Measurement and Analysis of Diamond Retention in CMP Diamond Pad Conditioners

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Introduction

This report investigates diamond retention characteristics of various CMP diamond pad conditioners. Diamond pad conditioners from 3M, *Vendor A* (a brazed diamond conditioner), *Vendor B* (an electroplated diamond conditioner), and *Vendor C* (a non-metallic CVD coated diamond conditioner) are compared using various tests that investigate the strength and durability of the bond. In Section I, the bond of the diamond to the substrate is evaluated using a high-pressure waterjet wash test. This "rapid aging" test demonstrates the failure mechanism of the diamond bond; it also quantifies the effective bond strength of the various conditioners. Section II utilizes a "pick" test to quantify the force necessary for release of diamond from the bond matrix of the 3M and *Vendor A* constructions.

I. High Pressure Waterjet Wash Test

1.1 Objective

To systematically evaluate the diamond bond strength and diamond fracture toughness of a variety of commercially available CMP diamond pad conditioners. This test applies tremendous force on the surface of the metal bond and diamond facets, thereby dislodging any loosely bonded metal or diamonds. Weak diamonds or diamonds that have been weakened by the manufacturing process will also fracture under the forces applied to them by the water stream. The number of diamond crystals removed is then quantified for comparison of the different type of bonding techniques. The manner of diamond loss is also qualitatively evaluated (i.e. diamond fracture, diamond bond issues).

1.2 Test Apparatus

The test apparatus consists of a high pressure washing head mounted to a Romeo Engineering Inc., (Fort Worth, TX, model AWJGU2) waterjet cutting and cleaning device. Water is delivered to the washing head via a high-pressure pump. The 45,000-psi supply water is split into four streams and passes through 6 mil and 9 mil orifices in the configuration shown in Figure1 (bottom view). The washing head (Jet Edge Inc. Mpls, MN P/N A3126) is rotated by a pneumatic motor at 1800 rpm, thereby producing a circular wash pattern that will contact the entire surface of the part as it is passed over it.

1.3 Test Procedure

The diamond abrasive sample is fixed in the waterjet tank. The head is then passed over the diamond abrasive sample at a rate of 25 IPM at a height of 3 inches. SEM micrographs are then taken of the washed and unwashed samples at 20X and 100X at a 60° angle.
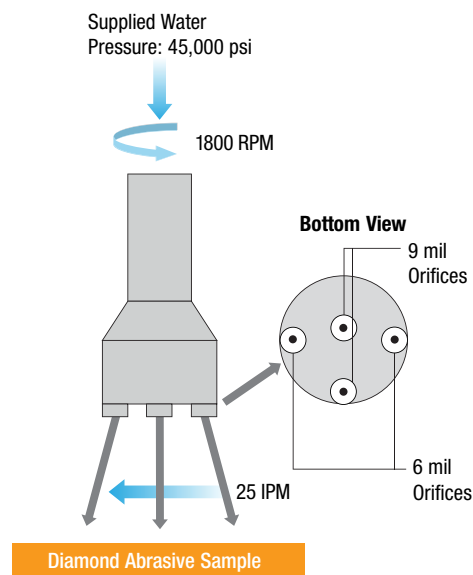

Figure 1. Washing apparatus schematic

1.4 Test Results

A number of diamond pad conditioners were subjected to the conditions described in the test procedure, including but not limited to 3M sintered material, *Vendor A, Vendor B,* and *Vendor C*. Summary results are included for each type of conditioner.

1.4.1 Vendor A

The product of brazing technology is shown in the pretest state in Figures 2 and 3. The diamond particles form a random/clustered pattern. The diamonds fall in this pattern due to the surface tension of the braze alloy used to bond the diamonds to the substrate as well as the random placement of the diamonds prior to brazing.

