PROPERTIES OF SINTERED SILICA FUME

Ján Majling, Peter Znášik, *Ivan Baník, *Juraj Veselský, Zdenek Hrabě

Department of Ceramics, Slovak Technical University, Radlinského 9, 812 37 Bratislava, Slovakia
*Department of Building Physics, Slovak Technical University, Radlinského 11, 813 68 Bratislava, Slovakia

Condensed silica fume – a by-product from silicon alloys industry is shown to be an interesting precursor to prepare highly porous, shaped ceramic articles. Compacts of silica fume of different green densities were sintered at 1000 °C. Thermal conductivity of received bodies ranges from 0.07 to 0.208 $W\ m^{-1}K^{-1}$, the compression strength ranges from 1.2 to 53.0 MPa, respectively.

INTRODUCTION

Condensed silica fume is a voluminous, high tonnage by-product in manufacture of pure silicon, or ferrosilicon alloys. It is utilised mostly as an active component in the preparation of concrete mixtures. It greatly improves the density, the mechanical strength and other properties of concrete structures [1, 2].

Silica fume is a precipitate from a gas phase what basically defines its properties. While not taking into account its chemical purity, which is, as of the technical product not too high, the silica fume fulfils nearly all othe very stringent requirements imposed on hightech ceramic powders. Literature data show that the condensed silica fume of the present origin consists of non-aggregated, amorphous particles of a spherical shape with the particle size <0.5 μm [3].

Sintering of powder compacts of silica fume with varying green densities is described in [4] with a particular reference to the rate of heating. The condensed silica fume with respect to the glassy nature of primary particles is expected to behave similarly at sintering as the silica xerogels [5, 6], of casted bodies of glass powders [7, 8]. The sintering will be accomplished by viscous flow. No information however is available in the literature as to the processing of dry silica fumes to low density compacts in order to prepare ceramic shaped articles.

In this contribution silica fume compacts were prepared at different (low) pressures and sintered at 1000 °C. Resulting bodies are characterized by their porosity, strength, and thermal conductivity.

EXPERIMENTAL

The condensed silica fume from the metalurgical plant in Mníšek pod Brdy (Czech Republic; SiO_2>98%, Al_2O_3, K_2O, CaO, MgO and Na_2O as the main oxide impurities) has been used as a starting material. The as received (dry) powder was primarily compacted in a steel die to square slabs (of a constant weight) of the edge of 50 mm and of the preselected height. The pressure at which compacts

were formed is expressed in relative volumes (V/V_0) to which silica fume was compressed from its loosely packed (tap) structure (V = pressed sample volume, V_0 = tap volume). The linear shrinkage of the separate sample was measured in the dilatometer (Netzsch 402 E) at a constant rate of heating (5°C/min) to derive the temperature for the isothermal sintering runs. All samples were in the next heated isothermally at 1000 °C in an electric muffle furnace in the static air.

The strength of samples was measured on spherical specimens, 10 mm in diameter, cut out of sintered bodies. The test specimens were encapsulated into flexible latex covers. The collapse of the skeletal structure of samples under the isostatic compression was measured via the Carlo Erba 1520 mercury porosimeter. The details of the adopted procedure of measurement are given in [9].

The thermal conductivity of samples was measured by a steady-state method at a laboratory temperature. A thermal flow density passed through a sample and the temperature difference on the sample were measured. The thermal conductivity of the sample was calculated from the measured data and the thickness of the sample [10].

The bulk density was calculated from the geometry and weight of samples. Their total porosity was calcualted using the true density of the material, which was approximated by the density of pure silica glass ($\rho = 2.2$ Mgm^{-3}).

RESULTS AND DISCUSSION

The dilatometric curve is given in Fig. 1. The linear shrinkage of the sample is accomplished in a relatively narrow temperature interval. Taking this dependence into account the fume compacts were heated at 1000 °C for 10, 20, 40 and 80 minutes, respectively.

The compaction of silica fume resulted in green bodies of well defined geometry and of smooth faces. The compacts received at the lowest pressure (V/V_0 = 0.59) had however a low manipulative strength.
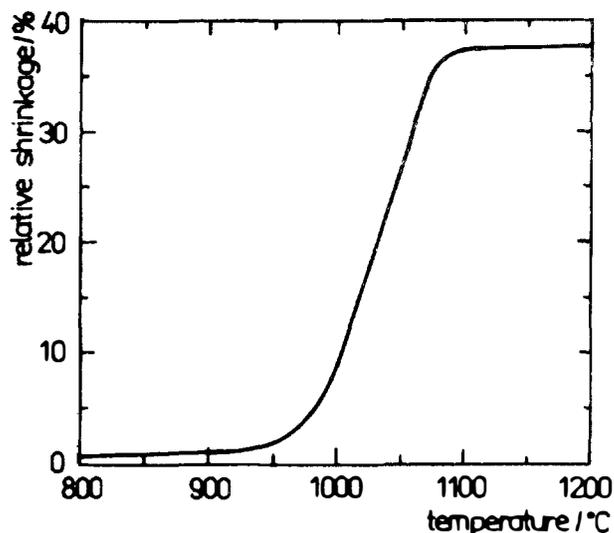
Fig. 1. Dilatometric Curve of the Silica Fume Sample.

