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DETERMINATION OF AUSTENITIZATION AND MARTENSITIC TRANSFORMATION TEMPERATURES OF M398 STEEL

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Abstract:

The article deals with dilatometric analysis of M398 steel. It is a chromium steel, produced by Böhler using MICROCLEAN powder metallurgy process. The investigated steel is high-alloy chromium and vanadium with a high carbon content. The steel is characterized by high strength, resistance to abrasive wear and corrosion. The result of the dilatometric analysis will be dilatation curves for selected cooling modes in order to determine the initial austenitization temperatures Ac_1 and Ac_3 and the beginning of the matrensitic transformation Ms. Since the steel is highly alloyed with Cr and V, its microstructure is formed by a very high content of carbides of the M_7C_3 and MC type. These carbides affect the resulting mechanical properties of the material M398 and predetermine its use for screws in injection molding machines in the plastics industry, where this steel has the highest use so far.

1 Introduction

1.1 Dilatometric analysis

Dilatometric analysis is an experimental method used to study the phase transformations of metals and their alloys. The method uses volume changes associated with phase changes and is based on recording the change in the length of the experimental sample due to the temperature during its heating and cooling [1].

By dilatometric analysis, we can also determine, in addition to the phase transformations of the material also the thermal expansion, the rate of course of the phase transformations, and the values of critical temperatures. The volume changes during the phase transformation arise due to the difference between the grid parameter of the original and the newly formed phase. In the case of steels, it is mainly the transformation of the α phase (ferrite, K8) to the γ phase (austenite, K12) during heating associated with austenitization and subsequent transformation of austenite γ to martensite, bainite or perlite during cooling. The lattice parameter γ - iron is approximately 3.65×10^{-10} m. The value of the grid parameter - iron depends on the temperature and increases up to the value of 2.9×10^{-10} m [2].

Another variation of dilatometric measurement can be the measurement of deformation processes under heat, in which we monitor the dependence of defor-

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mation and temperature of the examined sample. The authors of Krbaťa, Barényi, Eckert, Mikušová, deal with this topic in an article entitled: Hot Deformation Process Analysis and Modelling of X153CrMoV12 Steel.[3]
For the required measurement accuracy, it is necessary to use a suitable dilatometric device. Experimental measurements will be performed on a dilatometric device DIL 805. The output of the measurement will be a dilatation curve, representing the dependence of the change in length during heating and cooling of the examined sample. In the case of a phase change, the length does not change in proportion to the temperature change. The volume change of the sample will subsequently be reflected on the dilatation curve Fig.1, which will allow us to evaluate it.

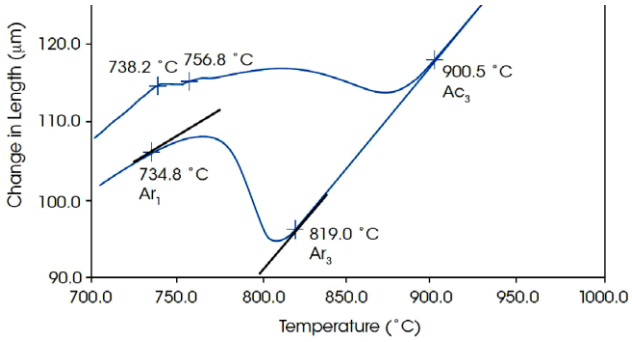


Fig. 1 Dilatation curve for medium alloy steel - high temperature range [4]

In the case of steels, these are mainly the limit temperatures in the ARA and IRA diagrams, which are important in the design and optimization of heat treatment processes [3, 4]. The main parameter influencing the shape of the resulting ARA diagram is the proportion of individual alloying elements. The secondary parameter influencing the shape of the ARA diagram is the height of the austenitization temperature. In Fig. 2 we can see how the individual alloys affect the final shape of the diagram.

