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Flexural strength of sapphire: Weibull statistical analysis of stressed area, surface coating, and polishing procedure effects

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The results of fracture testing are usually reported in terms of a measured strength, $\sigma_M = \overline{\sigma_i} \pm \Delta\overline{\sigma_i}$, where $\overline{\sigma_i}$ is the average of the recorded peak stresses at failure, and $\Delta\overline{\sigma_i}$ represents the standard deviation. This "strength" does not provide an objective measure of the intrisic strength since σ_M depends on the test method and the size of the volume or the surface subjected to tensile stresses. We first clarify issues relating to Weibull's theory of brittle fracture and then make use of the theory to assess the results of equibiaxial flexure testing that was carried out on a variety of sapphire specimens, at three mechanical test facilities. Specifically, we describe the failure probability distribution in terms of a characteristic strength σ_C—i.e., the effective strength of a uniformly stressed 1 cm^2 area—which allows us to predict the average stress at failure of a uniformly loaded "window" if the Weibull modulus m is available. A Weibull statistical analysis of biaxial-flexure strength data thus amounts to obtaining the parameters σ_C and m, which is best done by directly fitting estimated cumulative failure probabilities to the appropriate expression derived from Weibull's theory. We demonstrate that: (a) measurements performed on sapphire test specimens originating from two suppliers confirm the applicability of the area scaling law; for mechanically polished *c*- and *r*-plane sapphire, we obtain $\sigma_C \simeq 975$ MPa, $m = 3.40$ and $\sigma_C \simeq 550$ MPa, $m = 4.10$, respectively. (b) Strongly adhering compressive coatings can augment the characteristic strength by as much as 60%, in accord with predictions based on fracture-mechanics considerations, but degrade the Weibull modulus, which mitigates the benefit of this approach. And (c) Measurements performed at 600 °C on chemomechanically polished *c*-plane test specimens indicate that proper procedures may enhance the characteristic strength by as much as 150%, with no apparent degradation of the Weibull modulus. © *2004 American Institute of Physics*. [DOI: 10.1063/1.1782272]

I. INTRODUCTION

Sapphire (α-Al_2O_3) exhibits outstanding optical and mechanical properties that make this material highly attractive for manufacturing infrared-transmitting windows and domes capable of operating in adverse environments.[1] Sapphire is a well characterized material,[2] but requirements arising in the context of highly demanding defense programs have focused attention on the need to thoroughly investigate key features relating to the fracture strength, including ways of augmenting the strength. We intend to first clarify issues regarding the applicability of Weibull's theory of brittle fracture,[3] and then make use of the theory to assess the results of equibiaxial flexure testing for the specific purpose of investigating how the area subjected to tensile stresses affects the measured strength, and how the strength can be enhanced by means of compressive surface coatings or improved polishing procedures.

Published sapphire strength data are both abundant and confusing, primarily because the results of fracture testing are usually reported in terms of a measured strength,

$$\sigma_M = \overline{\sigma_i} + \Delta\overline{\sigma_i}, \tag{1}$$

where $\overline{\sigma_i}$ designates the arithmetic average of the recorded stresses at failure, and $\Delta\overline{\sigma_i}$ represents the standard deviation. This "strength" does not provide an objective measure of the intrinsic strength since σ_M depends on the test method as well as the size of the volume or the surface subjected to tensile stresses.[4] Also, it is common practice to interpret the strength data on the basis of a semiempirical expression derived from Weibull's statistical theory of fracture, i.e.,

$$P(\sigma) = 1 - \exp[-(\sigma/\sigma_N)^m], \tag{2}$$

which describes the cumulative failure probability P as a function of the applied tensile stress σ. This expression involves two parameters—the nominal strength σ_N and the Weibull modulus m—and both can be extracted from a set of experimental data by fitting the estimated failure probability to Eq. (2). The simplest method for performing this task consists of obtaining a least-squares fit to a linearized version of Eq. (2),

$$\ln[-\ln(1-P)] = -m\ln(\sigma_N) + m\ln(\sigma), \tag{3}$$

which yields the Weibull modulus from the slope and the nominal strength from the $\ln[-\ln(1-P)]=0$ intercept. Evidently, the strength σ_N does not take into account the potential impact of the test method (loading geometry and specimen size) and does not relate to the intrisic strength in an obvious manner.

At room temperature, test specimens made of sapphire fail as a result of tensile stresses acting on surface flaws; applicable models, therefore, may disregard volume effects,

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which greatly simplifies the task of describing the fracture strength. In Sec. II we address the problem of formulating Weibull's theory in a manner that accounts not only for the scatter of test data but also for the area effect in a concentric-ring test configuration. In this context, and upon defining a characteristic strength for biaxial loadings, we derive expressions for the failure probability distribution and the area scaling law, which allows us to predict the actual effective strength of a sapphire "window" if the Weibull modulus is available.

