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Phase Transformations in High Alloy Cold Work Tool Steel

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Phase transformations in the alloy tool steels have a crucial effect on the final properties of the steels. High alloy systems have different solidification conditions compared to construction steels. This paper deals with the phase evolution in high alloy tool steel in quasi-equilibrium state.

For analysis various methods such as differential thermal analysis, thermomagnetometry, light microscopy, scanning electron microscopy with energy dispersive analysis, X-ray diffraction analysis and dilatometry, were used. The analysed tool steel solidifies in three steps, and at lower temperatures secondary carbides are formed. Solidification begins at 1340 °C and is finished at 1208 °C. The Curie temperature of this steel is approximately 780 °C.

Keywords: high alloy cold work tool steel, differential thermal analysis, scanning electron microscopy, X-ray diffraction analysis, dilatometry

0 INTRODUCTION

The development of high alloy tool steel is important due to the requirement for achieving better mechanical and physical properties. High alloy tool steels of ledeburitic type produced by powder metallurgy (PM) contain a high amount of carbon and alloying elements (mainly V, Cr, Mo) that form carbides [1] to [3]. Conventional methods preparation of ledeburitic type tool steels (mould casting and forming) was influenced by the liquation and segregation processes, which lead to anisotropy of microstructure and properties of high alloy tool steels with high carbon content. This fact limits the applications of these steels as performance tools [4] to [8]. To improve the properties of the ledeburitic tool steels, the technology of powder metallurgy can be used [7], [9] and [10].

The main advantages of PM high alloy tool steel are the homogeneous distribution and fine size of carbides and uniform chemical composition in cross section, thereby better properties are achieved [11] and [12]. Due to specific properties cold work tool steels have found a wide spectrum of applications in various industries at blanking, forming, shearing, punching and other applications [13] to [15].

Solidification of ledeburitic steel in quasi-equilibrium conditions starts by austenite formation, then the formation of morphologically different MC, M_7C_3, M_6C and $M_{23}C_6$ carbides follows and the solidification is finished by eutectic reactions [16] to [19]. Some aspects of solidification of the high alloyed tool steels are described in [20].

In the case of rapidly solidified powders the solidification usually starts by the formation of solid solution with the dendritic, equiaxed and mixed types of solidification microstructures. However, due to recalescence effects also some amount of eutectic is usually formed [17] and [21] to [23]. For consolidation of powder the hot isostatic pressing (HIP) is often used. During HIP processing of rapidly solidified powder particles of high alloy tool steel significant changes in their microstructure and properties occur.

The differential thermal analysis technique/ thermomagnetometry is often used to determine the phase transitions including melting and solidification, liquation and formation of eutectics, recrystallization, dissolution and precipitation of new phases, solid-state transformation, and ferromagnetic to paramagnetic transition in a wide range of materials. Thermal analyses together with appropriate thermodynamic calculations can be used for analysis of such different materials as lead-free solders [24] and [25], carbon steels [26] and tool steels [27].

The aim of this work is the investigation of microstructure and phase transformations in selected high alloy cold work tool steel.

1 EXPERIMENTAL PROCEDURE

The investigated material was equivalent to high alloy cold work tool steel K390 Microclean. The chemical composition of steel is given in Table 1. Higher vanadium content ensures good tool wear resistance and high hardness (about 66 HRC) after heat treatment.

Analysed high alloy cold work tool steel has an extremely high wear resistance, outstanding toughness and high compressive strength. [15]

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Table 1. *Chemical composition of the investigated high alloy tool steel [wt. %]*

C	Cr	Mo	Si	V	W	Co
2.47	4.15	3.62	0.41	8.94	1.13	2.02

The compacts were prepared by HIP processing of the rapidly solidified powders. The parameters of processing were: 1100 °C, 100 MPa, during 90 minutes with protective gas Ar [28]. From compacts the samples for differential thermal analysis/ thermomagnetometry (DTA/TM) and dilatometry were prepared.

For simultaneous DTA and TM experiment the sample with total weight of 105 mg was prepared and Netzsch STA 409 CD apparature with heating up to 1600 °C in Ar protective gas (60 ml/min.) was used. The heating and cooling rates for measurement were 10 K/min, during three measurement runs. The differences in DTA curves in the 2nd run and in 3rd run were negligible. This fact indicates that the sample was in quasi-equilibrium state after the 1st run, so only the 2nd run was further analyzed.

