INDUCTION MELTING OF REFRACTORY OXIDE MATERIALS IN COLD CRUCIBLES

Yu. B. Petrov, A. V. Shul'kov, and I. V. Shurygina

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The induction melting of oxides in cold crucibles (IMCC) seems promising for the production of refractory materials and fuse-cast refractory products [1, 2]. The most commonly used IMCC method has been tested for melting oxide powders to produce a crystalline material. The justification for melting is the higher thermodynamic stability of the grains obtained after crushing and milling a crystalline material compared with a moist or dried powder. The ceramic made from the fused grains is characterized by low shrinkage and deformation on being fired, the constancy of its properties in use, and by good thermal-shock resistance.

The use of the IMCC method for melting oxides is justified by the high purity of the final product which retains the purity level of the initial batch since the noncontaminating induction methods of heating and melting are used in the equipment.

Characteristic features of IMCC of oxide materials are the initial warm up of the original batch until it melts using some other method of heating and the use in the actual melting of a frequency in the range of 0.4-10.0 MHz.

Of the current range of standard frequencies used in electrothermy in the IMCC method, we made use only of the 1.76- and 5.28-MHz frequencies. The industry produces two types of induction heaters with a frequency of 1.76 MHz and a power of 60 or 160 kW and one type with a frequency of 5.28 MHz and a power of 60 kW. We used these supply sources to design laboratory equipment for melting oxide materials. The special features of the design and operation of the apparatus are reported in this article.

The production of fused oxides by IMCC can be by "melting to a block" or "melting and discharging."

The melting into a block is done by batch melting the blocks in a thick-bottomed cold crucible or by the continuous melting of an ingot block in a through, cold crucible with a dummy instead of a bottom.

When the batch is melted to a polycrystalline block, it is particularly important that the productivity of the process is as high and the unit consumption of energy is as low as possible. These indices are most satisfactory in the continuous melting of a block (Fig. 1). The equipment for continuous melting is made in the form of a supporting framework inside which are arranged vertically a frame 1 with the dummy 2. Before the process begins, the dummy is brought inside crucible 3 and fusion begins. The preliminary fusion in the initial warm up is continued until block 4 is melted; the ultimate length of the block is determined by the length of screw mechanism 5 which moves the dummy.

During the process, the heating regime, the rate of supply of batch, and the rate of movement of the dummy must be compatible. A criterion of their compatibility is the constant position of the clear surface of melt relative to the plane of inductor 6. For this purpose, these parameters of the equipment are independently controlled.

The following are some special features of the melting equipment:

the internal surfaces of sections of the crucible are milled since the ingot moves relative to the immovable sections and it is necessary to eliminate any extra resistance to its movement;

the thickness of the walls of the sections is 3-5 mm because of their erosion at the level of the clear surface of the melt, whose position remains constant relative to the sections during melting;

the height of the crucible is $\sim 1.5D_i$-$2.0D_i$, where D_i is the diameter of the inductor; the lower end of the crucible is not less than $D_i/2$ below the plane of the inductor since at this distance the magnetic field

V. I. Ul'yanov (Lenin) Leningrad Electrical Engineering Institute. All-Union Refractory Institute. Translated from Ogneupory, No. 10, pp. 41-46, October, 1980.

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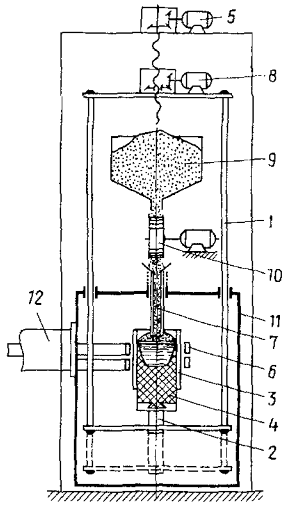

Fig. 1

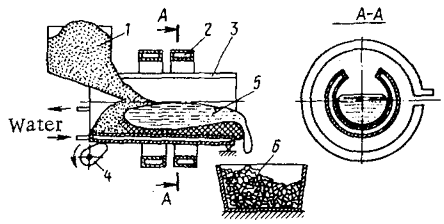

Fig. 2

Fig. 1. Layout of 60/5.28 IMCC equipment for melting blocks of fused oxides: 8) mechanism for adjustable movement of dummy; 9) bunker with batch; 10) batch doser; 11) working chamber; 12) power supply; remainder of symbols explained in text.

