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METHODOLOGICAL ASPECTS OF PLOTTING OF THERMOKINETIC DIAGRAMS OF TRANSFORMATION OF SUPERCOOLED AUSTENITE IN LOW-ALLOY STEELS

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Aspects of plotting of thermokinetic diagrams of transformation of supercooled austenite in low-alloy steels are analyzed critically using published data and results of own studies. Special attention is devoted to the methods of processing and visual representation of experimental data. Recommendations on representation of results for developing unified approaches to plotting and interpreting the diagrams obtained are developed.

Key words: thermokinetic diagram, supercooled austenite, low-alloy steel.

INTRODUCTION

In 1941 Soviet scientists A. V. Lopatin and A. V. Prokhorov suggested representation of the results of studies of special features of transformation of supercooled austenite in the form of thermokinetic diagrams [1]. These diagrams have turned out to be convenient enough for solving various problems of science and technology [2, 3] including the case of prescribing heat treatment modes for low-alloy steels produced at a commercial scale [4].

Progress in the outfit of research laboratories removes to a considerable degree the difficulties with obtaining enough volume of reliable experimental data but simultaneously makes it necessary to create identical conditions for the experiments and to observe the general principles of representation of results. It is said in one of the first domestic reference books presenting diagrams of decomposition of supercooled austenite plotted by A. A. Popov and L. E. Popova in 1961 [5] that "the appearance of the diagrams depends considerably on the method of their plotting and on the individual approach of various researchers to interpreting the experimental data obtained."

In foreign countries a uniform a approach to the study of transformations of supercooled austenite in low-alloy steels and to representation of the corresponding continuous cooling transformation diagrams is reflected in the ASTM A 1033 [6], which has an only recommendatory nature. In fact, this standard has been developed for unifying the test methods and forms of representation of results, which may later be used for simulating processes of heat treatment of low-alloy steels. In Russia such recommendations generalized as standard requirements are absent, which gives rise to a number practical and methodological problems concerning experiments and their interpretation.

The aim of the present work[2] consisted in developing a scientifically substantiated method for plotting thermokinetic diagrams of transformation of supercooled austenite in low-alloy steels.

METHODS OF STUDY

When a study has an aim to plot a thermokinetic diagram for a low-alloy steel, the transformation of supercooled austenite can be studied by different combinations of x-ray diffraction, thermal, magnetometric, and dilatometric analysis, measurement of hardness and electrical resistance. It is insufficient to use one of the mentioned methods, because each of them has disadvantages connected with the physical fundamentals of the method and with the techniques of its implementation.

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[2] In our work we have used results obtained at the Laboratory for Structural Methods of Analysis and Properties of Materials and Nanomaterials of the Collective Use Center of the Ural Federal University.
We do not claim to give an exhaustive consideration of the topic and will be grateful for critical comments on the material presented.

The dilatometric method of study is the most reliable and the simplest, which has been illustrated visually enough in monograph [7] in 1960. In the first turn, this is explainable by the fact that nonuniform changes in the linear sizes of specimens of low-alloy steels always accompany the phase transformation occurring in the metal upon changes in the temperature. Accordingly, the results of dilatometric tests plotted in the form of thermokinetic diagrams and supplemented with measurements of the hardness of the initial and the final microstructures and by their metallographic images give a virtually exhaustive picture of the transformations of supercooled austenite.

It is natural that when the interpretation of dilatometric curves is difficult, data on the occurring transformations can be obtained by other physical methods of research. It is interesting that the seemingly well known method of plotting thermokinetic diagrams has been called a "novel one" in a foreign publication [8].

In the case of low-alloy steels a full picture of transformations of supercooled austenite often requires implementation of relatively high cooling rates (over 10 K/sec). It seems that it is more expedient to use a simple dilatometer than a differential one due to the appearance of differences in the coolings rates of the test piece and of the standard [9].

We should mention the existence of various computational methods for plotting thermokinetic diagrams [10, 11], but they are always based on data obtained experimentally. Moreover, computational methods frequently do not allow for some factors affecting the occurrence of the transformation of austenite, for example, the size and the volume fraction of nonmetallic inclusions, the content of impurities in the metal, and its chemical inhomogeneity. In tests of a series of appropriately chosen test pieces these factors commonly affect the results identically and thus are not considered separately in further interpretation. For this very reason the computational methods of plotting of thermokinetic diagrams should be used carefully enough and only in exceptional cases.

