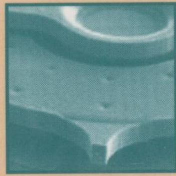

Fractography of Glasses and Ceramics IV

Edited by

James R. Varner
George D. Quinn

Ceramic Transactions
Volume 122

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Proceedings of the Fourth Alfred Conference on the Fractography of Glasses and Ceramics, held July 9–12, 2000, at Alfred University, Alfred, New York.

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Published by
The American Ceramic Society
735 Ceramic Place
Westerville, Ohio 43081
www.ceramics.org

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Cover photo: "Low magnification view of fractured polysilicon sample" is courtesy of S.J. Glass, D.A. LaVan, T.E. Buchheit, and K. Jackson, and appears as figure 12(a) in their paper "Strength Testing and Fractography of MEMS Materials," which begins on page 227.

Library of Congress Cataloging-in-Publication Data
A CIP record for this book is available from the Library of Congress.

For information on ordering titles published by The American Ceramic Society, or to request a publications catalog, please call 614-794-5890.

Printed in the United States of America.

4 3 2 1–04 03 02 01

ISSN 1042-1122
ISBN 1-57498-121-8

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Preface

This is the proceedings of the Fourth Alfred Conference on the Fractography of Glasses and Ceramics, held at Alfred University July 9–12, 2000. In the five years since the last conference, our understanding of fractography of glasses and ceramics has improved, and more people are using fractography in their work. Quite simply, it was time to meet again; past participants were asking us to hold another conference. The objectives of this and the previous three conferences (1986, 1990, and 1995) were to review the progress in understanding fundamental principles of fractography of brittle materials; to highlight applications of fractography in research, development, production, testing, and product failure analysis; and to promote discussion among diverse users of fractography of glasses and ceramics.

We believe these objectives were met. All presentations were high quality; participants heard over 30 presentations; discussion was lively and in-depth. There was a blend of seasoned fractographers and newcomers, people from academia/research facilities and from industry, explorers of fundamental phenomena and practitioners using fractography as a tool. The papers collected in these proceedings demonstrate much progress in this field. We were especially pleased with the session on edge chipping, in which a diverse collection of materials scientists, fractographers, and archeologists shared their common experiences and unique perspectives. This session exemplified the benefits of bringing together fractographers from different disciplines.

One measure of the success of the conference is the large number of participants who, when taking their leave at the end of the meeting, asked if we planned to do this again. We are encouraged by this response, and we do indeed plan to have the Fifth Alfred Fractography Conference in about four years.

Special thanks go to the Alfred people who worked so hard to make the participants feel comfortable during their stay here. We take pleasure in singling out Mrs. Marlene Wightman and her assistant, Mrs. Barbara Timbrook, for our expression of gratitude. They made all the local arrangements, managed registration, and kept an eye on the budget. Throughout all this, they maintained a marvelous sense of humor, making our task so much more enjoyable.

J.R. Varner

G.D. Quinn

Dedication

We dedicate the proceedings of the Fourth Alfred Conference on the Fractography of Glasses and Ceramics to Van Derck Fréchette, Prof. Emeritus of Ceramic Science at Alfred University. Prof. Fréchette passed away on March 20, 2001, at the age of 85, shortly before the proceedings manuscript was sent to the publisher.

Prof. Fréchette was the doyen of fractographers of brittle materials. He spent more than 50 years doing research in the field and serving as a consultant and expert witness. His book, *Failure Analysis of Brittle Materials*, is the standard reference for newcomers and seasoned practitioners alike. This book, and other publications in the field, elucidated the interpretation of fracture markings. Through his summer short course at Alfred University, he introduced hundreds of people to the mysteries and applications of fractography of glasses and ceramics. He was a teacher, and he loved to share his vast knowledge with his students. Prof. Fréchette knew how to tell a story, and his audiences loved hearing about his exploits as a fractography sleuth. We will miss his wit and humor.

The idea for the first conference was Prof. Fréchette's. He brought it up for discussion one day over coffee in his office. The year was 1985, and Prof. Fréchette thought that the time was ripe for people interested in failure analysis of brittle materials—glasses and ceramics—to have a conference of their own. He thought that we ought to be able to find enough people to make presentations and that the conference would attract enough participants to make it worthwhile. He was right, of course. We didn't start out with a series in mind, but the participants at the first conference asked for a second. So, three conferences later, the Alfred fractography conference series is world-renowned. We know that Prof. Fréchette took particular delight in this conference series as solid evidence of the growth of the field of fractography of glasses and ceramics.

