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Recenzované výzkumné články

Analysis of Phase Transformation Temperatures of Real Steel Grades

Analýza teplot fázových transformací reálných značek ocelí

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The paper deals with the study of phase transformation temperatures of two real grade steels. A series of thermal analysis measurements by Differential Thermal Analysis (DTA) and Direct Thermal Analysis (TA) were performed on two real grade medium carbon steel samples in low and high temperature regions. The eutectoid transition temperature (T_E), end of α-ferrite to γ-austenite transition temperature ($T_{\alpha\rightarrow\gamma}$), solidus temperature ($T_S$), peritectic transition temperature (T_P) and liquidus temperature (T_L) were determined. The results were verified by statistic evaluation and they were compared with theoretical calculations carried out by InterDendritic Solidification (IDS) and Thermo-Calc™ (2015b, TCFE8; TC) software.

The original experimental data of phase transformation temperatures were obtained by thermal analysis. Temperatures in low temperature region, the eutectoid transition temperatures and end of α-ferrite to γ-austenite transition temperatures, were determined only by differential thermal analysis method and verified by Thermo-Calc software. It can be said, that differential thermal analysis and Thermo-Calc are more versatile. All experimental values, show in general a high level of consistency and low level of variability. Both differential thermal analysis and direct thermal analysis are set correctly; the results are reproducible and comparable. The standard deviation of the results did not exceed 2 °C and variation coefficient did not exceed 0.3 %.

It is not possible to determine only one the most precise software or the one closer to the measured results. Besides one case, the software differed less (or it was equal to) 3 °C from each other. The comparison of thermo-analytical methods and software shows the following: in some cases, exceptional agreement, but in most cases the agreement remains poor. Therefore it is always vital to check the data by an experiment. The experimental temperatures obtained by the thermal analysis are expected to be used to optimize production (casting and solidification) and thermo-mechanical processing of the analysed steel grades.

***Key words**: thermal analysis; phase transformations; DTA; Thermo-Calc; IDS*

Předkládaná práce se zabývá studiem teplot fázových transformací dvou reálných značek ocelí metodou diferenční termické analýzy (DTA) a přímé termické analýzy (TA) v nízkoteplotní a vysokoteplotní oblasti. Stanoveny byly teploty eutektoidní transformace (T_E), teploty ukončení přeměny α-feritu na γ-austenit ($T_{\alpha\rightarrow\gamma}$), teploty solidu ($T_S$), teploty peritektické transformace (T_P) a teploty likvidu (T_L). Výsledky byly ověřeny statistickým vyhodnocením a diskutovány s teoretickými výpočty provedenými s využitím software InterDendritic Solidification (IDS) a Thermo-Calc™ (2015b, TCFE8; TC).

Originální experimentální data, tj. teploty fázových transformací, byla získána metodami termické analýzy. Teploty v nízkoteplotní oblasti, tedy teploty eutektoidní transformace a teploty ukončení přeměny α-feritu na γ-austenit, byly stanoveny pouze metodou diferenční termické analýzy a porovnány pouze s výsledky software Thermo-Calc. Lze tedy tvrdit, že diferenční termická analýza a Thermo-Calc mají všestrannější použití. Metody diferenční a přímé termické analýzy jsou nastaveny správně a výsledky jsou reprodukovatelné a porovnatelné. Výsledky termické analýzy vykazují vysokou míru konzistence a nízkou variabilitu. Směrodatná odchylka byla ve všech případech nižší než 2 °C a variační koeficient byl nižší než 0,3 %.

