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PREDICTION AND MEASUREMENT OF SELECTED PHASE TRANSFORMATION TEMPERATURES OF STEELS

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Abstract

The study deals with precise determination of phase transformation temperatures of steel. A series of experimental measurements were carried out by Differential Thermal Analysis (DTA) and Direct Thermal Analysis (TA) to obtain temperatures very close to the equilibrium temperatures. There are presented results from the high temperatures region, above 1000 °C, with focus on the solidus temperatures (T_S), peritectic transition (T_P) and liquidus (T_L) of multicomponent steels. The data obtained were verified by statistical evaluation and compared with computational thermodynamic and empirical calculations. The calculations were performed using 15 empirical equations obtained by literature research (10 for T_L and 5 for T_S), as well as by software InterDendritic Solidification (IDS) and Thermo-Calc (2015b, TCFE8; TC). It was verified that both thermo-analytical methods used are set correctly; the results are reproducible, comparable and close to equilibrium state.

Keywords: *Steel; DTA; Solidus; Liquidus; Thermo-Calc; IDS*

1. Introduction

Knowledge of phase transformation temperatures is important for understanding of the fundamental properties of steels. Increasing interest in phase transformations observed in steels is clearly evident by the growth in the number of publications, in which data, relevant to phase transformations, are systematically reported and assessed [1-5].

Modern economical production and processing of steel needs accurate phase transformation temperatures data to keep better control of the production metallurgy, especially the primary and secondary metallurgy processes, optimal setting of casting and solidification conditions, thermal and chemical homogenization of the melt [6]. Also, the phase transformation temperatures of steels are critical parameters for proper adjustment of other mathematical models (physical or numerical) that often uses such an input data for e.g. modelling of continuous casting [7].

Phase transformation temperatures may be investigated by several approaches [8], but among the most reliable techniques currently available involves thermal analysis [9]. However, there are many factors affecting the accuracy of the thermo-analytical measurement, e.g. procedure or method bias (different analytical procedures give different results), instrumental influences (detection limits of temperature sensors), sampling and sample preparation (size of the samples and temperature fields in sample, inhomogeneous chemical composition of the sample), environmental influences, experimental parameters (sample mass, sample geometry, heating and cooling rate, atmosphere, temperature range, crucible), evaluation methodology, etc. [10].

Another method for determination of phase transformation temperatures is calculation using one of so called “empirical equations”, available in the literature. The equations are based on extensive study of correlation between chemical composition and its impact on phase transformation temperatures. Although the most advanced approach used today are based on computational thermodynamic. Various computer software use e.g. CALPHAD [11] or PHASE FIELD [13] methods for calculating, among others, the phase transformation temperatures [12].

Dedicated to the memory of Professor Dragana Živković

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The methods are using databases with the stored assessed information and calculation results are dependent on correct thermodynamic data in the databases. However, low computing costs, accuracy and overall versatility of modern modelling software makes it more competitive to the experimental methods [14].

Even with the available information in current computational methods, requirements for accurate phase transformation temperatures data as well as insufficient experimental data in some systems, makes it still necessary to make further experimental investigation of phase transformation temperatures [15].

The aim of the paper was to obtain original accurate phase transformation temperatures: solidus temperatures (T_S), temperatures of peritectic transition (T_P) and temperatures of liquidus (T_L). The composition of the two steel grades evaluated, is shown in Table 1. The data were obtained by experimental measurement and calculations, and assessed in term of reproducibility, and comparability. Specifically, for calculations were used software Thermo-Calc [16] and IDS [17] and selected Empirical equations. The calculated results are compared with experimental measurements performed by differential thermal analysis (DTA) and direct thermal analysis (TA) [18, 19].

2. Experiment

Two medium-carbon steel samples were prepared from real steel castings. The composition of the samples is shown in Table 1. Steel 1 is standard construction steel grade. Steel 2 is tool steel, with considerably higher purity (except phosphorus) and higher alloying element content (e.g. Cr, Mo, Ni …).

The samples were machined to a desired shape for each equipment and method, then polished and cleaned by ultrasound in acetone. The mass of sample was 23 - 25 g for TA and approximately 200 mg for DTA.

