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Transition in material removal behavior during repeated scratching of optical glasses

R. THONGGOOM*, P. D. FUNKENBUSCH
Materials Science Program, Dept. of Mechanical Engineering, University of Rochester, Rochester, NY 14627

When indented or scratched, ceramics and glasses often exhibit distinct transition(s) in behavior as a function of the load on the abrasive or depth of its penetration. This behavior has important practical consequences in both material fabrication and wear. For example, so-called ductile mode grinding is dependent on reducing the depth of cut below a critical value so that a relatively damage free and smooth surface is produced. Transitions in behavior have been extensively studied using indentation and scratching on polished surfaces. However, in most practical wear, grinding, and polishing applications, scratching actually occurs on surfaces with existing damage.

In this study the behavior of three different optical glasses during repeated low-load scratching with a Berkovich diamond indenter is reported. A distinct transition point, corresponding to a change from ductile grooving to chipping along the scratch track, was observed as a function of the number of repeated passes (scratches). The critical number of passes was dependent on both the applied load and the material. Several different methods for identifying the transition point were studied and found to give consistent results.

1. Introduction

Scratching of brittle materials by hard abrasive particles is important in both materials fabrication (grinding and polishing processes) and wear. In both cases, the potential for a transition or transitions in behavior with increasing load or depth of indentation has long been recognized. In particular, for fabrication of high precision ceramic and glass components, there is currently great interest in development of grinding processes that induce minimum damage. In "ductile-regime grinding" the depth of cut is held below a critical value so that material removal occurs by a predominantly ductile mechanism, minimizing surface damage [1]. Similarly during wear it is recognized that the cumulative effect of many subcritical damage events can eventually lead to spallation and failure.

Much of our fundamental understanding of both grinding and wear process has been obtained through indentation experiments, in which a diamond indenter is pressed into a smooth (generally polished) material surface. These experiments are physically simple and can tell a great deal about the behavior of different materials. The effects of accumulating damage on a surface can also be studied by means of "indentation fatigue" experiments in which the indenter is cyclically loaded so that the same spot on the sample is repeatedly indented. For example, Banerjee and Sarkar [2–4] examined the initiation of cracks from the corners of Vickers indentations during repeated cycling of soda-lime glass. They found that the number of cycles required to initiate cracking was a strong function of the applied load. Most other indentation fatigue studies have emphasized the growth of cracks produced during the first loading cycle or by a preloading step. The technique has been applied to a wide variety of ceramics including alumina, zirconia, and glass, as well as to ceramic composites and thin films [5–15]. Failure in these studies is generally defined in terms of either the initiation of cracking from the corners of the indenter or the onset of chipping from the surface. The level of damage is generally quantified in terms of the growth of the indentation size (plastic deformation) and the chipped area.

Scratch testing introduces the element of transverse motion into testing and thus provides additional fundamental understanding of the material transport and removal processes associated with grinding, polishing, and wear. Typically, in a single-pass scratch test, a hard indenter is moved across the surface of the sample material. The induced tangential (friction) and normal forces as well as the resulting scratch morphology are obtained to determine material properties such as scratch hardness, scratch friction, abrasion, and wear resistance, coating-substrate adhesion, etc. [16–21]. However, single scratch testing does not capture the cumulative nature of these processes applicable in wear and grinding [22], in which each grit operates on a surface formed by the action of the preceding grits to produce the

Present address: Department of Chemistry, Faculty of Science, Mahidol University, Rama VI Road, Payathai, Bangkok 10400, Thailand final surface. Multi-pass scratch testing [23–27] can be used to provide information about the cumulative nature of the process, missing from single pass tests. In comparison with indentation and single pass scratch testing the number of published studies on multi-pass scratching is quite limited. Moreover, this literature is divided among several different geometries. The current study deals with repeated scratching, in which the indenter follows the same track during each pass. Alternative geometries include parallel scratching, where subsequent passes are made parallel to the original scratch (although sometimes the spacing between parallel scratches is less than the width of the groove), and intersecting scratches.

Several studies have used repeated scratching to examine the failure of hard coatings. For example, Bull and Rickerby [25] studied delamination of titanium nitride coatings applied to steel. Failure was judged to have occurred when an acoustic emission signal exceeded a threshold value. In contrast Bennet *et al.* [26], studying titanium nitride coatings on cemented carbide, found that acoustic emission did not provide a good indicator for failure, possibly because of masking emission signals from damage in the (brittle) substrate. Instead visible fragmentation of the coating was used. Both studies observed coating failure at values well below the nominal (single scratch) critical load, and a strong effect of applied load on the number of passes to initiate failure.