The aspect of choosing test pieces for dilatometric research is not trivial in the study of transformation of supercooled austenite, because the test pieces should characterize accurately enough the behavior of the steel from which they are fabricated. However, any commercial steel is characterized by this or that degree of nonuniformity of the distribution of alloying elements and impurities over the volume of the article [12], which may give different data for test pieces taken from its different parts. This is especially significant for small test pieces used when it is required to implement high cooling rates.

Thus, in the preparatory stage of the study of the kinetics of the transformation of supercooled austenite in a steel independently of the chosen method of study we should be very particular about the place of withdrawal of specimens from the article, their typical microstructure, and, if possible, the kind of the distribution of the main alloying elements and impurities over the length and the cross section.

If we determine that the specimens have homogeneous and identical initial microstructures, one measurement for each cooling rate will be sufficient, but it is necessary to fix the location of the critical temperatures (in heating with the same rate) and the error of their determination. If we doubt the uniformity of the distribution of chemical elements from specimen to specimen, it is desirable to make several measurements for each cooling rate and to compute the respective errors, the values of which should be stipulated in the description of the method of the experiment.

We have studied transformations of supercooled austenite in low-carbon steels of various compositions (over 30 grades, about 50 heats) staring with 2007 using the dilatometric method and cylindrical specimens about 3.0 mm in diameter and about 10.0 mm long. The equipment was a Linseis L78 R.I.T.A. quenching dilatometer. The data were accumulated and processed using the software supplied with the facility. The block diagram of the used dilatometer is similar to that of a dilatometer with an induction sensor presented in monograph [9]. The temperature in the tests was detected with the help of a preliminarily calibrated thermocouple of type S welded to the side surface of the studied specimens.

The specimens were heated in vacuum (about 10^{-2} Pa), which totally eliminated the formation of scale on their ends and hence the distortion of the readings of the dilatometer. The coolant was helium of grade B (according to TU-51-940–80, purity 99.99%), which was fed onto the specimens at different pressures.

When performing the experiments and representing the data obtained we encountered some methodological problems most of which will be reviewed below.

RESULTS AND DISCUSSION

Aspects of Methodology of Experimental Study of the Kinetics of Transformation of Austenite

Direct cooling of specimens is always preceded by a stage of heating to a temperature from the austenitic range (or between the critical points) and a hold. The time of the hold affects the occurrence of the transformation of austenite in subsequent cooling, but to a less degree than the heating temperature [2 – 4]. Note that in heating to room temperature at a rate of about 0.2 K/sec it is permissible to do without a hold at the austenization temperature [7]; in heating at a rate of about 100 K/sec the recommended hold is 300 sec [6]. In our opinion, in the case of the use of small specimens (about 3.0 mm in diameter and about 10.0 mm high) this time of austenization is quite enough for their heating.

The choice of the heating rate in experiments and determination of the critical temperatures are a matter for discussion. It is known [2 – 4, 13, 14] that the location of the criti-

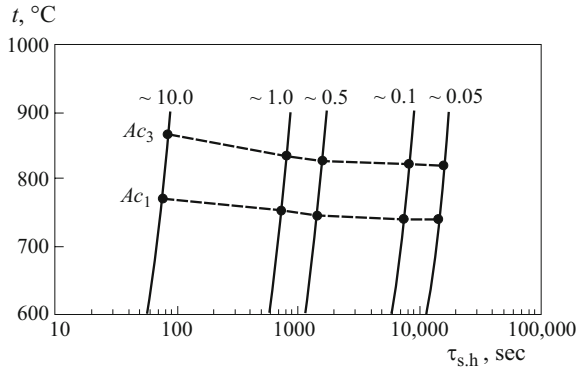

Fig. 1. Thermokinetic diagram of formation of austenite due to continuous heating (the numbers at the curves are the heating rates in K/sec) in a steel of type 26KhMFA (0.25% C, 0.85% Cr, 0.77% Mo, 0.05% V, 0.56% Mn, 0.25% Mn, 0.25% Si, 0.07% Ni, 0.01% Cu, 0.006% S, 0.009% P) ($\tau_{s.h}$ is the time from the start of the heating).

cal points changes depending on the heating rate (and on the type of the initial microstructure).

