Study of transverse deformation of porous alumina during uniaxial mechanical tests

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Abstract

In this article presents the results of laboratory tests and analysis of the mechanical behavior of Al_2O_3-based samples during mechanical uniaxial compression testing. The samples with different porosities are obtained via different sintering temperatures. It has been shown that an increase of strength and, accordingly, decrease in porosity are significantly determined the changes of the Poisson's ratio under loading, and this change begins long before the appearance of the first internal microcracks in the internal parts of the material, i.e., the appearance of excess volume. These results clearly show the phenomenon of dilatancy, with a sharp increase of effective Poisson's ratio.

Keywords: Al_2O_3, porosity, dilatancy, compression testing, transverse strain, X-ray diffraction

Kulcsszavak: Al_2O_3, porozitás, dilatáció, nyomószilárdság teszt, keresztirányú alakváltozás, röntgendiffrakció

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1. Introduction

The analysis of the current state of ceramic materials researching shows that the main interest is represented by the studies of correlation between porosity and strength of ceramics. The main researches of Al_2O_3-based ceramic materials are carried out in the field of compression testing, structure analysis, X-ray diffraction etc. [1-7]. There is much less attention given to the research of the dilatancy phenomenon, based on the Poisson's ratio change of Al_2O_3 samples with different porosities obtained at different sintering temperatures.

Identification of unstable crack growth threshold (threshold of absolute dilatancy) can be done with a good level of precision, but identification of the onset crack initiation (threshold of relative dilatancy) is not easy, especially in porous materials [8-9]. In last years various authors have proposed different methods for crack initiation threshold identification [10-11], for example, the change in Poisson's ratio as an indicator for establishing crack initiation threshold.

The increase in the specific volume of the material during the dilatancy process corresponds to an increase in the effective Poisson's ratio. Formally, during measurements, it is possible to obtain an increase in this value to 1 or more, although, as is known, for continuous media, the maximum possible value of the Poisson's ratio (the ratio of the values of longitudinal and transverse relative deformations) can be not more than 0.5. Ceramic materials and rocks very often have residual porosity, so under the influence of stresses, compaction of the material can occur, and at the same time, due to the anisotropy

of loading, the initial value of the Poisson's ratio can be very small. At high stresses, compaction is replaced by dilatancy with an increase in the effective Poisson's ratio and subsequent fracture.

Apparently, the appearance of microcracks in such materials occurs at stresses below the elastic limit, but in general, the loading diagram corresponds to an elastic-plastic body.

Literature data shows that when loading, for example, soil, there is a non-linearity not only in the volume deformability (compression), but also in shear deformation, which are caused not only by tangential, but also by normal stresses, with the appearance of the dilatancy effect, which in the case of crushing, i.e., micro-destruction, leads to the need to take into account other effects – hardening, deformation rate, etc.

However, cumulative studies of Al_2O_3 samples obtained at different sintering temperatures and comprehensively investigated by these methods with the data capturing of transverse deformation in situ, have not been properly carried out yet. The correlation between Poisson's ratio change and transverse strain has also not been thoroughly researched in this class of materials. It is known that Al_2O_3 exhibits a phenomenon of dilatancy under load, but the effect of this phenomenon has not been properly explored yet.

Thus, the **aim** of this paper is to study the internal structure of alumina samples with different porosities and their mechanical behavior under uniaxial loading tests.

2. Materials and methods

Ceramic samples in the form of rectangular parallelepipeds with the initial dimensions 36 x 7 x 6 mm were obtained by pressing commercially pure Al_2O_3 powder. It was mechanically processed in a ball mill in the span of 70 hours, which resulted in producing fine particles with a homogeneous size distribution.

The samples were sintered in the Nabertherm LHT 02/17 high-temperature muffle furnace. The various batches of samples were first slowly heated (within 6 hours) to the set temperatures of 1350°C, 1450°C, 1550°C and 1650°C. Then they were held for 1 hour and then slowly cooled down to 40°C. This was done with the purpose of obtaining Al_2O_3 samples with different porosities ranging in between 15 to 50%. Sintered Al_2O_3 samples were mechanically treated using the Struers Secotom-10 precision cutting machine to achieve their plane-parallel flatness. After the three-point bending tests, the samples were cut to 15 mm in height with the 7 x 6 mm cross section for compression testing and simultaneous measuring of transverse strain.

Both bending and compression tests were carried out using the Instron-1185 universal testing machine for determining flexural and compressive strength, technical elastic moduli and longitudinal strain values of the Al_2O_3 samples. Transverse strain was measured mechanically using a strain gauge device - multiturn measuring head - with an accuracy of 1 μm.

