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Utilization of thermal analysis to thermo physical properties study of real steel grades

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ABSTRACT

Purpose: This paper deals with determining the temperatures of phase transformations in real steel grades. It also includes the study of industrially produced steel grades using the methods of thermal analysis by experimental equipment STA 449 F3 Jupiter made by NETZSCH and Setsys 18TM made by SETARAM.

Design/methodology/approach: Selected methods of thermal analysis (DSC and DTA) enable to obtain the temperatures of phase transformations taking place in steel during the linear heating/cooling. Within the casting technology of steel, thermal analysis is used to determine the solidus temperature and especially the crucial liquidus temperature.

Findings: Experimentally obtained solidus and liquidus temperatures are higher in the DSC method (max. 3.8°C). The difference between the temperatures of phase transformation (T) running between the solidus temperature (TS) and liquidus (TL) for both methods (DTA and DSC) differ by a maximum of 3.2°C. The results from experimental measurements were compared with theoretical calculations of liquidus and solidus temperatures by different authors and with the computed results from thermodynamic database COMPUTHERM and also with temperatures from the equilibrium phase diagram of Fe-Cr-C. Experimentally obtained solidus temperatures are lower than the calculated equilibrium solidus temperatures. Experimentally obtained liquidus temperatures are in the range of temperatures obtained using computational relations. The temperatures mentioned in the equilibrium phase diagram (diagram for a particular steel grades was not found) are higher than experimentally obtained temperatures.

Research limitations/implications: The results of experimental studies can be used to refine the knowledge of basic physical properties of steel and for example replacement of the tabulated values or estimated values of phase transformation temperatures and thermal capacity. Furthermore, the obtained data will be implemented in the material databases of numerical programs used for the simulation of metallurgical processes.

Originality/value: On the basis of applied research in close collaboration with industry companies, the obtained data can contribute significantly to optimize the operating conditions, thereby increasing the efficiency of the steelmaking technology and final quality of cast steel.

Keywords: Steel; Thermal analysis; Solidus; Liquidus; DTA; DSC

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MATERIALS

1. Introduction

Thermo-physical and thermo-dynamical properties of steels are the subject of intense research, but the experimental data of these complex systems are still inadequate. Very important data are temperatures and latent heats of phase transformations, heat capacity, and surface tension. Knowledge of solidus and liquidus temperatures of the studied steels is one of the most important factors - especially in dealing with the processes involved in the casting and solidification. The determination of temperatures of phase transformations in the multicomponent systems, such as steel, is very difficult [1-3]. Possibilities of experimental determination of temperatures of phase transformations in high-temperature region, especially above 1000°C, are very complex, and there are several methods that provide reliable results. The principles of methods are described for example in [4]. The methods are generally based on the detection of temperature changes induced by heat-coloring process or on the detection of dimensional changes of the sample. Experimentally obtained data are necessary for the thermo-dynamic calculations and can also be used as input data for a number of mathematical and physical models [5,6].

2. Methods of thermal analysis

Methods of thermal analysis enable to obtain the temperatures of phase transformations taking place in steel during the linear heating/cooling. In the steel industry, thermal analysis can also be used to determine the solidus and liquidus temperatures. During experimental measurements, attention was focused on the determination of solidus and liquidus temperatures of the selected sample of steel. To obtain the solidus and liquidus temperatures were used following dynamic methods of thermal analysis [7]:

- Differential Scanning Calorimetry (DSC),
- Differential Thermal Analysis (DTA).

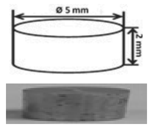
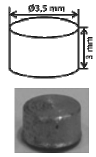
3. Experimental measurement, samples and experimental conditions

For experimental measurements of real samples were used tool steels with an approximate carbon content of 0.6 wt. %, [Cr] = 5 wt. %, and up to 2 wt. % of other alloying elements. The samples were cast on the continuous casting machine and processed to cylinders shape of appropriate size for each device and method (see Table 1). Before analysis, the samples were abraded and cleaned in acetone and by ultrasound.

DSC method is used for relatively slow heating of the sample (1 and 2 °C·min^{-1}). DTA method is then applied to heating rates of 10 and 15°C·min^{-1}. To obtain temperatures of phase transformations were used two experimental laboratory equipments:

- STA 449 F3 Jupiter Netzsch company - for the DSC method (Fig. 1),
- SETSYS 18_{TM} Setaram company - for the DTA method (Fig. 2).

