

Acta Materialia Transylvanica 2/2. (2019) 87–92. https://doi.org/10.33924/amt-2019-02-04 Hungarian: https://doi.org/10.33923/amt-2019-02-04 https://eda.eme.ro/handle/10598/31509



Examination of Heat Treatment on the Microstructure and Wear of Tool Steels

Enikő Réka FÁBIÁN,¹ László TÓTH,² Csenge HUSZÁK³

Óbuda University, Donát Bánki Faculty of Mechanical and Security Engineering, Budapest, Hungary

- ¹ fabian.reka@bgk.uni-obuda.hu
- ² toth.laszlo@bgk.uni-obuda.hu;
- ³ huszak.csenge@bgk.uni-obuda.hu

Abstract

The microstructure of the investigated X153CrMoV12 grade tool steel in delivered condition consisted of spheroidal matrix and primary carbides. The primary carbides were not dissolved under austenitisation time on either 1030°C or 1070°C. The microstructure and abrasion resistance of the steel changed due to quenching from different austenitisation temperatures. After conventional quenching from the higher austenitising temperature, there is more residual austenite in the steel than at quenching from the lower austenitisation temperature, which decreased the wear resistance. As a result of quenching from 1070°C followed by a multiple tempering process around 500 to 540°C, the retained austenite content is reduced and finely dispersed carbides are precipitated in the matrix, resulting in a higher matrix hardness and an increased wear resistance. After cryogenic treatment, the residual austenite content decreases compared to the conventional process, which leads to an increase in hardness and wear resistance.

Keywords: tool steel, austenitisation, retained austenite, precipitation, cryogenic treatment, tempering, wear resistance.

1. Introduction

The X153CrMoV12 grade steel is commonly used by tool makers as a raw material for blanking and punching tools, woodworking tools, shear blades for cutting light-gauge material, thread rolling tools, tools for drawing, deep drawing and cold extrusion, pressing tools for the ceramics and pharmaceutical industries, cold rolls (working rolls) for multiple-roll stands, measuring instruments and gauges and small moulds for the plastics industry where excellent wear resistance is required. This steel in delivered condition is annealed, the hardness is max. 250 HB. In this state the steel is relatively easy to be manufactured despite its high alloy content. The properties of the tool are achieved in the final heat treatment. There are differences in the recommendations for heat treatment, despite the fact that the X153CrMoV12 grade steel is widely used. Small dimension changes and high hardness is usually achieved after quenching from 930–960 °C [1]. Using high quenching temperature (1100 °C), secondary hardening appears in the material the maximum hardness result after tempering at 520–530 °C. The standard [2] recommendation for austenitisation is 1020 °C, and cooling in air. The standard for maximum hardness (over 62 HRC) recommends 970 °C for austenitisation, air quenching followed by tempering under 200 °C

Voestalpine is one of the world's leading tool steels, high-speed steels and special steels manufacturers. The company publication does not provide data on secondary hardening after the hardening process from 1030°C, but secondary hardening is expected at 520°C after hardening from 1070°C, so double tempering at this temperature is recommended before any nitration [3]. Interalloy recommends triple tempering at 500–550°C [4]. Cryogenic treatment is not mentioned by companies, nor by the standard, although when cooled to room temperature with this carbon con-

tent, the martensitic transformation is not completed [5]. While company data do not suggest a relationship between wear resistance and heat treatment parameters, the effects of deep cooling and tempering on mechanical and wear properties have been addressed by several researchers for similar cold forming tool steels [6–11]. The tool makers ask from the heat treatment shops only the Rockwell hardness, however, tool life, wear resistance, and toughness depend also on the heat treatment technology.

2. Test materials and methods

The chemical composition of the examined X153CrMoV12 grade steel is shown in Table 1.

Table 1. Chemical composition of the test material

С	Cr	Mo	v	Si	Mn	Fe
1.67	11.25	0.837	1.41	0.364	0.422	rest

Austenitization of the $20\times40\times20$ mm samples was carried out at 1030°C and 1070°C , respectively, in an Ipsen VFC type vacuum furnace with $300\times370\times200$ mm chamber size after a two-holding steps at $650\,^{\circ}\text{C}$ and $900\,^{\circ}\text{C}$. Some of the samples were quenched down to $40\,^{\circ}\text{C}$ conventionally, using nitrogen gas, and some samples were cryogenic treated in nitrogen to $-80\,^{\circ}\text{C}$ for 3 hours. The effects of simple and multiple tempering on the microstructure and wear resistance were studied for the conventional and cryogenic quenching.

