

COMPARISON OF LIQUIDUS AND SOLIDUS TEMPERATURES OF STEEL CAST INTO INGOTS IDENTIFIED BY DIFFERENT THERMO-ANALYTICAL METHODS

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Abstract

In the framework of optimising, correct setting of the casting process, knowledge of solidus temperature (Ts) and especially liquidus temperature (T_L) of produced steel grade is necessary. There are options to determine these temperatures: the use of empirical equations or calculations by specialised programs (thermodynamic databases). These calculations are based on the data of steel chemical composition and final temperature of phase transformation is then its reflection. T_s and T_L values then may not completely correspond to reality as well. It is very appropriate to employ a combination of different methods of thermal analysis and theoretical prediction for confrontation of experimental and calculated TL and Ts. Three modern devices for high temperature thermal analysis and two specialised programs of theoretical prediction are available at the Faculty of Metallurgy and Materials Engineering. Direct thermal analysis method (dirTA) and parallel currently mass-enhanced method of thermal analysis, differential thermal analysis (DTA), were used for determination of T_L and T_S of studied real steel grades. Simultaneous application of both methods allows to reduce significantly disadvantages of each method and recommend proper TL and Ts into the casting process and real conditions of industrial partner. Paper is focused on the discussion of T_L and T_S of steels cast into ingots (9 melts; 7 steel grades) analysed in the frame of a project TA0410035. Submitted evaluation refers an importance of the parallel utilization of different methods, their accuracy and reproducibility and also the divergences between T_L and T_S experimentally determined, empirically calculated and predicted by thermodynamic SWs' calculations.

Keywords: Steel, solidus temperature, liquidus temperature, thermal analysis, thermodynamic calculations

1. INTRODUCTION

Constantly increasing and strict requirements on quality of ingots and cast steel require a comprehensive approach to solve whole process of steelmaking. To ensure the cleanliness and micro-purity of steel [1, 2], the correct adjustment of the slag regime [3 - 5] during the secondary steel refining process plays a significant role. The assessment of strength characteristics is important for assuring the high quality of steel [6, 7]. A numerical simulation also has considerable importance in steelmaking process. The implementation of simulation results [8 - 11] can significantly affects the quality of produced steel. Thermodynamic properties of materials, especially for casting and solidification of the steel, the solidus (T_s) and liquidus (T_L) temperature are among the most crucial parameters [11, 12]. Precise knowledge of T_L is particularly important in relation to the superheat setting of steel before its casting. T_s is related with the solidification process and range of two-phase region between T_L and T_s, which is affected by segregation phenomena [13 - 17]. Knowledge of



these critical temperatures is necessary not only for the correct setting of the technology of steel casting and proper solidification of steel, but also for precise setting of simulation conditions of steel solidification.

This paper follows the previous research in the field of thermal analysis in the frame of the project TA0410035. Now, it summarise the results obtained for seven steel grades cast into ingots. The significance of parallel utilization of both presented methods of thermal analysis for an industrial practice of steel casting is evident.

2. METHODS USED FOR THE IDENTIFICATION OF SOLIDUS AND LIQUIDUS TEMPERATURES

Currently, it is possible to utilise a several dozens of thermo-analytical methods. Three of them are the most popular. In the field of thermal analysis, in the range of one half to three quarters of all professional works, these methods are employed: Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC) and Thermogravimetry (TG) [18-25]. Simultaneous combinations as TG / DTA and TG / DSC are often applied. In the past, Direct Thermal Analysis (direct measurement of the temperature change of the studied sample esp. under the linear cooling conditions) was also widely used [26]. This method is still applicable for measuring T_L and T_S of metallic materials.

Generally, thermal analysis (TA) [27 - 30] allows to monitor the changes in the study material by measuring selected physical properties in dependence on time or temperature (phase transformations, heat capacity etc.). TA methods are predominantly dynamic processes and allow to obtain information about the status change of the sample. These processes require a non-isothermal temperature regime (usually linear heating or cooling of the sample). Changes of the studied material either directly by measuring the selected physical properties or indirectly by measuring of the properties at the surrounding of the sample are determined.

A combination of two thermo-analytical methods (TA methods) - *Direct Thermal Analysis (DirTA)* and *Differential Thermal Analysis (DTA)* on two different professional systems for different sample mass (approx. 22 g, resp. 120-210 mg)) for study of T_L and T_S temperatures of investigated steels were used. Results from both TA methods (T_L and T_S temperatures) and the empirical calculations and predictions by modern commercially SWs for phase transformation temperatures determining are also compared. A combination of all approaches ensures the achievement of the maximum possible correctness of the results.

