THE METHODS FOR MEASUREMENT OF THE SLAGS MELTING TEMPERATURE

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Abstract
The submitted paper deals with methods of the melting temperature measurement, which is one of the most important physico – chemical properties of metallurgical slag systems. The paper is focused on two methods of melting temperature measurement using the high-temperature microscope and the thermocouple in Marsh furnace. The melting interval is measured using high temperature microscope while melting temperature is measured using the thermocouple in Marsh furnace. For experiment, slag systems similar to the Reactol 400/2 used as a refined tundish slag in Podbrezová Steel Plant were employed. It was found that both methods are suitable for measurement of the slag systems melting temperature to temperature 1450°C and using them gives reliable results.

Keywords: casting, slag properties, melting temperature, high – temperature microscope

1 Introduction

The efficiency of steel continuous casting is determinate in addition to technological factors by the using of the slag powdery mixtures for steel protection in crystallizer tanks of the continuous casting devices [1].The melted slags, defined as the solid material resulting from the interaction of flux and impurities in the smelting and refining of metals, represent a very important part in forming the basic physical and technological parameters of the final product not only during the production of the steel but also in another wide spectrum of metallurgical applications. Slags protect the metal and remove undesirable impurities [2-4].

The slags form a multicomponent system, which has a direct contact with melt. The composition of slag (composed mainly from oxides, alkaline and alkaline – earth), its chemical properties, melting temperature, viscosity, surface tension and its other properties influence the quality of produced metal, the intensity of production and the stability of running equipment. The chemical composition and the temperature strongly influence their structure. At present, the scientists lack comprehensive sets of information describing heterogeneous processes of slags at high temperatures, as data are obtainable only with difficulties [2, 5-9].

During the process of continuous casting, slags can be divided into:

a) cover and refined slags, which tend to be used in tundish, and
b) slags arising from mould fluxes in crystallizer.

In the process of continuous casting, the slags carry out the following important technological functions**:**

- protection of the metal against contact with the surrounding environment; thermal insulation
- absorption of inclusions floated out during the casting,
- stabilization of the process of casting
- show minimal corrosive effects [8, 10-12].

The effect of slags on the quality of the final product is given by the physico - chemical properties, which themselves depend on the changes in the structure of slags**.** The structure of the slags is in turn substantially influenced by the chemical composition and temperature of melting. [13-15].

The temperature is one of the major characteristics of the technological process of melting. The energetic or thermodynamic state of the material depends upon the heat-mass and physico – chemical transformations taking place during the melting [16, 17]. During the melting process, all of the energy added to a substance is consumed as a heat of fusion, and the temperature remains constant. Mixtures of substances whose components are insoluble in the liquid phase display a melting range (interval) [18].

It is possible to determine the melting temperature of oxidic system in several ways; however, any temperature determination requires a rigorous observation of any given sample [19-20]. One of the methods of measuring the melting temperature consists of measurement with the use of a thermocouple. This method has become very popular in metallurgy and serves an accurate (and only) source of information [16]. Several authors employ this method [21-23]. Another commonly used method of measuring the temperature of melting is the high – temperature analysis. It is a relatively simple method that requires small claim on the sample weight, but it remains rather time-consuming (**Fig. 1**) [24-29].

The present study deals with two ways of measuring the melting temperature of selected slag systems. Used methods provide reliable results, which are complementary.

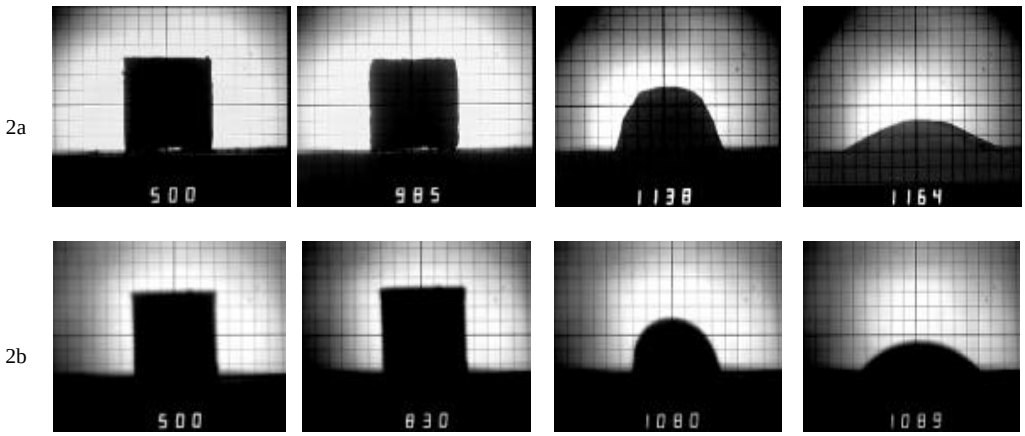

Fig.1 Melting characteristics of mould flux LCB 426 (2a) and LCB 416 (2b) determined by hot stage microscope [26]