SEM micrographs of the waterjet washed *Vendor A* sample (Figures 4 and 5) shows the effect of the high pressure waterjet washing on the *Vendor A* diamond to metal bond strength as well as the overall diamond strength. Large islands of diamond and metal bond were removed with the wash test. In some cases, partial diamond fragments were still attached to the substrate (Figure 5).
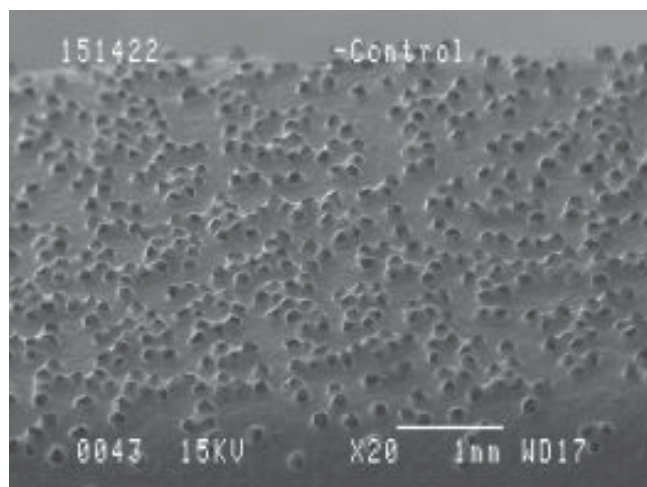

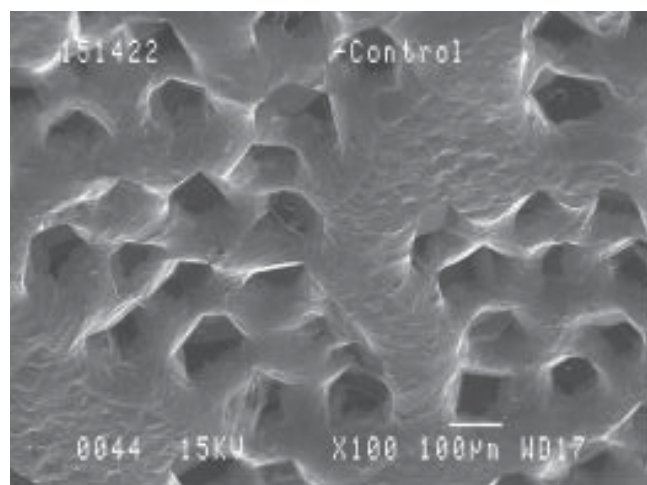Figure 2. *Vendor A* – Unwashed (20X)
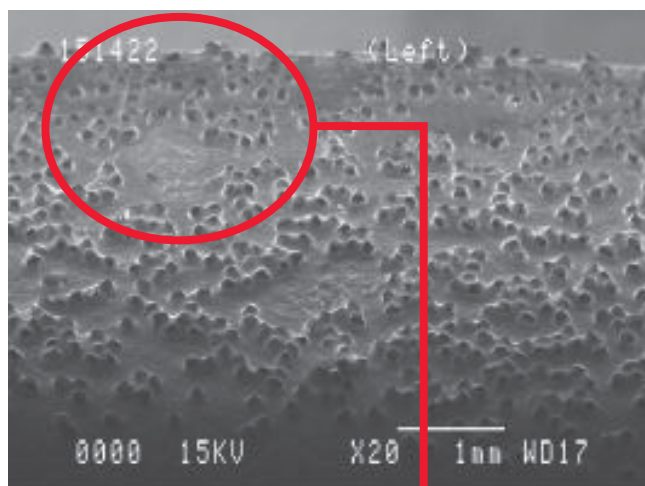

Figure 3. *Vendor A* – Unwashed (100X)

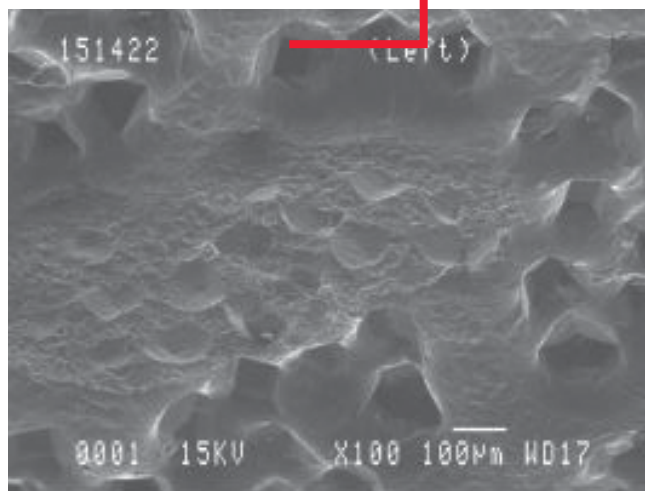Figure 4. *Vendor A* – Washed (100X)

Figure 5. *Vendor A* – Washed (20X)

1.4.2 3M Sintered Diamond (STD)