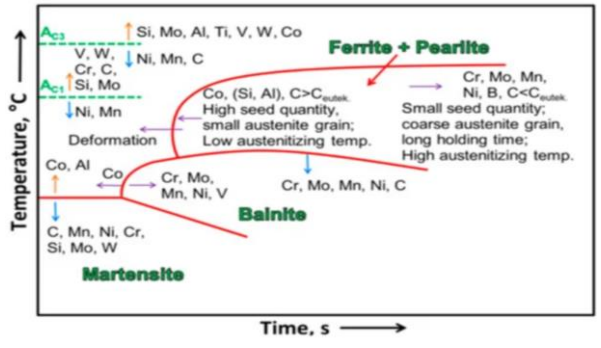


Fig. 2 Influence of alloying elements on the shape of the ARA diagram

2 Experimental details

2.1 Chromium tool steel M398

In industrial practice, a variety of tool steels are used in the manufacture of various components, which are subject to high stress and wear during processes of friction that have a large impact during their operating life.[5]
The investigated material M398 developed by BÖHLER focuses on the high requirements in the field of plastics processing. It is a high-carbon, martensitic steel, made of powder metallurgy. Thanks to the production method and chemical composition, the steel provides extremely high resistance to mechanical wear as well as corrosion resistance.
The prerequisite for the use of steel is the replacement of the currently used material M390 in the production of injection molding screws. Thanks to the high wear resistance of M398 steel, it would be possible to create screws enabling the processing of plastics with an increased content of glass fibers or to prolong the life of the screws. Other properties of M398 steel include high dimensional stability during heat treatment, good corrosion resistance, the possibility of polishing to a high gloss.
Table 1 shows the chemical composition provided by BÖHLER as well as the results of the spectral analysis provided by SPECTROLAB Jr. CCD device of the investigated M398 steel.

Table 1 Chemical composition of the M398 steel (wt. %)

	BÖHLER M398	Spectral analysis M398
C	2.70	2.65
Si	0.50	0.55
Mn	0.50	0.51
Cr	20.00	20.09
Mo	1.00	1.00
V	7.20	7.1
W	0.70	0.43

Figures Using the THERMOCALC software, a phase diagram of Fig. 3 and a diagram of the phase fractions of Fig. 4 of the examined steel with a carbon content of up to 3% were created. The created pair of diagrams is a useful tool in the analysis of expansion curves, as we can assume the formation of individual phases at given temperatures.

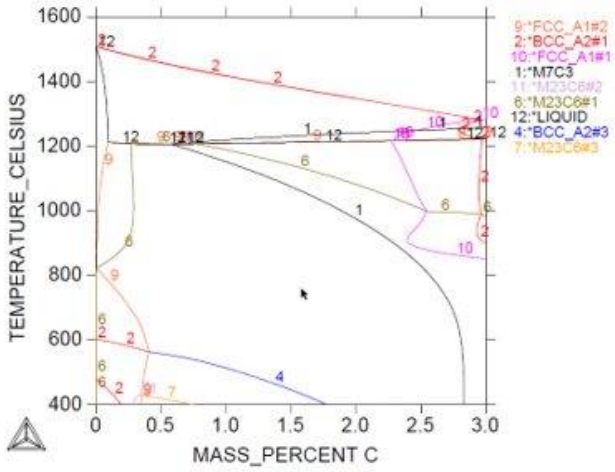


Fig. 3 Phase diagram of M389 steel with carbon content < 3%

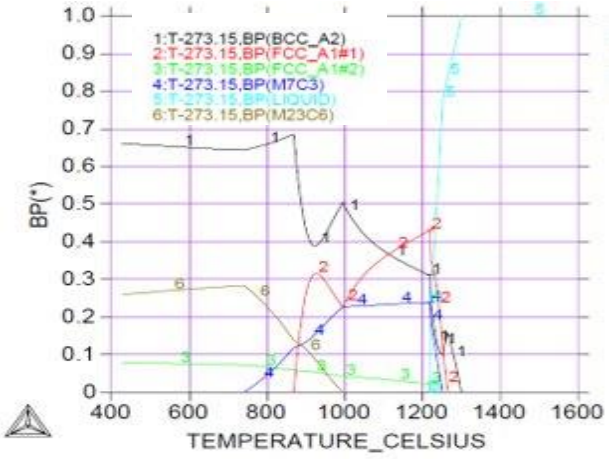


Fig. 4 Phase fractions

The main concept for increasing the macro-hardness is the high content of MC and M_7C_3 carbides, which can be observed in the microstructure itself provided by BÖHLER in Fig.5.

