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Recenzované vědecké články

Use of High-temperature Thermal Analysis for Determination of the Liquidus and Solidus Temperatures for Bearing Steels

Využití vysokoteplotní termické analýzy pro určení teplot solidu a likvidu u ložiskové oceli

doc. Ing. Karel Gryc, Ph.D.[1]; **doc. Ing. Bedřich Smetana, Ph.D.**[2]; **Ing. Michaela Strouhalová**[1]; **Ing. Monika Kawuloková, Ph.D.**[2]; **Ing. Simona Zlá, Ph.D.**[2]; **Mgr. Aleš Kalup**[2]; **doc. Ing. Markéta Tkadlečková, Ph.D.**[1]; **doc. Ing. Ladislav Socha, Ph.D.**[1]; **prof. Ing. Karel Michalek, CSc.** [1]; **Ing. Aleš Opler**[3]

[1] VŠB – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Department of Metallurgy and Foundry Industry, 17. listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

[2] VŠB – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Department of Physical Chemistry and Theory of Technological Processes, 17. listopadu 15/2172, 708 33 Ostrava-Poruba, Czech Republic

[3] VÍTKOVICE HEAVY MACHINERY, a.s., Ruská 2887/101, 703 00 Ostrava-Vítkovice, Czech Republic

The paper is devoted to comparison of the solidus and liquidus temperatures determined by methods of thermal analysis for industrially produced steel grade 100CrMo7. Two modern devices for high temperature thermal analysis were used. Method of direct thermal analysis (PTA) was applied on the device STA 449 F3 Jupiter with use of samples weighing approx. 22 g. The samples weighing approx. 120 to 210 mg were subjected to a differential thermal analysis (DTA) on the device Setaram SETSYS 18TM. Solidus and liquidus temperatures were obtained on the basis of evaluation of experimentally obtained curves during heating (DTA) and cooling (PTA - only liquidus temperature). The results of measurements using methods of thermal analysis were also confronted with the corresponding temperatures obtained from the industrial partner and with the temperatures obtained using specialised programs IDS and ThermoCalc. Identically as in earlier works it was proved that for more accurate determination of the liquidus and solidus temperatures of real steel grades it is very beneficial to use multiple methods of high-temperature thermal analysis. The liquidus temperature obtained by both methods (PTA and DTA) differs only by 1 °C. Variability of the results from individual experiments using different methods is also very low, proving thus the robustness of both methods used. The importance of using different methods of thermal analysis, their mutual verification, is also confirmed by the fact that theoretically determined temperatures differ from the experimentally determined values.

Key words: *bearing steel; solidus temperature; liquidus temperature; thermal analysis methods; thermodynamic database.*

Metody pro studium procesů spojených s výrobou oceli jsou založeny na znalosti termodynamických vlastností materiálů vyskytujících se v technologických uzlech. Teploty solidu (T_S) a likvidu (T_L) patří k nejdůležitějším parametrům, zejména pro odlévání a tuhnutí oceli. Přesná znalost T_L je zvláště důležitá pro nastavení přehřátí oceli před jeho odléváním. Teplota T_S se týká především procesu tuhnutí, kdy existence a rozsah dvoufázové oblasti mezi T_L a T_S ovlivňuje segregaci a jiné jevy spojené s procesem tuhnutí. Znalost těchto kritických teplot je důležitá nejen pro správné nastavení technologie odlévání a tuhnutí ocelového polotovaru, ale také pro přesné nastavení podmínek pro modelování tuhnutí oceli.

Tento článek navazuje na předchozí práce autorského kolektivu. Opět platí, že je zaměřen na možnosti zpřesnění těchto teplot (T_L, T_S) experimentálními metodami vysokoteplotní termické analýzy. Konkrétně byla analyzována ložisková ocel 100CrMo7. Pro tuto značku oceli je možné předpokládat, vzhledem k vysokému obsahu uhlíku (1 hm. %), že skutečná hodnota T_L se může výrazně lišit od teoretické T_L stanovené výpočtem. Očekává se, že na základě přesnější informace o teplotě likvidu je možno nastavit teplotu lití, a to tak, aby bylo dosaženo vyšší kvality ingotu, tj. menšího výskytu pórovitosti a makrosegregací.