The experimental samples were examined after metallographic preparation using an Olympus PMG3 light microscope (LOM) and a Jeol JSM 5310 scanning electron microscope (SEM). Primary examination was of the carbides size and distribution in the microstructure using nital. For the determination of retained austenite the Beraha'2 reagent (85 ml water, 15 ml HCl, 1 g K₂S₂O₅) proved to be the most suitable. Beraha'2 reagent colors the ferrite and martensite but not austenite and carbides. The primary carbides are clearly distinguishable from the matrix. The carbide precipitations in the matrix are spherical, and so could be distinguished from the residual austenite. The hardness of the carbides and of the matrix after different heat treatments was measured with Vickers hardness testing equipment type Buhler 1105.

For the wear resistance testing, a self-developed abrasive equipment was used [12] (Figure 1.). The used abrasive tool was a 20 mm diameter Al_2O_3 ceramic ball with polished surface. The

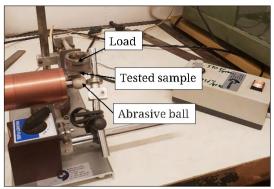


Figure 1. Abrasive equipment

loading force was provided by the sample which supported a load with 50 g.

The wear factor (K) was used as a measure of wear resistance, calculated from the volume mass loss (V_v), the sliding distance (s) and the normal load (F):

$$K = \frac{V_v}{s \cdot F} \ (mm^3/(m \cdot N)) \tag{1}$$

The volume mass lost is calculated from the diameter of the calotte formed during the wear (*d*) and the depth of the crater (*h*):

$$V_v = \frac{h \cdot \pi}{6} \left(\frac{3}{4} d^2 + h^2 \right), \quad (mm^3)$$
 (2)

The height of the abrasive calotte is calculated by a simple relationship between the radius *R* of the abrasive ball and the diameter of the calotte formed during the wear test:

$$h = R - \sqrt{R^2 - \left(\frac{d}{2}\right)^2} , mm \tag{3}$$

The sliding distance (*s*) depends on the time of the wear (*t*), on the radius of the abrasive ball (*R*) and its rotational speed (*n*) (4):

$$S = n \cdot 2 \cdot \pi \cdot R \cdot t \cdot m \tag{4}$$

For comparison with literature data [11–12], the speed was 570 rpm and the abrasion test time was 5 minutes.

3. Test results

The hardness of the tested X153CrMoV12 grade tool steel in delivered condition was 248 HV 1 on average and its microstructure consisted of a spheroidal matrix and primary carbides. The austenitisation temperature recommended by

Voestalpine Hungary Kft. strongly influences the microstructure and hardness of the treated material.

3.1. Effect of the austenitisation temperature

Examining the microstructure of the samples, it was found that the primary carbides did not dissolve during heating at 1070°C, as can be seen in the photos of Figures 2.a) and b). As shown in the photos of Figures 2.c) and d), quenching from a higher austenitization temperature resulted in more residual austenite than in the case of quenching from 1030°C. Hardness and wear resistance test results are fully consistent with the microstructure. When the sample was guenched from 1030°C, the average hardness of the matrix was 674 HV 1, while for the sample guenched from 1070°C it was 648 HV1. By increasing the austenitising temperature, the wear coefficient increased from $2.46\ 10^{-5}\ mm^3/Nm$ to $2.94\ 10^{-5}\ mm^3/Nm$ due to the higher retained austenite content. The distribution of retained austenite is always uniform in the matrix. The primary carbides appearance was similar after quenching from 1030°C and 1070°C. This microstructural similarity explains the phenomenon that the Rockwell hardnesses measured after quenching are the same (Table 2.).

Table 2. The effect of heat treatment parameters on hardness and wear resistance

Hardening		Tempering (°C)			HDC	1117.4	K
T _A (°C)	HRC	T ₁	T ₂	T ₃	HRC	HV 1	(mm ³ /Nm)
1030	62	_	_	_	62	674	2.46·10-5
1030	62	200	_	_	61	668	1.56·10-5
1070	62	_	_	_	62	648	2.94·10 ⁻⁵
1070	62	200	_	_	61	641	1.04·10-5
1070	61	520	_	_	60	663	2.22·10-5
1070	61	520	540	_	59	685	3.00·10 ⁻⁵
1070	61	520	540	500	59	748	2.46·10-5

3.2. Effect of tempering

The temperature and number of cycles of tempering following the quenching after austenitization at 1070 °C significantly influence the microstructure, hardness and wear properties of the X152CrMoV12 steel. The amount of fine dispersed carbides in the martensite is very small (photos