 T_L and T_S temperatures under the linear heating / cooling conditions, and also under cyclic experiments (2 heating and cooling cycles, under the same conditions) by DirTA were acquired. Only under the linear heating conditions, T_L and T_S temperatures by DTA were determined. Experimental results (T_L and T_S from DirTA and DTA) with empirically calculated T_L and T_S temperatures (by empirical equations of industrial partner VÍTKOVICE HEAVY MACHINERY a.s. (VHM)) were compared. T_S calculation in VHM is considered to be unreliable and in principle not applied. Experimental values were also predicted by modern commercial SWs IDS (Solidification Analysis Package), Thermo-Calc (database TCFE7) by own calculations (with regard to chemical composition available).

3. RESULTS AND DISCUSSION

Completely 9 melts of 7 steel grades are discussed. For two steel grades, the samples of two melts with a different chemical composition, but within the tolerance of the individual steel grade specifications were analysed. T_S and T_L temperatures were experimentally determined for each steel grade by both methods of thermal analysis (DirTA and DTA). The aim of the paper is not a detail analysis of the results for individual steel grade, but to achieve a comprehensive view on the results obtained not only by DirTA and DTA thermo-analytical methods, but also based on their confrontation with T_L , resp. T_S , predicted by empirical equations used in conditions of industrial partner (VHM) and with two thermodynamic professional SWs (IDS, Thermo-Calc). Therefore, T_S temperature by DTA under the conditions of linear cooling weren't identified. However, due to the higher sensitivity of the sensor, T_S temperature for all investigated steels by DTA, were also determined.



3.1. The analysis of the liquidus temperature

Experimentally and empirically determined T_L for analysed steel grades are summarized in **Table 1**. Experimentally captured T_L temperatures in the average form and as corrected values of the experimental conditions obtained from two correctly performed analyses are presented. Standard deviations (SD) for each average T_L temperature were also calculated to verify the reproducibility of the results.

Steel grade	DTA		dirTA						
	Heating	SD	Heating	SD	Cooling	SD	VHM	IDS	T-Calc
	1440	3			1441	3	1469	1451	1453
A	1444	0	1447	0	1433	5	1463	1448	1451
В	1465	0	1479	0	1468	1	1493	1474	1482
	1474	1	1480	0	1476	0	1491	1475	1480
С	1464	2	1473	1	1462	2	1485	1476	1477
D	1486	2	1486	1	1484	1	1496	1487	1488
E	1470	1	1475	1	1471	1	1488	1470	1475
F	1483	0	1487	0	1482	0	1496	1486	1487
G	1498	0	1501	1	1495	1	1506	1500	1504

Table 1 Liquidus temperatures obtained by different methods; (°C)

For the first investigated steel grade the results under the conditions of linear heating couldn't be identified by dirTA. High degree of reproducibility of the results achieved by both methods of TA due to very low values of standard deviations (SD) under the linear heating conditions is demonstrated. Under the linear heating conditions standard deviations are calculated in the range from 0 to 2 °C. The highest value of the standard deviation (3 °C) for the first steel grade was registered. The maximum standard deviation under the linear cooling conditions for the second steel grade is achieved. To obtain one final T_L temperature which should be recommended as optimal T_L temperature for the adjustment of steel casting technology the following approach has been chosen:

- 1) Experimental results with a high degree of reproducibility are more accurate than empirically calculated.
- 2) To avoid a threat (based on recommended temperatures by TA) of steel casting process, the highest TL temperature from the three TL experimentally determined (TA) temperatures (2x linear heating, 1x linear cooling) is recommended for each melt (grey mark and bolt font in **Table 1**).

Values got by dirTA method are selected for a final recommendation of T_L temperature from linear heating conditions for almost of all studied steel grades (also evident in **Table 1**). Values obtained under the linear cooling condition only for the first steel grade are recommended. T_L temperatures acquired under the conditions of linear heating were difficult to measure. The final T_L temperatures recommended for B, E, F steel grades also correspond to Thermo-Calc. For steel grade D the same results were achieved by DTA and DirTA. T_L divergences obtained by different methods of determination against recommended final T_L are shown in **Table 2**.