We honored Prof. Fréchette at the conference banquet with a brief program reviewing his accomplishments in fractography. Several participants spontaneously related personal stories about their interactions with Prof. Fréchette. There was a nice blend of good-natured humor and seriousness. All of this was a surprise to Van, and he was embarrassed by all the attention, even though he clearly enjoyed it. With a smile on his face, he told us that it sounded like a eulogy. We laughed, of course. Now, we look back and realize that he was right. We are deeply saddened that he will not be at future Alfred Fractography Conferences, but we are happy that we used that opportunity to express our affection and gratitude.

J.R. Varner

G.D. Quinn

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FRACTURE BEHAVIOR OF DOUBLE-ION-EXCHANGED GLASS

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ABSTRACT
Mechanical strength measurements have been made on double-ion-exchanged disc samples to determine reliability and dependence on stressing rate. The sodium aluminosilicate glass had been exchanged for 24 hours in KNO_3 at 500°C and then for 30 minutes at 400°C in a 30/70 mix of sodium nitrate and potassium nitrate to produce a stress profile with a compressive stress peak below the surface. Very narrow ranges in failure stress values were observed in all tests with Weibull moduli as high as 61. The results suggest an insensivity to the initial flaw size. Failure stress showed an anomalous slight increase with decreasing stressing rate. Fractographic examination revealed that the failure process involved a sequential activation of successively less severe flaws as the applied load increased. Fracture propagation from these flaws produced an array of shallow cracks on the surface loaded in tension. Ultimately, failure resulted from propagation of a very small flaw (dimensions of about one micrometer).

INTRODUCTION

This paper describes an assessment of double-ion-exchanged glass for use in an application that required survival of a glass disc for an extended period at very high stress and, subsequently, a very rapid and complete failure of the disc at only slightly higher stress. In that failure, the disc would be required to dice into fragments whose dimensions did not exceed 2 mm. This level of dicing would require some type of prestressing treatment. It can easily be achieved with a single exchange of an alkali-aluminosilicate glass and closely approximated with a thin, thermally-tempered sheet. However, with either of those treatments, the typical range of failure stresses[1] would be too broad to satisfy the first requirement.

The double-exchanged glass chosen for the study was stressed using a process developed at Pennsylvania State University and Univ. di Trento in Italy[2]. The authors of this process, Green, Tandon, and Sglavo, refer to the product as "Engineered Stress Profile (ESP) Glass."

The current work concerns the mechanical testing of the material and an evaluation of the failure characteristics using fractographic analysis. Because the mechanical behavior of the double-exchanged glass depends so critically on the details of the stress profile generated by the exchange treatment, that profile was measured and used to evaluate the failure process under a bending load.

MECHANICAL STRENGTH MEASUREMENTS

Objectives

The primary objective of the mechanical strength testing was to determine whether double-ion-exchanged glass had the potential to perform in the proposed application. Specific areas of interest were 1) failure stress distribution, 2) dependence of strength on strain rate due to environmental effects, and 3) fragment size.

In general, mechanical failure of a brittle material depends on two factors, 1) the size of a flaw present in the part and 2) the magnitude of the tensile stress at that flaw. Fracture propagation from that flaw requires that the stress intensity, K, exceed some value. Provided there is no subcritical crack growth, the requirement is that K must exceed the critical value, K_{IC}.

In a sample with no prestress, failure from a bending load occurs when the applied tensile stress activates the "worst" flaw in the region under that stress. In thermally tempered or ion-exchanged glass, the failure process is more complex. Failure requires the applied stress to be high enough that the K value of a flaw continues to exceed K_{IC} (or slightly less in the event of SCG) until the crack penetrates into the region of central tension[3]. For the stress profile produced by the ESP process, the stress varies from approximately zero at the disc surface to a very large compressive stress peak beneath the surface. Schaeffer[3] noted that such a profile should "hamper crack growth." Because of that increasing compressive stress, a flaw can begin to grow at some level of applied tensile stress, but stops growing after limited penetration. Additional growth requires an increase in the applied stress. Schaeffer suggested several processes that would produce that type of profile. Tandon and Green[4] presented a theory of how such a profile would affect the crack propagation during failure and showed how the beneficial effects of such a profile might be optimized.

Procedures

Measurements of mechanical strength were made on cylindrical disc samples using ring-on-ring loading. Specimens with nominal diameter of 25 mm were supported on an outer ring of diameter 18.8 mm and loaded with an inner ring of diameter 9.1 mm. Ring-on-ring loading was selected to reduce the possibility of failures from sample edges. It also provided sample geometry and mechanical loading approximating that anticipated in the proposed application.

All of the glass used in these tests was CGW0317 sodium aluminosilicate glass sheet from Corning, Inc. Sheets were supplied with nominal thickness of 2.79 mm (0.106 inch), 2.16 mm (0.085 inch), and 1.78 mm (0.070 inch). Disc samples were fabricated by sawing to approximate shape, then stacking and gluing the rough cut pieces into a bar that was then centerless-ground to size.

