

VISUALIZATION OF GRIT INTERACTIONS DURING THE DUCTILE TO BRITTLE POLISHING TRANSITION

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ABSTRACT

Dual strobe and single strobe once per revolution imaging techniques are employed on the Rapidly Renewable Lap (RRL) to study the ductile to brittle transition observed during the polishing of silicon and calcium fluoride. Analysis of video images during polishing, optical microscopy of the lapped surfaces and scanning electron microscopy (SEM) of the lapping film after polishing suggest the observed step increase in material removal rate with diamond size is due to an increase beyond the critical grit penetration depth necessary for crack initiation, the ductile to brittle transition commonly observed in ceramic and glass materials. A model derived for lateral crack extension in ceramics is applied to the brittle material removal regime here and found to agree with the experimental data. An explanation for apparent shifts in the ductile to brittle transition during lapping of ductile materials using lapping films of various compliances is also discussed.

INTRODUCTION

Lapping and polishing have a long history and are probably the oldest known manufacturing professions [Twyman (1942) and Evans *et. al.* (1998)]. They are considered abrasive processes and are used to produce surface finishes and profiles of extreme precision, and are still today critical to the successful manufacture of many modern products, including optical lenses, metallic bearing surfaces, automotive components, gages, semi-conductor substrates and printed circuit boards. Despite both past and more recent work investigating the relative influence of process parameters [Preston (1927), Twyman (1942), Moore (1974), Parks *et. al.* (1997), Evans *et. al.* (1998)], polishing processes are not well understood, being still highly dependent on the skill, experience and intuition of individual craftsmen. Successful automation of polishing processes will require this intuitive information to be captured in quantitative polishing process models, which would have significant impact on a number of important industries including optical and electronics manufacturing.

This work addresses the need for a better understanding of polish-

ing processes by further developing a polishing machine known as the Rapidly Renewable Lap (RRL), which was implemented on a 300 mm diameter conventional over-arm polisher. Past work on this apparatus by Parks *et. al.* (1997) and Evans *et. al.* (1998) provided new insights and some fundamental modeling of process performance trends for different lapping processes. Specifically, they showed that, for the polishing of two distinctly different materials, single crystal silicon and high-phosphorus electro-less nickel plating, on a 75 μm Mylar substrate, the rate of material removal is approximately independent of grit size using water-based diamond slurries in the range of 3 to 30 μm . In this primary polishing mode, described as ductile, two-body material removal, experimental evidence suggested that abrasives become embedded in the lap surface and remove material via sliding and plowing through the work material in a manner analogous to grinding. A diamond indentation model was assumed and a material removal rate equation was derived for widely spaced diamonds, given as,

$$\frac{dh}{dt} = k_1 \cdot v \cdot N \cdot d_w^2 = k_2 \cdot v \cdot N \cdot \frac{F_i}{H_w} \quad (1)$$

where dh/dt is the change in sample height with polishing time, k_1 and k_2 are constants, v is polishing speed, N is the number of diamonds, d_w is the grit penetration depth, F_i is the load per diamond and H_w is the work hardness. This is consistent with the Preston (1927) equation, showing that material removal rate is proportional to both load on the work and lap velocity. It can also be shown that both the Evans *et. al.* (1998) indention model and Preston's equation are consistent with the Archard (1953) wear model.

Two other primary modes of material removal observed in the Evans *et. al.* work were characterized by (1) a sudden drop in removal rate at diamond sizes of 3 μm and below for both silicon and nickel phosphorus, and (2) a sudden increase in removal rate at diamond sizes above 25 μm for the brittle silicon material, referred to as a "fracture mode lapping" process. Polishing in the low removal rate regime at small

diamond sizes was not addressed in detail by Evans *et. al.* (1998) but has subsequently been studied by Paul (2000) on the RRL for chemical mechanical polishing. However, a model was proposed for fracture mode lapping, based on the work by Evans and Marshall (1981), which showed reasonable agreement with polishing data of fused quartz lapped with alumina on Mylar films. Nevertheless, it was suggested that more detailed experiments should be done to refine the ductile and fracture mode models presented and determine their limits of validity. Further, it was suggested that alternative models based on fracture mode removal via rolling grits should be explored. In continuing the previous work then, the purpose of this paper is to study the transition in material removal mechanism during polishing from ductile, two-body removal to the fracture mode, with its sharp increase in material removal rate during this transition. Specifically, new grit visualization techniques are employed, which allow viewing of the lap and diamonds during typical polishing conditions in order to identify abrasive diamond movements relative to the lap, work and slurry. The fundamental question of whether or not the diamonds are rolling or sliding during this "fracture mode" lapping is addressed. Apparent departures from this model when more compliant polishing systems are used are discussed and explained.