Two thermal analysis methods were used for experimental investigation. The first method was Differential Thermal Analysis (DTA). The temperature of the test material is measured relative to that of an adjacent inert material. A thermocouple imbedded in the sample and empty corundum crucible are connected so that any differential temperatures generated during the heating cycle are graphically recorded as a series of peaks on a moving chart. The temperature of the analysed sample is measured relative to a reference sample. The result of the measurement is DTA curve.

The second method Direct Thermal Analysis (TA) uses one thermocouple inserted in the crucible with a sample. The result of the measurement is either a Heating curve (TAH) or a Cooling curve (TAC). The evaluation of the DTA and TA curves was carried out by the tangent interception method. Description of the equipment and adjustment of experimental conditions is described, e.g. in [20, 21].

- Setaram SETSYS 18 – DTA sensor (S-type tri-couple), (DTA);
- Netzsch STA 449 F3 Jupiter sensor (S-type, mono-couple), (TA).

The experiments were performed for high temperature region, this means temperature region above 1000°C. The experiments were performed in corundum crucibles in inert atmosphere of argon with purity higher than 99.9999 mol. %. Such high purity gas is accomplished by using Getter-gas purifier (MicroTorr Canister Purifier MC200).Heating rates were 10 $°C.min^{-1}$ (DTA) and 5 $°C.min^{-1}$ (TA). Measured temperatures were corrected on melting temperature of pure palladium (99.999 wt. %), on melting temperature of pure nickel (99.999 wt. %), on influence of the heating rate and on influence of the sample mass [22].

3. Calculations

The calculations were performed using two software. The first software Thermo-Calc (TC) is a flexible software, for all kinds of phase equilibrium, phase diagram and phase transformation calculations and thermodynamic assessments. TC software uses the CALPHAD approach. Thermo-Calc calculations were performed with TC v. 2015b, using TCFE8 database. TCFE covers the assessments of many important binary and ternary systems, as well as the iron-rich corner of some higher order systems,

Table 1. *Composition of steels [wt. %]*

Sample	C	Mn	Si	P	S	Cu	Ni	Cr	Al	$O_{soluble}$
Steel 1	0.368	0.758	0.278	0.009	0.0020	0.060	0.034	1.087	0.017	0.0017
Steel 2	0.380	0.400	0.940	0.010	0.0012	0.110	0.304	5.004	0.022	0.0003

Sample	N	Mo	V	Ti	Nb	Ca	Sn	As	Sb
Steel 1	0.0057	0.233	0.004	0.001	0.001	0.0016	0.005	0.003	0.0060
Steel 2	0.0100	1.152	0.404	0.002	0.005	0.0011	0.012	0.011	0.0024

within the 28-element framework. It can be used with satisfactory results for a range of different alloy types: e.g. stainless steels, tool steels, cast iron, etc. [16].

The second software InterDendritic Solidification (IDS) is thermodynamic–kinetic–empirical tool for simulation of solidification phenomena of steels. The software is based on PHASE FIELD method. IDS is valid for simulation of the solidification of low-alloyed steels and stainless steels. The model applies a thermodynamic substitutional solution model and a magnetic ordering model and Fick's diffusion laws to determine the stable solution phases, liquid, delta ferrite, eutectic ferrite and austenite, and their fractions and compositions as a function of temperature [17].

For Thermo-Calc calculations all phases were allowed for calculations. All elements shown in Table 1 were considered for the calculations, only Sn, As, Sb were excluded, because it's not defined in the databases. These three elements, in such low amount, have insignificant impact on the calculation results. Oxygen was also excluded from calculations. With oxygen included, the calculation became unstable. The software always either crashed or finished calculation when the temperature reached about 1300 °C. The issue is being further investigated.

The Empirical equations shown in Table 2 are based on the effect of contents of selected elements (weight % of elements) in the steel on liquidus and solidus temperatures. It can be seen from Table 2 equations, that there are different elements taken into account for the various calculations. Some equations include only effects of a few elements with main impact on the final phase transformation temperature, some equations include a wide variety of elements. For comparison, equation 6 has 21 elements included in the formula while equation 10 has only 5 elements. This can in some cases lead to the significantly higher deviation of calculated values of solidus and liquidus temperatures [15]. Calculation of peritectic transition temperature was not conducted by Empiric equations due to extensive complexity of the problem that exceeds the range of the paper [23, 24].

