

HEAT TREATMENT OF A P/M TOOL STEEL CONTAINING AN INCREASED NIOBIUM CONTENT

TOPLOTNA OBDELAVA P/M ORODNEGA JEKLA Z VISOKO VSEBNOSTJO NIOBIJA

Jan Šerák, Dalibor Vojtěch, Marketa Pavlíčková, Pavel Novák

Institute of Chemical Technology in Prague, Department of Metals, Technická 5, 166 28 Prague 6, Czech Republic
jan.serak@vseht.cz

Prejem rokopisa – received: 2003-10-01; sprejem za objavo – accepted for publication: 2003-11-18

The microstructure of a P/M tool steel with 2.5% C, 3.3% Si, 6.2%Cr, 2.2%Mo, 2.6%V, 2.6%Nb and 1.0%W was investigated for various heat-treatment conditions, with the aim of achieving an alloy with the maximum hardness. The heat treatment of the compacted semi-product consisted of soft annealing, austenitizing, hardening and tempering.

Key words: P/M tool steel, heat treatment, niobium

Opisani sta mikrostruktura P/M-jekla z 2,5 % C, 3,3 % Si, 6,2 % Cr, 2,2 % Mo, 2,6 % V, 2,6 % Nb in 1 % W in toplotna obdelava s ciljem doseganja največje trdote. Toplotna obdelava polproizvoda je obsegala mehko žarjenje, avstenitizacijo, kaljenje in popuščanje.

Ključne besede: P/M-jeklo, toplotna obdelava, niobij

1 INTRODUCTION

Hardness, adequate toughness and wear resistance are the properties generally required for tool steels. The increased demand for high-quality tool steels at the present time has resulted in the need to produce steels with larger amounts of carbon, as well as other alloying elements. However, increasing the level of the alloying elements tends to promote segregation effects and the coarsening of carbides, and hence a considerable reduction in the toughness results. One way to suppress segregation and coarsening is to use a powder-metallurgy (P/M) processes^{1,2}.

The demand for low-cost high-performance tool steels has resulted in a number of attempts to replace some of the expensive alloying elements (W, Mo) with niobium^{3,4}. However, niobium is a strong carbide former and modifies the solidification sequence of the steel. In the first stage of solidification, niobium-rich MC carbides are formed^{5,6}. In addition, increasing the niobium content in a steel up to a mass fraction of approximately 1.8 % causes an increase in the hardness, the wear resistance and the toughness compared to the material without niobium⁷. The affinity of niobium for carbon is greater than that of vanadium. As result, the presence of niobium increases the content of vanadium in the solid solution after austenitizing, and consequently a higher secondary hardness is obtained. In steels containing a mass fraction of more than approximately 2 % Nb, coarse NbC+ γ quasi-eutectic regions are formed⁸. These regions are very stable and do not change during high-temperature heat treatment. The presence of these regions in the microstructure is deleterious because they

affect the brittleness of the steel, but by using a powder-metallurgy process they can be prevented. In order to achieve an alloy with maximum hardness it is necessary to find the optimum heat treatment for the steel.

2 EXPERIMENTAL PROCEDURE

The investigated tool steel was prepared using a powder-metallurgy process. The powder was produced by atomizing the melt using a nitrogen flow at a pressure of 400–600 kPa. The chemical composition of the steel used in these experiments is shown in **Table 1**; it differs from that of commercial steels in terms of its niobium content.

Table 1: Chemical composition of the P/M steel

Tabela 1: Kemična sestava P/M-jekla

C	Si	Cr	Ni	Mo	V	Nb	W	N	O	P	S
2.50	3.30	6.20	1.42	2.20	2.60	2.60	0.98	0.03	0.04	0.03	0.02

Chromium, vanadium, molybdenum and niobium are the main carbide-forming elements present in steel. The contents of oxygen and nitrogen, which are of the order of hundreds of ppm, are in accordance with the method used for the powder preparation.

