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Electrical Conductivity Measurement of Oxides Melts

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Abstract

Notwithstanding on variety of existing processes of induction skull melting of oxides, electrical and thermal properties of oxides melts are practically unknown. From the other hand, liquid-phase synthesis of Hi-Tech oxides materials such as new ceramics, monocrystals and glasses, requires knowledge of melts properties up to 3700 K. Inasmuch as the main physical property, which influence to the melt power consumption at induction heating is electrical conductivity then study of electrical conductivity temperature dependence is necessary.

The principle and technical realization of the electrical conductivity measurement of melts are presented. The approach is based on induction melting in cold crucible technology and inverse electromagnetic problem solution. Analysis of the method accuracy via input data errors is presented. For increasing of Ill-posed problem solution was used over determination of set of equations. Conductivity of aluminum oxide melts for the temperature up to 3223 K is shown too.

Introduction

Oxides and its combinations form classes of major technical materials, which determine stage of development of energetic, metallurgy, mechanical engineering, communication and so on. Different processes of melting are widely used for oxides materials synthesis. However, induction melting in cold crucibles is beyond compare the best technology for producing high pure materials [1-3].

Method of induction melting of oxides in cold crucibles is known more than 40 years. But usually tasks of installations design and control of technological processes, which provide producing of high-quality materials are solving by empirical methods. From the other hand, deficiency of reliable data about electro- and thermophysical properties of oxides melts versus temperature limits of using of modern CAE systems [4]. Therefore study of high temperature melts properties, in particular electrical conductivity, is actual task.

For investigation of melt conductivity are commonly used contact methods [5, 6]. But all of them have essential limitation by temperature up to 2173 K when it takes to work in air atmosphere. To study of melts conductivity with temperature up to 3700 K in air there are approaches which are based on non-contact methods, but most of them is applied for metals [7-9]. There is attempt to estimate of electrical conductivity of oxides melts using parameters of induction melting in cold crucible [2], but suggested there approach requires measurement of inductor power factor and therefore this electrical conductivity measure method does not possess by well accuracy.

This present paper is the next stage of elaboration of noncontact method for estimation of electrical conductivity of high temperature melts [10-13]. Suggested method is based on induction melting in cold crucible technology with inverse solution of electromagnetic problem relative of electrical conductivity.

1. Inverse problem technique

Induction melting in cold crucible technology provides required temperature and chemical nonpolluting of the melt. At the same time, electrical and thermal parameters of induction system allow to judge about melt conductivity. On the base of our experience, power losses are more available from measurement technique point of view from them. Therefore inverse problem technique can use electromagnetic mathematical model of induction system and concept of the power balance:

$$\begin{cases} P_{bot}^{e}+P_{cc}^{e}+P_{me}^{e}+P_{cov}^{e}+P_{sh}^{e}=P_{bot}^{t}+P_{cc}^{t}+P_{cov}^{t}+P_{sh}^{t} \\ \begin{cases} 2\pi R_i \dot{E}_i + j\omega\int\limits_{S_1}\sigma_j \dot{E}_j M_{ij}dS + j\omega\int\limits_{S_2}\sigma_k \dot{E}_k\left(M_{ik}-M_{il}\right)dS = \dot{U}_{ind} \\ \dfrac{1}{\mu_0}\left(\dfrac{\partial^2 \dot{A}}{\partial R^2}+\dfrac{1}{R}\dfrac{\partial \dot{A}}{\partial R}+\dfrac{\partial^2 \dot{A}}{\partial z^2}-\dfrac{\dot{A}}{R^2}\right)-j\omega\left(\overline{\sigma}_{me}\right)_1\dot{A}=0 \end{cases} \end{cases}, \quad (1)$$

