STUDY OF SINTERING OF CERAMICS
BY MEANS OF HIGH-TEMPERATURE DILATOMETRY

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The kinetics of sintering of ceramic materials based on alumina, zirconia and of their composites was studied by means of the high-temperature dilatometer. It was found that the alumina-based ceramics sintered at higher temperatures than those based on zirconia, that at a heating rate of 10 °C min⁻¹ the resultant theoretical densities were comparable to those obtained at rates of 5 or 2 °C min⁻¹ , and that the resultant relative densities were higher with materials prepared by cold isostatic pressing than with those prepared by injection moulding. With the alumina and zirconia-based materials, the values of linear thermal expansion coefficients were identical with those specified in the literature.

INTRODUCTION

Sintering is a significant part of preparation of ceramic materials. Use of the high-temperature dilatometer for studying this process allows the course of expansion (or shrinkage) of the ceramic specimen to be followed in terms of temperature and time.

High-temperature dilatometry can also be employed in determining the coefficient of linear thermal expansion of sintered ceramics, which is a significant factor for example when different types of ceramic materials are joined.

The present study had the aim to describe the course of sintering of 4 types of ceramics based on alumina or zirconia, and of their composites (prepared by cold isostatic pressing and injection moulding) by means of high-temperature dilatometry, and to determine the linear thermal expansion coefficients of the materials after sintering.

EXPERIMENTAL PART

The following types of ceramic powders were used in the preparation of the ceramic specimens:

– powdered Al_2O_3, type RC-HP DBM (Reynolds Chemicals), abbreviated designation Rey,
– a composite of 85 wt.% Al_2O_3 - 15 wt.% ZrO_2, type ZTA 85 (Daiichi Kigenso), abbreviated designation ZTA,
– a composite of 80 wt.% ZrO_2 - 20 wt.% Al_2O_3, type ATZ 80 (Daiichi Kigenso), abbreviated designation ATZ,
– powdered ZrO_2 (stabilized with 5.15 wt.% Y_2O_3), type TZ-3YS (Tosoh). This type was employed for injection moulding and designated Tos-IM,
-- powdered ZrO_2 (stabilized with 5.15 wt.% Y_2O_3), type TZ-3YB (Tosoh). This type was employed for isostatic pressing and designated Tos-CIP.

The mean particle sizes were 0.6 μm (Rey), 0.7 μm (Tos-IM and ZTA) and 1.45 μm (ATZ) (figure 1). The particle size distribution of Tos-CIP powder was not measured because this powder, designed specifically for isostatic pressing, contained about 3.7 wt% of acrylic binder. Electron microscopy has confirmed the manufacturer's specification in that this powder consists of granules of 20 - 50 μm in size. The powder used for preparing these granules had a mean particle size of 0.8 μm (established on the Horiba analyzer).

The injection moulding of the materials was effected on the Allrounder 220M (Arburg) injection moulder. The ceramic suspensions were prepared with a content of 52.5 vol.% (ZrO_2) to 60 vol.% (Al_2O_3) of the solid phase. The technology of preparation of the specimens is described in [1]. Following extraction of the binder, the specimens were annealed in air atmosphere at 800 °C for 1 hour. The dilatometric specimens had the form of prisms (3.6mm × 4.7mm × 60mm) and cylinders (dia. 5.7 mm × × 60 mm). The injection-moulded specimens are designated here by the abbreviation IM, namely Rey-IM, ZTA-IM, ATZ-IM and Tos-IM.

The isostatic pressing of ceramic powders was carried out on the IP 4-22-60 press (Autoclave Engineering, Inc.). Except for the Tos-CIP type, the ceramic powders were first ground in a ball mill with an addition of 2.5 wt.% stearin for 24 hours. The press moulding took place under the pressure of the liquid medium (distilled water) 250 MPa, for one minute. Each mould contained two pressings in the form of prisms 120 mm × 60 mm × 15 mm in size. The green pressings were dried in air atmosphere at a temperature gradient of

10 °C hour^{-1} up to 800 °C and then annealed at 800 °C for 1 hour. The specimens prepared by cold isostatic pressing are designated here by the abbreviation CIP, namely Rey-CIP, ZTA-CIP, ATZ-CIP and Tos-CIP.

High-temperature dilatometry was performed on the dilatometer L70/1700 (Linseis) with vertical sample orientation. The specimen length was 15 – 17 mm and its maximum transverse dimension was 5.7 mm. The specimens were heated in a graphite furnace operating under a slight overpressure of nitrogen. The measuring system was of alumina and the specimens were placed in a sealed alumina tube in an air atmosphere. The heating was effected by three different temperature increase rates (10 °C min^{-1}, 5 °C min^{-1}, 2 °C min^{-1}) up to the maximum temperature (1530 °C for 2 hours for Rey and ZTA specimens, 1500 °C for 2 hours for ATZ and Tos specimens).

Electron microscopy of the sintered ceramic materials was carried out on the Jeol JX 840 electron microscope. The specimens were first polished and then thermally etched to visualize the grain boundaries (1400 °C for 10 min (Tos and ATZ), 1450 °C for 30 min (Rey and ZTA)).