In Secs. III–V, this model will be taken advantage of for the purpose of interpreting the results of "ring-on-ring" flexural testing that was carried out over a period of 20 years (1979–1999) at mechanical test facilities operated by the Southern Research Institute (SoRI), the University of Massachusetts (UMass), and the University of Dayton Research Institute (UDRI). Testing was conducted on specimens supplied by Union Carbide (now Saint-Gobain Crystals) and by Crystal Systems Inc., which will give us an opportunity to compare the performance of mechanically polished sapphire crystals obtained from two different vendors. The test data we rely on are documented in Refs. 5–7; note that the samples included in each of the lots we examined always fractured as a result of tensile stresses induced on the lower inner surface of the ring-on-ring fixture, thus implying that the data should be amenable to a Weibull statistical analysis as formulated in Sec. II. Specifically, the results of fracture testing performed on mechanically polished *c*- and *r*-plane specimens are examined in Sec. III and shown to be compatible with a two-parameter Weibull model in the sense that they obey the area scaling law surprisingly well. Experiments performed on compressively coated sapphire specimens are evaluated in Sec. IV, where we demonstrate that any strength improvement must be attributed to flaw internalization, in accord with predictions based on elementary fracture-mechanics considerations. In Sec. V we address the issue of sapphire strength enhancement by means of improved polishing procedures, in the light of biaxial flexure testing performed at 600 °C at the UDRI test facility.

The conclusions are stated in Sec. VI. In essence, the work we are reporting on here shows how to proceed in order to obtain reliable fracture statistical parameter values and illustrates how the Weibull model can be applied to predict the failure probability of sapphire windows subjected to biaxial tensile stresses. It emphazises how the effective—or measured—strength depends on the stessed area and demonstrates that the totality of data generated at SoRI, UMass, and UDRI leads to a coherent picture of key features relating to the strength of sapphire.

II. WEIBULL MODEL

The results of fracture-strength measurements performed on brittle materials are best modeled in the framework of Weibull's theory,[3] which postulates that the scatter in recorded strength data is controlled by the presence of randomly distributed defects. According to the two-parameter model, that is, if fracture can occur at any stress level, and on assuming that fracture originates at the surface, the cumulative failure probability of a test specimen subjected to a stress distribution $\sigma(x,y)$ on the surface under tension can be expressed as follows:

$$P = 1 - \exp\left\{ - \int_{\text{surf}} \left[\frac{\sigma(x,y)}{\chi} \right]^m dxdy \right\}, \tag{4}$$

where both the scaling parameter χ and the Weibull modulus m are material properties and, therefore, independent of the testing procedure or the specimen size. In a concentric-ring experimental configuration, the equibiaxial stresses (radial and azimuthal) acting on the tensile surface in the region delineated by the loading ring are essentially uniform, which implies that the integral expression reduces to

$$\int_{\text{surf}} \left[\frac{\sigma(x,y)}{\chi} \right]^m dxdy = S\left(\frac{\sigma}{\chi}\right), \tag{5}$$

where S represents the area subjected to the stress σ; the failure probability expression then reduces to

$$P(\sigma) = 1 - \exp\left[- S\left(\frac{\sigma}{\chi}\right)^m \right], \tag{6}$$

which enables us to predict how the stressed area impacts the distribution, if the two statistical parameters (χ and m) are available.

At this point, we may attempt to relate the scaling parameter to the measured strength as defined earlier, that is, the average stress at failure in a given test environment. For that purpose, and bearing in mind that $dP(\sigma)/d\sigma$ represents the failure probability density, or probability of a failure occurring at the stress level σ, we introduce the concept of an effective strength,

$$\overline{\sigma} = \int_0^\infty \sigma \left[\frac{dP(\sigma)}{d\sigma} \right] d\sigma, \tag{7}$$

which, in principle, matches the mean fracture stress $\overline{\sigma_i}$; with $P(\sigma)$ as in Eq. (6) the integration is straightforward and yields

$$\overline{\sigma} = \frac{\chi}{S^{1/m}} \Gamma\left(1 + \frac{1}{m}\right), \tag{8}$$

where $\Gamma(z)$ designates the gamma factorial function.[8] Equation (8) enables us to substitute the strength $\overline{\sigma}$ for the parameter χ and, thus, to express the cumulative failure probability as follows:

$$P(\sigma) = 1 - \exp\left\{ - \left[\Gamma\left(1 + \frac{1}{m}\right) \right]^m \left(\frac{\sigma}{\overline{\sigma}}\right)^m \right\}. \tag{9}$$

Now suppose that the surface under stress has an area s of one (1) unit. In that case the strength $\overline{\sigma}$ defines the characteristic strength,

$$\sigma_C = \frac{\chi}{s^{1/m}} \Gamma\left(1 + \frac{1}{m}\right), \tag{10}$$

which relates to the effective strength through

$$\overline{\sigma} = \frac{\sigma_C}{(S/s)^{1/m}}, \tag{11}$$

thus providing the area scaling law that applies to the effective—or measured—strength. Furthermore, on substituting the characteristic strength for the effective strength, the failure probability can be expressed in an explicit manner,

$$P(\sigma) = 1 - \exp\left\{ -\frac{S}{s}\left[\Gamma\left(1+\frac{1}{m}\right)\right]^m \left(\frac{\sigma}{\sigma_C}\right)^m \right\}, \tag{12}$$

keeping in mind that this expression assumes a uniform stress distribution.

