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Induction Skull Melting of Oxides and Glasses in Cold Crucible

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Abstract

The present paper is devoted to new results of experimental researches, which are received by realization of the tests at installations at ETP and ETU using cold crucibles. Research results of induction skull melting technology (ISMT) of zirconium dioxide in large volume and ISMT of phosphate (1200 °C) and silicate (1800 °C) glasses with bottom melt casting on installation ETU are submitted. The ISMT feature with the use of a transistor power supply and oxide melting in cold crucible, the influence of melt height, the inductor and some glass properties on conditions of bottom melt casting are discussed. The power characteristics from melting experiments and work conditions of the casting device are analyzed.

Introduction

Induction skull melting technology (ISMT) is a unique process based on contactless transfer of energy to the melted material by high frequency electromagnetic field of high intensity. In spite of conventional induction melting technologies, ISMT is also oriented to melt materials, which are not electrically conductive in solid phase. No chemical contact of the melt with crucible provides extremely high chemical purity of the process.

ISMT provides industrial realization of the processes with:

- Overheating the melt on 500 K and more above the melting point;
- No contact between oxide-coated melt and the crucible material, which ensures high chemical purity of melting process and crystallization;
- Running in different atmosphere or vacuum;
- High-temperature chemical synthesis (chemical reactor);
- Both periodic and continuous run mode of melting and crystallization [1].

By these advantages, ISMT is beyond competition to melt high pure oxides and ceramic materials, to make a glasses syntheses, to grow single crystals of high-temperature materials, etc. At melting and synthesis of glasses the special attention is given to periodicity of the process, when during one melt it is necessary to release the periodic casting of melted glasses. For both set four above mentioned processes were created in cooperation of the two institutes ETP and ETU. A copper cold crucible with a large diameter in order to grow single crystals of ZrO_2 stabilized by yttrium oxide (ETP) and a steel cold crucible with bottom melt casting for glass melting were tested.

1. Induction skull melting of oxides in cold crucibles

The ISMT setup installed at ETP consists of a high-frequency transistor generator, a capacitor bank with high frequency transformer, a process chamber with separate inductor and crucible or inductor crucible, a water cooling system and a set of sensors. The generator provides power up to 300 kW at operating frequency range of 80-350 kHz. Voltage at the inductor (as against the generator output voltage up to 460 V) can be increased by serial connection of capacitors in the bank and additional high frequency transformer installed before the inductor.

Fig. 1. “Big” cold crucibles with inductor

Fig. 2. Ingot in the “big” cold crucible

The cold crucible consists of 20 sections made of rectangular copper tubes (11x22 mm). The air gap between the crucible sections is less than 1 mm. The inner diameter of this crucible was increased up to 270 mm with height of 650 mm. The single-turn induction coil for this crucible has the inner diameter of 334 mm and the height of 250 mm. The crucible is accomplished with a massive copper water-cooled bottom, which is adjustable in position.

The main aim of first experiments at ETP was to investigate how ISMT process runs with high-frequency transistor generator as power supply [1]. Next set of experiments was to test a cold crucible as a melting device and to grow single crystals of ZrO_2 stabilized by yttrium oxide. Melting process was started by heating and burning of metal zirconium. Forming of the melt pool was controlled by power of the generator and charging the crucible by oxide powder. The temperature of the melt was controlled by the generator power as well. Transistor generator with a big reserve of power provided easy control of the process and overheating of the melt. To grow single crystals, a so-called crust above the melt, which consists of baked powder, has been formed. This crust reduces the radiation losses from the melt surface. Necessary cooling conditions for crystal growth were provided by very smooth reduction of power in the crucible an ingot of 70 kg was produced which is partly composed of single crystals of 50 mm length. The huge ingot taken out of the cold crucible is shown in Fig. 2.

2. Induction skull melting of glasses with bottom melt casting

ETU and ETP have continued their joint research into induction skull melting of glasses in cold crucible with bottom melt casting at ETU installations. This work began in 2000 and the results were published in [2, 3]. The main objective of this work is to study the conditions of bottom melt casting for various technical glasses and the impact of various melting parameters on the casting process. The present paper deals with the impact of melt pool level, melt temperature and glass composition on:

- automatic start of the melt casting process after the opening of the drain orifice
- feasibility of melt flow adjustment
- melt casting termination

In addition, the power characteristics of the melting process were calculated. A cold crucible described in [3] with a two-turn short inductor for phosphate glass melting and a three-turn tall inductor for silicate glass melting was used in the tests. A higher melt pool level and, accordingly, a taller inductor increase the melt pool volume in cold crucible and lead to:

- a more uniform heat distribution in the melt pool
- a greater amount of melt discharged in a single drain operation
- a smaller number of manipulations with the drain device

Fig. 3 shows the IMCC furnace for phosphate glass melting.

