Weibull Statistical Analysis of Sapphire Strength Improvement through Chemomechanical Polishing

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Abstract: Significant enhancements of the flexural strength of *a*- and *c*- plane sapphire by means of "super polishing" was first reported by McHargue and Snyder [*Proc. SPIE* **2013**, 135 (1993)]. The improvement was attributed to the removal of residual mechanical polishing damage. More recently, a comprehensive series of experiments was carried out by Crystal Systems for the specific purpose of assessing the effects of various polishing procedures on the high-temperature strength of *c*-plane sapphire. Subsequent testing at room temperature confirmed that chemomechanical polishing improves both the effective strength and the strength distribution. In this contribution we take advantage of the methodology previously used by Klein et al. [*Opt. Eng.* **41**, 3151 (2002)] to perform a correct Weibull statistical analysis of biaxial flexure-strength data generated in the course of Crystal Systems' investigations. We demonstrate that chemomechanical polishing procedures can improve the high-temperature characteristic strength of *c*-plane sapphire by 150 % and the room-temperature Weibull modulus by 100 %.

1. INTRODUCTION

Sapphire ($\alpha-Al_2O_3$) exhibits outstanding optical and mechanical properties that make this material highly attractive for manufacturing infra-red (IR) transmitting windows and domes capable of operating in adverse environments.[1] Sapphire is a well characterized material,[2] but requirements arising in connection with highly demanding defense programs have focused attention on the need to investigate ways of augmenting the strength, especially at elevated temperatures. Since the surface quality of ceramic materials has a major effect on the mechanical strength, we may expect that the strength of sapphire can be enhanced by means of improved polishing procedures. In this regard, significant results were first reported by McHargue and Snyder,[3] who demonstrated that a "super polishing (SP)" technique developed by Laser Power Optics improves the strength of both *a*- and *c*-plane sapphire test specimens. Specifically, they reported that "much" improvement was found in terms of

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failure in the low-cumulative-failure regime (low or moderate tensile strresses) and "some" improvement at higher levels of applied stress. This improvement in fracture strength was attributed to the removal of residual mechanical polishing damage as a result of the SP treatment.

In 1999, a comprehensive series of experiments was carried out by Crystal Systems for the specific purpose of assessing the effects of various polishing procedures on the high-temperature flexural strength of *c*-plane sapphire; these experiments are documented in Ref. 4. A summary, which was presented at the 8th DoD Electromagnetic Windows Symposium,[5] concluded that Aspen treatment[6] of a smoothly polished sapphire specimen has the potential to improve the high-temperature strength by 35–40 %. More recently, some room-temperature tests have been performed,[7] but the results have not yet been disseminated. The biaxial flexure-strength data[4,7] we are evaluating in this paper concern HEMlite-grade material produced by Crystal Systems;[1] the data were obtained at the University of Dayton Research Institute (UDRI) on a Instron test instrument using a SiC ring-on-ring fixture with inner and outer rings of 15.88 mm and 31.75 mm diameter, respectively. The object of this paper is to present a comprehensive analysis of these data—an analysis based on Weibull's theory of brittle fracture[8]—and, thus, to describe the effects of super polishing in terms of intrinsic material properties, i.e., the characteristic strength and the Weibull modulus.[9] This procedure allows us to obtain proper expressions for the failure-probability distribution that take the area effect into account and, therefore, to predict the actual allowable stress of super-polished sapphire windows.

In this context, we intend to first (Sec. 2) clarify issues regarding the application of the Weibull theory, keeping in mind that the theory is predicated on tensile-stress initiated fracture at strength-limiting surface flaws. Specifically, 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 configuration. Furthermore, upon defining the characteristic strength for biaxial loadings, we derive an appropriate expression for the failure-probability distribution, which should allow us to assess the strength of sapphire windows on a realistic basis. In Sec. 3, this model will be taken advantage of to address the issue of sapphire strength enhancement by means of improved polishing procedures, as seen in the light of biaxial flexure strength testing performed at 600 ^{o}C.[4] The results of room-temperature testing,[7] which involves smaller sample populations, will be evaluated in Sec. 4, where we demonstrate that the strength improvement is consistent with our high-temperature observations as well as prior room-temperature work performed at the Oak Ridge National Laboratory (ORNL).[3] Finally, the conclusions are stated in Sec. 5.

