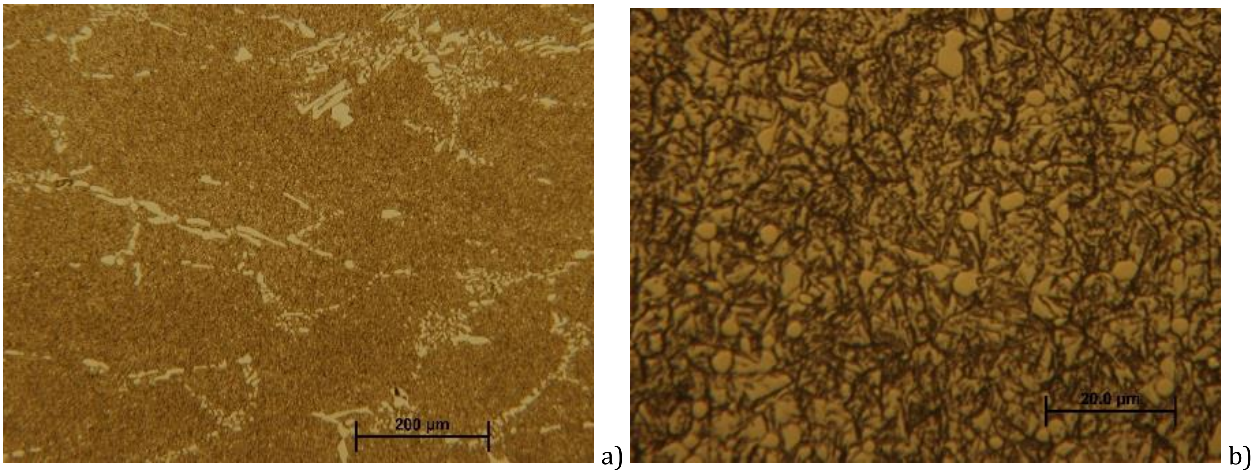


Figure 5. Micrograph of quenched and tempered sample at 510°C. a) $N_{original}$=100x b) $N_{original}$=1000x

The microstructure of a specimen tempered three times is show in *Figure 6.*

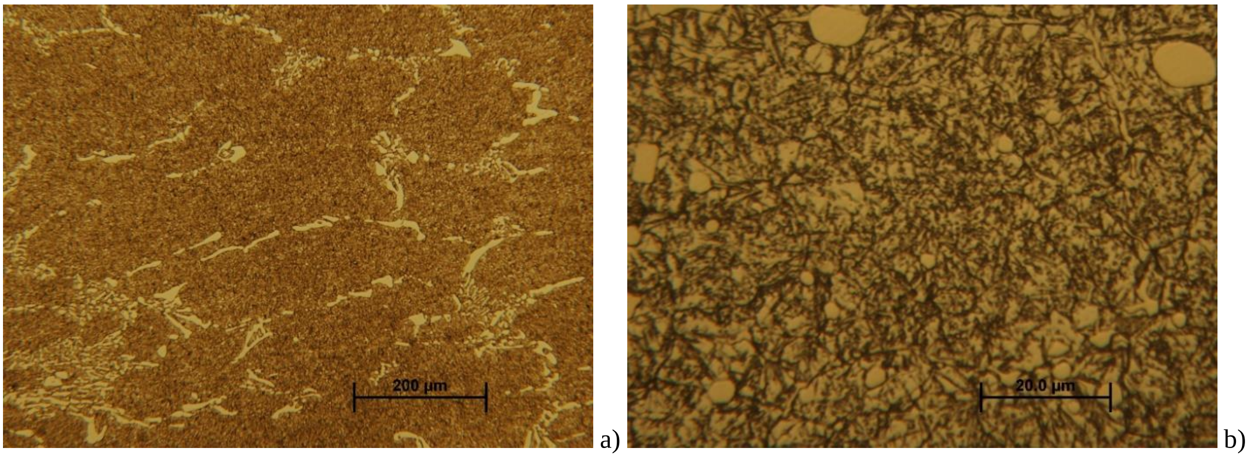


Figure 6. Quenched and triple tempered sample micrographs a) $N_{original}$=100x b) $N_{original}$=1000x

The study the microstructure of the conventionally quenched and triple tempered sample by optical microscope in high resolution show a dense precipitation of carbides and tempered martensite (Figures 6. b). The quantity of retained austenite is reduced compared with the sample tempered one time as it can be seen in Figure 6.b) and Figure 5. b).

The microstructure of cryogenically treated and tempered sample at 510°C is presented in Figure 7. In cryogenically treated sampled retained austenite quantity is decreased comparing with conventional quenched samples like in Figure 5. b) and Figure 7. b). The carbides precipitation are fines and their distribution are homogeneous in etched zones.