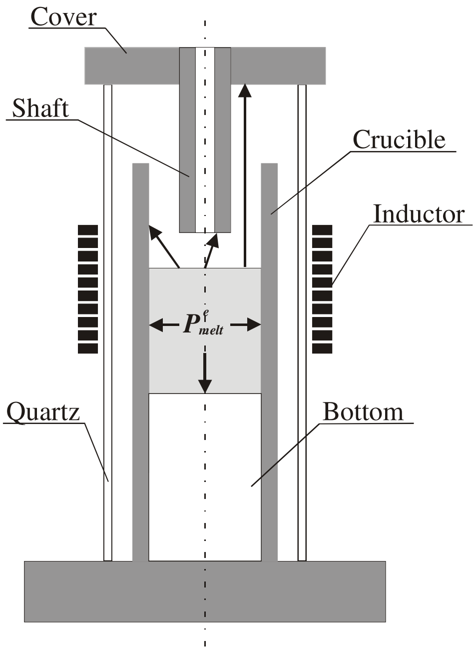

Fig. 1. Scheme of the induction system

were, P_{bot}^{t}, P_{cc}^{t}, P_{cov}^{t}, P_{sh}^{t} - calorimetry power in the bottom, crucible, cover and shaft; P_{bot}^{e}, P_{cc}^{e}, P_{me}^{e}, P_{cov}^{e}, P_{sh}^{e} - electrical losses in the bottom; crucible; cover; shaft and power in the melt, Fig. 1. Other symbols at (1) are generally accepted. It was used here mathematical model on the base of 2D integro-differential approach [14], but it is possible to use any available one. Fig. 2 illustrates us solution search at equation system (1). Commonly the system (1) has two decisions (points 1 and 2, see Fig. 2) where thermal balance condition is realized. To provide of one-valuedness solution is additionally used inductor current or power losses values as input data. As far as this method is formulated as inverse problem solution, it takes to estimate sensitivity of desired quantity from input data deviation. Thus it needs to estimate of accuracy of the electrical conductivity investigation method.

2. Accuracy of the method

Summarized error of the electrical conductivity investigation method includes errors of inverse problem approach δ_{ip} and mathematical model δ_{mat}:

$$\delta_{sum}=\delta_{ip}+\delta_{mat}.$$

At the same time error of inverse problem approach is:

$$\delta_{ip}=\left(2\delta_{cal}+\delta_{geom}\right)\cdot W_{ip},$$

were δ_{cal} - total error of calorimetry; δ_{geom} - measurement error of induction system geometry and W_{ip} - transfer function of the inverse problem. This transfer function is determined as:

$$W_{ip} = f\left(\left\{\frac{R_{melt}}{\Delta_{melt}}, \frac{H_{melt}}{H_{ind}}\right\}, \delta_{cal}, \delta_{el}, \delta_{geom}\right),$$

were R_{melt} - melt pool radius; Δ_{melt}- skin layer in the melt; H_{melt} - melt pool depth; H_{ind} - inductor height; δ_{el} - error of voltage or current measurement. The systematic error values and possible neglected losses, which can arise as a result of imperfection design of real induction system is presented at Tab. 1. Presence of hardware systematic errors produces of possible solution areas, Fig. 3. Analysis of inverse problem solution sensitivity via all input data deviations gives us dependence of inverse problem approach error, Fig. 4.

Tab. 1. Grading of errors

Kind	**Sign**	**Error, %**
Flow meter		0.5
Thermocouple or resistor temperature sensor		0.25
Analog-to-digit converter		0.1
Systematic error of calorimetry		0.85
Additional nonregistered power losses		1.2
Total error of calorimetry	δ_{cal}	**2.05**
Measurement error of induction system geometry	δ_{geom}	**2.5**
Inverse problem solution on the base of analytical electromagnetic mathematical model	δ_{ip}	**10 ÷ 35**
Numerical mathematical model	δ_{mat}	**5 ÷ 10**
Uniform of melt temperature distribution		not accounted
Total error of the method	δ_{sum}	**15 ÷ 45**

Results analyses of our physical and numerical experiments show that power sources in oxides melt are distributed in area without heavy temperature gradients. From the other hand buoyancy convection decreases the temperature gradients too and these phenomena allows us to put assumption about uniform electrical conductivity of oxides at the melt pool.

