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# Weibull statistical analysis of flexure breaking performance for alumina ceramic disks sintered by solar radiation heating

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**Abstract**

Densified alumina ceramic disk specimens were prepared through sintering compacted alumina powders by concentrated solar beam in inert Ar gas atmosphere and their flexure breaking behaviour was evaluated by the ring-on-ring jig test under application of equibiaxial stress. Slope $m$ of Weibull plot for $MOR$ (modulus of rupture) values of these specimens was comparable to that of the reference alumina disk specimens prepared by sintering in an electric furnace under comparable conditions (1600°C for 30 min) whereas mean $MOR$ level of the former was slightly lower than that of the latter. Thus, the failure mechanism of the specimens sintered by solar radiation heating and that of the specimens sintered in the electric furnace were concluded to be practically indistinguishable. This evidence guarantees that alumina ceramic sintered bodies, with quality comparable to those obtained through traditional sintering process, can be prepared by solar heating in spite of quite fast rate of heating and cooling realised in the solar furnace. © 2000 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

*Keywords:* Alumina ceramics; Equibiaxial strength; Weibull statistics; Solar furnace

## 1. Introduction

Solar furnace proved to be a convenient facility for preparing refractory carbides and carbonitrides in relatively short reaction period (typically 30 min) due to its satisfactorily high heat flux (about 1350 $kW/m^2$ from natural solar radiation 800 $W/m^2$ to yield measured temperature up to 1650°C) [1–3]. Rate of heating to the steady state reaction temperature and rate of cooling therefrom are also considerably high in the solar furnace processing. This feature is generally favourable in the production of refractory carbide or carbonitride powders from mixture of metal powders and carbon powders, but this same feature might lead to introduction of undesired thermal stresses and resultant cracking for the sintered body prepared in the solar furnace.

In this work, alumina disk specimens were prepared from compacted pellet of alumina powders under standard solar furnace operation condition (holding at 1600°C for 30 min; heating rate ca. 45°C/min; cooling rate ca. 30°C/min) and their flexure breaking behaviour under application of equibiaxial stress was analysed in terms of Weibull statistics. The mechanical strength of ceramic materials is acknowledged to be critically dependent on microstructure and defects and, as such, it is inherently stochastic in nature, being subjected to influences of the process parameters. Therefore, the test results were evaluated by Weibull statistics.

Values of modulus of rupture ($MOR$) of the solar processed alumina ceramic disk specimens were measured and compared with those of the reference alumina disk specimens prepared by the traditional laboratory sintering method. Mean $MOR$ level as well as the slope $m$ of the Weibull statistics applied on the experimental data for the former were comparable to those for the latter.

## 2. Experimental

High purity Alumina powder (99.7% $Al_2O_3$) CT3000SG supplied by Alcoa (Canada) was used as the

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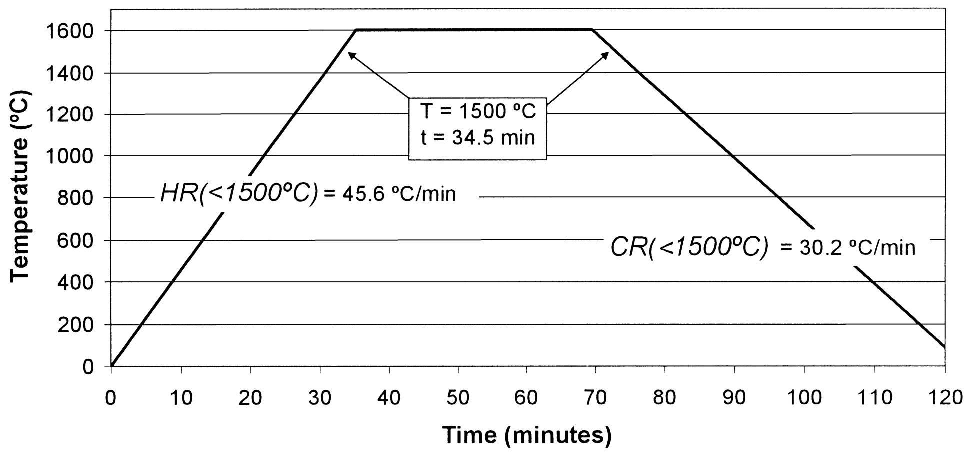


Fig. 1. Schematic temperature profile during the heat process in the solar furnace.

starting material for the present experiments. Mean particle size $d_{50}$ of the CT3000SG powder was 0.5 μm. It was compacted into disk (diameter 12 mm and thickness 3 mm) by applying a pressure of 20 MPa at ambient temperature. Then, each disk was irradiated in a solar furnace at Uzbek Academy of Sciences (Tashkent, Uzbekistan). Fig. 1. shows schematic temperature profile during the processing and Table 1 summarises the detail heating conditions of the valid 20 test runs together with the measured $MOR$ values. $HR(<1500°C)$ refers to the heating rate up to 1500°C; $t(>1500°C)$ represents the period of time in which the specimen temperature was higher than 1500°C; $t(1600°C)$ is the period of time in which the specimen temperature was held at 1600°C; and $CR(<1500°C)$ is the cooling rate below 1500°C. Fluctuation of these process parameters was unavoidable due to weather condition during the solar irradiation, whereas the irradiation was commenced when the sky looked generally clear. Temperature of the specimen was measured by type B Pt-Rh thermocouple being in contact with the backside of each alumina disk specimen.