On the Fig. 6 we can observe the effect of tempering temperatures on the resulting hardness of the material M398. As the figure shows, the highest hardness is reached after cooling the material to negative temperatures. With this material, the cooling temperature following hardening is set at -70 °C, with a residual austenite value of less than 1% (Fig. 7). Tempering temperatures in the range of (200 - 300) °C are suitable when the material is designed for high corrosion resistance. For materials that have not been cooled to sub-zero temperatures, there is an area in the tempering temperature range of (540 - 560) °C where the material is most resistant to wear. For materials that are frozen, this area is shifted between (510 - 530) °C.

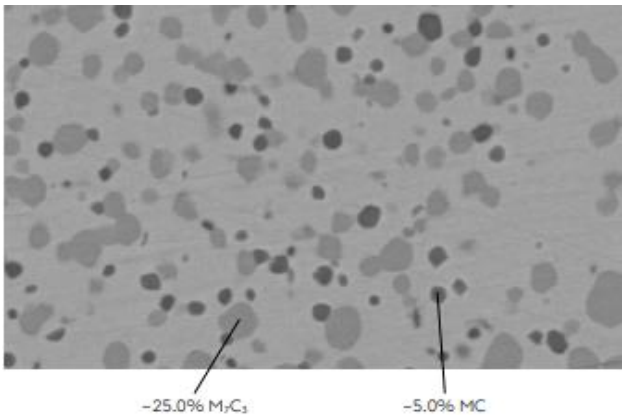


Fig. 5 Microstructure of M398 steel [6]