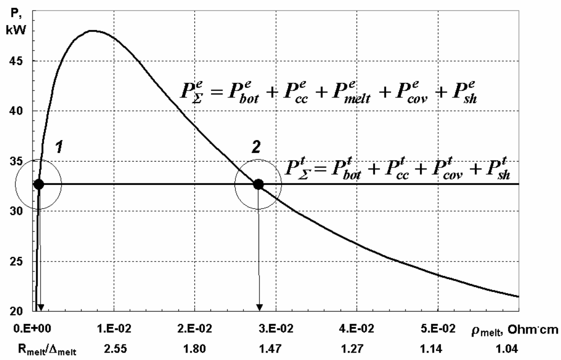

Fig. 2. Search of solution on the base of thermal balance

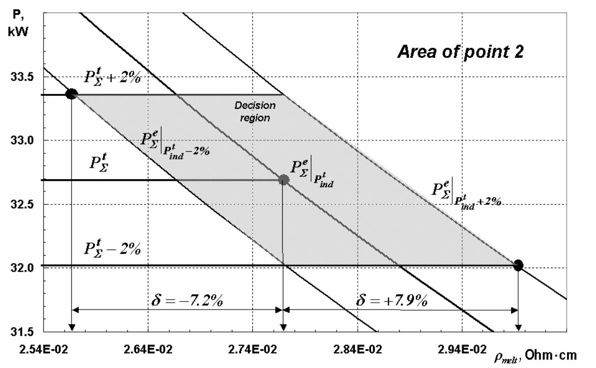

Fig. 3. Decision region around point 2

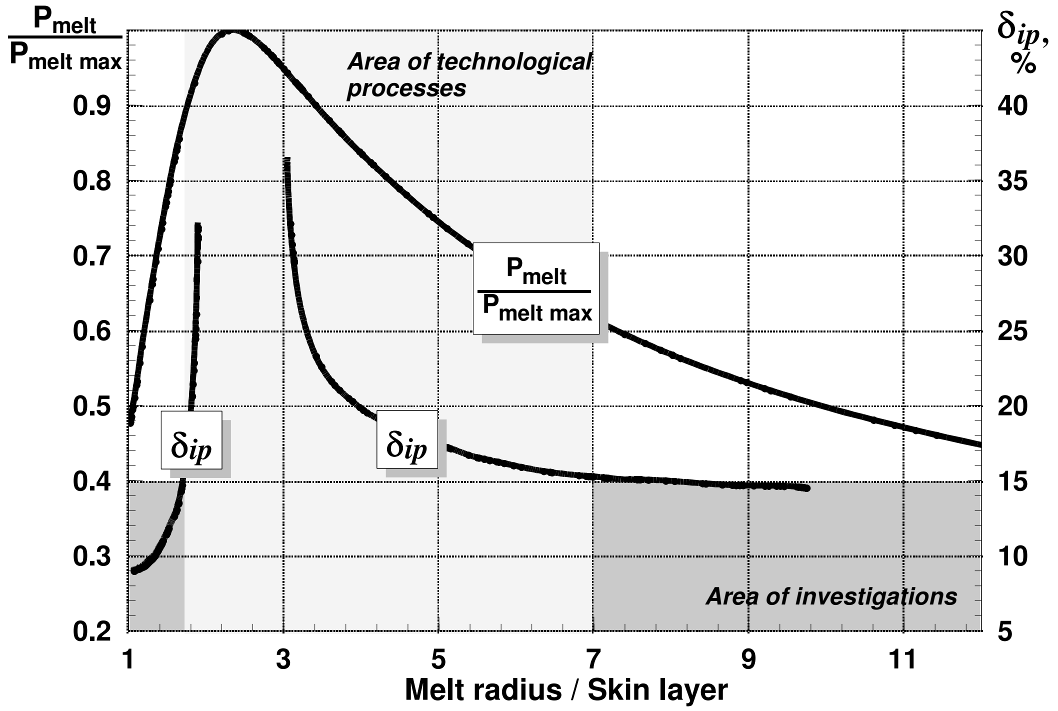

Fig. 4. Inverse problem solution error

3. Results

For estimation of electrical conductivity value of aluminium oxide melt series tests were done. Basic dimensions of induction systems, experimental data and results of numerical analysis tree of tests are presented in Tab. 2. The values of the melt electrical conductivity are related to the temperature on the melt surface.