A proper Weibull analysis of biaxial flexure strength data thus amounts to obtaining the two statistical parameters—σ_C and m—that control the failure of test specimens originating from the same lot and having the same surface finish. The procedure involves three steps as follows:

(1) Rank by ascending order $(i=1,2,\ldots,n)$ the recorded stresses at fracture and assign cumulative probabilities of failure according to $P_i=(i-0.5)/n$, where i is the rank, and n is the number of broken samples.[4]

(2) Fit the $\ln[-\ln(1-P_i)]$ versus $\ln(\sigma_i)$ data points to a straight line, which provides not only a visual assessment of the validity of the two-parameter model but also a direct estimate of the Weibull modulus and the nominal strength [see Eq. (3)]. This greatly facilitates implementing the next step, but keep in mind that such estimates can deviate significantly from the true values considering that a linear least-squares analysis places inordinate weight on the low-strength data points.

(3) Fit the P_i versus σ_i data points to Eq. (12), which is best done by means of a bivariate, nonlinear regression based on the Marquardt-Levenberg (M-L) algorithm,[9] on choosing initial parameter values that reflect the results of step 2. If successful, the procedure yields both the Weibull modulus, which defines the scatter in fracture stresses, and the characteristic strength, which defines the intrinsic strength of the material; this procedure can be "programmed" to yield relevant uncertainties at any prescribed level of confidence.

To illustrate, I propose to do an analysis of the fracture-test results for planar disks of Crystal Systems sapphire cut in the a-plane (90 deg orientation) that are recorded in Ref. 10. The tests were conducted at the UDRI large-ring fixture (inner-ring diameter: 1.59 cm) on 14 samples, 38.1 mm in diameter and 2 mm thick, ground and polished to a flatness of a few 0.6328 μm waves and an rms roughness of better than 10 Å. As seen in Fig. 1(a), the "Weibull plot" confirms that the two-parameter model applies—at the 95% confidence level there is a single outlier—and yields 567 ± 10 MPa for the nominal strength (63.2% failure probability) and 6.32 ± 0.54 for the Weibull modulus. At the 95% confidence level, the M-L fit displayed in Fig. 1(b) then points to $\sigma_C=595\pm13$ MPa and $m=5.71\pm0.79$, which translates into an effective strength of about 528 MPa for a uniformly stressed area of 1.98 cm^2 [see Eq. (11)], in agreement with the measured mean stress at failure ($\overline{\sigma_i}=528$ MPa).

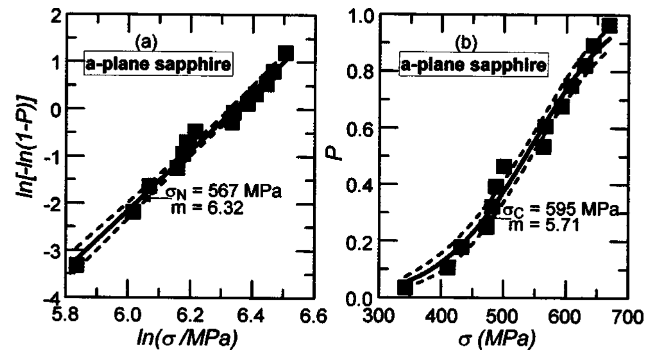

FIG. 1. Analysis of flexural strength data (Ref. 10) for a-plane sapphire; the broken lines delineate the 95% confidence bands. (a) Weibull plot, the nominal strength σ_N referring to a stressed area of 1.98 cm^2, and (b) failure probability as a function of the applied stress. The fit yields the statistical parameter values recorded in Sec. II.

III. AREA EFFECTS

The strength data we examine in this section concern mechanically polished sapphire obtained from two suppliers: Union Carbide (Czochralsky gown) and Crystal Systems (Schmid-Viechnicki grown). In 1979, eight disks of c-plane (0 deg) sapphire measuring 50.8 mm in diameter and 2.54 mm in thickness, grown and polished at Union Carbide, were subjected to biaxial fracture testing at SoRI (inner-ring diameter: 1.91 cm);[5] the measured strength was $\sigma_M=703\pm242$ MPa. More recently (1996), eight disks of c-plane sapphire, 25.4 mm in diameter and 2.54 mm in thickness, made of HEMlite-grade material[1] and delivered with a 60/40 scratch/dig polish, were fracture tested in the small-ring fixture (inner-ring diameter: 1.06 cm) at UDRI;[6] the measured strength was $\sigma_M=1061\pm372$ MPa. The Weibull plots displayed in Fig. 2(a) indicate that the two-parameter model should be applicable. The cumulative failure probabilities are plotted in Fig. 2(b). On performing bivariate regressions based on a theoretical failure-probability expression as in Eq. (12), with $S/s=1.85$ and $S/s=0.876$ for SoRI and UDRI experimentation, respectively, we find that $\sigma_C=946\pm166$ MPa, $m=3.40\pm1.53$ and $\sigma_C=1001\pm78$ MPa, $m=3.41\pm1.61$ are appropriate numbers for the statistical parameters, which demonstrates that both the characteristic