Table 1.
Steel samples with dimensions specified for each analysis

Sample	NETZSCH STA 449 F3 Jupiter	SETARAM Setsys 18_{TM}
Dimensions		

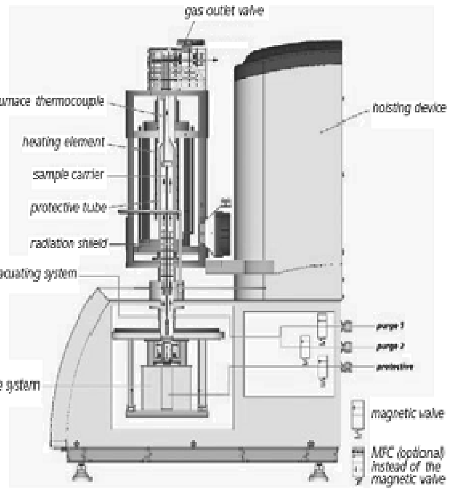


Fig. 1. NETZSCH STA 449 F3 Jupiter

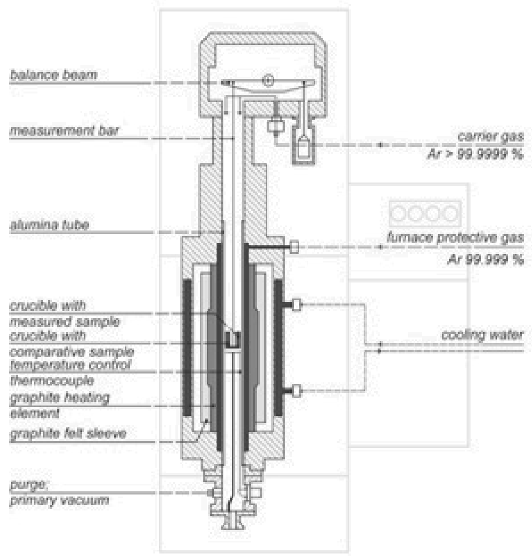


Fig. 2. SETARAM Setsys 18_{TM}

S type measuring rod for TG/DSC (thermocouple: Pt/PtRh 10%) was used to obtain of temperatures of phase transformations at the STA 449 F3 Jupiter equipment. The samples were analysed in the corundum (Al_2O_3) crucibles. Weight of analysed samples was about 300 mg. Constant dynamic atmosphere - inert argon with purity of 99.9999% - was maintained during measurement.

S type measuring rod for TG/DTA (thermocouple: Pt/PtRh 10%) was used to acquire temperatures of phase transformations with use of SETSYS 18_{TM} equipment. Samples were analysed in alumina (Al_2O_3) crucibles with capacity of 0.100 ml. Weight of analysed steel samples was approximately 200 mg. No reference sample was used during measurements. Constant dynamic atmosphere - inert argon with purity of 99.9999% - was maintained during measuring.

4. Results and discussion

Temperatures of phase transformations were obtained on the basis of DSC and DTA curves evaluation. Figure 3 shows, that in the region between the solidus and liquidus temperatures occurs further phase transformation, marked T - its nature is discussed below. Temperatures of phase transformations of steel obtained by methods of thermal analysis are listed in Table 2. Liquidus temperatures (Table 2) obtained by DSC and DTA methods were corrected with respect to the experimental conditions [8].

Table 2.
Experimentally obtained phase transformation temperature

Method	Heating rate	T_S	T	T_L
	°C·min^{-1}		°C	
DSC	1	1348.4	1441.1	1468.2
DSC	2	-	1442.3	1465.5
DTA	10	1341.0	1444.3	1464.4
DTA	15	1340.1	1442.8	1465.2

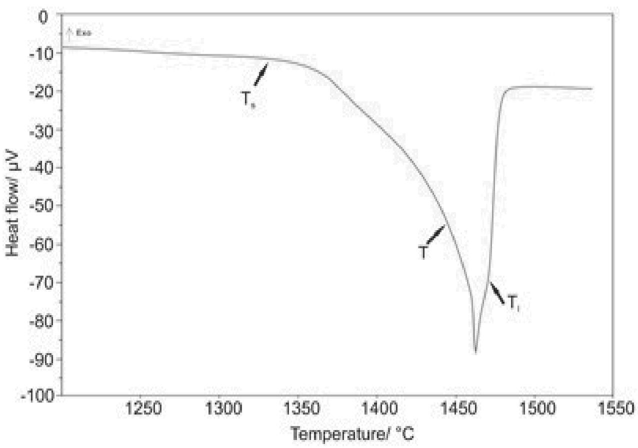


Fig. 3. DTA curve (10°C·min^{-1})

The exact determination of phase transformation temperatures is often very difficult. For this tool steel, the determination of solidus temperature was difficult. The melting of the samples was carried out initially very slowly (Fig. 3). The difference between the results of the solidus temperature (T_S) using DTA method is 0.9°C. The difference between the temperatures of phase transformation (T) running between the solidus temperature (T_S) and liquidus (T_L) for both methods (DTA and DSC) differ by a maximum of 3.2°C.

