

MECHANICAL PROPERTIES AT HIGH TEMPERATURE OF
HIGH-PURITY Al_2O_3

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ABSTRACT

The mechanical properties at high temperature of high-purity Al_2O_3 fabricated using seven kinds of commercial Al_2O_3 powder, which were prepared by the Bayer-process and from aluminium compounds, were investigated. The density, grain size and its distribution of sintered samples were different from each powder. Impurity content and compositions influenced on the strength, the creep strain rate and the internal friction at high temperature. And also, the grain size distribution affected the strength and the creep strain rate.

INTRODUCTION

Al_2O_3 ceramics are one of the typical oxide ceramics and promising for engineering applications, because of its desirable properties such as high refractoriness, good wear resistance and chemical stability¹⁾. However, Al_2O_3 ceramics show relatively rapid strength degradation at high temperature. This is considered to be influenced on the plastic deformation by the grain boundary-sliding, which is mainly caused by glassy phase softening²⁾. Thus, it is expected that these undesirable properties are improved by means of reducing impurities.

While many investigators have studied the mechanical properties at high temperature such as the strength and the creep resistance³⁻⁵⁾. They have scarcely investigated the effect of high purification of Al_2O_3 (>99%) on those properties.

In this study, the mechanical properties at high temperature of Al_2O_3 , which were fabricated from commercial high-purity Al_2O_3 powder, were investigated.

EXPERIMENTAL PROCEDURE

Seven kinds of commercial high-purity Al_2O_3 powders were selected as starting powders. Samples A, C and D were prepared by the Bayer-process, and samples B, E, F and G were from aluminium compounds. The particle size of samples B, E, F and G was smaller than that of samples A, C and D, and the particle size distribution of samples B, E, F and G was narrower than samples A, C and D. These powders, as received, were isostatically pressed at 100MPa and sintered at 1650°C for 2h in air.

The density was measured by Archimedes method using distilled water. The Young's modulus was evaluated by the flexural vibration method⁶⁾. The internal friction was evaluated by the torsional vibration method⁶⁾. The flexural strength was measured at R. T. to 1400°C in air by 3-point bending test on a 30mm span using 3x4x40mm test specimens at cross head speed of 0.5mm/min. The tensile surfaces of the testing specimens were ground by #600 grid diamond wheel. Creep tests were carried out at 1300, 1400 and 1450°C in air by 4-point bending method. The major and minor spans were 30 and 10, respectively, and the strain rate and stress-exponent were calculated using Eqs. (1) and (2)⁷⁾.

$$\sigma = \frac{3P(L-1)}{2BD^2} \left(\frac{2n+1}{3n} \right) \quad \text{-----(1)}$$

$$\dot{\epsilon} = \frac{2D(n+2)}{(L-1)(L+1(n+1))} \dot{y} \quad \text{-----(2)}$$

where σ is the maximum stress, P is the applied load, n is the stress exponent and \dot{y} is the deflection rate at the load point. B and D are

the width and thickness of specimen and L and l are the major and minor spans, respectively.

The chemical compositions of the sintered samples were analyzed by the Inductively Coupled Plasma Emission Spectrometry.

The microstructure was observed by Scanning electron micrograph (SEM) after thermal etching, and the average grain size of each sample was determined by the intercept method⁸⁾.

RESULT AND DISCUSSION

Table 1 shows the density, Young's modulus, flexural strength and average grain size of each sample, and Table 2 shows the chemical compositions. SEM of thermally etched-surface after polishing shows in Fig. 1. Each sample had 98% of theoretical density or greater value.

Table 1 Density, Young's modulus, flexural strength and grain size for each sample

		A	B	C	D	E	F	G
Density (g/cm ³)		3.92	3.96	3.91	3.93	3.96	3.97	3.96
Young's Modulus (GPa)		363	378	367	365	383	383	386
Flexural Strength (MPa)	R. T.	348	326	342	375	334	448	482
	1400°C	86	185	152	178	181	238	217
Grain Size (μm)		7.9	11.2	6.9	8.3	6.3	7.4	7.2

