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Strength of pure alumina ceramics above 1 GPa

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ABSTRACT

Up to now, commercially available alumina ceramics were claimed to have strength between 400 and 550 MPa. However, our study shows strength ~ 2 times higher for commercially available alumina than commonly believed. The average and characteristic strength, measured on 31 pure alumina ceramic discs by ball on three balls (B3B) test, were 1205 ± 93 MPa and 1257 MPa, respectively, with a Weibull modulus of m = 11.8. Tested specimens were in form of discs with a diameter of 5 mm and thickness 0.5 mm. The grain size distribution of the alumina is bimodal with an average grain size of ~ 850 nm measured at the surface. The fracture reveals a mixed transgranular / intergranular failure mode. To avoid incorporation of additional flaws, the discs were tested as sintered. The characteristic flexural strength measured in B3B was recalculated according to Weibull theory for standard 4-point bending bars of size 3 × 4 × 45 mm as bend 856 MPa. The measured strength of nearly 900 MPa shows the potential of strength for high purity alumina ceramics.

1. Introduction

Alumina is perhaps the most widely studied and used technical ceramics. Properties such as bio-inertness and chemical resistivity together with good mechanical strength make it applicable in various fields. The strength of alumina ceramics typically varies between 450 and 550 MPa. Therefore, alumina belongs to the intermediate ceramics, between ZnO (~ 100 MPa) and Si_3N_4 (~ 900 MPa) [1] tested by the standard 4-point bending test (4Pt). For example, pure alumina, with > 99% relative density prepared from high purity alumina TM DAR by pressure-less sintering, reached the strength of 520 MPa [2]. In order to obtain the higher strength of alumina the zirconia-toughened alumina (ZTA), alumina-toughened zirconia (ATZ) [3–5] or SiC composites were developed [6–8]. To summarize, composites made out of alumina and second phase materials (in order to increase the mechanical strength) reached strength values close to 1 GPa [9–12]. As it is well known, green body processing and sintering have a great impact on mechanical properties. Therefore, very careful preparation even of pure alumina can result in a much higher strength than is ordinarily observed and e.g. reported by Mizuta et al. [9], Koike et al. [12] and Mata-Osoro et al. [13]. In all cases, high purity TM DAR alumina was used. In the work of Mata-Osoro [13], green samples were prepared by slip casting followed by high vacuum sintering and reached a bending strength of 700 MPa measured by 4-point bending test. Mizuta conducted vacuum-pressure slip-casting and hot isostatic pressing (HIP) to obtain fully dense samples with an average bending strength of 786 MPa [9]. Even better mechanical properties were reached by centrifugal compaction of green body followed by sintering in air. Such a preparation of green body leads to a strength of 1330 MPa after sintering, as presented by Koike [12]. These studies show the potential of monolithic alumina ceramic in terms of mechanical strength. However, it is worth to mention, that until today the statistical evidence is still missing, according to the author's knowledge.

The aim of the research is to provide evidence that the mechanical strength of high purity alumina is in the high strength range for ceramic materials and not in the middle, as usually presented. The sintered material reached a two-times higher strength than today's state of the art.

2. Experimental

2.1. Materials and instrumentation

All samples were produced by injection moulding and were then pre-sintered at 900 °C for 2 h in air. The high purity alumina powder used was an unknown but commercially available powder. A reference batch (samples marked as A) was pressure-less sintered (PS) to the closed porosity and then followed by hot isostatic pressing (HIP). The sintering and HIP regime used for reference batch A was the same as used for commercially available products. Two additional sintering cycles in air without further HIP treatment were developed by Empa. In both cases, the samples were pressureless sintered at 1500 °C. The samples are noted as E-A-0 (without dwell time) and E-A-2 (with 2 h dwell time at maximum temperature). The heating and cooling rate was

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Table 1
Content of impurities of investigated alumina and previously used TM DAR for comparison.

Impurities	Si [ppm]	Fe	Na	K	Ca	Mg
Al_2O_3	4	4	3	1	3	1
TM DAR	13	9	3	2	2	1

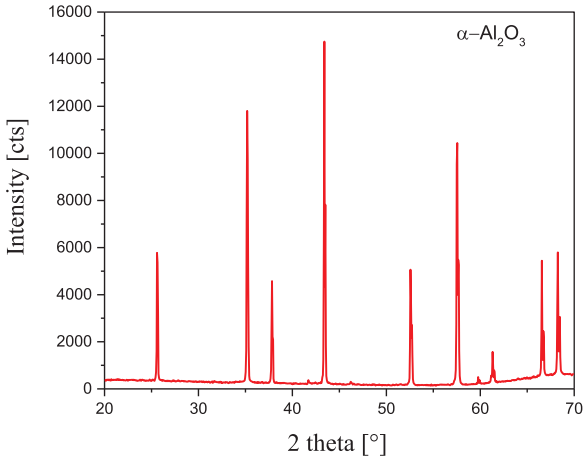

Fig. 1. XRD analysis of the investigated material.

5 °C/min in both cases. Sintering at Empa was utilized in a vertical dilatometer TMA 402 F1 from NETZSCH-Gerätebau GmbH (Graz, Austria). In all three cases, the sintered samples are disc-shaped with a diameter of 5.10 ± 0.03 mm and thickness of 0.47 mm ± 0.01 mm.