Table 2. *The equations for calculation of liquidus and solidus temperatures based on the effect of contents of selected elements [wt. %] in steel*

Liquidus temperatures [°C]		
No.	**Ref.**	**Empirical Equation**
1	[25]	$T_L = 1537 - 88(\%C) - 25(\%S) - 5(\%Cu) - 8(\%Si) - 5(\%Mn) - 2 (\%Mo) - 4(\%Ni) - 1.5(\%Cr) - 18(\%Ti) - 2(\%V) - 30(\%P)$
2	[26]	$T_L = 1536 - \{90(\%C) + 6,2(\%Si) + 1.7(\%Mn) + 28(\%P) + 40(\%S) + 2.6(\%Cu) + 2.9(\%Ni) + 1.8(\%Cr) + 5.1(\%Al)\}$
3	[27]	$T_L = 1536 - 8(\%C) - 7.6(\%Si) - 3.9(\%Mn) - 33.4(\%P) - 38(\%S) - 3.7(\%Cu) - 3.1(\%Ni) - 1.3(\%Cr) - 3.6(\%Al) - 0(\%Sn)$
4	[28]	$T_L = 1809 - 78(\%C) - 7.6(\%Si) - 4.9(\%Mn) - 34.4(\%P) - 38(\%S) - 4.7(\%Cu)$
5	[29]	$T_L = 1809 - \{100.3(\%C) - 22.4(\%C)^2 - 0.16 +13.55(\%Si) -0.64(\%Si)^2 + 5.82(\%Mn) + 0.3(\%Mn)^2 + 4.2(\%Cu)\} - 273.15$
6	[30]	$T_L = 1811 - 72.6(\%C) - 7.1(\%Si) - 5.7(\%Mn) - 24.4(\%P) - 33.4(\%S) - 2.1(\%Cr) - 2.8(\%V) - 6.2(\%Al) - 8.4(\%Cu) - 4.3(\%Ni) - 2.8(\%Co) - 8.6(\%Nb) - 4.2(\%Ta) - 8.7(\%Ti) - 9(\%As) - 1(\%W) - 6.9(\%Sn) - 54.7(\%N) - 0.1(\%Mo) - 55.3(\%O) - 89(\%B)$
7	[31]	$T_L = 1537 - 73.1(\%C) - 2.5(\%Al) - 1.5(\%Cr) - 4.0(\%Mn) - 5(\%Mo)- 3.5(\%Ni) - 30(\%P) - 14(\%Si) - 45(\%S) - 4(\%V)$
8	[32]	$T_L = 1538-\{31.15(\%C)^2 + 62.645(\%C) + 0.609(\%Si)^2 + 2.0678(\%Si-0.0674(\%Mn)^2 + 5.3464(\%Mn)+20(\%P)^2 + 9(\%P) - 1.7724(\%S)^2 + 24.775(\%S) + 1.1159(\%Nb)^2 + 5.3326(\%Nb) - 0.0758(\%Ca)^2 + 3.1313(\%Ca) + 0.0379(\%Ni)^2 + 5.2917(\%Ni) + 0.6818(\%Cu)^2 + 2.5955(\%Cu) + 0.0214(\%Mo)^2 + 3.2214(Mo) + 0.0359(\%Cr)^2 + 1.1402(\%Cr) + 10.797\}$
9	[33]	$T_L = 1536 - 78(\%C) + 7.6(\%Si) + 4.9(\%Mn) + 34(\%P) + 30(\%S) + 5(\%Cu) + 3.1(\%Ni) + 1.3(\%Cr) + 3.6(\%Al) + 2(\%Mo) + 2(\%V) + 18(\%Ti)$
10	[34]	$T_L = 1537 - 78(\%C) - 7.6\%(Si) - 4.9(\%Mn) - 34.4(\%P) - 38(\%S)$

Table 2 is continued on the next page.