The density and Young's modulus of samples B, E, F and G prepared from aluminium compounds were higher than those of samples A, C and D by the Bayer-process, and grain size distribution of samples B, E, F and G were narrower than samples A, C and D. These differences are attributed to powder characterizations such as particle size, particle size distribution, chemical compositions and properties of formed bodies

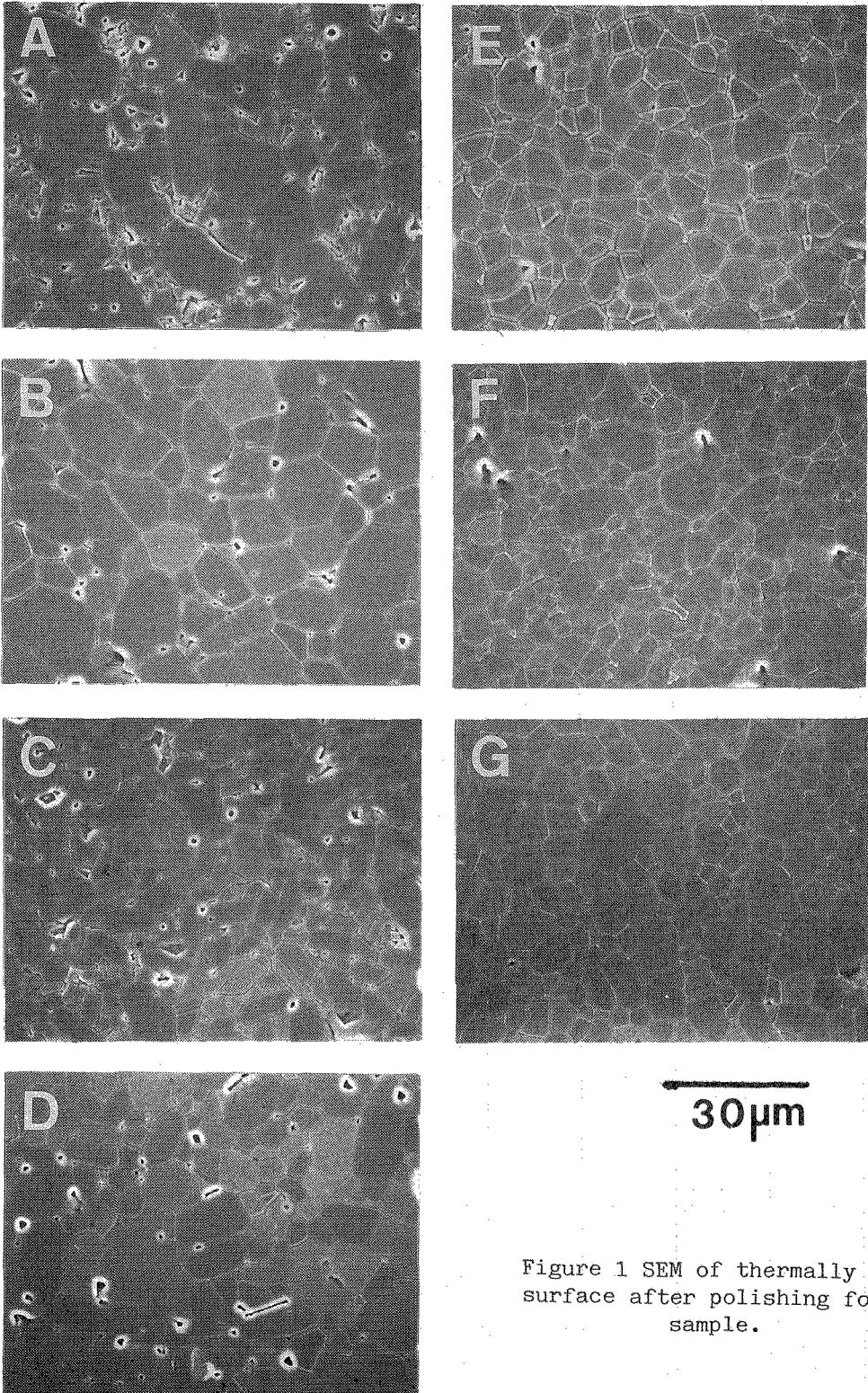


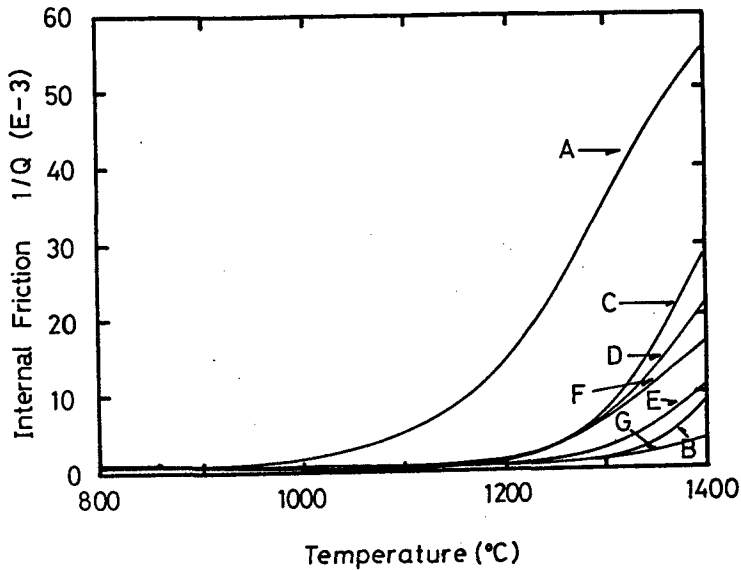
Figure 1 SEM of thermally etched surface after polishing for each sample.

Table 2 Chemical compositions for each sample

	A	B	C	D	E	F	G
Al_2O_3 (wt%)	99.79	99.99	99.83	99.94	99.99	99.94	99.99
SiO_2 (ppm)	769	51	306	25	48	99	37
Fe_2O_3 (ppm)	137	12	135	110	12	9	N. D.
CaO (ppm)	353	4	150	32	N. D.	N. D.	7
MgO (ppm)	470	N. D.	529	348	6	491	25
Na_2O (ppm)	241	N. D.	505	54	13	35	19
K_2O (ppm)	95	N. D.	10	N. D.	2	5	N. D.

such as the compactness and uniformity. Impurity of samples prepared from aluminium compounds was less than that of samples prepared by the Bayer-process. Especially, as sample A containing much impurity, it was suggested to contain much glassy phase at grain boundaries, because impurity such as alkali and alkali earth oxides react with SiO_2 to form glassy phase at high temperature.