Table I.

The Bulk Densities of Sintered Compacts ($Mg\ m^{-3}$).

Time of sintering / min	V/V_0			
	0.59	0.51	0.42	0.34
10	0.334	0.337	0.435	0.537
20	0.342	0.378	0.450	0.542
40	0.434	0.392	0.492	0.671
80	0.492	0.592	0.655	0.845

Table II.

The Thermal conductivity of Sintered Compacts at room Temperature ($W\ m^{-1}K^{-1}$).

Time of sintering / min	V/V_0			
	0.59	0.51	0.42	0.34
10	0.075	0.071	0.090	0.096
20	0.072	0.086	0.089	0.099
40	0.091	0.091	0.109	0.129
80	0.102	0.122	0.142	0.208

Table III.

The Bulk Porosity of Sintered Compacts (%)

Time of sintering / min	V/V_0			
	0.59	0.51	0.42	0.34
10	84.9	83.0	80.3	75.5
20	84.5	82.9	79,6	75.5
40	80.4	82.3	77.8	69.6
80	77.7	73.2	70.4	61.8

The bulk densities of sintered compacts are given in Table I, the thermal conductivity at room temperature is given in Table II, the porosity of samples in Table III and their compressive strengths in Table IV. The non-standard method to determine the strength in compression was applied in this case because the highly porous bodies with fine pores and homogeneous pore distribution are especially suitable for this kind of measurement. The results on the absolute scale are comparable to the results received in axial compression tests. From given data, the particular dependencies have been generated graphically which are shown in Figs. 2-4. The respective functional surfaces were not further numerically smoothed.

The densification of samples comes out essentially from two different sources, from the mechanical packing of particles by an external pressure to receive the green structure and from the rearrangement of particles by the viscous flow during sintering. The quan-

titative description of the densification process and changes in the pore size distribution is in the course of the further study.

The strength of sapmles correlates with their bulk densities. In the area of low primary compaction ($V/V_0 \sim 0.59$, 0.51) and short sintering time (10, 20, 40 min.) it is very low and the material has only a marginal manipulative strength similar to the manipulative strength of the green body (Fig. 3). The increase in strength is more pronounced either at higher compaction, or at longer periods of sintering.

The surface of the Fig. 3 is more or less concave what means that the densification mechanisms are not "mutually supportive" in the strength increase.

The superposition of changes of the total porosity and changes of the thermal conductivity is given in Fig. 4. In comparing thermal conductivites along individual lines of equal porosity, it can be seen that thermal conductivites of samples at which the bulk density was attained at lower primary compression and longer heating tend to be always higher than thermal conductivities of more strongly compressed

Table IV.

The Compressive Strength of Sintered Compacts (MPa).

Time of sintering / min	V/V₀			
	0.59	0.51	0.42	0.34
10	1.2	1.2	2.7	5.0
20	1.3	1.4	3.7	9.9
40	2.5	2.8	4.0	11.3
80	7.7	12.0	24.0	53.0
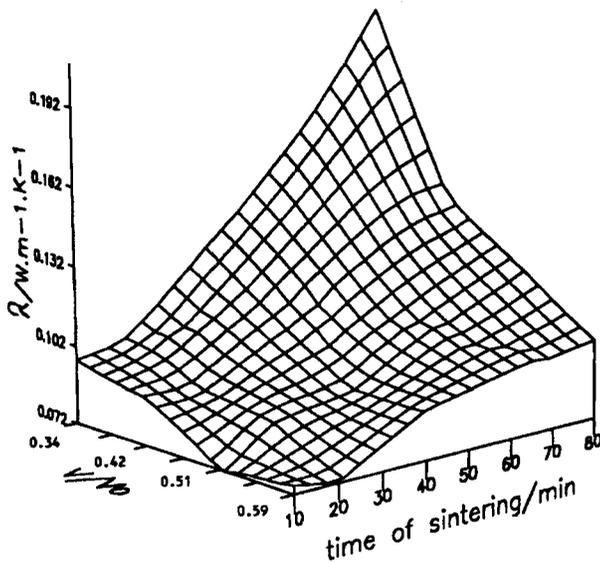
Fig. 3. *Strength of Sintered Compacts in Dependence on Time of Sintering and Primary Compression.*
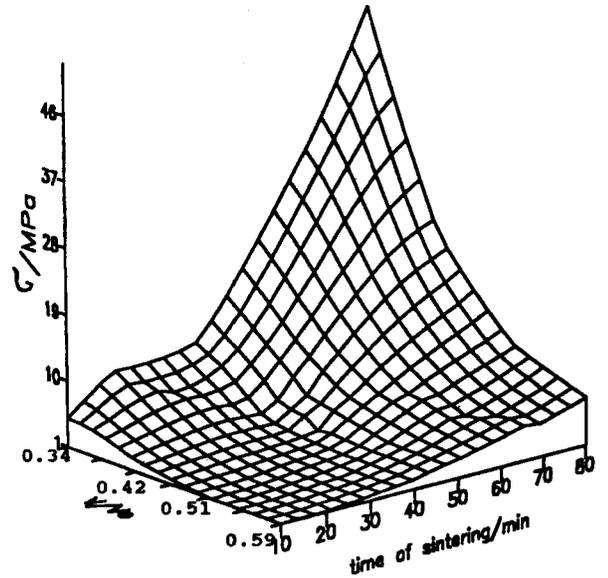
Fig. 2. *Thermal Conductivity of Sintered Compacts in Dependence on Time of Sintering and Primary Compression.*
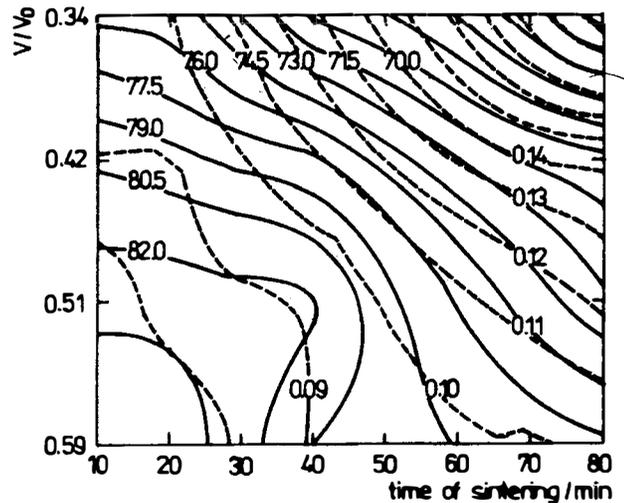
Fig. 4. *Superposition of Changes of the Total Porosity and Changes of the Thermal Conductivity of Sintered Compacts in Dependence on Time of Sintering and Primary Compression (————— – Total Porosity, %, —— —— —— – Thermal Conductivity, $Wm^{-1}K^{-1}$).*