Xie and Hawthorne [27] studied the behavior of a plasma-sprayed alumina coating applied to a mild carbon steel, with an emphasis on the detailed morphology of the coating wear. Single-pass, parallel pass, and repeated pass scratch tests were performed with different indenters. Friction coefficient, acoustic emission, and specific wear rate (volume removed divided by the load and scratch length) were monitored. Single and parallel scratching produced micro-scale dislodgement and fragmentation, evidenced by small angular debris particles. In contrast, repeated scratching produced thin platelet shaped debris, presumably as a result of cracking along splat boundaries. The specific wear rate during repeated scratching was found to be much higher than that during single scratching and showed an initial increase followed by a subsequent decline. During parallel scratching the wear rate showed a very large increase when the width of the scratches became greater than the separation between them, demonstrating the effects of making the second scratch through material already heavily strained by the first scratch.

Xu and Jahanmir [28] studied transitions in the mechanism of material removal in bulk polycrystalline alumina during repeated scratching. Behavior was characterized primarily by microstructural examination of the scratches, including estimation of the removal volume from profilometer traces. Removal volume was found to be small until a critical number of passes was reached, after which it increased rapidly. This was attributed to a transition from microcracking along grain boundaries to grain dislodgement, and a model was developed to predict the critical number of passes as a function of grain size and load. In a later study [29] a second transition, from grain dislodgement to lateral crack chipping, was observed with increased number of passes at high loads.

In this study, the scratching behavior of a set of optical glasses is reported, with a special emphasis on observations of a transition in behavior during repeated scratching at low applied loads.

2. Experimental procedure

2.1. Apparatus and materials

Scratch testing was performed using a Nano Indenter XP [MTS Nano Instrument, Innovation Center] equipped with a nanoscratch attachment. For each scratch test, the specimen surface profile before scratching was obtained by pre-scanning the sample surface with the indenter under a very low load (20 uN). During scratching, the depth, applied load, and frictional force generated between the sample and the indenter were all measured. After scratching, the surface profile of the specimen was again obtained by post-scanning under conditions identical to those used in the pre-scanning. Berkovich indenters, oriented in an edge-leading configuration, were used for all tests. Because of the large number of scratches involved in the repeated scratching experiments, damage to the indenter was of concern. To monitor this a standard hardness test on a standard fused silica sample was performed after each test. When the values from a standard procedure were out of an acceptable range ($\pm 5\%$), the indenter was replaced.

Experiments were performed on three different optical glasses, chosen to represent a relatively wide cross section of those encountered in practice, with different mechanical properties [30] and different behavior during grinding [31, 32]. SF55 is a soft glass normally "easy" to remove. BK7 is a harder glass, which can be ground over a wide range of conditions but presents some representative processing challenges. Fused silica is also hard and is generally considered to be difficult to grind because of the high loads required and its tendency to glaze/blunt grinding tools. It also represents a classic example of an "anomalous" glass, expected to have distinctly different mechanical and fracture behavior [33].

2.2. Single scratch measurements

To determine the critical load required to initiate brittle chipping for each glass, single scratch testing was performed with the normal load continuously increasing from 20 to 150,000 uN along the length of a 0.5 mm scratch. The transition was identified by a rapid increase in the penetration occurring at a particular load. The critical load was defined as the load at which a sudden drop in the final penetration (after unloading) started. The test was performed twice for each specimen and the average values taken. The point of onset was also confirmed by optical microscope examination of the scratch surfaces following testing. Table I summarizes the critical loads determined using this procedure.

TABLE I Critical loads obtained from single scratch testing with an increasing load

Material	Critical load (mN)
SF55	100
BK7	130
Fused silica	70

2.3. Repeated scratch testing

All repeated scratch tests were performed under a constant normal load. A speed of 10 um/sec was used in order to minimize frictional heating at the contact between the indenter and the sample. Scratches were 500 microns long. As a check on consistent system operation, all repeated scratches were performed as a part of a three scratch set, consisting of a single-pass scratch, the multi-pass (repeated) scratch, and a final single-pass scratch. Scratches within each set were parallel and each set was separated from other sets by a distance of 1.0 mm. Scratches were conducted under different loads defined specifically for each glass (all less than critical load identified from single scratch testing), and with a maximum number of 25 repeats for the central (multi-pass) scratch. As noted by Bennett *et al.* [26], an advantage of scratch testing is that it samples a line rather than a point. Therefore, for analysis the measured values for each pass were averaged over the length of the scratch.

After testing, the samples were cleaned in an ultrasonic bath for approximately 10 min by placing the specimen in a container with no liquid. Then the specimen was air-blown to remove any remaining loosely attached debris and the damage assessed using an optical microscope. For each test condition, microscopic assessment was performed after completion of a full set of repeated passes [either 20 or 25 repeats]. In addition some tests were terminated after a smaller number of repeats to permit microscopic examination.