The temperature dependence of the internal friction of each sample is shown in Fig. 2.



The temperature at which the internal friction increases and the internal friction value were different from each sample. The temperature at which the internal friction of sample A containing much impurity and sample B, E and G containing less impurity increases was 900 and 1200°C, respectively. And the internal friction of sample A at 1400°C was 10 times increased as high as that of sample G. In the samples containing the same impurity content, the temperature at which the internal friction of all samples increases was same, but the internal friction value was different. These differences are considered to depend on the glassy phase content and compositions to be formed by impurity⁶⁾. It is suggested that sample A contains much low molten glassy phase at grain boundaries and sample G contains less glassy phase than samples B and E.

Figure 3 shows the temperature dependence of the flexural strength of each sample.

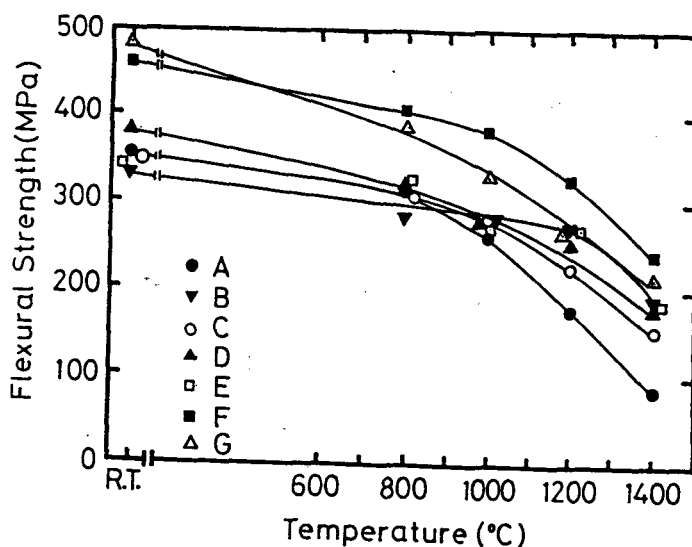


Figure 3 Temperature dependence of the flexural strength for each sample

The strength of samples F and G at R. T. was higher than other samples. The strength at R. T. is thought to depend on not the grain size,