The relative density of the ceramic materials was determined in three ways: by measuring the dimensions and weighing the specimens (method 1) [2], by measuring water absorption (method 2) [2] and by the double weighing method [3]. The former two methods were used on specimens before dilatometric measurement, the last on sintered ceramic specimens after dilatometry.

The grain size of sintered ceramic materials was determined according to Czech standard ČSN 42 04 63 [4]. Use was made of the linear method based on establishing the grain size according to the number of grains in a unit area of polished sample.

A significant part of the results were obtained by evaluation of dilatometric curves. The dilatometer employed recorded the values of expansion (or contraction) of the specimen (ΔL (mm)) in terms of time t and temperature T. By means of a program software the values were recalculated to a dependence of relative expansion $\varepsilon = \Delta L / L_0 \times 100$ (%), where L_0 (mm) is the initial specimen length) on temperature T (°C). The following quantities were evaluated by means of the curve $\varepsilon = f(T)$:

T_1 (°C), which is the temperature of shrinkage onset ($[d\varepsilon/dT]_{T_1} = 0$),
T_2(°C), which is the temperature of shrinkage slow-down ($[d^2\varepsilon/dT^2]_{T_2} = $ max.),
T_3(°C), which is the temperature of highest shrinkage rate ($[d\varepsilon/dT]_{T_3} = $ min.),
v_{max} (% °C^{-1}), which is the highest rate of shrinkage ($v_{max} = [d\varepsilon/dT]_{T_3}$).

The diagram showing the way of determining T_1, T_2, and T_3, and the rate v_{max}, is shown in figure 2.
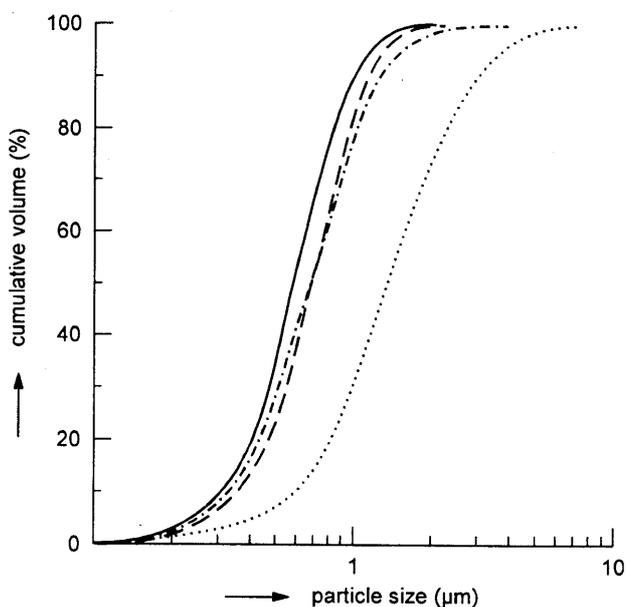

Figure 1. Particle size distribution of the ceramic powders. ——— Rey, — — — ZTA, · · · ATZ, — · — · Tos - IM
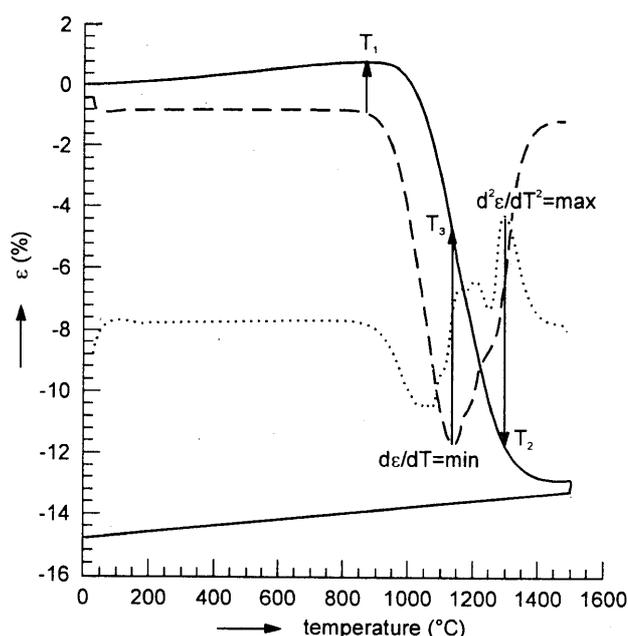

Figure 2. Determination of temperatures of shrinkage onset T_1, shrinkage slow-down T_2, and maximum shrinkage rate T_3 from the expression $\varepsilon = f(T)$.
——— ε, — — — $d\varepsilon/dT$, · · · $d^2\varepsilon/dT^2$

Also determined were the following quantities:

ε_1 (%), which is the micrometer-measured shrinkage ($\varepsilon_1 = (L_1 - L_0) \times 100 / L_0$, (%), where L_1 (mm) is the

specimen length measured with the micrometer after the experiment),

p_{rel} (%) is the relative specimen density after the experiment, established by the double weighing method.