The microstructure of sample obtained from DTA experiment after standard metallographical procedure and chemical etching in 3% Nital was observed using the Neophot 32 light microscope and Jeol JSM-7600F scanning electron microscope (SEM). The experimental technique of scanning electron microscopy and energy dispersive X-ray spectroscopy (EDS) was used to characterize the composition of the phases in the steel after DTA.

X-ray diffraction analysis was carried out by means of Philips PW 1810 X-ray diffractometer with Co anode ($\lambda CoK\alpha = 1.72091 \times 10^{-10}$ m) and secondary monochromator. The measuring step was 0.02°, for each step the holding time 10 s was used. For X-ray diffraction analysis the sample from DTA experiment was used.

To characterize the phase transformations in solid state the dilatometry analysis of bulk sample prepared from the HIP compact was used. The initial sample length was 9.58 mm. The analysis was performed using Netszch 402 C dilatometer in Ar protective gas with heating rates of 3 K/min, during two measurement runs. Also, in this case only the second run was further analysed.

2 RESULTS

2.1 Microscopy

The microstructure of the sample after DTA obtained by light microscopy is shown in Fig. 1. It can be seen that the microstructure is dendritic, however, each dendrite consists from carbide eutectic colonies. At the boundaries of eutectic colonies (Fig. 2) a secondary skeleton eutectic is present.

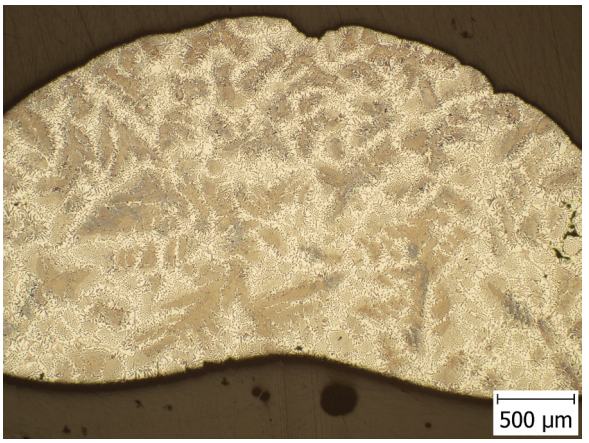


Fig. 1. *Microstructure of the tool steel after DTA*

For the more detailed interpretation of the microstructure SEM and EDS mapping techniques were used. On the base of element distribution (Fig. 3) it is shown that eutectic colonies contain the vanadium carbide and ferrite. The white carbides localized at the boundaries between eutectic colonies are on the base of molybdenum carbide. The eutectic colonies are rich in molybdenum and chromium. Cobalt and tungsten are distributed uniformly in all phases in material.

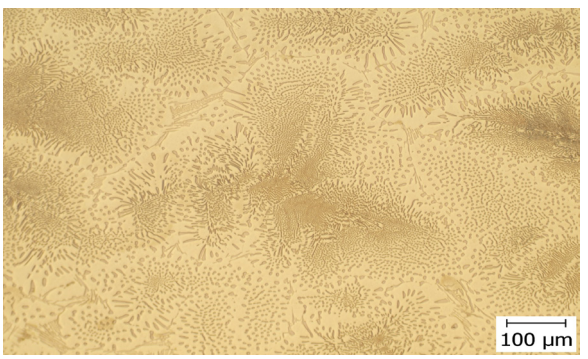
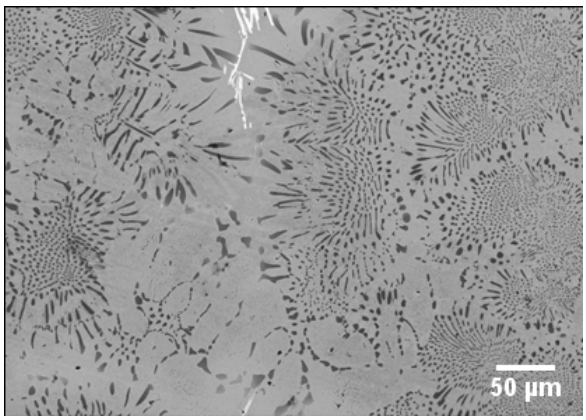


