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COMPARISON OF METHODS FOR PHYSICAL DETERMINATION OF PHASE TRANSFORMATIONS TEMPERATURES

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Various physical methods for the determination of phase transformation temperatures were compared using C-Mn and C-Mn-V-Nb steels. The measurement using temperature scanner, variously located thermocouples, dilatometer, different thermal analysis (DTA) and anisothermal plastometric test were completed. The specimens were heated to 1 323 K and 1 473 K in the case of the C-Mn-V-Nb microalloyed steel. The aim of the different heat treatment were to obtain different levels of precipitates' dissolution. It was found that the better particles' distribution and precipitation due to the cooling lead to the enlargement of the two-phase region in the material. The good agreement of result gained by used methods was achieved. We found that all used methods can be used for common steels, but the temperature scanner seems not to be precious enough for microalloyed steels.

Key words: phase transformations temperatures, dilatometer, differential thermal analysis

INTRODUCTION

The classic thermal analysis represents the oldest and simplest method for determination of the melting point and solidification temperature of pure materials and further characteristic temperatures in case of alloys. It is based on registration of temperature changes in dependence on the heat released and/or absorbed during the phase transformation. Temperatures (T) of the system are measured in dependence on time (t). The phase transformation in progres shows itself by a typical anomaly on the curve T = f(t). This method is not very sensitive in case of the phase transformations realized in the solid state (they are less "thermally-coloured"), unlike the differential thermal analysis [1-3] and/or the examination by means of dilatometer [4-7].

In this contribution the various ways for the determination of phase transformation temperatures during cooling of steel were utilized, from the simplest thermal analyses by means of temperature scanners and/or thermocouples to more exact methods of the differential thermal analysis, the dilatometry and the plastometric tests. The aim was to compare the accuracy of results obtained by various methods and judge the possibilities of application of these methods in the laboratory conditions. The two low carbon steels were chosen as a base material, one of then was microalloyed by vanadium and niobium – Table 1.

Table 1 **Chemical composition of steels / wt. %**

	C	Mn	Si	P	S	V	Nb
C-Mn	0,25	0,76	0,360	0,008	0,007	-	
C-Mn-V-Nb	0,18	1,31	0,364	0,011	0,009	0,09	0,05

THERMAL ANALYSIS BY MEANS OF TEMPERATURE SCANNER

Thermal analysis was carried out by measurements of surface temperatures of specimens, cooled on air, using the temperature scanner [8]. The specimen with dimensions 40 x 90 x 120 mm was heated in the furnace at the temperature of 1 323 K for 7 minutes. After extracting the sample was immediately put under the temperature scanner and temperature was monitored during cooling. One more temperature was used in the case of

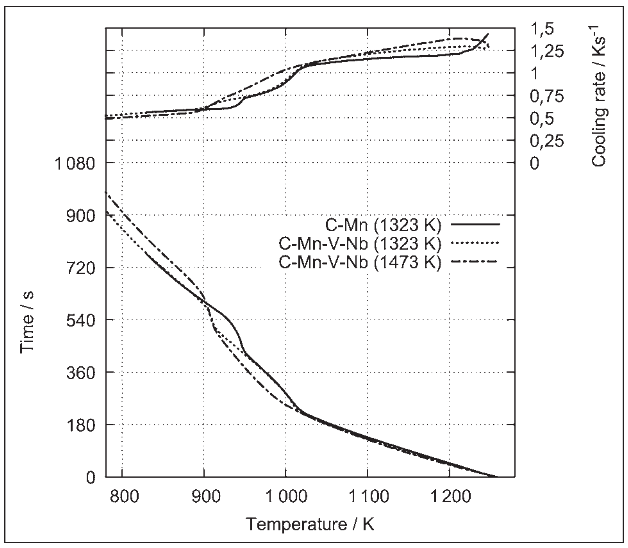

Figure 1 Cooling curves measured by means of temperature scanner

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C-Mn-V-Nb steel, where the specimen was heated to 1 473 K and kept there for 30 minutes to ensure dissolving of precipitates [9-10]. The cooling curves, determined for particular experiments by the temperature scanner, are shown in Figure 1. The average cooling speed within a particular experiment was approx. 0,5 K/s, at higher temperatures (i.e. in the region of austenite) it was naturally higher.

THERMAL ANALYSIS BY MEANS OF MEASUREMENTS BY THERMOCOUPLES

In measurements of the phase transformation temperatures the thermocouples drilled into the volume of the investigated specimen has often been used – i.e. unlike the temperature scanner, by means of the thermocouple no surface temperature is measured. In our case two thermocouples of type K with thickness 2 mm were used. One of them was placed in the hole drilled into the middle of the specimen the other was placed just under the surface. The used specimen had the same size as in case of the measurement by means of the temperature scanners. It means that the same average cooling speed was measured, approximately 0,5 K/s. Of course, the cooling speed was higher at higher temperatures and at the surface of the specimen.

