REDUCING THE FIRING DEFORMATION OF CORUNDUM CERAMICS

L. A. Dergaputskaya and I. G. Orlova

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The production of vacuum-tight corundum ceramics of complex configuration entails difficulties owing to their considerable shrinkage and deformation during firing. It has been shown [1] that it is possible to produce a corundum ceramic with an open porosity of 17-18% before firing and a firing shrinkage of 6-7%. The interrelation between the shrinkage and deformation [2] suggests that a decrease in the shrinkage of the ceramic during high-temperature firing will result in a corresponding decrease in deformation.

Experimental confirmation of this theory was sought by an investigation of the kinetics of the shrinkage, deformation, and change in porosity over the temperature range 1300-1750°C for three types of corundum ceramic produced by different methods.

The starting material for all specimens was fine-ground alumina with added magnesium oxide (0.1-0.2%). In production version I, the alumina was calcined at 1550°C, and in version II and III at 1750°C. All specimens were fired at 1300°C. In version III, the specimens were impregnated (after firing at 1300°C)

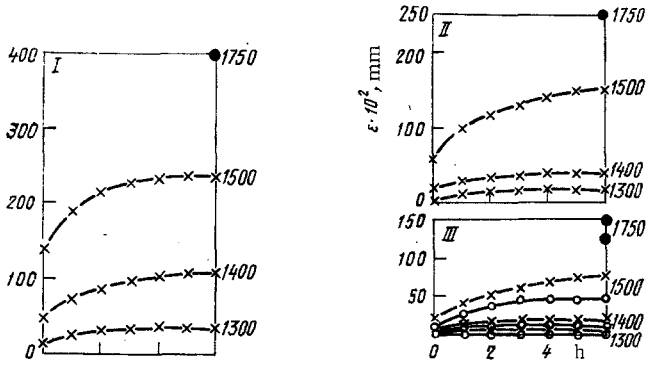

Fig. 1. The kinetics of the deformation % over 1300-1500°C of corundum specimens produced in versions I, II, and III. The firing temperature, °C, is stated against each curve. The deformation at 1750°C is that after isothermal holding for 6 h.

TABLE 1. Characteristics of Corundum Specimens Produced in Three Different Versions

Production version	Production method	Properties of the specimens after firing at 1300°C	
		open porosity, %	apparent density, g/cm^3
I	Cast from alumina calcined at 1550°C	33,0	2,60
II	Cast from alumina calcined at 1750°C	25,0	2,85
III	Cast from alumina calcined at 1750°C and impregnated	14,0—22,0	3,15—2,95

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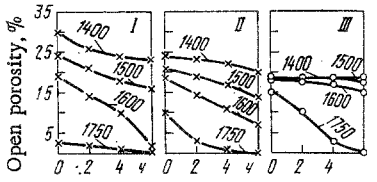

Fig. 2

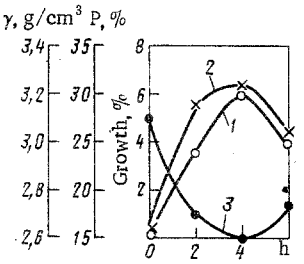

Fig. 3

Fig. 2. The kinetics of the decrease in the open porosity P of corundum specimens produced in versions I, II, and III. The firing temperature, °C, is stated against each curve.

Fig. 3. The variation of the growth (1), open porosity P (2), and apparent density γ (3) of specimens of version III with the holding time at 1400°C. The specimens were impregnated with a salt containing aluminum and potassium.

with melts of various aluminum-containing salts followed by the dissociation of the latter. The result was that the initial porosity of the specimens fired at 1300°C differed significantly and was at minimum for specimens produced by version III (Table 1).

The kinetics of sintering was studied over the temperature range concerned on cylindrical specimens 12 mm in diameter and 20 mm in height. The kinetics of the deformation of the specimens under their own weight over 1300-1500°C was investigated by a known method [3] on bars measuring 5 × 5 × 80 mm. The deformation at 1750°C was expressed in terms of the deflection of similar bars placed on corundum supports the distance of which was that used by Kainarskii et al. [3]. The specimens were heated in these experiments to 1750°C in an open-flame surface with a holding time of 6 h.

The method of producing the specimens influences their firing shrinkage and deformation to a significant extent. For specimens produced from alumina calcined at 1750°C the shrinkage during holding at various temperatures over 1400-1750°C decreases from 2.8-12% to 1.5-9.1%, and the deformation from 1.1-4.0 mm to 0.2-2.5 mm (Table 2, Fig. 1). The impregnation of the specimen results in a further decrease in the shrinkage to 6.5-8.4% at 1750°C and in the deformation to 1.25-1.50 mm. The deformation is thus reduced by a factor of two and a half as a result of the change in the production technology.

The ultimate open porosity of the specimens was zero regardless of the production method but the rate of decrease in the porosity is at maximum at the higher temperatures (Fig. 2).