The ion-exchange treatment consisted of an initial exchange for 24 hours at 500°C in pure potassium nitrate. Discs were then given a second exchange for 30 minutes at 400°C in a 30/70 mix of sodium nitrate and potassium nitrate. After the ion exchange, the discs were examined with a low-power binocular microscope. This examination included the use of a polariscope to detect possible stress risers associated with flaws. Many of the discs showed significant stresses at scratches and edge chips. To limit the possible effect of these potential flaws on the strength measurements, the discs were oriented during the strength test so that, when possible, the flaws were located on the face that was loaded in compression.

The location of the fracture origin and the details of the dicing process were expected to be important to the understanding of the mechanical behavior of the double-exchanged samples. Therefore, to the extent possible without influencing the results of the stress measurement, attempts were made to keep the disc fragments together following the failure. This was accomplished by applying transparent adhesive tape to both faces of the disc. The tape extended beyond the edge of the disc so that the two sheets of tape were bonded together.

For tests at low stressing rates, growth of cracks can be substantially enhanced by subcritical crack growth (SCG). To ensure that the effect of SCG was being evaluated in the testing, it was considered necessary to provide direct access of water vapor in the air to the disc face placed in tension during the test. To provide that access, a circular section of the tape, within the inner ring, was removed from the tensile surfaces of samples prior to testing at low stressing rates.

Sets of discs were tested at stressing rates ranging from 0.3 MPa/sec to 7,000 MPa/sec. High-rate testing was done under stroke control while slow-rate testing was under load control. Failure stress values were calculated from loads using Shetty's equation for ring-on-ring loading.[5]

The first series of strength tests was conducted on discs with nominal thickness of 2.79 mm. One group of 20 each discs was tested at a stressing rate of 4270 MPa/sec while a second group was tested at 0.43 MPa/sec. Subsequent testing included measurements over a wide range of stressing rates on discs of 2.16 mm thickness and some additional testing on discs with thickness of 1.78 mm. The choice of 20 samples for the initial tests on the 2.79 mm thick discs was made to ensure a reasonable statistical representation for the strength values. In subsequent tests on the thinner samples with different strain rates and other conditions, generally, six specimens were measured at each stressing rate.

Test Results

Figure 1 shows the Weibull plot for 20 discs tested at a stressing rate of 0.43 MPa/sec. The data show good fit to a straight line with a slope (Weibull modulus, m) of 61 and a characteristic strength of 544 MPa. This modulus value is much higher than typical values (5 to 10) for annealed glass or for glass with a single exchange treatment. For tests with sample sets of five discs, lower Weibull moduli were obtained. The lowest was a value of 25 in a sample set of 2.16 mm thick discs tested at 0.62 MPa/sec.

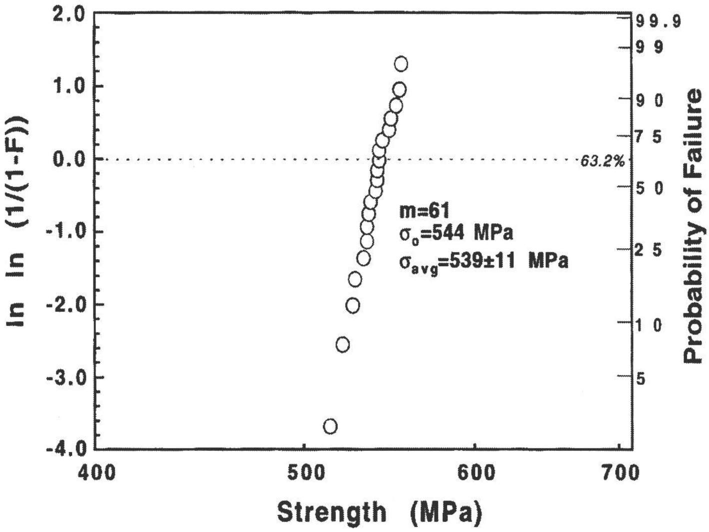

Fig. 1 Weibull plot of failure stresses in 2.79 mm thick discs (stressing rate, 0.4 MPa/sec.

Figure 2 summarizes the data for the mechanical strength testing on disc samples (at three different disc thicknesses). The data points in Figure 2 are mean values and the error bars indicate one standard deviation. The most significant feature of these plots, other than the narrow range of failure stresses at any condition, is the decrease in failure stress with increasing stressing rate. Note, in particular, the data for the 2.16 mm thick samples represented by the filled circle symbols. That decrease is in marked contrast to the typical behavior of glass. In general, for annealed glass and for prestressed glass with peak compressive stress at the sample surface, strength increases with stressing rate.[1] In those materials, the increase is explained in terms of the effect of subcritical crack growth (SCG) on strength. SCG lowers the strength of glass, and the reduction is most pronounced at the lowest stressing rates where SCG is most effective. Conversely, at high stressing rates, SCG is less effective and strengths are greater.