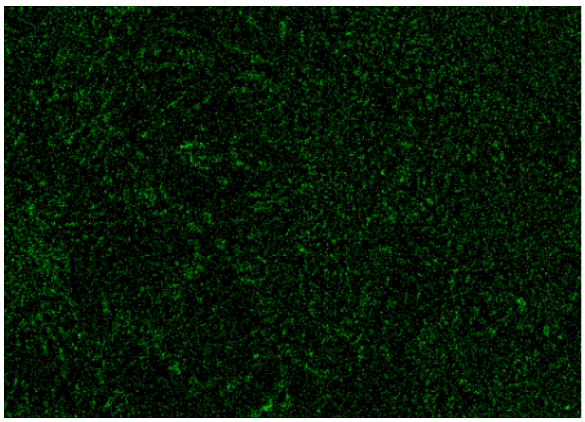
Fig. 2. *Detail of the microstructure*

2.2 X-ray Diffraction Analysis

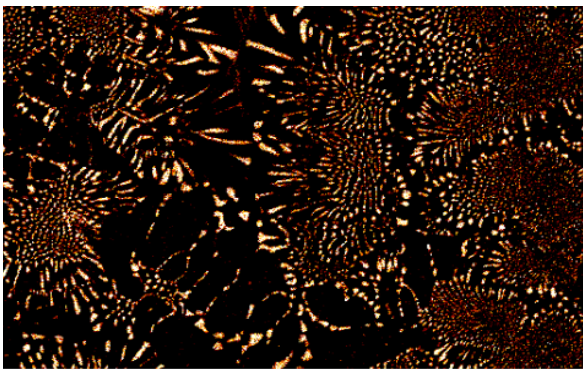
X-ray diffraction pattern of sample after DTA is shown in Fig. 4. The following phases were observed: ferrite, MC carbide (vanadium type), M_2C and M_6C carbides (molybdenum types). Due to mixing of different substitutional atoms in carbide lattices the diffraction patterns of carbides are slightly shifted, compared to ICDD database [29] and [30].



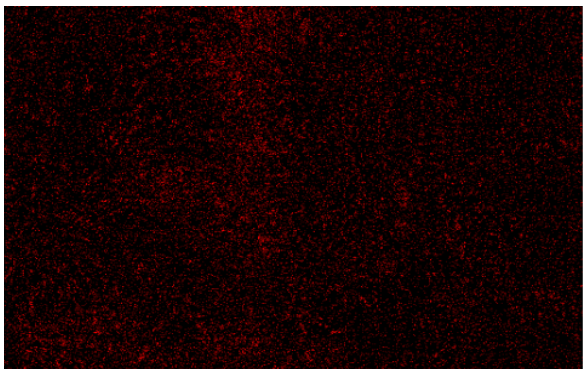
Mapped image (SEM)