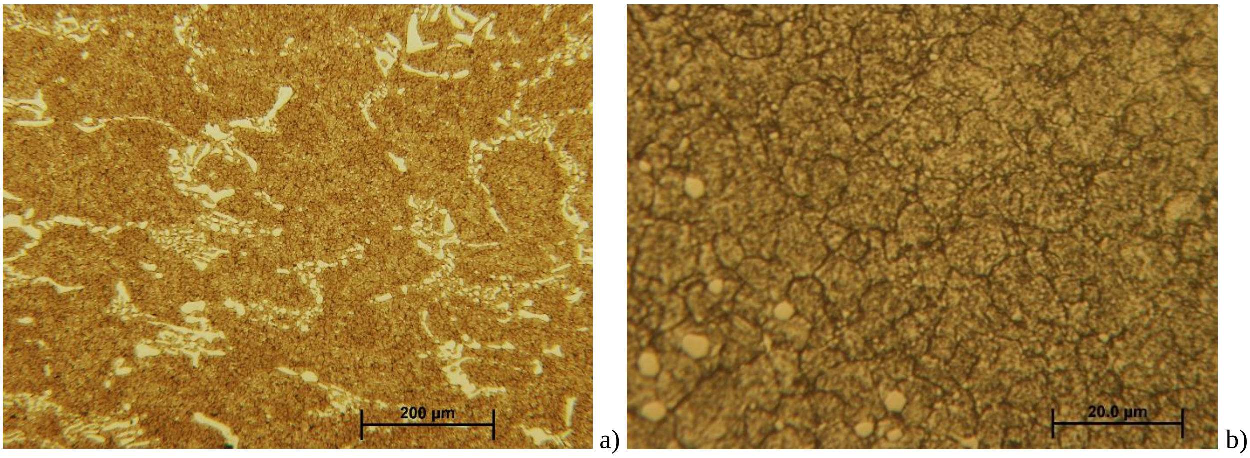


Figure 7. Microstructure of cryogenically treated and tempered sample at 510°C
a) N $_{original}$=100x b) N $_{original}$=1000x

Studying the microstructure of cryogenically treated and three time tempered samples (Figure 8) were observed partially reducing primary carbides sizes and their morphology became more spherical. The distribution of the precipitated carbides is denser and the martensite are full tempered. The retained austenite is minimalized.

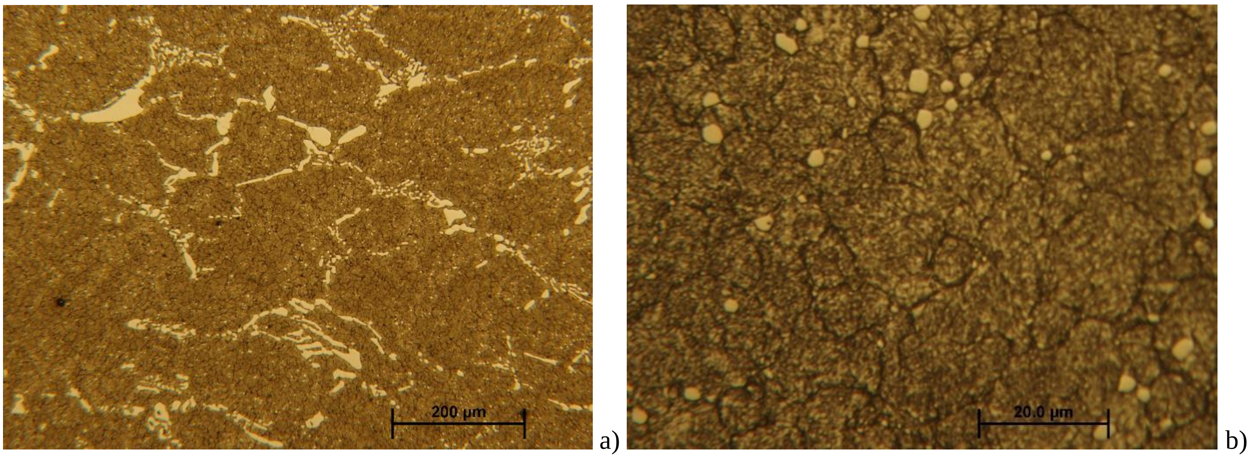


Figure 8. Cryogenically treated and triple tempered sample micrographs. a) N $_{original}$=100x b) N $_{original}$=1000x

The microstructure changings after different heat treatment technologies are reflected in the hardness values. Austenitizing temperatures, cryogenic temperatures tempering temperatures and the hardness evolutions are given in Tab.2.

Studying the microhardness of etched zones were observed that cryogenically threating increase the hardness due to reduction of retained austenite content. In case of conventional quenching increasing the tempering temperature to 510°C the hardness has been increased too. The reason is that the secondary carbides precipitations begins.

After the three times tempering the hardness continues to increase due to the secondary carbides precipitations.