NATURE OF THE DUCTILE TO BRITTLE TRANSITION IN POLISHING

In order to visualize the movement of abrasive diamonds relative to the lap, work and slurry, it was first necessary to identify a material which exhibited a ductile to brittle transition like silicon ($E= 168$ GPa, $H_{knoop} = 917$ kg/mm², $K_c = 0.7$ MPa-m^{0.5}) [Anstis *et. al.* (1981) and Chiang *et. al.* (1982)], but which was transparent to visible light to allow viewing of the lap/work interface through the work. Calcium fluoride ($E= 76$ GPa, $H_{knoop} = 178$ kg/mm² [100], $K_c = 0.28$ MPa-m^{0.5}) [Holloway (1973) and Becher *et. al.* (1978)], was identified as a candidate likely to have the desired properties because of its brittle nature and transparency to wavelengths of 0.15 – 9 μ m. It also has a single crystal structure like silicon and is inexpensive. Subsequently, a series of polishing trials was conducted with water-based diamond slurries on Mylar film using work samples of single crystal calcium fluoride (CaF₂) in a <100> orientation. These tests were performed to determine whether or not a ductile to brittle transition similar to silicon exists in this material. Polishing experiments were also repeated on silicon and nickel phosphorus to confirm the previous test results conducted by Evans *et. al.* (1998). These experiments were done on the same over-arm, 300 mm diameter, polisher previously used in their experiments, at identical test conditions, that is, at a load of about 1 kg/mm², 35 rpm lap speed, 4 cm stroke arm length, and 10 strokes/minute arm oscillation speed. These data are plotted together in Figure 1. The estimated uncertainties of the material removal rates plotted in Figure 1 are approximately ± 0.02 μ m/min based on at least five data points at each condition for the previous data (DR/CE) and fifteen data points at each condition for the new data (RM). The results show that the previous data (DR/CE) and the new data (RM) agree well, within the estimated uncertainty of the tests. Further, they indicate that the modes of material removal for calcium fluoride are similar to silicon,

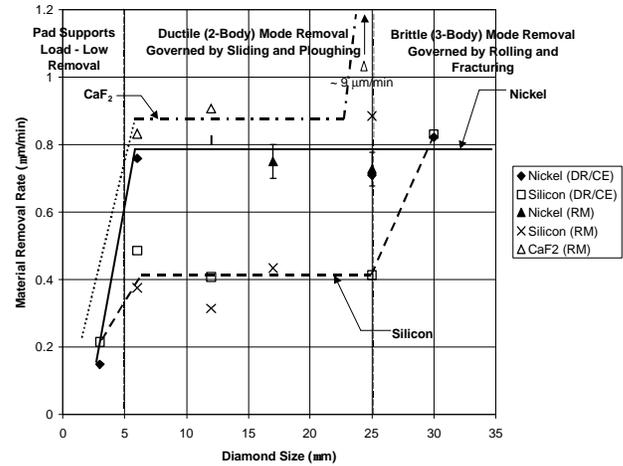


FIGURE 1 – POLISHING OF SI, NI-P AND CaF₂ USING DIAMOND IN AQUEOUS SLURRIES ON MYLAR FILM, REMOVAL RATE VS. DIAMOND SIZE, VELOCITY = 35.2 RPM, PRESSURE = -0.90 G/MP² (NOTE: LINES ARE DRAWN AS VISUAL AIDS ONLY).

exhibiting a ductile to brittle polishing transition between 12 and 25 μ m. The step increase in removal rate during this transition is higher for the calcium fluoride, however, showing about a factor of 10 times higher removal rate (8.4 μ m/min vs. 0.86 μ m/min), relative to a factor just above 2 times for silicon (0.82 μ m/min vs. 0.41 μ m/min). Furthermore, comparing removal rates within each removal regime, the removal rate of calcium fluoride is about 2 times higher than silicon in the ductile regime (0.86 μ m/min vs. 0.41 μ m/min), but about 10 times higher than silicon in the brittle regime (8.4 μ m/min vs. 0.82 μ m/min). Overall, it is most important to note that, over the entire diamond size range studied, these brittle materials both show three distinct material removal regimes, as illustrated in Figure 2. In the first regime, at diamond sizes below about 3 μ m for these polishing systems, material removal is low, likely due to the fact that the diamonds support only a portion of the load on the total sample. In this regime, presumably the remainder of the load is supported both by hydrodynamic fluid and lapping film asperities, which contribute little or no material removal at

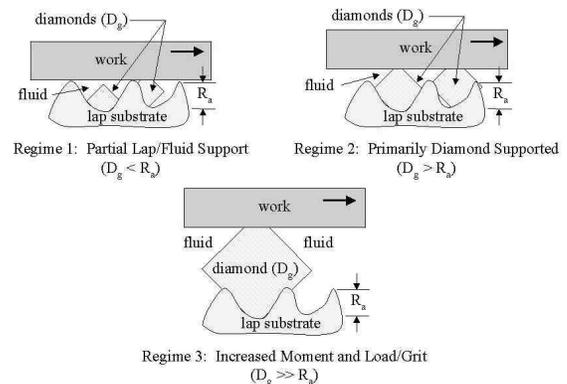


FIGURE 2 – THREE MATERIAL REMOVAL REGIMES OBSERVED USING THE RAPIDLY RENEWABLE LAP, WHERE R_a = AVG. SURFACE ROUGHNESS AND D_g = GRIT DIAMETER.

