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Influence of humidity on the flexural strength of alumina

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Abstract

The influence of humidity on the flexural strength of alumina was investigated. The strengths tested at relatively high relative humidities (>45%) were smaller than those tested at relatively low relative humidities (<45%) as much as ~10%. It was demonstrated by a round robin test that the influence of the humidity could produce a problem in the reproducibility of the strength data. It was discussed, that the problem in the reproducibility can be avoided by testing and using the inert strength in the comparison of strength data. It was recommended that, to test the inert strength, the strength measurement be made after the specimens had been coated with oil to protect them from contact with environmental moisture.

Keywords: Al_2O_3; Humidity; Strength

1. Introduction

In many ceramics, cracks grow slowly at stress intensity factors below a critical value required for a fast fracture. This sub-critical crack growth relates to a stress-assisted chemical reaction at the tip of cracks between material constituents and environmental constituents (especially moisture).[1,2] The sub-critical crack growth occurs on loading in strength testing. Therefore, testing conditions can influence the measured strength data in association with this sub-critical crack growth. For example, a higher crosshead rate yields a higher strength value because of a reduced sub-critical crack growth until breaking.[3] Since the kinetics of sub-critical crack growth is significantly influenced by the content of moisture in the environment,[1] the humidity in the testing laboratories can also influence the measured strength.[4] However, investigations of the influence of humidity on the strength of ceramics are rare. McMahon[5] reported that the flexural strength of alumina decreases with the increase of the relative humidity. However, his results were based on a limited number of test specimens. In this paper, we report the influence of relative humidity on the flexural strength of alumina in more detail.

2. Experimental procedure

Alumina of 99.5% purity (Coors AD995) was used in this investigation. The material was received in the form of plate, 10×10×1 cm. The plates were cut and ground into flexure specimens of 3×4×40 mm or 3×4×50 mm. Coarse grinding was done with a 400-grit resin-bonded diamond wheel at the rate of 15 μm per pass. Final grinding of at least 30 μm was done with the 800-grit wheel at the rate of 2 μm per pass. About 0.12 mm of specimen edges were chamfered with the 800-grit wheel at the rate of 2 μm per pass. Specimens machined from several plates were randomized to avoid a plate-to-plate variation in the strength data.

All strength testing was done at room temperature. Two four-point semi-articulation fixtures having different spans were used: one had the outer and inner spans of 30 and 10 mm, and the other, 40 and 20 mm, respectively. Both the fixtures had rotating roller bearings. The relative humidity of the laboratory was controlled during the testing. During the season when the testing was done, the ambient relative humidity in the laboratory ranged from 60 to 80%. A range of relative humidity lower than this ambient value was controlled

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by using a dehumidifier. The use of the dehumidifier increased the laboratory temperature, but the increase was less than 8°C in any case. To test an inert strength, some specimens were tested after their surfaces were coated with liquid paraffin. The liquid paraffin may contaminate moisture. To get rid of the moisture, it was heat-treated at 170°C for 2 h. Non-heat-treated liquid paraffin was also used for the purpose of comparison. A minimum of 10 specimens were tested for each testing condition.

3. Results and discussion

Preliminary tests revealed that the alumina was appropriate for our investigation in terms of the consistency of strength data. The coefficient of variation (standard deviation/average) of the flexural strength data was only ~6%. Unbiased Weibull modulus estimated by maximum likelihood method with the strength data of 30 specimens was ~20.

Fig. 1 shows the change of flexural strength tested using the fixture having the spans of 30 and 10 mm at a constant crosshead rate of 0.5 mm/min as a function of relative humidity. The data point at the relative humidity of zero presents the inert strength tested after the specimens coated with heat-treated liquid paraffin. The value of this inert strength was 330 MPa. The strengths tested at low relative humidities (< 45%) were similar to this inert strength. However, the strengths tested at higher relative humidities (> 45%) were in the range from 296 to 298 MPa, similar to each other but significantly smaller than the inert strength (as much as ~10%).

Fig. 2 shows the change of strength as a function of crosshead rate at various conditions. The crosshead rates were varied to be 0.005, 0.05 and 0.5 mm/min. When the specimens were coated with heat-treated liquid paraffin, the strength did not change with the crosshead rate and the inert strengths were obtained at all the crosshead rates. When the specimens were coated with non-heat-treated liquid paraffin, the strength was similar to the inert strength at the crosshead rates of 0.05 and 0.5 mm/min, but significantly smaller at the rate of 0.005 mm/min. When specimens were tested at relative humidity 40%, the strength was similar to the inert strength only at the crosshead rate of 0.5 mm/min, and decreased with the decrease of the crosshead rate. At the humidity of 70%, the strength was not similar to the inert strength at any crosshead rates and decreased again significantly with the decrease of the crosshead rate.