Bylo zjištěno, že není možné určit přesnější software nebo software, jehož výsledky by byly blíže k naměřeným hodnotám. Vyjma jednoho případu se výsledky IDS a Thermo-Calc mezi sebou lišily méně než o 3 °C. Shoda výsledků *získaných využitím software a experimentálních výsledků termických analýz je značně proměnlivá: v některých případech výborná, v jiných případech je naopak velmi nízká. Z tohoto důvodu je nutné vždy ověřit výpočet software experimentem. Existuje reálný předpoklad využití získaných výsledků k optimalizaci výroby (odlévání a následného tuhnutí) a tepelně-mechanického zpracování analyzovaných značek ocelí.*

***Klíčová slova**: termická analýza; fázové transformace; DTA; Thermo-Calc; IDS*

Material science and general knowledge about thermo-physical properties of steels are getting more and more importance due to an increasing pressure on steel industry to reduce price of steel to a minimum. The most promising way chosen by the companies is reduction of costs, with particular focus on energy savings [1, 2]. It is widely known, that production of steel is energetically demanding, therefore it is necessary to define requirements and to analyse real steel samples in detail in order to achieve precise and efficient production process.

Theoretical calculations using specialised software are becoming increasingly important due to their overall efficiency. A decision cannot be made, though, based purely on calculation. The accuracy of the calculation depends on the used model and correct data in its databases. Lack of data or faults in databases result in unpredictable errors of calculation, and therefore it is recommended always to check the calculated results by experimental measurement [3, 4].

Significant thermo-physical properties of steels are, among others, temperatures of solidus, liquidus, eutectoid, peritectic and magnetic transitions [5, 6]. The aim of this paper is to obtain these key thermo-physical data from two real steel grades by experiment and by theoretical calculations, then to assess the calculated and experimental data in terms of reproducibility, to evaluate the comparability of the analytical methods used and to revise the substitutability of the thermal analysis by software.

Analytical methods and calculations

Thermal analysis covers a wide range of methods used to determine the physical or chemical properties of material as it is heated, cooled or held at constant temperature. This provides analytical information on the fundamental properties of materials [7, 8]. The experiments were carried out by two thermo-analytical methods: Differential Thermal Analysis (DTA) and Direct Thermal Analysis (TA).

Differential Thermal Analysis (DTA) is a thermo-analytical method, in which the temperature effects are studied during continuous linear heating or cooling in controlled atmosphere [9]. The temperature of the analysed sample is measured relative to a reference sample. A reference sample can be standard material (e.g. Pd) or an empty crucible. The result of the measurement is a DTA curve [10].

Direct Thermal Analysis (TA) is a thermo-analytical method, in which the direct measurement of temperature of the sample is carried out during its heating or cooling in controlled atmosphere [11, 12].

The amount of heat involved and the temperature, at which these changes take place, are characteristic for the changes in the steel structure: Eutectoid Transition (T_E), End of α-Ferrite to γ-Austenite transition ($T_{\alpha\rightarrow\gamma}$), Solidus ($T_S$), Peritectic transition (T_P), Liquidus (T_L) [13].

Theoretical calculations

Theoretical calculations were performed by Thermo-Calc^{TM} [14] (TC) and InterDendritic Solidification [15] (IDS) software. The Thermo-Calc is a sophisticated software using CALPHAD approach and it includes many databases, which are necessary to its calculations. The IDS software is based on kinetics and thermodynamic calculations and it is utilized for the determination of temperature dependencies for thermo-physical properties of steels.

The IDS calculations (SW) reported problems with calculations for studied steel samples below temperatures of 1000 °C due to exceeded concentration limits of some elements. Extensive tests were performed to find the problematic elements, but with no success. Only limited content of most of the elements enabled calculation in this temperature region, but results were incorrect and not corresponding with the experimental results. For IDS calculations Sn, As, Sb were not included, because they were not defined in the IDS database, and O was excluded due to the defective results with high divergence in respect to experimental values.

The Thermo-Calc calculations were performed on TC v. 2015b, using TCFE8 database. All the determined elements were used for calculation; Sn, As, Sb were not defined in the TCFE8 database and they were not taken into account. Oxygen was excluded due to its impact on stability of calculation in terms of calculation time and results obtained.

Impact of phases allowed in TC calculation was tested as well. It is recommended to exclude only the phases that we are certain about that they cannot be found in the sample during its analysis. In this case, metastable equilibrium during experiment was achieved; therefore diamond and graphite phases were excluded. In general, restriction of one or two phases, that were present at some point during calculation, resulted in no effect on the calculated temperatures. Only the amount of phases differed. When calculation was restricted to the main phases only (FCC, BCC, cementite, and liquid), the results were affected by significant error. Solidus temperature was the most affected by restriction of phases. Moreover, the calculation often became unstable and ended in the range of T_S point. Elimination of the phases had no practical impact on duration of calculation. The best results were obtained if all the phases, except for diamond and graphite phases, were allowed; therefore this setting was used as a default for calculation of important temperatures.