DIFFERENTIAL THERMAL ANALYSIS

Another used method was the differential thermal analysis (DTA). Each physical change or chemical reaction that is sufficiently strong can generate a temperature effect, called peak, on the DTA curve. From this peak it is possible, under convenient conditions, to deduce the temperature of the transformation in progression, its heat of reaction and speed of the ongoing process. DTA was carried out on the equipment Setaram SETSYS 18TM for C-Mn and C-Mn-V-Nb steels heated at the speed of 0,083 K/s to the temperature of 1 323 K with dwell of 10 minutes at this temperature, and steel C-Mn-V-Nb was heated moreover to the temperature of 1 473 K with dwell of 30 minutes at this temperature. The cooling speeds were 0,33 K/s and 0,66 K/s. An example of the processed DTA curves in the evaluation programme for steels cooled from the temperature of 1 323 K can be seen in Figure 2.

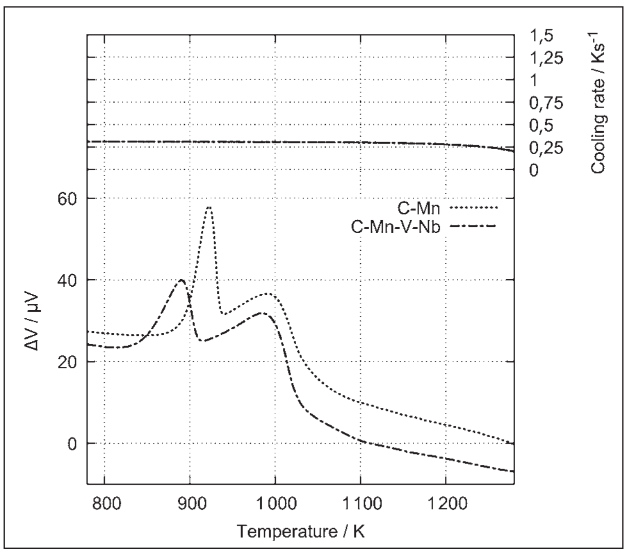

Figure 2 Results of DTA for cooling from temperature 1 323 K, cooling speed 0,33 K/s

DILATOMETRIC ANALYSIS

Measurement of phase transformations is also possible by means of dilatometer. The experiment was carried out on the dilatometer DIL 402 of the company Netzsch. Equipment is of a horizontal design; the specimen is located in the homogeneous temperature field of the furnace. The identical temperature in the whole specimen is ensured due to it and ensure the precise measurement.

The same mode as in previous methods were used. In Figure 3 the cooling curves showing the relation between the length change and temperature for both steels can be seen.

DETERMINATION OF PHASE TRANSFORMATIONS TEMPERATURES BY MEANS OF TORSION TEST

The anisothermal interrupted torsion tests, implemented on the plastometer Setaram-Vítkovice, make it possible to study the influence of the dropping temperature and the actual phase composition of the tested material on its deformation resistance.

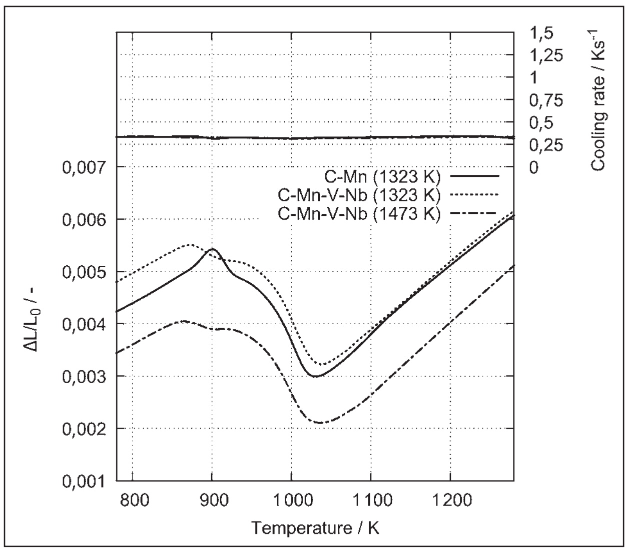

Figure 3 Temperature dependence of length change during cooling of particular steels (speed 0,32 K/s)

The specimen repeatedly undergoes specific semi-constant deformations during cooling. The mean flow stress is recalculated from the recorded torque for particular deformations and the dependence of the deformation resistance on the forming temperature can be

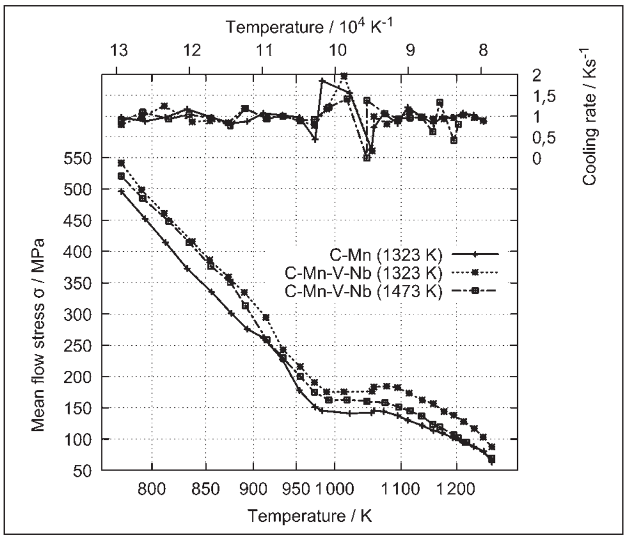

Figure 4 Temperature dependence of mean flow stress on temperature for particular steels with respecting influence of austenitizing temperature

evaluated [11-12]. The well-known fact that ferrite has lower deformation resistance than austenite on comparable conditions [13] is used for evaluation of the phase transformation temperatures in case of steels, Figure 4.