2. DATA ANALYSIS METHODOLOGY

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,

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

where $\overline{\sigma_i}$ designates the arithmetic average of the recorded stresses at failure, and $\Delta\overline{\sigma_i}$ is the standard deviation. This "strength" does not represent an objective measure of the intrinsic strength since σ_M depends on the test method as well as the volume or the surface subjected to tensile stresses.[10] Also, it is common practice to interpret the strength data on the basis of a semi-empirical expression derived from Weibull's theory, 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 estimated failure probabilities 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 $\ln[-\ln(1-P)] = 0$ (or $P = 63$ %) intercept. Evidently, the strenght σ_N does not take into account the impact of the test method—loading geometry and specimen size—and does not relate to the intrinsic strength in an obvious manner.

In effect, the results of fracture-strength measurements performed on brittle materials are best modeled in the framework of Weibull's original theory,[8] which postulates that the scatter in recorded strength data is controlled by the presence of randomly distributed defects. According to his 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 test specimens subjected to a stress distribution $\sigma(x, y)$ on the surface under tension can be expressed as follows:

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

where both, the scaling parameter χ and the Weibull modulus m, are true material properties and, therefore, independent of the testing method 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,[2] which implies that the integral term of Eq. (4) reduces to $S(\sigma/\chi)$, where S measures the area subjected to the stress σ; the failure-probability expression then reduces to

$$P(\sigma) = 1 - \exp\left[-S\,(\sigma/\chi)^m\right] \ , \tag{5}$$

which enables us to predict how the stressed area impacts the distribution, if the statistical parameters χ and m are available. For that purpose we may attempt to relate the scaling parameter to the measured strength, that is, the average stress at failure in a given test environment. Bearing in mind that $dP(\sigma)/d\sigma$ represents the probability of a failure occurring at a stress of magnitude σ, we introduce the concept of an *effective* strength,

$$\overline{\sigma} = \int_0^\infty \sigma\left[\frac{dP(\sigma)}{d\sigma}\right] d\sigma \ , \tag{6}$$

which, in principle, matches the measured average strength $\overline{\sigma_i}$; with $P(\sigma)$ as in Eq. (5) the integration is straightforward and yields

$$\overline{\sigma} = \frac{\chi}{S^{1/m}}\Gamma\left(1+\frac{1}{m}\right) , \tag{7}$$

where $\Gamma(z)$ designates the gamma factorial function.[11] Now suppose that the surface under tension has an area s of one (1) unit; in that case the strength $\overline{\sigma}$ defines the *characteristic* strength,

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

which leads to

$$P(\sigma) = 1 - \exp\left\{-\frac{S}{s}\left[\Gamma\left(1+\frac{1}{m}\right)\right]^m \left(\frac{\sigma}{\sigma_C}\right)^m\right\} , \tag{9}$$

for the failure probability, bearing in mind that this expression assumes a uniform stress distribution.

A proper Weibull analysis of biaxial flexure strength data thus amounts to obtaining the 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:

- Rank by ascending order ($i = 1, 2, \ldots, n$) the rcorded 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.[10]
- Fit the $\ln[-\ln(1-P)]$ vs. $\ln(\sigma_i)$ 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 be aware that such estimates can deviate significantly from the true values considering that a linear least-squares fit places inordinate weight on the low-strength data points.
- Fit the P_i vs. σ_i data points to Eq. (9), which is best done by means of a bivariate, nonlinear regression based on the Marquardt-Levenberg (M-L) algorithm, 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.

