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Citation for published version (APA):

With, de, G. (1986). Note on the relation between the compressive strength of debased alumina and its use as hot-pressing die material. *Journal de Physique. Colloque*, 47(C1), 667-671.
<https://doi.org/10.1051/jphyscol:19861102>, <https://doi.org/10.1051/jphyscol;19861102>

DOI:

[10.1051/jphyscol:19861102](https://doi.org/10.1051/jphyscol:19861102)
[10.1051/jphyscol;19861102](https://doi.org/10.1051/jphyscol;19861102)

Document status and date:

Published: 01/01/1986

Document Version:

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
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NOTE ON THE RELATION BETWEEN THE COMPRESSIVE STRENGTH OF DEBASED ALUMINA AND ITS USE AS HOT-PRESSING DIE MATERIAL

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Resumé - La durabilité des céramiques d'alumine employées dans les matrices de pressage à chaud est très variable. C'est pourquoi la résistance à la compression de diverses céramiques d'alumine a été déterminée en fonction de la température. Les résultats sont analysés dans leurs rapports avec la microstructure et avec la morphologie des fractures. Il n'y a aucune corrélation entre les données obtenues et la durabilité des matériaux au pressage à chaud. Cette propriété semble être déterminée exclusivement par l'homogénéité de la céramique et par son absence de défauts.

Abstract - Durability of alumina ceramics when used as die material in compressive loading varies widely. The compressive strength of various alumina ceramics was therefore determined as a function of temperature. The results are discussed in terms of the microstructure and fracture morphology. The (hot-pressing) durability of the materials does not correlate at all with the compressive strength data obtained. Instead, this property seems to be determined entirely by the homogeneity and lack of flaws in the ceramic.

I - INTRODUCTION

In our laboratory during hot-pressing experiments in the temperature range of 800 to 1200°C and at pressures of 0.05 to 0.15 GPa, major differences in durability were found between various alumina ceramics. The prime property to study then seems to be the compressive strength of these alumina ceramics. Contrary to the tensile (or bending) behaviour of ceramics the compressive strength has received only scanty attention. A general discussion on the interpretation of the compressive strength of ceramics has been given by Rice (1). He relates compressive strength to hardness. Data for our particular ceramics were lacking, however, and therefore the compressive strength was measured as function of temperature.

II- EXPERIMENTAL

Although the measurement of the compressive strength, S_C , is conceptually simple, experience with this type of measurement is not wide spread. A broad discussion on the experimental problems is given by Sines and Adam (2).

For three different types of debased alumina specimens were cut of length 18 mm and diameter 6 mm. These were tested between alumina die's (diameter 40 mm) at a strainrate of about $0.3 \cdot 10^{-4} \text{ s}^{-1}$ at various temperatures in air. No interface mate-

rial was used. The measurements were done on a Tinius Olsen Electomatic universal testing machine in a Kanthal wound alumina tube furnace temperature controlled by a microcomputer. The ceramics were also tested at room temperature in water, in order to check on effects of the environment on the compressive strength, S_C . The fracture surfaces of the compression specimens were examined by scanning electron microscopy (SEM).

The microstructure was revealed by optical microscopy (OM) and SEM after polishing (4-7 μm diamond) and thermal etching in air at 1250°C for 4 hours. The grain size distribution was determined by linear intercept measurements from approximately 700 grains. The overall chemical composition was determined by chemical analysis while X-diffraction was used to determine the nature of the second phase. The density, q , was determined from weight and geometric data. The longitudinal wave velocity, v_L , at 10 MHz and the transverse wave velocity, v_S , at 20 MHz were determined at room temperature by the pulse-echo technique using Panametrics 5223 equipment. Young's modulus, E , and Poisson's ratio, ν , were calculated from q , v_L and v_S according to the usual formulae for isotropic materials. The fracture toughness, K_{Ic} , at room temperature was measured in a three-point bending set-up in dry air (dew point - 42°C) using specimens of size 3*9*45 mm³ at a cross-head speed of 1.0 mm/min. A notch with a relative depth of 0.15 and a width of about 150 μm was sawn in each specimen. Precracking was done by a Knoop indentation (5 N load) at the notch root on both sides of the specimen.

Blocks of the different materials normally used at hot-pressing die's typically of 100 mm diameter and 50 mm height, were tested ultrasonically using Sonic Mark IV equipment. Test frequencies used were 5, 10 or 15 MHz with transducers having a diameter of 1/4 or 1/2 inch.