Použily se dva moderní přístroje pro vysokoteplotní analýzu. Metoda přímé termické analýzy (DirTA) byla aplikována na velké ocelové vzorky (hmotnost cca. 22 g) v experimentálních podmínkách na zařízení Netzsch STA 449 F3 Jupiter. Druhé zařízení, Setaram SETSYS 18TM, bylo použito pro metodu diferenční termické analýzy (DTA) a pro

malé vzorky (hmotnost cca 120 – 210 mg). Na základě vyhodnocení experimentálně získaných křivek během ohřevu (DTA) a chlazení (DirTA, pouze T_L) byly získány teploty T_S a T_L. U obou použitých metod činil rozdíl pro teplotu T_L pouze 1 °C. Variabilita výsledků získaných z jednotlivých měření v rámci používaných metod byla rovněž velmi nízká. Tato skutečnost potvrzuje robustnost obou používaných metod termické analýzy. Bylo provedeno porovnání naměřených teplot T_S a T_L s jejich hodnotami získanými od průmyslového partnera VÍTKOVICE HEAVY MACHINERY, a.s., i s teplotami T_S a T_L získanými pomocí specializovaného softwaru specializovaných IDS a ThermoCalc. Využití výše popsaných metod termické analýzy vedlo ke stanovení teplot solidu (1280 °C) a likvidu (1441 °C) pro ocel 100CrMo7. Naměřená teplota se liší od teoreticky stanovené teploty o desítky stupňů a dokonce i více než o 100 °C. Teoreticky vypočtená teplota (IND) T_S je přibližně o 155 °C nižší nežli naměřená teplota. Teplota T_L pak byla vypočtena průmyslovým partnerem o 28 °C vyšší, než je ve skutečnosti.

Klíčová slova: *ložisková ocel; teplota solidu; teplota likvidu; metody termické analýzy; termodynamická databáze*

Methods for studying processes related to steelmaking are based on the knowledge of thermodynamic properties of materials occurring in the given technological nodes. Solidus (T_S) and liquidus (T_L) temperatures belong to the most critical parameters, especially for casting and solidification of steel. Exact knowledge of T_L is particularly important for setting of the superheating temperature of the steel before its casting. T_S is then related mainly with the solidification process as such, when in the two-phase region between T_L and T_S the segregation phenomena take place. Knowledge of these critical temperatures is important not only for the correct setting of the given casting technology and solidification of the steel intermediate product, but also for precise setting of conditions for modelling of the course of steel solidification.

This paper builds on numerous previous works, for example [1]. It focuses again on possibilities of more precise determination of these temperatures (T_L, T_S) using experimental methods of high-temperature thermal analysis. This time the focus was concentrated on the bearing steel 100CrMo7 and on determination of its liquidus and solidus temperatures. It can be assumes that due to the very high C content, the true value of the liquidus temperature may be significantly different from the theoretical temperature determined by calculation. It is expected that by obtaining more precise accurate information about the liquidus temperature it would be possible to adjust the casting temperature so as to achieve higher quality of the ingot, i.e. lower scope of porosity and of macro-segregation.

1. Used methods of thermal analysis and theoretical calculations

The term thermal analysis [2] can be explained as group of methods, which enable monitoring of changes in the investigated substance by measurement of selected physical properties in dependence on time or temperature (phase transition, heat capacity, dissociation, etc.). Thermal analysis is from the perspective of the steel industry used mainly for determination of T_L and T_S.

Methods of thermal analysis represent mostly dynamic processes, the aim of which is to obtain information about change of the sample state. These processes require a non-isothermal temperature mode, which is generally constant heating or cooling of the sample [2, 3]. Changes in the state of the studied material are determined both directly by measurement of the selected physical properties, and indirectly by measurement of the properties of atmosphere surrounding the sample.