3. HIGH-TEMPERATURE DATA

The raw data we rely on in this section are recorded in Appendix A of Ref. 4. Disks of c-plane sapphire made by Crystal Systems, which measured 38 mm in diameter and 1 mm in thickness, were sent to four fabricators to be polished to a scratch/dig specification of 60/40 using various polishing procedures. Polishing performed by Elcan simulated the process used to fabricate IR missile domes and will be referred to as "mechanical" polish; polishing performed by General Optics, Insaco, and Meller made use of soft compliant pads to further reduce the surface roughness and will be referred to as "chemomechanical" polish. After annealing at 1200 ^oC, each of the sets of specimens that turned out to be suitable for detailed analysis was subjected to a proprietary treatment at Aspen Systems, which is known to enhance the high-temperature compressive strength of sapphire.[6] Biaxial flexure strength testing was carried out at a temperature of 600 ^oC at the UDRI large-ring fixture on inserting Graphoil^{TM} sheets of 0.13-mm thickness and, thus, to minimize the compressive contact stress;[13] subsequent fractography confirmed that the specimens failed in tension, away from the loading ring, which implies that the data should be amenable to a Weibull statistical analysis. The measured strengths, i.e., $\overline{\sigma_i} \pm \Delta\overline{\sigma_i}$, are displayed in Fig. 1, and strongly suggest that the chemomechanical polishing process enhances the flexural strength of c-plane sapphire.

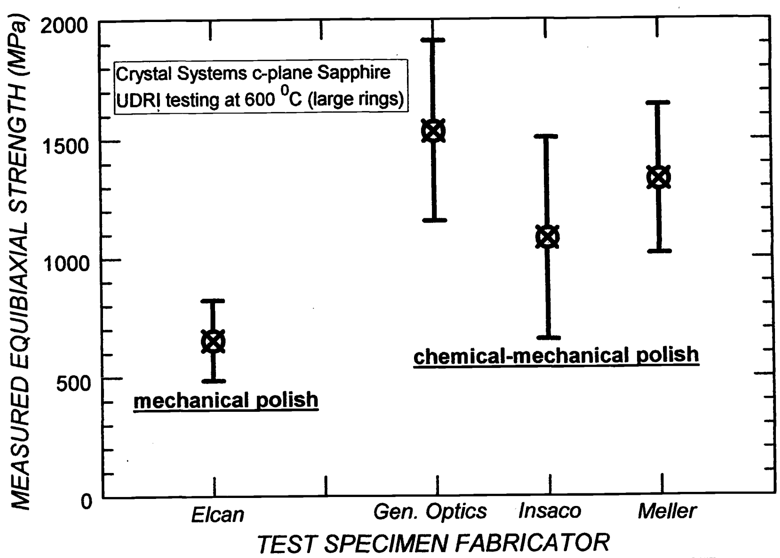

FIG. 1. *Equibiaxial flexure strength of c-plane sapphire at 600 ^oC as measured at the UDRI large-ring facility; the error bars are indicative of the standard deviation based on sample populations of eight.*

Figure 2 illustrates our analysis of the recorded 600 ^oC test data for mechanically (Elcan) polished material; the analysis was carried out according to the procedure outlined in Sec. 2 and relying on commercially available software.[14] The conventional Weibull plot on the left-hand side demonstrates that the eight data points obey Eq. (3) at the 95 % confidence level, thus indicating that a two-parameter model should be appropriate. An M-L fit to Eq. (9) with S/s set equal to 1.98—the uniformly stressed area measured in square centimeter—as displayed on the right-hand side then yields

$$\sigma_C = 708 \pm 13 \text{ MPa} \qquad \text{and} \qquad m = 5.77 \pm 0.72 \,, \tag{10}$$

which confirms that the intrinsic strength of c-plane sapphire decreases at elevated temperatures but suggests that the strength distribution may improve (see Sec. 4). In this connection, it is noteworthy that subsequent testing at 600 ^oC on a small set of "routinely"