impurity content and compositions, but the density and Young's modulus. Samples F and G are suggested to have less defects than other samples. The remarkable strength degradation of samples A and B at high temperature revealed above 900 and 1200°C, respectively. These temperature are closely related to the temperature at which the increases of the internal friction value⁶⁾. The strength degradation at high temperature is suggested to be influenced on the plastic deformation by the grain boundary-sliding which was caused by glassy phase softening²⁾. The impurity content of sample G was the same as that of samples B and G, and the internal friction value of sample G was lowest of all. The temperature dependence of the strength indicated a similar trend to that of sample A, however, this degradation was attributed to not impurity compositions, but the grain size distribution and small grains.

The steady-strain creep vs. stress from 5 to 200MPa at 1300, 1400 and 1450°C for each sample is shown in Figs. 4(A), (B) and (C). The strain rate of samples containing less impurity was low, and that of samples containing much impurity was high. These results indicated that the strain rate was in proportion to the internal friction value. However, sample G showed the low internal friction at high temperature, the strain rate was higher than that of samples B and E. It is considered that the strain rate depends on not influence of impurity content and compositions, but that of the grain size distribution and small grains, as discussed about the strength. Exponent for sample A, at low stresses, the stress exponent, n was 1 to 1.2. This result indicated that the deformation of each sample is mainly diffusional creep and slightly influenced by the grain boundary-sliding and/or dislocation climb. As the grain size exponent, m of samples B and D was 2.3, the creep mechanism is suggested to be Nabarro-Herring mechanism. The stress exponent of sample A increased with increasing temperature. And other samples showed a similar trend to sample A. It is considered that the deformation at high temperature and at high stresses is mainly caused by the grain boundary-sliding as same as the strength degradation.

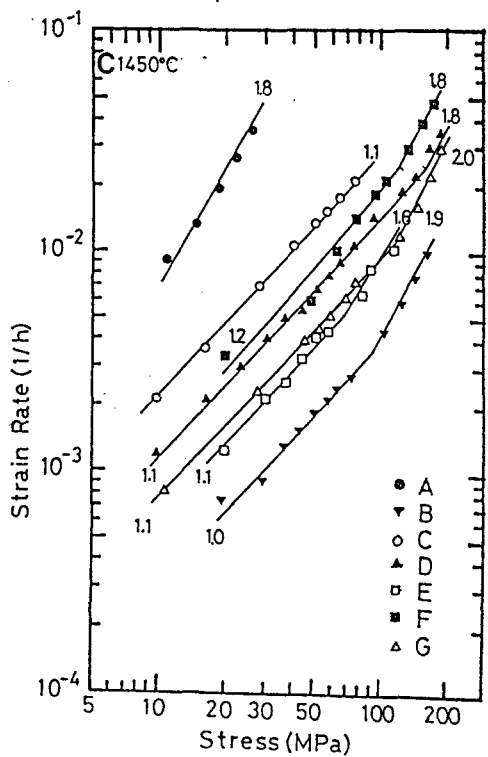
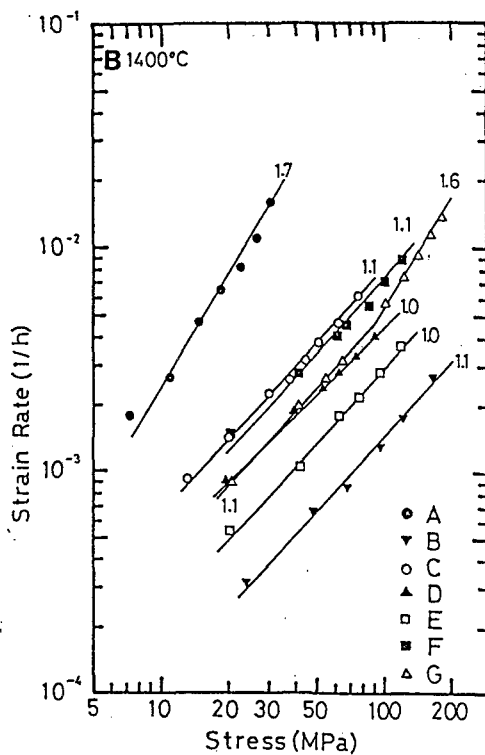
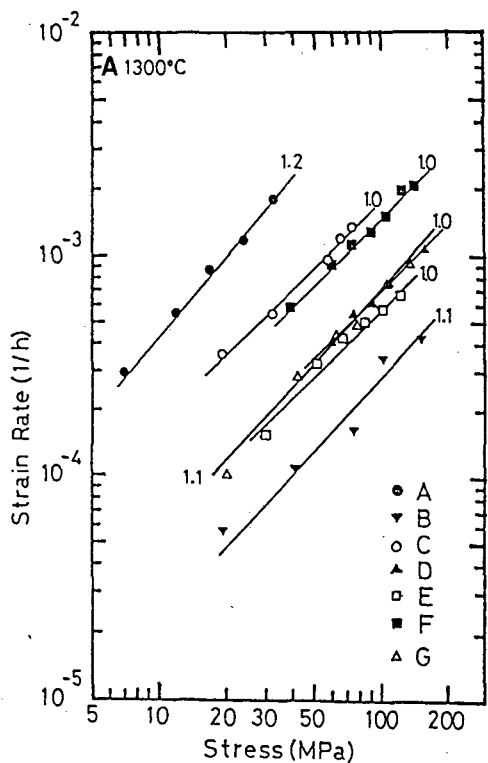