Several dozens of thermo-analytical methods exist. Three of them are important. These three methods are used in one half up to three-quarters of all professional works concerning thermal analyses [4-10]. These methods are the following ones: differential thermal analysis (DTA), differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TG). Simultaneous methods also exist in combinations TG/DTA and TG/DSC. In the past the method of direct thermal analysis, based on direct measurement of the change of the sample temperature, especially during controlled cooling, was also used very often [11]. This method has been and still is used mainly for measurements of T_L and T_S of metallic materials.


a) Netzsch STA 449 F3 Jupiter   b) Setaram SETSYS 18_{TM}

Obr. 1 Zařízení pro vysokoteplotní termickou analýzu
Fig. 1 Equipment for high-temperature thermal analysis

As it was already mentioned in the previous work [1], at the Faculty of Metallurgy and Materials Engineering at the VŠB – Technical University of Ostrava a new laboratory for modelling of processes in liquid and solid phase was established within the project RMSTC (Regional Materials Science and Technology Centre). This laboratory was, among other things, equipped with

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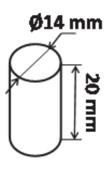
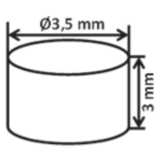
new equipment for high-temperature thermal analysis – Netzsch STA 449 F3 Jupiter (Fig. 1a). Another device used in this laboratory for high-temperature thermal analysis is Setaram SETSYS 18_{TM} (Fig. 1b).The equipment Netzsch STA 449 F3 Jupiter was used for measurements using the PTA method - direct measurement of sample temperature in dependence on time (large samples, sample mass approx. 22 g), see Tab. 1. Values of the liquidus temperature during the cooling mode were obtained, and also in the mode of cyclic experiments (2 cooling cycles were performed under identical conditions). The solidus temperature could not be determined by this device and this method due to low sensitivity of the temperature sensor, which was reflected in the ambiguity of the cooling/heating curve).

Setting of temperature modes of cooling was as follows: the sample was heated to the temperature, at which the sample completely in the liquid phase. From this temperature it was cooled down at the cooling rate of 5 or 10 °C·min^{-1} (first cooling) until complete solidification, and it was then again re-heated to the complete melting and then cooled down again at the cooling rate of 5 or 10 °C·min^{-1} (second cooling). The device Setaram SETSYS 18_{TM} with DTA method, the principle of which is measurement of the temperature difference between the sample and the reference, was used for determination of the liquidus and solidus temperatures (small samples, sample mass approx. 120 to 210 mg) only during heating. Dimensions of the samples are also given in Tab. 1. Heating itself of the sample was carried out at higher heating rate (30 °C·min^{-1}) from the temperature of 20 to 1 200 °C, and then from 1 200 to 1 600 °C at the heating rate of 10 °C·min^{-1}. Temperatures T_L and T_S were not determined from the DTA curves obtained during cooling. Thanks to the higher sensitivity of the sensor it was possible to determine in the analysed steel grade also the solidus temperature.

Tab. 1 Rozměry vzorků ocelí pro použité metody vysokoteplotní termické analýzy
Tab. 1 Dimensions of steel samples for used high-temperature thermo-analytical methods

Sample for device:	**STA 449 F3 Jupiter**	**Setaram SETSYS 18_{TM}**
Dimensions:	 	 

Chemical composition of the investigated bearing steel 100CrMo7 according to the specification of the industrial partner - VÍTKOVICE HEAVY MACHINERY, a.s. (VHM) is given in v Tab. 2.

Tab. 2 Chemické složení ložiskové oceli dle specifikace
Tab. 2 Chemical composition of bearing steel according to the specification

Element	**Element content (mass %)**
C	0.93 – 1.05
Si	0.15 – 0.35
Mn	0.25 – 0.45
P	max. 0.025
S	max. 0.015
Cr	1.65 – 1.95
Mo	0.15 – 0.30
Cu	max. 0.30
Al	max. 0.050

Apart from the above mentioned methods of thermal analysis we obtained from the VHM also T_L and T_S determined on the basis of calculation relationships used in steel shop of this industrial partner. These values were further complemented by our own calculations made with the inclusion of complex available chemical composition in specialised programs IDS (Solidification Analysis Package) and ThermoCalc (database TCFE7) and they are collectively presented in Tab. 3.