The coefficient of linear thermal expansion (α_{tech}) is defined by equation:

$$\alpha_{tech} = \frac{\Delta L}{L_0 \times (T\text{-}20)} \quad (°C^{-1}) \,, \tag{1}$$

where the significance of quantities ΔL, L_0 and T is the same as in the previous paragraph. In determining these quantities, the specimens were heated by a temperature increase rate of 10°C min^{-1} up to 1600 °C. The mean value of the linear thermal expansion coefficient over the interval of 800 °C to 1500 °C ($\overline{\alpha}_{tech}$) was calculated as an arithmetic mean of values α_{tech} for the respective temperatures of 800 °C, 900 °C, ... 1500 °C.

RESULTS

Properties of the ceramic materials after annealing at 800 °C for 1 hour

A comparison of the experimental values of relative densities of the materials after annealing at 800 °C for 1 hour, including standard deviations s and the number of measurements n is given in table 1.

The results show that the relative density after annealing ranged from 61 % to 67 %, with the sole exception of material Tos-CIP which had a relative density of about 48 %. The relative density values established from water absorption were always higher than those determined by measuring the specimens, the differences ranging from 0.3 % (Tos-IM) to 3.6 % (ATZ-CIP).

Properties of the ceramic materials after sintering

The series of sintering experiments performed was concerned with determining the dependence of expansion

(or shrinkage) of the specimens in terms of temperature T and time t. A list of the experiments is given in table 2, where the values found by analyses of curves $\varepsilon = f(T)$ are listed.

The values show that the position of the "quick sintering" interval ($<T_1$, $T_2>$) did not depend on the method of sample preparation nor on the rate of temperature increase. In the case of materials based on alumina (Rey, ZTA) the position of the interval was shifted by 100 – 150 °C towards higher temperatures, as compared to materials based on zirconia (Tos, ATZ). The mean value of the maximum shrinkage rate (independent of the sintering heating rate) was lower in the case of injection-moulded specimens (by 0.004 % °C^{-1} to 0.011 % °C^{-1}), and this maximum rate occurred at higher temperatures than with isostatically pressed specimens.

The maximum sintering rate was exhibited by specimens Tos-CIP and Tos-IM (-0.08 % °C^{-1}), the minimum one by specimens Rey-CIP and Rey-IM (-0.04 % °C^{-1}). The temperature of maximum shrinkage rate increased in the order ATZ (~1150 °C), Tos (~1270 °C), Rey (~1390 °C), ZTA (~1470 °C).

Table 2 also shows that the temperature of maximum shrinkage rate was increasing with the heating rate (with the exception of ATZ-CIP), but also that the heating rate had no effect on the value of maximum shrinkage.

The values of relative specimen expansion and of relative density (cf. table 2) were independent of the heating rate, but depended on the specimen preparation method. The values of relative shrinkage and those of relative density were in all cases higher with specimens prepared by isostatic pressing (CIP) than with the injection-moulded ones (IM). This fact is also illustrated by the diagram in figure 3 for the ATZ material. A similar behaviour was exhibited by specimens Rey and ZTA. An analogous comparison of relative expansion of isostatically pressed and injection-moulded zirconia specimens was not possible owing to the great difference in the green relative density of the two types of specimens (cf. table 1).

Table 1. Relative densities of the ceramic materials after annealing at 800 °C for 1 hour.

| material | CIP | | | | IM | | | |
| | method 1 | | method 2 | | method 1 | | method 2 | |
	p_{rel0} (%)	s(%)/ n	p_{rel0} (%)	s(%)/ n	p_{rel0} (%)	s(%)/ n	p_{rel0} (%)	s(%)/n
Reynolds	63.09	0.64/ 3	64.74	0.03/ 2	61.39	0.65/ 2	64.34	0.57/ 2
ZTA	65.13	1.11/ 2	67.19	0.08/ 2	63.83	0.22/ 7	65.11	0.19/ 3
ATZ	61.02	0.42/ 5	64.58	0.13/ 5	62.05	0.24/ 3	64.00	0.24/ 3
Tosoh	47.85	0.62/ 4	48.81	0.12/ 2	62.26	0.25/ 7	62.57	0.72/ 3

Table 2. Characteristic of dilatometric curves and properties of the ceramic materials after sintering