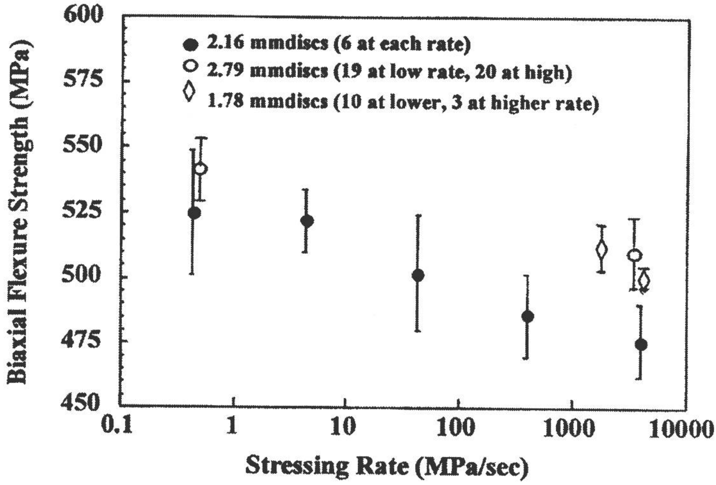

Fig. 2 Summary plot of failure stress data for disc samples.

The differences in failure strengths for discs of different thickness are also somewhat anomalous. Changes in thickness of the discs should produce a small effect on the strength because of the change in the stress profile with thickness for a given ion-exchange treatment. Provided the ion-exchange treatment is the same, the shape of the profiles is the same from the surface to the point where tensile stress is essentially constant. However, to maintain equilibrium, the area in the tensile region must equal that in the compressive region to have mechanical equilibrium. For the thinner disc, the tensile stress must, therefore, be higher and, as a consequence, the magnitude of the compressive stress will be reduced. Because the failure stress depends on the magnitude of the maximum compressive stress, the strength should decrease with thickness. That trend is apparent in comparing the data for the 2.79 mm samples and that of the 2.16 mm thick samples. The apparent increase in strength of the 1.78 mm discs compared with the 2.16 mm discs may indicate that the deformation of the thinner plates was great enough that elastic analysis of the stress was inappropriate or that the exchange conditions were slightly different.

FRACTOGRAPHIC EXAMINATION OF STRENGTH TEST SPECIMENS

Fragmentation Patterns

In general, the fracture patterns of the strength test specimens featured an array of radial cracks emanating from a point with additional cracks at roughly right angles to the radial cracks. An example (Disc #59) is shown in Figure 3. The black arc marks the position of the inner load ring during the test. This was a low-stressing-rate specimen, but one of the few low-rate specimens in which most of the fragments remained in place. For most of the low-strain-rate specimens with the tape removed from the area of high tensile stress, almost all of the fragments in that section were ejected from that area. In those discs, the location of the origin was inferred from the orientation of the cracks in the recovered outer zone. Figure 4 is a micrograph of one of the low-stressing-rate discs (#44) showing the fragments captured on the annular piece of tape between the outer load line and the open circle. In almost every case in the testing of the 2.79 mm thick discs, the tape was cut by the outer load ring so that the circular section of the tape within that ring and the attached fragments fell away from the rest of the disc. In Figure 4, only the taped zone within that circle is shown. The intersection of the dotted lines in Figure 4 indicates the position of the fracture origin.

In Figure 3, the central point of the radial crack array, the origin, is located in the maximum tensile zone, approximately 1.0 mm from the load line of the inner ring. With one exception (Disc #49) in which the failure initiated at the disc edge, all of the origins of the first 40 discs tested were found in an annular region within 2.5 mm of the load line of the inner ring. No failures originated in the 4.0 mm diameter central region, nor were there any fracture origins on the load line of the inner ring. There is an implication, in this preferential location of the origins, that the tensile stress generated in the ring-on-ring loading is not uniform within the inner load ring but is slightly higher in the annular region near the load line.

Failure Origins

In Figure 3, a dashed line marks a pair of cracks running along the same line, in opposite directions from the origin. The presence of this relatively well defined pair of cracks is a common feature of fragmentation in prestressed glass. It occurs because the first fracture extending through the region of central tension from the origin runs in diametrically opposite directions, and, even though branching is occurring, these cracks often continue in a straight line. In what follows, it will be apparent that other fracture events occur in the failure of double exchanged glass before the dicing process begins. Nevertheless, for convenience, the first fracture in the dicing process will be referred to as the "primary fracture" and the cracks as "primary cracks."