Tab. 3 Teoreticky stanovené T_L a T_S
Tab. 3 Calculated liquidus and solidus temperatures

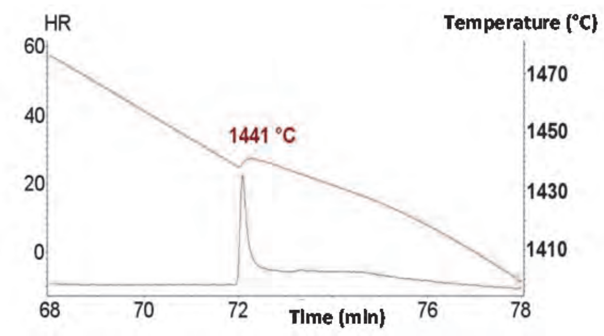
Source	T_S	T_L
	(°C)	
VHM	1 125	1 469
IDS	1 319	1 451
ThermoCalc	1 318	1 453

2. Results and their discussion

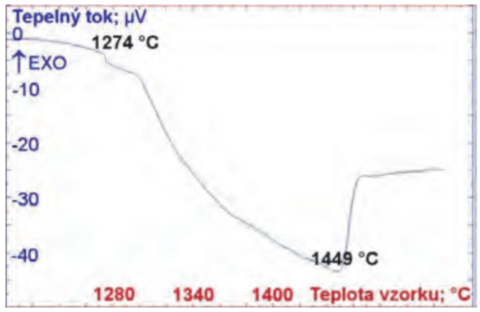
Fig. 2 shows examples of results of individual experiments carried out with use of two above-mentioned methods of thermal analysis on both laboratory devices during more precise determination of T_S and T_L of the investigated steel.

Fig. 2a) shows the cooling curve obtained by PTA method with use of the device Netzsch STA 449 F3 Jupiter. A sample weighing approx. 22 g was cooled down at the cooling rate of 10 °C·min^{-1}. The minimum of the heat flow assigned in this curve to the temperature of 1 441 °C was assessed as T_L. Then the sample temperature decreases gradually due to reduction of the share of exothermic effect in the next course of phase transformations.

Fig. 2b) shows the DTA curve obtained with use of the device Setaram SETSYS 18_{TM} – this time during heating, namely at the heating rate of 10 °C·min^{-1}. On the basis of experience with the evaluation of DTA curves the temperatures T_S = 1 274 °C and T_L = 1 449 °C were identified on this curve.



a) Křivka ochlazování vzorku oceli 100CrMo7 na zařízení Netzsch STA 449 F3 Jupiter
a) Cooling curve for steel sample from experiment realised on Netzsch STA 449 F3 Jupiter device



b) DTA křivka ohřevu vzorku oceli 100CrMo7 na zařízení Setaram SETSYS 18_{TM}
b) DTA curve from heating of steel sample on Setaram SETSYS 18_{TM} device

Obr. 2 Příklady křivek získaných při vysokoteplotní termické analýze studované oceli 100CrMo7
Fig. 2 Examples of curves obtained during high-temperature analysis of 100CrMo7 steel grade

Determination of T_S and T_L of this steel was preceded by a number of methodological experiments. Among others, on both devices with use of both methods their calibration was performed using the samples of standard – nickel of purity 4N5, the melting point of which (1 455 °C) is exactly known. Deviations obtained during experiments with nickel of high purity then allowed us to quantify the temperature correction expressing the effect of experimental conditions on the values of T_S and T_L of real steel grades. Tab. 4 presents the corrected T_S and T_L for individual valid experiments, including basic statistical analysis (average, standard deviation, variation coefficient).