Table 2 continues from the previous page

Solidus temperatures [°C]		
No.	**Ref.**	**Empirical Equation**
11	[25]	T_S = 1535 - 200(%C) - 12.3(%Si) - 6.8(%Mn) - 124.5(%P) - 183.9(%S) - 4.3(%Ni) - 1.4(%Cr) - 4.1(%Al)
12	[26]	T_S = 1536 - {415.3(%C) + 12.3(%Si) + 6.8(%Mn) + 124.5(%P) + 183.9(%P) + 4.3(%Ni) + 1.4(%Al) + 4.1(%Al)}
13	[27]	T_S = 1536 - 251(%C) - 12.3(%Si) - 6.8(%Mn) - 123.4(%P) - 183.9(%S) - 3.3(%Ni) - 1.4(%Cr) - 3.1(%Cu) - 3.6(%Al)
14	[28]	T_S = 1809 - 175(%C) - 20.5(%Si) - 6.5(%Mn) - 500(%P) - 700(%S) - 273.15
15	[29]	T_S = 1809 - {415.5(%C) + 12.3(%Si) + 6.8(%Mn) + 124.5(%P) + 183.9(%S) + 4.1(%Al)} - 273.15

4. Results and discussion

Based on DTA and TA analysis measurements, the following temperatures of phase transition temperatures were determined: temperature of solidus (T_S), temperature of peritectic transition (T_P) and temperature of liquidus (T_L). DTA analysis was performed with three pieces of samples. Evaluated was DTA curve during heating only. Typical DTA curve is shown at Figure 1. The TA analysis was performed on two pieces of samples. Results of TA analysis were discussed separately for heating curves (TAH, Figure 2) and cooling curves (TAC, Figure 3), due to, in some cases, substantial differences of results.

The difference is caused mainly due to the arrangement of the equipment, sample mass and sensitivity of the used sensors. The TA heating and cooling curve is also affected by inhomogeneous temperature field, release and absorption of latent heat during ongoing phase transition, possible decarburisation, and contact of sample with sensor or crucible. Furthermore, the evaluation of obtained curves can be difficult in cases, where heat effects overlap or there isn't sharp deviation from the base line. Also faster cooling because of a smaller sample could alter the solidification behaviour of the iron, affecting the undercooling.

The results of selected empiric equations for T_S and T_L are shown in Table 3. The summary of thermal analysis results and software calculations are shows Table 4. Statistic evaluation of obtained experimental results was performed by mean value, standard deviation and variation coefficient expressed in %. All measurements, in general, show high level of consistency and low level of variability. The standard deviation of the results does not exceed 9 degrees of Celsius and variation coefficient does not exceed 0.62 % (Table 4).

It can be seen that some variability between obtained phase transformation temperatures exists. The values obtained by thermal analysis measurements were determined based on standardized methodology. For this paper, it can be stated that measured results are the most accurate to the real phase transformation temperatures of studied steels. Therefore to determine final phase transformation temperatures, the mean values were

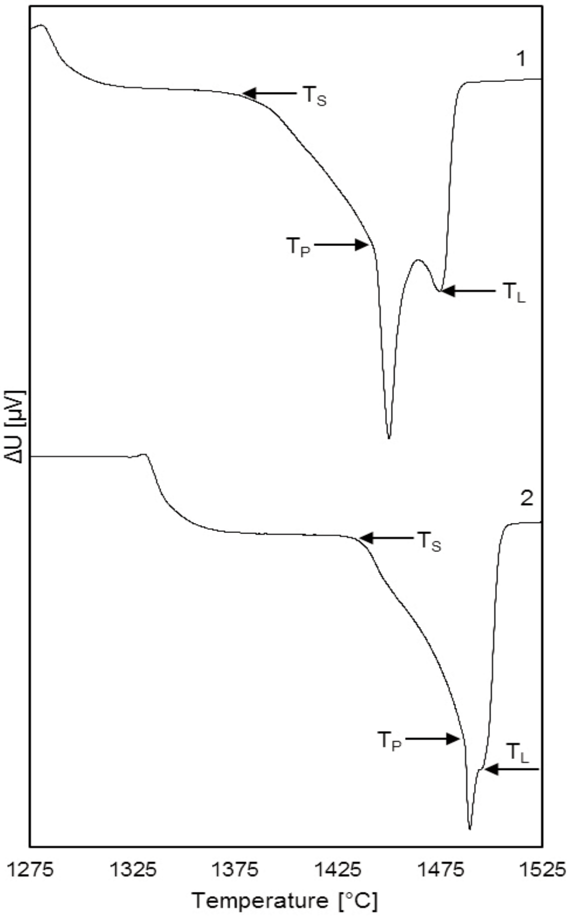

Figure 1. *DTA curves, heating, steel 1 and steel 2*

calculated from DTA, TAH and TAC results. This mean values of thermal analysis results are determined as final valid real phase transformation temperatures.