The 3M sintered diamond material utilizes patented techniques for controlled placement of diamonds in a corrosion resistant sintered metal bond matrix. The unwashed 3M STD sample is shown in Figures 6 and 7. SEM micrographs of the 3M conditioning disk-washed sample (Figures 8 and 9) shows minimal loss of diamond. In this test, scattered individual diamonds are removed from the metal bond matrix. In the typical region depicted in Figure 8, only 1 diamond out of 189 was removed over an area of about 26 mm^2.
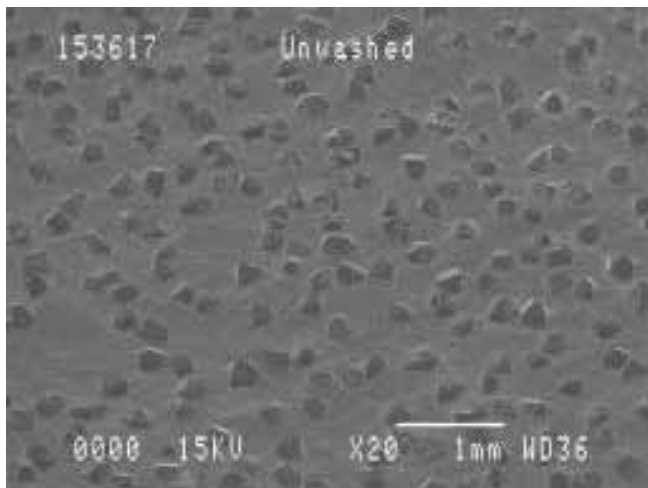
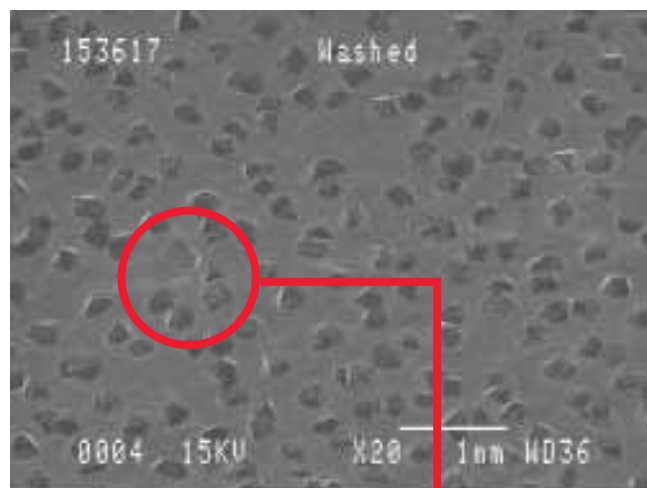Figure 6. *3M Std.* Unwashed (20X)

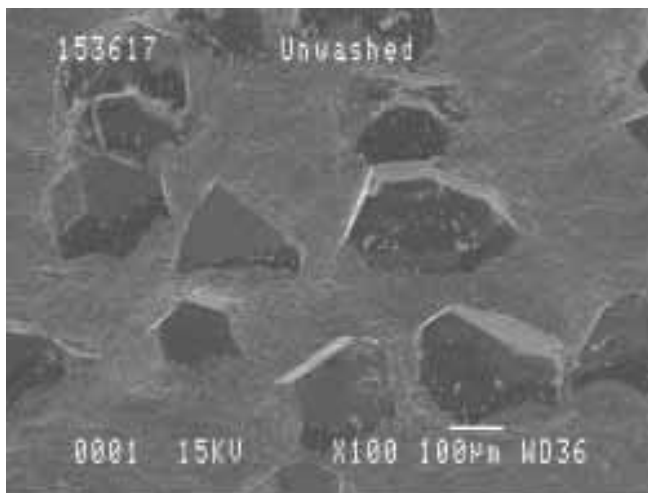Figure 8. *3M Std*. – Washed (20X)