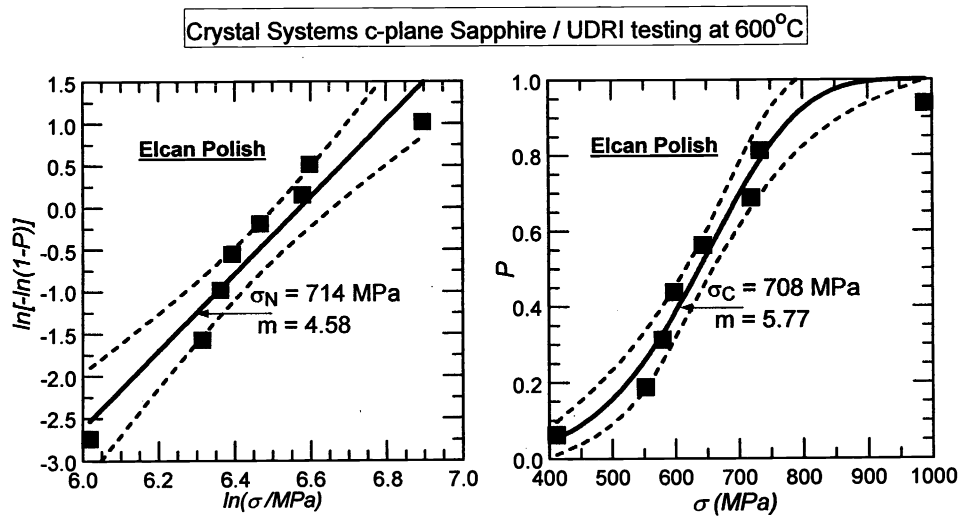

FIG. 2. Weibull statistical analysis of flexural strength data recorded at 600 °C for mechanically polished c-plane sapphire; the broken lines delineate the 95 % confidence bands.

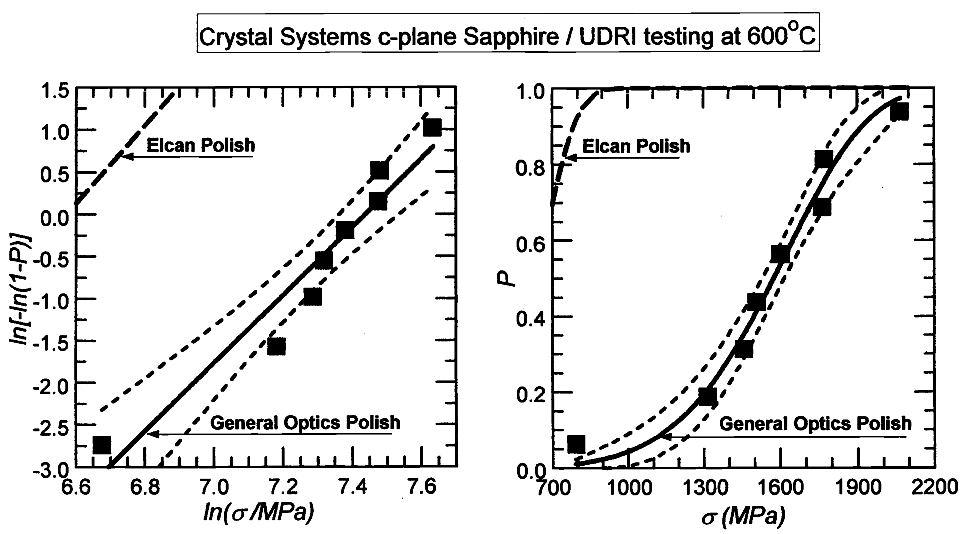

FIG. 3. Weibull statistical analysis of flexural strength data recorded at 600 °C for chemo-mechanically (General Optics) finished c-plane sapphire; the "Elcan Polish" lines refer to mechanically polished material as in Fig. 2 and emphasize the benefit of super polishing.