Experiment

Medium carbon steels were prepared from real steel castings. The samples were machined to a desired shape for each equipment and method, then polished and cleaned by ultrasound in acetone. The mass of the samples was 23 – 25 g for TA and approximately 200 mg for DTA. The S1 sample contained 0.368 wt. % and the S2 sample contained 0.646 wt. % of carbon. Description and setting of equipment is described e.g. in [15].

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

The experiments were performed for low and high temperature region separately, in order to eliminate impacts of decarburization and to ensure, that all phase transitions and heat effects were easily identifiable. The experiments were performed in corundum crucibles in an inert atmosphere of Ar (6N). Heating rates were 10 °C·min^{-1} (DTA) and 5 °C·min^{-1} (TA). The measured temperatures were corrected by melting temperature of pure palladium (5N), by melting temperature of pure nickel (5N), by influence of the heating rate and by the influence of the sample mass.

Results and Discussion

Based on the results of DTA and TA analysis (Figs. 1 – 3), the following temperatures of the phase transitions were determined: Eutectoid Transition (T_E), End of α-Ferrite to γ-Austenite transition ($T_{\alpha\rightarrow\gamma}$), Solidus ($T_S$), Peritectic transition (T_P), Liquidus (T_L). Experimental and also theoretical temperature values are presented in Tab. 1. Statistic evaluation of the obtained experimental results was performed by mean values, standard deviation and variation coefficient. All measurements, in general, show high level of consistency and low level of variability. The standard deviation of the results does not exceed 2 degrees of Celsius and variation coefficient does not exceed 0.3 %. Fig. 1 presents DTA curves in low temperature region, and Fig. 2 presents DTA curves in high temperature region.

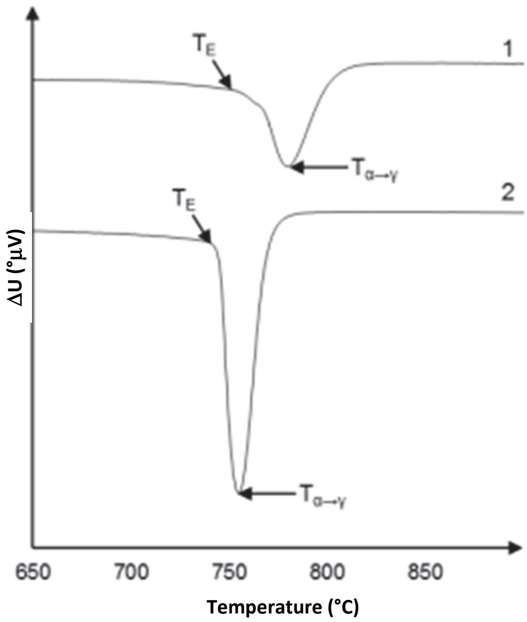

Fig. 1 DTA curves, low temperature region; steel grade 1 and 2
Obr. 1 DTA křivky, nízkoteplotní oblast; ocel jakosti 1 a 2

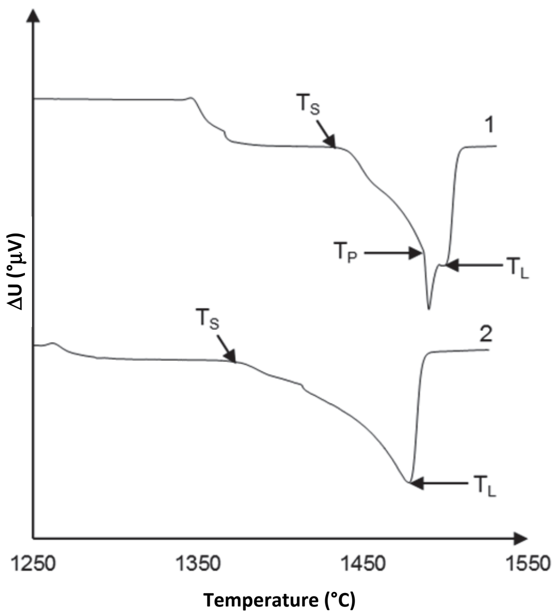

Fig. 2 DTA curves, high temperature region; steel grade 1 and 2
Obr. 2 DTA křivky, vysokoteplotní oblast; ocel jakosti 1 a 2

