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STUDY OF PHASE TRANSFORMATIONS IN Cr-V TOOL STEEL

ŠTUDIJ FAZNIH PREMEN V ORODNEM JEKLU Cr-V

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The properties of steels are very dependent on the phases present in the microstructure. The wear resistance and thermal stability of tool steels are achieved with the presence of different types of carbides. So the chemical composition and heat treatment play crucial roles in optimizing the properties of tool steels.

The phase transformations in Cr-V tool steel were analyzed during the heating from room temperature up to 1100 °C using DTA and dilatometry. After soft annealing the microstructure of the investigated steel consists of a ferritic matrix and M_7C_3 and MC carbides, as determined with SEM, EDX and XRD techniques. The experimental results were compared to the computational results (Thermo-Calc).

Keywords: phase transformation, Cr-V tool steel, Thermo-Calc

Lastnosti jekla so močno odvisne od faz, ki so v mikrostrukturi. Odpornost proti obrabi in toplotna stabilnost orodnih jekel se dosežeta z različnimi vrstami karbidov. Kemijska sestava in toplotna obdelava imata ključno vlogo pri optimiranju lastnosti orodnih jekel.

DTA in dilatometrija sta bili uporabljeni za preučevanje faznih premen v orodnem jeklu Cr-V med ogrevanjem od sobne temperature do 1100 °C. Tehnike SEM, EDX in XRD so potrdile, da po mehkem žarjenju mikrostrukturo preiskovanega jekla sestavlja feritna osnova ter karbidi M_7C_3 in MC. Eksperimentalni rezultati so bili primerjani z izračunanimi (Thermo-Calc).

Ključne besede: fazna premena, orodno jeklo Cr-V, Thermo-Calc

1 INTRODUCTION

At present Cr-V ledeburitic steels are often used for the manufacturing of cutting, forming and other tools in the industry. To meet the industrial requirements for high stability and reliability, they have to withstand the wear and plastic deformation. They are usually produced with powder metallurgy. This technology enables us to obtain a uniform carbide size and distribution and also to produce the materials with particular compositions that cannot be prepared by conventional casting. The technology of powder metallurgy provides good results in achieving high homogeneity. The phase composition, microstructure and mechanical properties of ledeburitic tool steels are determined by the matrix and the type, quantity, size and distribution of the carbides.[1] The properties of the material thus depend on the microstructure obtained after heat treatment.

The phase transformations during heating can be described with thermodynamic modeling using the CALPHAD method.[2–4]

Thermal-analysis techniques are suitable for an experimental determination of the phase transformations in materials. For steels, the DTA and dilatometry techniques are often used.[5,6]

Carbide phases have a different thermal stability and some of them are dissolved during the austenitizing in the solid state.[7,8] Bílek et al.[9] investigated the phase composition of Cr-V tool steel after heat treatment, using SEM+EDX, a quantitative analysis of the microstructure and hardness measurements. They found two types of carbides in the microstructure of Cr-V tool steel. Carbide M_7C_3 was partially dissolved during the austenitizing at 1000 °C and it completely disappeared after the austenitizing at 1100 °C. Carbide MC is more stable and starts to dissolve only at a temperature higher than 1200 °C.

The aim of this paper is to enhance and explain the results published previously by Bílek et al.[9] using additional experimental techniques (XRD, DTA and dilatometry) and thermodynamic calculations (Thermo-Calc).

2 EXPERIMENTAL WORK

2.1 Sample material

Cr-V tool steel was used as the experimental material with the chemical composition of w(C) = 2.1 %, w(Si) = 1 %, w(Mn) = 0.4 %, w(Cr) = 6.8 %, w(Mo) = 1.5 %, w(V) = 5.4 %, balanced by Fe.[10]

The sample was initially annealed at the temperature of 900 °C for 1 h and then slowly cooled down to room temperature.

The X-ray diffraction (XRD) analysis was accomplished with a Panalytical Empyrean X-ray diffractometer. A cobalt anode (U = 40 kV and I = 40 mA) with a parallel-beam X-ray mirror was used. The sample was measured with a PIXcel3D detector at room temperature in the angular range of 45–110° with a step size of 0.0131°. The microstructure and chemical composition of the phases were analyzed with a JEOL JSM 7600F electron microscope equipped with a secondary and back-scattered electron detector and a MAX 50 EDX detector from Oxford Instruments. The differential thermal analysis (DTA) was done using a NETZSCH STA 409CD simultaneous thermal analyzer in an inert gas (Ar 6.0) with a magnetic frame enabling also the measurements of the magnetic transition in the temperature range of 100–1100 °C, at the heating rate of 10 K/min. The dilatometry measurements were performed using a NETZSCH DIL 402C dilatometer. The inert-gas atmosphere (Ar 6.0) in the temperature range of 100–1000 °C and different heating rates (2, 5, 8, 12, 16, 20, 25) K/min were used. The thermodynamic calculations were done using the Thermo-Calc software and TCFE6 thermodynamic database.