The powder was compacted using a hot isostatic pressing (HIP) method. The steel capsule (length 200 mm and diameter 40 mm) containing the powder was evacuated. The HIP processing was performed at 1120 °C under an argon pressure of 147 MPa for 4 h. The residual porosity of the hot isostatically pressed product was negligible.

The heat treatment of the compacted semi-product consisted of soft annealing, austenitizing, hardening and tempering. The soft annealing was performed at 780 °C for 8 h with subsequent slow cooling to 680 °C, and then holding at this temperature for 2 h. Next, the steel was slowly furnace cooled to room temperature. The conditions of soft annealing were selected with the aim of obtaining a desirable growth of carbide particles. After this treatment, the hot isostatically pressed semi-product was cut into samples of size approximately 5 mm × 10 mm × 10 mm. To characterize the influence of the heat treatment on its properties, the steel was austenitized for 30 min at temperatures ranging from 1000 °C to 1150 °C, after which it was cooled in nitrogen with a pressure of 500 kPa to 600 kPa in a vacuum furnace. As a final step, the hardened steel was tempered three times at various temperatures in the range 150–700 °C with intermediate air-cooling.

The hardened and tempered samples were then investigated with optical, scanning and transmission electron microscopy. EDAX microanalysis and Rockwell hardness testing were also used.

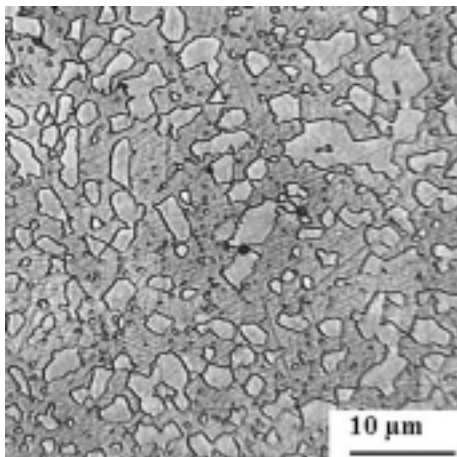


Figure 1: Microstructure of the steel after soft annealing
Slika 1: Mikrostruktura jekla po mehkiem žarjenju

3 RESULTS AND DISCUSSION

The microstructure of the investigated alloy is shown in **Figure 1**. It consists of carbide particles that are and uniformly distributed in the matrix and surrounded with the matrix. The carbide particles are of two different sizes

EDAX microanalysis was used for to determine theation of chemical composition of the carbides. The X-ray maps in **Figure 2** show that chromium is the main constituent of the coarse carbides. The size of these particles ranges from approximately 5 μm to 10 μm. Vanadium, niobium and molybdenum are present mainly in the smaller carbide particles of with diameters less than 3 μm. Vanadium and niobium preferably tend to form MC carbides. The number of smaller V-, Nb- and Mo-rich particles is much higher than that of the coarse Cr-rich particles.

3.1 Hardening

The microstructure of the hardened samples consists of particles uniformly distributed in the matrix of the solid solution, as can be seen in **Figure 3**.

The microstructure of the hardened steel is similar to that of the soft annealed steel with two types of particles. The coarse particles of irregular shape, see **Figure 4**, were identified using TEM as Cr₇C₃, and the smaller particles (**Figure 5**) as NbC.

The volume fraction of the primary carbide particles in the steel depends on the austenitizing temperature, and the volume fraction of the carbides decreases with the increasing temperature of austenitizing, due to their dissolution in the austenite. The finest carbides were present in the steel hardened at 1050 °C.

3.2 Tempering

The final properties of many highly alloyed tool steels are adjusted by tempering at relatively high temperatures (500–600 °C), where the peak of the secondary hardness occurs. To find the optimum