Fig. 2. Layout of equipment for melting a discharge melt.

strength is low and the density of the heat source in the melt is negligible and therefore there is no melting of the coating which could lead to a constriction;

the flow rate of the water in the sections is between 1.0-1.5 m/sec and was chosen so as to allow for the need to prevent film boiling in the flow cooling the wall of the sections; if the minimum consumption of water is a criterion, e.g., where there is a scarcity of water, then the minimum possible flow rate v_{min}, m/sec, can be determined from the formula

$$v_{min} \geqslant 5.5 \cdot 10^{-10} p^{-2/3} q_{max}^2, \quad (1)$$

where p is the water pressure in the cooling system, N/m^2; q_{max}, maximum unit heat flow from the melt to the water, W/m^2, determined by calculation for the cross section of the crucible at the level of the clear surface of melt.

At a constant power supply to the melt, the productivity of the IMCC equipment is limited by the heat-exchange conditions between melt and batch. The heat exchange in the throat zone is limited and unstable for the following reasons: firstly, with a low-power generator, it is almost impossible to heat a large bulk of melt and to obtain the required heat flow to the batch; secondly, as a result of the batch sintering above the melt, a dome is formed which acts as a vapor condenser. As a result, the transverse section of the crucible is covered by a strong crust which prevents the batch subsiding. In order to prevent the formation of the domes and to

TABLE 1. Indices of IMCC Process for Several Oxides*

Material to be fused	Cross-sectional area of crucible, cm^2	Productivity of process, kg/h	Theor. required unit energy, kW·h/kg	Unit consump. of energy, kW·h/kg	Total eff. of process, %
CaO†	65	3,5	8,5	1,1	12,8
MgO	80	6,0	9,5	1,0	10,5
Al_2O_3	50	10,8	2,8	0,95	33,9
ZrO_2†	50	2,0	7,0	0,7	10,0
Y_2O_3	95	25,0	2,3	0,55	23,9
Sm_2O_3†	50	2,5	5,3	0,35	6,6
Dy_2O_3†	50	4,0	4,1	0,3	7,3
CeO_2	80	9,0	4,5	0,53	11,7
$3Al_2O_3 \cdot 2SiO_2$	65	9,0	3,7	>0,7	>18,9
$3Al_2O_3 \cdot 2SiO_2$	156	12,0	5,2	>0,7	13,5
80% ZrO_2+15% Al_2O_3+5% SiO_2	50	3,2	6,2	0,65	10,5

*Similar indices of the IMCC process were given in [5], where crucibles of cross-section 400 and 900 cm^2 were used.

†Batch process in a thick-bottomed crucible.

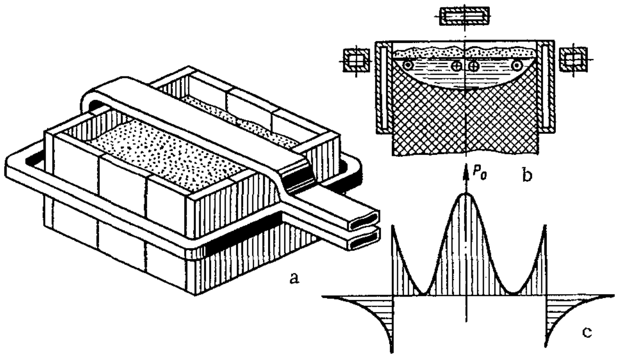

Fig. 3. Inductor with a transverse strip in the equipment for melting a large block: a) crucible with inductor; b) contours of current in the melt; c) distribution curves of unit power through the melt.

eliminate any possible stoppage of the melting process, it was recommended in [3] that a water-cooled plunger 7 be used (Fig. 1) and by this means the throat region of the melt can be perforated either manually or with a mechanical drive. If the plunger is made in the form of a tube, its internal cavity can also act as a batch feed for the melt.