Tab. 4 Korigované T_S a T_L získané pomocí metod termické analýzy
Tab. 4 Corrected T_S and T_L obtained by used thermo-analytical methods

Device	Method	Conditions of experiment			Results					
		Sample	Mode	Heating/cooling rate	Liquidus temperature T_S			Solidus temperature T_L		
				°C·min^{-1}	°C	Statistical values		°C	Statistical values	
SETSYS	DTA	V1C9	H	10	1296	Average	1 280	1443	Average	1 440
		V1C10			1275			1438		
		V1C11			1271	Standard deviation	10	1435	Standard deviation	3
		V1C2			1277	Variation coefficient	0.78 %	1442	Variation coefficient	0.20 %
JUPITER	PTA	V1.1	C1	5	-			1442		
			C2		-			1441		
		V1.3	C1	10	-			1439	Average	1 441
			C2		-			1435		
		V1.4	C1		-			1440	Standard deviation	3
			C2		-			1441	Variation coefficient	0.20 %
		V1.5	C1		-			1446		
			C2		-			1440		

Pozn.: H – ohřev, C1 – ochlazování v 1. cyklu, C2 – ochlazování v druhém cyklu
Note: H – heating, C1 – 1st cycle of cooling, C2 – 2nd cycle of cooling

As already mentioned above, small samples with mass of 120 - 210 mg were used for experiments with the DTA method during their heating. The values of T_S and T_L were taken from the obtained from DTA curves and they were subsequently corrected. From the corrected values obtained during heating of individual samples an average was then calculated (1280 °C), which is considered to be the real T_S of the given steel grade. T_S determined by the DTA method show higher standard deviation (10 °C) and also higher variation coefficient (0.78 %). This variability is, however, generally accepted to be negligible. In the case of T_L the variability is even lower and its average value (1 440 °C) can thus also be regarded as relevant.

As it is evident from the example in Fig. 2b, during cooling of large samples of this steel it was not possible to determine reliably T_S, since during cooling no sufficient thermal effect appeared on these large samples. In the case of use of the method of direct thermal analysis it was possible to read for the investigated steel only T_L values directly from the cooling curve, while variability of individual measured values was again very low. The obtained values that were corrected with respect to experimental conditions can be considered relevant. Final T_L determined by this method (1 441 °C) was recalculated the average of the values obtained in a series of all relevant experiments performed using the PTA method. For practical reasons it is preferable to consider higher value of the T_L as a result of high temperature thermal analysis, it means T_L = 1 441 °C.

Tab. 5 Srovnání experimentálně získaných a teoreticky stanovených T_L a T_S

Tab. 5 Comparison of experimentally obtained and theoretically determined T_L and T_S

Source	Solidus temperature T_S	Liquidus temperature T_L	ΔT_S	ΔT_L
	(°C)			
Thermal analysis	1280	1441	X	X
VHM	1125	1469	-155	28
IDS	1319	1451	39	10
ThermoCalc	1318	1453	38	12

Comparison of T_S and T_L determined by correctly set methodology of high thermal analysis with the values determined by calculation shows that the real T_S and T_L in case of the steel 100CrMo7 significantly differ from the theoretically determined values. The differences between theoretically calculated and measured values is summarised in Tab. 5.

The most significant differences of both temperatures (T_S and T_L) was obtained by comparison of the experimentally determined values with the values used in the conditions of our industrial partner, wherein the difference of T_S was 155 °C and that of T_L was 28 °C. Theoretically calculated temperatures (IDS or Thermo-Calc) are very close to each other. They differ less from the experimentally determined T_S and T_L than the values provided by the industrial partner, in spite of that the differences are not quite negligible (39; 38; or 10 and 12 °C). In all cases, the deviation is higher in case of T_S than in case of T_L.

Conclusions

The paper presented methods of comprehensive verification of solidus and liquidus temperatures for the steel grade 100CrMo7. Two methods of thermal analysis were used. On the device STA 449 F3 Jupiter we applied method of direct thermal analysis (PTA). On the device Setaram SETSYS 18_{TM} the samples were subjected to differential thermal analysis (DTA).

The acquired findings can be summarised in the following points:

1. Both used methods of thermal analysis showed a high degree of reproducibility of results, which proves the correct setting of the experimental methodology.
2. Correctness of the measured liquidus temperatures T_L is accentuated by their almost identical average obtained by different methods with very different masses of the samples, when the difference between them was 1 °C.
3. For the tested steel grade 100CrMo7 it was not possible to identify the thermal effect on the cooling curve of large sample using direct thermal analysis, which confirms correctness of the approach taken, when several methods were used for determination of the solidus temperature T_S and the liquidus temperature T_L of real industrially produced steels.
4. For the steel grade 100CrMo7 the following temperatures were determined with use of the methods of thermal analysis: T_S = 1 280 °C, T_L = 1 441 °C.
5. The measured temperatures differ from the theoretically determined values by of tens and even more than 100 °C. Theoretically determined T_S is approx. by 155 °C lower than the measured one; T_L was then determined in the operating conditions by 28 °C higher than it corresponds to reality.