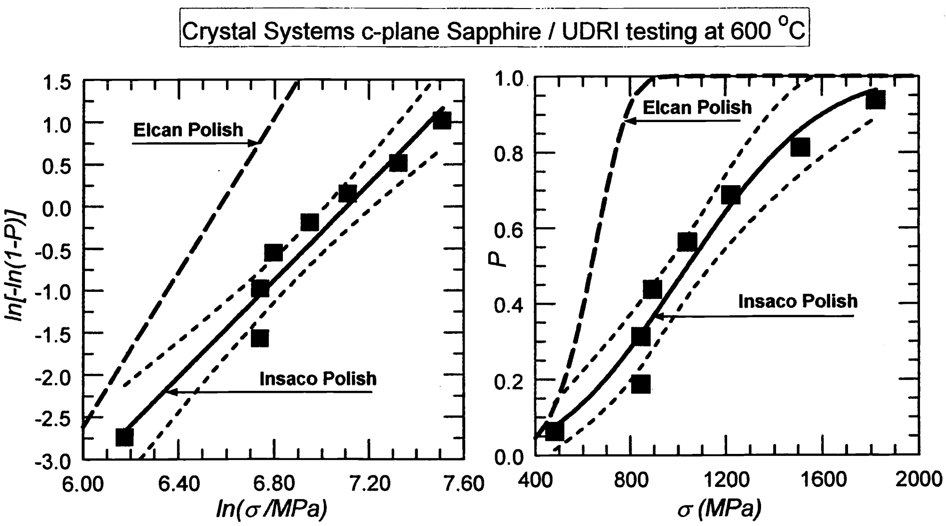

FIG. 4. Weibull statistical analysis of flexural strength data recorded at 600 °C for chemomechanically (Insaco) finished c-plane sapphire; the "Elcan Polish" lines refer to mechanically polished material as in Fig. 2 and emphasize the benefit of super polishing.

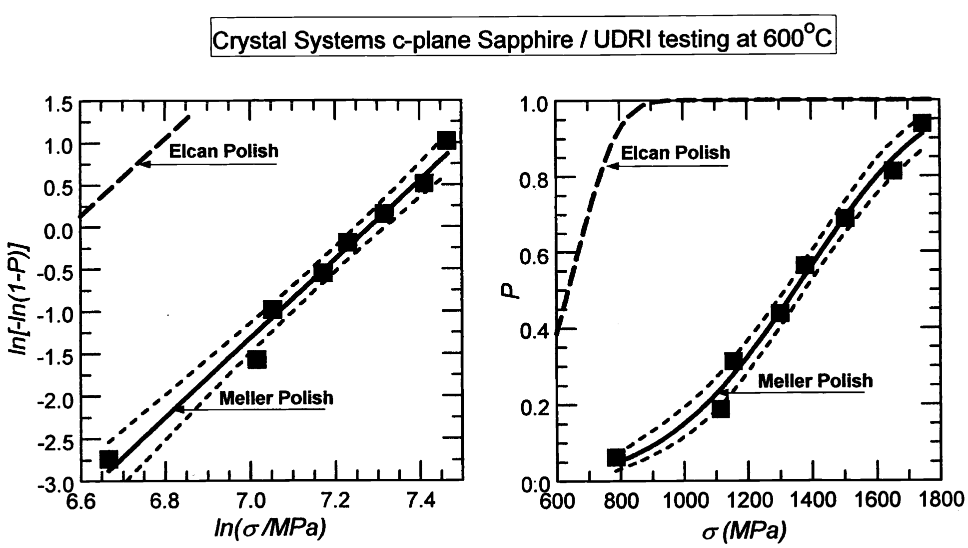

FIG. 5. Weibull statistical analysis of flexural strength data recorded at 600 °C for chemomechanically (Meller) finished c-plane sapphire; the "Elcan Polish" lines refer to mechanically polished material as in Fig. 2 and emphasize the benefit of super polishing.

polished specimens points to $m = 5.16 \pm 0.91$,[15] in essential accord with a Weibull modulus as stated in Eq. (10).

Figures 3, 4, and 5 illustrate a Weibull statistical analysis of the three sets of chemomechanically polished c-plane sapphire that were tested at 600 °C; the polishing procedures differed in terms of the final step, and t-testing that we performed indicates that each set of data originated from a distinct population. The Weibull plots confirm that the recorded stresses at fracture obey a two-parameter model, especially in the case of Meller-polished specimens (see Fig. 5), which should allow us to compare the "performance" of the three polishes on a reliable basis.

Table 1 summarizes the results of this analysis of polishing effects on the biaxial flexure strength of Aspen-treated c-plane sapphire, at elevated temperatures. The two Weibull parameters (σ_C and m) best describe the effects of the surface treatment; evidently, the chemical finish substantially enhances the characteristic strength—without degrading the Weibull modulus—and must be attributed to the reduction in surface roughness. In this regard, we note that the best strength improvement ($\sigma_C \simeq 1740$ vs. 710 MPa) was achieved with a General Optics polish. The improvement reflects the measured rms roughness (≤ 1 vs. 10 nm) and appears to be indicative of the surface flaw depth, in the sense that a strength ratio of about 2.5 correlates with the square root of the roughness ratio considering that, in a mode I loading environment, the theoretical fracture strength obeys the relation[10]

$$\sigma_f = \frac{K_{Ic}}{1.12\sqrt{\pi}\sqrt{c}} , \tag{11}$$

where K_{Ic} denotes the intrinsic fracture thoughness, and c measures the depth of the largest surface flaws. The strength improvement of Meller and Insaco finished specimens does not correlate as well with the roughness (see Table 1), but it is remarkable that X-ray topography reveals lower dislocation densities in the Meller specimens, which may explain the discrepancy. It is not clear, however, why the Weibull modulus of Insaco-polished material experiences a substantial degradation compared to material polished at Elcan, General Optics, or Meller.