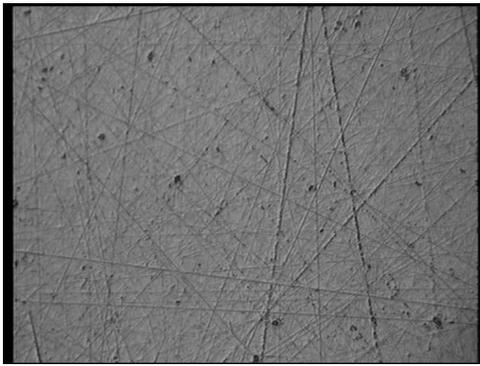


FIGURE 3(A) AU-PD COATED SILICON SURFACE AFTER POLISHING FOR 5 MINUTES WITH 12 μm DIAMOND AQUEOUS SLURRY AT 35 RPM (W/OSCILL.), 1 G/MM², 400X.

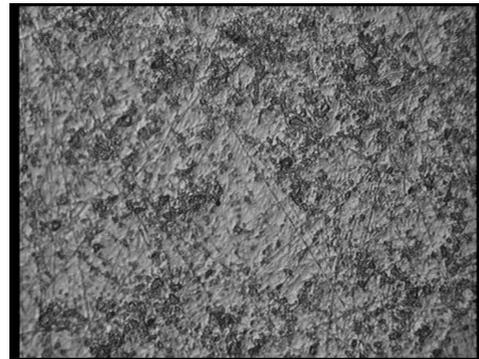


FIGURE 4(A) AU-PD COATED SILICON SURFACE AFTER POLISHING FOR 5 MINUTES WITH 25 μm DIAMOND AQUEOUS SLURRY AT 35 RPM (W/OSCILL.), 1 G/MM², 400X.

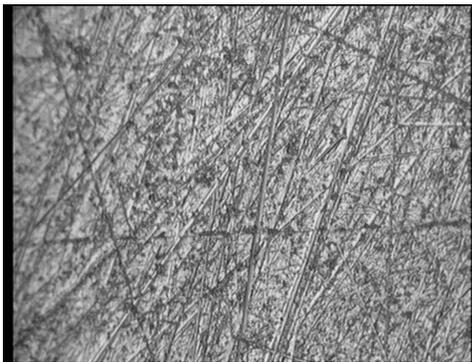


FIGURE 3(B) AU-PD COATED CaF_2 SURFACE AFTER POLISHING FOR 15 MINUTES WITH 12 μm DIAMOND OIL-BASED SLURRY AT 30 RPM (27.9 CM/S), 1 G/MM², 400X.

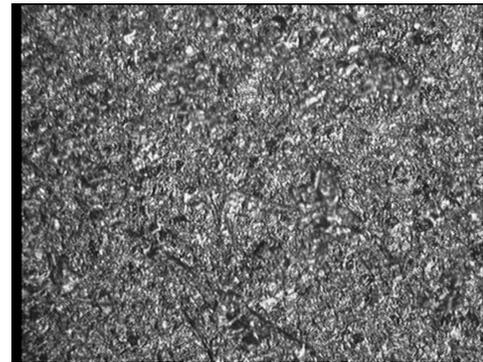


FIGURE 4(B) AU-PD COATED CaF_2 SURFACE AFTER POLISHING FOR 15 MINUTES WITH 25 μm DIAMOND OIL-BASED SLURRY AT 30 RPM (27.9 CM/S), 1 G/MM², 400X.

these polishing conditions. In the second regime, the diamonds support virtually all of the total load on the sample, resulting in a higher removal rate relative to the first regime. Here removal rate is approximately independent of grit size for both the brittle and ductile materials tested. As previously stated, experimental evidence suggests that this material removal regime is governed by ductile removal, as described by eqn. (1), and is generally characterized by a network of smooth "v- grooves" or "scratches," as shown in Figures 3(a) and 3(b). In this material removal regime, generally little or no evidence of grit rolling or work fracturing occurs, although Figure 3(b) indicates slightly more damage present in the calcium fluoride sample, likely due to its lower fracture toughness relative to silicon, and possibly to some pre-existing sub-surface damage from previous runs at higher diamond grit sizes. The third material removal regime illustrated in Figure 2 is one in which material removal increases significantly (Figure 1) and the surface of the workpiece in the case of brittle materials, becomes spalled and fractured as shown in Figures 4(a) and 4(b). Despite the undesirable surface conditions imparted to the work in this material removal regime, it has been unclear what physics are involved or what grit/slurry/work interactions occur to generate the types of surfaces depicted in these figures. Based on previous evidence, it has been hypothesized that the abrasive grits "are loose, and may move relative to one another, and possibly rotate" in what was referred to as a three-body material removal regime

[Misra *et. al.* (1982) and Evans *et. al.* (1998)]. This hypothesis suggests that between the second and third material removal regimes shown in Figure 1, there exists a sliding to rolling transition as grit size is increased, above which grit rolling predominates causing a large number of work fracture events. One can readily envision a relatively larger moment imparted to the diamond grit shown for regime 3 in Figure 2 as the work passes over it, which lends credibility to such a scenario. If this sliding to rolling, or embedding to rolling transition exists then it could explain the sudden step increase in material removal and the source of observed fracturing of the work surface as diamond size is increased. Further, such a rolling condition should create observable "track" marks or periodic indentations on either the lapping films or the work surfaces, or both, as has periodically been observed on unpublished polishing data for quartz and silicon using aluminum lapping film.