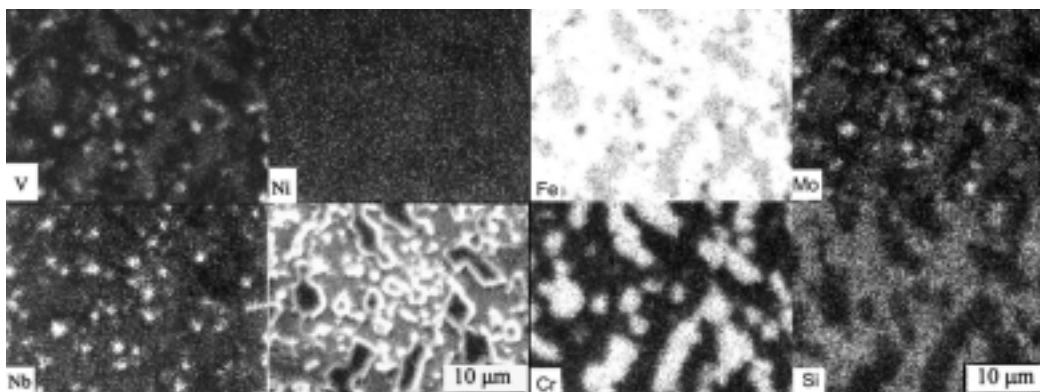


Figure 2: Distribution of alloying elements in the investigated steel (X-ray maps)
Slika 2: Porazdelitev legirnih elementov v jeklu (specifični rentgenski posnetki)

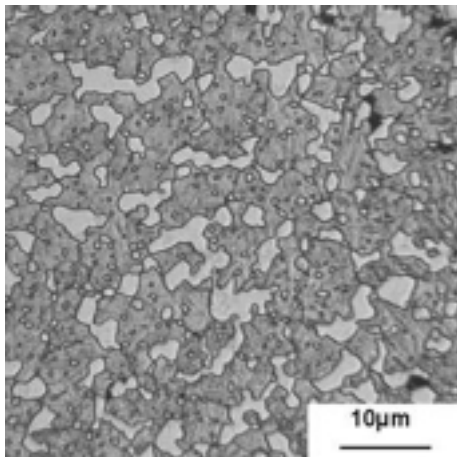


Figure 3: Microstructure of the hardened steel (austenitizing temperature of 1050 °C)

Slika 3: Mikrostruktura kaljenega jekla (temperatura avstenitizacije 1050 °C)

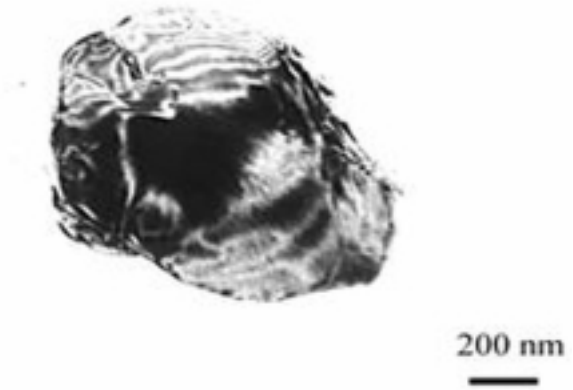


Figure 5: Primary NbC carbide particle in the hardened steel (TEM-dark field)

Slika 5: Primarno karbidno zrno v kaljenem jeklu (TEM temno polje)

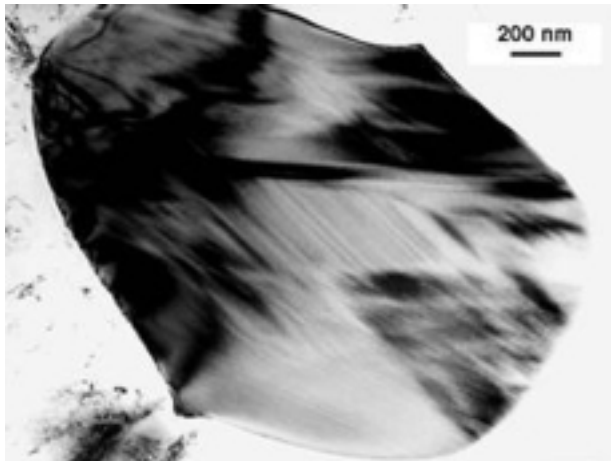


Figure 4: Primary Cr_7C_3 carbide particle in the hardened steel (TEM-bright field)

Slika 4: Primarno zrno karbida Cr_7C_3 v kaljenem jeklu (TEM, svetlo polje)

tempering conditions for our material, the hardened samples were tempered (tempering time: 3×1 h) at temperatures from 150 °C to 700 °C and detailed investigations of the microstructure, the phase composition and the mechanical properties after tempering were performed.