Like the increase in the open porosity at the start of isothermal heating at 1400°C, this phenomenon is probably connected with the continuing elimination of the volatile components of the aluminum-containing salt (more particularly, K_2O) at 1300-1600°C. In the case of certain salts used as additive the specimens grew by up to about 6% and the porosity increased and the density decreased appreciably during isothermal holding at 1400°C (Fig. 3). This process reached its development peak after 4 h holding following which sintering set in. When deciding the technological parameters account had to be taken of the fact that a

TABLE 2. The Production Method as a Factor in the Shrinkage, Deformation, and Open Porosity of the Specimens after 6 h Holding at Temperatures from 1300 to 1750°C

Firing temp., °C	Properties of specimens produced by versions (see Table 1)								
	I			II			III		
	γ, %	ε·10², mm	P, %	S, %	ε·10², mm	P, %	S, %	ε·10², mm	P, %
1300	—	32,4	33,0	—	19,7	25,0	—	3,6—7,2	17,5—22,5
1400	2,8	110,0	24,0	1,5	41,4	20,8	0,5—1,1	10,8—21,6	19,0—21,2
1500	6,5	238,0	16,0	4,5	150,0	14,4	1,0—1,9	36,0—75,6	17,7—17,6
1600	9,5	—	1,5	7,6	—	7,6	2,6—6,0	—	14,8—8,5
1750	12,0	400,0	0	9,1	250,0	0	6,5—8,4	125,0—150,0	0

*S) Shrinkage, ε) deformation, and P) open porosity.

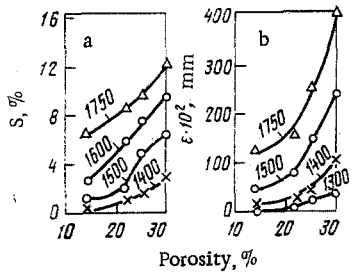

Fig. 4

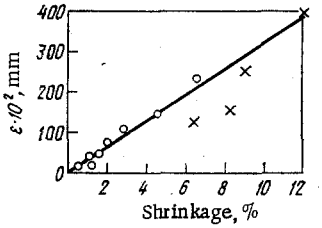

Fig. 5

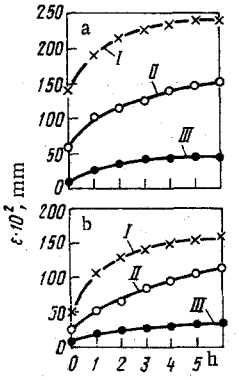

Fig. 6

Fig. 4. The porosity of the intermediate product as a factor in the shrinkage S (a) and deformation ε (b) of corundum specimens fired at 1300-1750°C. The firing temperature, °C, is stated against each curve.

Fig. 5. The interrelation of the shrinkage and deformation ε of the corundum specimens: ○) deformation data obtained over the temperature range 1300-1500°C by a known method [3], ×) deformation expressed in terms of the deflection after firing at 1750°C.

Fig. 6. The effect of firing in stages on the kinetics of the deformations at 1500°C of specimens of versions I, II, and III: a) direct heating, b) heating with a step at 1400°C.

higher temperature and the impregnation with salts containing aluminum and potassium may result even in the destruction of the specimens.

As expected, the porosity of the intermediate product proved to be the principal factor in the influence of the production technology on the shrinkage and deformation of the specimens. A decrease in this porosity results in a sharp decrease in the firing shrinkage and deformation (Fig. 4) a finding which is confirmed by the known interrelation [2] of these two processes (Fig. 5).

It has been shown [2] that firing in stages is an effective method of reducing deformation. In the present investigation too the deformation after firing at 1500°C was considerably lower when the temperature

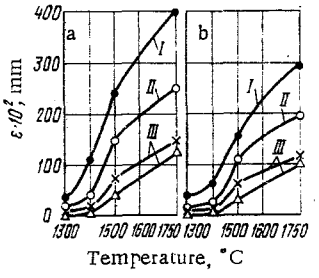

Fig. 7

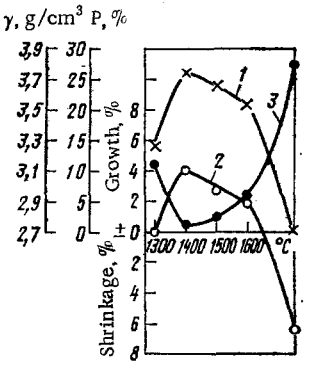

Fig. 8

Fig. 7. The temperature dependence of the deformation ε of specimens of versions I, II, and III in direct (a), and stepwise (b) heating: ×) specimens impregnated with ammonia alum, Δ) specimens impregnated with a 1/3 mixture of potassium alum and ammonia alum.

Fig. 8. The variation of the porosity P (1), linear dimension (2), and apparent density γ (3) of specimens of version III with the temperature after 6 h isothermal holding in stepwise heating. The specimens were impregnated with a salt containing aluminum and potassium.

was increased with a steps of 100°C (Fig. 6), i.e., 37, 27, and 30% for specimen versions I, II, and III, respectively.

When specimens are fired in a single stage to 1500°C and then again fired at 1750°C their deformation is also lower (by 21-27%) than in direct heating at 1750°C (after calcining at 1300°C) (Fig. 7).

Firing in stages prevents the cracking of specimens impregnated with certain salts but the specimens continue to grow to 1400°C and resume their original dimensions only at 1650°C as a result of sintering which sets in at 1400°C (Fig. 8). The porosity of specimens fired at 1600°C remains very high, however (about 20%) and it decreases to zero only after firing at 1750°C while the density increases to 3.8 g/cm^3.

The guideline when choosing the aluminum-containing salt should not be merely the amount by which the porosity of the intermediate product can be reduced but also the volumetric changes in the subsequent heating process. The best salt is one which produces no increase in the volume when it dissociates.

This investigation showed that the use of alumina calcined at 1750°C for the production of the ceramic developed at the Ukrainian Scientific-Research Institute for Refractories, and the impregnation of the intermediate product with an aluminum-containing salt makes it possible to reduce the deformation to half of that of specimens produced by the usual technology for a ceramic of this type. Firing in stages also helps to reduce deformation (by 21-27%) but to a considerably lesser extent than the change in the method of producing the specimens.

This method should be recommended for use in the manufacture of thin-walled complex-shape corundum products requiring a high degree of precision in dimensions and configuration.

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