The solar furnace of Uzbek Academy of Sciences was composed of 62 segments of heliostat mirrors. After the first heliostat was turned on to start heating up the specimen, the other 61 heliostats were successively turned on, one after another, at 3 min intervals. When all 62 heliostats were turned on, the maximum temperature 1600°C was reached.

Table 1
Characteristic process parameter values of the 20 valid test runs and the measured values of $MOR$ (modulus of rupture)

| Test run no. | $HR$ (<1500°C) (°C/min) | $t$ (>1500°C) (min) | $t$ (1600°C) (min) | $CR$ (<1500°C) (°C/min) | $MOR$ (MPa) |
|---|---|---|---|---|---|
| 1 | 69.2 | 33.8 | 27.9 | 28.8 | 144.28 |
| 2 | 52.2 | 37.0 | 32.1 | 28.8 | 158.26 |
| 3 | 44.9 | 36.3 | 30.4 | 24.7 | 146.10 |
| 4 | 42.8 | 40.0 | 31.3 | 28.3 | 182.19 |
| 5 | 38.2 | 31.7 | 8.5 | 28.3 | 177.14 |
| 6 | 36.7 | 34.8 | 31.8 | 29.3 | 126.65 |
| 7 | 43.5 | 32.4 | 30.7 | 25.8 | 108.55 |
| 8 | 49.8 | 35.1 | 31.5 | 32.1 | 114.74 |
| 9 | 38.2 | 38.0 | 32.5 | 31.2 | 136.06 |
| 10 | 42.1 | 34.8 | 29.5 | 31.7 | 114.59 |
| 11 | 42.1 | 35.8 | 31.7 | 29.5 | 152.69 |
| 12 | 53.0 | 36.2 | 31.0 | 33.2 | 114.98 |
| 13 | 48.8 | 32.0 | 30.3 | 34.5 | 106.38 |
| 14 | 39.6 | 33.2 | 30.8 | 30.1 | 106.74 |
| 15 | 44.2 | 32.8 | 31.0 | 32.1 | 120.88 |
| 16 | 50.7 | 33.9 | 31.3 | 36.0 | 141.87 |
| 17 | 56.2 | 31.6 | 30.3 | 30.2 | 96.76 |
| 18 | 41.5 | 33.3 | 30.6 | 28.6 | 133.11 |
| 19 | 41.5 | 33.7 | 31.0 | 29.7 | 121.82 |
| 20 | 37.4 | 32.9 | 30.8 | 31.1 | 120.52 |
| Mean value | 45.6 | 34.5 | 29.7 | 30.2 | 131.22 |
| Standard deviation | 7.9 | 2.2 | 5.1 | 2.7 | 23.50 |

Comparable number of reference alumina disk specimens (24) were prepared in laboratory using an electric furnace (holding for 30 min at 1600°C; $HR(<1600°C) = CR(<1600°C) = 20°C/min$).

The equibiaxial tests on the disk specimens were carried out in an Instron machine equipped with a self-aligning ring-on-ring jig, shown in Fig. 2, using a cross-head speed of 0.5 mm/min. The fracture stress (Modulus of Rupture, $MOR$) in equibiaxial conditions is calculated according to [4]:

$$MOR = \frac{3 \cdot F_r}{2 \cdot \pi \cdot e^2} \cdot \left\{ (1+\nu) \cdot \ln\left(\frac{R_2}{R_1}\right) + (1-\nu) \cdot \frac{R_2^2 - R_1^2}{2 \cdot R_0^2} \right\}$$

where $F_r$ refers to the rupture load, $e$ the specimen thickness, $\nu$ the Poisson's ratio of the specimen material (0.26), $R_1$ the inner (upper) ring radius (3.8 mm), $R_2$ the outer (lower) ring radius (11 mm), and $R_0$ the specimen radius.

## 3. Results and discussion

The determined values of $MOR$ for the solar processed specimens are listed in Table 1 and the corresponding Weibull plot for these results is given in Fig. 3. On the other hand, Fig. 4 shows the Weibull plot for the reference specimens. The mean value of fracture stress obtained for the reference specimens was $MOR = 166.1 \pm 29.2$ MPa, which was higher than that attained for the solar-sintered specimens (see Table 1). Probable cause leading to the lower mean value of $MOR$ for the solar-sintered specimens than for the specimens prepared by traditional laboratory furnace is the induction of greater internal stresses in the solar-sintered disks due to higher heating and cooling rates attained in the solar furnace.