| specimen	heating rate ($°C \ min^{-1}$)	T_1 ($°C$)	T_2 ($°C$)	T_3 ($°C$)	v_{max} ($\% \ °C^{-1}$)	ε_1 ($\%$)	ρ_{rel} ($\%$)
Rey-CIP05	5	967	1426	1362	-0.044	-13.65	100.0
Rey-CIP10	10	985	1460	1390	-0.044	-13.72	100.2
Rey-IM05	5	977	1493	1390	-0.037	-12.21	99.5
Rey-IM10	10	1004	1476	1423	-0.036	-12.18	98.9
ZTA-CIP05	5	1043	1495	1450	-0.061	-12.49	99.3
ZTA-CIP10	10	1052	1492	1467	-0.059	-12.55	98.9
ZTA-IM05	5	1066	1500	1465	-0.056	-12.20	98.8
ZTA-IM10	10	1072	1485	1490	-0.053	-12.22	98.3
ATZ-CIP05	5	893	1295	1156	-0.051	-14.59	98.5
ATZ-CIP10	10	901	1328	1139	-0.054	-14.69	99.3
ATZ-IM05	5	900	1299	1138	-0.047	-13.33	97.4
ATZ-IM10	10	919	1322	1164	-0.046	-13.37	98.0
Tos-CIP02	2	879	1344	1225	-0.077	-21.66	99.0
Tos-CIP05	5	874	1334	1263	-0.080	-21.28	99.8
Tos-CIP10	10	897	1357	1284	-0.083	-21.19	101.5
Tos-IM05	5	904	1313	1273	-0.077	-13.57	97.8
Tos-IM10	10	928	1339	1286	-0.072	-13.50	98.0
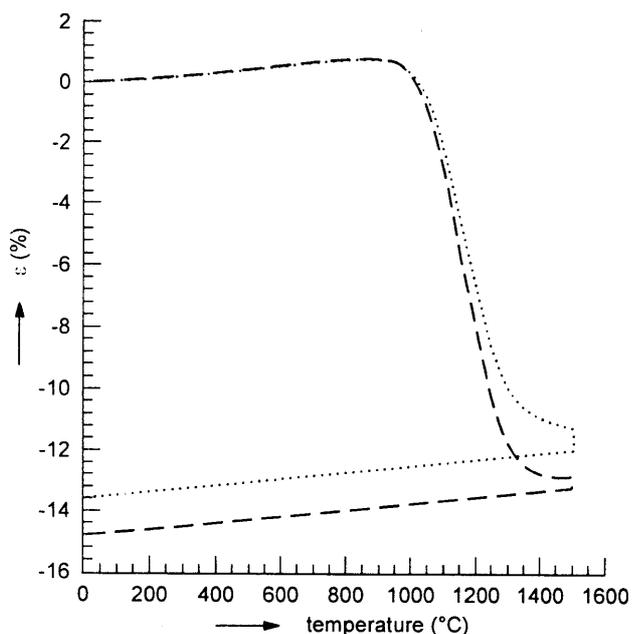
Figure 3. Diagram of relative expansion of ATZ specimens in terms of temperature and technology of preparation.
— — — ATZ-CIP05, · · · · ATZ-IM05

Figure 4. Relative expansion of ZrO_2 specimens versus temperature and rate of sintering heating (technology of preparation - isostatic pressing).
— — — TOS-CIP02 (2 °C min⁻¹), · · · · TOS-CIP05 (5 °C min⁻¹), — · — · TOS-CIP10 (10 °C min⁻¹)

The relative thermal expansion of isostatically pressed specimens vs. temperature and heating rate is plotted in figure 4. The diagram indicates that the resultant relative shrinkage did not depend on the heating rate.
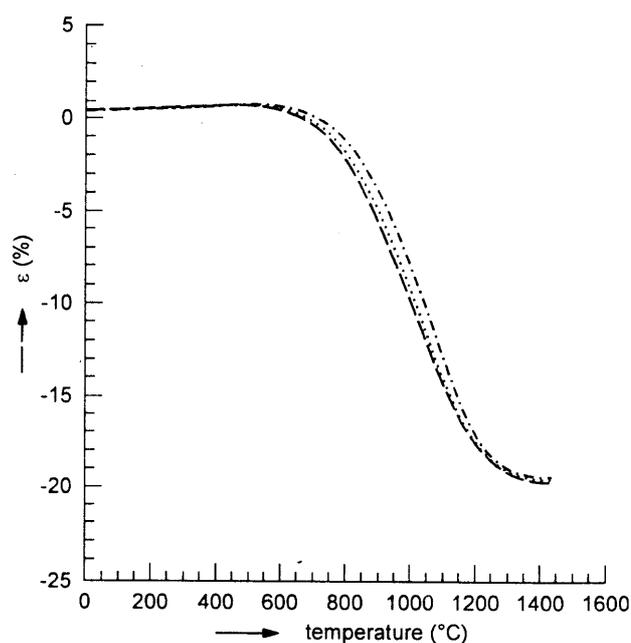
A diagram of the relative expansion of injection-moulded specimens vs. temperature and type of material is shown in figure 5. It indicates that the resultant relative shrinkage of specimens was greater with ATZ and Tos than with the Rey and ZTA specimens.
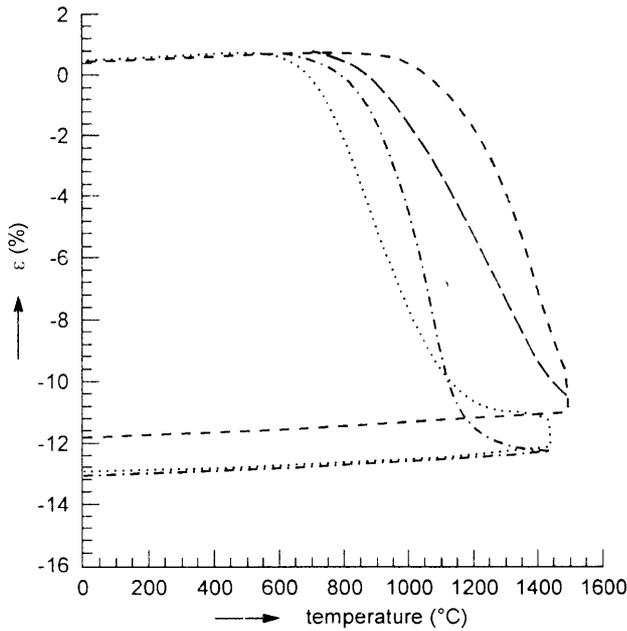
Figure 5. Relative expansion of specimens versus temperature and type of material (temperature gradient 5 °C min⁻¹; technology of preparation - injection moulding).
— — REY-IM05, — — — ZTA-IM05, · · · · ATZ-IM05, — · — · TOS-IM05