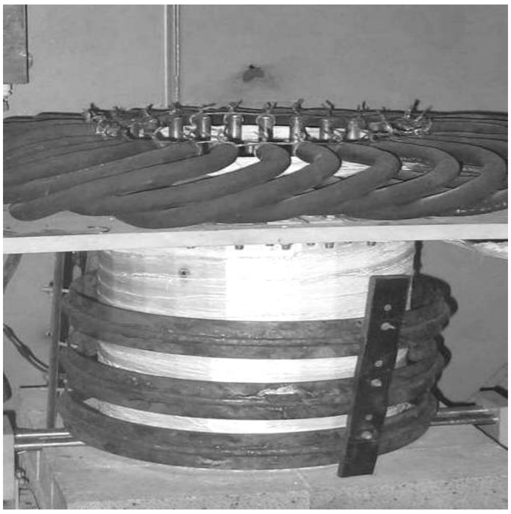

Fig. 3. External view of the IMCC furnace for phosphate glass melting

In the installation the drain device was upgraded in order to improve its cooling, to reduce the effort required to open and close the drain orifice and to enable the adjustment of melt flow during casting. TXA-type thermocouples with steel cores were installed above the ceramic bottom to measure the temperature of near-bottom melt layers. Temperature measurements of the melt pool surface were taken with the help of an optical pyrometer. For the IMCC-5 installation a VChG-11-60/1.76 vacuum-tube oscillator (oscillating power – 60 kW; current frequency – 1.76 MHz) was used. Calorimetric measurements of the furnace were performed during the test in order to define power characteristics of the melting process. During these measurements, a water-cooled copper cover was placed on the cold crucible. The melt pool height was determined by the skull level on a submersible copper probe that was used for this purpose.

2.1. Phosphate glass melting

A three-component phosphate glass frit was used in the melting process. Thermocouple junction point was approximately 10 mm above the crucible bottom and approximately 40 mm from the cold crucible side. The results of calorimetric measurements and oscillator modes are set out in Tab. 1. The characteristics of melt casting conditions are presented in Tab. 2. Changes in thermocouple readings over time are shown in Fig. 4.

Tab. 1. Results of calorimetric measurements of the furnace and oscillator modes

Time, s	T_{melt}, ºC	H_{melt}, mm	I_g, A	U_{ind}, kV	U_a, kV	I_a, A	P_{cc}, kW	$P_{o\ cc}$, W/cm2
4140	1000	130	0.946	2.56	5.58	7,0	18.91	15,44
8280	1200	175	1.486	2.47	5.19	9.8	23.12	14,03
11820	1280	143	-	-	-	-	21.94	16,29

In Tab. 1 are: U_{ind} – inductor voltage; T_{melt} – maximal temperature on the melt surface, ºC; H_{melt} – melt pool level, mm; P_{cc} – power in the cold crucible, including bottom sections.

Tab. 2. Characteristics of melt casting conditions

Casting #	T_{start}, sec	T_{cast}, sec	m_{melt}, kg	M_{mid}, kg/min	T_{melt}, °C
1	15	30	8	32	1180
2	10	98	7,2	4,4	1280

In Tab. 2 are: T_{start} – time between drain orifice opening and the start of melt casting; T_{cast} – melt casting time; m_{melt} – the mass of drained melt; M_{mid} – average mass flux during casting.