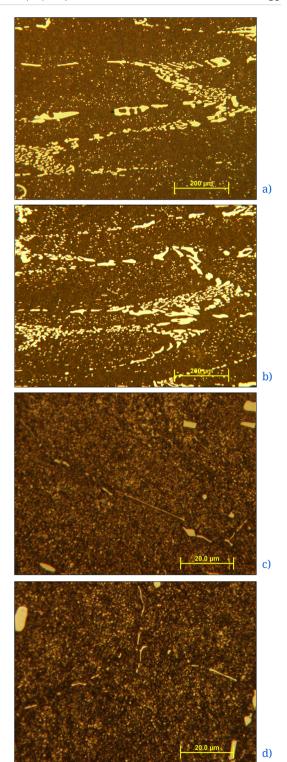


Figure 2. The effect of the hardening temperature on the microstructure; LOM image. Etching agent: Beraha-2. a) c) T_{aust} = 1030 °C b) d) T_{aust} = 1070 °C

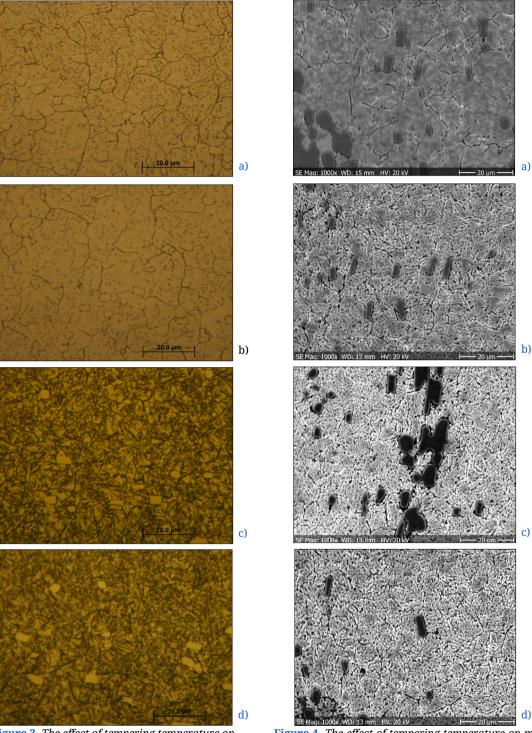


Figure 3. The effect of tempering temperature on microstructure; LOM images. T_{Aust} = 1070 °C. Etching agent: 2% Nital. a) quenched, b) T_{temper} = 200 °C, c) T_{temper} = 520 °C, d) T_{temper} = 520 °C + 540 °C + 500 °C

Figure 4. The effect of tempering temperature on microstructure. SEM images. Etching agent: 2% Nital. a) T_{temper} = 200 °C, b) T_{temper} = 520 °C, c) T_{temper} = 520 °C + 540 °C, d) T_{temper} = 520 °C + 540 °C + 500 °C

in Figure 3.b), Figure 4.a)) when the sample is tempered at 200 °C after quenching. After tempering at high temperature (520 °C) the result is fine dispersed carbides precipitations, but retained austenite can be detected as shown in the photos of Figure 3.c) and Figure 4.b) Multiple high-temperature tempering processes result in a higher quantity of fine, dispersed carbides in the microstructure while the residual austenite content decreases, as shown in the photos of Figures 3.c), d) and 4.c), d) The characteristic of the primary carbides does not change significantly.

Changes in microstructure due to tempering at 500–550°C increase the hardness of the matrix. Due to multiple tempering around 500–550°C, the hardness of the matrix increased to 750 HV 1 (Table 2.).

3.3. The effect of cryogenic treatment

High-temperature tempering is required for tools that would be nitrided [1, 3]. Nitrided products typically require dimensional accuracy, so a large amount of residual austenite is not allowed. In our investigations it was found that the residual austenite content of the cryogenic treated workpiece is low even after a single high-temperature tempering process compared to the conventional treated sample (Figure 5.a), 2.c)). The triple high-temperature tempering process decreases the residual austenite content further (Figure 5.).

Due to cryogenic treatment the hardness of the matrix increase, and its positive effect on the wear factor is clearly demonstrated (Table 3.).

