DETERMINATION OF SOLIDUS AND LIQUIDUS TEMPERATURES IN THE LOW CARBON STEEL USING THREE DEVICES FOR HIGH-TEMPERATURE THERMAL ANALYSIS AND SPECIALIZED PROGRAMS

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Abstract

The paper is devoted to a discussion of the results obtained by the simultaneous use of three methods of thermal analysis in three different specialized devices with the aim to precise solidus and liquidus temperatures. The low carbon as-cast steel (130 mm round format) from Continuous Casting Machine No. 3 in ArcelorMittal Ostrava a.s. (CCM No. 3) was studied. The series of experiments with large samples (approx. 23 g) based on direct thermal analysis was carried out on Netzsch STA 449 F3 Jupiter device. The differential scanning calorimetry (DSC) method with special 3D sensor for other samples (approx. 1 g) was applied on Setaram MHTC 96 device. The third device (Setaram SETSYS 18$_{TM}$) was used for solidus and liquidus temperatures determination by differential thermal analysis method on small steel samples (approx. 150 mg). Thermo analytically determined solidus and liquidus temperatures were then compared with values obtained by calculations (specialized Thermocalc, IDS and CompuTherm software) and with liquidus temperature currently defined in CCM No. 3 practice for continuous casting of discussed steel grade.

Keywords: steel, round billet, solidus temperature, liquidus temperature, thermal analysis, software

1. INTRODUCTION

Optimization of any crucial parameters of steelmaking technology should leads to significant lowering of production cost and to better quality of steel products. It is necessary to succeed in tough competition.

In the refining processes, optimizing the slag regimes [1, 2], thermal and chemical homogenization of the melt [3] or filtration of steel [4, 5] is very important to solve. Works toward optimizing the process of solidification of heavy forging ingots [6, 7] were implemented in the casting and solidification of steel. Finally, an attention is focused on physical and numerical modelling of steel flow in the tundish [8, 9], the computer support of mould diagnostics [10], and on utilisation of wastes from steel industry [11].

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 [12,13] 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 [14,15].

2. THE METHODOLOGY AND EXPERIMENTAL CONDITIONS

As described in previous papers [16], the Laboratory for Modelling of Processes in the Liquid and Solid Phases is equipped with three modern devices for thermal analysis. The knowledge of real plant conditions is necessary for proper selection of material for sample preparing. In the frame of solved project (TA03011277) that is devoted to optimising the casting conditions for specific steel grades casted into round billets, close cooperation between university's and steel plant's specialists is very important.

So, the cross section positions for samples taken from round billets (**Fig.1**) (d=130 mm) are drawn in **Fig.2**.

Fig. 1 Round billets (d=130 mm) taken from CCM. No. 3 for sampling

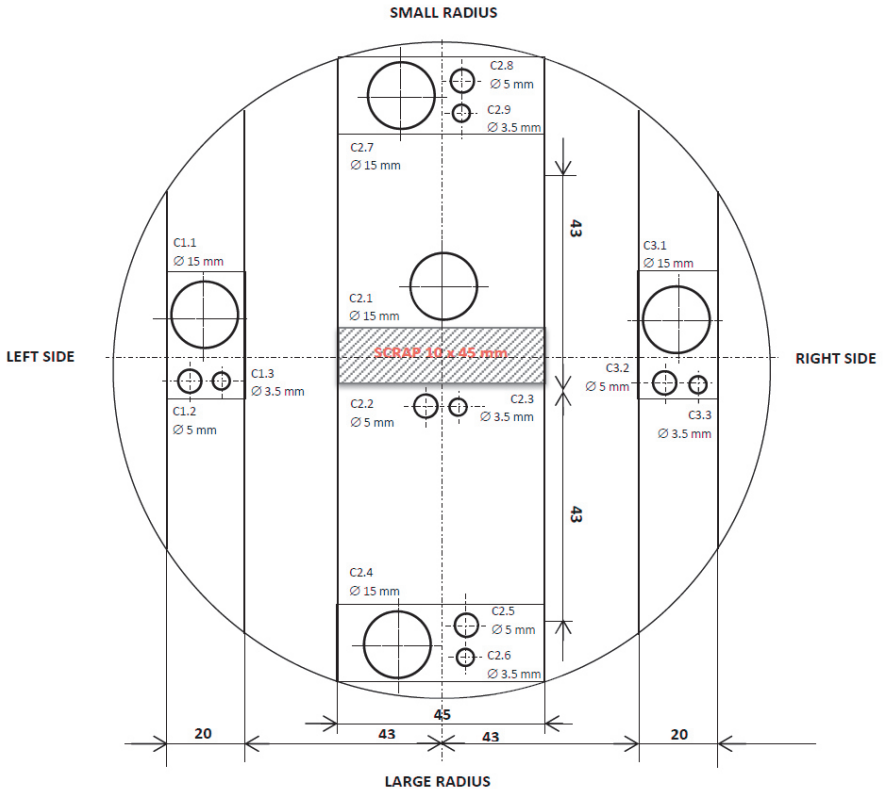

Fig. 2 Positions of steel samples for thermal analysis experiments

Fig. 3 Sticks with different diameter for all three thermo analytical methods

Final sticks (**Fig.3**) delivered from steel plant were then cut to final length of samples for individual devices and thermo analytical experiments.