As seen in Figs. 3 and 4, Weibull modulus $m$ (slope of the fitted curve to the experimental data) for the solar processed specimens and that for the reference specimens were comparable to one another. This is indicative of the fact that the failure mechanism of the former and that of the latter were almost identical to one another. That is, the type and the density of defects in the alumina disk specimens prepared by solar radiation heating are almost the same as those in the reference alumina specimens produced by traditional laboratory sintering procedure, in which $HR$ and $CR$ values were approximately half than those in the solar furnace. This evidence gives guarantee that alumina sintered bodies with

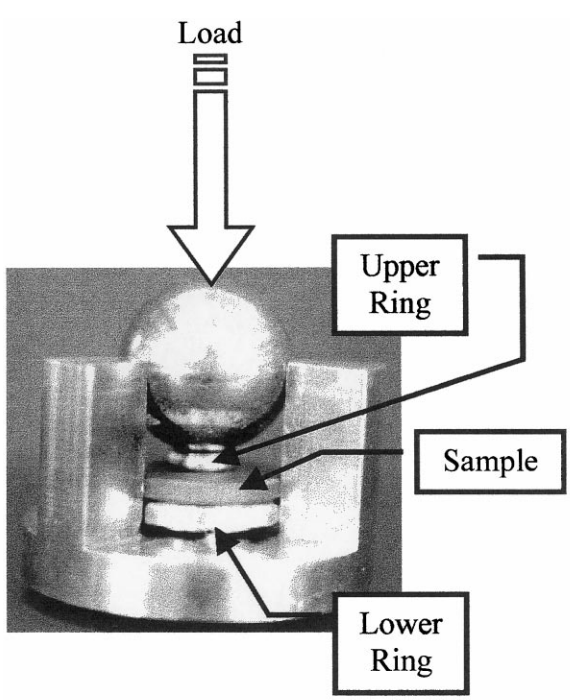


Fig. 2. Aspect of the self-aligning ring-on-ring jig used for equibiaxial tests for disk specimens.

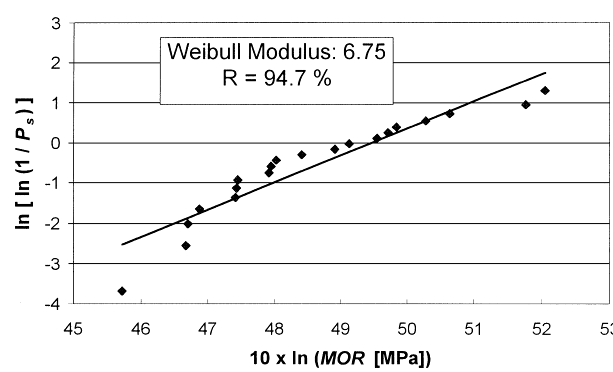


Fig. 3. Weibull plot of $MOR$ for the solar processed specimens. $P_s$ represents the probability of survival with $P_{sj}$ of the $j$th $P_s$ (in ascending order of $MOR$) defined as $P_{sj} = 1-(j-0.5)/N$, where $N$ is the total number of tested specimens. R is the data correlation factor.

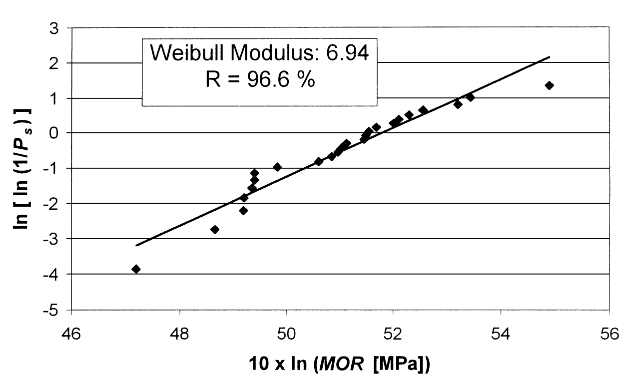


Fig. 4. Weibull plot of $MOR$ for the reference specimens sintered in a laboratory furnace with $HR(<1600°C) = CR(<1600°C) = 20°C/min$.

quality comparable to those prepared by the traditional method might be produced in the solar furnace.

## 4. Concluding remarks

The present preliminary experimental evidences indicated that a solar furnace is acceptable as sintering furnace for consolidated ceramic components production as well as a furnace for producing non-consolidated refractory carbides and carbonitrides [1–3]. In the present work, standard heating and cooling operation of the solar furnace was practised and this led to considerably higher rate of heating and cooling than that in a traditional electric furnace. By retarding somehow the rate of heating and cooling in the solar-sintering process, mechanical properties of the produced ceramic sintered bodies might be improved.

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