Phase composition was determined by X-ray diffractometer Panalytical MRX 4 with accelerator detector from PANalytical B.V. (Almelo, Netherland). The source of X-ray radiation was a copper cathode and the measurement was performed according to the instruction of the manufacturer with the standard program. The diffraction pattern was analyzed with the X-Pert HighScore software.

The microstructure was examined by scanning electron microscopy (SEM) (VEGA Plus 5136 MM, Tescan instruments, Czech Republic) on as sintered surfaces and on fractured surfaces. The material was not further polished or etched before microstructure investigation. The mean grain size of alumina in the sintered samples was determined by the linear intercept method, measuring at least 250 intercepts (software LINCE, TU Darmstadt, Germany), and using a correction factor of 1.56 according to [14].

The biaxial strength was determined using the ball-on-three-balls (B3B) test method. The strength was calculated according to equation [1,15]:

$$\sigma_{B3B} = f^* \frac{F}{t^2} \tag{1}$$

Where σ_{B3B} is the strength [MPa]; F the applied force [N] and t [mm] the thickness of the specimen. The dimensionless factor f depends on the ratio of thickness to the radius of disc, $t/R = \alpha$, the ratio of the support

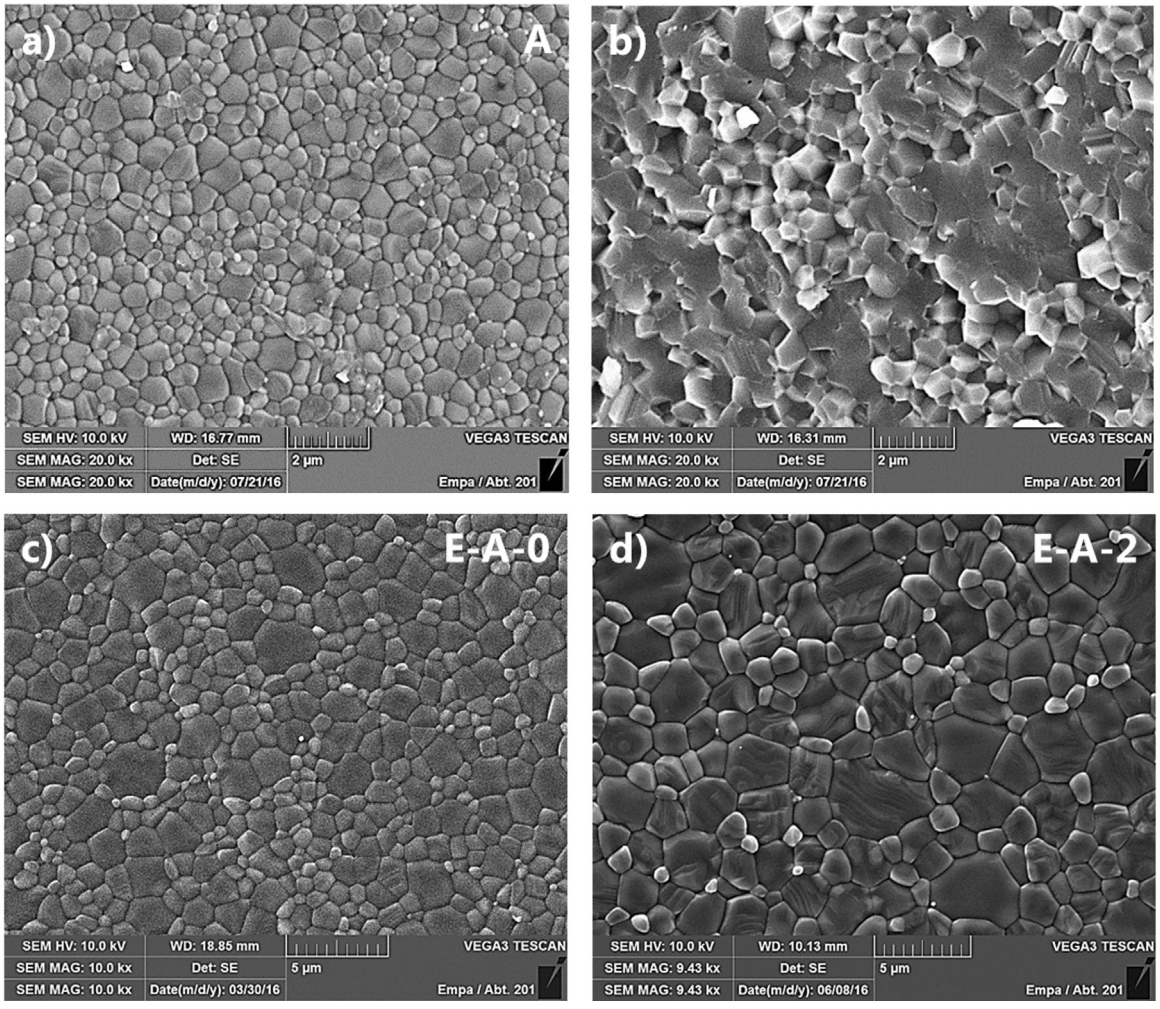

Fig. 2. Microstructure of sample A a) from as sintered surface and b) fracture surface and only from as sintered surface in samples c) E-A-0, and d) E-A-2.