Liquidus temperature (T_L) for DSC method differs by 2.7°C. The difference between the liquidus temperature (T_L) obtained using the DTA method is only 0.8°C. At the sight of results, which are shown in Table 2, it is also evident that the temperature interval between solidus and liquidus temperatures in the range of measurements is still in the range of about 120°C to 124°C.

Theoretical calculations of liquidus and solidus temperatures by different authors and also by the thermodynamic database Computherm were carried out for comparison of results from experimental measurements, Table 3.

Calculations of liquidus and solidus temperatures according to the various authors typically don´t include all the elements detected by chemical analysis. Most of the mathematical relationships don´t include boron, oxygen, nitrogen or hydrogen. With liquidus temperature, there is the difference between the minimum and maximum temperature of 18°C. The average equilibrium (calculated) liquidus temperature is 1468.5°C.

Table 3.
Calculated temperatures of liquidus and solidus

Calculation	Sample	
	T_S	T_L
Computherm	1358	1461
Myslivec	-	1471
Šmrha [9]	-	1464
CLECIM	-	1461
TECTIP	-	1463
Dubovick	-	1476
Aymard	-	1465
Wensel	-	1479
Voest Alpine	-	1463
VSŽ Košice	-	1478
Štětina [10]	1360	1473

Experimentally obtained solidus temperatures are lower than the calculated solidus temperatures. This difference may exist due to the influence of all elements, which are not included in the calculation. Experimentally measured liquidus temperatures are in the range of temperatures obtained using computational relations.

Furthermore, the experimentally obtained temperature readings were compared with the temperatures of the equilibrium phase diagram of Fe-Cr-C (Fig. 4). Based on the analysis of Fe-Cr-C equilibrium phase diagram and DTA curves can be further assumed that in the temperature range 1440-1475°C occur to other phase transformation, which can be expressed as follows: Fe ht + L → Cr, Fe + L - in the Table 3 is the phase transformation temperature marked (T). The Fe-1.8C-Cr (Fe-0.4 wt.% C-Cr) equilibrium phase diagram [11] is shown in Figure 4. The same phase diagram has not been found in the available literature - there is no diagram corresponding to specified steel grade (for sample of a given chemical composition).

For the chromium content (5.2 at.%), which corresponds to steel sample, the following temperatures were subtracted from the equilibrium diagram: T_S = 1395°C, T = 1465°C, T_L = 1485°C. Temperatures in the diagram are higher than the experimentally obtained temperature. With regard to the chemical composition of steel sample, it must be taken into account that presented equilibrium phase diagram shows sectional view only of the ternary system Fe-C-Cr. The presence of other elements in steel (in the minimum quantities) may significantly shift the boundary of equilibrium curves in the diagram.

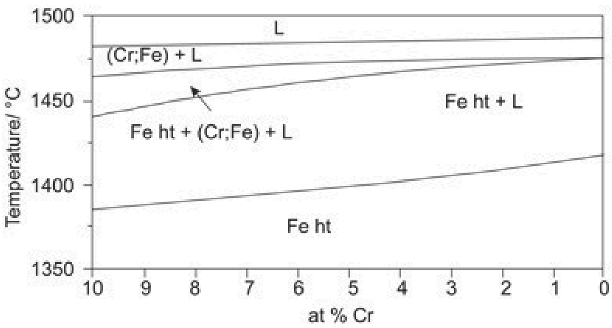


Fig. 4. Fe-1.8C-Cr equilibrium phase diagram

5. Conclusions

Using methods of DTA and DSC were obtained solidus temperature, liquidus temperature and other temperature of phase transformation between these temperatures for samples of commercially produced tool steel with an approximate carbon content of 0.6 wt.% [Cr] = 5 wt.%, and other alloying elements summarily to 2 wt.%. DSC method was used for the relatively slow heating of the sample (1 and 2°C·min^{-1}). DTA method was applied at a heating rate of 10 and 15°C·min^{-1}. Experimentally obtained solidus and liquidus temperatures (DSC method) are higher (up by 3.8°C). The difference between the temperatures of phase transformation (T) running between the solidus temperature (T_S) and liquidus (T_L) for both methods (DTA and DSC) differ by a maximum of 3.2°C.

The presented work has shown that it is still necessary to study thermophysical and thermodynamic properties of steel. Within the project RMSTC using modern equipment STA 449 F3 Jupiter NETZSCH and SETARAM Setsys 18$_{TM}$ can be thoroughly researched other key aspects of the steel production in steel mills, when arises the possibility of significant improvement of conditions for the control of metallurgical processes, remove of non-metallic inclusions, surface and internal quality of billet, reducing the number of failures of technological equipment for continuous casting and others.

The results of experimental studies can be used to refine the knowledge of basic physical properties of steel and for example to replace the tabulated values or estimated values of phase transformation temperatures and thermal capacity. Furthermore, the obtained data can be implemented in the material databases of numerical programs used for the simulation of metallurgical processes.

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