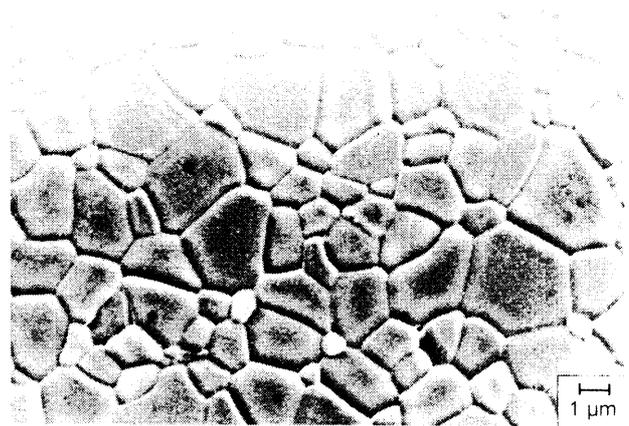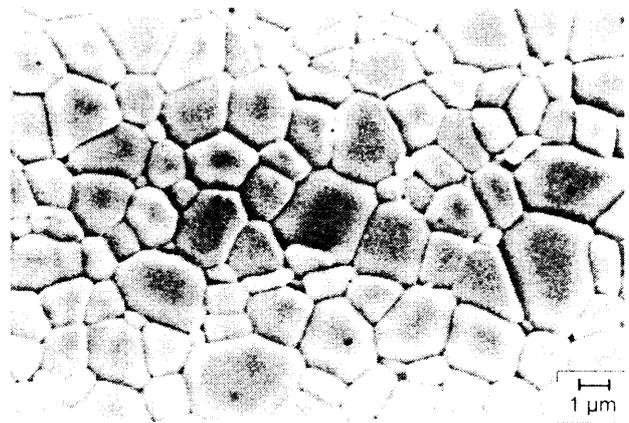

Figure 6. Microstructure of thermally etched Al_2O_3 of specimen Rey-CIP05 (temperature gradient 5 °C min⁻¹; technology of preparation - isostatic pressing).

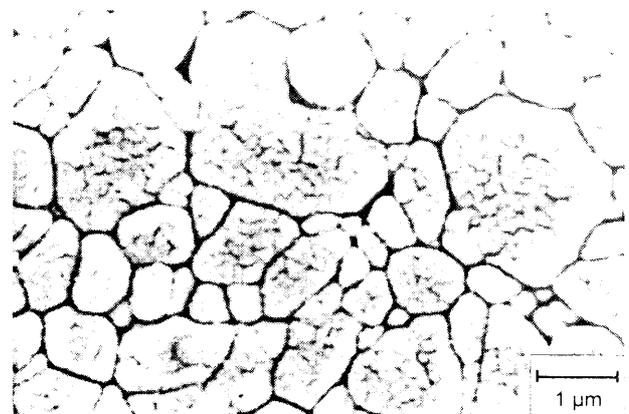Figure 7. Microstructure of thermally etched Al_2O_3 of specimen Rey-IM05 (temperature gradient 5 °C min⁻¹; technology of preparation - injection moulding).

Figure 8. Microstructure of thermally etched ZTA of specimen ZTA-CIP05 (temperature gradient 5 °C min⁻¹; technology of preparation - isostatic pressing).

Several polished and thermally etched sections of the ceramic materials were prepared for studying the structure of the materials after sintering. Their micrographs are shown in figures 6 through 10.

Figure 6 shows the set-off structure of isostatically pressed alumina ceramic material sintered at a heating rate of 5 °C min⁻¹. The grains are sintered and no pores are discernible on the micrographs. The statistically determined value of mean grain size for isostatically pressed specimens of Rey-CIP05 had the value $d = 1.8$ µm. The injection-moulded specimen Rey-IM05 had a similar structure (figure 7) and a mean grain size $d = 1.6$ µm.

Figure 8 illustrates the structure of ZTA ceramic material prepared by isostatic pressing and sintered at a rate of 5 °C min⁻¹. The material was composed of sintered large alumina grains and small zirconia grains.

Figure 9 shows the structure of ATZ ceramic material prepared by isostatic pressing and sintered at a heating rate of 5 °C min⁻¹. There are sintered small grains of ZrO_2 and large grains of Al_2O_3.