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

- Direct Thermal Analysis - Netzsch STA 449 F3 Jupiter.
- Differential Thermal Analysis (DTA) - Setaram SETSYS 18^{TM},
- Differential Scanning Calorimetry (DSC) - Setaram MHTC.

The principles of these methods are described for example in [17].

In the frame of solved project, round billets from several heats of different steel grades was taken and prepared by above described way to these sticks and finally cut to an individual dimensions for different types of thermal analysis and used Al_2O_3 crucibles.

Above mentioned dynamic thermal analysis methods were used to determine the T_S and T_L under different heating/cooling conditions and also for experiments focused on latent heat and/or heat capacity determination. Data obtained from thermo analytical measurements are then compared with theoretical calculations and used for setting of numerical simulations of steel solidification and/or implemented into steel making practice.

3. RESULTS AND DISCUSSION

This paper is focused on discussion of results for one selected low carbon steel grade with approximately 0.08 wt.% C and 0.6 wt.% Mn.. Aim was to compare:

- Results for samples from different positions in cross section of billet (**Fig.2**).
- Results for studied steel grade obtained from different thermo analytical methods.
- Results of thermal analysis method with computed data and T_L value from control system operating in real plant conditions.

3.1 Direct thermal analysis

From the viewpoint of real conditions, it is necessary to take into account the steel as very heterogeneous material. So, the most experiments was realised on big samples (approx. 24 g) taken from different cross section billet's positions. Results are summarized in **Table 1**.

It is evident that values of TS are in narrow interval from 1478 to 1483 °C without any significant dependence on position in cross section of continuously cast billet. Only very slow growth of TS in the direction from the left to the right side of billet cross section is visible from individual Mean Values. Final TS (Direct TA/Mean Value) is 1480 °C.

Generally, measured TL are also in narrow interval from 1521 to 1523 °C. But, there are two TL values (1516 °C - Left Side/No. 2; 1525 °C - Small Radius/No. 1). These differences could be connected with heterogeneity of studied steel grade in the as-cast state.

Simultaneously with thermal analysis, the micro cleanliness analysis also for here discussed steel grade was realised. An attention was focused on comparison of oxide and sulphide non-metallic inclusion distribution in the different position of samples in cross section of billet [18]. Results showed very good micro cleanliness in whole billet cross section. No significant differences were found. On the left side was a little higher content of sulphide based non-metallic inclusions and a little lower content of oxide based non-metallic inclusions. Next, an analysis devoted to study of segregation processes is in progress now.

Table 1 Experimental temperatures of solidus T_S and liquidus T_L from direct thermal analysis

Method	Position/Sample No.	Heating Rate; °C.min^{-1}	T_S ; °C	T_L ; °C
Direct Thermal Analysis	Left Side/No. 1	5	1479	1521
	Left Side/No. 2	5	1478	1516
	Left Side/No. 3	5	1480	1522
	Left Side/Mean Value	**x**	**1479**	**1519**
	Billet Centre/No. 1	5	1479	1522
	Billet Centre/No. 2	5	1481	1522
	Billet Centre/No. 3	5	1481	1521
	Billet Centre/Mean Value	**x**	**1480**	**1522**
	Right Side/No. 1	5	1482	1522
	Right Side/No. 2	5	1483	1523
	Right Side/Mean Value	**x**	**1482**	**1522**
	Small Radius/No. 1	5	1479	1525
	Direct TA/Mean Value	**x**	**1480**	**1521**

3.2 Different thermal analysis and Differential Scanning Calorimetry

Currently, a DTA method is applied during studying the phase transformation of various kinds of material, including steel and other alloys, resp. metals. Small steel samples (approx. 173 g) where analysed by DTA method. Third thermo analytical method - DSC - was used on bigger steel samples (approx. 1.2 g). Very sensitive 3D DSC sensor was used. Results from both mentioned methods are summarised in **Table 2**.