3. Results

3.1. Microscopic examination

All three of the glasses tested showed a transition from a ductile, grooving, behavior to a brittle, chipped, behavior with increasing load and number of repeated passes.

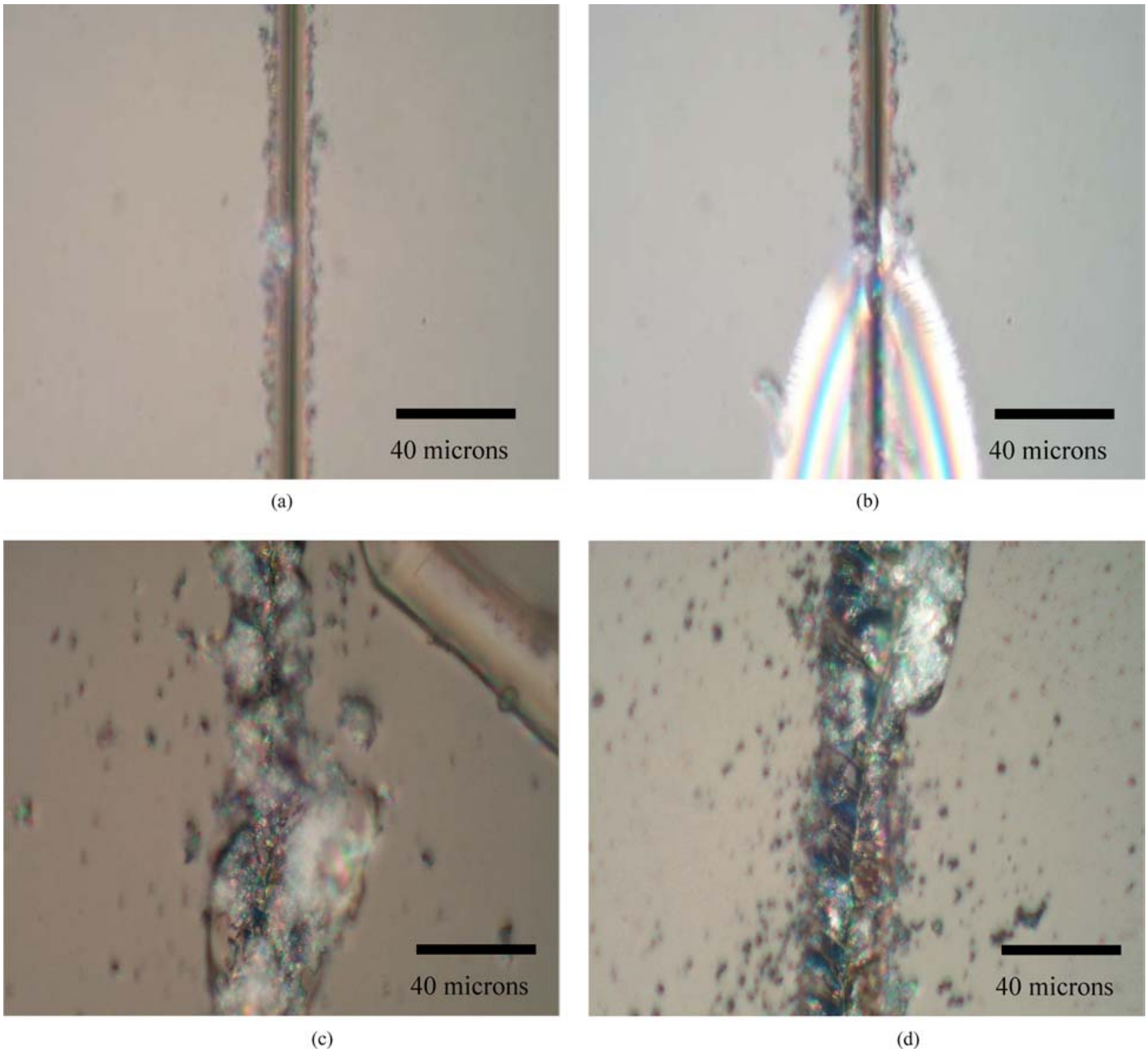

Figure 1 Optical micrographs of BK7 scratched under a normal load of 60 mN after (a) 3, (b) 4, (c) 10, and (d) 25 repeated passes.

TABLE II Summary of results on transition during multi-pass scratching

Materials	Load (mN)	Critical number of repeated passes: Contact depth	Removal volume	Coefficient of variation	Fracture mode (after all passes)
SF55	10	–	–	Decreasing	Smooth
	20	–	–	Decreasing	Smooth
	30	–	–	Decreasing	Smooth
	35	–	–	Increasing	Intermittent
	40	–	–	Increasing	Intermittent
	60	5–6	7–9	4	Fractured
BK7	20	–	–	Decreasing	Smooth
	40	12	11–13	10	Fractured
	60	4	4–6	2	Fractured
Fused silica	5	5	9	5	Intermittent
	20	3	5	2	Fractured
	30	2	4	2	Fractured
	40	1	2–3	1	Fractured

Fig. 1 illustrates this transition for BK7 scratched with a 60 mN normal load with different numbers of repeated passes.

