Wear of Fluorapatite Single Crystals: I. A Method for Quantitative Evaluation of Wear

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A quantitative method for characterizing the surface failure of nonmetallic single crystals under sliding was evaluated. It was found that strain rate, load, and slider design influenced the wear of natural fluorapatite single crystals. High loads and slider designs with small radiuses and sharp angles resulted in the deepest penetration.

The effect of dentifrices and toothbrushing on the wear of enamel, exposed dentin, and cementum has been the topic of extensive experimental research.[1–3] Little attempt has been made, however, to study the mechanisms that control the surface failure of enamel under an applied stress that results primarily from sliding. To identify the wear phenomenon of enamel at the level of slip or cleavage interactions or both requires a simplified system that is not influenced by variable or undetermined effects (for example, dentifrice composition and the rate of brushing).

Single crystals have been used in industry for the past decade to study the phenomenon of wear.[4–6] Bowden, Brookes and Hanwell[7,8] studied the plowing friction and surface damage of diamond and MgO single crystals. By sliding conical diamonds with various apical angles on known crystallographic surfaces in specified directions, they related frictional anisotropy and surface damage to specific slip and cleavage interactions.

The purpose of this study was to evaluate a method for characterizing the surface failure of nonmetallic single crystals under sliding, by an investigation of the wear of natural fluorapatite (*F*) single crystals. This paper describes an experimental procedure and examines the variables of track width, track depth, and area as quantitative measures of wear.

Materials and Methods

The method of producing wear scars was based on the experiments reported by Bowden, Brookes, and Hanwell.[7,8] In the present study, diamond sliders of known geometry were slid across the basal surfaces of natural *F* single crystals in a dry environment. Because only gross effects were sought initially, the effect of crystallographic direction on failure properties was not evaluated. Measurements were based on a series of three one-traversal passes on each of two crystals for a given condition.

Three dependent variables (track width, track depth, and area) were evaluated with respect to four factors (specimen, slider speed, load, and slider design) by means of a factorial design with replications. Analysis of variance[9] and multiple comparisons[10] were used to estimate the effects of factors and their interactions. Factors examined in this experimental design are given in Table 1.

F single crystals* (about one fourth inch in diameter) were sliced perpendicularly to the growth axis, ground flat, and subjected to a four step polishing procedure. The final grit used was AB Gamma Polishing Alumina on AB Microcloth.† An average roughness of a sample of the polished crystals was obtained with the aid of a profilometer.‡ Before being scratched, the crystals were treated in 2-propanol and in distilled

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* Southwest Scientific Co., Hamilton, Mont.

† Buehler, Ltd., Evanston, Ill.

‡ Model AE pilotor with QC amplifier, Micrometrical Mfg. Co., Ann Arbor, Mich.

TABLE 1
EXPERIMENTAL DESIGN FACTORS

Factor	Levels	Comments
Specimen	2	2 crystals (3 replications per crystal)
Slider speed	2	0.025 and 0.076 cm/sec
Load	5	10, 25, 50, 100, and 500 gm (A_1 through A_5, respectively)
Slider design	5	75° cone, 0.018 cm radius 75° cone, 0.064 cm radius 104° cone, 0.005-0.008 cm radius 123° cone, 0.005-0.008 cm radius 143° cone, 0.005-0.008 cm radius (B_1 through B_5, respectively)

water, and placed in a vacuum desiccator for 30 minutes (29 mm Hg). All crystals then were stored in the desiccator at atmospheric pressure until use. Comparison of the natural apatite in powdered form with a synthetic *F** was made by means of infrared spectroscopy (IRS)† and X-ray diffraction.‡

The apparatus used in this investigation to scratch the surface of a specimen and measure the friction force§ consisted of mechanisms that can be categorized as follows: surface grinder, loading jig, friction transducer, diamond sliders, and sample holder.

A precision surface grinder|| that was modified and tested in previous friction and wear studies[11] was used to provide a horizontal, linear motion to a specimen mounted on the grinding table. The hydraulic movement afforded a smooth and vibration-free motion at the speeds investigated (Table 1).