However, another reasonable approach is to consider the model derived by Evans and Marshall (1981) for the volume of material removed due to the extension of lateral cracks in ceramics. Their work derives from previous work by Lawn and Evans (1977) who considered the critical threshold conditions required for the growth of median cracks in ceramics from sub-surface flaws. More recent work by Blake and Scattergood (1990) applies the Lawn and Evans derived relationship to

single-point diamond turning of germanium and silicon. The Evans and Marshall model assumes that some initial cracking precursors are present at the work surface, from which lateral cracks form normal to the relative motion of a fixed particle sliding or abrading through the work. Applying their model to polishing in the fracture mode at constant normal load per grit and work velocity, removal rate can be written,

$$\frac{dh}{dt} = k_3 \frac{(E/H)^{4/5}}{K_c^{1/2} \cdot H^{5/8}} \quad (2)$$

where k_3 is a material-independent constant, E is modulus of elasticity of the work material, H is material hardness and K_c is fracture toughness of the work material. If this model holds for fracture mode polishing, we should be able to use it to compare the removal rates for both silicon and calcium fluoride at a constant normal load per grit and work velocity. It should be noted, however, that application of this relation to the current polishing configuration assumes sufficient grit exposure above the lapping film so as not to move the effective material removal polishing mechanism into either the ductile mode or into the first material removal (partial lap supported) polishing regime. This situation might occur if, for example, a more compliant lapping film is used, allowing the grits to penetrate more deeply into the lapping film and thus lowering the effective grit size during polishing. Plugging the values of E , H , and K_c given previously for silicon and calcium fluoride into eqn. (2), we obtain a material removal ratio for calcium fluoride relative to silicon of about 9 times. This compares well with the value of 10 times greater material removal previously quoted from Figure 1, giving some credibility to the application of the Evans and Marshall (1981) model to fracture mode polishing. It would then also suggest that the cause of work fracturing in this material removal regime occurs via fixed diamonds sliding through the work material, as assumed by the model, rather than by rolling diamonds. However, to test this analytical reasoning, polishing experiments were conducted, using calcium fluoride to allow visualization of diamond movements under typical polishing removal regimes.

EXPERIMENTAL APPARATUS AND APPROACH

The basic experimental set-up used for grit visualization experiments is shown in Figures 5(a) and (b). Three variations of this set-up were used in order to explore the feasibility and limitations of visualizing diamond grit movements during polishing. In all cases, a three-station, 300 mm diameter, bench top polishing machine, fitted with a 10 pore/cm Rapidly Renewable Lap [Evans *et al.* (1998)], was used to allow viewing from above, which would have been difficult on the over-arm polisher. In the first variation, a CCD video camera was mounted on top of a long standoff microscope, which was focused on the lap surface. The video camera generated a once per frame pulse, which was used to trigger an electronic flash (strobe). Interactions during polishing were recorded by the CCD video camera, from which a video signal was feed to both a VCR and a computer equipped with video capture software. Subsequent analysis of both the VCR recording

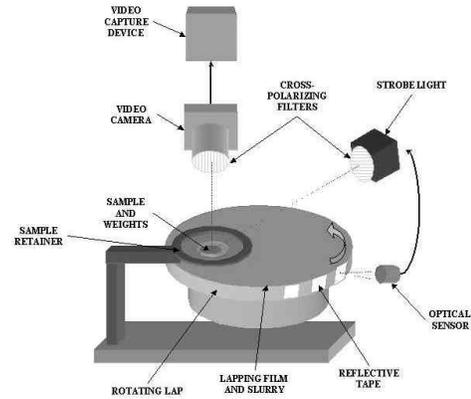


FIGURE 5(A) EXPERIMENTAL RIG USED TO STUDY GRIT VISUALIZATION AND DIAMOND IMAGING.