The issue of verification of the T_L and T_S requires more complex approach using several methods. Larger differences in respect to the calculated values can be expected especially in the case of special steels with high content of carbon or of alloying elements. In the final phase industrial scale experiments should be performed, which would enable adjustment of the casting technology itself, so that there significant operational savings could be achieved, by possible reduction of the temperature of steel overheating. Furthermore, it is advisable to implement the obtained results into numerical simulations aimed at optimisation of casting processes of steel casting and solidification, which would lead to achievement of more accurate results corresponding to real operating conditions.

Acknowledgements

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Závitové tyče na 3D měření výrobků

Středojemná válcovna ArcelorMittal Ostrava začíná využívat 3D měření svého unikátního výrobku, závitových tyčí. Zařízení v hodnotě přibližně 1 mil. Kč, které navrhl a vyvinul tým zlepšovatelů, zkrátí čas potřebný pro měření tyčí, čímž se zvýší celkový objem výroby. Měření navíc bude podstatně přesnější: tyče dokáže změřit na tisíciny milimetru. V dubnu se bude zařízení testovat a po vyhodnocení se jeho aplikace v případě úspěchu rozšíří.

„*Při výrobě závitových tyčí na středojemné válcovně jsme měli nízké hodinové výkony. Vyhodnocení prostojů nám ukázalo, že nejvíce času ztrácíme při seřizování trati a odběru vzorků*,“ vysvětluje Michaela Roubíčková z oddělení trvalého zlepšování, která s myšlenkou 3D měření přišla.

Dosavadní postup, kdy musel operátor změřit digitálním posuvným měřítkem sedm požadovaných rozměrů, trval až čtyři minuty a výsledky byly v řádech desetin až setin milimetru. „*Požadavky zákazníků jsou stále náročnější, to nás přimělo přemýšlet i nad měřením, které bude přesnější. Proto jsme se rozhodli jít cestou 3D měření, díky němuž jsme schopni měřit s přesností na tisíciny milimetru*,“ doplňuje Roubíčková. Spolu s kolegy z úseků automatizace a řízení výroby pak na míru navrhla a sestrojila hliníkovou konstrukci, do níž je umístěn profilometr a servomotor. Zařízení vloženou tyč naskenuje a zobrazí obraz, včetně naprogramovaných rozměrů.

„*Máme za sebou první zkoušky na třech typech profilů. Měření je daleko přesnější a rychlejší, vše je tedy dle našich představ a plánů i po praktické zkoušce*,“ hodnotí první výsledky Roubíčková. V případě, že se 3D měření v ostrém provozu osvědčí, bude se aplikovat i u ostatních profilů. Předpokládaná návratnost investice je cca 4 měsíce. Není do ní ale započítán čas ušetřený během seřizování trati, který se zjistí právě v průběhu zkoušek. Je proto pravděpodobné, že skutečná návratnost bude ještě kratší.

Středojemná válcovna je součástí závodu 14 – Válcovny v ArcelorMittal Ostrava. V době svého vzniku v 80. letech byla co do výše investic druhou největší investicí v Československu, hned za jadernou elektrárnou Temelín. Pořizovací cena válcovny byla 7 miliard Kčs. Válcovna dnes vyrábí široký sortiment za tepla válcovaných dlouhých výrobků – jemnou a střední profilovou ocel základních tvarů, žebírkové tyče pro výztuž do betonu, rovnoramenné úhelníky, tyče profilu U a speciální profily. Jako jediná v ČR vyrábí také tyče s průběžným závitem používané ve stavebních konstrukcích, důlních dílech, tunelech či podzemních stavbách, a to bez nutnosti svařování nebo drátování.

- Z tiskové zprávy 12. dubna 2016 -