K. GRYC, B. SMETANA, M. ŽALUDOVÁ, K. MICHALEK, M. TKADLEČKOVÁ,
J. DOBROVSKÁ, L. SOCHA, L. VÁLEK, R. PACHLOPNIK

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THERMAL ANALYSIS OF HIGH TEMPERATURE PHASE TRANSFORMATIONS OF STEEL

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The series of thermal analysis measurements of high temperature phase transformations of real grain oriented electrical steel grade under conditions of two analytical devices (Netzsch STA 449 F3 Jupiter; Setaram SETSYS 18_{TM}) were carried out. Two thermo analytical methods were used (DTA and Direct thermal analysis). The different weight of samples was used (200 mg, 23 g). The stability/reproducibility of results obtained by used methodologies was verified. The liquidus and solidus temperatures for close to equilibrium conditions and during cooling (20 °C/min; 80 °C/min) were determined. It has been shown that the higher cooling rate lead to lower temperatures for start and end of solidification process of studied steel grade.

Key words: steel, thermal analysis, liquidus temperature, solidus temperature

INTRODUCTION

Steel production is one of the most important industries. However, for a steel company to succeed in tough competition, it is necessary to constantly optimize the production process itself. The optimization should lead not only to improvement in the quality of the final product, but also to increase productivity and reduce overall production costs. The precise identification of the particular physical and thermo-physical properties is the one of the possible methods of steel production optimization. Physical properties of substances in the course of reactions taking place in the operating conditions can often differ from theoretically determined (calculated/tabulated) values. In this case, it is not possible to guarantee the optimal course of the optimization process. Improper process management, in the worst case, can also lead to significant losses.

In the refining processes, optimizing the slag regimes [1], thermal and chemical homogenization of the melt [2] or filtration of steel [3] is very important to solve. Works toward optimizing the process of solidification of heavy forging ingots [4] are currently being implemented in the casting and solidification of steel studies.

The methods of study of metallurgical processes are also based on knowledge of thermodynamic properties of materials occurring in a given technology nodes. 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. These temperatures are critical parameters for proper adjustment of models (physical or numerical) or in the final stage of applied research of the real process. It is significantly affecting the final quality of the as-cast steel (billets or ingots).

Therefore, this paper is devoted to discussion of findings obtained during the utilization of dynamic thermal analysis methods [5-8] to identify the solidus and liquidus temperatures of selected steel grade. Generally, it is not so easy to identify the phase transformations occurring in such multicomponent systems like steels [9-12].

THERMAL ANALYSIS METHODS AND STEEL SAMPLES

New Laboratory for Modelling of Processes in the Liquid and Solid Phases within the project RMSTC was formed at the Faculty of Metallurgy and Materials Engineering at the VŠB-Technical University of Ostrava in Czech Republic.

This Laboratory has also acquired new equipment for high-temperature thermal analysis – Netzsch STA 449 F3 Jupiter (Figure 1).

The conditions for initiation of intensive research activities in the field of dynamic thermal analysis methods for steel are based on years of experience of team members with the issue of laboratory studies of metallurgical processes and the ability to use other equipment of this type – Setaram SETSYS 18_{TM} (Figure 2) create.

This paper discusses methods and results of thermal analysis of samples (Table 1) taken from the grain oriented electrical steel, with very low carbon content (0,04 wt. %) and high content of [Si] = 3 wt. %.

K. Gryc, B. Smetana, M. Žaludová, K. Michalek, M. Tkadlečková, J. Dobrovská, L. Socha, VŠB-TU Ostrava, Faculty of Metalurgy and Materials Engineering, Ostrava, Czech Republic
L. Válek, R. Pachlopnik, ArcelorMittal Ostrava a.s., Research, Ostrava, Czech Republic