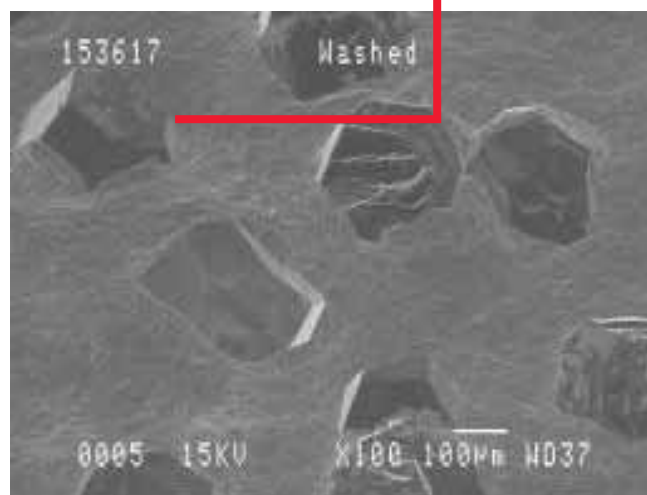Figure 7. *3M Std*. – Unwashed (100X)

Figure 9. *3M Std*. – Washed (100X)

1.4.3 Vendor B

The *Vendor B* diamond pad conditioner is shown in its unwashed or pretest state in Figures 10 and 11. These disks utilize standard plating technologies on a perforated metal substrate. This technique randomly drops diamonds onto the substrate and mechanically holds them in place with an electroplated nickel bond.

The effect of the high-pressure waterjet wash can be seen in Figures 12 and 13. The result of this test shows that a large number of diamonds have been removed from the disk,

either by diamond fracture and/or poor diamond adhesion to the substrate. Due to the large amount of diamond loss, it is very difficult to get an exact count from the SEM micrograph (Figure 12). However, Figure 13 was used to get a more accurate count of diamond loss, then the area was taken into account to get an approximate number (approximately 125). The amount of diamond loss would be even larger if the area of the perforations was included.
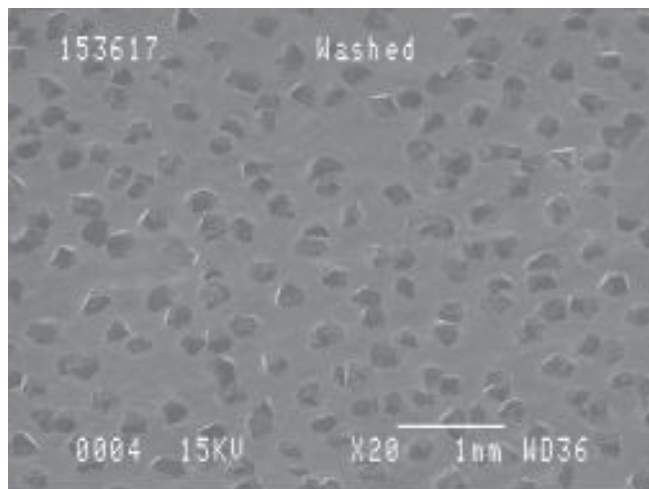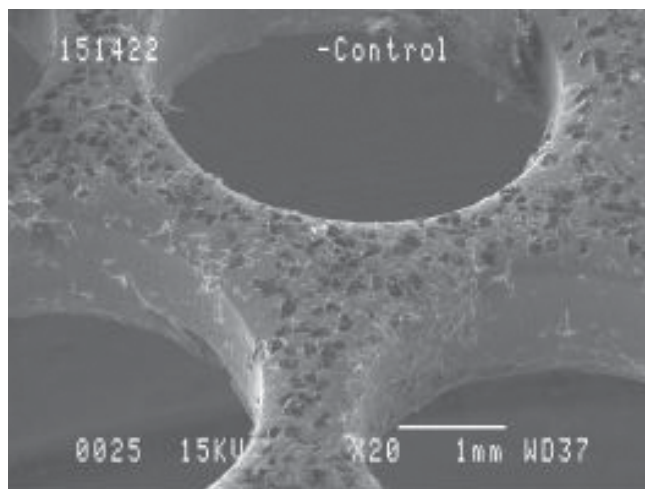
Figure 8. *3M Std.* – Washed (20X)
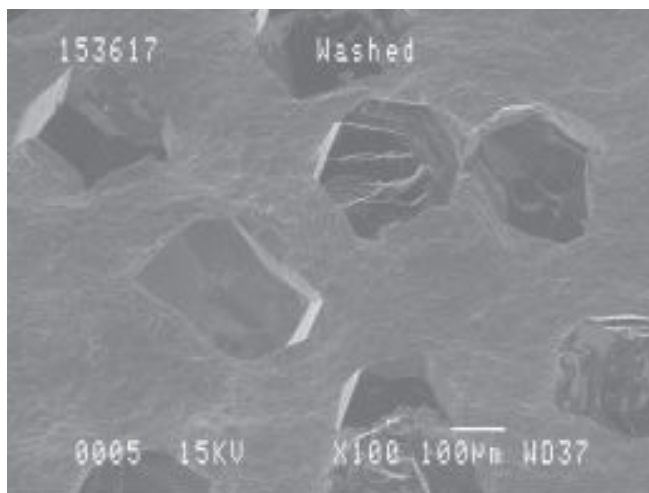