The difference of measured values and software calculations covered a fairly wide interval 0 - 17 °C. Very good compliance (±5 °C) was reached for both software in case of steel 1 and the T_P of steel 2. The TC provided, for steel 2, T_S and T_L results with difference ±17 °C. Also, the software differed from each other, 3 cases were even beyond range ±10 °C. To summarize the above: Compared to the measured values, the theoretical calculations by IDS software provided much more consistent results. Thermo-Calc software and IDS software are providing, in some cases, relatively good calculation results, and its reliable tool for verification of measured data, but it is always vital to check the calculated data with an experiment.

4.1 Solidus temperature

Determination of the start of melting by both methods are, in this case of steel grade, very depended on deflection of the base line. Therefore it was difficult to determine the initiation of melting. In case of cooling, the temperature of solidus was strongly dependant on the creation of secondary (austenite) phase, and temperature of solidus can be more affected by nucleation process. The temperatures of solidus, obtained by DTA and TA methods, shows in case of steel 1 good agreement, in case of steel 2

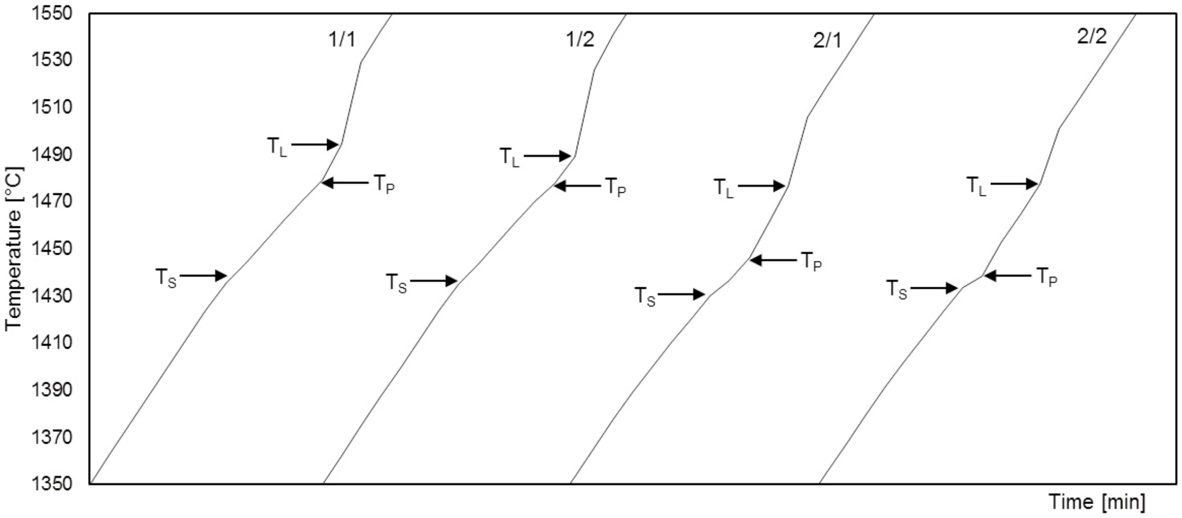

Figure 2. *TA; Heating curves (TAH); steel 1 and steel 2 / 1st and 2nd cycle*

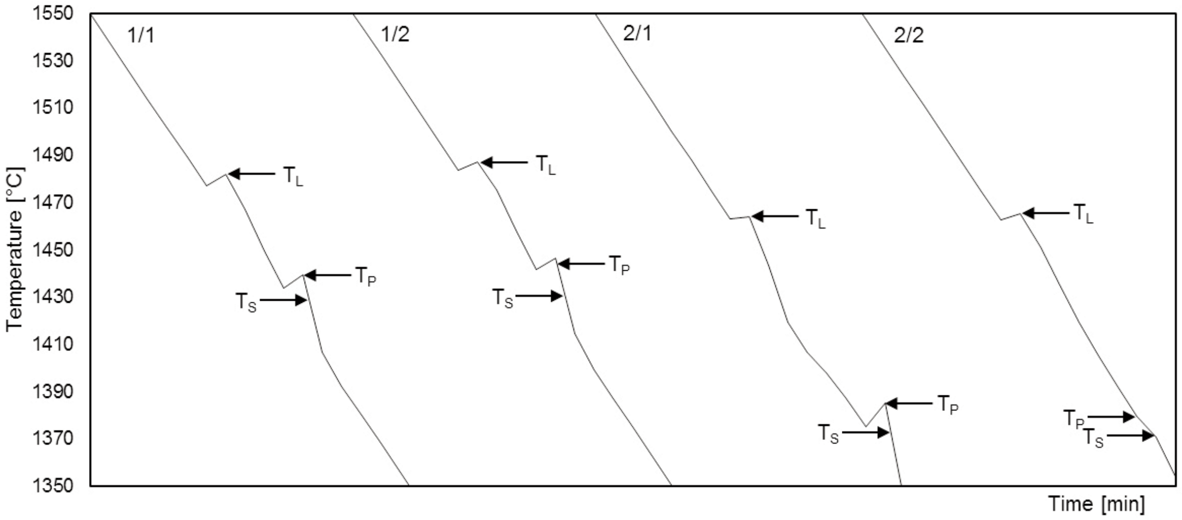

Figure 3. *TA; Cooling curves (TAC); steel 1 and steel 2 / 1st and 2nd cycle*

relatively high standard deviation 4 – 9 °C. Moreover, there is 3 °C difference between TC and IDS for steel 1 software calculation, while 16 °C for steel 2 calculation. However, compared to final results both software are providing solid results, except for significant 17 °C difference between final result and TC in case of steel 2. The statistics and software calculations suggests, that the values are reliable, although the DTA and TAC measurement, in case of steel 2, might be less accurate.