2 Material and experimental methods

2.1 Material data

Experiments were designed to study the melting temperature of modeled slag systems. Systems were done by combining natural materials with pure oxides. The new system chemical composition calculations were done by program HSC 5.1, whose composition is close to the composition of REACTOL 400/2 slag used as a refining slag in the basin of Podbrezová Steel Plant. The source material for both simulated systems was dolomite limestone, which was modified by dissociation on dolomite lime. 35 % Al_2O_3 and SiO_2 4 % (the first slag system) were added to the dolomite lime. 33 % Al_2O_3 and 10% clay lime were added to the second slag system. Chemical composition of REACTOL 400/2 as well as the chemical composition of the first and second system are shown in **Table 1**.

Table 1 Chemical composition of REACTOL 400/2 and two modeled slag systems

Label samples	Content [weight %]					
	CaO	**MgO**	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**Sum**
REACTOL 400/2	48,8	18,3	6,70	24,4	1,8	100
1. Modeled slag system	45,69	22,04	5,58	25,77	0,62	99,67
2. Modeled slag system	45,53	21,70	6,65	24,86	1,03	99,77

2.2 Experimental methods

The experiment was realized using the high-temperature LEITZ Wetzlar microscope, which is possible to use to the temperature 1823 K. The temperature of the sample is registered by digital thermometer and displayed on the screen below the sample. Melting range at this methodology is determined according to ISO 540:1995, the temperature at which a melt the first time appears (for instance, drops on the sample surface, melt meniscus on the contact pad – a sample) to the temperature when the sample reaches a spherical shape (outdoor dome), whose surface is without ridges.

To verify and possibly complement the experimental results, the method of measuring the melting of debris using a thermocouple in Marsh furnace with working temperatures up to 1450 °C was used. The temperature measurement is performed by thermocouple, indication PtRh10 - Pt, which is attached to the transducer and transmits the voltage from the thermocouple temperature. The measurement principle is that the sample is placed into ceramic sample boat on the base of Al_2O_3, in the middle of the furnace, 10 minutes at a temperature of about 1400 °C, and then the sample is gradually ejected after 1 cm (8 - times), at which temperatures are read.

3 Results and discussion

According to the ternary diagram CaO - MgO - Al_2O_3 at temperature 1400 °C, the composition created from three major elements of modeled samples was used. (**Fig. 2** – Melting area indicated by a dashed line for both modeled systems). This composition has not been used in practice because another additives (Fe_20_3, SiO_2) are present in the sample of modeled slag systems which have influence on the melting temperature. Therefore it was necessary to determine the melting temperature of system included 5 components.

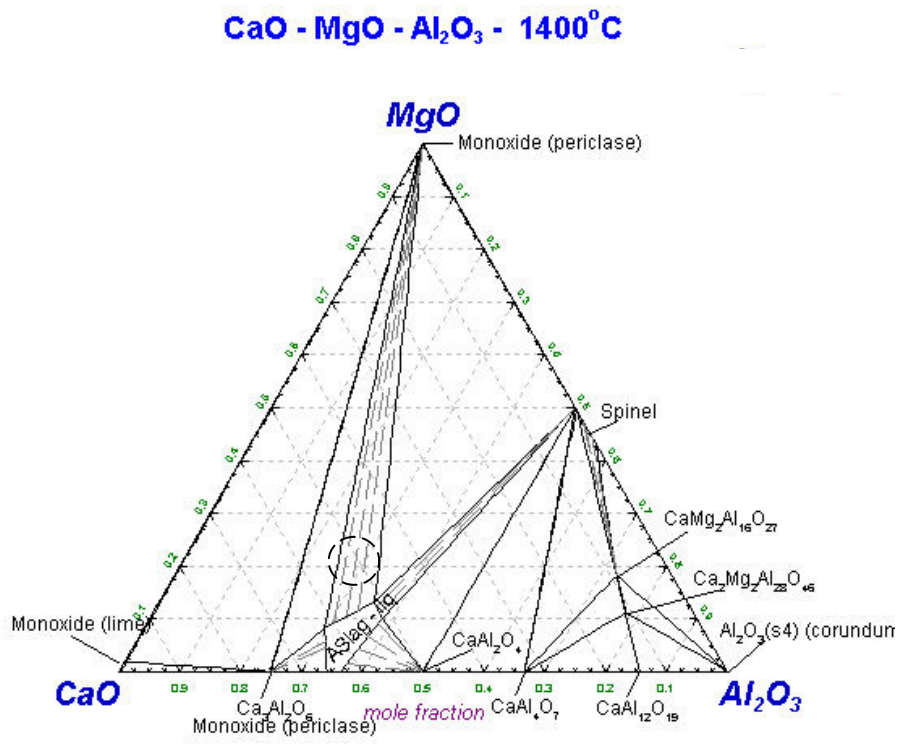

Fig.2 Ternary diagram of $CaO – MgO – Al_2O_3$ system at 1400 °C [27]