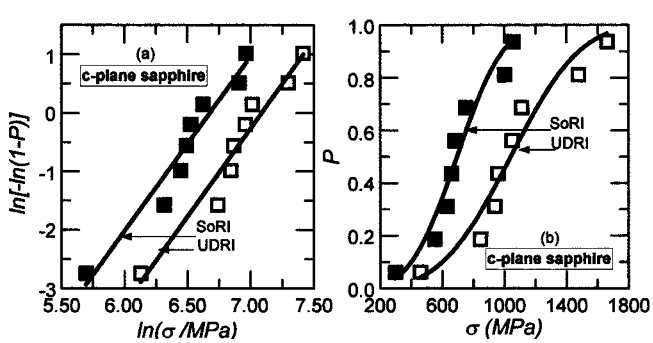

FIG. 2. Analysis of flexural strength data (Refs. 5 and 6) recorded at the Southern Research Institute and the University of Dayton Research Institute for c-plane sapphire. (a) Weibull plots, and (b) failure probabilities as a function of the applied stress. Applicable statistical parameter values are listed in Table I.

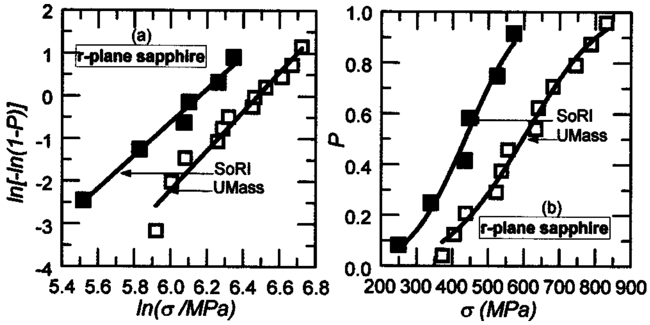

FIG. 3. Analysis of flexural strength data (Refs. 5 and 6) recorded at the Southern Research Institute and the University of Massachusetts for *r*-plane sapphire. (a) Weibull plots, and (b) failure probabilities as a function of the applied stress. Applicable statistical parameter values are listed in Table I.

strength and the Weibull modulus are essentially identical for the two lots. Furthermore, we note that in terms of effective and measured strengths we have

$$\frac{\overline{\sigma}(\text{UDRI})}{\overline{\sigma}(\text{SoRi})} \simeq \frac{1040}{695} \simeq \frac{\overline{\sigma_i}(\text{UDRI})}{\overline{\sigma_i}(\text{SoRI})}, \tag{13}$$

in accord with the area scaling law [see Eq. (11)].

Work performed at SoRI (Ref. 5) also included six disks of *r*-plane (60 deg) sapphire grown and polished at Union Carbide and having the same dimensions as the *c*-plane specimens; the measured strength was $\sigma_M = 427 \pm 118$ MPa. In addition, we have data[6] for 12 disks of *r*-plane sapphire, 25 mm in diameter and 1 mm thick, that were cut from a boule grown by the heat-exchange method at Crystal Systems, polished to a scratch/dig specification of 80/50, and tested for strength at the UMass concentric-ring facility (inner-ring diameter: 0.953 cm); the measured strength was $\sigma_M = 595 \pm 150$ MPa. Figure 3(a) shows that the failure probability obeys the two-parameter Weibull model, thus providing suitable initial values for performing the nonlinear regressions displayed in Fig. 3(b). At the 95% confidence level, these regressions yield the following values: $\sigma_C = 558 \pm 54$ MPa, $m = 4.09 \pm 1.47$ for the lot tested at SoRI, and $\sigma_C = 545 \pm 14$ MPa, $m = 4.10 \pm 0.52$ for the lot tested at UMass. Characteristic strengths and Weibull moduli are in good agreement, thus demonstrating that the area scaling law applies remarkably well, even for material created by different techniques; in effect, we have

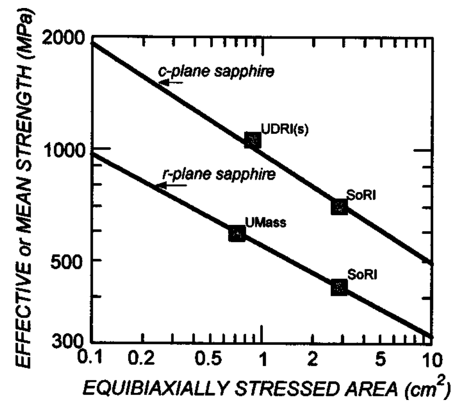

FIG. 4. Effective strength of mechanically polished *c*- and *r*-plane sapphire as a function of the equibiaxially stressed area. The straight lines are as obtained from Eq. (11) with statistical parameters (most probable values) as listed in Table I. The data points are mean strengths as measured at the Southern Research Institute, the University of Massachusetts, and the University of Dayton Research Institute small-ring fixture.

$$\frac{\overline{\sigma}(\text{UMass})}{\overline{\sigma}(\text{SoRI})} \simeq \frac{592}{432} \simeq \frac{\overline{\sigma_i}(\text{UMass})}{\overline{\sigma_i}(\text{SoRI})} \tag{14}$$

precisely as expected. Effective strengths of 432 and 592 MPa agree with $\overline{\sigma_i} = 427$ MPa as measured at SoRI, and $\overline{\sigma_i} = 595$ MPa as measured at UMass, which again confirms that the picture emerging from this analysis is not only coherent but clarifies issues arising in the context of assessing the strength of sapphire at room temperature.