III - RESULTS

In table 1 the main characteristics of the various ceramics are presented. Major differences between the materials are found in the amount and nature of the additives and in the grain size. In all cases the grain size distribution was multi-modal log-normal and the overall modal grain size, D , is indicated. The differences between the materials are also reflected by the values of K_{Ic} . A significant decrease of the compressive strength in water was observed.

Table 1: Characteristics of the various alumina ceramics

Material	A	B	C
$q(\text{g}/\text{cm}^3)$	3.85	3.86	3.80
X-ray (2nd phase)	spinel	-	spinel
$D(\mu\text{m})$	15	31	6.4
wt % Si	0.050	0.10	1.9
wt % Ca	0.046	0.020	0.31
wt % Mg	0.58	0.037	0.27
$E(\text{GPa})$	369	369	347
ν	0.236	0.235	0.240
$K_{Ic}(\text{MPa}\cdot\text{m}^{1/2})$	4.10(0.22)	383(0.08)	5.66(0.20)
$S_C(\text{GPa})$, air	2.11(0.11)	1.90(0.13)	1.83(0.21)
$S_C(\text{GPa})$, water	1.33(0.14)	0.96(0.20)	1.67(0.33)

Sample standard deviations are given in parentheses.

At low temperatures the specimens of all materials essentially exploded forming powder, many tiny chips and some larger pieces. In the temperature range of 1000 to 1200°C the specimens were mostly sheared in the middle.

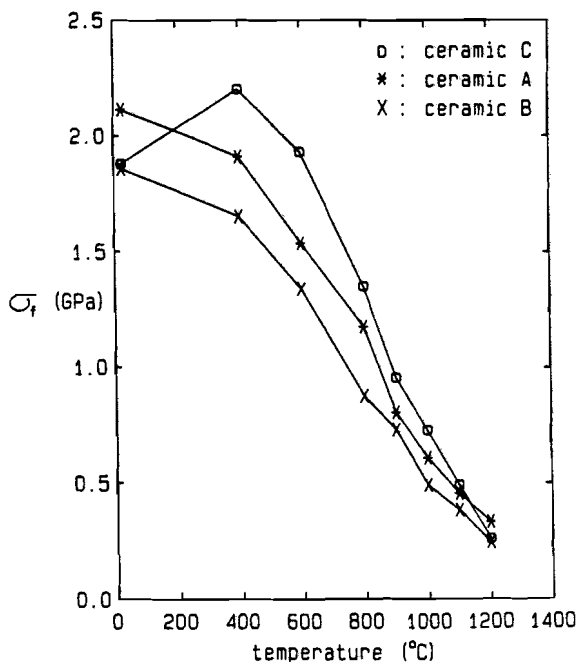


Fig. 1. Compressive strength of the various ceramics as a function of temperature.

For all ceramics clear twinning phenomena were observed upto 800°C. These phenomena were occasionally observed at much higher temperatures. In general the fracture mode was mainly transgranular below about 800°C, changing to mainly intergranular above that temperature.

On the fractographs various microfracture phenomena were observed. Microcracks due to twinning or originating from grain boundary or triple junction voids but also from pores within a single grain. In a number of cases the linking of microcracks could be identified. In fig. 2 some of these phenomena are shown.