Tab. 2. Experimental and calculations data

Basic dimensions, cm	T^{*}_{me}, K	f, MHz	U_{ind}, kV	I_{ind}, A	$P^{t}_{cc}+P^{t}_{cov}$, kW	P_{ind}, kW	σ, (Ohm× cm)$^{-1}$	$\frac{R_{melt}}{\Delta_{melt}}$	δ_{sum}, ±%
H_{melt}=11.1 R_{melt}=7.6 H_{ind}=8.6 R_{ind}=11.0 W=3	2573	1.853	-	-	37.71	0.90	0.47	1.41	16.53
H_{melt}=5.0 R_{melt}=3.30 H_{ind}=9.30 R_{ind}=12.6 W=4	2953	1.744	5.316	316	-	3.30	1.00	0.87	17.36
H_{melt}=7.20 R_{melt}=2.65 H_{ind}=6.50 R_{ind}=4.70 W=5	3223	1.830	-	-	19.79	2.22	1.64	0.91	16.21

* Brightness temperature (λ=0.65 μm).

Reasoning from that oxide aluminium melt is characterized by ionic kind of electrical conductivity and for the overheating of the melt up to 150-200 K above the melt temperature, number of charge carriers as simple ions is became much more than complex one [15]. So, it can be assumed that the electrical conductivity is thermally activated and varies according to the equation:

$$\sigma = \sigma_0 \cdot e^{-\frac{E_\sigma}{RT}}, \qquad (2)$$

where σ_0 preexponential factor, R - the gas constant, E_σ - activation energy of electrical conductivity and T - temperature.

Activation energy is determined as:

$$E_\sigma = \frac{d(ln(\sigma))}{d(T^{-1})}.$$

Fig. 5 shown that activation energy $E_\sigma = const$ with a fair accuracy. It allows us to find all coefficients at equation (2) as the next: σ_0 =243 Sm/cm; E_σ =134496 J·mole; R =8.314 J·mole/K.

The whole set of data include the results obtained from the participants on the three samples from the measurements at three temperatures. These tests results end exponential relation of electrical resustivity of aluminium oxide are plotted in Fig. 6.

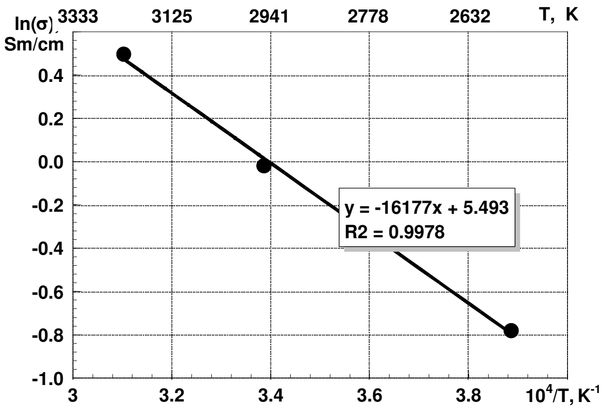

Fig. 5. Variation of the melt electrical conductivity and its approximative representation by linear dependence

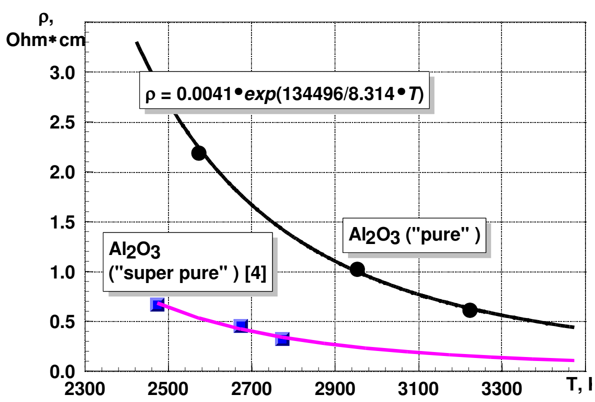

Fig. 6. Electrical resistivity of aluminium oxide different purity via temperature

CONCLUSION

For estimation of electrical conductivity of oxides melts on the base of induction furnace with cold crucible the complex of hard and software was designed. Noncontact investigation of melts electrical conductivity of oxides, carbides, nitrides, borides, metals and so on in wide temperature range is advantage of the proposed method.

This method can be applied to investigation of melt singular points too [16, 17].

References

[1] Petrov Yu. B., Ratnicov D. G. "*Cold Crucibles*" - M.: Metallurgy, 1972. (Russian).