FIGURE 5(B) EXPERIMENTAL RIG USED WITH DUAL STROBE SET-UP W/REFLECTIVE TAPE ON LAP O.D. FOR STROBE TRIGGERING.

and computer captured images was performed to study grit interactions between both the lap and the work sample. The image produced on each frame exposure of the camera was sharp despite lap rotation because of the short duration of lap illumination by the strobe light, but the area of the lap photographed was uncontrolled. Fluorescein dye was mixed with the lapping slurry in early tests using this set-up with a Mylar lapping film in an attempt to fluoresce the diamonds. Subsequent tests used aluminum foil lapping film and cross polarizing lenses to optimize contrast between the diamond grits and the unworn areas on the highly reflective aluminum foil lapping film. This set-up was used primarily to determine the feasibility of viewing diamond interactions during polishing and to optimize the size of diamonds capable of being imaged. In the second variation, two strobes, each with different color filters, were positioned to illuminate the lap in close succession. A set-up utilizing this technique is shown in Figure 5(b). The delay between strobe flashes, which was controlled by the operator, was set so that each video camera frame captured was superimposed with two differently colored images, one from each strobe illumination. In this mode, a CCD video

camera frame rate of 8 ms was generally used, enough time to capture approximately two images per frame at a strobe delay of 3 ms. Triggering of the strobes was first done with the video camera, but later with reflective tape read by an optical sensor, which allowed viewing of specific portions of the lap repeatedly with time. Using this set-up, an attempt was made to view diamond, slurry and lap feature movements during polishing. The third variation employed a single high intensity strobe, triggered using reflective tape read by an optical sensor at multiple, but identical, locations during each revolution of the lap. A high intensity strobe was used allowing for better lap illumination. Cross polarizing filters, mounted on both the strobe and microscope lenses, were also used allowing for the optimization of contrast between the diamond grits and the worn and unworn areas of the aluminum foil lapping film. This set-up was used to study the movement of diamonds during each lap rotation as well as the formation of worn "islands" on the high spots of the lap at discrete locations during the first several rotations of the lap at various polishing conditions. Together with SEM analysis of the lap surface, these tests proved to be the most beneficial in clarifying the movement of the diamonds during polishing in the two material removal regimes studied.

Because of the slight solubility of calcium fluoride in water, virtually all polishing tests using grit visualization were conducted using oil-based slurries on the bench top polisher. In addition, aluminum foil was used as the lapping film, since previous results showed that alleged grit rolling tended to occur more readily with this film relative to the more compliant Mylar film. Given this change in polishing system and apparatus relative to the earlier tests presented in Figure 1, a set of preliminary tests was conducted at identical test conditions in order to identify any potential change in the nature of the ductile to brittle transition for calcium fluoride on aluminum foil. These tests confirmed the same ductile to brittle transition in calcium fluoride as had previously been observed and no discernable shift in the location of the transition relative to grit size. Successive polishing experiments were then conducted on calcium fluoride in the ductile range using a 12 μm diamond slurry, and in the brittle range using a 25 μm diamond slurry. In each of these polishing trials, a lapping charge consisting of 3 ml of slurry and 3 ml of white mineral oil were used. Polishing runs were usually 3 minutes in duration, except for the once per revolution tests, which were run for 1 minute each. All runs used a sample load of 1 g/mm², as tests on the over-arm polisher had used (Figure 1), and a lap speed of 30 rpm corresponding to an average sample velocity of 27.9 cm/s. This was the maximum speed of the three-station bench top polisher, and approximates the lap speed previously used during tests with the over-arm polisher.

GRIT VISUALIZATION RESULTS AND DISCUSSION

In employing the dual strobe technique during polishing, whereby two images were captured at a preset delay in time on a single CCD video frame, some difficulty was initially encountered in identifying specific diamonds in each portion of a given double image due to variations in lap topography and strobe intensity and

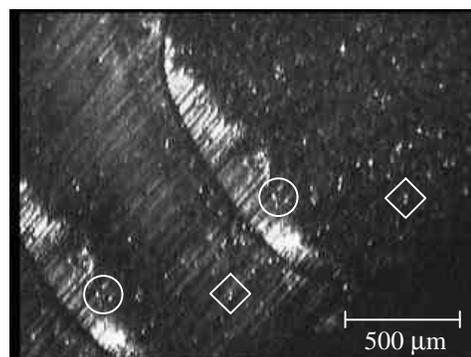


FIGURE 6 DOUBLE IMAGING TECHNIQUE USING A SINGLE STROBE SHOWS DIAMONDS STAY FIXED DURING 3 MS INTERVAL.

positioning. To overcome this problem, a polishing run with 45 μm diamonds, clearly in the fracture mode for calcium fluoride, was performed using a single high speed strobe of lower intensity. An example of an image captured using this approach is shown in Figure 6, which is a double image captured with a single white light strobe at 3 ms intervals during the polishing of calcium fluoride on aluminum film with 45 μm diamonds at a sample load of 1 g/mm² and a lap speed of 14 cm/s. The two lighter regions are identical locations on the lap at each time interval. The light to dark interface to the upper right of these regions represents the transition from a low (bright) area on the lap, shown untouched by diamonds in the slurry, to a high spot (dark) on the lap, where diamonds have created multiple indentations in the aluminum foil and can be seen embedded in the islands. Two specific clusters of 45 μm diamonds at each time interval are pointed out in this photo, showing no apparent diamond movement on the island during this time interval. In general, after review of many similar photos, it can be stated that no discernable diamond movement was observed on the lap high spots at these polishing conditions, despite the large size of the diamonds used during polishing. This finding is further confirmed by Figures 7(a) and 7(b), which are SEM images of aluminum foil lap samples taken from the used lapping film after polishing with 45 μm diamonds and passively rinsing with acetone. Figure 7(a) shows the



FIGURE 7(A) LEADING EDGE OF PLASTICALLY WORKED AREA OF AL FILM AFTER 3 MINUTES OF POLISHING CaF_2 WITH 45 μm DIA., 28 CM/S, 1 G/MM², 20KV, 102X.