samples heated for a shorter time. The densification, mechanisms then contribute unequall to the porosity distribution what is likely to have an interesting implication for the technology of these materials.

CONCLUSIONS

The behaviour of silica fumes at their compaction and sintering densification was studied. It is expressed by changes in the bulk density, thermal conductivity, and the mechanical strength of sintered bodies with respect to their processing conditions.

The results show that sintered compacts with thermal conductivity at the level of 0.01 $Wm^{-1}K^{-1}$ have

a moderate strength (4–5 MPa). The highly sintered compacts with a strength in the interval of 25–50 MPa have thermal conductivity at a level of 0.15–0.20 $Wm^{-1}K^{-1}$.

The received materials have an advantageous strength at their relatively high porosity. They may have therefore interesting engineering properties.

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VLASTNOSTI SPEKANÉHO KREMIČITÉHO ÚLETU

JÁN MAJLING, PETER ZNÁŠIK, *IVAN BANÍK, *JURAJ VESELSKÝ, ZDENEK HRABĚ

Katedra keramiky, skla a cementu, Slovenská technická univerzita, Radlinského 9, 812 37 Bratislava
**Katedra stavebnej fyziky, Slovenská technická univerzita, Radlinského 11, 813 68 Bratislava*

Kremičitý úlet je vedľajším produktom pri výrobe čistého kremíka a ferosilíciových zliatin. Pozostáva z neagregovaných, amorfných častíc guľovitého tvaru s veľkosťou <0.5 μm. Cieľom práce bolo overenie lisovateľnosti kremičitého úletu (Mníšek pod Brdy; $SiO_2 > 98\%$, Al_2O_3, K_2O, CaO, MgO a Na_2O ako hlavné oxidické prímesy) pri relatívne nízkych lisovacích tlakoch a charakterizácia niektorých dôležitých vlastností spekaných kompaktov. Zlisované telieska presných geometrických tvarov boli izotermicky spekané pri teplote 1000 °C po dobu 10, 20, 40 a 80 minút. Získané spekané kompakty sú charakterizované objemovou hmotnosťou, tepelnou vodivosťou a pevnosťou.

Výsledky ukazujú, že spekané kompakty s tepelnou vodivosťou na úrovni 0.10 W $m^{-1}K^{-1}$ majú pevnosti v tlaku okolo 4–5 MPa. Telieska s pevnosťami v intervale 25–50 MPa majú tepelnú vodivosť na úrovni 0.15–0.20 $Wm^{-1}K^{-1}$. Získané materiály sú vysoko pórovité, majú zaujímavé úžitkové vlastnosti a môžu byť východiskom pre alternatívne využitie kremičitého úletu.

Obr. 1 Dilatometrický záznam vzorky kremičitého úletu.

Obr. 2. Závislosť tepelnej vodivosti spekaných kompaktov od času spekania a primárneho zhutnenie lisovaním.

Obr. 3. Závislosť pevnosti v tlaku spekaných kompaktov od času spekania a primárneho zhutnenia lisovaním.

Obr. 4. Superpozícia zmien celkovej pórovitosti a zmien tepelnej vodivosti spekaných kompaktov v závislosti od času spekania a primárneho zhutnenia lisovním (——————— - celková pórovitosť, %, —— —— —— – tepelná vodivosť, $Wm^{-1}K^{-1}$).