

SKULL MELTING TECHNOLOGY FOR OXIDES AND GLASSES

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Introduction. The induction skull melting technology (ISMT) is an excellent method to melt low conductive materials like oxides and glasses. Purest materials could be produced at extremely high melting temperatures above 3000°C [1]. The new melting installation at ETP takes advantage of ISMT. For first experiments with the new installation at ETP YBCO was chosen because of the low liquidus temperature of 1200°C and low electrical resistivity of the melt. The next experiments were carried out with stabilised zirconium oxide (ZrO₂-Y₂O₃) with a liquidus temperature of about 2700°C. As a third material, glass was melted. Two different crucible concepts were used, the well known cold crucible and the new inductor crucible concept.

1. New melting installation at ETP. ETP has installed a new multifunctional melting furnace, as shown in Fig. 1, with a high frequency transistor generator as power supply. Its maximum output power is about 320 kW and output voltage is about 460 V. The series connection of capacitors and the use of an additional high frequency transformer allow a doubled or even quadrupled converter voltage at the inductor. With the aid of the compensation unit frequency is variable in a range from 80 to 350 kHz.

A measurement data acquisition system is available at ETP in order to record essential electrical and thermal data. With the help of flowmeters and temperature sensors all heat losses can be determined calorimetrically. The temperature of the melt is measured by a pyrometer at the free melt surface.

First melting experiments were carried out a cold crucible. Fig. 2 shows the crucible made of 20 segments each of two water-cooled copper tubes. The inner diameter of the crucible is 150 mm.



Fig. 1. New melting installation at ETP.



Fig. 2. Cold crucible and inductor.

Table 1. Electrical resistivity of YBCO and ZrO₂ at melting temperature.

Name	Consistency	Resistivity in $\Omega\cdot\text{cm}$	Melting temperature in $^{\circ}\text{C}$
YBCO	Mixture of fine powder 5.62 Y ₂ O ₃ – 45.13BaO – 49.25 CuO	0.11.	~ 1200
	Crushed material		
ZrO ₂	Fine powder	0.01	~ 2700

2. Melting experiments in a cold crucible. Three melting experiments were carried out in the cold crucible which are listed in Table 1, including their electrical resistivities at melting temperature.

2.1. Starting process. In order to start the melting process, a conductive material must be embedded in the initial powder. In the case of YBCO, a graphite ring has been placed on the powder surface. After forming a melt pool when it is able to heat itself by eddy currents the ring is taken out of the melt. The melting process of ZrO₂ can be started with metallic zirconium. Pieces of zirconium with a diameter of about 6 mm have been put on the stamped initial material. Afterwards the metal, approximately 40 g in weight, has been covered with additional powder. If zirconium is used as a starting material for melting of ZrO₂, it will not be required to remove the metal because oxidized zirconium (ZrO₂) does not contaminate the melt.

2.2. Melting process. After coupling to the electromagnetic field further the material must be gradually added into the crucible. The density of the melting material increases enormously when the state changes. In the case of ZrO₂ more than in four times the amount of the initial material has to be charged additionally during the melting process.

While melting down, the surface of the melt should be closed with the initial material in order to decrease the radiation losses. The skull melting process runs faster.

2.3. Process control. Simultaneously power or voltage has to be adjusted. At the beginning higher voltages are needed to extend the melt volume. Then voltage must be reduced carefully with the increasing melt volume because, otherwise the skull between the melt and the crucible wall becomes too small and the melt can break through. On the other hand, voltage must be high enough and because of that the melt does not freeze.

Fig. 3 shows the free melt surfaces of both YBCO (left) and ZrO₂ (right) in steady state. The ZrO₂ melt has more than 3000°C, the YBCO melt has merely about 1300°C. Parts of the crust forming above the melt surface are visible in Fig. 3 (right), too.