Figure 10. V*endor B* – Unwashed (20X)

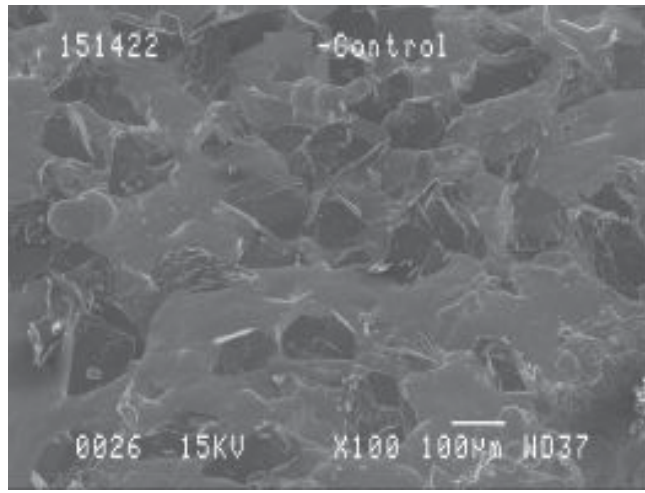Figure 9. *3M Std.* – Washed (100X)

Figure 11. *Vendor B* – Unwashed (100X)

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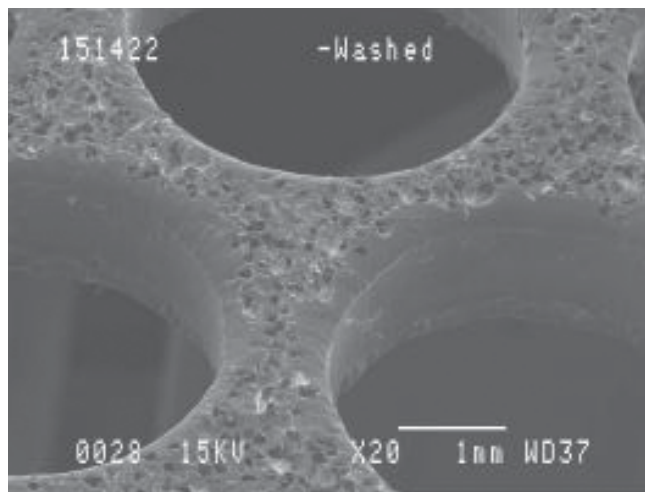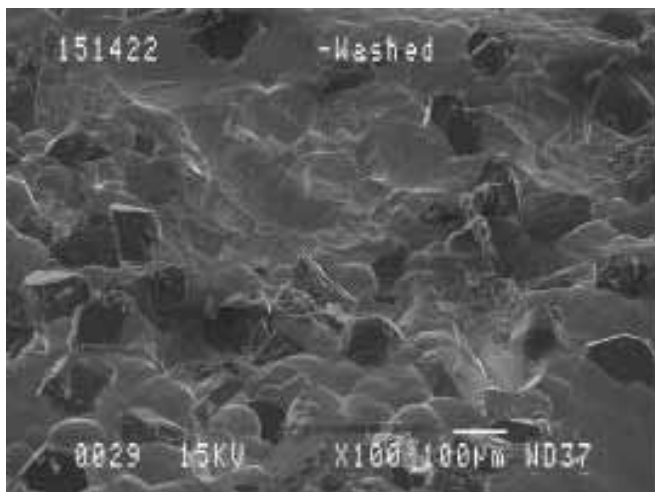

Figure 12. *Vendor B* – Washed (20X)

Figure 13. *Vendor B* – Washed (100X)