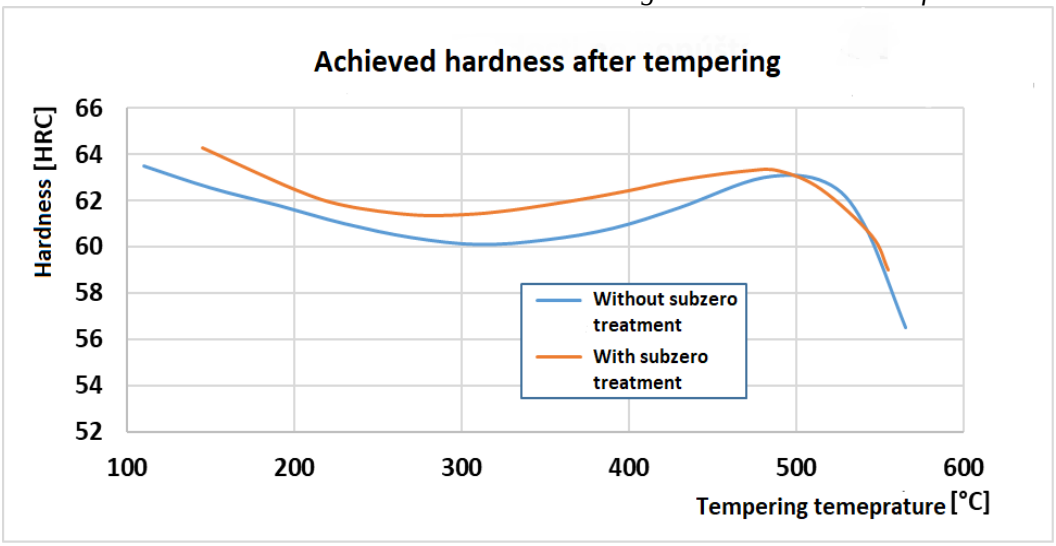


Fig. 6 Graph of achieved hardness after tempering of M398

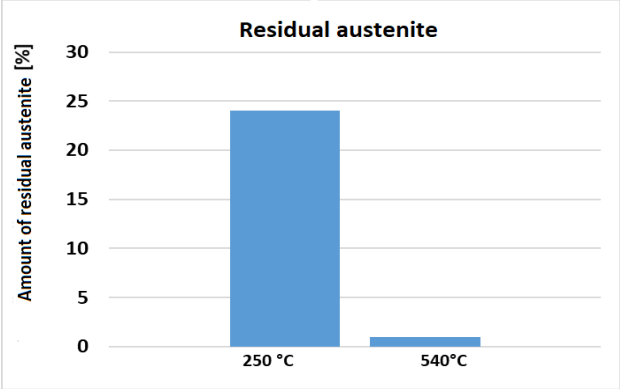
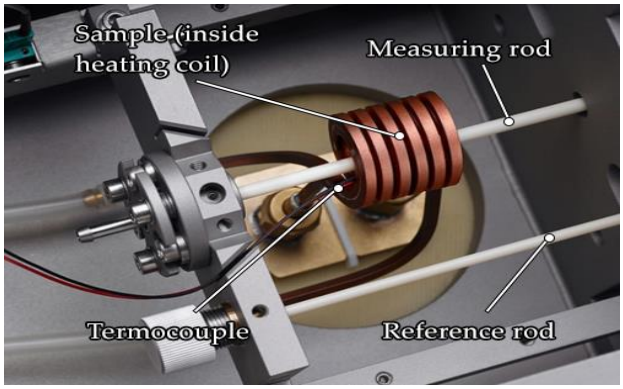


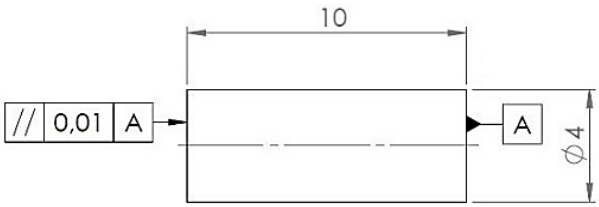
Fig. 7 amount of residual austenite after heat treatment

2.2 Methodology of dilatometric analysis using dilatometer DIL 805A

The DIL805A / D dilatometer is a laboratory device that is used either to measure and record expansion curves or to measure hot deformation resistances. It is intended for physical modeling of heat treatment processes (805A) or hot metal forming processes (805D) [5,6]. An experimental sample of the prescribed shape and dimensions is placed in a working chamber (Fig. 8a) between the Al_2O_3 tips. The tips are connected to a precision extensometer, which thus records changes in length during the execution of the set temperature cycle. [7]



a)



b)

Fig. 8 a) Working chamber of dilatometric device DIL805A, b) parameters of experimental sample for DIL805A

Welded to the sample are high temperature resistant conductors based on high fusible metals (Pt, Pt + Rh), connected to a thermocouple for recording and regulating the temperature inside the chamber. [7]
The first step of dilatometric measurement is to set the temperature mode and its parameters using the device software. The system makes it possible to carry out one or more successive temperature cycles, consisting of heating, possible holding at temperature and cooling. After inserting the sample and connecting the thermocouple to the system, the chamber is closed and evacuated. The phases of heating the sample and holding at temperature take place in a vacuum (5×10^{-3} mbar). The sample inserted inside the coil is heated by induction heating. At the beginning of the cooling phase, the heating is switched off and a cooling gas is pressurized into the chamber - most often H_2, N_2 or Ar. During the whole process, the temperature of the sample and its change in length due to temperature with a resolution of 0.05 µm / 0.05 °C are recorded very accurately. The graphical representation of this record is the dilatation curve. The step changes in the dimension on the curve represent phase changes (PF → A, A → B, A → M, etc.). The dilatometer software has tools for reading the temperatures of the beginning and end of these phase changes, most often in the form of a tangent at the point of beginning of change (first derivative dl / dT) or second derivative dl / dT. [7]
An experimental samples with dimensions according to Fig. 8b. were prepared for dilatometric analysis of M398 steel. Subsequently, 3 dilatometric measurements representing rapid cooling were performed on a DIL805A dilatometer. The measurement itself has three phases, heating, endurance, cooling. The heating rate of the sample was constant in all modes. Heating was performed at a rate of 1 °C/s followed by holding at 1150 °C for 30 min with cooling modes according to the parameters specified in Tab.2. The initial and final cooling temperatures were constant for all modes (T_{max} = 1150 °C, T_{min} = 50 °C). Cooling was performed using H_2 gas with ambient temperature (approx. 23 °C).