Results shown in Figs. 1 and 2 indicate that the flexural strength of alumina is influenced significantly by the relative humidity as well as crosshead rate. Specifically, the influence of relative humidity implies that the strength tested in an uncontrolled environment may not be an intrinsic material property but a humidity-dependent extrinsic property. The influence of the humidity, in turn, can produce a problem in terms of the reproducibility and repeatability of the strength data, because the humidity is different from place to place and changes from time to time even in the same place.

In fact, we have performed a round robin test (RRT) on the flexural strength of alumina. We present the results briefly here, because they may demonstrate an example of the problem in the reproducibility of the strength data. Eight domestic laboratories including our laboratory and two foreign laboratories (one from Shanghai, China and the other one from Bangkok, Thailand) took part in the round robin. We prepared the specimens of the same alumina as that used in the present investigation and distributed 30 specimens to each laboratory. All the laboratories tested the strength at a constant crosshead rate of 0.5 mm/min using semi-articulating four-point fixtures with rotating roller bearings. All domestic laboratories used the fixtures having outer and inner spans of 30 and 10 mm, respectively,

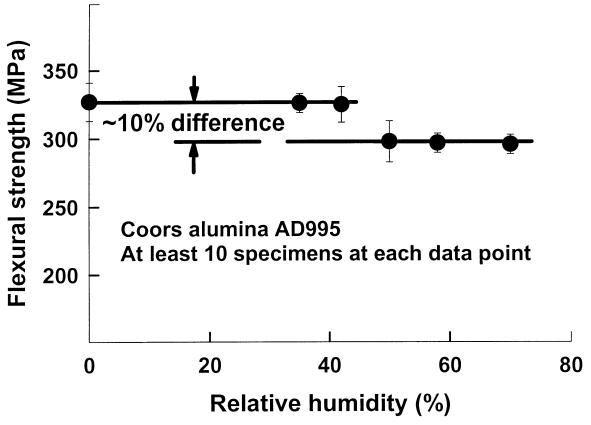

Fig. 1. Change of flexural strength as a function of relative humidity. Error bars represent 95% confidence intervals.

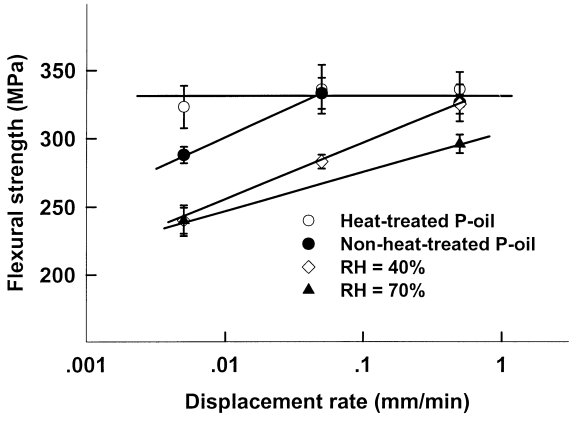

Fig. 2. Change of flexural strength as a function of crosshead rate at various conditions. Error bars represent 95% confidence intervals.

whereas the two foreign laboratories used the fixtures having the outer and inner spans of 40 and 20 mm, respectively. Our laboratory used both the fixtures. The strengths from domestic laboratories are shown in Table 1 and by Weibull plot in Fig. 3. The strengths from the laboratories A–E and our laboratory ranged from 321 to 334 MPa, well consistent with each other and to the inert strength shown in Fig. 1. However, the strengths from the other two laboratories, F and G, were 308 and 309 MPa, respectively, significantly smaller.

The strengths from the two foreign laboratories and our strength tested by the fixtures having outer and inner spans of 40 and 20 mm, respectively, are shown in Table 1 and in Fig. 4. Our strength tested at the relative humidity of 35% was 338 MPa, slightly higher than the strength tested at the same relative humidity using the fixture having the outer and inner spans of 30 and 10 mm. However, 95% confidence intervals of the two strength data were overlapped, indicating that the difference between the strengths tested by the two fixtures was not significant. [This is attributable to high Weibull modulus (~20) of this particular alumina. Estimation based on Weibull volume calculation predicts that the difference in strength for two test geometries is only ~3.2%.] However, the strengths from the two foreign laboratories and our strength tested at the relative humidity 75% were again significantly smaller (< 300 MPa).