Table I summarizes the results of our analysis of equibiaxial flexure testing that was performed on *c*- and *r*-plane sapphire disks, manufactured and polished by two independent vendors. The measured strengths are believed to be representative of commercially available products, and the statistical parameters suggest that their “pedigree” may not play as important a role as previously surmised. Specifically, we note that the characteristic strength of *c*-plane sapphire, at room temperature, is of the order of 975 MPa (140 kpsi) and the Weibull modulus is close to 3.4. The strength of *r*-plane sapphire is seen to be substantially lower ($\sigma_C \simeq 550$ MPa $\simeq 80$ kpsi), and so is the scatter ($m \simeq 4.1$). Based on these numbers, the area scaling law as formulated earlier, i.e.,

$$\overline{\sigma} = \frac{\sigma_C}{(S/s)^{1/m}} \simeq \overline{\sigma_i}, \tag{15}$$

leads to the situation depicted in Fig. 4, which illustrates how the effective strength—or average stress at failure for a set of

TABLE I. Stressed area dependence of the measured strength and relevant Weibull statistical parameters of *c*- and *r*-plane sapphire at room temperature.

Orientation	Vendor	Stressed area (cm^2)	Measured strength (MPa)	Characteristic strength (MPa)	Weibull modulus
c-plane	Union Carbide	2.85	703 ± 242	946 ± 166	3.40 ± 1.53
	Crystal Systems	0.876	1061 ± 372	1001 ± 78	3.41 ± 1.61
r-plane	Union Carbide	2.85	427 ± 118	558 ± 54	4.09 ± 1.47
	Crystal Systems	0.713	595 ± 150	545 ± 14	4.10 ± 0.52

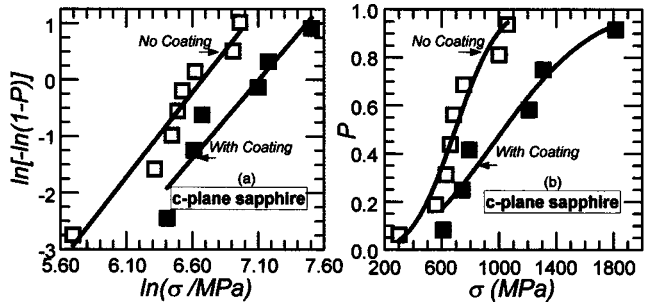

FIG. 5. Analysis of flexural strength data (Ref. 5) for *c*-plane sapphire, with and without surface coating. (a) Weibull plots, and (b) failure probabilities as a function of the applied stress. Applicable statistical parameter values are listed in Table II.

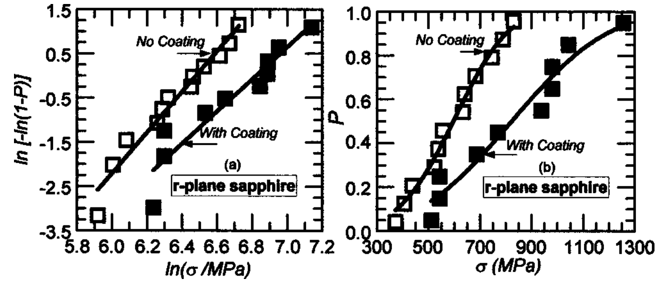

FIG. 6. Analysis of flexural strength data (Ref. 6) for *r*-plane sapphire, with and without surface coating. (a) Weibull plots, and (b) failure probabilities as a function of the applied stress. Applicable statistical parameter values are listed in Table II.

identical specimens tested under identical conditions—depends on the stressed area in a concentric-ring experimental configuration. The data points represent the measured mean strengths σ_i as recorded in the course of fracture testing that was conducted over a period of 18 years at three mechanical test facilities.

IV. COATING EFFECTS

In this section we address the issue of sapphire strength improvement by means of compressively stressed surface coatings[11] and demonstrate that the enhancements we observe are in general agreement with predictions based on simple fracture-mechanics considerations as presented in the Appendix. The surface treatments that were carried out at Ceramic Finishing Company on Union Carbide made *c*-plane sapphire disks consisted in creating either an alumina-silicate based glaze fired at 1400 °C or a mullite layer obtained through vacuum deposition of SiO and subsequent reheating at 1100 °C. Three specimens of glaze-coated and three specimens of mullite-coated *c*-plane sapphire were fracture tested at SoRI with the coated surface subjected to tensile stresses; since the nature of the coating should make no difference in terms of achievable strength enhancement, we assume that the measured stresses at fracture belong to a single stochastic ensemble with $\sigma_M = 1079 \pm 453$ MPa based on the σ_i's listed in Ref. 5. The two Weibull plots displayed in Fig. 5(a) demonstrate that the coatings do enhance the strength. The nonlinear fitting procedure [see Fig. 5(b)] yields the following numbers for the statistical parameters of coated *c*-plane sapphire: $\sigma_C = 1636 \pm 462$ MPa and $m = 2.43 \pm 1.17$, which imply that the coating gives rise to a significant increase in characteristic strength but at the cost of more scatter in measured strengths. Consequently, it is not obvious how to properly assess the enhancement in terms of a theoretical fracture strength as considered in the Appendix; if, however, we rely on the ratio of the two characteristic strengths or the two measured strengths, with and without coating, we have