1.4.4 Vendor C

A photo of the *Vendor C* disk is shown in figure 14. This photo shows the complete failure of the CVD diamond bond to the silicon wafer. This vendor utilizes a CVD diamond coating process to bond diamond particles (50 micron) to a silicon wafer substrate. The high pressure waterjet stream was strong enough to cause brittle failure of the CVD coating at the CVD-Silicon interface. In the summary of the diamond loss, *Vendor C* will be listed as 200 diamonds removed (full scale on the graph for complete diamond loss).
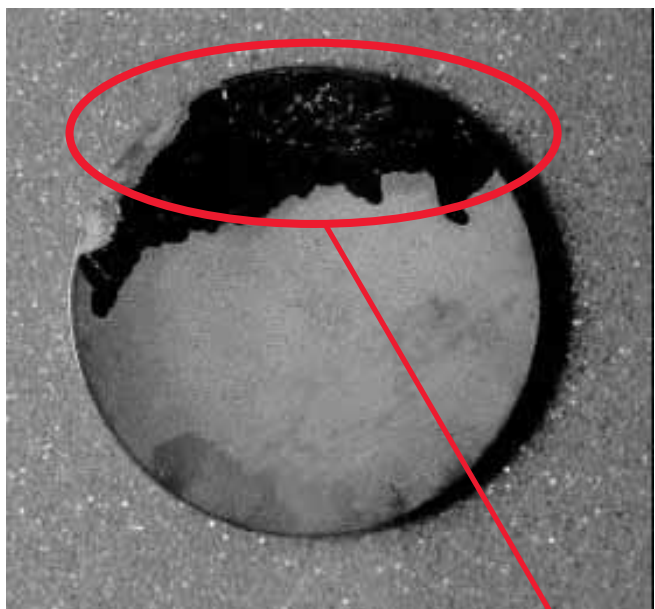

Figure 14. *Vendor C* – Washed   Washed area

1.5 Conclusion

A quantitative result from the high-pressure Waterjet Wash test is shown in Figure 15. This graph shows the number of diamonds removed from the field of view in the Washed 20X SEM photos. Note: *Vendor C* is listed as 200, as previously noted.

Under these very extreme conditions, the 3M sintered diamond material has an order of magnitude lower susceptibility to diamond loss than the next best product, *Vendor A*.
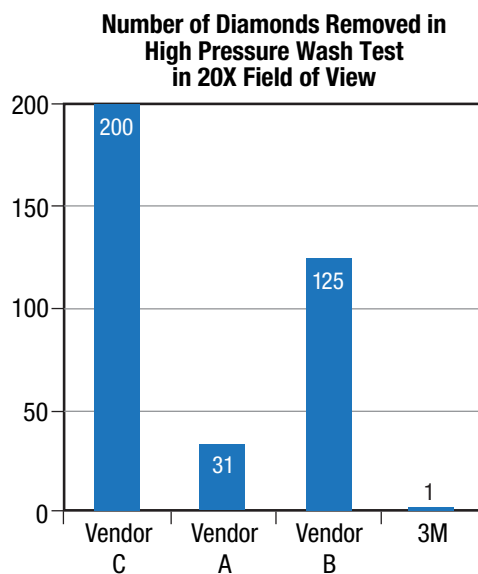

Figure 15. Diamond loss comparison for various diamond pad conditioners

II. Mechanical Test for Diamond Retention

2.1 Objective

To measure and compare the force necessary to remove a diamond particle from the metal bond matrix of various CMP diamond conditioners.

2.2 Test Apparatus

The test apparatus consists of a hardened steel pick that is attached to a digital force gage (Shimpo model FGV-50A). A schematic of the apparatus is pictured in Figure 15. In order to place the hardened steel pick on a diamond, it is necessary to do this alignment under the microscope (typically 10-40X magnification).

2.3 Test Procedure

The diamond pad conditioner that is being tested is clamped to the work surface. The hardened steel pick is place on a single diamond while viewing the placement through the microscope. Force is then applied to the diamond at a 45° nominal angle. The force to dislodge or crush the diamond is then recorded from the force gage.
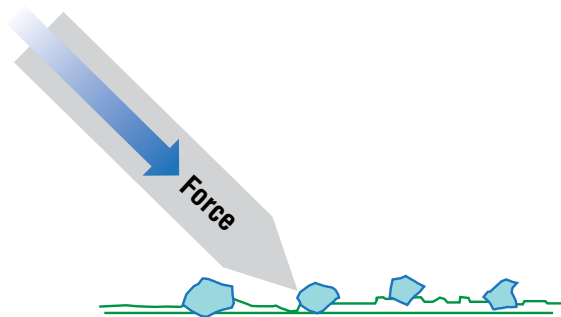
Figure 16. Schematic of Diamond Pick Experimental Procedure

2.4 Test Results

The break strengths measured with the pick held by hand at about 45° are given below in Table 2.1. In tests of this kind, it is particularly useful to use Weibull statistics in analyzing the results. This approach is generally most applicable if the failure strengths being represented by this distribution function are essentially determined by a population of flaws, in this case in the diamonds or at the diamond-metal interface. Examples of "flaws" might include inclusions within diamonds, interface defects, or even unusually small areas of contact between diamond and matrix.