4. Conclusion

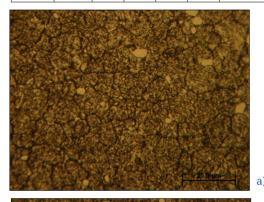
Increasing the austenitisation temperature reduces the hardness of the martensitic matrix after quenching due to the increasing amount of residual austenite. Multiple high-temperature tempering increases the hardness of the sample quenched from 1070 °C, partly due to the reduction of residual austenite content and partly due to the finely dispersed precipitates. Cryogenic treatment causes a good reduction in the amount of residual austenite and an increase in wear resistance.

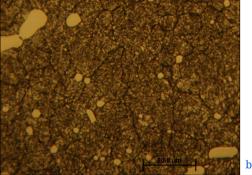
Acknowledgments

The authors would like to thank the Hungarian state and the European Union for the financial support of our work in the framework of the project EFOP-3.6.1-16-2016-00010.

Table 3. The effect of deep cooling on matrix hardness and wear factor

Austeni- tisation	Cool- ing (°C)	Tempering (°C)				Wear fac- tor (mm³/N·m)	
(°C)	T	T ₁	T ₂	T ₃	HV1 mátrix	K·	
1070	20	510	_	ı	663	4.29·10 ⁻⁵	
1070	-80	510	_	-	746	2.66·10-5	
1070	20	510	480	480	695	3.00·10 ⁻⁵	
1070	-80	510	480	480	738	2.24·10-5	





5. **ábra**. The effect of deep cooling on texture LOM images Etching: 2% nital, $T_{quench} = -80 \, ^{\circ}\text{C}$ a) $T_{temper} = 510 \, ^{\circ}\text{C}$, b) $T_{temper} = 510 \, ^{\circ}\text{C} + 480 \, ^{\circ}\text{C} + 480 \, ^{\circ}\text{C}$

References

- [1] Smóling K., Czeglédi L.: Szerszámacélok kézikönyve. Szabvány Kiadó, Budapest, 1977. 94–95.
- [2] MSZ EN ISO 4957: 2018. Szerszámacélok
- [3] Voestalpine Böhler Edelstahl GmbH & Co KG: Szerszámacélok, nemesacélok, 45. http://www.boehler.hu/media/productdb/down-loads/K110DE.pdf (accessed on: 2019. 01.28)
- [4] Interalloy Engineering Steels and Alloys: Product datasheets, Tool steel D22011 Inter-alloy Pty Ltd (accessed on: 2019. 02.15)
 http://www.interlloy.com.au/our-products/tool-steel/d2-tool-steel-x153crmoy12

- [5] Capdevila C. et al.: Determination of Ms temperature in steels: A Bayesian Neutral Network Model. ISIJ International, 42. (2002) 894–902. https://doi.org/10.2355/isijinternational.42.894
- [6] Gavriljuk V. G., Theisen W., Sirosh V. V.: Low-temperature martensitic transformation in tool steels in relation to their deep cryogenic treatment. Acta Materiala, 61. (2013), 1705–1715. https://doi.org/10.1016/j.actamat.2012.11.045
- [7] Das D., Dutta A. K., Toppo V., Ray K. K.: Effect of deep cryogenic treatment on the carbide precipitation and tribological behaviour of D2 steel. Materials Manufacturing. Process, 22. (2007) 474–480. https://doi.org/10.1080/10426910701235934
- [8] Molinari A., Pellizzari M., Gialanella S., Straffelini G., Stiasny K. H.: Effect of deep cryogenic treatment on the mechanical properties of tool steels. Journal of Materials Processing Technology, 118. (2001) 350–355.
 - https://doi.org/10.1016/s0924-0136(01)00973-6

- [9] Das D., Sarkar R., Dutta A. K., Ray K. K.: Influence of sub-zero treatments on fracture toughness of AISI D2 steel. Materials Science Engineering A, 528. (2010) 589–603. https://doi.org/10.1016/j.msea.2010.09.057
- [10] Das D., Dutta A. K, Ray K. K.: Sub-zero treatments of AISI D2 steel: part II. Wear behaviour. Materials Science and Engineering A, 527. (2010) 2194–2206.
 - https://doi.org/10.1016/j.msea.2009.10.071
- [11] Kumar S., Nagaraj M., Khedkar N. K., Bongale A.: Influence of deep cryogenic treatment on dry sliding wear behaviour of AISI D3 die steel. Materials Research Express, 5/11. (2018) 116525, 1–9. https://doi.org/10.1088/2053-1591/aadeba
- [12] Kovács T., Dévényi L.: Kopásvizsgálati eljárás fejlesztése. Anyagok világa, 5/1. (2004). https://www.kfki.hu/~anyag/tartalom/2004/dec/05 KT DL.pdf