DISCUSSION OF RESULTS

Utilization of different methods for determination of the phase transformation temperatures led to plenty of results. The results for the temperature A_{r1} (final temperature of the austenite - ferrite transformation) and A_{r3} (start temperature of the austenite - ferrite transformation) are summarised in Table 2. The DTA 0,3 and DTA 0,6 represent using the cooling rates 0,33 K/s and 0,66 K/s, TC means the use of the thermocouple respectively. Line average gives a mean value of all used methods and line interval gives a difference between the highest and lowest value of the used methods.

Table 2 **Temperatures A_{r1}/ A_{r3} gained by various methods – all in K**

	C-Mn (1 323 K)	C-Mn-Nb-V (1 323 K)	C-Mn-Nb-V (1 473 K)
DTA 0,3	935/ 1 040	909/ 1 030	898/ 1 038
DTA 0,6	923/ 1022		883/ 1 039
Torsion	932/ 1056	913/ 1 056	889/ 1 056
Scanner	946/ 1043	911/ 1 033	911
TC	933/1037	903/ 1038	907
Dilatometer	921/ 1043	914/ 1048	899/ 1 053
Average	932/ 1040	912/ 1 042	898/ 1 047
Interval	25/ 34	17/ 26	28/ 18

A good accordance is found in case of the temperature A_{r1} for the steel C-Mn-Nb-V cooled from 1 323 K, and in the case of the temperature A_{r3} for the steel C-Mn-Nb-V cooled from 1 473 K, where the sub-range of the compared temperatures does not exceed 17 K and 18 K, respectively.

Interesting results were found out by evaluation of the microalloyed steel. This steel heated to 1 473 K shows lower values of the temperature A_{r1} than the steel heated to 1 323 K. Dissolving of the precipitates led to increase in the range of the two-phase structure by 19 K; this fact was confirmed by both methods – both DTA, and dilatometer [14]. The thermal analysis carried out by means of the temperature scanner was not sufficiently sensitive. Nevertheless, the thermal analysis by means of temperature scanners is still adequate for examination of phase transformations (austenite → ferrite) of common steels.

In case of DTA a very small thermal effect is observed at the temperatures from 1 100 to 1 028 K for C-Mn-V-Nb steel, cooled from the temperature of 1 323 K, this effect apparently corresponds to the gradual precipitation of the minority phase, probably V(C,N), or it could be also partly connected with a change of magnetic properties. Partial overlapping of thermal effects will occur.

A similar phenomenon may be observed also for the steel C-Mn-V-Nb at 1 101 – 1 024 K, cooled from the temperature of 1 473 K. Besides, the small pointed thermal effect is observed at the temperatures of 1 155 - 1 143 K, compared with the steel cooled from 1 323 K. The peak is observed in the austenite region, and it corresponds most likely to precipitation of Nb(C,N).

Of course, the phase transformation temperature is relatively lowered at higher cooling rates. It may be explained by the kinetics of the process and detection capabilities of the devices. Different rates of the heating and/or cooling process can also influence the kinetics of phase transformations (and/or the mechanism of the transformation). For that reason, the phase transformations taking place at different temperature change rates can be detected at various temperature intervals [15, 16].

SUMMARY

Several different methods were used for the determination of the phase transformations temperatures in the course of cooling the low carbon and microalloyed steel. It is evident from comparison of the obtained results that all methods enabled a plausible determination of these temperatures.

The thermal analysis by means of the temperature scanners appears to be very effective due to its simplicity. The measurement of surface temperatures can be negatively influenced by scaling of the specimen. The method of measurements by means of thermocouples is rather more exacting for the preparation; however, its advantage consists in a possibility of examination the temperatures inside the cooled body. These procedures, just as using the anisothermal interrupted torsion test, based on the measurement of temperature relation of deformation resistance characteristics, are not sufficiently sensitive to the kind of delicate effects that are caused e.g. by precipitation processes. For examination of these phenomena, as well as the phase transformations, the special methods with higher sensitivity – the dilatometry and above all DTA – are generally more appropriate; they are, however, much more demanding on their performance and the evaluation of the experiment.

The simple analysis of surface temperatures, using the temperature scanner, which should represent the less accurate method, due to the assumed inhomogeneity of temperature fields and scaling of specimens, gives the results comparable with the results of much more exacting specialized procedures in case of the quick determination of the phase transformations temperatures. The disadvantage of this method is lesser sensitivity to features of less pronounced structure-forming processes and a limited range of the cooling speeds that may substantially be regulated only by the weight of the measured specimen.

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Note: The responsible translator for English language is B. Škandera, Dobrá, Czech Republic.