The main aim of the experiment was to use a microscope with high range temperature to determine the melting interval of modeled systems.

The sample of the first system was studied in the range from 15 °C to 1452 °C. At temperature to 1333 °C there was no noticeable change in shape and volume of the sample. Melting of the sample leads to the fact that the sample can acquire a barrel shape, and between the sample and the pad can be formed meniscus or edges can be rounded on the sample. In our case, the creation of the first barrel is visible at 1360 °C. This temperature (solidus temperature) can be regarded as the beginning of the sample melting. According to the norm DIN 5173, the melting interval ends reaching a hemispherical shape of the original cube, in our case the temperature 1420 °C (temperature of liquidus). Since the temperature 1430 °C there has been flow of samples.

To verify the temperature reaction course of slag systems samples, melting was carried out by Marsh furnace. The course of sample melting was studied in the cross section in 1 cm intervals from left to right. **Fig. 3** shows the course of melting and thermal gradient of the first sample of slag system and the intended melting rate of the first modeled slag system. Figure also shows temperature running of the first modeled slag system used by high tempertaure microscope.

The course of melting is visible in the first sample simulated system (Fig. 3) and can provide visual softening temperature, melting and flow. An interval of softening was defined from 1345 °C to 1362 °C, whereby softening temperature is 1345 °C. Melting temperature was determined

on 1362 °C and the interval of melting is estimated from 1362 °C to 1375 °C. The flowing temperature was considered on 1375 °C and over this temperature the sample was fluent. **Fig. 4** shows the course of melting and thermal gradient of the second sample of slag system, the intended melting rate of the first modeled slag system and alsothe temperature running of the second modeled slag system used by high tempertaure microscope.