The loading jig might be described most simply as a counterbalanced, mechanical parallelogram with oil-impregnated bronze pin-bearings to provide a frictionless vertical motion. The end of the jig directly below the center of the loading plate was tapped to accept the friction transducer. The lower end of the friction transducer was machined to hold the shaft of the diamond slider by means of a set screw. The loading jig, transducer, and diamond slider are shown assembled in Figure 1.

Industrial grade diamonds of five different geometries (Table 1) were designed and prepared# for use as sliders. The diamonds were mounted on steel shafts and given a gem polish by the manufacturer. Crystallographic orientation of the diamonds was not specified. Before use, the diamonds were given a surface treatment similar to that received by the apatite crystals.

The specimen holder, for tests in a dry environment, was machined from aluminum with a center hole capable of accommodating a single crystal mounted in plastic. Care was taken to mount the surface of the specimen parallel to the grinding table.

To obtain an accurate measurement of speed, a traveling microscope¶ was used to measure the length of the wear tracks. The time of each run was recorded from a stopwatch to the nearest 0.1 second.

The average track width of the wear scars was measured from photographs developed from 4 × 5 inch negatives** exposed at an appropriate objective lens magnification (50 × to 500 ×) on a metallograph.†† The metallograph was calibrated to obtain the true magnification of the negatives.

The values of track depth and area were calculated from the corresponding values of track width and slider geometry (in metric units) with the aid of a computer program. The area computed was based on the assumption that only the front half of the slider contributed to the sliding process.

Results

Comparison of the natural apatite with the synthetic product by means of IRS and X-ray diffraction showed only slight differences in parameters. In the IRS analysis, the ν'_3 (P-O stretch of ionic PO_4^{-3}) peak at 1,000 cm^{-1} and the ν'_4 (P-O bending) peak at 560 cm^{-1} were missing

* X-13283 fluorapatite, General Electric Co., Cleveland, Ohio.

† Model 337 grating infrared spectrophotometer, Perkin-Elmer Corp., Norwalk, Conn.

‡ Vertical diffractometer, Philips Electronic Instruments, Mt. Vernon, NY.

§ The measurement of friction force will be reported in a later paper.

|| Grand Rapids 250, Gallmeyer and Livingston Co., Grand Rapids, Mich.

Wheel Trueing Tool Company, Detroit, Mich.

¶ Gaertner Scientific Corp., Chicago, Ill.

** Panatomic-X Film, Eastman Kodak Co., Rochester, NY.

†† Aristophot, Ernst Leitz, Wetzlar, Ger.