Above RRT results indeed demonstrate the problem in the reproducibility of strength data: some laboratories obtained the strengths comparable with each other and to the inert strength but some other laboratories obtained the strengths significantly smaller. Unfortunately, we could not correlate the strengths from laboratories with the relative humidity, because none of these laboratories except our laboratory recorded the relative humidity at the time of strength testing. However, examinations of data sheets from the domestic laboratories revealed that the laboratories A–E tested the strength in the dry season, while the laboratories F and G tested the strength in the rainy season. Therefore, the problem in the reproducibility of strength data was attributable to the difference between the humidities at the two groups of laboratories. Because the data from the two foreign laboratories were similar to our data tested at high humidities (>45%) (Fig. 1), it is also believed that the humidities at these foreign laboratories were relatively high at the time of testing.

To characterize the inherent material strength, it is required to test the humidity-insensitive inert strength. If the inert strengths were tested and used in making data comparison, the problems of repeatability and reproducibility can also be avoided. The inert strength can be tested either by controlling the relative humidity sufficiently low or by coating the specimens with oil to prevent them from contact with environmental moisture. Because it may be costly to control the relative

Table 1
Summary of round robin test results

Laboratory	Fixture spans (mm/mm)	Relative humidity (%)	Strength (MPa)[b]
A	30/10	n.m.[a]	328 ± 7.2
B	30/10	n.m.	334 ± 5.8
C	30/10	n.m.	329 ± 6.3
D	30/10	n.m.	326 ± 5.9
E	30/10	n.m.	321 ± 6.9
Our lab.	30/10	35	326 ± 5.9
F	30/10	n.m.	308 ± 8.6
G	40/20	n.m.	309 ± 8.8
Our lab.	40/20	35	338 ± 6.6
Our lab.	40/20	75	280 ± 6.2
H	40/20	n.m.	296 ± 6.8
I	40/20	n.m.	298 ± 7.9

[a] Not measured.
[b] Average and 95% confidence limits are shown.

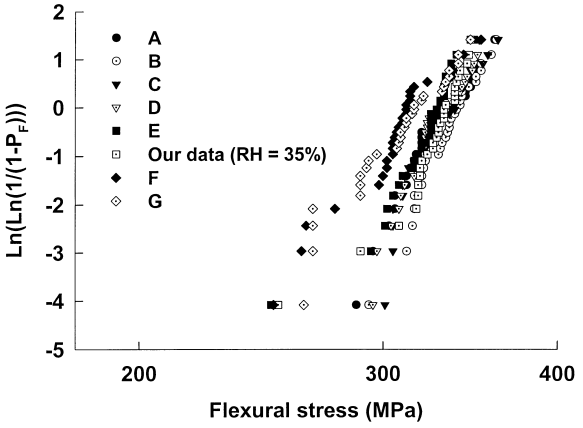

Fig. 3. Weibull two-parameter graphs for the round robin strength data as measured with fixtures having outer and inner spans of 30 and 10 mm, respectively.

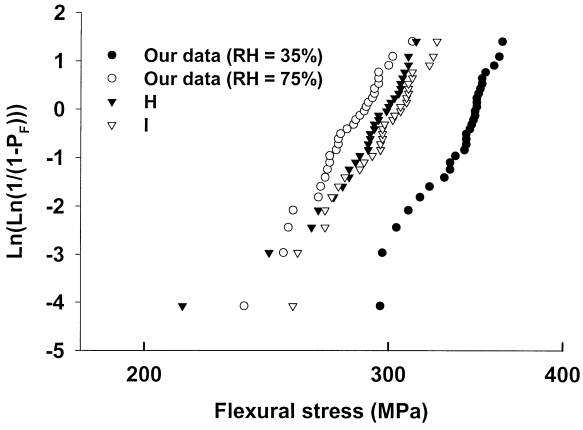

Fig. 4. Weibull plots for the round robin strength data as measured with fixtures having outer and inner spans of 40 and 20 mm, respectively.

humidity, it is more desirable to use the oil to test the inert strength.

Finally, in this paper, we reported the influence of humidity on the flexural strength observed in alumina. However, the influence of humidity is not limited to the present alumina only. We have also studied the influence in silicon nitride and silicon carbides. The influence existed in silicon nitride of which the microstructure contains a grain boundary glassy phase, whereas it did not in sintered and siliconized silicon carbides.

4. Conclusion

The strength of alumina tested at high relative humidities ($>45\%$) is smaller than that tested at low relative humidities ($<45\%$) as much as $\sim 10\%$. Such a large influence of humidity produces a problem in the repeatability and reproducibility of strength data. The problem in the repeatability and reproducibility can be avoided by testing and using the inert strength in the comparison of strength data. It is recommended that, to test the inert strength, the strength measurements be made after coating the specimen surfaces to protect them from contact with environmental moisture.

Acknowledgements

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