Within the various applications of TA methods for studied steel grades, the divergences of determined T_L temperature exceed -5 °C or even -10 °C (**Table 2**). Considering the above presented methodology of recommendation of final T_L temperature, these divergences are negative. Currently in real conditions (VHM), the empirically calculated T_L for most studied steel grades is higher by tens of degrees Celsius and ranges from 5 to 28 °C. T_L values predicted by IDS are in some cases higher (up to 10 °C), in other are lower (max.



to -5 °C). In three cases, the identical T_L temperatures as the recommended T_L by Thermo-Calc were predicted. For other steel grades, a positive divergence (2 - 4 °C) was found. The first steel grade has a divergence 12 °C.

Stool grade	DTA	dir	ТА	VHM	IDS	T-Calc	
Steel grade	Heating	Heating	Cooling		103	I-Calc	
0	-1		0	28	10	12	
A	-3	0	-14	16	1	4	
В	-14	0	-11	14	-5	3	
	-6	0	-4	11	-5	0	
С	-9	0	-11	12	3	4	
D	0	0	-2	10	1	2	
E	-5	0	-4	13	-5	0	
F	-4	0	-5	9	-1	0	
G	-3	0	-6	5	-1	3	

Table 2 Divergences from the final recommended T_L obtained by different methods; (°C)

3.2. The analysis of the solidus temperature

Solidus temperature appears to be less significant than T_L in the operational conditions of steelmaking. However, its importance can't be ignored, especially in the case of casting of heavy ingots. The main reason is that in the two-phase region between T_L and T_S temperatures, the conditions supporting a number of processes with a negative impact on the quality of cast ingot exist. Correct setting of T_S and T_L temperatures can significantly affects the results of numerical simulations whose are necessary to recommended interventions to optimise the casting process. T_S temperatures are summarized in **Table 3**.

Steel grade	DTA		dirTA						
	Heating	SD	Heating	SD	Cooling	SD	VHM	IDS	T-Calc
	1280	10					1125	1319	1318
A	1307	0	1308	12	1412	5	1097	1302	1309
	1365	8	1385	4	1385	8	1354	1382	1404
В	1397	2	1405	6	1410	13	1354	1386	1395
С	1362	1	1393	3	1417	18	1282	1381	1381
D	1432	2	1436	4	1437	6	1358	1417	
E	1374	1	1389	5	1394	7	1334	1361	1311
F	1421	1	1412	1	1435	5	1316	1423	1413
G	1445	2	1447	2	1437	12	1390	1438	1451

Table 3 Solidus temperatures obtained by different methods; (°C)

It wasn't possible to determine T_s temperature for the first steel grade by DirTA. It wasn't possible to calculate T_s temperature for steel grade D by Thermo-Calc. Standard deviations of T_s temperature identification by TA methods are already higher, although for most steel grades analysed under the conditions of linear heating by DTA, standard deviations only from 0 to 2 °C have been observed. Due to the generally problematical determination of T_s temperature, standard deviations can be considered as satisfactory. Higher differences in



standard deviations can be related with slightly different character of the solidification process of individual steel samples.

As the final recommended T_s (from TA analysis) for practical use in conditions of industrial partner, the lowest values (grey mark and bolt font in **Table 3**) in accordance with the above approach were selected. Most of them under the linear heating conditions by DTA have been achieved. Exceptions are steel grades F and G for whose T_s temperatures obtained by DirTA method as final were recommended. The values of D and F steel grades are marked in italic form in **Table 3** due to both T_L and T_s temperatures for the same thermal analysis method and same conditions (linear heating) were selected as industrially applicable. More complicated situation of final T_s determination is evidently based on the fact that the empirical equations (VHM) nor SWs predictions (IDS, Thermo-Calc) didn't fit the final recommended T_s temperatures. Divergences from finally recommended T_s temperatures are shown in **Table 4**.

Chaol grada	DTA	dir	TA		IDC	T-Calc	
Steel grade	Heating	Heating	Cooling	VHM	IDS	I-Calc	
А	0			-155	39	38	
A	0	1	105	-210	-5	2	
В	0	20	20	-11	17	39	
D	0	8	13	-43	-11	-2	
С	0	31	55	-80	19	19	
D	0	4	5	-74	-15		
E	0	15	20	-40	-13	-63	
F	9	0	23	-96	11	1	
G	8	10	0	-47	1	14	

Table 4 Divergences from the final recommended T_S obtained by different methods; (°C)

Furthermore, it is also possible to observe that divergences of T_S temperature obtained by different methods from the final recommended values are more pronounced than in the case of T_L identification (usually in the order of tens of degrees of Celsius for individual melts). However, there are steel grades where these differences are less than 10 °C and where the T_S obtained by different methods from the final recommended value differ by the hundreds of degrees of Celsius. Due to the procedure of recommendation of final T_S temperatures - recommended T_S values are always higher than other thermal analysis methods' results.