The authors of ASTM A 1033 recommend determination of critical temperatures in heating and cooling in a dilatometric study for individual series of specimens from one batch [6]. This seems to be connected with the exceptionally low recommended rate of heating of the specimens (28 K/h) starting with 700°C for determining the temperatures Ac_1 and Ac_3. Under specific conditions such rate of heating may be accompanied by inheritance of structure [15], which will affect the transformation of supercooled austenite in subsequent cooling due to the change in the size of austenite grains. Despite this fact, this approach seems to be virtually perfect from the standpoint of maximum possible closeness to isothermal conditions, at which we are supposed to determine the temperatures Ac_1 and Ac_3.

One more possible variant of heating rate in a dilatometric study is described in a necessary supplement to GOST 14080 and in a supplement to GOST 14081 and 14082 on the method of determination of the temperature coefficient of linear expansion of precision alloys. For the alloys with CLTE $\geq 3 \times 10^{-6}$ K^{-1} the rate of heating in the dynamic mode should not exceed $v_h = 200$ K/h. The recommended heating rate is $v_h = 150$ K/h, but it is permissible to perform testing at $v_h = 600$ K/h in a temperature range of 300 – 900°C. A heating rate of about 200 K/h has been implemented in the Chevenard dilatometer, which is a quite popular instrument in research laboratories.

In the actual practice, decrease in the heating rate below some value does not lead to a noticeable change in the critical temperatures of low-alloy steels (see Fig. 1 as an example). In addition, in heat treatment of actual articles the mentioned conditions (28 K/h) are attained rather rarely in heating to the temperatures of the austenitic range.

In our opinion, it is expedient to determine the critical temperatures using heating at a rate of about 0.1 K/sec from 700°C to the austenization temperature. This makes it possible to detect the transformation of supercooled austenite in cooling on the same specimens and to shorten the time of accumulation of data required for plotting a thermokinetic diagram.

One more methodological aspect for discussing is prescription of cooling modes for the studied steel, in particular, when it is necessary to choose the law for cooling the specimens, i.e., an exponential or a linear one. There is no doubt that in practical heat treatment the law of cooling of articles is an exponential one, and it seems logical to specify the same law of cooling specimens in a laboratory study. However, cooling at a linear rate has an undoubted advantage, because it is reproducible in any appropriately equipped research laboratory. If possible, a linear cooling rate should be implemented at the start of the transformation. If otherwise, we may arrive at a situation when as a result of a positive heat effect accompanying the transformation the temperature of the end of the process turns out to be higher than the temperature of its start, and it will be impossible to plot a thermokinetic diagram.

Different Approaches to Processing and Representing Data of Dilatometric Studies

There are at least three widely used methods for determining the critical temperatures from dilatometric curves. In the most accurate of them the position of the critical temperature is determined as the place of detachment of the tangent to the curve drawn to the region on which the transformation has not yet started and the specimen undergoes conventional expansion (compression) or the transformation has already finished and we observe only expansion (compression) of the newly formed phase and of the old phase, the transformation of which has stopped temporarily for some reason [7]. This method allows us to determine the moments of the very start of the transformation and the moments corresponding to its final or temporary stop.

The method for plotting thermokinetic diagrams of transformation of supercooled austenite in low-alloyed steels consists in cooling of specimens heated to the temperature of austenization and detecting the moments of the start, end, or temporary stop of formation of products of the transformation of supercooled austenite, which are then marked on the experimentally obtained cooling curves (Fig. 2). Here we arrive at the next methodological question, which is from what temperature the researcher should draw the cooling curves.