The statistical parameter values listed in Table 1 can be exploited to predict the failure-probability distribution at 600 °C of windows made of c-plane sapphire having surface finishes as characterized in Ref. 5. If subjected to uniform biaxial stresses over an area of 1 cm^2, and on assuming that compressive failure can be avoided, the cumulative failure probability of such windows derives from Eq. (9), i.e.,

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

which leads to the distributions displayed in Fig. 6. It is seen that, in the low-failure probability regime ($P \leq 1\%$), Meller as well as General Optics developed chemomechanical polishing procedures enhance the allowable stress of Aspen-treated c-plane sapphire by at least a factor of 2 ($\sim$ 700 vs. 300 MPa for mechanically polished material), thus demonstrating the potential benefit of super polishing in the context of contemplated defense applications.

TABLE 1. Polishing effects on the measured strength and relevant Weibull statistical parameters of c-plane sapphire at 600 °C.

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

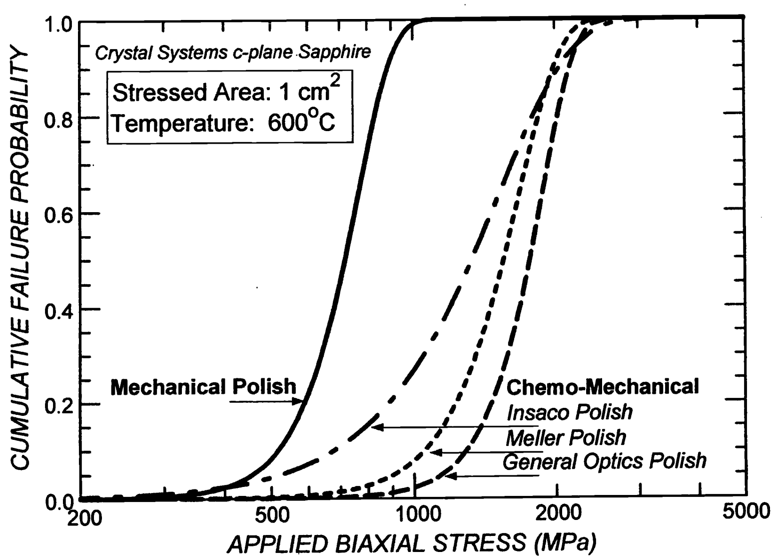

FIG. 6. Cumulative failure probabilities of mechanically and chemomechanically polished c-plane sapphire, at 600 °C; the distributions assume stressed areas of 1 cm^2 and Weibull statistical parameters (most probable values) as listed in Table 1.

4. ROOM-TEMPERATURE DATA

The test data we are examining in this section are recorded in Ref. 7 and concern two separate batches of 38-mm diameter, 2-mm thick c-plane sapphire disks. The first batch was polished using the "routine" procedure commonly employed for fabricating small optical windows; the second batch was polished using a 2–step diamond polishing method followed by a final polish with colloidal silica, thus producing "low-damage" surfaces. Both batches were subjected to high-temperature annealing prior to room-temperature flexure-strength testing at the UDRI large-ring facility ($S = 1.98$ cm^2).

