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Contact-Induced Strength Degradation of Thermally Treated Al_2O_3

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FINE-GRAINED alumina can be strengthened (typically from ≈400 to ≈700 MPa) by quenching from temperatures of 1500 to 1700°C.[1–5] There is evidence (viz. end deflection of treated rods after slotting,[3] thermal stress analysis of the quenching process,[6] and fracture mirror measurements[7]) that the strengthening results from a residual compressive stress in the surface. However, it is not unequivocally established that there is a complete correlation between surface compression and strength improvement. In this context, Gupta[8] showed that heating alumina at the above temperatures causes a certain amount of flaw healing. It is therefore uncertain that the initial strengthening effect in treated surfaces will be preserved under stringent in-service conditions; mechanical surface damage can introduce new dominant flaws, and thereby degrade the strength characteristics.[9–14]

The purpose of this note is to investigate the strength characteristics of untreated and quenched alumina rods (3 mm in diam., 30 mm long)* after they have been subjected to surface-degrading contact events. Following the approach developed in a previous study of thermally tempered glass,[14] the characteristics of the quenched rods can be predetermined using indentation fracture parameters (including residual stress) obtained from calibration indentation/strength tests.

Experimentally, contact damage was produced by indenting the rods with a Vickers pyramid, with loads of 2 to 100 N. This loading produces half-penny median cracks which expand about the contact point on planes containing the load axis and an indentation diagonal.[15] The strength was then measured in 4-point flexure with the indented side of the rod in tension. Comparative data are shown in Table I for four groups of nonindented and indented rods; the groups represent different thermal treatments. More comprehensive strength-degradation data were collected for the as-received and the oil-quenched rods; these data are plotted in Fig. 1 as a function of indentation load.

Table I. Strength and Residual-Stress Data for As-Received and Thermally Treated Al_2O_3

Treatment	Measured strength (MPa) Nonindented ($P=0$)	Indented ($P=100$ N)	Residual stress (MPa)
As-received	440 ± 28	123 ± 16	0 ± 12
Furnace-cooled*	503 ± 42	135 ± 7	2 ± 12
Air-cooled*	572 ± 49	170 ± 8	34 ± 15
Oil-quenched*	684 ± 38	252 ± 31	106 ± 30

*From 1550°C.

Theoretical analysis of the strengths of treated rods follows from the fracture mechanics of the indentation and flexure tests.[14] The characteristic radius c of a well-developed half-penny crack produced by an indentation load P is given by

$$P/c^{3/2}=(K_c/\chi)(1+2\sigma_R c^{1/2}/\pi^{1/2}K_c) \tag{1}$$

where σ_R is the residual compression stress, K_c is the critical stress-intensity factor, and χ is a dimensionless indenter constant. For surfaces in which such a crack constitutes the dominant flaw, the subsequent strength in flexure is

$$\sigma=\pi^{1/2}K_c/2c^{1/2}+\sigma_R \tag{2}$$

Elimination of the crack length from Eqs. (1) and (2) yields a relation between the strength and the indenter load:

$$P=(\pi^{3/2}/8)(K_c^4/\chi)[\sigma/(\sigma-\sigma_R)^4] \tag{3}$$

Prediction of the strengths of treated rods using Eq. (3) then requires that K_c, χ, and σ_R be known. The parameters K_c and χ were found from two calibration runs on as-received rods. In the first run, surface traces of indentation cracks were measured as a function of indenter load (30 cracks): in conjunction with Eq. (1) (with $\sigma_R=0$), this gave $K_c/\chi=P/c^{3/2}=(28\pm1)$ MPa m$^{1/2}$. In the second run, the best fit of Eq. (3) (with $\sigma_R=0$) to the data in Fig. 1 for as-received rods gave $(\pi^{1/2}/2)(K_c^4/\chi)^{1/3}=\sigma P^{1/3}=(600\pm30)$ MPa N$^{1/3}$. These two equations solve simultaneously to give