On the face of it, it seems logical to depict them right from the temperature of heating. In this case the final user of thermokinetic diagrams can immediately determine the time for which it is necessary to cool the article to some temperature of the start of the transformation in order to obtain as a result the required set of structures and the specified hardness. In addition, this technique of drawing cooling curves seems to be the only one for plotting thermokinetic diagrams

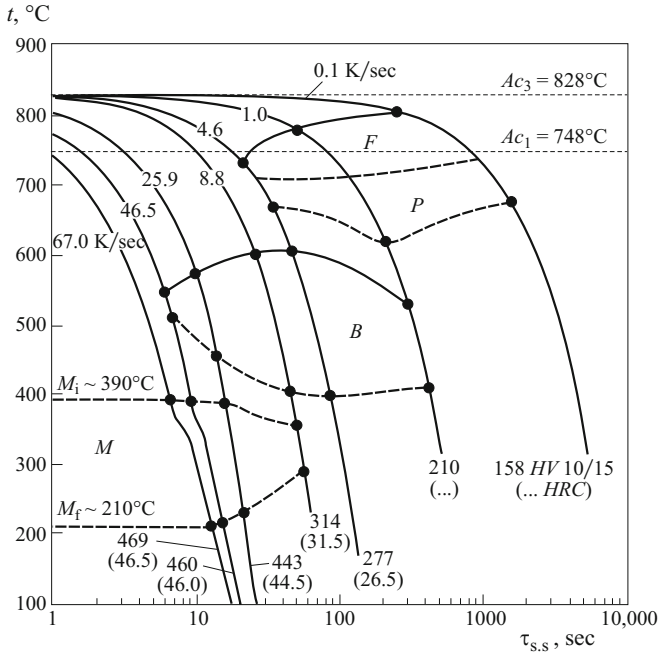

Fig. 2. Thermokinetic diagram of transformation of supercooled austenite in a steel of type 26KhMFa (0.25% C, 0.97% Cr, 0.17% Mo, 0.05% V, 0.60% Mn, 0.28% Si, 0.06% Ni, 0.01% Cu, 0.009% S, 0.005% P) ($\tau_{s.s}$ is the time from the start of supercooling).

for low-alloy steels cooled from a temperature of the two-phase region.

In another variant the cooling curves are drawn from the temperature Ac_3. The time spent for cooling the specimen from the temperature of heating to Ac_3 is subtracted from the total cooling time [6, 7]. We can give the following considerations in favor of this variant of plotting thermokinetic diagrams.

1. Above the temperature Ac_3 austenite does not transform and becomes supercooled only after some cooling below the temperature Ac_3.

2. When the temperature of austenization of a low-alloy steel is varied, a causeless shift of the thermokinetic diagram to the right or to the left, which is not connected with growth or decrease in the stability of supercooled austenite, is eliminated but rather is explained by a change in the temperature from which the curves are drawn.

In our opinion, the choice of the temperature from which to draw the cooling curves remains with the researcher, but the specimens should be cooled according to a linear law, which will make it possible to reconstruct the thermokinetic diagram to another form if necessary.

CONCLUSIONS

1. To make the use and comparison of thermokinetic diagrams plotted by various authors more convenient it is desirable to describe the used method of study and to give the grade or description of the device used for the work, to present the chemical composition of the steel and its contamination with nonmetallic inclusions according to GOST 1778, the conditions and the scheme of withdrawal of specimens and their sizes, a typical microstructure of the studied specimens, the kind of the distribution of the main alloying elements, the rate of heating of the specimens in the determination of the temperatures Ac_1 and Ac_3 and the duration of the hold at the heating temperature, and the size of the former austenite grains in accordance with GOST 5639.

2. On the very thermokinetic diagram it is desirable to mark the critical temperatures Ac_1 and Ac_3, the rates of cooling of the test pieces in the temperature range 'Ac_1 – start of the transformation,' the ranges of formation of different structures (for example, *F* – ferrite, *P* – pearlite, *B* – bainite, *M* – martensite) and, if possible, the Brinell hardness of the specimens according to GOST 2999.

3. To finish the representation of the results we may present images of typical microstructure of specimens, which have been obtained after cooling, and data on the content of the structural components.

RESUME

We treat the process of plotting and representation of thermokinetic diagrams of transformation of supercooled austenite in low-alloy steels as a complex study, the results of which in the case of their appropriate representation can be used successfully for developing the theory of phase transformations in solid state, mathematical simulation of various production processes, and solution of specific practical tasks of heat treatment of articles fabricated at commercial scale.

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