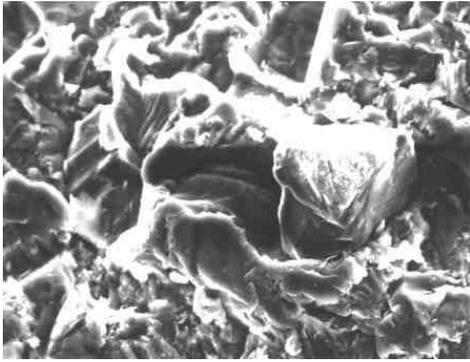


FIGURE 7(B) EMBEDDED GRIT SHOWING EVIDENCE OF A BRIEF ROLL AFTER 3 MINUTES OF POLISHING CAF_2 WITH 45 μm DIA., 28 CM/S, 1 G/MM², 20KV, 1.02KX.

leading edge of a high spot (island) on the aluminum film, which has been plastically worked from multiple indentations of diamonds. Several diamonds can be seen embedded on the island, and a single diamond is shown embedded in the lower undamaged area of the aluminum film at upper left in the photo. Figure 7(b) shows an enhanced view of one of the diamonds embedded in an island. It appears as though the diamond has initially started to roll during polishing, but has then embedded in the film. However, no evidence of sustained rolling is shown in either photo. A review of several samples in this manner did indicate one instance of a rolling grit as it approached a high spot on the lap, indicated by periodic "track" marks. Nevertheless, the predominant feature observed in the majority of samples that were studied for the 45 μm polishing condition was embedded grits and not rolling grits.

Since polishing tests using the smaller grit sizes at 12 and 25 μm could not fully resolve diamond movement using the dual strobe double imaging technique, once per revolution tests using a single high intensity strobe were conducted, described previously as the third experimental variation used. These tests, which show lap

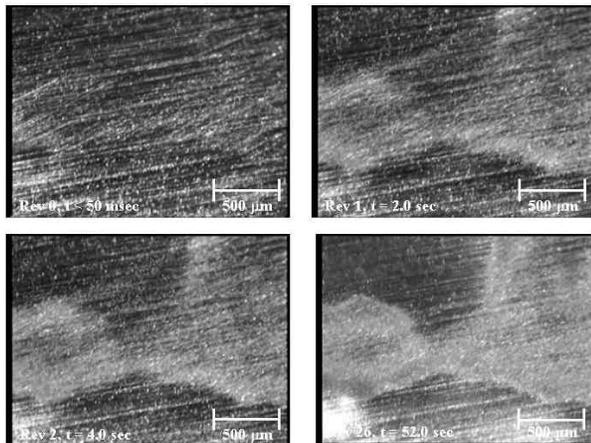


FIGURE 8 SUCCESSION OF ONCE PER REVOLUTION IMAGES SHOWING PROGRESSION OF AN "ISLAND" AT A HIGH SPOT ON AL LAPPING FILM DURING POLISHING W/12 μm DIA., 28 CM/S, 1 G/MM².

changes at a specific location in successive lap rotations, were performed to further discern diamond, slurry, and lap interactions during the first minute of polishing with 12 and 25 μm diamonds. Figure 8 is an example of the series of photos obtained from such a polishing run using 12 μm diamonds, each showing 1.5 mm by 2 mm images of the lap in sequence from left to right and top to bottom for the first 26 revolutions (52 seconds) of lap rotation. Development of the islands can be clearly seen as polishing progresses, gradually revealing an impression of the full "worn" area on the lap. The first photo in the series at upper left seems most revealing in terms of diamond movement in that it shows the first few marks, which appear to be scratches on the surface of the aluminum foil lapping film occurring in just the first few milliseconds of lapping. (Note that in all such once per revolution runs, the lap was circumferentially positioned so that the attached reflective tape triggered the optical sensor and strobe, taking the first image in just the first several milliseconds of the run.) Closer inspection of each photo revealed that diamonds visible during a given revolution were often not visible in a subsequent revolution in the same location on the lap, or were not visible at all in subsequent revolutions. This suggested diamond movement from rotation to rotation, but still did not fully clarify the nature of the diamond movement. That is, does diamond movement take place while the diamonds are between the polishing sample and the lap, which would suggest the diamonds roll, or do they temporarily embed in the film and then release after the polishing sample passes? SEM photos of the lap surface taken after one minute of polishing at 12 μm , subsequent to rinsing loose swarf and diamonds with acetone, appear to answer this question, as shown in Figures 9(a) and (b). They suggest that diamonds predominantly embed, either temporarily or permanently, in the lap rather than roll when polishing at a diamond size of 12 μm . Figure 9(a) shows that a large number of diamonds stay embedded in the aluminum film despite the acetone rinse, some of the more visible of which are pointed out by the highlighted arrows. Other indentation marks around these embedded diamonds are plastically deformed areas on the lap where diamonds have temporarily embedded in the aluminum and then fallen out. Closer inspection of the lapping film shows that additional diamonds