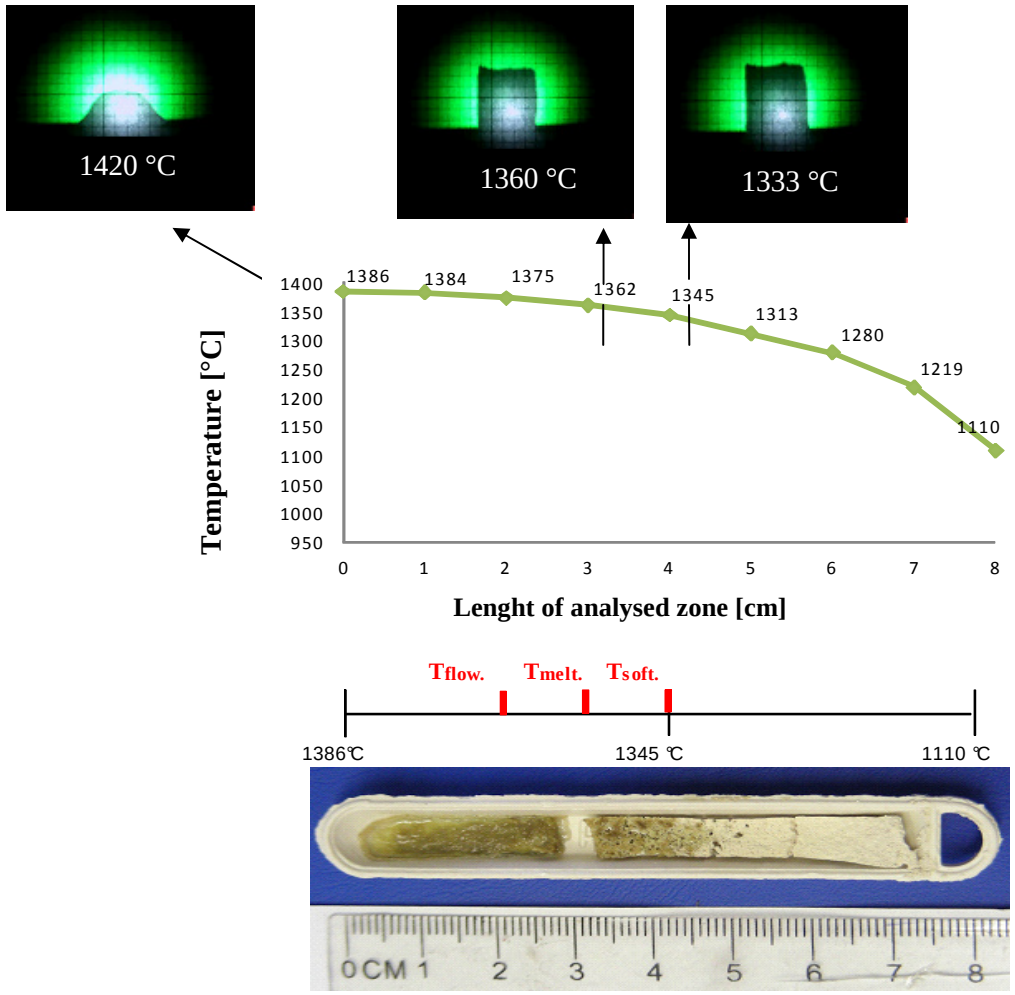

Fig.3 Temperature course of the 1. modeled slags system using Marsh furnace and high-teperature microscope

On a sample of the second slag system can be visually determined the temperature of softening, melting and flow sample (Fig. 4). In that case, an interval of softening was defined from 1349 °C to 1367 °C, whereby softening temperature is 1349 °C. The melting temperature was determined on 1367 °C and the interval of melting is estimated from 1367 °C to 1383 °C. The flowing temperature was considered on 1383 °C and over that temperature the sample was fluent.
Even in that sample is assumed that up to the temperature 1330 °C there was no noticeable change in shape and volume of the sample. The first visible changes were observed at 1335 °C

when samples were sintering, and the meniscus was created. At temperature 1366 °C there is a visible creation of the first barrel. The interval of melting is estimated from 1366 °C to 1422 °C. In this interval, there was a change in sample shape on hemispherical. After reaching the liquidus temperature (the melting interval end) 1422 °C, the melt was homogeneous, and above that temperature the sample flowed.