Table 2 Input cooling parameters for selected temperature modes of dilatometric analysis of M398 steel

Cooling mode	Cooling time T_{max} to T_{min}			Cooling rate	T_{max} [°C]	T_{min} [°C]
	t [s]	*t* [min]	*t* [hr.]	*v* [°C/s]		
1	11	0.18	0	100	1150	50
2	110	1.83	0	10	1150	50
3	220	3.67	0	5	1150	50

3 Results and discussion

The method for determining the limit temperatures Ac_1 and Ac_3 is shown in Fig. 9. The phase transformation is reflected in the expansion curve as a step change in the length of the experimental sample as a function of temperature. The initial temperature Ac_1 corresponds to the temperature at which the expansion curve begins to deviate from the linear expansion during heating due to the onset of austenite formation. Subsequently, the temperature Ac_3 is defined as the temperature at which the expansion curve begins to regain a linear character during heating. The average value obtained from all three dilatometric measurements for M398 steel is Ac_1 = 955 °C and Ac_3 = 1085 °C. The figure shows the derivation of the heating curve, which is used to determine the beginning and end of the austenitization temperatures Ac_1 and Ac_3. Also, in the figure we can observe a step change of the derivative curve at 710 °C and a return to its linear direction at 735 °C. This short deviation records the dissolution of M_7C_3 type carbides, which is also shown in Fig. 4.