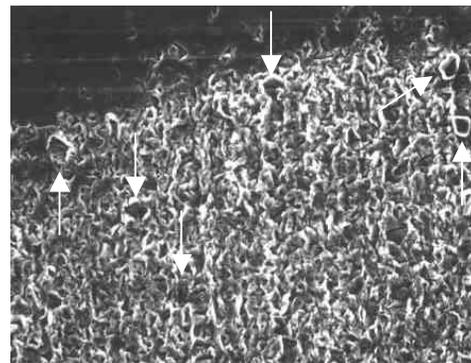


FIGURE 9(A) TRAILING EDGE OF PLASTICALLY WORKED AREA OF AL FILM AFTER 1 MINUTE OF POLISHING CAF_2 WITH 12 μm DIA., 28 CM/S, 1 G/MM², 20KV, 520X.

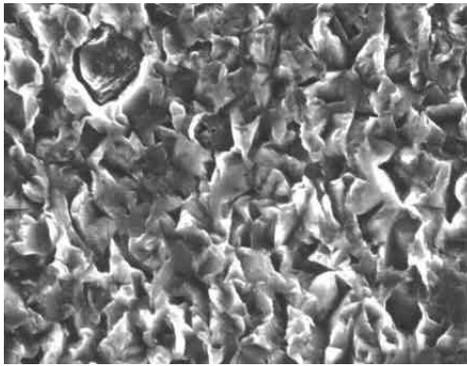


FIGURE 9(B) PARTIALLY HIDDEN EMBEDDED DIAMOND IN AL FILM AFTER 1 MINUTE OF POLISHING CAF_2 WITH 12 μm DIA., 28 CM/S, 1 G/MM², 20KV, 2.05KX.

are embedded in the film, but are barely visible, even at much higher magnifications, as shown in Figure 9(b). It should be noted that there appears to be some indication of sliding diamonds in Figure 9(a), seen as light grooves in the vertical direction with a width approximately equal to the diamond diameter. Although not dominant, this was observed on several different island areas studied at this grit size. However, closer inspection indicated the cause of these grooves to be from sliding diamonds rather than rolling diamonds. Inspection of other areas of the worn lap not shown revealed diamonds embedded throughout the film with various protruding grit heights. This suggests a third possible explanation for the changing visibility of diamonds in successive lap revolutions might be that some become embedded deeply enough into the aluminum film so as to be visible in one revolution and hidden in subsequent revolutions. In considering both images, it is apparent that a combination of temporarily embedding and permanently embedding diamonds is likely during polishing. This situation is consistent with the ductile indentation model, but suggests that some of the diamonds in a given polishing size "act" smaller to varying degrees based on how far they are embedded into the lap. This might explain why grooves of varying width and depth are seen on the polished sample after polishing in the ductile mode (Figures 3(a) and (b)).

Once per revolution polishing tests similar to the ones described above were also performed using 25 μm diamonds. An example of such a run is given in Figure 10, which is a series of photos each showing 1.5 mm by 2 mm images of the lap in sequence from left to right and top to bottom for the first 26 revolutions (52 seconds) of lap rotation. Development of the islands can be clearly seen with polishing as demonstrated with the 12 μm diamonds. The diamonds themselves are also more discernable in the photos using this size diamond. The first photo again shows some apparent "scratching" of the lapping film. A gradual "wearing" of the aluminum film, forming islands at a high spot in the lap as polishing progresses, are also visible. Closer inspection of each photo again revealed that some diamonds visible during a given revolution were often not visible in a subsequent revolution in the same location on the lap, or were not visible at all in subsequent revolutions. To clarify diamond movement, many SEM photos

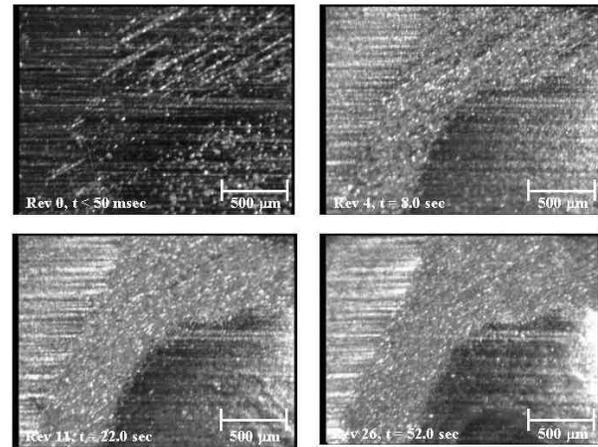


FIGURE 10 SUCCESSION OF ONCE PER REVOLUTION IMAGES SHOWING PROGRESSION OF AN "ISLAND" AT A HIGH SPOT ON AL LAPPING FILM DURING POLISHING W/25 μm DIA., 28 CM/S, 1 G/MM².