Due to positioning and alignment limitations, it was not possible to return a sample to continue repeated scratching after microscopic examination. Therefore most samples were examined only after completion of the full (20 or 25) repeat sequence. Behavior for each glass at each load was, therefore, microscopically characterized in terms of its appearance at the end of the 25 repeat sequence. These results are summarized in Table II as falling into one of three categories. "Smooth" indicates continued smooth grooving similar to that shown in Fig. 1a. "Fractured" behavior indicates extensive chipping all along the scratch track similar to the behavior in Figs 1c and d. "Intermittent" indicates that the final scratch track had alternating sections exhibiting both behaviors, i.e. some sections with smooth grooving and some with chipping.

3.2. Contact depth

For each pass during multi-pass scratch testing, four parameters were calculated from the data to characterize material behavior and look for evidence of a transition in material removal behavior. The first of these was the contact depth, defined as the depth of the scratch during testing (i.e. with the load applied).

The average contact depth (averaged over the length of the scratch) versus the number of repeated passes on BK7 glass is shown in Fig. 2 for various normal loads. The behavior observed with this parameter could be divided into two groups, both of which are seen in this figure. The first, which for BK7 occurred only a normal load of 20 mN, was defined by a steady increase in the depth with increasing number of passes but with the rate of increase decreasing with the number of passes. In this case the scratch track remained a smooth groove throughout testing or showed an intermittent pattern. The second group, which for BK7 occurred for normal loads of 40 and 60 mN, exhibited an s-curve type

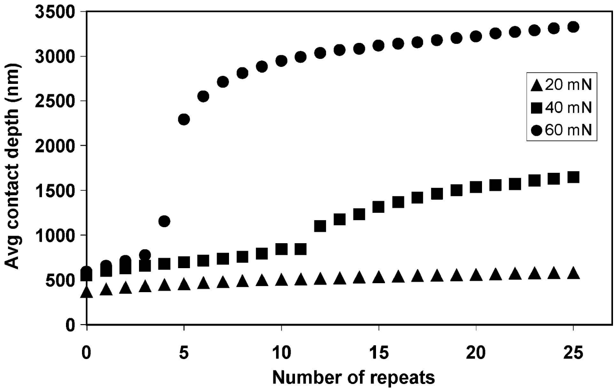

Figure 2 Average contact depth vs. number of repeated passes at different normal loads for BK7 glass.

behavior. There was an initially slow increase in the average contact depth, followed by a sudden increase, and then a return to a slowly increasing depth. These scratch tracks exhibited extensive chipping upon the completion of the test.

Both SF55 and fused silica samples exhibited the same behaviors, although for fused silica at the highest load tested (40 mN) a rapid increase in contact depth occurred immediately (i.e. on the first repeated pass) obscuring the s-shape of the curve. For all samples exhibiting an s-shaped curve, the number of passes corresponding to the transition was taken as the point where the contact depth showed a sudden increase. These results are summarized in Table II.

3.3. Force ratio

Surprisingly, repeated scratching had little effect on the force ratio, defined as the ratio of the tangential to normal force averaged over the scratch length. Fig. 3 shows the data for SF 55, which is typical of that obtained for all of the glasses. Normal load also had a relatively small effect on force ratio, but there was a trend for the ratio to be slightly smaller at smaller normal loads. Hence, force ratio could not be used to predict the transition.

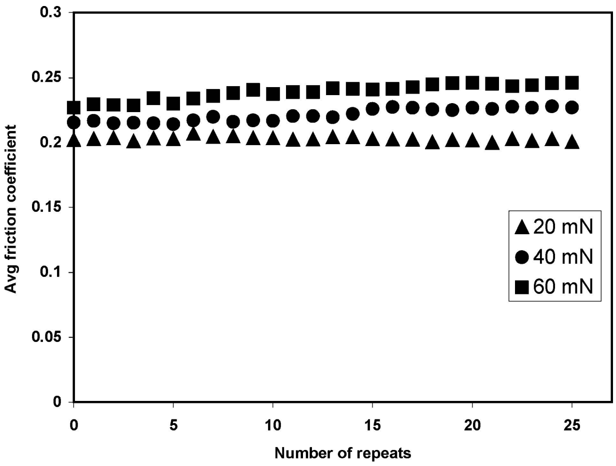

Figure 3 Average friction coefficient versus the number of repeated passes for different normal loads on SF55 glass.