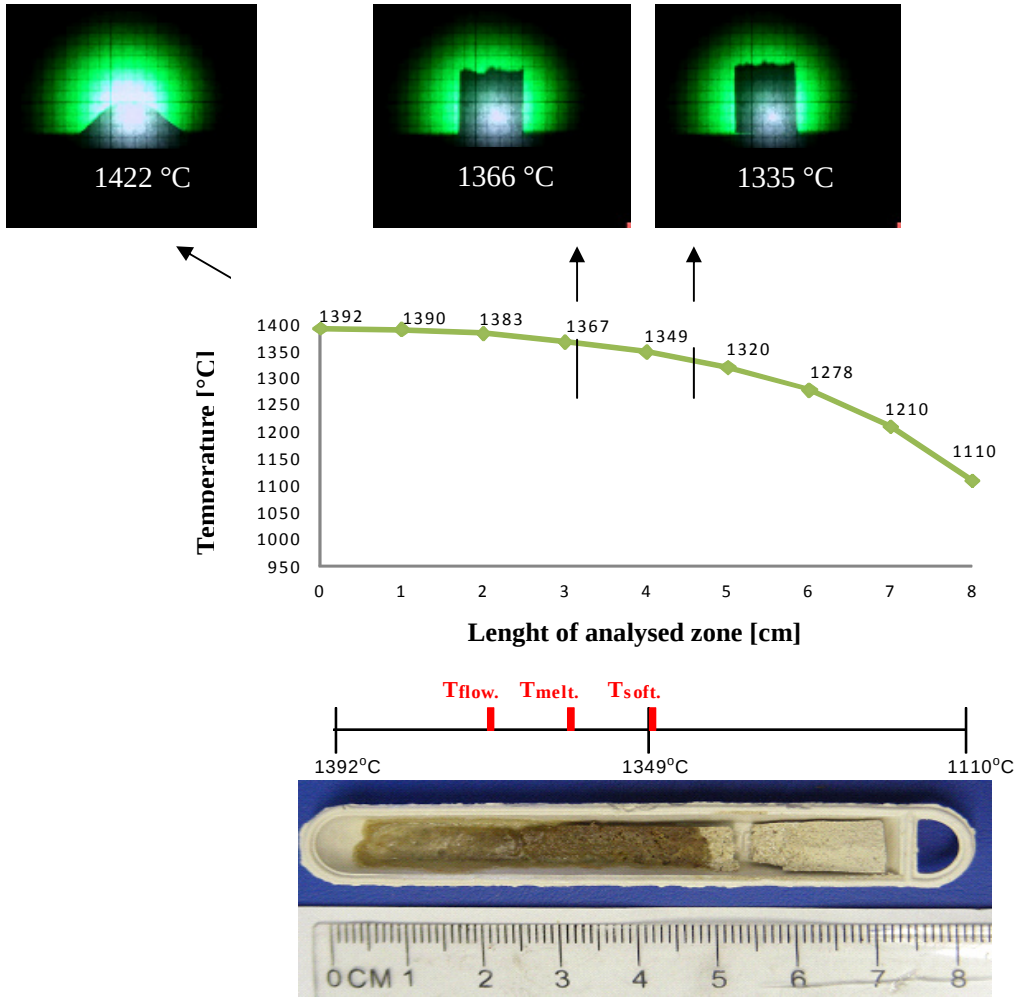

Fig.4 Temperature course of the 2. modeled slags system using Marsh furnace and high-teperature microscope

Comparison of melting and flowing temperatures within the first flow slag system by Marsh furnace and melting interval obtained by high-temperature microscope is graphically shown in **Fig. 5**, as long as **Fig. 6** shows a comparison of temperatures during the melting and flow of the second slag system measured by Marsh furnace and melting interval obtained by high-temperature microscope.

Comparison of the results from the high temperature microscope with the results achieved by measuring in Marsh furnace for the first sample slag system (Fig. 5) shows that the melting

range of the sample measured by Marsh furnace was from 1362 °C to 1375 °C, even though it was determined visually, lies in interval measured on the high-temperature melting microscope (1360 °C - 1420 °C). For the second sample slag system (Fig. 6) the melting range was determined from 1367 °C - 1383 °C and also lies in the interval measured on the high-temperature melting microscope (1366 °C - 1422 °C).

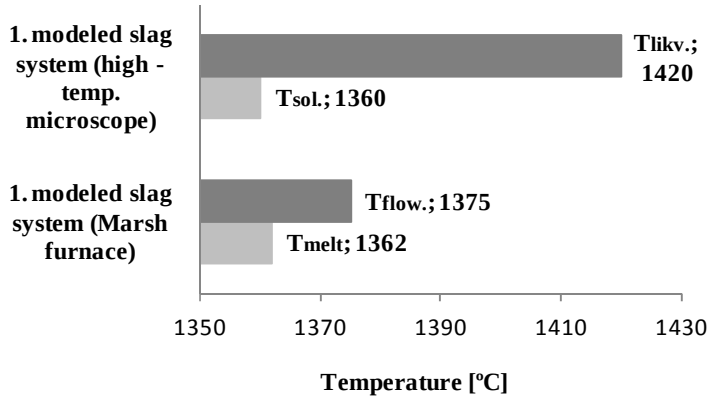

Fig.5 Comparison of measured temperature for the 1. modeled slag system

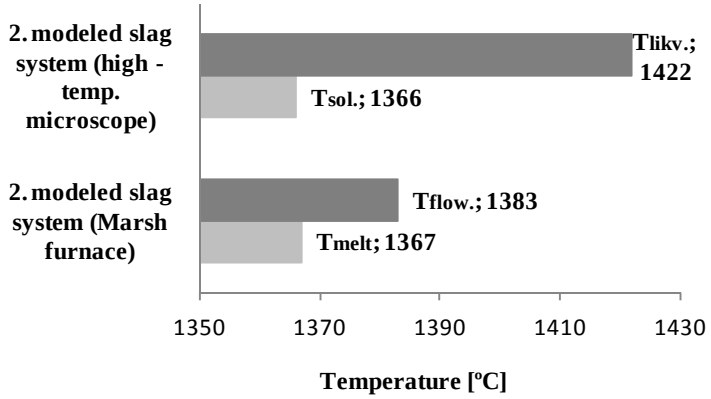

Fig.6 Comparison of measured temperature for the 2. modeled slag system

4 Conclusion

The aim of described experiment was to assess the suitability of use two methods designed to determine the melting temperatures of slag systems and use the high temperature microscope Leitz Wetzlar brand and by Marsh furnace. Achieved experimental results can be formulated as follows:

- determined melting interval by high temperature microscope is about 47 °C higher for the first slag system, and for the second slag system about 40 °C.
- determined flow temperature indicated by producer for REACTOL 400/2 is up to 1430 °C for a sample of the first system was determined on 1375 ° C and the second system on 1383 °C (results from Marsh furnace).

Based on the findings, it can be concluded that both methods are suitable for use in measurement of slag systems melting temperature to a temperature 1450 °C and also that the melting ranges of used samples measured in Marsh furnace lie in melting interval determined by high temperature microscope.

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