$$\frac{\sigma_f^*}{\sigma_f} \simeq \frac{1636}{946} \simeq \frac{1079}{703} \simeq 1.63, \tag{16}$$

which would indicate that the enhancement was of the order of 60% and, therefore, that the coatings performed as expected.

More recently, ten specimens of coated Crystal Systems *r*-plane sapphire, 25 mm in diameter and 1 mm thick as in Sec. III, were fracture tested at the UMass ring-on-ring facility for the specific purpose of assessing the strength improvement achieved through coatings prepared at Materials Systems, Inc. The coatings consisted of an 0.3 μm thick SiO_2 layer directly deposited on the polished sapphire substrate and subjected to heat-treatment at 1200 °C, which gave rise to a high level of compressive stress. The recorded stresses at fracture are as listed in Ref. 6 $(\sigma_M = 825 \pm 253$ MPa$)$ and displayed in Fig. 6 as filled data points. The Weibull plot on the left suggests that the data are of acceptable "quality;" the nonlineat fit on the right yields the following parameter values: $\sigma_C = 744 \pm 48$ MPa, $m = 3.25 \pm 0.89$. Evidently, and in accord with the SoRI data for *c*-plane material, the coating appears to be beneficial with regard to the characteristic strength but detrimental in terms of the Weibull modulus. Implications from the point of view of the theoretical strength are difficult to quantify, but if we proceed as earlier and write

$$\frac{\sigma_f^*}{\sigma_f} \simeq \frac{744}{545} \simeq \frac{825}{595} \simeq 1.38, \tag{17}$$

the ratios are indicative of a strength improvement of about 40%, which is less than expected for an ideal coating (see the Appendix).

Table II summarizes the results of this investigation of the effects of compressive coatings on the flexural strength of *c*- and *r*-plane sapphire. The availability of the two statistical parameters, σ_C and m, can be taken advantage of to predict the failure probability distribution of a uniformly stressed window made of *c*- or *r*-plane sapphire having a similar surface finish as the test specimens. Figure 7 displays the cumulative failure probability of coated and uncoated windows, on assuming a stressed area of 1 cm^2—in other words, as obtained from the expression

$$P(\sigma) = 1 - \exp\left\{ - \left[\Gamma\left(1 + \frac{1}{m} \right) \right]^m \left(\frac{\sigma}{\sigma_C} \right)^m \right\} \tag{18}$$

with statistical parameters as listed in Table II (most probable values). Since design methodologies for windows and domes made of brittle materials and subjected to tensile loads require reliable information on the failure probability as a function of the applied stress, this procedure provides the

TABLE II. Surface coating effects on the measured strength and relevant Weibull statistical parameters of *c*- and *r*-plane sapphire at room temperature.

Orientation	Coating	Vendor	Measured strength (MPa)	Characteristic strength (MPa)	Weibull modulus
c-plane	No		703±242	946±166	3.40±1.53
	Yes	Ceramic Finishing	1079±453	1636±462	2.43±1.17
r-plane	No		595±150	545±14	4.10±0.52
	Yes	Materials Systems	825±253	744±48	3.25±0.89

best means of ascertaining the failure probability at the lower tail-end of the distribution. In this regard, it is seen that coatings provide little useful strength improvement, primarily because of the degraded Weibull modulus. At the 5% failure probability level, Fig. 7 indicates that, in the *r*-plane orientation, mechanically polished sapphire may tolerate equibiaxial tensile loads of about 300 MPa, whereas coated *c*-plane sapphire windows should be able to function with minimal risk of catastrophic failure at stress levels of up to 500 MPa, as long as the stressed area does not exceed 1 cm^2.

V. POLISHING EFFECTS

In 1999, a comprehensive series of experiments was carried out by Crystal Systems for the purpose of assessing the effects of the polishing procedure on the high-temperature strength of *c*-plane sapphire.[7] In-house made disks, which measured 38 mm in diameter and 1 mm in thickness, were sent to four vendors to be polished to a scratch/dig specification of 60/40 using various polishing techniques as described in Ref. 7. After annealing at 1200 °C, each of the sets of specimens that turned out to be suitable for detailed analysis was subjected to a proprietary treatment at Aspen Systems,[12] which is believed to enhance the high-temperature compressive strength of sapphire. Biaxial flexure strength testing was carried out at a temperature of 600 °C at the UDRI large-ring fixture (loading-ring diameter: 1.59 cm); Grafoil™ sheets were used to minimize the compressive contact stress[13] and, thus, to suppress potential rhombohedral twinning caused by compressive loading at the ring contact. Subsequent fractography confirmed that the specimens failed in tension, away from the loading ring. In this connection, we reemphazise that the Weibull model as presented in Sec. II is predicated on tensile-stress initiated fracture at strength-limiting surface flaws.