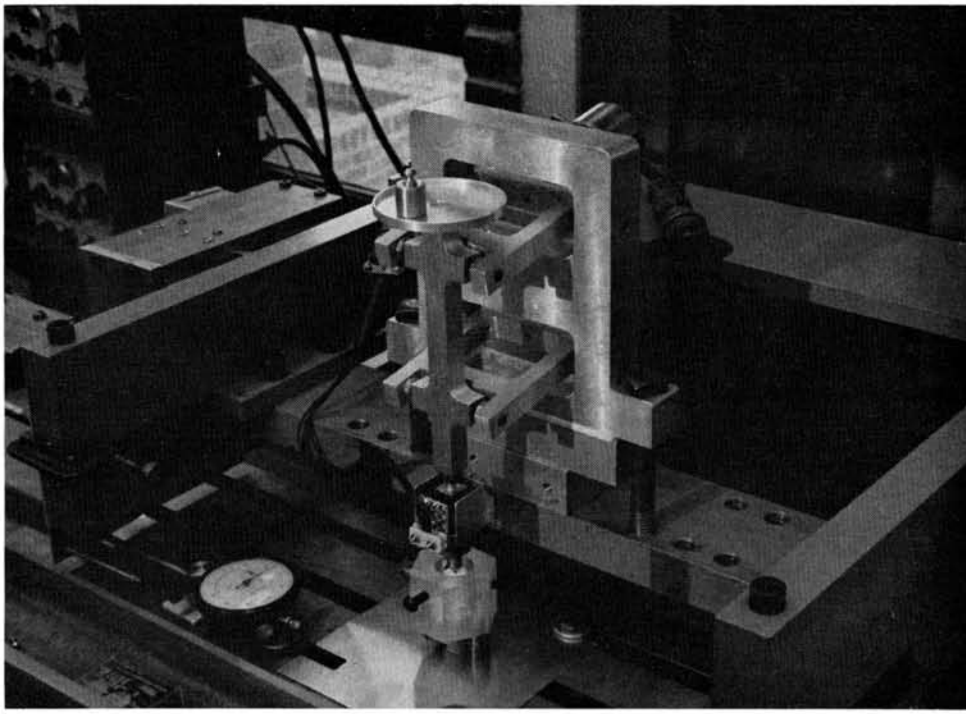

FIG 1.—Assembled loading jig, transducer, and diamond slider.

with the natural apatite. In the X-ray analysis, slight shifts in some *D*-values[12] were found for the natural *F* (Table 2).

An average surface roughness of 0.020 micrometers (μm) (rms) with a standard deviation of 0.007 μm was found for a sample of five crystals traced in two mutually perpendicular directions. No significant directional effect caused by polishing was observed.

The effect of specimen on track width was examined at two levels (two crystals) for five slider designs and five loads with three replications per cell. Unless otherwise indicated, the factorial designs analyzed have a slider speed of 0.025 cm/sec and six replications per cell. The null hypothesis (H_o) that there were no differences between the effects at two levels of the specimen was evaluated for the measured variable track width.

The main effect of crystals was not significant ($F = 0.16 < F_{(1,60)} = 4.00$) at the 0.05 level.[13] First order and second order interactions were not significant. There were no differences between the two levels of the specimen, and consequently, the data from three replications on each of two crystals was combined and considered as six replications for the analysis of the

TABLE 2
COMPARISON OF POWDER—DIFFRACTION (*D*) VALUES

NBS Standard[12]		Synthetic Fluorapatite		Natural Fluorapatite	
D-Value	I/I_o*	*D*-Value	I/I_o	*D*-Value	I/I_o
3.442	40	3.440	22	3.440	47
2.800	100	2.801	100	2.805	100
2.772	55	...	...	2.780	65
2.702	60	2.702	69	2.706	67
2.624	30	2.625	23	2.629	29
1.837	30	1.838	25	1.838	39

* I/I_o, relative peak intensity where maximum peak intensity equals I_o equals 100.

load and slider design factors. The coefficient of variation for the data taken before it was combined was 12%.

The effect of specimen on track depth and area was examined. The main effect of crystals on track depth was not significant ($F = 1.24 < F_{(1,60)} = 4.00$) at the 0.05 level. The main effect of crystals on area also was not significant ($F = 0.02 < F_{(1,60)} = 4.00$) at the 0.05 level. The coefficients of variation for these data were 16 and 29%, respectively. In each instance first order and second order interactions were not significant.

The effect of slider speed on track width was examined at two levels (0.025 and 0.076 cm/sec) for five slider designs at a 500 gm load. The main effect of speed was significant ($F = 7.91 > F_{(1,40)} = 7.31$) at the 0.01 level. In general, the lower speed resulted in a larger track width for the two speeds studied. The first order interaction was significant ($P < 0.025$), which indicated that speed and slider design were not independent. The coefficient of variation for this data was 6.1%.

To rank the speeds with respect to constant slider design, Duncan's New Multiple Range Test[10] was used as shown in Table 3. The speeds were significantly different ($P < 0.05$) only for slider designs B_3 and B_4 for the track width variable.

The effect of slider speed on track depth and area was examined as described previously. The main effect of speed on track depth was significant ($F = 13.8 > F_{(1,40)} = 12.6$) at the 0.001 level. The lower speed resulted in a larger track depth. The main effect of speed on area also was significant ($F = 14.3 > F_{(1,40)} = 12.6$) at the 0.001 level. The lower speed resulted in a larger area. The coefficients of variation for these data were 12 and 22%, respectively. In each instance the first order interaction between speed and slider design was significant.