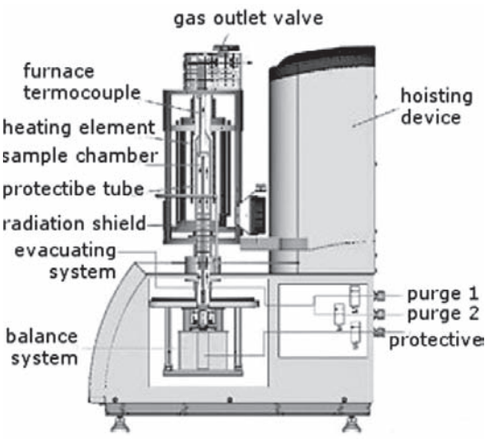


Figure 1 Netzsch STA 449 F3 Jupiter

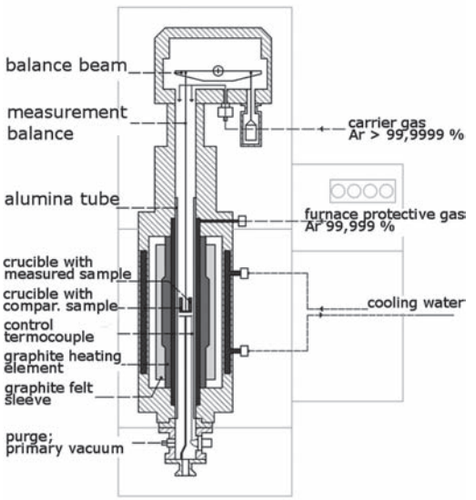


Figure 2 Setaram SETSYS 18$_{TM}$

Two methods for dynamic thermal analysis were used to measure the solidus (T_S) and liquidus (T_L) temperatures:

- Differential Thermal Analysis (DTA) – Setaram SETSYS 18$_{TM}$,
- Direct Thermal Analysis - Netzsch STA 449 F3 Jupiter.

The principles of both methods are described for example in [13].

RESULTS AND DISCUSSION

Both above mentioned dynamic thermal analysis methods were used to determine the T_S and T_L close to equilibrium in the frame of studied grain oriented electrical steel grade.

Three direct thermal analysis was realized on the big steel samples (about 23 g) and the T_S and T_L were obtained by analysing of the heating curves (heating rate 30 °C/min). The four measurements for close to equilibrium T_S and T_L temperatures were also realized by differential thermal analysis on small steel samples (about 200 mg) during their heating by heating rate 10 °C/min). Liquidus temperatures were then corrected according to generally accepted methods [14]. Measured T_S and T_L are summarized in the Table 2.

Based on data in the Table 2, it can be stated that there is low variability between individual results for close to equilibrium temperatures (standard deviations: 2,5 °C for T_S and 1,6 °C for T_L) independently on used method and mass of samples. It means that methodology of measurement is set correctly and results are fully reproducible. Based on mean values, the T_S and T_L were for selected steel grades were identified: 1 479 °C and 1 496 °C.

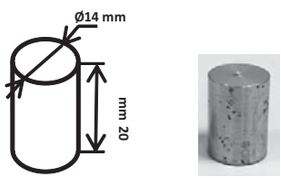
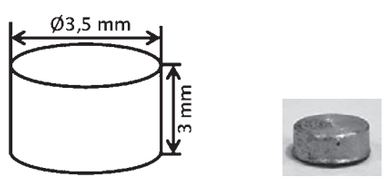
Table 2 **Experimental settings and measured T_S and T_L for close to equilibrium conditions**

Method, Measurement No.	Sample mass / mg	Heating rate / °C/min	T_S / °C	T_L / °C
Direct thermal, 1	22 913,3	30	1 476,0	1 496,7
Direct thermal, 2	23 691,2	30	1 475,3	1 497,0
Direct thermal, 3	23 539,4	30	1 477,8	1 493,4
DTA, 1	198,3	10	1 480,8	1 495,5
DTA, 2	206,4	10	1 481,1	1 495,9
DTA, 3	205,6	10	1 481,0	1 498,5
DTA, 4	207,9	10	1 480,5	1 496,4

From the viewpoint of technology of continuous casting of studied steel grade, it should be useful to know the phase transformation temperatures during solidification process taking place under non equilibrium conditions – intensively cooled bloom caster and secondary cooling zone. The solidification of continuously cast steel starts and ends under such non equilibrium conditions.