Nr	Austenitizing temperature	Cryogenic treatment	Tempering 1		Tempering 2		Tempering 3	
		T(°C)	T(°C)	HV1	T(°C)	HV1	T(°C)	HV1
1	1070 (°C)	-	200	641	-	-	-	-
2	1070 (°C)	-	510	663	-	-	-	-
3	1070 (°C)	-	510	-	480	-	480	695
4	1070 (°C)	-80	510	746	-	-	-	-
5	1070 (°C)	-80	510	-	480	-	480	738

Table 2. Heat treatment process and hardness evolution

The cryogenic treatment consequence is the higher hardness due to retained austenite reduction.

The best properties were obtained after tempering three times at high temperature.

Conclusion

In cryogenically treated sampled retained austenite quantity is decreased comparing with conventional quenched samples. Cryogenic treatment accelerate the decomposition of martensite and modify the precipitation behaviour of secondary carbides. The reason for increasing hardness due to the cryogenic treatment is related to the conversion of the retained austenite to martensite and the sediment of the tiny nanometer-sized carbides, and more appropriate distribution of carbides so the transformation of retained austenite to martensite and the secondary carbide precipitation are the main mechanism responsible for the properties improvement of this steel quality.

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References

[1] L. Kirkhorn – V. Bushlya – M. Andersson – J.E. Stahl (2013) *The influence of tool steel microstructure on friction in sheet metal forming*. Wear. 302 pp. 1268–1278.

[2] https://www.bohler-edelstahl.com/en/

[3] V. Leskovsek – B. Podgornik (2012) *Vacuum heat treatment, deep cryogenic treatment and simultaneous pulse plasma nitriding and tempering of P/MS390MC steel.* Mater. Sci. Eng. A 531, pp. 119–129.

[4] J Hollomon – L. Jaffe (1945) *Time- temperature relations in tempering steel.* Trans AIME. 162 pp. 223- 249.

[5] Hougardy, H.P. (1992) *Description and Control of Transformations in Technical Applications*. Steel: A Handbook for Materials Research and Engineering – Volume 1: Fundamentals, Springer-Verlag, Berlin. pp. 167-200.

[6] G.Roberts – G.Krauss – R.Kenedy (1998) *Tool Steels* , 5th ed. ASM International, Metals Park, OH, USA.

[7] K.E. Thelning (1984) *Steel and Its Heat Treatment*. 2nd ed. Butterworts, London.

[8] R.E. Reed-Hill – R. Abbaschian (1992) *Physical Metallurgy Principles*. 3rd ed., PWS Publishing Company , Boston.

[9] R.G. Bowes (1974) *Heat Treatment Metallurgy.* 1, pp. 29-32.

[10] D. Das – A.K. Dutta – V. Toppo – K.K. Ray (2007) *Effect of deep cryogenic treatment on the carbide precipitation and tribological behavior of D2 steel*. Materials Manufacturing. Process. 22., pp. 474-480.

[11] A. Molinari – M. Pellizzari – S. Gialanella – G. Straffelini – K.H. Stiasny (2001) *Effect ofdeep cryogenic treatment on the mechanical properties of tool steels*. J. Mater.Process. Technol. 118 pp.350–355.

[12] M.A. Hamidzadeh – M. Meratien – A. Saatchi (2013) *Effect of cerium and lanthanium on the microstructure and mechanical properties of AISI D2 tool steel*. Mater. Sci. Eng. Struct. Mater. Prop. Microstruct. Process. 571 pp. 193-198.

[13] D. Das – A.D. Dutta – K.K. Ray (2009) *Optimization of the duration of cryogenic processing to maximize wear resistance of AISI D2 steel*. Cryogenics 49 pp. 176-184.

[14] D. Das – R. Sarkar – A.K. Dutta – K.K. Ray (2010) *Influence of sub-zero treatments on fracture toughness of AISI D2 steel.* Mater. Sci. Eng. A 528 pp. 589-603.

[15] D. Das – A.K. Dutta – K.K. Ray (2010) *Sub-zero treatments of AISI D2 steel*: *part II*. Wear behavior, Materials Science and Engineering A, 527 2194-2206.

[16] L. Toth (2018) *Examination of the properties and structure of tool steel EN 1.2379 due to different heat treatments.* European Journal of Materials Science and Engineering. 3 (3) pp. 165-170.

[17] A.I. Tyshchenko – W. Theisen – A. Oppenkowski (2010) *Evaluation of factors influencing deep cryogenic treatment that affect the properties of tool steels*. J. Mater. Process. Technol. 210, pp. 1949–1955.

[18] V. G Gavriljuk – W. Theisen – V.V. Sirosh (2013) *Low-temperature martensitic transformation in tool steels in relation to their deep cryogenic treatment*. Acta Mater. 61 pp. 1705–1715.