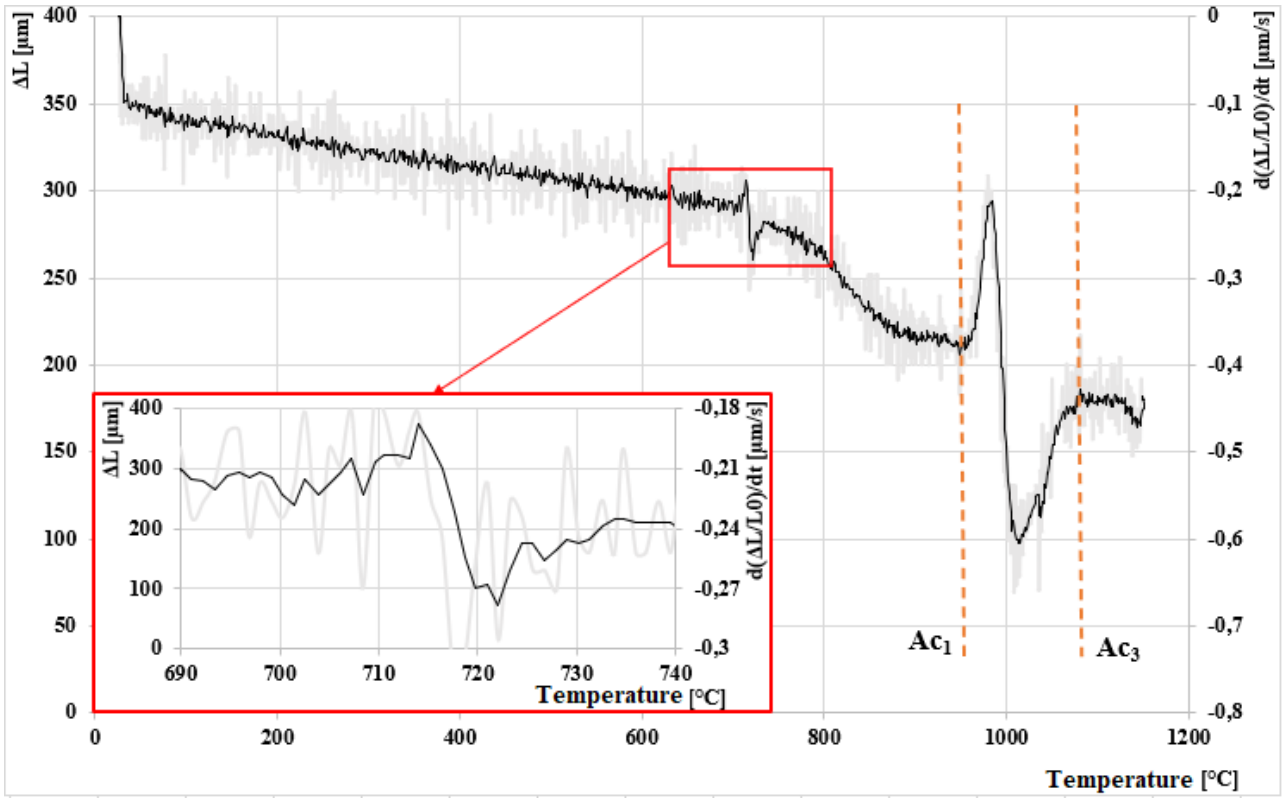


Fig. 9 Derivation of the dilatation heating curve

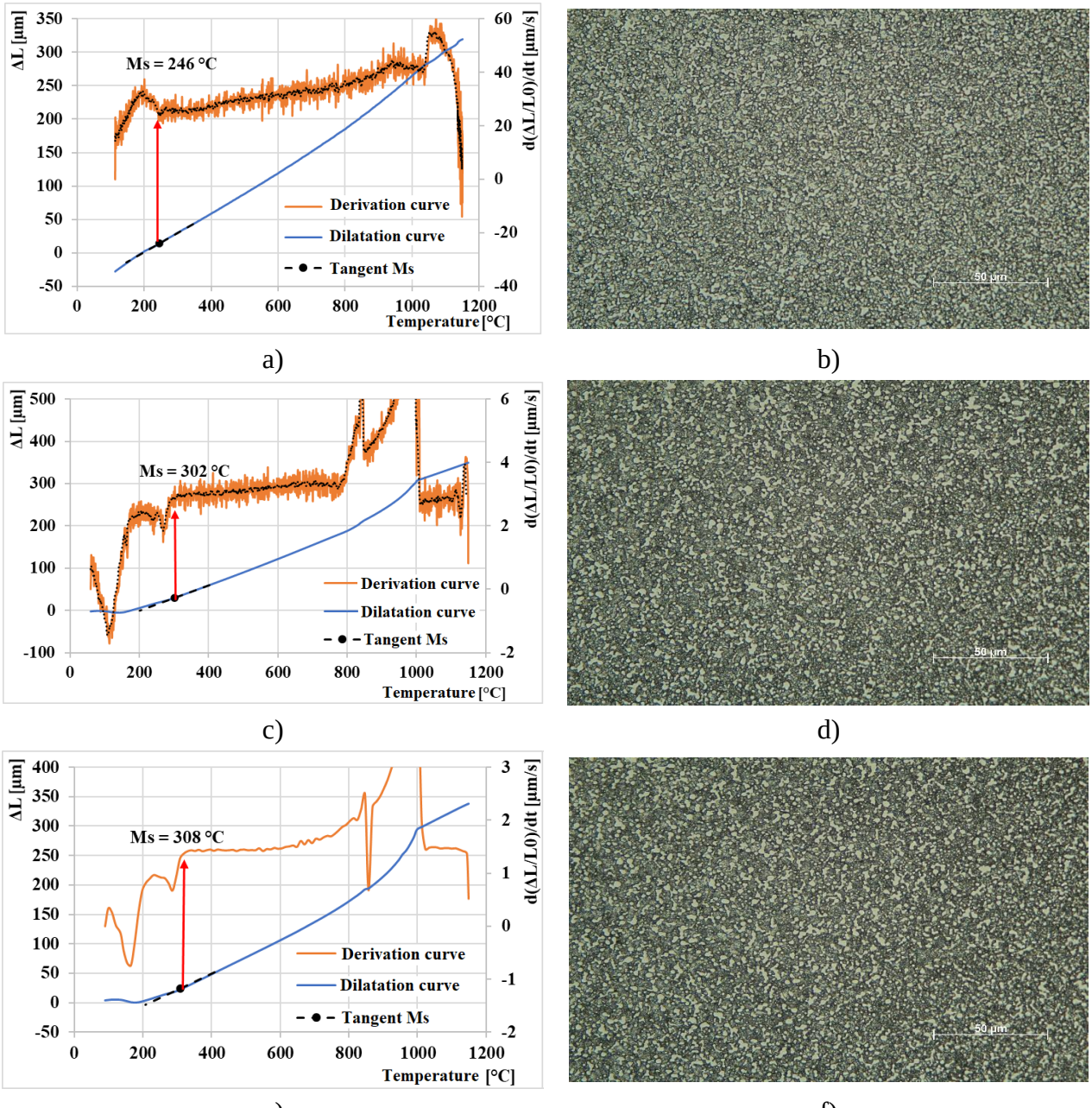


Fig. 10 Dilatation curves for selected cooling modes

The expansion curve from the austenitization temperature of 1150 °C is shown in Fig. 10a. An expansion curve cooled at 100 °C/s and its derivatives were used to determine the initial temperature of martensite formation and its value is Ms = 246 °C. The dashed line in the figure is a tangent copying the linear part of the expansion curve. The point of deviation between the tangent and the curve is considered to be the initial temperature Ms. This point also corresponds to the Ms temperature determined from the derivation curve. The final microstructure obtained is shown in Fig. 10b and is fully formed by a carbide-containing martensitic matrix.

Another expansion curve at a cooling rate of 10 °C/s is shown in Fig. 10c. Given the dilatation curve, two types of martensite probably formed in the structure of the material. Since the initial temperature Ms reached 302 °C, this shows an increase in the initial temperature Ms by 55 °C compared to the previous expansion curve. Due to the decreasing cooling rate, the temperature Ms cannot have an increasing character [10]. Likewise, this deflection cannot represent the beginning of the formation of a bainitic trans-

formation because the investigated M398 steel is highly alloyed with chromium and vanadium, and these two elements according to Fig. 2 move the entire ARA diagram to the right. The microstructure of the sample tax is shown in Fig. 10d. it is also formed by a martensitic matrix with a high carbide content.
The same paradox occurred in the last sample examined, which was cooled at a rate of 5 °C/s (Fig. 10e). Here, the initial temperature of Ms reached 308 °C. At a temperature of about 860 °C, a step change in the derivative curve can also be seen. This change is probably related to the transformation of the FCC austenitic lattice to a BCC lattice as shown in Fig. 4. The resulting microstructure is shown in Fig. 10f.