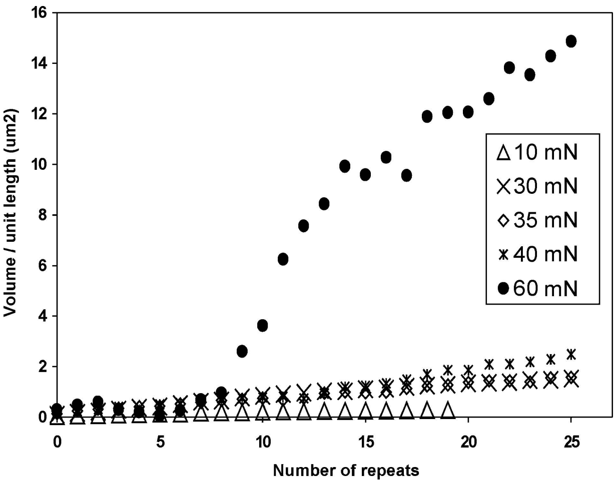

Figure 4 Removal volume per unit scratch length vs. number of repeated passes at different normal loads for SF55 glass.

3.4. Removal volume

The removal volume per unit length of the scratch was calculated based on the final scratch depth (with the load removed) by assuming that the shape of the scratch conformed to the shape of the Berkovich indenter with perfect geometry. The calculated removal volume per unit length as a function of the number of repeated passes on SF55 glass is plotted in Fig. 4 for various normal loads. Again there is an obvious division of the behavior into two groups. The first group (normal loads of 10, 20, 30, 35, 40 mN) shows that, as the number of repeated passes increases, the volume of material removed per unit length also steadily increases, with the effect becoming stronger as the normal load increases. The second group (normal load of 60 mN) shows a more interesting behavior; i.e., the curve can be divided into two parts. From 0 to 6 repeated passes there is again a slow increase. However somewhere between 7 and 9 passes there is a sharp increase. This is taken as the transition point. The other two glasses exhibited similar behavior. Results are summarized in Table II.

3.5. Coefficient of variation

The coefficient of variation is defined as the standard deviation divided by the average. In this study we have determined the coefficient of variation of the contact depth using the average and the standard deviation of the measurements made along the scratch track during each indenter pass. This parameter may be thought of as providing some measure of the roughness along the bottom of the scratch. We believe this is the first time this approach has been used for this application.

Fig. 5 shows the results obtained for SF55 glass. In this case three different behaviors are observed. (Note that lines have been drawn through the 30, 40, and 60 mN data to make the trends easier to see.) At the lowest loads (10, 20, and 30 mN) there is a steady decline in the coefficient of variation, corresponding to a smoothing of the scratch track. At the highest load tested (60 mN) the coefficient shows a slight initial decline followed by a sharp rise. In this particular case there is a peak and the coefficient subsequently declines, however in some cases the rapid increase is followed by a plateau rather than a peak. Finally two of the data sets (35 and 40 mN) exhibit a ragged increase in the coefficient of variation, without a single clearly defined transition point. Data for the other two glasses exhibited similar behaviors. Results are for the coefficient of variation are summarized in Table II. For those cases where there was no clear transition point, the overall trend (increasing or decreasing) is shown.

4. Discussion

4.1. Identification of transition point

In this study four quantitative parameters for identifying the transition in behavior were evaluated: the contact depth, the force ratio, the volumetric removal, and the coefficient of variation of contact depth. Except for the force ratio, which showed no identifiable transition point, data from these techniques is summarized in Table II, along with the result of microscopic observation following the completion of the repeated