The structure parameters and detailed microstructural study of the samples were investigated using the X-ray diffractometer with filtered CuK_α radiation. For the analysis of the polished sample surface the Vega 3 SBH scanning electron microscope was used.

3. Results and discussion

The porosity of the sintered samples varies from 48% at 1350 °C down to 16% at 1650 °C, which correlates well with the already existing data [12]. *Fig. 1a* shows the typical polished surface of the Al_2O_3 sample with 16% porosity. The grain size in the samples obtained at all sintering temperatures varies from 2 to 6 microns. At the same time, as the sintering temperature increases, the percentage of grains with a size of 4 to 6 microns increases. The pore sizes in the samples vary from 1 to 7 microns; for samples with a sintering temperature of 1350 °C and 1450 °C, the main pore size is from 1 to 4 microns, and for the highest temperatures the percentage of pores with a size of 4 to 7 microns increases, which coincides with the literature data [12-13]. The dependence of the porosity on the sintering temperature of the Al_2O_3 samples is shown in the *Fig. 1b*. As can be seen, the porosity of the samples decreases almost linearly with the increase of the sintering temperature, which corresponds with the literature data [13]. Extrapolation of this curve using *log* function to determine the temperature at which the porosity of the sample will smaller when 1% one obtained this temperature approximately equal 1850 °C.
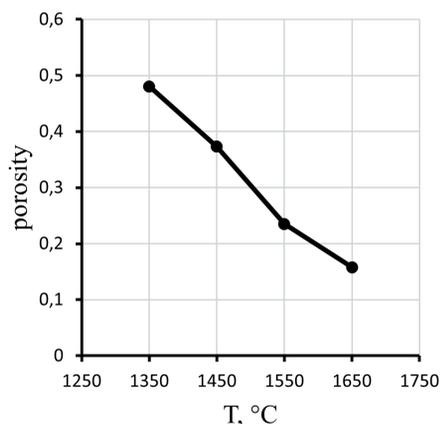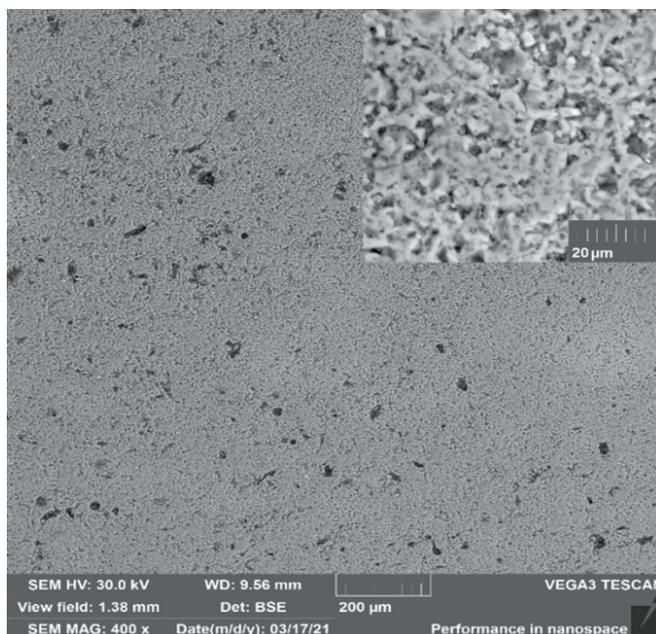
Fig. 1   Polished surface of the Al_2O_3 samples with 16% porosity (a); Dependence of the porosity of the Al_2O_3 samples vs. sintering temperature (b).
1. ábra   A 16%-os porozitású Al_2O_3 minták csiszolt felülete (a); Az Al_2O_3 minták porozitása a szinterelési hőmérséklettől függően (b).