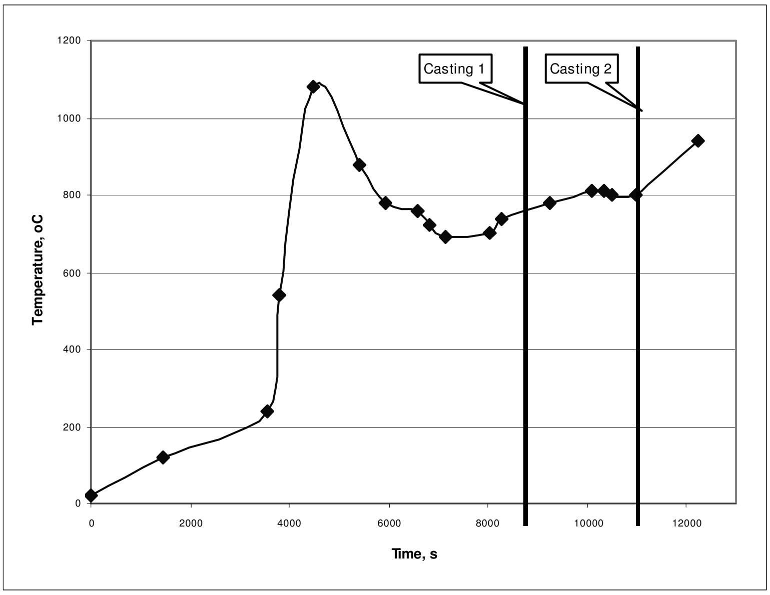

Fig. 4. External view of the IMCC furnace for phosphate glass melting

It was possible to effectively control the melt stream flow. As in the course of the draining operation the temperature in the pool was growing, the oscillator mode was adjusted downwards. A photo of the melt stream is shown in Fig. 5. When the draining operation was completed, the orifice was successfully closed. The view of the melt pool surface after the casting operation is shown in Fig. 6. When the furnace was disassembled, it was discovered that the glass was not fused with the cold crucible ceramic bottom.

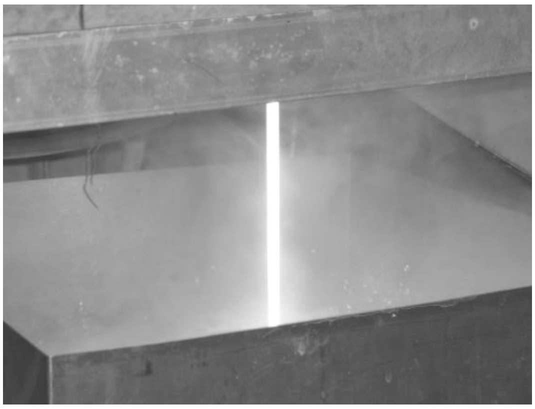

Fig. 5. Molten glass stream

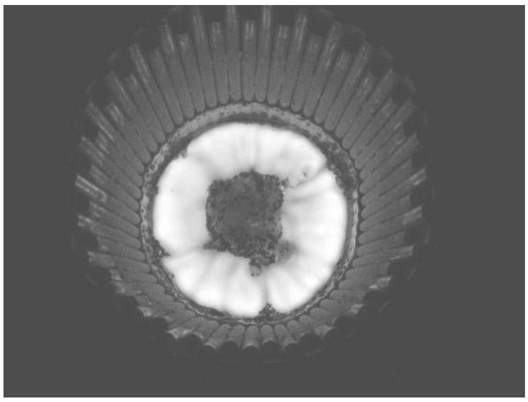

Fig. 6. Melt pool condition after melt casting

2.2. Silicate glass melting

Four-component aluminosilicate cullet prepared in advance was used in the melting process. Three thermocouples were installed at a height of 10 mm above the ceramic bottom at different radiuses in order to measure the temperature of near-bottom melt layers: TC1 – in the central zone of cold crucible; TC2 – at the medium point on the crucible radius; TC3 – at a distance of 30 mm from crucible side sections. Oscillator modes and results of calorimetric measurements of the furnace are presented in Tab. 3. Melt casting characteristics are shown in Tab. 4. Changes in thermocouples readings over time are shown in Fig. 7.

Tab. 3. Results of calorimetric measurements of the furnace and oscillator modes

#	U_a, kV	I_a, A	T_{melt}, °C	H_{melt}, mm	U_{ind}, kV	P_{cc}, kW	P_{ind}, kW	P_{cov}, kW	P_{bus}, kW	$P_{a\ ind}$, kW	cosφ
1	6.77	10.2	1700	105	2.52	21.435	0.318	1.484	0.190	23.24	0.0613
2	7.59	10.3	1660	124	2.6	25.129	0.374	2.084	0.267	27.59	0.0684
3	6.75	9.05	1440	124	2.26	21.469	0.364	1.837	0.212	23.67	0.0776

In Tab. 3 are: P_{cc}, P_{ind}, P_{cov}, P_{tube}, P_{bus} – heat and electrical losses in the cold crucible, inductor, cover, and inductor buses, kW; $P_{a\ ind}$ – active power applied to the inductor and calculated using the following formula:

$$P_{aind} = P_{cc} + P_{ind} + P_{cov} + P_{tube} \quad (1)$$

Inductor power factor was determined through the following formula:

$$\cos\varphi = \frac{P_{aind}}{U_{ind}^2 \cdot 2 \cdot \pi \cdot f \cdot C_{cb}} \quad (2)$$

where f = 1.846 MHz – current frequency; C_{cb} = 5,15 nF – capacitor bank capacity.