The structure of a zirconia ceramic material prepared by isostatic pressing and sintering at a rate of 5 °C min⁻¹ is shown in figure 10. The sintered zirconia grains had a mean grain size of $d = 0.4$ µm. A similar structure was exhibited by specimens sintered at a heating rate of 2 °C min⁻¹ and 10 °C min⁻¹, the mean grain size being identical for all three heating rates (0.4 µm).
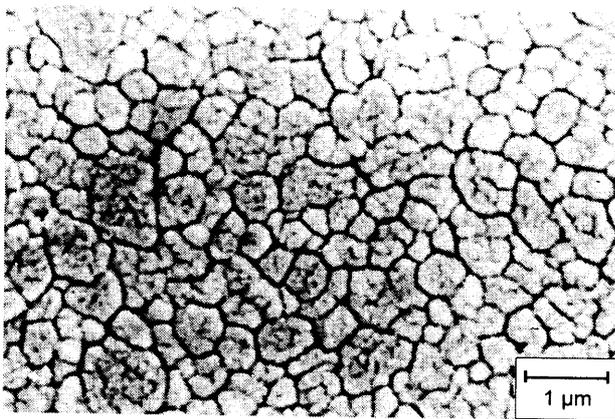
Figure 9. Microstructure of thermally etched ATZ of specimen ATZ-CIP05 (temperature gradient 5 °C min⁻¹; technology of preparation - isostatic pressing).
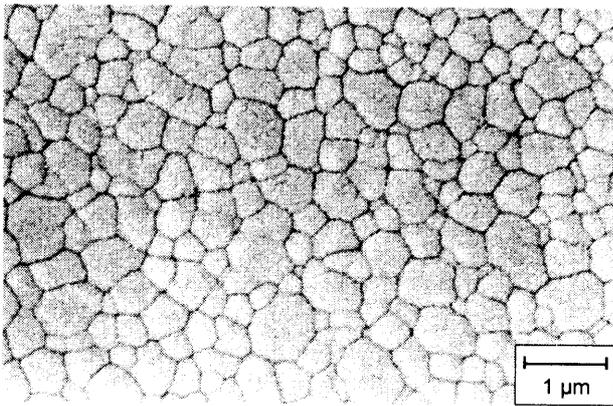

Figure 10. Microstructure of thermally etched ZrO_2 of specimen Tos-CIP05 (temperature gradient 5°C min⁻¹; technology of preparation - isostatic pressing).

Thermal expansion of the sintered ceramics

Figure 11 shows the temperature dependence of relative expansion and of the coefficient of linear thermal expansion of sintered specimens prepared by isostatic pressing. The mean coefficients of linear thermal expansion of the alumina-based materials (Rey-CIP, ZTA-CIP) over the interval of 800 °C to 1500 °C had the value of $\bar{\alpha} = 9 \times 10^{-6}$ K⁻¹, those of the zironia-based materials (ATZ, Tos) the value of $\bar{\alpha} = 11 \times 10^{-6}$ K⁻¹.

DISCUSSION

The structure of ceramic materials annealed at 800 °C for 1 hour was described by means of relative density. Determination of ρ_{rel} from water absorption is accurate if all pores in ceramics are open. The values of

ρ_{rel} determined by means of water absorption were always higher than the values derived from dimension measurements, namely by 0.3 % to 3.6 %. These differences may have been due to:

- the presence of a small amount of closed pores,
- the presence of narrow points in the pore network through which capillary forces did not allow water to penetrate,
- the possibility of removing a small amount of water from surface pores by wiping off the specimens before weighing.
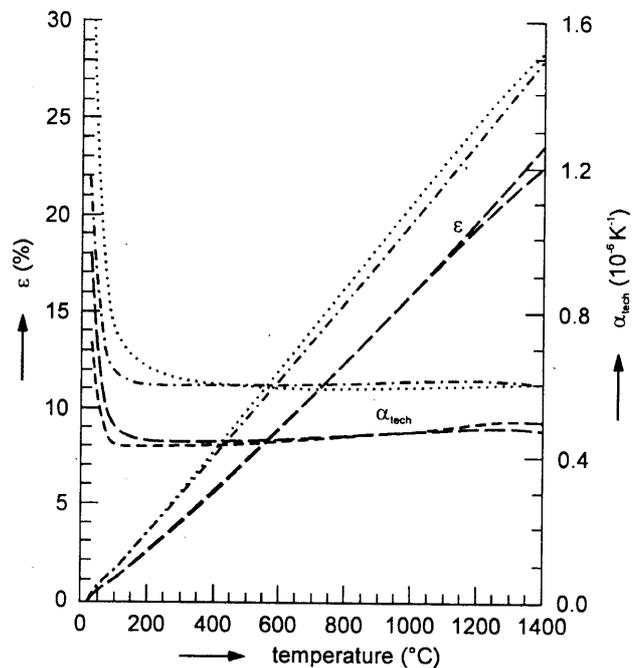

Figure 11. Relative expansion and coefficient of thermal expansion of sintered isostatically pressed specimens vs. temperature.
— — Rey-CIP, — — — ZTA-CIP, · · · · ATZ-CIP, — · — · TOS-CIP

For the individual types of material, the "interval of quick sintering", the name proposed in the present study for the temperature region between the onset of sintering (T_1) and the sintering slow-down (T_2) was independent of the technology of specimen preparation and of the heating rate. In the case of ZrO_2 specimens, the distinctly lower initial relative density of the green isostatically pressed specimens (Tos-CIP) resulted in a broadening of the interval, which means that the sintering began at a lower temperature and slowed down at a higher temperature compared to specimens with a higher green relative density (Tos-IM). However, the maximum relative rate of sintering had values comparable for both types, i.e. the lower green relative specimen density

resulted in a lengthening of the time required for sintering, but not in a higher sintering rate.