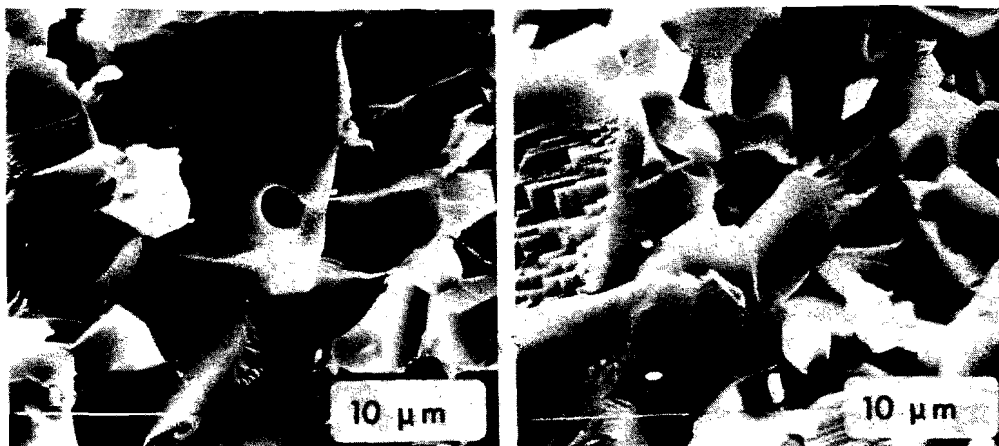


Fig. 2. Microfracture phenomena as detected on the fracture surfaces.
 Left : microfracture originating from a pore inside a grain.
 Right: twinning linked to microfracture originating from a grain boundary pore.

In fig. 1 the compressive strength of the various ceramics is presented. At room temperature the compressive strength correlates well with the fracture toughness. Apart from the maximum in the compressive strength values for one of the materials, no large differences in temperature dependence between the various ceramics are observed. The relative change with temperature R ($\cong 1/\sigma_c \cdot d\sigma_c/dT$) was about $1.1 \cdot 10^{-3} \text{ K}^{-1}$ in the temperature range of 400 to 1200°C.

The data for ceramic C are actually the average of data from two nominally the same materials but produced as rod and plate. As should be the case, the microstructure, chemical composition, mechanical properties and fracture morphology were the same within experimental accuracy. Consequently the data obtained were averaged.

IV - DISCUSSION OF PREVIOUS DATA AND MEASUREMENTS

From the scarce literature data one can distinguish two classes of alumina ceramics as far as S_c is concerned. Rather pure materials with an S_c value typically above 3 GPa and an R value of about $5 * 10^{-4} K^{-1}$ (3-8) and the less pure materials with S_c typically around 2 GPa and an R value of about $1 * 10^{-3} K^{-1}$ (4-9). (The material of Nash (9), although not explicitly stated, probably contains also SiO_2 since he used commercially available materials).

The values obtained for our rather impure ceramics (see table) at room temperature are typically 2 GPa and thus consistent with those of the second class. Also the R value of our ceramics is similar to the rate observed by Dawihl and Dörre (4) for their silicate containing material. Consequently the conclusion must be that for these ceramics the flaw density is higher (lower strength value) and that cavitation is easier (larger rate of decrease).

Most studies of the compressive strength of alumina were done on translucent material (Lucalox), particularly by Lankford (5-8). A significant strain rate effect was observed (5) but an influence of the environment, so manifestly present in tensile testing, was not found. In testing these rather pure materials at room temperature, twinning was observed at least to some extent over a widely varying strain-rate range (5). The twinning led to microcracking starting mostly at grain boundaries. Microcracks also started at voids at triple junctions. After nucleating some growth of the microcracks took place. The coalescence of the microcracks then led to catastrophic fracture. At higher temperatures cavitation in the (supposedly glassy) grain boundary phase was said to take place. Growth and coalescence again led to catastrophic fracture. The observed maximum in compressive strength is explained by a decreased effectiveness of the twinning mechanism while the grain boundary phase is not yet easy flowing (6).

All the phenomena discussed extensively by Lankford for translucent alumina were observed on these debased alumina's as well. In particular the suggestion that pores are probably also a source of microfracture (7) has been found true.

A maximum in S_c was observed for ceramic C only. This is probably related to the large amount of SiO_2 mainly present in the grain boundary phase. The fracture toughness of debased alumina shows a maximum at about 800°C as shown by Davidge and Tappin (10), which is explained by a reduction of the crack tip stress by viscous flow of the glassy second phase, effectively increasing energy dissipation. At slightly higher temperature viscous grain boundary sliding, introducing pores, results in a strength decrease. The viscous flow dissipation mechanism is likely to operate in compressive fracture as well, besides the already mentioned twinning and cavitation mechanisms, but only if enough glassy grain boundary phase is present. The absence of a maximum in S_c for the other ceramics is probably due to the much easier cavitation as compared with translucent alumina.

Contrary to the results of Lankford (5), a significant environmental effect was observed by Nash (9). For a 97.5% pure alumina the value of S_c dropped from 1.7 GPa when tested in air to 1.2 GPa when tested in a physiological salt solution. Similarly for 99.5% pure material, S_c dropped from 2.2 GPa to 1.7 GPa. A significant decrease in S_c was also observed in our case. This probably due to the fact that the specimens used by Nash (and ours) were ground on the outer surfaces while for the specimens of Lankford these surfaces were as-fired. The density of the flaws on the outer surfaces, sensitive to the environment, is much smaller in the latter case thus reducing the environmental effect.