2.2 Results

The XRD analysis (**Figure 1**) confirms that only MC and M_7C_3 carbides are present as the secondary phases in the ferrite at room temperature.

Figure 2a illustrates the microstructure of Cr-V tool steel after annealing, with the coarse M_7C_3 particles (some of these particles are in an extraordinary fine form) and finer MC particles in the ferritic matrix.

In the EDX mapping mode the distribution of vanadium (**Figure 2b**) and chromium (**Figure 2c**) can be seen.

The phase transformations in the investigated steel during heating are shown with the DTA and thermomagnetometry curves in **Figure 3**. The magnetic transition of the ferrite from the ferromagnetic to paramagnetic state is detected at 754.0 °C from the thermomagnetometry curve and at 753.6 °C from the DTA curve. The next peak on the DTA curve with the onset at 855.3 °C corresponds to the austenitization process.

These results were verified with thermodynamic calculations using the Thermo-Calc software. **Figure 4** shows the temperature dependence of the volume fractions of individual phases. It can be seen that the amount of the M_7C_3 carbide starts to decrease already during the ferrite-to-austenite transformation, in the temperature range of 814–837 °C.

After the completion of the ferrite-to-austenite transformation, the dissolution of M_7C_3 in austenite continues until its completion at about 1200 °C.

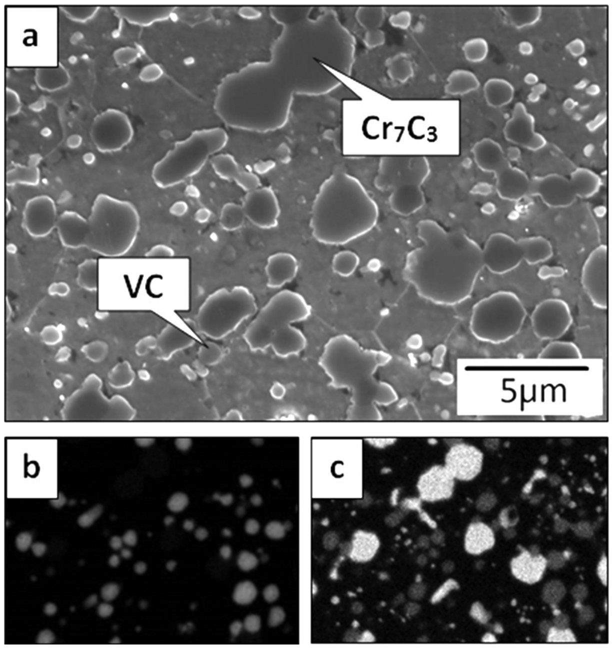

Figure 2: Microstructure of the ledeburitic Cr-V tool steel in the soft-annealing state: a) overview (SEM), b) EDX map of vanadium from **Figure 2a**, c) EDX map of chromium from **Figure 2a**

Slika 2: Mikrostruktura ledeburitnega orodnega jekla Cr-V v mehko žarjenem stanju: a) SEM, b) EDX-razporeditev vanadija s **slike 2a**, c) EDX-razporeditev kroma s **slike 2a**