The selected empiric equations shows high deviation from measured values, range of solidus temperatures lies for steel 1 within 1371 – 1455 °C interval. Similarly, the steel 2 range 1354 - 1442 °C. This makes difference 84 °C or 88°C respectively. Standard deviation of empiric equations is 37 °C for both steel grades. However for steel 1, the equation 11 and in particular 13 seems to be possibly suitable for calculations. The selected empiric equations for solidus calculations, presented in this paper, are not suitable for precise determination of solidus temperatures and should be used only for approximate estimation of T_S. The determined solidus temperatures for steel 1: T_S = 1435 °C and for steel 2: T_S = 1385 °C.

Table 3. *Results of empiric equations calculations [°C]*

Equation	Steel 1		Steel 2	
T_L/T_S	T_S	T_L	T_S	T_L
1	1450	1496	1435	1435
2	1371	1497	1354	1485
3	1432	1525	1416	1515
4	1455	1501	1442	1496
5	1373	1494	1362	1486
6		1500		1348
7		1500		1478
8		1493		1482
9		1516		1527
10		1502		1498
Mean value	1432	1500	1416	1485
St. Deviation	37	10	37	48
Var. Coef. [%]	2.57	0.65	2.59	3.25

4.2 Peritectic transition temperature

The measured peritectic transformation temperatures between DTA and TAH shows 10 °C difference for steel 1 and 8 °C difference for steel 2. This values are in relatively good agreement and is consistent with deviations of data published in [24]. The TAC values are considerably deflected to the rest of results, although the standard deviation was only 6 °C or 8 °C respectively. The difference from DTA is more than 34°C or 58 °C respectively for both steels, and from TAH 24 °C or 50 °C respectively. It needs to be emphasized, that the both steels software calculations are within ±3 °C range from these results.

The literature [35, 36] suggests that nucleation in cooling regime is energetically more demanding rather than during heating. The issue of nucleation of primary solid phase exists in this case from melt to delta ferrite. This can lead to minor distortion of results. The TAC results present in this paper shows high deviation and are not reproducible. Therefore it is not recommended to use the results of peritectic transition temperatures gained from TAC. The determined peritectic transformation temperatures, in this case excluding the incorrect TAC results, are for steel 1: T_P = 1484 °C and for steel 2: T_P = 1444 °C.