After melting, the cooling down process was commenced by switching-off the generator promptly. In the case of ZrO₂ voltage was reduced in small steps in several times before the generator was switched off. After cooling down the solidified material was removed from the crucible. The cross-sections of the ingots show clearly the areas of melted and non-melted material. The skull layer between the solidified melt and the non-melted powder is visible, too. Fig. 4 illustrates the ingots of both solidified YBCO (left) and ZrO₂ (right).

After the generator is switched off the YBCO melt solidifies fast from the outside towards the centre. Thereby different effects cause voids in the ingot. The material structure changes in such a way that the density increases. In addition gas production occurs, for example, carbon dioxide, which cannot leak from the

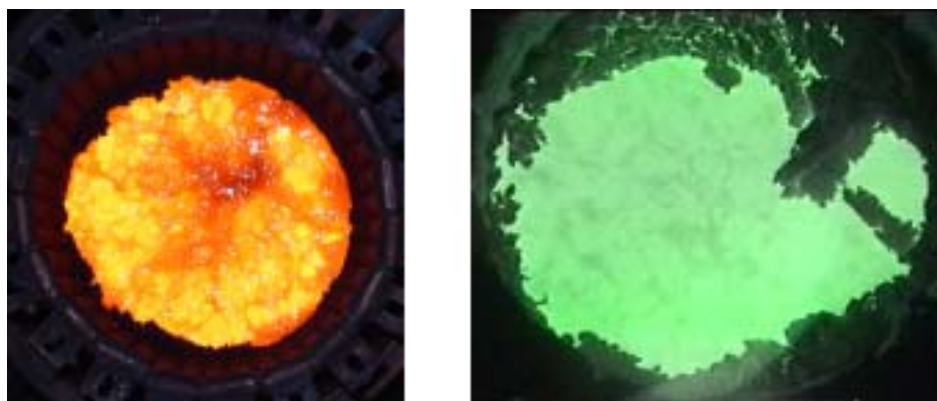


Fig. 3. Melt of YBCO (left) and ZrO_2 (right) in steady state.

inside.

2.4. Experiment results. The cross-section of the ZrO_2 ingot in Fig. 4 (right) shows spontaneous crystallisation of a lot of small single crystallites that has started from the outside towards the centre because of the significantly slower cooling down caused by the voltage reduction in small steps. There are different reasons why only teeny crystals have been grown: 1. Zirconium oxide has not been stabilised. 2. Impurities in the zirconium oxide powder make impossible growing of bigger crystals. 3. The cooling down process is still too fast.

The core of the ingot looks similar to the YBCO ingot with some voids. This structure has been expected because the generator was switched off after a certain time period.

3. Melting experiments in the inductor crucible. Different experiments are carried out in an inductor crucible for melting of glasses. Beside the simpler design, the inductor crucible offers some other advantages compared to the cold crucible like a higher electric efficiency and lower losses. The diameter of the inductor crucible is 250 mm and it contains a volume of 6 l. The first glasses which were melted in the crucible, are high conductive glasses with a melting temperature of $1200^{\circ}C$. For the starting process a gas burner is used. The glass was prepared for melting with a mixture of powder and small glass pieces. The control of the process for starting and melting was investigated by a simulation program which was developed at the University of Latvia [3]. This simulation tool allows to



Fig. 4. Ingots of YBCO (left) and ZrO_2 (right) after cooling down.



Fig. 5. Solidified ingot of glass in the inductor crucible (left) and after removal from the inductor crucible (right).

simulate the electromagnetic, thermal and melt flow behavior during the starting and melting periods. The results of the simulation were taken into account for the design of the inductor crucible and for the control parameters of the process. Fig. 5 shows the results of the first glass melting experiments.

4. Conclusions. First induction skull melting experiments have been carried out successfully with the new melting installation at ETP in Hannover. Two different crucible concepts were used, the well known cold crucible technique and the new inductor crucible technique. In the cold crucible two different oxides, YBCO with a low melting temperature ($\sim 1200^{\circ}\text{C}$) and ZrO_2 with a high melting temperature ($\sim 2700^{\circ}\text{C}$) were melted successfully. The inductor crucible was used for first melting experiments of glass.

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