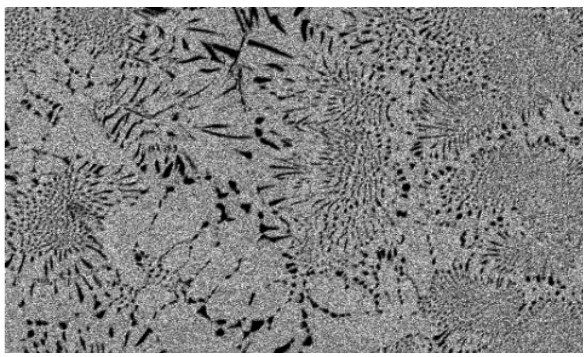
C Kα1,2



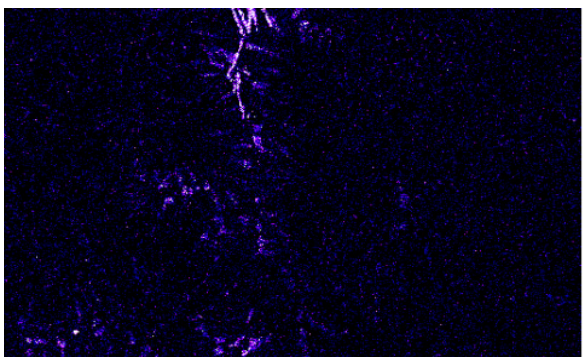
V Kα1



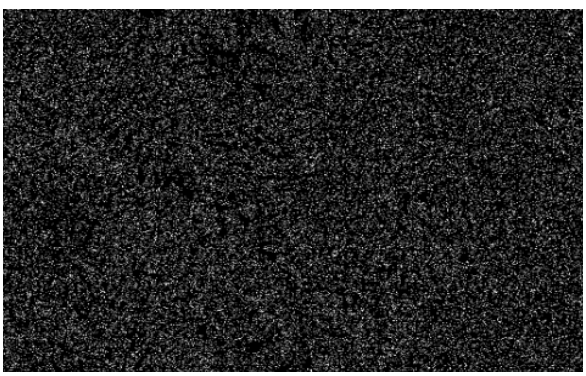
Cr Kα1



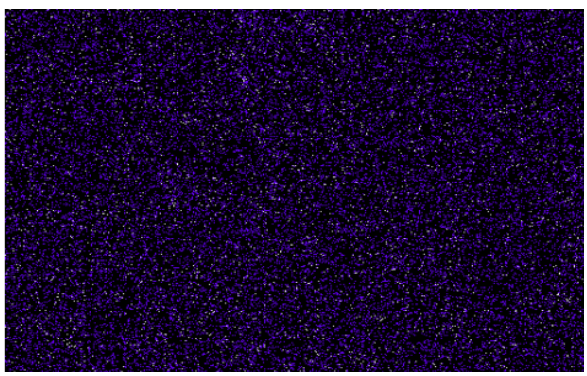
Fe Kα1



Mo Lα1



Co Kα1



W Lα1

Fig. 3. *Element mapping of high alloy tool steel, showing distribution of C, V, Cr, Fe, Mo, Co and W*

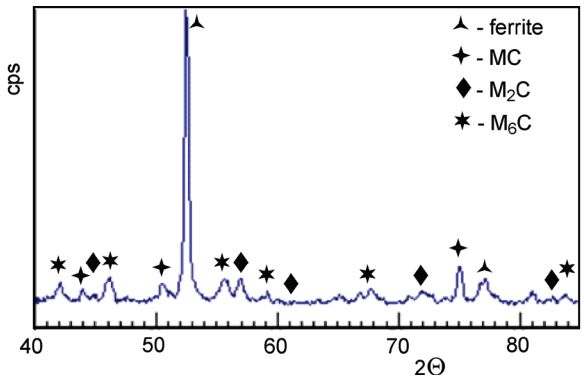


Fig. 4. *X-ray diffraction pattern*

2.3 Differential Thermal Analysis and Thermomagnetometry

Figs. 5 and 6 show DTA curves during heating and cooling of sample, respectively. The first peak in Fig. 5 represents the transformation from ferromagnetic to the paramagnetic state (781 °C). The second endothermic peak (onset 856 °C) can be considered as the transformation of ferrite to austenite. Next peaks characterize the melting of the present phases. The melting begins at 1209 °C, and the end of melting the temperature is about 1389 °C.

2.4 Dilatometry Analysis

The dilatometry curve of analysed tool steel is shown in Fig. 7. The onset around temperature 660 °C is possibly caused by dissolving the secondary carbides in matrix. The steep decrease in length corresponding to the transformation of ferrite to austenite is present at about a temperature of 832 °C.

DTA curve during cooling (Fig. 6) shows that solidification proceeds in three steps. Solidification probably begins by austenite formation from