The results also show that the zirconia-based materials (Tos, ATZ) sintered at lower temperatures than those based on alumina (Rey, ZTA). On the one hand, this may be due to the chemical character of the materials, while the composites retained the properties of their majority component, and on the other, to the particle size. According to the particle size distribution (figure 1), all the types of materials had similar particle size about 1 μm; however, the manufacturer of the zirconia powders (Tosoh) specifies for the zirconia powder a crystallite size of 0.036 μm (Tos-IM) and 0.027 μm (Tos-CIP). These crystallites then formed primary particles and aggregates about twenty or thirty times larger which were measured by the particle size analyzer.

The lower values of the maximum rates of sintering (and the higher temperatures at which they occurred) for injection-moulded specimens correlated with the lower values of final shrinkage and with the resultant relative density of these specimens as compared to the isostatically pressed ones. The values of relative expansion ε were measured with a great accuracy, but for the purpose of mutual comparison of the individual types of materials the value is affected by the differences in the green relative densities of the materials. The resultant relative density of the specimens allowed the individual materials to be compared. The resultant relative density ρ_{rel} was also calculated from the known values of ε_1 and from green relative density $\rho_{rel\,0}$. On the assumption of an isotropic shrinkage it is possible to derive equation:

$$\rho_{rel} = \rho_{rel\,0} \times \left(\frac{100}{100 + \varepsilon_1} \right) (\%) \ . \tag{2}$$

The values of relative expansion, of the resultant relative density measured and those of the calculated resultant relative density are listed in table 3.

The deviations of the experimental from the theoretical values ρ_{rel} were due in particular to:

- inaccuracy of measuring ρ_{rel} (double weighing method),
- inaccuracy of measuring $\rho_{rel\,0}$ (specimen measuring and weighing method, water absorption),
- non-isotropic behaviour in the course of shrinkage.

The experimental and the calculated values of ρ_{rel} showed that the specimens prepared by isostatic pressing attained a higher degree of sintering than those prepared by injection moulding. Evidence of this difference was not found on the micrographs of specimens after sintering (compare figures 6 and 7). For the purpose of comparing the porosity, it will be necessary to prepare thermally

untreated polished sections from various parts of the sintered specimens.

The thermally etched structures of the specimens served for determining the shape and size of grains in the material. The planar section passes through the grain volume at a statistically random distance from the grain centre, and the interpolated straight line intersects the planar pattern formed as an intersection of the cut plane with the grain, again at a statistically random distance from the geometrical centre of the pattern. Therefore the value $\bar{d}$ is not a quantity describing the grain size in an absolute way, being used solely as a relative comparable quantity. The absolute grain size, which would allow a comparison with the grain size of the initial powder to be made, can be calculated from the $\bar{d}$ value on assuming, for the sake of simplicity, the grains to be of spherical shape (the sphere diameter being designated $\bar{D}$). The experimentally established $\bar{d}$ value is a statistical estimate of mean lengths of all randomly chosen secants through spheres $\bar{D}$ in diameter. Calculation of this mean value yields:

$$\bar{d} = \left(\frac{\pi}{4} \right)^2 \times \bar{D} \ (\mu m) \ . \tag{3}$$

On the basis of experimental measurement, the particle size $\bar{D}$ can be estimated to be equal to:

$$\bar{D} = \left(\frac{4}{\pi} \right)^2 \times \bar{d} = 1.62 \times \bar{d} \ (\mu m) \ . \tag{4}$$

The absolute grain size of Al_2O_3 grains after sintering was then $\bar{D} = 2.9$ μm (Rey-CIP05) and 2.6 μm (Rey-IM05), which means that by sintering the diameter of Al_2O_3 grains was increased 4 to 5-times.

The absolute grain size of ZrO_2 after sintering was $\bar{D} = 0.6$ μm (Tos-CIP02, 05, 10), that is similar value as the size of aggregates of the initial powdered ZrO_2.

The values of the linear thermal expansion coefficient of the sintered ceramic materials were determined for the specimens prepared by isostatic pressing. Their values for Al_2O_3 ($\bar{\alpha}_{tech} = 9 \times 10^{-6}$ K^{-1}) and ZrO_2 ($\bar{\alpha}_{tech} = 11 \times 10^{-6}$ K^{-1}) agreed with the literary data in [5] where the following values are given:

Al_2O_3 : $\alpha = 9.2 \times 10^{-4}$K^{-1} for the direction parallel with crystallographic axis c,
$\alpha = 8.3 \times 10^{-4}$K^{-1} for the direction perpendicular to crystallographic axis c.
ZrO_2 : $\alpha = 10.6 \times 10^{-6}$ K^{-1}

The values of α established for the ZTA composites ($\bar{\alpha}_{tech} = 9 \times 10^{-6}$ K^{-1}) and ATZ ($\bar{\alpha}_{tech} = 11 \times 10^{-6}$ K^{-1}) showed that the thermal expansion of the composites was controlled by the thermal expansion of their majority phase.