The X-ray diffraction studies of the sample surfaces has shown, *Fig. 2a*, that the angles positions of diffraction peaks correspond to the rhombohedral structure of alumina and almost does not change with increasing temperature, which coincides with the literature data [14]. Only the width of X-ray lines had changed with increase of sintering temperature, therefore we have calculated the coherent diffraction domains sizes (CDD) for (012)-line with smallest angle diffraction, *Fig. 2b*, from which one can see that dependency of the coherent diffraction domains sizes for different sintering temperatures of the Al_2O_3 samples are increase with the increase of the sintering temperature and CDD changes from 450 angstroms at the lowest sintering temperature up to 610 angstroms at the highest temperature of 1650 °C. On this figure a dash line is showing the initial powder's CDD, which equals approximately to 400 Å, therefore extrapolation of this dependence on lower temperature had obtained that CDD changes after heating of the samples only after 1250 °C.
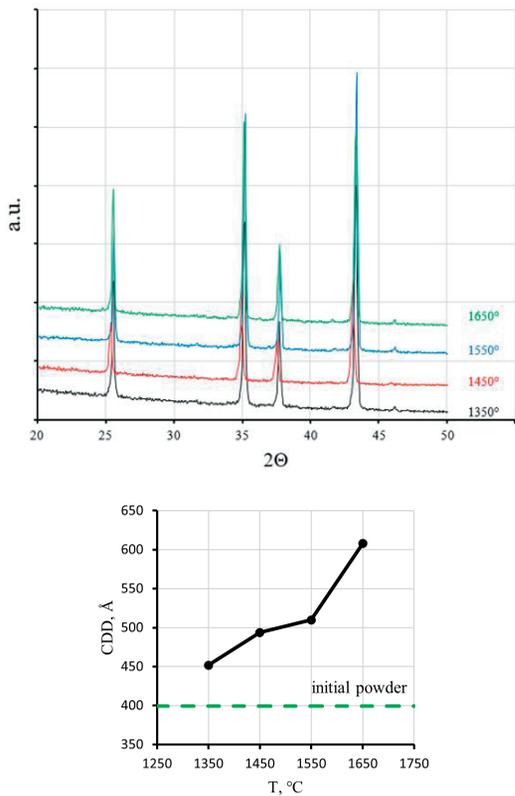
Fig. 2 X-ray diffraction patterns of the Al_2O_3 samples sintered at different temperatures (a); Dependence of the coherent diffraction domain (CDD) on the sintering temperature (b)

2. ábra Különböző hőmérsékleteken szinterelt Al_2O_3 minták röntgendiffrakciós mintázata (a); A koherens diffrakciós domén (CDD) mérete a szinterelési hőmérséklettől függően (b)

Fig. 3 shows the bending (a) and compressive (b) strengths of samples. The values of flexural strength vary between 8 MPa at 1350 °C and 87 MPa at 1650 °C with an almost linear increase and correlate with porosity of the samples (*Fig. 1b*). The correlation between the compressive strength and the sintering temperature of the alumina samples is shown in the *Fig. 3b*, the values are varied between 28 and 229 MPa. As one can see from these data the well-known correlation between the compressive and bending stresses occurs: compressive stresses exceed the bending stresses by 3 times.
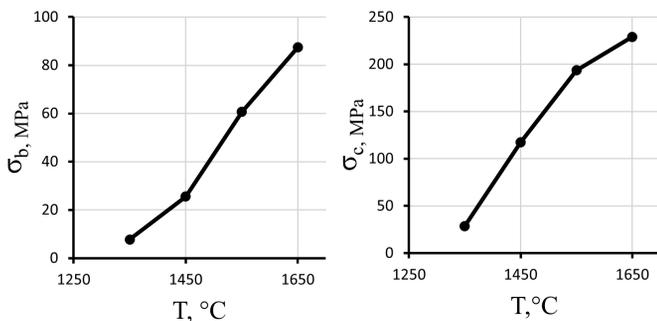

Fig. 3 Dependency of the bending (a) and compression strengths (b) vs. the sintering temperature

3. ábra A hajlító (a) és a nyomószilárdságok (b) szinterelési hőmérséklettől függően

A typical stress-strain curve during the uniaxial compression test is shown on the *Fig. 4*. The slope of curve in its linear part stipulates the technical Young's moduli of sintered samples,

which is equal approximately 10.5 GPa. This modulus is rather small, probably due to a high porosity of sintered ceramics, but the samples sintered at the highest temperature and having the lowest porosity have the highest modulus of elasticity.

In this figure one can see some special region on stress-strain curve: small interruption of increasing stresses, marked with an arrow, which, as a rule, stipulate an internal micro-fault. This fact may be interpreted as due to the dilatancy effect when compressing the ceramic.
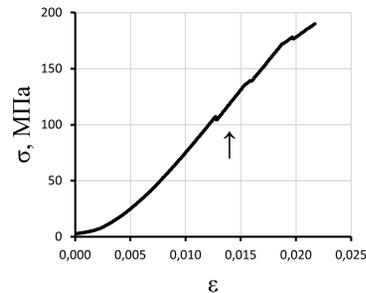

Fig. 4 Typical stress – strain curve for the uniaxially compressed Al_2O_3 samples; sintering temperature 1450 °C.