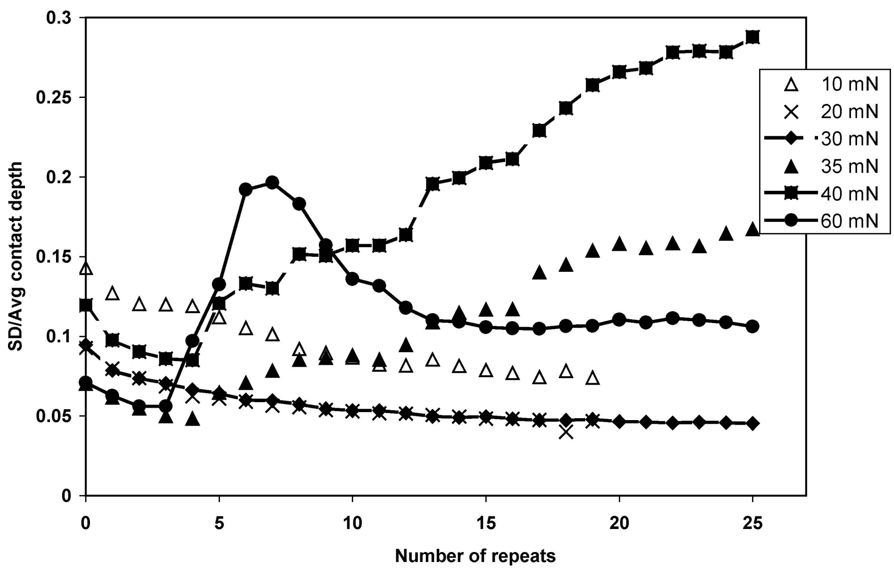

Figure 5 Coefficient of variation of the contact depth vs. number of repeated passes at different normal loads for SF55 glass.

scratch test (i.e. after 20–25 repeats). Results for the various techniques are generally consistent in identifying both the occurrence of a transition and the number of passes required, although there are minor differences in the number of repeats identified as marking the transition.

Average contact depth measurement identified a clear transition point for all samples identified as fractured during microstructural observation. A small, but noticeable, transition was also observed for the fused silica sample with intermittent behavior. However, no transition was apparent for the intermittent SF55 samples.

The transition in removal volume with number of passes (e.g. Fig. 4) is very similar to that observed by Xu and Jahanmir [28] in polycrystalline alumina. A clear transition point was more difficult to determine with this technique as shown by the need to include a range of values in Table II. In addition, the critical number of passes identified by this method tended to be somewhat higher than that from the other parameters. Like the contact depth measurement, the removal volume observed a transition for all samples with a fractured morphology following repeated scratching as well as for the intermittent fused silica sample, but did not show a distinguishable transition for the intermittent SF55 samples.

The coefficient of variation of the contact depth was effective in producing a sharply defined transition point for all of the samples showing a final fractured morphology as well as the intermittent (5 mN) fused silica sample. In addition, although it was not possible to identify a single clear transition point for the other intermittently fractured samples (SF55 at 35 and 40 mN), this measurement was able to distinguish these samples from the samples that showed no transition using the overall trend in the data (increasing coefficient vs. decreasing coefficient). In effect, the coefficient of variance provides a measure of the roughness occurring at the very bottom of the scratch, a value that would be very difficult to obtain by other means. Initially repeated travel of the indenter along the scratch surface tends to gradually smooth the track. The transition to more brittle behavior is, however, clearly marked by a sharp increase in the unevenness of the scratch.

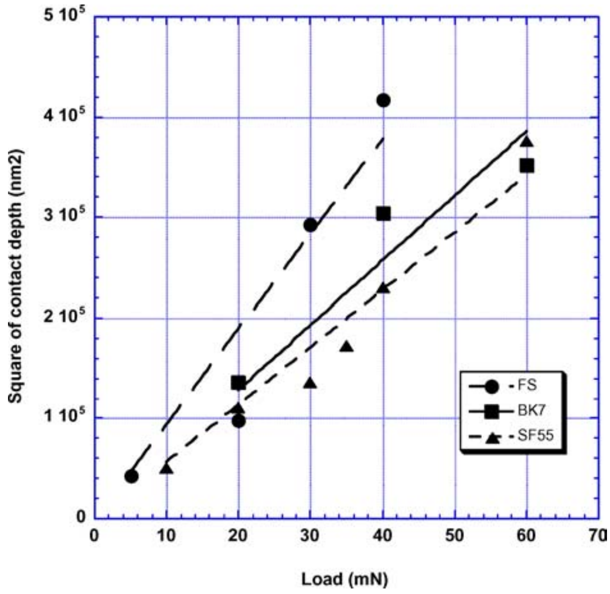

Figure 6 Square of the contact depth vs. load for three glasses.

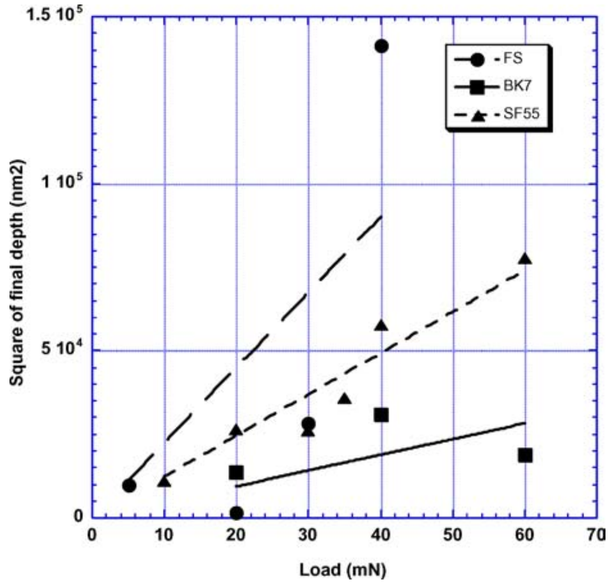

Figure 7 Square of the final scratch depth vs. load for three glasses.