were again taken of the lapping film after polishing, following a rinse with acetone to remove loose swarf and diamond debris. Figures 11(a) and (b) are representative of the lapping film condition observed after polishing with 25 μm diamonds. Note that they are similar to the condition observed when polishing

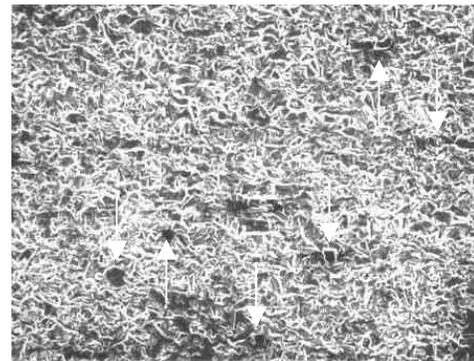


FIGURE 11(A) CENTER OF AN ISLAND IN WORKED AREA OF AL FILM AFTER 1 MINUTE OF POLISHING CAF_2 WITH 25 μm DIA., 28 CM/S, 1 G/MM², 20KV, 200X.

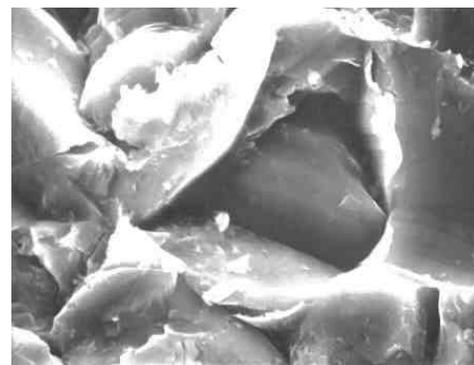


FIGURE 11(B) PARTIALLY HIDDEN EMBEDDED DIAMOND IN AL FILM AFTER 1 MINUTE OF POLISHING CAF_2 WITH 25 μm DIA., 28 CM/S, 1 G/MM², 20KV, 5.1KX.

with 12 μm diamonds. Figure 11(a) shows several visible diamonds embedded at the center of an island on the aluminum lapping film. Figure 11(b) shows that additional diamonds are actually present in the lap, but are difficult to resolve unless viewed at higher magnifications. Overall, the condition of the lap appears to be about the same as it was when polishing with 12 μm diamonds, with no obvious evidence of a transition to grit rolling. So, despite the observed ductile to brittle transition in material removal mechanism (Figure 1) and polished sample appearance (Figures 3(a) and (b) versus Figures 4(a) and (b)) after polishing at both 12 and 25 μm , respectively, no apparent change in the manner in which the diamonds interact with the lap is observed. It therefore seems reasonable to represent the brittle removal mode, above the observed polishing transition, using the Evans and Marshall model, which assumes embedded grits sliding (not rolling) through the work. Further, these results provide compelling evidence that fracturing occurs as a result of crack extension and propagation in the brittle material removal mode, as grits slide through the work material, rather than by grit rolling, even at relatively large grit diameters.

CONCLUSIONS

Based on the analytical evaluation and experimental evidence obtained, the following conclusions are made regarding this work:

- (1) A ductile to brittle transition is observed for the polishing of calcium fluoride in a diamond size range, and therefore load per diamond range, similar to that found for the polishing of silicon. That is, approximately above 25 μm , a transition from ductile to brittle material removal is observed.
- (2) When polishing at large (45 μm) diamond sizes, clearly in the brittle material removal range for calcium fluoride, dual strobe imaging techniques appear to show that the diamonds stay fixed at high spots in the lap, despite the presumed increase in rolling moment imposed on the diamonds.
- (3) When polishing calcium fluoride at both 12 and 25 μm sizes, once per revolution observation of the lapping film indicates diamond movement on the lap high spots during successive revolutions of the lap. However, SEM images show that the diamonds embed in the lap. While many diamonds embed temporarily, giving rise to a multitude of plastically deformed indentation areas on the aluminum lapping film, a significant number embed permanently, protruding at varying heights in the worn lap.
- (4) Given that the degree to which a particular diamond embeds in a lapping film will be a function of lapping film material properties, including its yield point and elasticity (compliance), as well as abrasive shape and type, it is likely that the transition from ductile to brittle material removal, observed for the polishing of calcium fluoride and silicon on aluminum foil, could change significantly for other abrasive or lapping film combinations.
- (5) No significant difference in the manner in which the diamonds interact with the lap at 12 and 25 μm diamond sizes was observed, despite the observed change in material removal mechanism and condition of the polished sample when polishing at these two

diamond sizes. Since the evidence suggests that the diamonds stay predominantly fixed in the lap and slide through the work, the removal mechanism in the brittle mode is apparently due to lateral fracture as the grit slides through the work, rather than from grit rolling. An analytical evaluation of removal rates in the brittle polishing regime for silicon and calcium fluoride indicates that this model is consistent with the model for brittle removal previously put forth for single point diamond turning and grinding of ceramic materials.

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