Utilisation of the empirical equations applied in conditions of industrial partner always lead to lower T_s values. With the use of thermodynamic SW, some calculated values were higher, other lower than the final recommended T_s obtained based on thermal analysis methods.

3.3. Analysis of two-phase region between T_L and T_S

To illustrate an importance of determination of T_s was further proceeded to compare the temperature interval between the two-phase region between T_L and T_s (**Table 5**).

Values of the temperature interval from T_L to T_S recommended by the thermal analysis in column TA in **Table 5** can be registered. Temperature intervals achieved by each other type of prediction (VHM, IDS, Thermo-Calc) in the next three columns are demonstrated. The last three columns show values of the differences between the values of Thermal Analysis (TA) and values of the intervals obtained by the theoretical calculations / predictions.



Conclusively, differences in the prediction of the two-phase temperature interval for individual steel grades within the individual prediction method exist. The range of the temperature interval of two-phase region identified by the TA methods, as well as SW predicted values, is in the order of tens or hundreds of degrees of Celsius. The temperature intervals of two-phase region obtained by empirical prediction (VHM) for the individual steel grades are always in the order of hundreds of degrees of Celsius.

		VHM	IDS		Divergences from TA values			
Steel grade	TA			T-Calc	VHM	IDS	T-Calc	
	161	344	132	135	183	-29	-26	
A	140	366	146	142	226	6	2	
	114	139	92	78	25	-22	-36	
В	83	137	89	85	54	6	2	
С	111	203	95	96	92	-16	-15	
D	54	138	70		84	16		
E	101	154	109	164	53	8	63	
F	75	180	63	74	105	-12	-1	
G	64	116	62	53	52	-2	-11	

Table 5 Range of two-phase temperature intervals for individual steel grades by the use of different methods of their identification; (°C)

When comparing the temperature intervals of the range of the two-phase region, determined by the empirically calculations, it is possible to find close temperature intervals and conversely wider temperature intervals than were experimentally determined by TA methods (**Table 5**). These differences are again the most significant in the case of empirical calculations (VHM), where the temperature interval between T_L and T_S is always estimated to be wider than measured one. Range of two-phase regions by both SWs predicted were in some cases close in other ones they were wider than determined by TA methods.

4. CONCLUSION

The paper presented results of determination of liquidus and solidus temperatures for various steel grades cast into ingots by different methods. Two methods of thermal analysis were employed. On Netzsch STA 449 F3 Jupiter experimental system, method of direct thermal analysis (dirTA) was applied. Steel samples by Differential Thermal Analysis (DTA) were analysed by Setaram SETSYS 18_{TM} experimental system. Results of thermal analysis methods with the predictions of the solidus and liquidus temperatures calculated by empirical equations supplied by industrial partner (VMH) and with the results of calculations with sophisticated programs Thermo-Calc IDS were compared.

Acquired knowledge can be summarised as follows:

- Because the real analysis performed using standardized methods on real samples, not by means of empirical calculations, T_L and T_S temperatures obtained by thermal analysis methods were selected for further evaluation. In particular, T_L temperatures show a high degree of reproducibility of results.
- It is obvious, that the parallel application of different methods of thermal analysis and different mass of the steel samples makes it possible to compare the results of these analyses and then recommend the more critical and proper T_L and T_S values.
- 3) Divergences between T_L temperatures for individual steel grades across the different used methods of prediction as were compared were generally less problematic that T_S determination. However, these divergences can't be generally considered insignificant even in the case of T_L.



4) Temperature ranges from T_L to T_S in most cases show significant differences depending on the choice of the used method of prediction. Here, the impact of choosing the final recommended T_L and T_S is the most significant.

These findings indicate that the problematics of verification of T_L and T_S temperatures requires a comprehensive approach using multiple methods of solution. More significant differences against calculations can be expected especially for special steels grades with a high content of carbon or alloying elements. Finally, the operational experiments in real plant conditions should be proceed to adjustment of the casting technology to gain savings not only in the field of superheat temperature of steel before casting.

Furthermore, it is appropriate to implement measured results into numerical simulations focused on the optimization of steel casting technology and solidification of the steel. It should lead to more accurate results corresponding to real conditions.

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