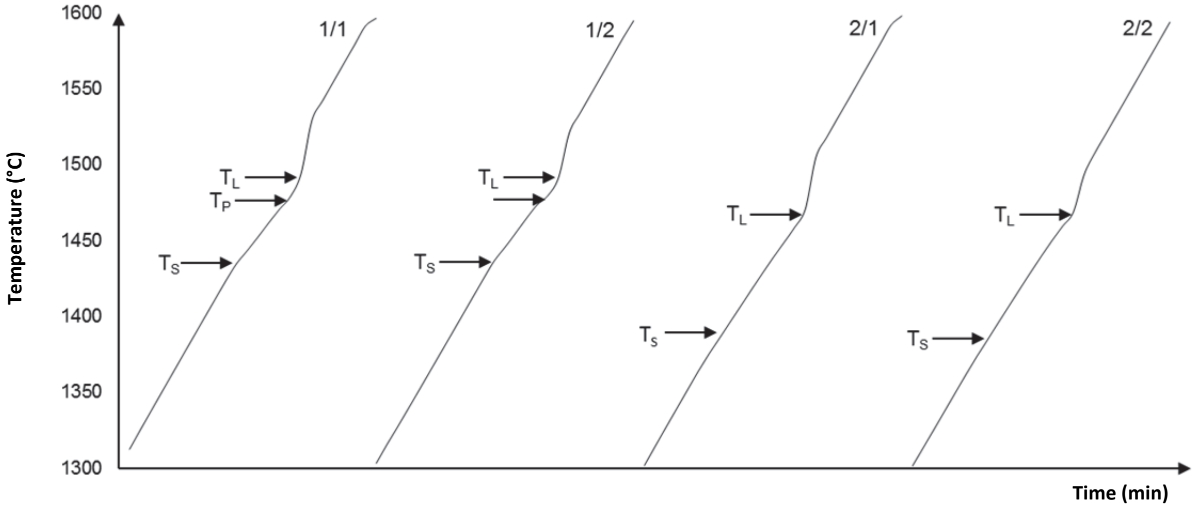

Fig. 3 TA heating curves; steel/cycle; steel grade 1 and 2; 1st and 2nd cycle
Obr. 3 TA křivky ohřevu; jakost/cyklus; ocel jakosti 1 a 2; první a druhý cyklus

For both figures, the curve 1 represents the steel 1 and the curve 2 represents the steel 2. Fig. 3 presents only results obtained by heating process of the steel 1 (curves 1/1, 1/2), and the steel 2 (curves 2/1, 2/2). Tab. 1 presents all the measured and calculated results for both steel grades. Two cycles were performed. Each cycle consisted of heating and cooling step in the high temperature region. All the steps were analysed and significant temperatures were determined, but due to the high standard deviation and variation coefficient, the results from cooling step were excluded. Theoretical calculations were focused on determination of all experimental temperatures obtained by DTA and TA. Calculations in high temperature region corresponded with the experimental results. The low temperature region was calculated only by TC software.

For the low temperature region two important transition temperatures T_E and $T_{\alpha\rightarrow\gamma}$ were evaluated. The experiments in the low temperature range were performed by DTA method only, because the TA method was not suitable for measurements in low temperature region due to the lower distinctiveness of the temperature effects on the heating or cooling curve. It was not possible to determine the temperature of magnetic transition (Currie Temperature), heat effects of phase transitions covered completely each other.