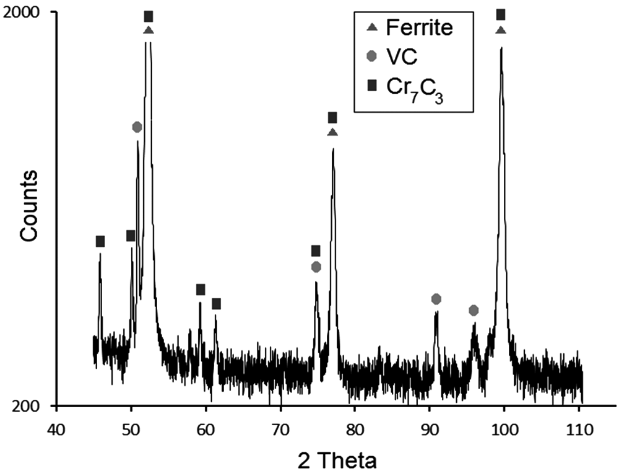

Figure 1: Diffractogram of the annealed sample measured at room temperature

Slika 1: Rentgenski difraktogram žarjenega vzorca, posnet pri sobni temperaturi

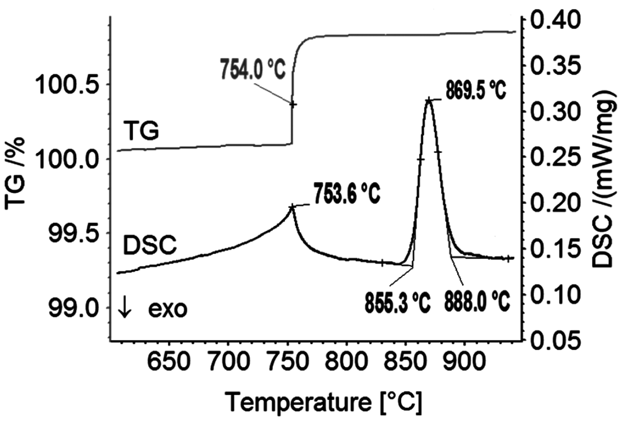

Figure 3: DTA and TG curves of the as-prepared tool-steel powder at the heating rate of 10 K/min

Slika 3: Krivulji DTA in TG prahu orodnega jekla pri hitrosti ogrevanja 10 K/min

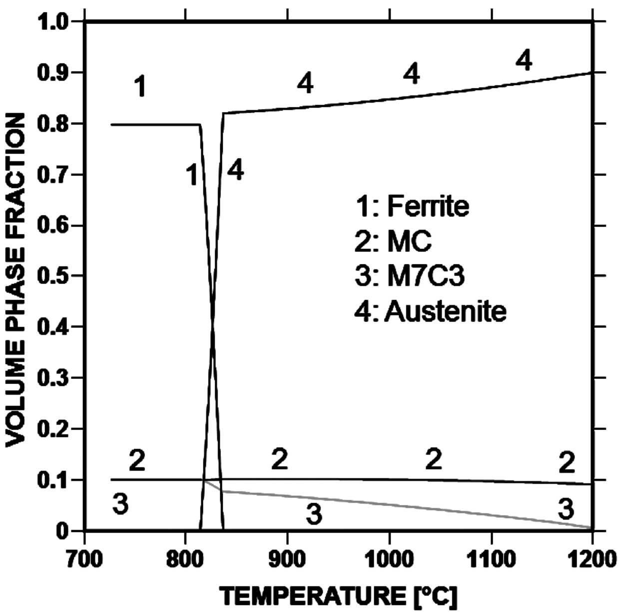

Figure 4: Volume-phase fractions in dependence on the temperature calculated with Thermo-Calc

Slika 4: Volumenski delež faz v odvisnosti od temperature, izračunane s Thermo-Calc

Figure 5 shows the dilatometry curves of Cr-V tool steel during heating (2 K/min) and cooling (3 K/min). By analyzing the curve of the thermal-expansion coefficient, the characteristic temperatures of the ferrite-to-austenite transformation were determined. The transformation start and finish temperatures during the heating are thus 843.0 °C and 872.6 °C, respectively. The transformation start and finish temperatures during the cooling were determined as 760.2 °C and 732.3 °C, respectively.

The other dilatometry measurements were performed at different heating rates and the results regarding the transformation start and finish temperatures of the ferrite-austenite transformation are summarized in **Table 1** and **Figure 6**.

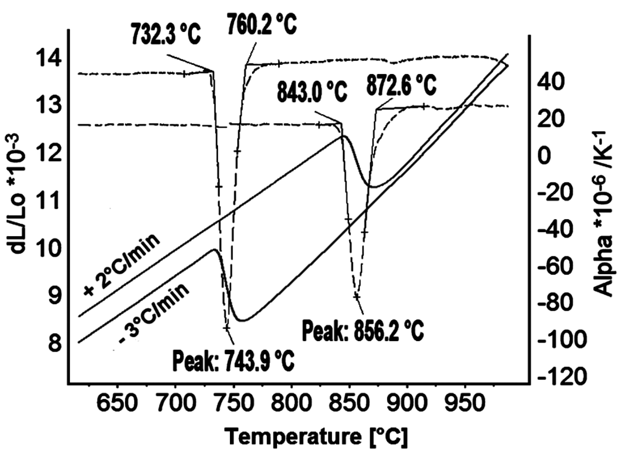

Figure 5: Dilatometric curves at the heating rate of 2 °C/min and the cooling rate of 3 °C/min