In order to eliminate incomplete melting at the center of the ingot, it is necessary to select the relative diameter m of the crucible within the limits

$$1.0 \leqslant m \leqslant 7.0; \tag{2}$$

where m is determined from the formula

$$m = \frac{D_c}{\Delta_p \sqrt{2}}, \tag{3}$$

where D_c is the diameter of the crucible, m; Δ_p, depth of penetration of current into the melt, m;

$$\Delta_p = 503\sqrt{\frac{\rho}{f}}; \tag{4}$$

ρ is the electrical resistivity of the melt, $\Omega \cdot$ m; and f is the generator frequency, Hz.

If $m < 1$, the electrical efficiency of the inductor is reduced and there is danger of the melt "freezing." If $m > 7$, it is necessary to overheat the melt, which is not always possible and, moreover, has an unfavorable effect on the coating and the electrical strength of the gaps between the sections of the crucible.

Table 1 gives the data on the melting of several oxides in cold crucibles of various cross sections. Table 1 gives for the characteristic the total unit energy consumption in IMCC, ΔW, which was experimentally measured, and the theoretically required energy of melting, ΔW_t, which was determined from the formula

$$\Delta W_t = \frac{H_m - H_{293} + \Delta H}{3600M}, \tag{5}$$

where H_m is the enthalpy of the oxide at the melting temperature, kJ/mole [4]; ΔH, unit heat of melting kJ/kmole; M, molar mass of the oxide, kmole.

The ratio $\Delta W_t/\Delta W$ corresponds to the total efficiency of the equipment. It depends on the nature of the oxide and the melting conditions. It is clear from Table 1 that the total efficiency is lowest when rare-earth oxides are being melted and highest when corundum is melted.

For comparison when oxides are melted in arc furnaces, the total efficiency is from 20 to 80%. Although the unit energy consumption in the IMCC is greater than in arc melting, we must remember that the induction melting is used with pure materials which do not lose this quality after being melted. Moreover, the specific consumption of the batch materials in induction melting is 1.01-1.05 kg for 1 kg of fused oxide and this is lower than when the arc method is used. Hence, it follows that it would be technically and economically viable to carry out the IMCC method for melting oxide into a block.

Melting with discharge is done by accumulating the melt in a crucible and discharging it into a casting mold or drum.

Melting with discharge makes it possible to reduce the size of the production equipment and to organize a continuous horizontal process which is more convenient than the vertical arrangement in industrial conditions. Melting with discharge eliminates the labor-intensity operation of crushing the block since the jet of melt breaks up on falling and sets in the form of tiny drops.

Since the energy is dissipated only in the liquid phase in the IMCC method, the take-off of melt may lead to a reduction in the electrical efficiency of the inductor and the damping of the process. Therefore, when nonconducting oxides are melted in the discharge process, there must always be a small amount of melt left in the crucible.

By the term "electrically conducting oxides" we mean materials with a sufficiently high electrical conductivity in the solid state making it possible to carry out the induction melting without creating the initial volume of melt by means of other energy sources. In the fusion of such materials (stabilized ZrO_2, chromites of rare-earth elements, etc.) the take-off of melt does not lead to a significant reduction of efficiency since the remaining coating on the walls of the crucible is electrically conducting, has a high temperature, and provides the initial heating of new portions of batch and further melting.

The best results of discharge melting were obtained in equipment which consists of a horizontal water-cooled container (Fig. 2). In order to reduce the probability of electrical breakdown, the container is not made in sections in the melting space. The batch arrives from bunker 1 into the region under inductor 2 where the melt is accumulated while container 3 is in the horizontal position. The container is then tilted 5-10° using mechanism 4 and melt 5 flows away into the cooling chamber 6 through special nozzle fittings. The advantage of the equipment includes the possibility of producing the melt with the heating switched on and also moving the container under the inductor in order to reheat the jet.