Table 2 Experimental temperatures of solidus T_S and liquidus T_L from DTA and DSC

Method	Position/Sample No.	Heating Rate; °C.min^{-1}	T_S ; °C	T_L ; °C
DTA	Left Side/No. 1	10	1493	1523
	Billet Centre/No. 1	10	1491	1522
	Billet Centre/No. 2	10	1494	1524
	Billet Centre/No. 3	10	1492	1524
	Billet Centre/Mean Value	**x**	**1492**	**1523**
	Right Side/No. 1	10	1491	1523
	DTA/Mean Value	**x**	**1492**	**1523**
DSC	Left Side/No. 1	5	1487	1523
	Left Side/No. 2	5	1486	1522
	Left Side/Mean Value	**x**	**1487**	**1523**
	Billet Centre/No. 1	5	1486	1523
	Billet Centre/No. 2	5	1486	1522
	Billet Centre/Mean Value	**x**	**1486**	**1523**
	DSC/Mean Value	**x**	**1486**	**1523**

Table 2 shows that T_S and T_L determined based on DTA method are very similar. Difference between maximum and minimum T_S/T_L values is only 3 °C/2 °C and it corresponds. From this viewpoint, the difference 1 °C for T_S and T_L determination based on applied DSC method is marginal. So, it is not possible to discuss the dependence of T_S/T_L on cross section position in billet when DTA and/or DSC method is used.

3.3 Comparison of experimentally determined T_S and T_L temperatures with calculated values

Above, results of thermo analytical methods were discussed individually. **Table 3** summarise experimentally obtained T_S/T_L Mean values from specified methods and values calculated by specialized programs (Thermocalc, IDS, Computherm). Finally, the T_L value defined in control system of CCM No. 3 for discussed steel grade is stated too.

Table 3 Comparison of experimental T_S and T_L with calculated values

Method	T_S ; °C	T_L ; °C
Direct TA/Mean Value	1480	1521
DTA/Mean Value	1492	1523
DSC/Mean Value	1486	1523
Thermocalc[1]	1480	1525
IDS[2]	1485	1525
CompuTherm[3]	1490	1527
AMO[4]	X	1521

[1] SW Thermocalc ver. 3.1, database TCFE7.
[2] SW Solidification analysis package, not included following elements: V, Ti, B, Nb, Sn, Al, N, O.
[3] SW Computherm, not included following elements: B, Sn, Al, N, O.
[4] ArcelorMittal Ostrava a.s. calculation.

Solidus temperatures determined by different methods drawn in **Table 3** show very interesting agreement between individual results from thermal analysis methods and computed values. For example, T_S from direct thermal analysis fits to T_S value computed by Thermocalc. Focused on T_L, it can be stated that calculated results correspond to each other. Calculated T_L values are up to 6 °C higher than measured ones. T_L obtained from thermal analysis are closer to the T_L value that is set for selected steel grade in the real plant condition as a crucial parameter for continuous casting operations.

CONCLUSIONS

Presented paper was devoted to the determination of solidus (T_S) and liquidus (T_L) temperatures in the low carbon steel. Three different methods of thermal analysis were employed (Direct thermal analysis, DTA, DSC). Based on chemical composition of this steel grade, the calculations of T_S and T_L were realised by three specialised software (Thermocalc, IDS, CompuTherm). Results were discussed. Finally, experimentally and thermodynamically obtained T_L results were compared with T_L value currently used in real steel plant conditions as one of the most crucial parameter for setting of continuous casting process.

The difference between T_S and T_L values obtained from direct thermal analysis of selected steel grade samples taken from different round billet cross section positions show any small differences. So, the influence of possible heterogeneity in round billet cross section has marginal impact on T_S and T_L in the case of this steel grade and billet cross section dimension. But, such influence cannot be generally excluded and should be taken into account in the future works. The cleanliness and segregation in round billets are simultaneously studied in the frame of this project.

The differences of T_S and T_L determined by different thermo analytical methods are not significant. Generally, obtained T_S interval is wider. While, values of T_L are almost the same with marginal variability. Therefore, all three applied methodologies of thermal analysis are set correctly. Thermo-analytically determined T_S and T_L are fully reproducible and can be considered as real values for concretely studied steel grade.

The three computation methods for T_S and T_L were employed. The results of such T_S calculations are in thermo analytically measured interval. The calculated T_L values are higher up to 6 °C against measured ones.

For direct real plant utilisation, it is very important to compare obtained results for T_L (thermo-analytically determined and calculated) with T_L value that is currently used in plant practice for selected steel grade. It is necessary to be sure that any recommendations for real casting conditions are based on correctly obtained data. Only based on all above described results and realised comparison, it is possible to continue in discussion on possible changes of T_L setting during continuous casting of steel.

In the case of this steel grade, T_L used in steel plant is quite the same as measured. So, the discussion on optimising the continuous casting process can be moved almost to the other crucial parameters, e.g.: casting speed, primary and secondary cooling etc. Therefore, our team is employing determined T_L and T_S values and other experimentally obtained thermo physical parameters into numerical modelling of studied continuous casting process.

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