The DTA method for the steel 1 gave the average T_E = 758 ± 1 °C and $T_{\alpha\rightarrow\gamma}$ = 782 °C. The calculated results for the steel 1 T_E = 729 °C and $T_{\alpha\rightarrow\gamma}$ = 781 °C. For the steel 2 the average T_E = 746 ± 1 °C and $T_{\alpha\rightarrow\gamma}$ = 757 ± 2 °C. In comparison with the calculated results T_E = 717 °C and $T_{\alpha\rightarrow\gamma}$ = 739 °C.

The calculated eutectoid transition of the steel 1 is approx. 29 °C below the measured value, which is the same difference as for the steel 2. The end of α-ferrite to γ-austenite transition for the steel 1 is only 1 °C below the measured value, while for the steel 2 the difference is 18 °C.

In the high temperature region three transition temperatures T_S, T_P and T_L for the steel 1 were obtained, while only T_S and T_L for the steel 2. Peritectic transition was not observed in the steel 2. Also SW's calculations (IDS and TC) confirmed this fact.

On the basis of DTA method the following transition temperatures were determined for the steel 1: T_S = 1431 ± 1 °C, T_P = 1489 °C and T_L = 1492 ± 1 °C. TA method determined the following: T_S = 1435 ± 2 °C, T_P = 1479 °C and T_L = 1493 °C. Using IDS and TC the following was calculated: T_S = 1437 °C, T_P = 1484 °C, T_L = 1497 °C and T_S = 1440 °C, T_P = 1486 °C and T_L = 1487 °C. On the basis of DTA method the following transition temperatures were determined for the steel 2: T_S = 1363 ± 1 °C and T_L = 1472 °C. TA method determined the following: T_S = 1372 ± 1 °C and T_L = 1472 ± 1 °C. Using IDS and TC the following was calculated: T_S = 1383 °C, T_L = 1477 °C and T_S = 1381 °C, T_L = 1478 °C.

The results in the high temperature region are less clear than in the low temperature region. Both methods, the DTA and TA, report high level of consistency with each other, the difference between all corresponding temperatures is below or equal to 10 °C. In fact, the liquidus difference for both steel grades is below or equal to 1 °C. Therefore it can be assumed, that both methods are comparable and the results are reliable and reproducible.

As for the software, it is not possible to determine only one most precise or closer to the measured results. In the case of solidus, the TC presents better agreement for the steel 2 and worse agreement to the steel 1 compared to IDS. Also the calculated results of solidus by TC and IDS show lower deviation from TA than DTA for both steel grades.

In comparison with the measured values of T_L, the calculated results of the steel 1 by TC are approx. 5 °C below the measured value, while 5 °C above by IDS.

Hutnické listy č. 2/2017, roč. LXX
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This is the only case, where the software differed by more than 3 °C from each other, if the software reached result at all, or the result was obviously faulty. The calculated T_P of the steel 1 is 3 °C below the measured value.

The differences of theoretical and experimental values might have been caused by Thermo-Calc's calculation due to elements restriction (excluded O, Sn, As, Sb) and equilibrium state of all calculated values. Experimental values were obtained from measurements with real steel and all the measurements were not in complete equilibrium. Also, the difference of heat conductivity of the reference and the sample can be reflected in shifted transition temperatures, because the sensors were located on the surface of the crucible or the sample, where the temperature can be exceeding the real transition temperature, while most of the volume of the samples volume is yet below the transition temperature.

Also, the difference of heat conductivity of the reference and the sample can be reflected in shifted transition temperatures, because the sensors were located on the surface of the crucible or the sample, where the temperature can be exceeding the real transition temperature, while most of the volume of the samples is yet below the transition.