Figure 4 Steady-strain creep vs. stress at (A);1300°C, (B);1400°C and (C);1450°C for each sample.

CONCLUSIONS

Mechanical properties at high temperature of Al_2O_3 , which were fabricated from commercial high-purity Al_2O_3 powder, were investigated.

1) The density, Young's modulus, the grain size and the grain size distribution of each sample were different from each powder.

2) The temperature dependence of the internal friction was influenced by impurity content and compositions.

3) The strength at R. T. was independent of the grain size and impurity.

4) The strength degradation and creep strain rate at high temperature were in agreement with the internal friction. But, in case of the wide grain size distribution, this relation was not necessarily supported.

REFERENCES

1. H. Abe, et al., "Engineering Ceramics," pp. 22-3, in Ceramic Science Series, vol. 5. GIHODO, Tokyo, 1984.
2. Y. Fukuura, "Recent progress of High-purity Al_2O_3 ," pp. 73-95, in Proceedings of Raw Material Divisional Meeting of the Ceramics Society of Japan, Tokyo, 1979.
3. T. Nishikawa, T. Nishida, Y. Okamoto and K. Asano, "Diffusional Creep of Polycrystalline Al_2O_3 Doped with MgO," J. Ceram. Soc. Jpn., 87 [2] 46-54 (1979)
4. R. M. Cannon, W. H. Rhodes and A. H. Heuer, "Plastic Deformation of Fine-Grained Alumina:I, Interface-Controlled Diffusional Creep," J. Am. Ceram. Soc., 63 [1-2] 46-58 (1980)
5. W. Blumenthal and A. G. Evans, "High-Temperature Failure of Polycrystalline Alumina:II, Creep Crack Growth and Blunting," J. Am. Ceram. Soc., 67 [11] 751-59 (1984)
6. K. Matushita, "Internal Friction Investigation of Ceramics and Fiber Reinforced Ceramics," pp. 63-83, in Doctoral Thesis, 1990.

7. G. W. Hollenberg, G. R. Terwilliger and R. S. Gordon, "Calculation of Stresses and Strains in Four-Point Bending Creep Tests," J. Am. Ceram. Soc., 54 [4] 196-9 (1971)
8. R. L. Fullman, "Measurement of Particle Size in Opaque Bodies," [3] 447-52 (1953)
9. E. M. Passmore, R. M. Springgs and T. Vasilos, "Strength-Grain Size Porosity Relations in Alumina," 48 [1] 1-7 (1965)