The testing of five routinely polished specimens resulted in a measured strength $\sigma_M = 820 \pm 294$ MPa, which substantially exceeds the measured strength of mechanically polished c-plane sapphire at elevated temperatures (see Table 1). A Weibull plot as displayed in Fig. 7 (left-hand side) points to a modulus of about 3, in good agreement with the results of previous testing[9] that was done at the UDRI small-ring facility ($S = 0.876$ cm^2). An M-L fit to estimated cumulative failure probabilities (see Fig. 7, right-hand side) then yields

$$\sigma_C = 1042 \pm 144 \text{ MPa} \qquad \text{and} \qquad m = 2.68 \pm 0.83 \tag{13}$$

for the characteristic strength and the "true" Weibull modulus, in agreement—within the uncertainty limits—with the results of our previous work ($\sigma_C = 1001 \pm 32$ MPa, $m = 3.41 \pm 0.66$) and, thus, substantiating the area scaling law.[9]

The room-temperature testing of four "low damage" specimens resulted in $\sigma_M = 1260 \pm 207$ MPa, and the Weibull plot on the left-hand side of Fig. 8 suggests that the data should be amenable to a two-parameter Weibull analysis,* albeit a sample population of four implies poor statistical reliability. The analysis (see Fig. 8, right-hand side) yields

$$\sigma_C = 1393 \pm 64 \text{ MPa} \qquad \text{and} \qquad m = 6.06 \pm 1.77\ , \tag{14}$$

which indicates that the chemomechanical finish described in Ref. 7 not only enhances the characteristic strength but also narrows the fracture-strength scatter. It goes without saying that additional room-temperature testing will be required to substantiate this evidence.

To illustrate the impact of low-damage polishing on the room-temperature performance, we display in Fig. 9 the calculated failure probability as a function of the applied biaxial stress for routinely polished and super-polished material, on setting the stressd area equal to 1 cm^2. For mechanically polished c-plane sapphire this means

$$P(\sigma) \simeq 1 - \exp\left\{ - \left[\Gamma\left(1.328\right)\right]^{3.05} \left(\sigma\,/1022\right)^{3.05} \right\} \tag{15}$$

on inserting average Weibull parameter values into Eq. (12); for low-damage polished material we assume that the parameters are as specified in Eq. (14), bearing in mind that these are tentative numbers. It is seen that, in terms of allowable biaxial tensile stresses ($P \leq 1$ %), Fig. 9 suggests ~ 300 MPa for a routine polish and ~ 700 MPa for a low-damage

*At the 65 % confidence level, that is, in terms of standard deviation, there are no outliers.

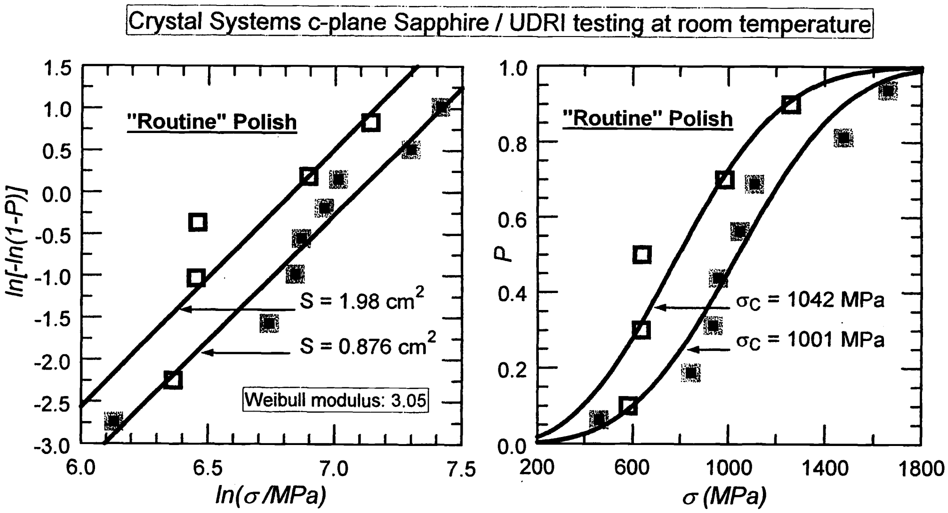

FIG. 7. Weibull statistical analysis of flexural strength data recorded at room temperature for routinely polished c-plane sapphire; the analysis demonstrates that the area scaling law applies surprisingly well considering the limited number of test specimens.