However, it must be stated that metallographic analysis using an optical microscope is insufficient. For a qualitative evaluation of the resulting expansion curves it is necessary to use an electron microscope, which will be equipped and a chemical analysis of EDS.

4 Conclusion

The paper describes dilatometric analysis of tool steel M398. The theoretical part of the article is supplemented by several thermo-mechanical-chemical properties of the investigated material M398. The study of the expansion behavior of the steel was performed at three different cooling rates of 100, 10 and 5 °C/s from an authentication temperature of 1150 °C. Dilatation results are supplemented by metallographic analysis of experimental samples using an optical microscope.
The following conclusions can be drawn from this work:

1) The temperature value Ac_1 = 955 °C and Ac_3 = 1085 °C were determined from all three measurements and their average value was determined. These temperatures reach higher values than conventional tool steels due to the high content of Cr and V-based alloying elements.

2) At a cooling rate of 100 °C/s, a martensitic matrix with a high carbide content is formed in the resulting structure. Carbides are formed on the basis of M_7C_3 and MC, which must be proved using an electron microscope and chemical analysis of EDS elements.

3) At cooling rates of 10 and 5 °C/s, two types of high carbide martensite were likely to form in the resulting structure. These carbides probably bound carbon and other alloying elements, while a different type of martensite began to form in their immediate area, which must be proved using an electron microscope and chemical analysis of EDS elements.

5 Acknowledgement

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