Table 4. *Results of software calculations and measurement [°C]*

Analysis	Statistics	Steel 1			Steel 2		
		T_S	T_P	T_L	T_S	T_P	T_L
IDS		1437	1484	1497	1386	1441	1474
Thermo-Calc		1440	1486	1487	1402	1443	1451
DTA	Mean Value	1431	1489	1492	1365	1448	1465
	St. Deviation	1	0	1	9	1	0
	Var. Coef. [%]	0.09	0.00	0.08	0.62	0.07	0.03
TA Heating	Mean Value	1435	1479	1493	1385	1440	1474
	St. Deviation	2	0	0	4	1	1
	Var. Coef. [%]	0.11	0.00	0.03	0.29	0.03	0.03
TA Cooling	Mean Value	1439	1455	1487	1385	1390	1468
	St. Deviation	5	6	0	8	8	1
	Var. Coef. [%]	0.33	0.42	0.03	0.58	0.55	0.05
Determined temperature		1435	1484	1492	1385	1444	1468

4.3 Liquidus temperature

The determination of liquidus temperature was considerably less complicated than T_S or T_P in previous sections. The results of measurement are reliable, with low standard deviation and variation coefficient. The difference between DTA and TA is within ±5 °C for steel 1 and ±9 °C respectively. The steel 1 T_L result is consistent with the results published in work [37], where steel with carbon content 0.35 wt. % provided T_L = 1494 °C and paper [20], where low carbon steel also with 0.35 wt. % of carbon provided T_L = 1499 °C. As for steel 2, the paper [37] presents similar alloyed steel with 0.69 wt. % of carbon and T_L = 1471 °C.

The TC and IDS software in case of steel 1 showed 10 °C difference from each other, while relatively high difference 23 °C in case of steel 2. However software calculations compared to measurement shows relatively narrow temperature interval, where boundary values are given by both software (the measured values are within this interval). The agreement between measured values and software calculations is good. The determined liquidus temperatures are for steel 1: T_L = 1492 °C and for steel 2: T_L = 1468 °C.

The empiric calculations are providing, compared to solidus temperatures, seemingly more consistent results shown in Table 3, but consistency is only achieved for steel 1. Also, it needs to be pointed out that, compared to measured values, the equations 3 and 9 shows for steel 1 relatively high deviation and when excluded, the standard deviation of rest of the empiric equations is 3 °C with variation coefficient 0.22.

The steel 2 shows 5 times higher variability of empiric equation calculations than steel 1 with standard deviation 48°C. In this case, the equation 6 needs to be excluded due to more than 100 °C deviation of liquidus temperature from the measured values. However even after this step the standard deviation is 24 °C and variation coefficient 1.65. In general, no empiric equation listed in this work can be recommended for calculation of liquidus temperature of steel 2.

The cause of significantly greater problems with theoretical calculations for steel 2 lies probably in higher content of alloying elements. These are causing problems mainly for calculations by empiric equations. It is too difficult to characterize all the issues of alloying elements influence on phase transformation temperatures by one simple equation. Therefore, as well as the T_S values, it is recommended to use empiric equations only for approximate determination of T_L or T_S respectively.

5. Conclusions

The aim of the paper was to obtain key phase transformation temperatures from two real steel grades: temperature of solidus (T_S), temperature of peritectic transition (T_P) and temperature of liquidus (T_L).

	Steel 1	Steel 2
T_S	1435	1385
T_P	1484	1444
T_L	1492	1468

The results (temperatures T_S, T_P and T_L) were obtained by DTA and TA method, refined, compared and verified with theoretical calculations performed using TC and IDS software and selected empiric equations.

Compared to the measured values, the theoretical calculations by IDS software provided much more consistent results. Thermo-Calc software and IDS software are providing, in some cases, relatively good calculation results, and its reliable tool for verification of measured data, but it is always vital to check the calculated data with an experiment.

It is too difficult to characterize all the issues of alloying elements influence on phase transformation temperatures by one simple equation. Therefore it is recommended to use empiric equations only for approximate determination of T_L or T_S respectively.

All experimental values, in general, show high level of consistency and low level of variability. It was shown that both thermo-analytical methods used are set correctly; the results are reproducible, comparable and close to equilibrium. Obtained experimental temperatures by the thermal analysis can be used to optimize production and processing of analysed steel grades.

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