Table 3. Value of resultant relative expansion, measured and calculated relative densities of the sintered ceramic materials.

| material | measured density and shrinkage | | calculated density | |
	ε_l (%)	ρ_{rel} (%)	method 1 ρ_{rel} (%)	method 2 ρ_{rel} (%)
Rey-CIP05	-13.65	100.0	97.9	100.5
Rey-CIP10	-13.72	100.2	98.2	100.8
Rey-IM05	-12.21	99.5	90.7	95.0
Rey-IM10	-12.18	98.9	90.6	95.0
ZTA-CIP05	-12.49	99.3	97.1	100.2
ZTA-CIP10	-12.55	98.9	97.3	100.4
ZTA-IM05	-12.20	98.8	94.3	96.2
ZTA-IM10	-12.22	98.3	94.3	96.2
ATZ-CIP05	-14.59	98.5	97.9	103.6
ATZ-CIP10	-14.69	99.3	98.2	104.0
ATZ-IM05	-13.33	97.4	95.3	98.3
ATZ-IM10	-13.37	98.0	95.4	98.4
Tos-CIP02	-21.66	99.0	99.5	101.5
Tos-CIP05	-21.28	99.8	98.1	100.1
Tos-CIP10	-21.19	101.5	97.8	99.7
Tos-IM05	-13.57	97.8	96.4	96.9
Tos-IM10	-13.50	98.0	96.2	96.7

CONCLUSION

The following results have been obtained by studying the sintering of ceramic materials based on Al_2O_3, ZrO_2 and of their composites:

- the alumina-based ceramic materials sintered at higher temperatures than those based on zirconia,
- at heating rates of 10 °C min⁻¹ the resultant relative densities obtained were comparable to those achieved with rates of 5 °C min⁻¹ and 2 °C min⁻¹,
- the resultant relative densities were higher with materials prepared by isostatic pressing than with materials prepared by injection moulding.

High-temperature dilatometry was used for determining the mean values of the linear thermal expansion coefficient of the sintered ceramic materials over the interval of 800 – 1500 °C:

$\overline{\alpha}_{tech}$ (Rey, ZTA) = 9×10^{-6} K⁻¹,
$\overline{\alpha}_{tech}$ (Tos, ATZ) = 11×10^{-6} K⁻¹.

Further study of the given subject matter should be aimed in particular at:

- the kinetics of sintering of these materials at higher heating rates,
- the relationship between the resultant relative densities and the microstructure of the specimens,
- the sintering of specimens controlled by the rate of shrinkage (RCS).

The results obtained should serve for optimizing the temperature schedules for firing the given types of materials.

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STUDIUM SLINOVÁNÍ KERAMICKÝCH MATERIÁLŮ POMOCÍ VYSOKOTEPLOTNÍ DILATOMETRIE

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Vysokoteplotní dilatometrie keramických vzorků na bázi Al_2O_3, ZrO_2 a jejich kompozitů (ZTA85 a ATZ80), vyrobených izostatickým lisováním a injekčním vstřikováním, přinesla tyto poznatky o jejich slinování:

- použité keramické materiály na bázi Al_2O_3 slinovaly při vyšších teplotách než materiály na bázi ZrO_2
- výsledná relativní smrštění byla vyšší u vzorků na bázi ZrO_2 (ZrO_2 a ATZ80) než u vzorků na bázi Al_2O_3 (Al_2O_3 a ZTA85)
- při rychlostech ohřevu 10°C min⁻¹ bylo dosaženo srovnatelných výsledných relativních hustot jako při teplotních gradientech 5 °C min⁻¹, resp. 2 °C min⁻¹
- výsledné relativní hustoty byly vyšší u materiálů připravených izostatickým lisováním než u materiálů připravených injekčním vstřikováním
- střední velikost částic Al_2O_3 vzrostla při slinování (5 °C min⁻¹, prodleva 2h při 1530 °C) z 0,6 μm na 2,6 μm (injekčně vstřikovaný vzorek) resp. na 2,9 μm (izostaticky lisovaný vzorek)
- střední velikost částic izostaticky lisovaného ZrO_2 po slinování (1500 °C / 2 h) byla 0,6 μm při všech třech rychlostech ohřevu (2 °C min⁻¹, 5 °C min⁻¹, 10 °C min⁻¹)

Byly stanoveny střední hodnoty koeficientu teplotní roztažnosti slinutých keramických materiálů v intervalu 800 - 1500 °C:

$\overline{\alpha}_{tech}$ (Al_2O_3, ZTA85) = 9×10^{-6} K⁻¹
$\overline{\alpha}_{tech}$ (ZrO_2, ATZ80) = 11×10^{-6} K⁻¹