Thus, based on requirements of industrial partner, the T_S and T_L during cooling were studied. Differential thermal analysis on small steel samples was used (cooling rates 20 and 80 °C/min) – Table 3.

Table 1 **Steel samples with dimensions specified for each analysis**

Sample for method:	NETZSCH STA 449 F3 Jupiter	SETARAM Setsys 18$_{TM}$
Dimensions:		

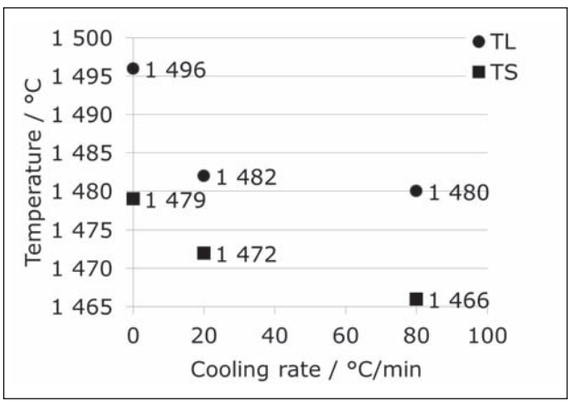


Figure 3 Results of realised thermal analysis measurements for close to equilibrium temperatures and for non-equilibrium conditions – cooling

Table 3 shows that the final values of T_S and T_L for twice repeated measurements for the same cooling rate are comparable. So, the results are fully reproducible.

Table 3 **Experimental settings and measured T_S and T_L for non-equilibrium conditions (cooling)**

Method, Measurement No.	Sample mass / mg	Cooling rate / °C/min	T_S / °C	T_L / °C
DTA, 1	198,3	20	1 472,0	1 480,8
DTA, 2	206,4	20	1 471,6	1 483,8
DTA, mean 1, 2	**---**	**20**	**1 472**	**1 482**
DTA, 3	205,6	80	1 465,0	1 480,3
DTA, 4	207,9	80	1 466,4	---*
DTA, mean 3, 4	**---**	**80**	**1 466**	**1 480**

* Unable to identify from cooling curve.

Figure 3 summarizes all determined T_S and T_L for studied steel.

The start of solidification is very similar for both cooling rates and such T_L are lower than T_L for close to equilibrium conditions. But, non-equilibrium conditions during cooling down the steel sample led to significant differences between the slower and faster cooling rates. Faster cooling down of the steel during its solidification leads to the lower T_S.

It can be seen (Figure 3) the decreasing the T_L and T_S for increasing cooling rate against the close to equilibrium conditions. These differences could be critical for correct setting of superheat of selected steel grade before its continuous casting.

CONCLUSION

The series of thermal analysis measurements of high temperature phase transformations of real grain oriented electrical steel grade under conditions of two analytical devices (Netzsch STA 449 F3 Jupiter; Setaram SETSYS 18_{TM}) were carried out. Two thermo analytical methods were used (DTA and Direct thermal analysis). The crucial temperatures (liquidus and solidus) for solidification process under different heating/cooling conditions were studied.

It was shown that methodologies of both used methods are set correctly: results are reproducible and comparable for both used analytical devices for identification of T_L and T_S close to equilibrium. These temperatures were determined for studied steel grade:

T_S = 1 479 °C;

T_L = 1 496 °C.

Focused on industrial conditions – continuous casting of steel, it could be useful for optimizing the superheat to identify the start and end of solidification process under different cooling rates. The DTA results for these non-equilibrium liquidus and solidus temperatures determination were used. These temperatures differ against equilibrium values.

Cooling rate 20 °C/min:

T_L = 1 482 °C;

T_S = 1 472 °C.

Cooling rate 80 °C/min:

T_L = 1 480 °C;

T_S = 1 466 °C.

Thermal analysis is very useful method for determination of high temperatures phase transformations in steel.

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