4.2. Dependence of damage on load and number of passes

Damage produced by repeated scratching at subcritical loads may be considered in terms of three stages. During the initial pass, the indenter penetrates to a depth expected to scale in a simple fashion with the applied load, although elastic effects can complicate this function, particularly at low loads/depths. During the second stage, repeated scratching causes a gradual increase in the scratch depth and width. If the load is sufficiently low (and/or the number of cycles small enough) this process will ultimately determine the total damage done by repeated scratching. However, if a critical number of passes is reached there is a transition from ductile grooving to chipping and the volumetric removal undergoes a rapid increase.

During the initial pass it is anticipated that the contact area should scale approximately linearly with applied load (assuming the hardness is independent of load). For the current geometry, this means that a linear relationship is anticipated between the load and the square of the scratch depth. Figs 6 and 7 show the square of the contact depth (i.e. depth during scratching with load applied) and the square of the final depth (depth after scratching), respectively, vs. the applied load for all three glasses. Note that the least-squares lines in these plots have been forced to pass though the origin. Data for the contact depth is seen to show an excellent fit to this simple prediction. Final depth data, however, shows the effect of smaller absolute values and increased scatter. The SF55 data still fits quite well but, the BK7 and fused silica data are badly scattered. (Because it is collected by passing the indenter along the scratch track with only a small nominal load, final depth data tends to be more subject to scatter due, for example, to

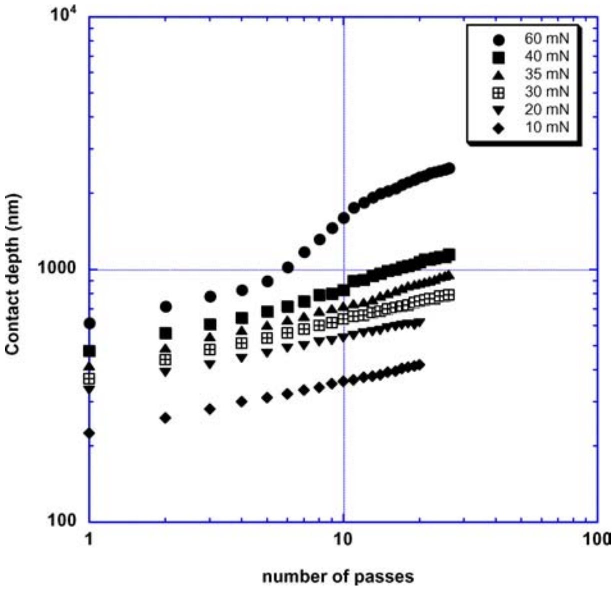

Figure 8 Log–log plot of contact depth vs. number of passes for SF55 at various loads.

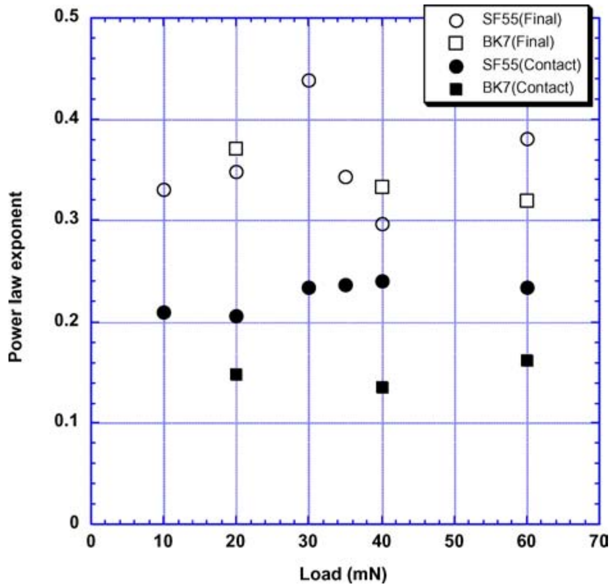

Figure 9 Exponent obtained at different applied loads by fitting depth vs. number of passes data to a power-law equation.

small pieces of debris or other irregularities along the track.)

Following the initial scratch, additional repeats cause a gradual deepening of the track, but at a decreasing rate. We have found that the current data can be well represented by a power law fit, with an exponent that is dependent on the glass, but independent of the load. Fig. 8 shows a log-log plot of contact depth vs. number of passes for the SF55 glass at different loads. It is apparent that, prior to the transition in behavior associated with chipping, the data for each load is nearly linear, and that there is little difference in slope on the log-log plot for different loads. Contact depth data for BK7 glass showed similar behavior, as did final depth data for both SF55 and BK7. (No assessment was possible for fused silica due to the rapid onset of chipping.)