4. ábra Az 1450 °C-on szinterelt, egytengelyesen összenyomott Al_2O_3 minták tipikus feszültség - alakváltozás görbéje

In *Fig. 5* the similar stress-strain dependence for the sample sintered at 1350 °C and the correlation between longitudinal – transverse strains measured during the compression test. The slopes of the linear approximations for the longitudinal-transverse strain curve will define the effective Poisson's ratio. At the initial stage of loading the effective Poisson's coefficient has a relatively good value 0.16 and correlate with table data, but at the strain of 0.0125 it sharply changes up to 1.6. This means that during the sample compression the appearance of an excess internal volume in the sample takes place even before the macrofracture effect, forming in internal parts of the sample [15]. These results clearly show the phenomenon of dilatancy, with a sharp increase of effective Poisson's ratio.
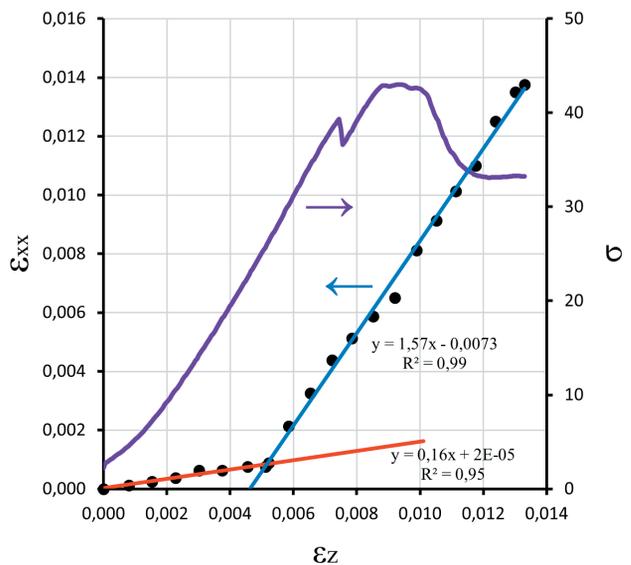

Fig. 5 Stress-strain curve and the corresponding "longitudinal – transverse" strains for sample sintered at 1350 °C

5. ábra A feszültség-alakváltozás görbe és a megfelelő „hosszirányú - keresztirányú" alakváltozás az 1350 °C-on szinterelt minták esetén

Fig. 6a shows the dependence of the change in the effective Poisson's ratio vs. density of the sintered samples. From the obtained **Δν** values one can see that an increase in strength and, accordingly, decrease in porosity significantly determines the changes of the Poisson's coefficient under loading, and this change begins long before the appearance of the first internal microcracks in the volume of the material, i.e., the appearance of excess volume. Therefore, the dilatancy can occur more easily with the increase in temperature. A similar pattern is observed in the analysis of CDD (*Fig. 6b*), i.e., with the growth of CDD, the formation of internal defects (microcracks) that cause the dilatancy effect can more easily occur in the material.
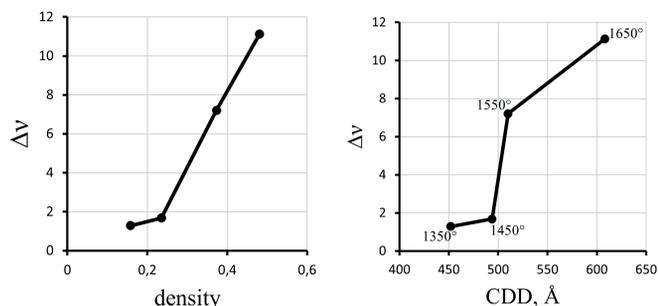

Fig. 6   *Correlation between Poisson's ratio difference (Δν) and density of the samples (a); Correlation between Poisson's ratio difference (Δν) and coherent diffraction domain (CDD) (b).*
6. ábra   *A Poisson-féle aránykülönbség (Δν) és a minták sűrűsége közötti összefüggés (a); Korreláció Poisson-aránykülönbség (Δν) és koherens diffrakciós domén (CDD) között (b).*

4. Conclusions

It has been observed, that under uniaxial compression the transverse strain measuring allows a determination of Poisson's coefficient for ceramic samples under investigation.

It has been shown that an increase of strength and, accordingly, decrease in porosity are significantly determined the changes of the Poisson's ratio under loading, and this change begins long before the appearance of the first internal microcracks in the internal parts of the material, i.e., the appearance of excess volume. These results clearly show the phenomenon of dilatancy, with a sharp increase of effective Poisson's ratio.

Acknowledgments

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