Figure 8(a) displays a Weibull plot derived from the recorded 600 °C fracture-test data for mechanically polished ("Elcan" polish) *c*-plane sapphire. It is seen that the eight data points obey Eq. (3) at the 95% confidence level, thus demonstrating that the data are amenable to a two-parameter Weibull analysis. The analysis [see Fig. 8(b)] yields $\sigma_C = 708 \pm 13$ MPa and $m = 5.77 \pm 0.72$, which confirms that the flexural strength of *c*-plane sapphire decreases at elevated temperatures, but so is the data scatter. Polishing performed by the three other vendors (General Optics, Insaco, and Meller) involves a chemomechanical process as a final step to reduce the surface roughness. The fracture-test data were found to be suitable for detailed evaluation, and Fig. 9 illustrates the results of the Weibull analysis for material processed by Meller Optics, Inc. Evidently, the chemomechanical final polish substantially enhances the strength, and must be attributed to the improved roughness of the surface subjected to tensile stresses.

Table III lists the results of our analysis of polishing effects on the biaxial flexure strength of "Aspen"-treated *c*-plane sapphire. The best strength improvement ($\sigma_C = 1739 \pm 25$ MPa versus 708 ± 13 MPa in the absence of chemomechanical processing) was achieved with a "General Optics" polish and did not entail any degradation of the

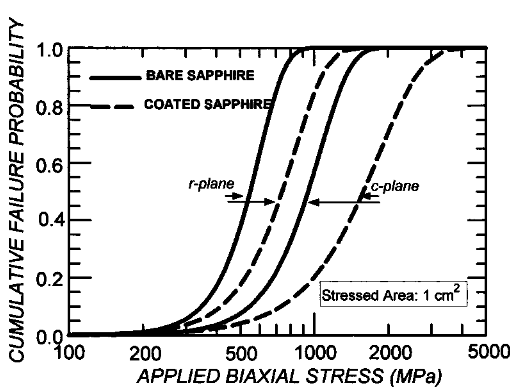

FIG. 7. Cumulative failure probabilities of mechanically polished bare and coated *c*- and *r*-plane sapphire. The distributions reflect Eq. (18)—i.e., assume a uniformly stressed area of 1 cm^2— with statistical parameters (most probable values) as listed in Table II. Note that, in the low-failure probability regime, the strength enhancement appears to be minimal.

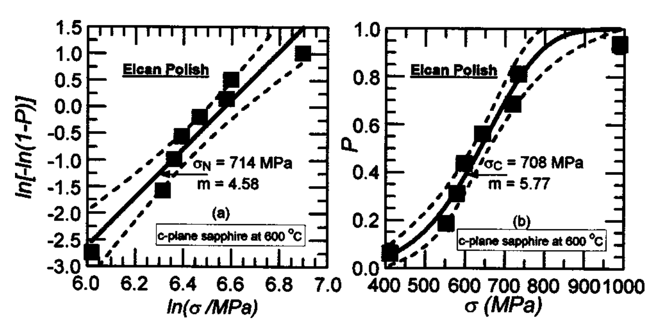

FIG. 8. Analysis of flexural strength data (Ref. 7) recorded at 600 °C for mechanically polished *c*-plane sapphire; the broken lines delineate the 95% confidence bands. (a) Weibull plot, the nominal strength σ_N referring to a stressed area of 1.98 cm^2, and (b) failure probability as a function of the applied stress. The fit yields the statistical parameter values listed in Table III.

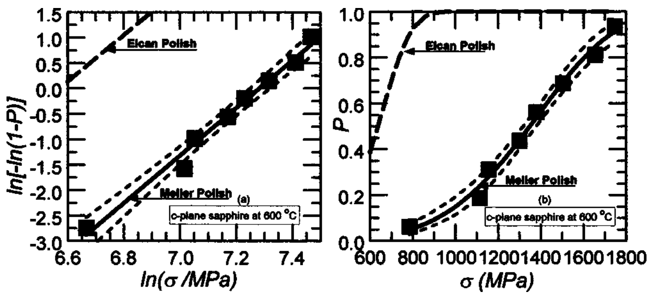

FIG. 9. Analysis of flexural strength data (Ref. 7) recorded at 600 °C for chemomechanically finished *c*-plane sapphire; the broken lines delineate the 95% confidence bands. (a) Weibull plot, which demonstrates the effectiveness of the "Meller" polish, and (b) failure probability as a function of the applied stress. The fit yields the statistical parameter values listed in Table III.