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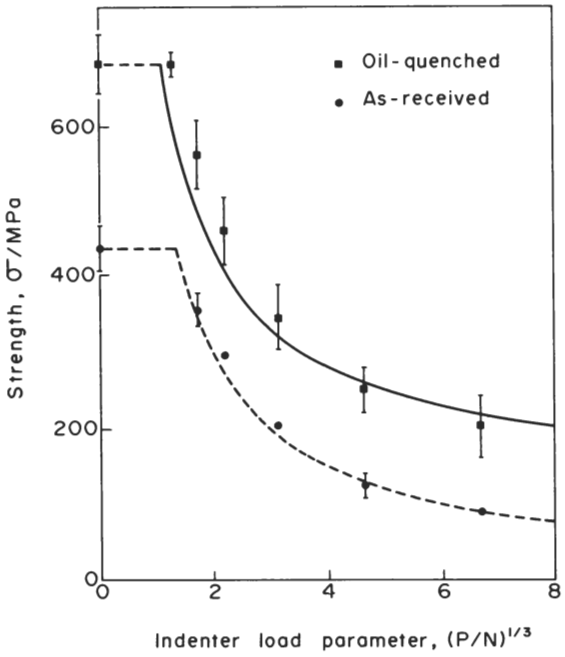

Fig. 1. Strength as a function of Vickers indentation load for as-received and oil-quenched Al_2O_3 rods. Data points with error bars represent mean and standard deviation of 4 to 10 tests. *Horizontal broken lines* represent strength cutoff at measured strength of nonindented surfaces; *broken curve* is best fit of Eq. (3) with $\sigma_R = 0$ to data for as-received surfaces; *solid line* is strength of oil-quenched rods predicted from Eq. (3).

$K_c = (2.23 \pm 0.14)$ MPa m$^{1/2}$,* and $\chi = 0.080 \pm 0.008$. The magnitude of σ_R was determined by direct application of Eq. (1),[18] using indentation crack lengths measured at specified loads and the constant K_c/χ as evaluated above; these values are listed in Table I. The strengths of the oil-quenched rods predicted by Eq. (3) are represented as the solid curve in Fig. 1; a cutoff corresponding to the strength of the nonindented surface occurs at low loads, the indentation cracks in this region becoming less severe than preexisting flaws.[14]

The results in Table I show differences in strength response in the indented and nonindented states relative to the magnitude of the residual stress. From Eq. (2) the strengthening effect of the heat treatment can be expressed as

$$\sigma_T - \sigma_O = (\pi^{1/2} K_c/2)(c_T^{-1/2} - c_O^{-1/2}) + \sigma_R \tag{4}$$

where the subscripts T and O refer to treated and as-received surfaces. At $P = 100$ N, where the dominant flaws are indentation cracks introduced after heat treatment, the observed strengthening $\sigma_T - \sigma_O$ is given, within experimental error, by σ_R; the second term in Eq. (4) is negligibly small. However, at $P = 0$, $\sigma_T - \sigma_O$ is considerably greater than σ_R for all of the treatments. In this case the dominant flaw is provided by preexisting damage and is therefore susceptible to modification during heat treatment[19]: the data are consistent with a subsidiary strengthening mechanism involving flaw healing (from $c_O \approx 19$ μm in the as-received surfaces to $c_T \approx 11$ μm in the treated surfaces), or perhaps relief of localized residual stresses about the damage sites.*

The strength data in Table I and Fig. 1 have certain implications concerning the prospective structural applications of thermally treated ceramics. In view of the large strength losses resulting from severe contact events, strength tests on laboratory-prepared surfaces cannot always be used as a sole guide to in-service performance. The indentation fracture approach provides a sounder basis for strength design. It may be noted in Fig. 1 that, although treated surfaces tend to suffer greater absolute strength loss than as-received surfaces, the strength ratio of treated to as-received surfaces actually improves with increasing load. Therefore, the quenched surfaces are better suited to resist degradation in more hostile environments. These conclusions, although relating specifically to static tests using a Vickers indenter, are expected to carry over to more general contact situations involving, for example, impacts with irregular particles.[12,13]

†This value of K_c is considerably lower than values measured by other independent techniques on similar alumina (Ref. 16), presumably because of the omission from the present fracture mechanics of residual-stress effects about the elastic/plastic indentation (Ref. 14). This is readily verified by annealing indented specimens, which removes the discrepancy (Ref. 17). The value of K_c from indentation/strength tests may therefore be considered as an "effective K_c," but is nevertheless adopted here because the subsequent indentation/strength experiments in treated rods are conducted under identical conditions.

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