TABLE 3
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR SLIDER SPEED DIFFERENCES

Slider Design	Speed (cm/sec)	Mean (μm)
B_1	0.025–0.076	49.3– 54.7*
B_2	0.025–0.076	56.4– 59.6*
B_3	0.076–0.025	83.6–107.0
B_4	0.076–0.025	90.0–106.0
B_5	0.076–0.025	75.5– 86.5*

Note: Dependent variable, track width.
* No significant difference at the 95% level.

The speeds were ranked with respect to constant slider design for the variables track depth and area as described for track width. In each instance, the speeds were significantly different ($P < 0.05$) only for slider designs B_3 and B_4. Typical values of track depth and area under these conditions were 22 µm and 100×10^{-6}cm^2, respectively.

The effect of load on track width was examined at five levels (A_1 through A_5) for five slider designs. In conjunction with this, the critical track width† was calculated for each slider design (Table 4). The 500 gm load represented a different plowing situation from the other loads because the mean track widths for slider designs B_3 through B_5 exceeded the corresponding critical track widths of the 500 gm load. Therefore, values for the 500 gm load were treated separately from the other values in the statistical analysis of the data.

The main effect of load on track width was significant ($F = 186 > F_{(3,60)} = 6.17$) at the 0.001 level. In general, lower loads resulted in lower values of track width over the range studied, including the 500 gm load. The first order interaction also was significant at the 0.001 level, which indicated that load and slider design were not independent. The coefficient of variation for this data was 8.1%.

The loads were ranked with respect to constant slider design as shown in Table 5. The values for the 500 gm load are included for comparison, but were not ranked statistically. With several exceptions, the loads were significantly different ($P > 0.05$) within a given slider design. Log-log plots of mean track width vs load were straight lines for the designs studied, although the values for the 500 gm load were larger than would have been anticipated by this relationship for slider designs B_3 through B_5. Log-log plots of mean track width vs load for slider designs B_1 and B_4 are shown in Figure 2. The location of the critical track width of slider design B_4 corresponded to a critical load of about 250 gm. The slopes of these curves for designs B_1 and

† Critical track width is defined as that width below which only the hemispherical part of a slider contributes to sliding, and above which both the hemispherical and conicial parts of a slider contribute to sliding.

TABLE 4
CRITICAL TRACK WIDTHS

Slider Design	Critical Track Width (μm)
B_1	282.0
B_2	1,010.0
B_3	78.3
B_4	48.5
B_5	40.4

B_4 were 0.445 and 0.640, respectively. The values of track width for the 50 and 100 gm loads were reversed in order for design B_2.

The main effect of load on track depth was significant ($F = 420 > F_{(3,60)} = 6.17$) at the 0.001 level. In general, higher loads resulted in higher values of track depth over the range studied, including the 500 gm load. With two exceptions, the mean track depth values were 5 to 1,000 times the mean surface roughness value. The mean effect of load on area also was significant ($F = 89.9 > F_{(3,60)} = 6.17$) at the 0.001 level. Higher loads resulted in higher values of area over the range of loads studied. The coefficients of variation for

TABLE 5
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR LOAD DIFFERENCES

Slider Design	Ranking of Means (μm)				
	A_1	A_2	A_3	A_4	A_5
B_1	8.85	16.9	19.2	24.5	49.3
B_2	13.6	22.0	45.0	34.6	56.4
B_3	8.18	10.1	17.2	33.8	107
B_4	6.58	11.7	20.0	28.4	106
B_5	7.16	9.23	22.8	28.0	86.5

Note: Dependent variable, track width. Underscore indicates no significant difference at the 95% level.