One feature of melting in the horizontal container is the presence of a significant open area of heated material under the inductor and this causes high losses by radiation and may be the reason for the electrical breakdown in the melting space. Taking this into account, it would be sensible to make the container in the form of a cylinder with a wide opening.

In discharge melting it is possible for the current which leads to erosion of the walls to flow along the surface of the melt between the opposite points of the cross section of the container. In order to eliminate this current, or at least to lessen its force, the level of the melt when it has built up must not rise above the horizontal axis of the container.

Using the horizontal container, we melted $LaCrO_3$ and $NdCrO_3$ for use later to prepare electric heaters; we also melted a eutectic of the forsterite–spinel system ($2MgO \cdot SiO_2 - MgO \cdot Al_2O_3$) in order to study the possibility of making casts of this melt. The heat losses when melting was done in the horizontal container are greater than in the vertical crucible and therefore the unit consumption of energy in discharge melting is 2-3 times more than melting into a block with the same process productivity. It is possible to reduce the consumption by use of high-speed melting and in discharge melting the use of crucibles and containers of a larger capacity.

Manufacture of Shaped Blanks.* The cross section of the IMCC-produced block ingots is determined by the shape of the crucible. This makes it possible to obtain figured blanks by means of an appropriate arrangement of the sections of the crucible. It must be pointed out that in arc melting the production of shaped goods is only possible by casting the melt into a mold.

The IMCC equipment for melting shaped blanks is subdivided into equipment for making complex blocks (e.g., the lining blocks for the furnace masonry) and for blocks with cavities (e.g., pipes and muffles).

The equipment for producing complex blocks is the equipment for continuous melting similar to that shown in Fig. 1. The particular specifications for the quality of the blocks such as a relatively smooth surface and well formed edges can be met by using a slightly hotter melt in order to produce thin and ductile shells and by ensuring that the corners are filled as well as possible.

In order to increase the cross section of the melted ingot and ensure that its core is fully melted, we can recommend here the use of an inductor of the particular design shown in Fig. 3, which is called an inductor with a transverse strip. Its special feature is that the contours of the inducting and induced currents

*V. M. Ganyuchenko and I. P. Rublevskii took part in the work.

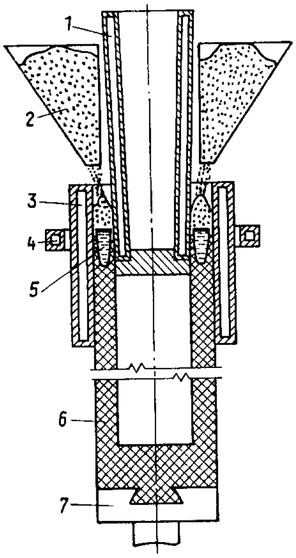

Fig. 4

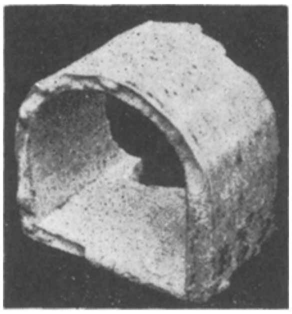

Fig. 5

Fig. 4. Layout of IMCC equipment for melting pipeline blanks: 1) tapered core; 2) bunker with batch; 3) cold crucible; 4) inductor; 5) melt in gap; 6) pipelike blank; 7) dummy.

Fig. 5. An arched muffle detail made of Al_2O_3 melted by IMCC.

are not coaxial with the ingot (Fig. 3b and c). The presence of an inductor lead (the transverse strip) over the melting tank ensures that induced currents appear across the end planes of the tank. Since the density of these currents is roughly double that in the peripheral contour on the side walls of the tank, the melt in the center of the tank is heated more than that in the periphery. This ensures thorough melting of the core of the ingot and at the same time helps to intensify the heat emission from the melt to the newly arriving batch, i.e., increases the process rate.