The tempering curves of the samples austenitized at various temperatures are shown in **Figure 6**.

As expected, the austenitization temperature of 1000 °C was insufficient to obtain a high final hardness. The sample austenitized at this temperature reached a maximum hardness that was slightly below 60 HRC after tempering at 500 °C. The highest hardness of 65 HRC was measured on the samples austenitized at 1050 °C and 1100 °C. The hardness of the sample austenitized at 1050 °C is relatively high and increases slowly with increasing tempering temperature until it reaches a

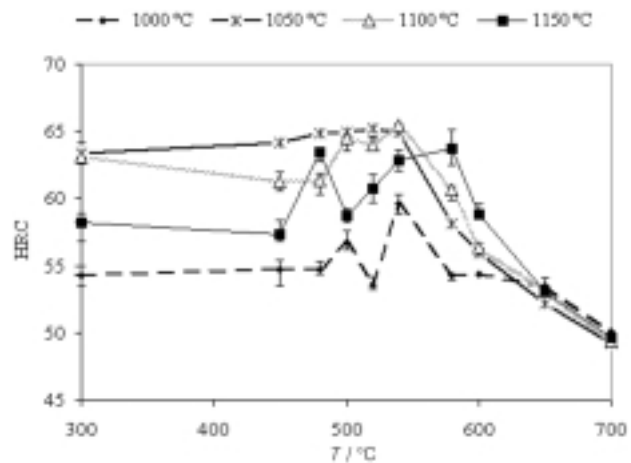


Figure 6: Influence of the heat treatment on the hardness of the steel
Slika 6: Vpliv toplotne obdelave na trdoto jekla

maximum of 65 HRC at 540 °C. Higher tempering temperatures lead to a steep reduction in the hardness due to the loss of coherency and the coarsening of the precipitated carbide particles. During the austenitization at 1100 °C, the austenite becomes more saturated with carbon and the alloying elements, and therefore a pronounced secondary hardness peak between 480 °C and 550 °C is observed. The maximum of 65 HRC was achieved after tempering at 540 °C, whereas for samples austenitized at 1150 °C, a maximum hardness of only 64 HRC after tempering at 580 °C was obtained.

3.3 Optimal heat treatment

In accordance with the results in this investigation, the optimum conditions for the heat treatment of the P/M tool steel in order to obtain the maximum hardness are:

1. Austenitization at temperatures from 1050 °C to 1100 °C for 30 min (nitrogen, vacuum furnace).

2. Quenching in a high-pressure nitrogen flow (500–600 kPa).
3. Tempering at a temperature of 540 °C for 3 × 1 h (air).

ACKNOWLEDGMENT

This work was supported by the research projects EUREKA 2728 UPLETOOLS and MSM 223100002.

4 REFERENCES

¹H. Takigawa, H. Manto, N. Kawai, K. Homma, Powder Metallurgy 24 (1981) 4, 196

²P. Matteazzi, F. Wolf, Mat. Sci. Eng. A 248 (1998) 19

³R. Riedl, S. Karagöz, H. Fischmeister, F. Jeglitsch, Steel Research 58 (1987) 8, 339

⁴L. A. Dobrzański, A. Zarychta, M. Ligarski, Journal of Mat. Proc. Technol. 63 (1997) 531

⁵S. Kheirandish, Y. H. K. Kharrazi, S. Mirdamadi, ISIJ International 37 (1997) 7, 721

⁶S. Kheirandish, S. Mirdamadi, Y. H. K. Kharrazi, Steel Research 68 (1997) 10, 457

⁷M. Fiset, K. Peev, M. Radulovic, J. Mat. Sci. Let. 12 (1993) 615

⁸S. Kheirandish, S. Mirdamadi, Y. H. K. Kharrazi, Metall 53 (1999) 6, 339