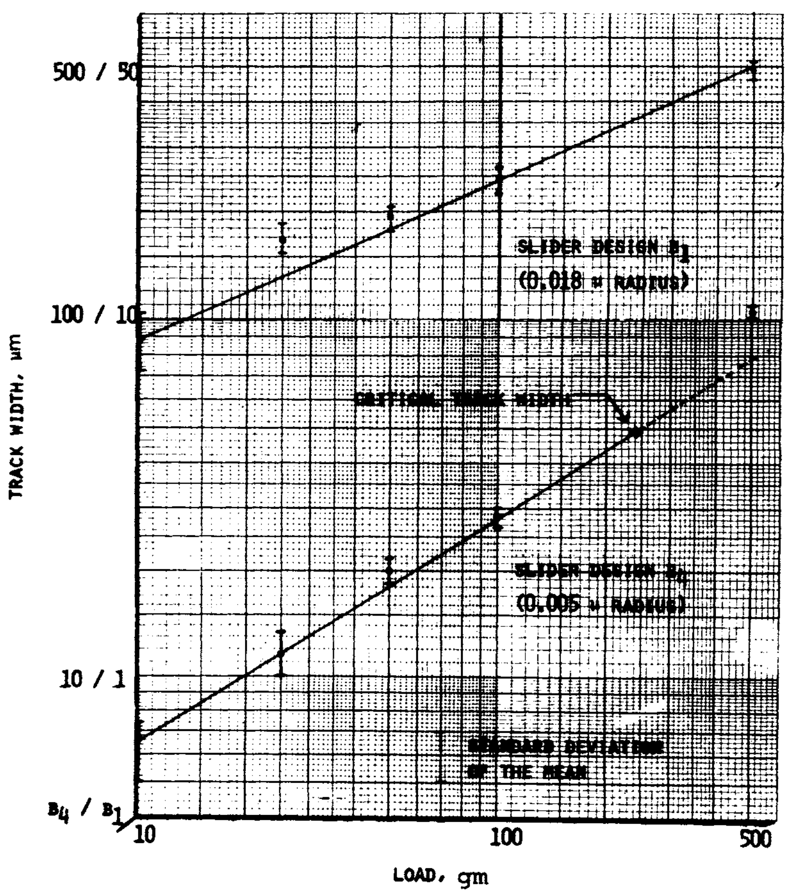

FIG 2.—Relationship between track width and load for two slider designs ($\mu = \mu$m).

these data were 11 and 19%, respectively. In each instance the first order interaction between load and slider design was significant at the 0.001 level.

The loads were ranked with respect to constant slider design for the variables track depth and area (Tables 6, 7). The values for the 500 gm load are included for comparison, but were not ranked statistically. In both instances, values for the 500 gm load were an order of magnitude larger than values for the 100 gm load for designs B_3 through B_5.

The effect of slider design on track width was examined at five levels (B_1 through B_5) for five loads. The main effect of slider design was significant ($F = 44.8 > F_{(4,60)} = 5.31$) at the 0.001 level. Slider designs with smaller radiuses resulted in lower values of track width with the designs studied. The coefficients of variation for this and the following data are identical to those reported for load.

The designs were ranked with respect to constant load, as shown in Table 8. The values for the 500 gm load are included for comparison and were ranked based on the data presented in Table 3. There appeared to be no significant difference ($P < 0.05$) among the means of slider designs B_3 through B_5, with the exception of the 500 gm load values. At loads below 100 gm, these designs resulted in the lowest track widths. At the 500 gm load, however, those slider designs with large radiuses (B_1 and B_2) resulted in low values of track width and were not significantly different ($P < 0.05$) from each other.

The main effect of slider design on track depth was significant ($F = 116 > F_{(4,60)} = 5.31$) at the 0.001 level. The main effect of slider design on area also was significant ($F = 27.5 > F_{(4,60)} = 5.31$) at the 0.001 level. Ranking of slider designs with respect to constant load for the variables track depth and area is shown in Tables 9 and 10, respectively. Significant differences among means could be observed only for the higher loads in each instance. In contrast with track width values, the higher track depth values were observed with slider designs B_3 through B_5. Higher area values were observed with slider designs B_3 through B_5 for the 500 gm load.