In the case of an inductor with the transverse strip, Eq. (2) remains and Eq. (3) is transformed to the form

$$m = \frac{B}{2\Delta_p\sqrt{2}}, \tag{6}$$

where B is the larger cross section of the crucible, m.

The intrinsic inductance of the inductor with the transverse strip is almost a quarter of that of the single-coil coaxial inductor for the same area of coil and, therefore, it is not possible to replace one inductor with another in the melting equipment without correcting its matching to the generator.

A more complicated design of melting equipment for the production of blocks with cavities is shown in Fig. 4. In order to mold the cavity it is necessary to use a core which also consists of sections arranged in accordance with the contour of the internal cavity. The process of melting the block occurs in the gap between the crucible and the core. Hence the conditions for the successful completion of the induction melting are:

$$\delta \geqslant \Delta_p + 2\xi \tag{7}$$

and

$$\alpha_m \geqslant \delta, \tag{8}$$

where δ is the width of the melt gap, m; ξ, thickness of the shell, m; and α_m, depth of the molten bath, m.

A particular feature of melting in the gap is the divergence from Eq. (2), characteristic of melting ingots of complex cross section. In this case there is no limit to the maximum value of the parameter m and therefore the transverse dimensions of the ingot with a cavity are limited only by the power of the generator.

In the construction of the core, the following must be taken into account: a downward taper of roughly 1-10% caused by the shrinkage of the ingot as it cools; the possibility of collapsing across a section if the core is squeezed when the ingot crystallizes; the possibility of independent movement relative to the immobile crucible when selecting the optimum level of installation of the core; and the use of screens to protect the pipes bringing water to the core since these pass through the zone of radiation from the melt.

We melted shaped blocks under laboratory conditions in high-frequency equipment one of which had a power of 60 kW and a frequency of 5.28 MHz while the other had a rating of 120 kW at 1.76 MHz. In the first equipment we obtained rectangular mullite, corundum, and baddeleyite–corundum blocks of maximum section 200 × 220 mm and also pipe-shaped blocks and arched muffles of corundum with a maximum section of 220 × 250 mm and a wall thickness of 20 mm (Fig. 5). In the second equipment we melted blocks of magnesial spinel, forsterite, and mullite with a maximum section of 300 × 300 mm and length up to 1600 mm.

In order to prevent descruction by thermoelastic stresses, the blanks were heat treated or cooled slowly.

CONCLUSIONS

It is shown that the use of induction melting of oxide materials in a cold crucible makes it possible to achieve a high-productivity process for melting into blocks or with discharge for the production of fused material for industrial refractories and also to provide fused blanks for lining blocks and arched muffles.

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GRANULATION OF REFRACTORY OXIDES AND THE PROPERTIES OF THE GRANULES PRODUCED

I. G. Orlova, I. N. Rudyak,
N. M. Chudnova, N. M. Sharova,
and A. A. Grebenyuk*

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Spherical ceramic compounds are now becoming more and more widely used as heat-exchange compounds [1-4], milling compounds for milling refractory and other materials [5, 6], briquettes for the preliminary firing of crock [7], as a catalyst carrier in the chemical industry [8], etc.

The spherical compounds are made in presses of various design [1, 9], from plastic masses in roller-pressers [6, 10], and by casting from aqueous suspensions [11]. In order to obtain spherical compounds in the form of granules, a granulator is used as the shape-forming machine: this granulator is simple to make and use and has an excellent productivity. Using it we can obtain 5-30 mm granules made of nonplastic masses without any change or replacement of the molding element of the equipment. However, information on the granulation process is limited and deals mainly with iron-ore materials [12] and therefore the present article gives some results of a study of the granulation of Al_2O_3 and ZrO_2 powders and the properties of the granules so produced.

*Deceased.

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