A comparison of P_{bus} and $P_{a\ ind}$ values gives a difference of not more that 1%, which does not have a significant impact on cosφ. In future research, it will be necessary to make a correction in cosφ calculations for voltage reduction in oscillator buses and to adjust the C_{cb} value.

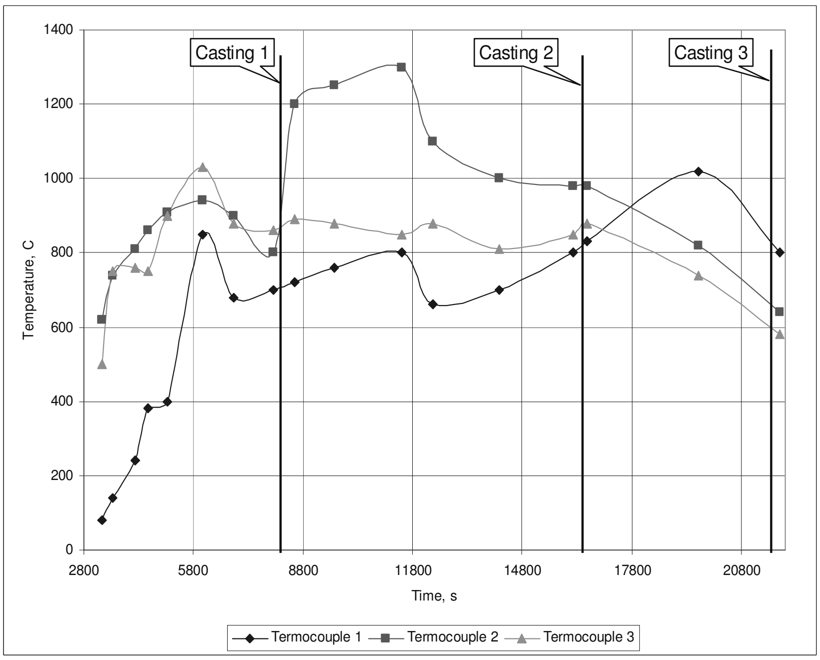

Fig. 7. Changes in bottom thermocouple readings over time

Tab. 4. Characteristics of melt casting conditions

Casting #	T_{start}, sec	T_{cast}, sec	m_{melt}, kg	M_{mid}, kg/min	T_{melt}, °C
1	10	64	9,3	8,7	1700
2	25	32	9,1	17,1	1660
3	10	360	12,5	2,1	1440

When the furnace was disassembled, is was found out that silicate glass did not interact with the ceramic in between water cooled tubes, behaving similarly to phosphate glass. However, in places where ceramic was not cooled, glass fused with ceramic.

2.3. Discussion of tests results of glass melting

The use of a taller inductor enabled an increase in the mass of drained glass melt and did not lead to any problems at the beginning of phosphate glass casting at temperatures of 1180 – 1280ºC. The upgraded drain device as a whole was proved to be operational in the conditions of hard silicate glass melting at temperatures up to 1700ºC.

Thermocouple readings showed that molten glass temperature generally did not rise above 700-800ºC for phosphate glass and above 900-1000ºC for silicate glass before melt casting. Multiple bottom casting of glass melt and melt stream flow adjustment were successfully performed. Ceramic crucible bottom with a water-cooled drain device may be used for melting opaque glasses. Water-cooling should be applied to the entire surface of the crucible bottom for melting optical or transparent glasses, however, gaps between tubes may be increased to 10-25 cm.

During this test it was observed that the melt pool height has an impact on the temperature of central near-bottom melt layers which, in turn, determines the conditions for melt casting. This is a result of inductor voltage increase during casting and a higher specific volumetric power in the melt generated by tube oscillator used the source of power supply.

Conclusions

A set of melting experiments of oxides in cold crucibles was carried out using induction skull melting technology (ISMT). A high-frequency transistor generator was used as power supply in the melting installation at the Institute for Electrothermal Processes (ETP), University of Hanover. Following processes were investigated successfully: test of “big” cold crucible as a melting device and growing single crystals of ZrO2 stabilized by yttrium oxide.

The present task of glass melting included the upgrading and successful testing of a bottom drain device for multiple melt casting from the ISMT furnace at the Saint-Petersburg Electrotechnical University (ETU). The operability of this device was tested in the conditions of melting low-temperature phosphate and high-temperature silicate glasses with various melt pool heights and melt temperatures. Various melting parameters and conditions for molten glass casting were determined.

References

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