TABLE 6
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR LOAD DIFFERENCES

Slider Design	Ranking of Means (μm)				
	A_1	A_2	A_3	A_4	A_5
B_1	0.06	0.22	0.26	0.43	1.7
B_2	0.04	0.10	0.41	0.25	0.64
B_3	0.16	0.20	0.58	2.3	25
B_4	0.11	0.34	1.0	2.1	22
B_5	0.12	0.17	1.0	1.5	11

Note: Dependent variable, track depth. Underscore indicates no significant difference at the 95% level.

TABLE 7
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR LOAD DIFFERENCES

Slider Design	Ranking of Means (10^{-6} cm²)				
	A_1	A_2	A_3	A_4	A_5
B_1	0.33	1.2	1.5	2.4	9.7
B_2	0.74	2.1	8.1	5.0	13
B_3	0.31	0.39	1.2	4.6	120
B_4	0.17	0.55	1.6	3.4	160
B_5	0.24	0.34	2.1	3.0	100

Note: Dependent variable, area. Underscore indicates no significant difference at the 95% level.

Discussion

The discrepancies noted during comparison of the natural apatite with the synthetic product indicate the presence of impurities in the natural crystal. Although it would be improper to assume that single crystals of natural and synthetic apatite would yield the same data under similar experimental conditions, it is probable that the trends observed would be similar. It is not known whether the behavior of F single crystals is indicative of the behavior of enamel under similar conditions.

The purpose of the polishing and surface treatment procedures was to standardize the crystals. However, different levels of surface roughness[4] and different surface treatments[6] might be expected to produce different results. Buckley,[6] for example, has shown that the number and depth of surface and subsurface cleavage cracks for single crystals of calcium fluoride were sensitive to the environmental species present as well as to the load.

A basic assumption underlying the use of analysis of variance and multiple comparisons is that all sample means have statistically equal variances.[14] When this assumption is not met, the power of these statistical tools is lessened, and true differences among means may not be observed.

TABLE 8
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR SLIDER DESIGN DIFFERENCES

Load		Ranking (μm)				
A_1	Slider design	B_4	B_5	B_3	B_1	B_2
	Mean	6.58	7.16	8.18	8.85	13.6
A_2	Slider design	B_5	B_3	B_4	B_1	B_2
	Mean	9.23	10.1	11.7	16.9	22.0
A_3	Slider design	B_3	B_1	B_4	B_5	B_2
	Mean	17.2	19.2	20.0	22.8	45.0
A_5	Slider design	B_1	B_5	B_4	B_3	B_2
	Mean	24.5	28.0	28.4	33.8	34.6
A_5	Slider design	B_1	B_2	B_5	B_4	B_3
	Mean	49.3	56.4	86.5	106	107

Note: Dependent variable, track width. Underscore indicates no significant difference at the 95% level.

The assumption of equal variances was violated in several instances where values ranged over several orders of magnitude. These statistical tools represent a convenient means of data analysis and impose a conservative restriction on interpretation of the results.

As stated earlier, it was assumed that the effect of crystallographic direction on failure properties was of less importance than other factors. The low F values found in comparisons of two crystals with three replications (of arbitrary sliding direction) imply that this assumption was valid for the variables examined. Yet, anisotropic effects have been observed by several investigators for a number of crystals,[5,7,8] including the hexagonal crystal sapphire.[15]

A strain rate effect on the variables track width, track depth, and area was observed under conditions of a 500 gm load for slider designs B_3 and B_4. Under these conditions, the wear scar was influenced by the conical and hemispherical sections of the sliders, and much deeper penetration occurred than with other designs. For designs B_3 and B_4, the observed track widths were twice that of the respective critical track widths. No strain rate effect was observed for designs and loads in which the observed track widths were less than the critical track widths. Buckley[6] observed that the amount of track deformation of calcium fluoride decreases with increased sliding speed.