A least-squares fit was applied to determine the power-law exponent for SF55 and BK7 at each load for both the contact and final depth data. For those tests where there was no transition from smooth grooving, the exponent was obtained using data for all of the passes. For those tests where either chipping or intermittent behavior was observed (see Table II), only data from well before the transition was used. In all but one case, the fit to a power law was excellent, with correlation coefficients (R) exceeding 0.99. (The single exception occurred for the final depth of BK7 at 60 mN, where a small number of data points and relatively large scatter combined to give an R value of only 0.87.) Fig. 9 summarizes the power-law exponent obtained. The value is seen to be dependent on the glass, but is essentially independent of the load.

Combining the initial scratch depth (dependent on the load and glass type–Figs 6 and 7) with the power-law dependence on number of passes (exponent dependent on glass type but not load–Fig. 9) provides a simple method for estimating the scratch depth during multipass scratching in the smooth grooving regime.

At sufficiently high loads and/or large numbers of repeated passes the scratch track undergoes a transition from smooth grooving to chipping. Identification of this transition point was discussed above. Fig. 10, shows the number of repeated passes required to initiate chipping as a function of load for the three glasses tested. To make this plot values based on the contact depth parameter from Table II have been used and the critical load for zero repeats (i.e. single scratch) has been taken from the single scratch data (Table I). The overall shape of these curves is similar to those reported for repeated indentation fracture experiments at sub-critical loads [2–4].

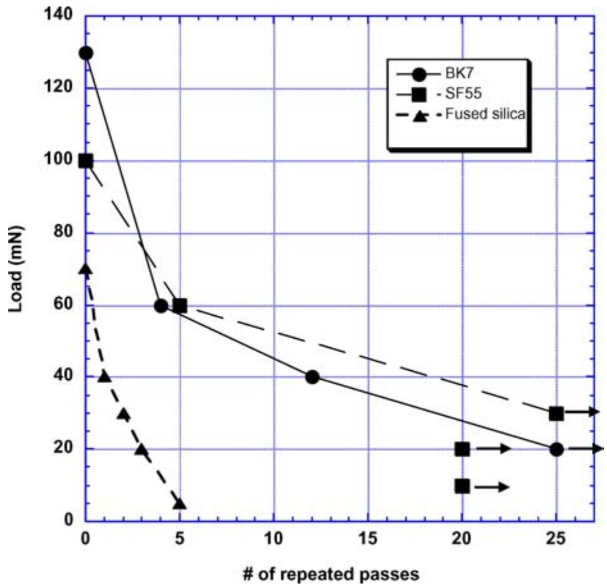

Figure 10 Load vs. critical number of repeated passes for three glasses. Arrows indicate samples which did not undergo a transition even at the maximum number of passes tested.

Two particular features in Fig. 10 are worth noting. First there is a very strong effect of repeated scratching on the load at which chipping occurs. In BK7, for example, after four additional passes the critical load is already less than half that measured in the single scratch test (60 vs. 130 mN). SF55 shows a similar drop, while fused silica has an even steeper decline. Second, chipping was observed for the fused silica glass even at the lowest load tested ($<10\%$ of the critical load for single scratching). However at the lowest load(s) tested no chipping was observed for SF55 and BK7 at the lowest load(s) tested even at the maximum number of repeated passes. This suggests the possibility of a threshold load for multiscratch chipping in these materials, although additional testing is necessary to confirm this behavior.

5. Summary and conclusions

- Repeated scratch experiments have been conducted on three optical glasses at loads below the critical load required to initiate chipping with a single scratch.
- Four different parameters were examined for their ability to identify a transition in behavior from smooth grooving to chipping during repeated scratching. Three of these parameters (contact depth, calculated removal volume, and coefficient of variation of the contact depth) were able to identify clear transition points in close agreement with each other and consistent with microscopic examination of the scratches following testing. The fourth parameter (force ratio) did not show an identifiable transition.
- We believe this is the first use of a parameter based on the coefficient of variation of depth for this purpose. This parameter provides some gauge of the roughness produced at the bottom of the scratch during multi-pass testing.
- The square of the contact depth for the first scratch in each series scaled with the applied load for all glasses, as expected from geometrical considerations (contact area).
- Scratch depth during subsequent passes (below the transition point) closely followed a power law dependence on the number of passes. The power-law exponent was dependent on the glass, but independent of the load.
- All three glasses showed a sharp decrease in the load required to initiate chipping with a small increase in the number of passes. Chipping was observed for fused silica even at the lowest load tested. However, the behavior of SF55 and BK7 glass suggested the possibility of threshold loads below which no transition in behavior would occur during multipass scratching.

Acknowledgments

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