Test	3M	Vendor A	Test	3M	Vendor A
1	19.58	10.79	16	27.75	8.16
2	38.5	7.53	17	53.56	8.5
3	20.48	14.59	18	40.54	10.45
4	16.56	5.73	19	37.75	10.86
5	27.98	6.85	20	28.22	8.8
6	18.31	10.82	21	24.5	
7	51.24	5.67	22	37.46	
8	54.39	7.57	23	35.79	
9	48.84	11.73	24	52.11	
10	41.02	6.43	25	25.68	
11	9.71	8.57	26	30.13	
12	60.88	4.77	27	57.36	
13	51.66	12.76	28	20.85	
14	28.67	10.88	29	11.85	
15	50.9	13.07	30	33.5	

Table 2.1. Raw Data from Pick Tests (Values are applied force, in pounds).

The analysis involves plotting the data on a Weibull probability chart, in which the cumulative probability of failure is plotted against the break strengths.[1]

[1] To do this, an estimate of the probability of failure based on the measured break strengths is needed. Such estimates are done automatically in many statistical software packages. One method is to rank the break strengths in ascending order, and then estimate the cumulative probability with the equation pi = (i–0.3)/(n+0.4), where i is the rank and n is the total number of measurements. Example: For the 8th sample out of 20 samples, p= 37.7%. For more information, see *Fracture Mechanics of Ceramics*, Vol. 3, R. C. Brandt, 1978, pg. 9-13.

Weibull Probability Plot
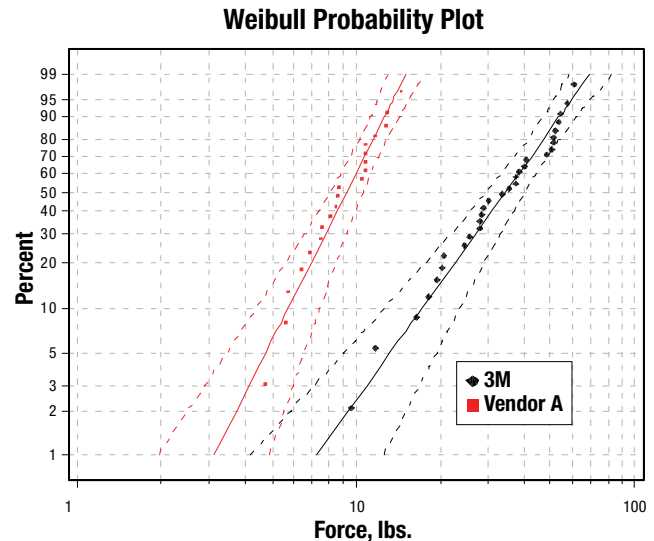
2.5 Conclusion

From this graph one can see that at the force needed to break 50% of the *Vendor A* diamond bonds, only about 2% would fail in the 3M product. The 3M disk exhibits an average break force of 35.2 lbs., approximately 3.8 times the corresponding strength of the *Vendor A* product (average break force of 9.2 lbs.). Because of the high strength of the diamond-metal interfaces in the 3M product, only a small fraction of the measured failures (about 13%) actually resulted from diamonds popping out under the applied stress. More often, the test ended with diamond fracture. This change of failure mode should not be surprising given that all but 2 of the individual diamond bonds tested on the 3M product withstood higher forces than any of the individual tests of the *Vendor A* product.

IV. Summary

The results of the high-pressure waterjet wash test have shown that the 3M Diamond Pad Conditioner and the *Vendor A* (brazed) product were superior to the *Vendor B* (electroplated) and *Vendor C* (CVD diamond bonded) products for retention of diamond to the pad conditioner substrate material. The 3M pad conditioner and the *Vendor A* pad conditioner were then subjected to the Diamond Pick Test (section II.). The pick test clearly demonstrated that the 3M pad conditioner had superior diamond adhesion characteristics as compared to the *Vendor A* product.

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