[2] Petrov Yu. B., "*Induction melting of oxides*" - M.: Metallurgy, 1972. (Russian).

[3] Petrov Yu. B., Kanaev I. A."*Induction furnaces for melting of oxides*" - L.: Polyteknica, 1991. (Russian).

[4] C. Gross, W. Assmus, A. Muiznieks et al. "*Power Consumption of skull Melting, Part II: Numerical Calculation of the Shape of the Molten Zone and Comparizon with Experiment*" // J. Cryst. Res. Technol., 1999, vol. 34, pp. 329 - 338.

[5] M.Constantinescu, G. Jitianu, M.Brichi, C.Vuap, M.Jlteanu. Properties of some glass-ceramics in molten and solid state. Romanian academy, Institute of physical chemistry "I.G.Murgulescu" Spl.Independentei 202, Bucharest 77208, Romania. High tmperature Naterial Procesecc 6 (2002) p. 75-86.
[6] A. Silnqa and B. Haugsdal. Electrical conductivity measurements of corrosive liquids at high temperatures. Department of Chemistry, University oj'Os10, P. 0. Box 1033, 0315 Oslo, Norway/Rev. Sci. Instrum. 64 (2), February 1993, P 532-537.
[7] Yuu Ono, Cheng-Qing Zheng, Francois G. Hamel et al. "*Experimental investigation on monitoring and control of induction heating process for semi-solid alloys using the heating coil as sensor*" // J. Meas. Sci. Technol., 2002, vol. 13, pp. 1359 - 1365.
[8] Won-Kyu Rhima) and Takehiko Ishikawa. Noncontact electrical resistivity measurement technique for molten metals. *Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California 91109.* REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 69, NUMBER 10 OCTOBER 1998.
[9] Georg Loh.ofer. Electrical resistivity measurement of liquid metals. Meas. Sci. Technol. 16 (2005) 417–425.
[10] Pozniak, A. Pechenkov. "*Special tools for investigation and controlling of induction skull melting processes*". Proceedings of the Int. Colloq. "Modelling for Saving Resources" // Riga, Latvia, May 2001, pp. 158 - 163.
[11] I. Pozniak, A. Pechenkov. "*An Approach of Electrical Conductivity Estimation of Technical Materials Melts*" // 8th Int. Conf. on Microwave and High Frequency Heating. Bayreuth, Germany, September 3-7, 2001.
[12] I.V. Pozniak, A.Yu. Pechenkov, A.N. Shatunov. "Induction Furnace with Cold Crucible as a Tool for Investigation of High Temperature Melts". 9th KOREAN-RUSSIAN INTERNATIONAL SYMPOSIUM ON. Science & Technology / KORUS 2005, June 26 ~ July 2, 2005 Novosibirsk State Technical University , Novosibirsk, Russia.
[13] I.V. Pozniak, A.Yu. Pechenkov, A.N. Shatunov, Yu.I. Blinov. "Inverse techniques for analyzing and control of cold crucible induction melting".Proceedings of the Int. Colloq. ACTUAL PROBLEMS OF THE INDUCTION HEATING (THEORY&APPLICATIONS) APIH-05. St.Petersburg, 24 -26 May, 2005.
[14] I. Pozniak, A. Pechenkov, B. Nacke and T. Behrens. "*Mathematical Modelling and Investigation of Induction System with Slitted Crucible*" // Proceedings of the Int. Scient. Colloq. "Modelling for Electromagnetic Processing", Hannover, Germany, March 24-26, 2003, pp. 151 - 156.
[15] Bentley R. E, *Jones T.P.* - J. Amer. Ceram. Soc., 1975, v. 11, №2, p. 30-36.
[16] Sung S. Kim, Thomas H. Sanders. "*Thermodynamic Modeling of the Isomorphous Phase Diagrams in the* Al_2O_3–Cr_2O_3 *and* V_2O_3–Cr_2O_3 *Systems*" // J. Am. Ceram. Soc., 84 [8], pp.1881–84 (2001).
[17] Skrybin V. G., Novokhatsky I.,Usatuk I. I. "*About structure trancformation in ionic melts*".// III All Russian conf. "Structure and properties of metal and slag melts". Part. 3. "Investigation of slag melts". Sverdlovsk, 1978, pp. 128-131. (Russian).

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