V - RELATION WITH HOT-PRESSING PRACTICE

Comparing the values of S_c with the pressures normally applied during hot-pressing it is clear that the latter are generally much lower. Nevertheless fracture occurs. The order of longer durability in (hot-pressing) practice is: A \gg B \gg C. This order does correlate weakly with the room temperature compressive strength

(order: $A > B = C$) but this correlation is irrelevant since hot-pressing is done at higher temperatures. It does not correlate with the high temperature compressive strength ($A = B = C$). Slow crack growth may be important since at room temperature a clear environmental effect was observed. More important seems to be, however, the homogeneity of the material when delivered in large blocks. For material A the as-delivered blocks used for hot-pressing are essentially free of macroflaws (as tested by pulse-echo technique) but the other ceramics often show some large inhomogeneities probably due to extruding or pressing (fig. 3). Failure of the blocks during hot-pressing is often originating from these defects. This makes the S_C measurements irrelevant. The processing of the ceramics is thus, again, shown to be of vital importance even in the case of compressive loading which is usually considered as a safe type of loading.

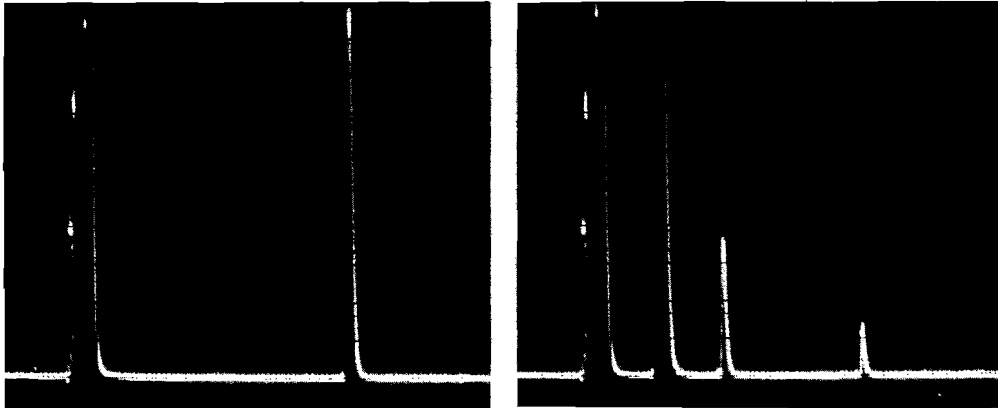


Fig. 3. Ultrasonic pulse echo patterns (A-scans) of a particular block of ceramic B taken at 10 MHz and gain 63 dB.

Left : proper pattern from the edge showing only the transmitting pulse (left) and backside echo (right).

Right: pattern from the centre showing also two defects.

VI - ACKNOWLEDGEMENT

Many thanks are due to Mr. H.C. Smulders for the compressive strength measurements and Mr. C.J. Geenen for the SEM photography.

REFERENCES

- /1/ Rice, R.W., Mater. Sci. Res. Vol. 5 (1970) 195.
- /2/ Sines, G. and Adam, M., "Fracture Mechanics of Ceramics", Vol. 3, R.C. Bradt, D.P.H. Hasselman and F.F. Lange, eds., Plenum, NY, 1978, page 403.
- /3/ Ryshkewitch, E., "Oxide Ceramics: Physical Chemistry and Technology", Acad. Press. NY. 1960.
- /4/ Dawihl, W. and Dörre, E., Ber. Dtsch. Keram. Ges. 41 (1964) 85.
- /5/ Lankford, J., J. Mater. Sci. 12 (1977) 791.
- /6/ Lankford, J. and Davidson, D.L., ICM 3, Vol. 3, Cambridge, UK, 1979, page 35.
- /7/ Lankford, J., J. Mater. Sci. 16 (1981) 1567.
- /8/ Lankford, J., J. Mater. Sci. 18 (1983) 1666.
- /9/ Nash, A., J. Mater. Sci. 18 (1983) 3571.
- /10/ Davidge, R.W. and Tappin, R.W., Proc. Brit. Ceram. Soc. 15 (1970) 47.