All three variables were sensitive to load and slider design. The track width was related to the load by an equation of the form $L = \psi w^n$, where L and w are the load and width, respectively, for slider designs B_1 and B_4. Such a relationship has been discussed by Steijn[4] for single crystals of NaCl, LiF, and MgO. Values of n reported for these materials were 2.27, 2.33,

TABLE 9
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR SLIDER DESIGN DIFFERENCES

Load		Ranking (μm)				
A_1	Slider design	B_2	B_1	B_4	B_5	B_3
	Mean	0.04	0.06	0.11	0.12	0.16
A_2	Slider design	B_2	B_5	B_3	B_1	B_4
	Mean	0.10	0.17	0.20	0.22	0.34
A_3	Slider design	B_1	B_2	B_3	B_4	B_5
	Mean	0.26	0.41	0.58	1.0	1.0
A_4	Slider design	B_2	B_1	B_5	B_4	B_3
	Mean	0.25	0.43	1.5	2.1	2.3
A_5	Slider design	B_2	B_1	B_5	B_4	B_3
	Mean	0.64	1.7	11	22	25

Note: Dependent variable, track depth. Underscore indicates no significant difference at the 95% level.

TABLE 10
RESULTS OF DUNCAN'S NEW MULTIPLE RANGE TEST FOR SLIDER DESIGN DIFFERENCES

Load		Ranking (10^{-6} cm^2)				
A_1	Slider design	B_4	B_5	B_3	B_1	B_2
	Mean	0.17	0.24	0.31	0.33	0.74
A_2	Slider design	B_5	B_3	B_4	B_1	B_2
	Mean	0.34	0.39	0.55	1.2	2.1
A_3	Slider design	B_3	B_1	B_4	B_5	B_2
	Mean	1.2	1.5	1.6	2.1	8.1
A_4	Slider design	B_1	B_5	B_4	B_3	B_2
	Mean	2.4	3.0	3.4	4.6	5.0
A_5	Slider design	B_1	B_2	B_5	B_3	B_4
	Mean	9.7	13	100	120	160

Note: Dependent variable, area. Underscore indicates no significant difference at the 95% level.

and 2.45, respectively. The values of n for designs B_1 and B_4 on single crystals of F were 2.25 and 1.56, respectively, as calculated from the reciprocal of the slopes in Figure 2. Although not shown in Figure 2, the data for slider designs B_3 and B_5 were approximated well by the relationship for slider design B_4. Such curves may be characteristic of the hemispherical contribution of the sliders up to a point determined by the critical track width and apical angle of the design. A slider design with a sharp apical angle (eg, B_4) can be expected to deviate from this linear log-log relationship more dramatically than one with a blunter angle (eg, B_5) at loads above their critical load for designs of similar radiuses.

Penetration caused by sliding was relatively low below the critical track width for all slider designs, but above this value, there was a considerable increase in penetration for the sharper angles. However, below the critical track width, those sliders with smaller radiuses resulted in larger penetration at a given load. These results are similar to those observed by Bowden and Brookes[8] in their study of MgO single crystals, and by Steijn[4] in his study of copper.

Values for track depth and area were not as effective as track width in ranking the data because of the propagation of error in their respective calculations. In spite of this, both were valuable in explanations of the observed behavior, and should not be discarded. Values obtained for slider design B_2 were questionable in several instances, and this was thought to be the result of the effect of first order asperities on the large diameter slider.

Conclusions

A quantitative method for characterizing the surface failure of nonmetallic single crystals under sliding was evaluated by an investigation of the wear of natural fluorapatite single crystals. The method consisted of sliding a diamond of specific design on the basal surface of natural fluorapatite single crystal in a dry environment, and required apparatus characterized as follows: surface grinder, loading jig, friction transducer, diamond slider, and sample holder.

The variables track width, track depth, and area were effective in ranking data obtained under varying conditions of slider speed, load, and slider design. Although less accurate because of propagation of error, the variables track depth and area were valuable in explaining observed behavior.

A strain rate effect was observed at a 500 gm load for slider designs with small radiuses and sharp apical angles. In these designs a lower sliding speed resulted in increased surface damage.

Track width was related to the load by a linear log-log relationship under those conditions of load and design where the critical track width had not been exceeded. Above this critical value, track width was related to the sharpness of the apical angle of the diamond.

Penetration was favored by slider designs of small radiuses, and for the same slider, by higher loads.

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