Tab. 1 Experimental and calculated results (°C)
Tab. 1 Experimentální a vypočtené výsledky (°C)

Method	Evaluation	S1					S2			
		T_E	$T_{\alpha\rightarrow\gamma}$	T_S	T_P	T_L	T_E	$T_{\alpha\rightarrow\gamma}$	T_S	T_L
TC		729	781	1440	1486	1487	717	739	1381	1478
IDS				1437	1484	1497			1383	1477
DTA	Mean Value	758	782	1431	1489	1492	746	757	1363	1472
	Standard Deviation	1	0	1	0	1	1	2	1	0
	Variation Coefficient (%)	0.12	0.00	0.09	0.00	0,08	0.13	0.27	0.06	0.03
TA	Mean Value			1435	1479	1493			1372	1472
Heating	Standard Deviation			2	0	0			1	1
	Variation Coefficient (%)			0.11	0.00	0.03			0.06	0.03
TA	Mean Value			1439	1455	1487			1433	1461
Cooling	Standard Deviation			5	6	0			4	3
	Variation Coefficient (%)			0.33	0.42	0.03			0.27	0.19

Conclusions

Thermal analysis of two real medium carbon steel samples was performed. Phase transition temperatures were obtained for concrete steels and new original experimental data were obtained. The obtained results (temperatures T_E, $T_{\alpha\rightarrow\gamma}$, T_S, T_P and T_L) were refined, compared and verified with theoretical calculations performed using TC and IDS software. Only the DTA method was used for measurement in the low temperature region and only by using TC it was possible to calculate and verify the experimental results from the low temperature region.

It is not possible to determine only one the most precise software or the one closer to the measured results. The calculated solidus results by TC and IDS show lower deviation from TA than DTA for both steel grades. With the exception of one case, the software differed by less than (or was equal to) 3 °C from each other. The TC is considered to be more versatile. The theoretical calculations by Thermo-Calc and IDS software are providing, in some cases, relatively good calculation results, but it is always vital to check the data by an experiment.

All experimental values, show in general high level of consistency and low level of variability. The standard deviation of the results did not exceed 2 °C and variation coefficient did not exceed 0.3 %. It was shown that both thermo-analytical methods used were set correctly; the results are reproducible and comparable. The experimental temperatures obtained by the thermal analysis will be used to optimize production and processing of the analysed steel grades.

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Podniková rada ThyssenKrupp vyvíjí tlak

Westdeutsche Allgemeine *10.01.2017*

Šéf podnikové rady koncernu Thyssenkrupp Wilhelm Segerath se vyjádřil proti jednostranným ústupkům vůči Tata Steel při fúzi ocelářských divizí. „Nemůžeme souhlasit, že při konsolidaci budou ohroženy jen naše provozy. Jen pouhý pokus vyvolá naše masivní protesty," řekl Segerath. Převzetím ztrátových britských provozů Tata Steel nedává sloučení žádný smysl. Segerath se odvolával na snahu Tata Steel najít s britskými odbory řešení pro vysoké penzijní závazky a pro největší ocelárnu v Port Talbot. Management navrhl dosavadní penzijní fond uzavřít a nahradit ho novým modelem. Současně bylo přislíbeno provozovat obě vysoké pece v Port Talbot ještě minimálně po dobu pěti let. Segerath na to říká: „Dostanou- li oni 5 let, budeme my chtít minimálně deset".

Reformní zápal u Thyssenkrupp

Börsen-Zeitung *20.01.2017*

Šéf Thyssenkrupp Heinrich Hiesinger bude na valné hromadě akcionářů čelit kritice, protože přestavba koncernu přešlapuje na místě a slabost vlastního kapitálu je velmi nebezpečná. Jen s velkým úsilím si může podnik dovolit vyplatit akcionářům navržených 85 mil. € (15 centů na akcii). Současně kýžená fúze ocelářské divize s evropskou částí indického koncernu Tata Steel, která by umožnila dekonzolidaci divize a jejích penzijních závazků, nepokračuje dostatečnou rychlostí. To vyvolává kritiku institucionálních investorů, například Union Investment: „Thyssenkrupp je stále ještě kolos na hliněných nohou". Koncern musí napřít všechny síly na transformaci obchodního modelu a likvidaci zadlužení, aby si zajistil dlouhodobou další existenci. Odvrhnutí ocelářské divize by bylo osvobozujícím odkopem, budoucnost totiž leží v obchodu s technologiemi a ne s ocelí, míní Union Investment. Tento názor šéf koncernu Heinrich Hiesinger plně sdílí, nezbývá mu však nic jiného, než vyčkat, až Tata Steel oddělí od britské části podniku své penzijní závazky.