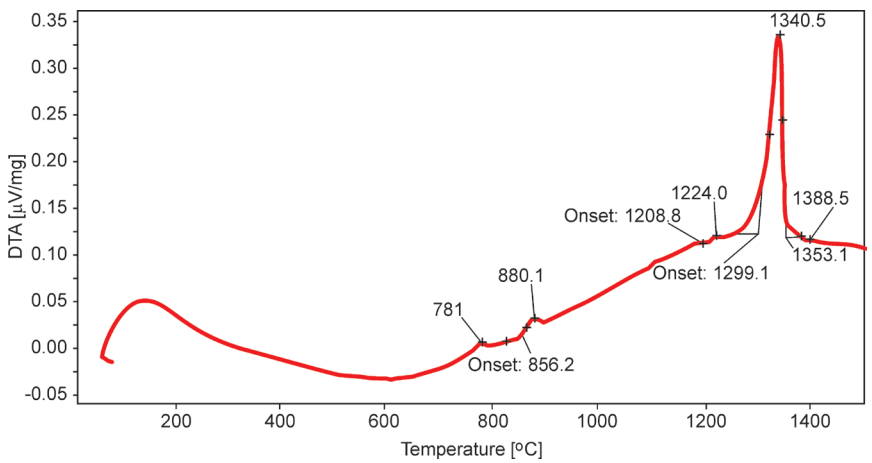


Fig. 5. *DTA curve of the investigated high alloy tool steel during heating*

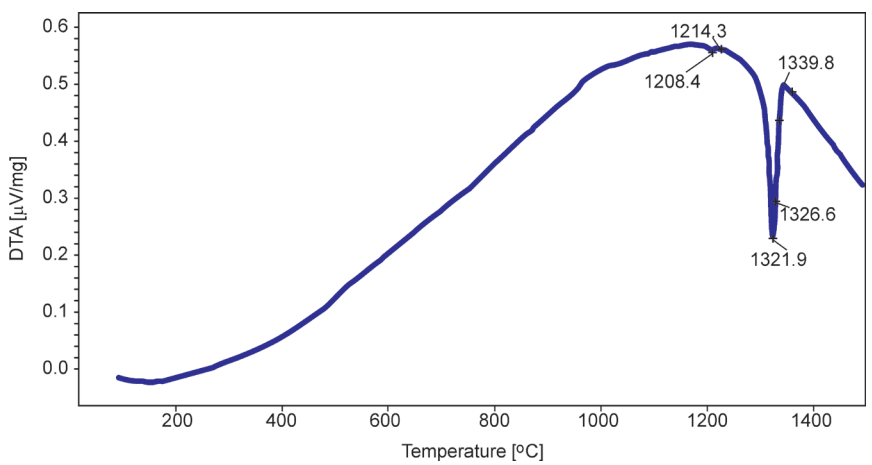


Fig. 6. *DTA curve of the investigated high alloy tool steel during cooling*

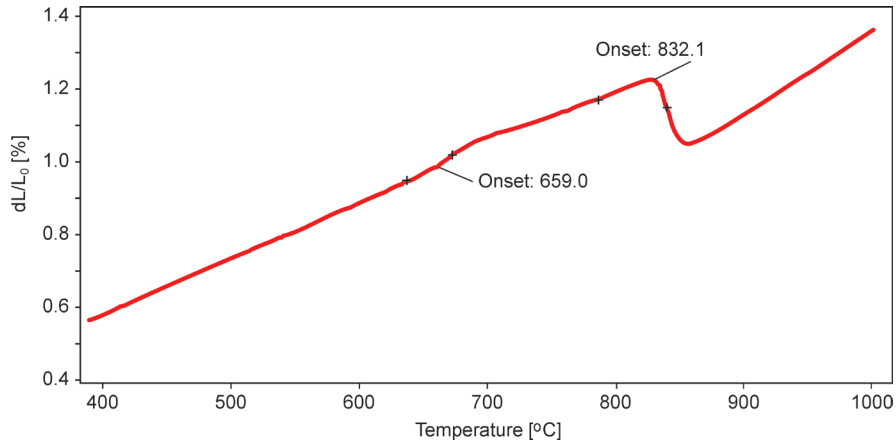


Fig. 7. *Dilatometry curve of analysed tool steel during heating*

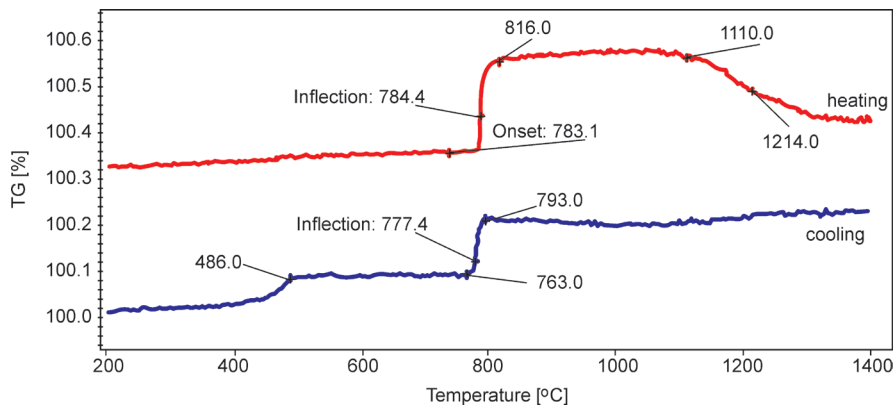


Fig. 8. *Thermomagnetometry curves*

undercooled melt at a temperature of 1340 °C. The solidification continues with two eutectic reactions. During the first reaction probably eutectic with vanadium carbides is formed (at 1327 °C). During the second reaction the eutectic with molybdenum carbides is formed (at 1214 °C). The solidification is finished at a temperature of 1208 °C. Fig. 8 shows the TM curve during heating and cooling of sample. About the temperature 780 °C the transformation from paramagnetic to ferromagnetic state is seen. During heating the curve slowly decreases from 1110 °C, probably due to dissolution of paramagnetic MC carbides. The cooling curve shows that at 486 °C also some secondary carbides form.

3 DISCUSSION

During heating of the high alloy cold work tool steel K390 Microclean the first secondary carbides start to dissolve in matrix at 660 °C. The transition from ferromagnetic to paramagnetic state takes place at 781 °C (determined by DTA) or 783 °C (determined by TM). Then, the transformation of ferrite to austenite at a temperature of 856 °C (determined by DTA) occurs. This temperature is higher than the temperature of ferrite to austenite transition determined by dilatometry (832 °C), probably due to higher heating rate at DTA measurements. Melting process of tool steel begins at temperature 1209 °C and continues in three steps. The first step is probably the melting of molybdenum carbide eutectics at 1209 °C. They are localised on the boundary of eutectic colonies Melting of the material continues by melting of vanadium carbide eutectic colonies. Finally, melting finishes when the last amount of austenite is dissolved in the melt at temperature 1389 °C.

Also, solidification of the material proceeded in three steps. In undercooled melt the austenite grains grow at the temperature 1340 °C. Next, solidification continues by the evolution of eutectic colonies based on the vanadium carbide (starts at 1327 °C). Molybdenum is diffusing to the melt and in the