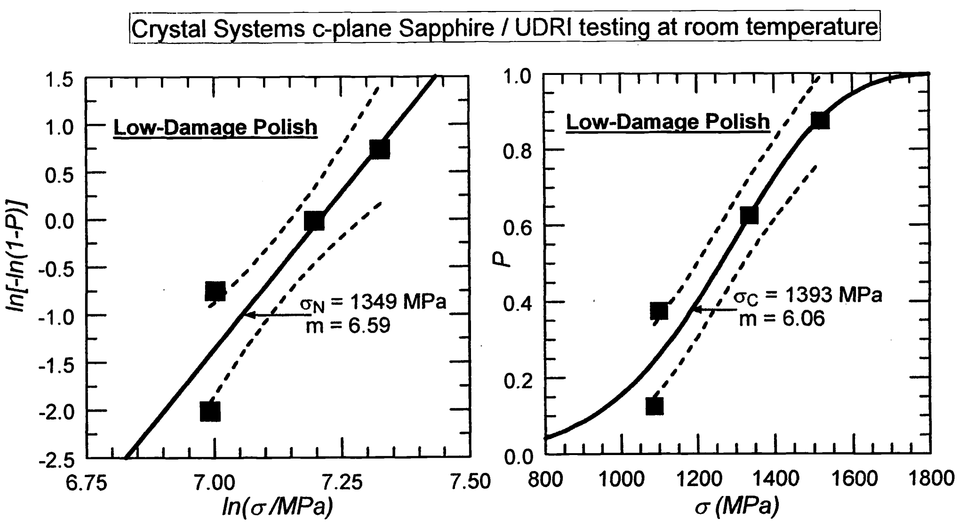

FIG. 8. Weibull statistical analysis of flexural strength data recorded at room temperature for low-damage polished c-plane sapphire; the broken lines delineate the 65 % confidence bands.

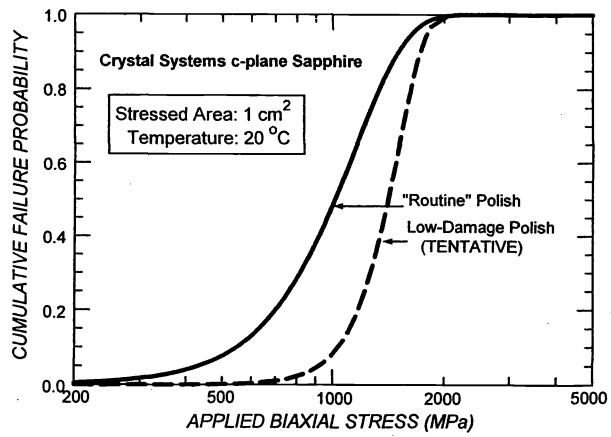

FIG. 9. *Cumulative failure probability of routinely polished and "low-damage" polished c-plane sapphire for stressed areas of 1cm*^{2}*; the statistical parameters of super polished material are as given in Eq, (14) but may be subject to revision.*

polish—in other words, an improvement of 130 %, in surprising agreement with our observations at 600 °C (see Sec. 3). At this point, it should be of interest to compare this evidence with the results of McHargue and Snyder,[3] who performed 4-point bending tests at ORNL on HEMlite-grade specimens having as-received, annealed, and SP (rms roughness: 1.1 nm) surfaces. Upon extrapolating conventional Weibull plots, they concluded that, in the 0-deg orientation (*c*-axis perpendicular to the sample plane), the tensile stress at the 1 % failure probability level should be 90 MPa for simply annealed material but may increase to 170 MPa after super polishing. To the extent that these extrapolated numbers can be relied upon, they point to an improvement of the order of 90 %, which turns out to be quite comparable with our results; as mentioned in the Introduction, McHargue and Snyder also report a "slight improvement at the highest applied stresses," which again fits the situation displayed in Fig. 9, thus confirming the significant improvement of the Weibull modulus induced by chemomechanical polishing.

5. CONCLUSIONS

- A Weibull statistical analysis of equibiaxial flexure-strength data amounts to obtaining the parameters σ_C and m, which is best done by fitting estimated cumulative failure probabilities to the $P(\sigma)$ expression as formulated in Eq. (9).
- A proper evaluation of high-temperature flexural strength data collected on Aspen-treated *c*-plane sapphire specimens 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.
- Ring-on-ring testing performed at room temperature on a small number of *c*-plane sapphire specimens strongly suggests that super polishing enhances both the characteristic strength and the Weibull modulus, thus resulting in substantial improvement ($\sim$ 100 %) of the allowable tensile stress, in accord with previous ONRL findings.

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