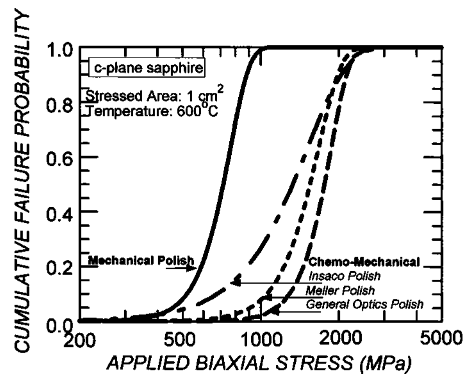

FIG. 10. Cumulative failure probabilities of mechanically and chemomechanically polished *c*-plane sapphire, at 600 °C. The distributions reflect Eq. (18)—i.e., assume a uniformly stressed area of 1 cm^2— with statistical parameters (most probable values) as listed in Table III. Note that, in the low-failure probability regime, chemomechanical polishing can result in strength enhancements of at least a factor of 2.

Weibull modulus; this improvement reflects the measured rms roughness (⩽1 nm versus 10 nm) and appears to be indicative of the surface flaw depth, in the sense that a strength ratio of about 2.4 correlates with the square root of the rms roughnesses (see the Appendix). The strength improvement of "Meller" and "Insaco" polished specimens does not correlate as well with the roughness (see Table III), but it is noteworthy that, according to Schmid *et al.*,[7] the "Meller" specimens exhibit lower dislocation densities, which may explain the discrepancy. As was done in Sec. IV, the statistical parameter values can be exploited to predict the failure probability distribution at 600 °C of windows made of *c*-plane sapphire having surface finishes as specified in Table III. If subjected to uniform biaxial tensile stresses over an area of 1 cm^2, and on assuming that compressive failure can be eliminated, the cumulative failure probability of such windows derives immediately from Eq. (18), which leads to the distributions displayed in Fig. 10. It is seen that both the "Meller" and the "General Optics" polish can improve the high-temperature strength of "Aspen"-treated sapphire by at least a factor of 2, even in the low-failure-probability regime, i.e., at failure probability levels of less than 5%.

VI. CONCLUSION

Since ring-on-ring testing is the preferred method for measuring the strength of optical ceramics,[2] the characteristic strength—as specified in this paper—represents a true material property; together with the modulus m, this strength σ_C enables us to evaluate the cumulative failure probability as a function of the applied biaxial stress, given the surface area under uniform stress. A Weibull statistical analysis of equibiaxial flexure-strength data thus amounts to obtaining the parameters σ_C and m, which is best done by fitting estimated P_i versus σ_i data to the $P(\sigma)$ expression as formulated in Eq. (12). This procedure avoids distorting the distribution through logarithmic linearization and can be implemented by performing a bivariate regression that yields most probable values and applicable uncertainties at any specified confidence level. In this light, we conclude the following.

(i) The wide spread of published sapphire strength data reflects the poor modulus of this material and must be attributed not only to crystallographic or morphological features but also to the impact of the area effect on fracture strength data.

(ii) Strongly adhering compressively stressed coatings can augment the characteristic strength of sapphire windows by as much as 60%, in accord with theoretical speculations; the broader dispersion of the strength data, however, mitigates the potential benefit of this approach.

(iii) A proper evaluation of high-temperature flexural strength data collected on "Aspen"-treated *c*-plane sapphire demonstrates that chemomechanically finished surfaces can improve the characteristic strength of mechanically polished specimens by as much as 150%, with no apparent degradation of the Weibull modulus.

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TABLE III. Polishing effects on the measured strength and relevant Weibull statistical parameters of *c*-plane sapphire at 600 °C.

Polish	Vendor	Roughness (rms nm)	Measured strength (MPa)	Characteristic strength (MPa)	Weibull modulus
Mechanical	Elcan	~10	653±169	708±13	5.77±0.72
Chemical	General Optics	⩽1	1534±378	1739±25	6.07±0.66
Chemical	Meller	⩾2.5	1330±313	1534±18	4.86±0.31
Chemical	Insaco	⩾1	1082±425	1350±79	2.80±0.42

APPENDIX

In ceramic materials such as sapphire, stress-induced fracture originates at preexisting surface flaws originating from machining damage. In a mode-I loading environment, the theoretical fracture stress obeys the relation[4]

$$\sigma_f = \frac{K_{Ic}}{1.12\sqrt{\pi}\sqrt{c}}, \tag{19}$$

where K_{Ic} denotes the intrinsic fracture thoughness of the material, and c measures the depth of the largest surface flaws. Now consider the case of a surface with a compressive coating able to withstand the applied tensile load. From a fracture-mechanics point of view,[14] this implies that (a) the original surface flaws are now embedded cracks, and (b) these cracks are being acted upon by the tensile stress σ_s generated at the coating/surface interface. Consequently, the relevant fracture stress should be

$$\sigma_f^* = \frac{K_{Ic}}{\sqrt{\pi}\sqrt{c/2}} - \sigma_s, \tag{20}$$

which leads to the conclusion that the strength enhancement reflects the ratio

$$\sigma_f^*/\sigma_f \simeq 1.12\sqrt{2} = 1.58 \tag{21}$$

since the substrate stress at the interface can be ignored for thick substrates (see the Appendix in Ref. 6). In other words, well adhering coatings resulting in complete flaw internalization may enhance the theoretical strength by as much as 60%, in apparent agreement with previously reported strength measurements performed on Y_2O_3-coated ZnS wafers.[15]

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