Slika 5: Dilatometrijska krivulja pri hitrosti ogrevanja 2 °C/min in pri hitrosti ohlajanja 3 °C/min

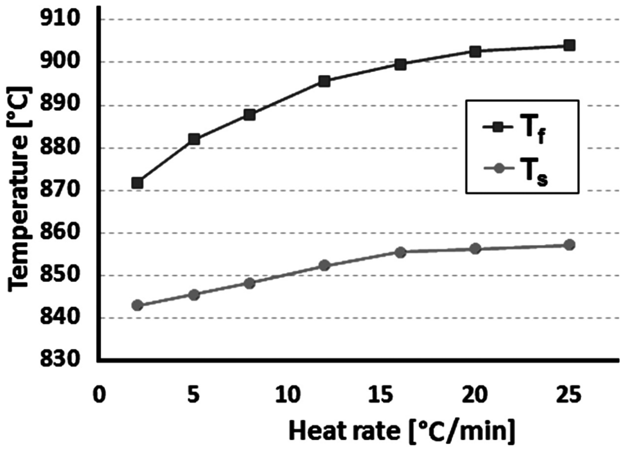

Figure 6: Transformation start and finish temperatures of the ferrite-austenite transformation in dependence on the heating rate

Slika 6: Temperature začetka in konca pretvorbe ferit-avstenit v odvisnosti od hitrosti ogrevanja

Table 1: Start/finish temperatures of the ferrite-to-austenite transformation at different heating rates, with the calculated temperature range of the transformation

Tabela 1: Temperature začetka – konca pretvorbe ferita v avstenit pri različnih hitrostih ogrevanja z izračunanimi temperaturnimi območji pretvorbe

Heating rate	2 °C/min	5 °C/min	8 °C/min	12 °C/min	16 °C/min	20 °C/min	25 °C/min
T_s/°C	843.0	845.5	848.2	852.3	855.5	856.3	857.1
T_f/°C	871.8	881.9	887.8	895.6	899.6	902.6	904.0
ΔT/°C	28.8	36.4	39.6	43.3	44.1	46.3	46.9

3 DISCUSSIONS

In this work it was confirmed that the microstructure of Cr-V tool steel after soft annealing consists of a ferritic matrix and M_7C_3 and MC carbides. M_7C_3 starts to dissolve during the ferrite-to-austenite phase transformation and a complete dissolution of M_7C_3 in the austenite occurs at the temperature of about 1200 °C. The MC carbide is more stable and its amount remains unchanged from room temperature up to 1200 °C. The temperature range of the ferrite-to-austenite phase transformation determined with dilatometry is 843.0–872.6 °C using a slow heating rate (2 °C/min) and the transformation during cooling occurs in the temperature range of 760.2–732.3 °C. The temperature range of this phase transformation calculated with Thermo-Calc is 814–837 °C. If the heating rate in dilatometry measurements increases (2–25 °C/min), the transformation start temperature also increases from 843.0 °C to 857.1 °C, while the transformation finish temperature increases from 871.8 °C to 904.0 °C and the temperature range of this transformation also increases from 28.8 °C to 46.9 °C.

The results for the thermal stability of M_7C_3 and MC carbides in Cr-V steel are in agreement with and explain the results published previously.[10] The future work will be focused on developing a kinetic model of the ferrite-austenite phase transformation in this system using the dilatometry data. The dissolution of M_7C_3 in austenite will be modeled using the Dictra software to enhance the knowledge about these kinetic processes occurring in Cr-V tool steel during heating.

4 CONCLUSIONS

The microstructure and phase transformations in Cr-V tool steel were analyzed using experimental and computational techniques. The main results are summarized as follows:

- the microstructure of Cr-V tool steel after soft annealing consists of a ferritic matrix and M_7C_3 and MC carbides
- M_7C_3 starts to dissolve during the ferrite-to-austenite phase transformation and is completely dissolved in the austenite at 1200 °C
- MC is more stable and its amount does not change from room temperature up to 1200 °C
- the phase transformation of ferrite to austenite proceeds in the temperature range of 843.0–871.8 °C, determined at the low heating rate by dilatometry by using the dilatometry data obtained at different heating rates, a kinetic model of the ferrite-to-austenite phase transformation will be proposed in the near future, and the kinetics of the dissolution of M_7C_3 in austenite will be calculated using the Dictra software.

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