last step of solidification the molybdenum carbide eutectics at the boundaries are formed. The cooling in solid state shows only two transitions in TM curve. The first change is the transition from paramagnetic to ferromagnetic state at 793 °C and the second one is the formation of the secondary carbides in the matrix at 486 °C. The resulting microstructure is mainly dendritic where most dendrites consist from vanadium carbide eutectics. At the boundaries molybdenum carbide eutectics are localised. The small amount of austenite dendrites was also present. This type of microstructure is quite differrent compared to [11], [16] to [18], [22], [23], [27], [28], [31] and [32], where microstructures containing a large amount of austenite dendrites or a large amount of primary carbides in dependence from the chemical composition of the ledeburitic tool steels are present.

4 CONCLUSIONS

The aim of the article is to describe the phase transformations in high alloy cold work tool steel K390 Microclean during slow heating and cooling. These conditions are different compared to those used in production of the alloy steels, but they enable a description of the solidification and phase transitions in quasi-equilibrium conditions.

The microstructure of high alloy cold work tool steel has a dendritic morphology, however, dendrites consists from eutectic colonies based on the vanadium carbides. On the boundaries there are localised molybdenum carbide eutectics. The transformation of austenite to ferrite occurs at about 830 °C. The transition from ferromagnetic to paramagnetic state is about 780 °C.

The knowledge about the quasi-equilibrium phase transformations in high alloy cold work tool steel can help to a better understanding of processes occurring in the material during the heat treatment. The obtained results extend the data obtained by others authors related to ledeburitic tool steels [1], [11], [16], [17], [23], [27], [31] and [32]. The results may be useful for the next thermodynamic analysis of phase transitions using the Thermo-Calc and Dictra software.

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