Test Results

Thermal Spray Coating Test Results - Kermetico

Center: Stonehouse Technology Center (SHTC)

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Responsible Eng.: B. Madsen

1. Device Under Test (DUT)

Several different Tungsten Carbide (WC) HVOF/HVAF sprayed coatings from different coating vendors were tested to determine the:

- Vickers micro-hardness
- Erosion rate of the coating under a slurry jet testing environment.
- Micro-structure of the coating under a SEM.

The vendors and coating types to be tested are:

TABLE 1 - COATING VENDORS TESTED

Supplier	Chemical Composition	Coating Process	Supplier Coating Definition
Supplier A	86%WC, 10%Co, 4% Cr	HVOF	XXXX
Kermetico	86%WC, 10%Co, 4% Cr	HVAF	WC104A
Supplier C	86%WC, 10%Co, 4% Cr	Special Process	XXXX
Supplier D	86%WC, 10%Co, 4% Cr	HVOF	XXXX
Supplier E	86%WC, 10%Co, 4% Cr	HVOF	XXXX
Supplier F	3-9% Co, WC Balance	HVOF	XXXX

**Same coating can be applied from other global locations*

Each coating type has been sprayed onto circular and square test coupons with the dimensions and specifications defined in section 1.1 and 1.2.

Test Results

1.1 Tungsten Carbide (WC) Coated Circular Test Sample Coupon

- Uncoated Coupon Dimensions: Ø1 in. (Ø 25.4mm), 0.25 in. (6.35mm) thick – refer to Figure 1
- Substrate material: 410-13 Cr Stainless steel (SST), 80 KSI Yield Strength
- Coating Material and Type: Tungsten Carbide Coating (WC) – Process and composition varies according to supplier.
- Coating Thickness: 0.016 ± 0.003in. (400 μm) before grinding (grinding only required for hardness tests).

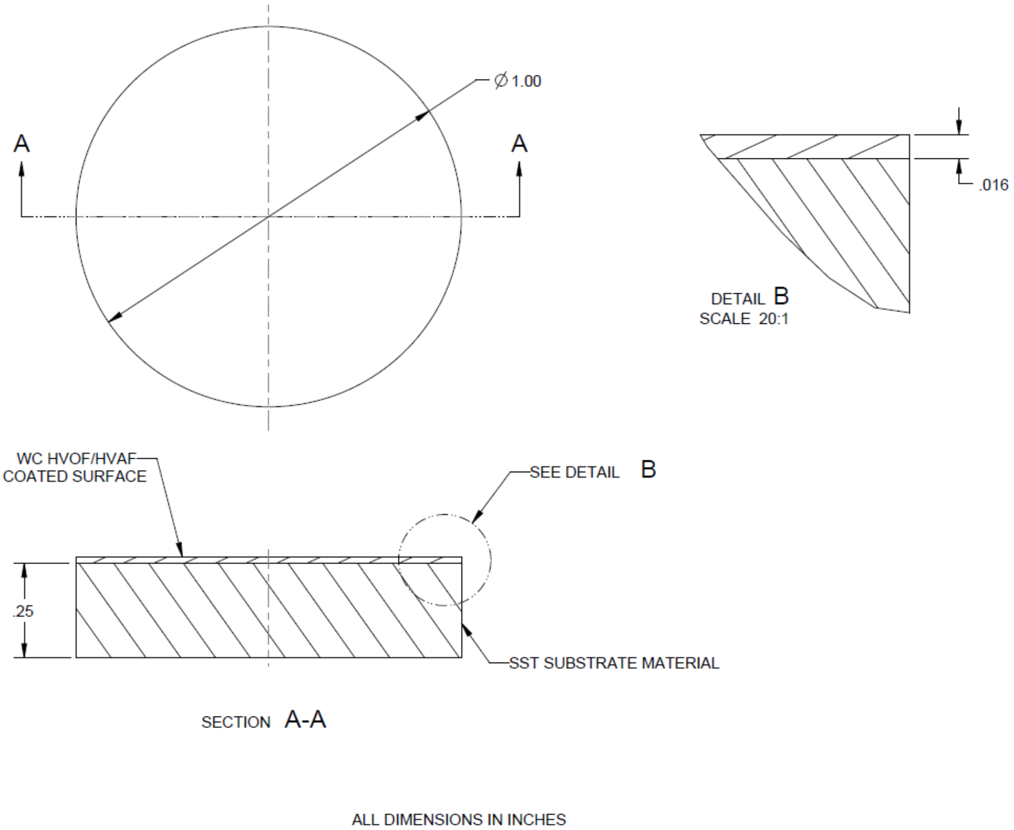


FIGURE 1 - CIRCULAR TEST COUPON DIMENSIONS

Test Results

1.2 Tungsten Carbide (WC) Coated Square Test Sample Coupon

- Uncoated Coupon Dimensions: 1.97in. X 1.97in. X 0.197in (50mm X 50mm X 5 mm) – refer to Figure 1
- Substrate material: 410-13 Cr Stainless steel (SST), 80 KSI Yield Strength
- Coating Material and Type: Tungsten Carbide Coating (WC) – Process and composition varies according to supplier.
- Coating Thickness: 0.016± 0.003 in. (400 µm) - Coating should be left in as sprayed condition (no grinding or polishing required).

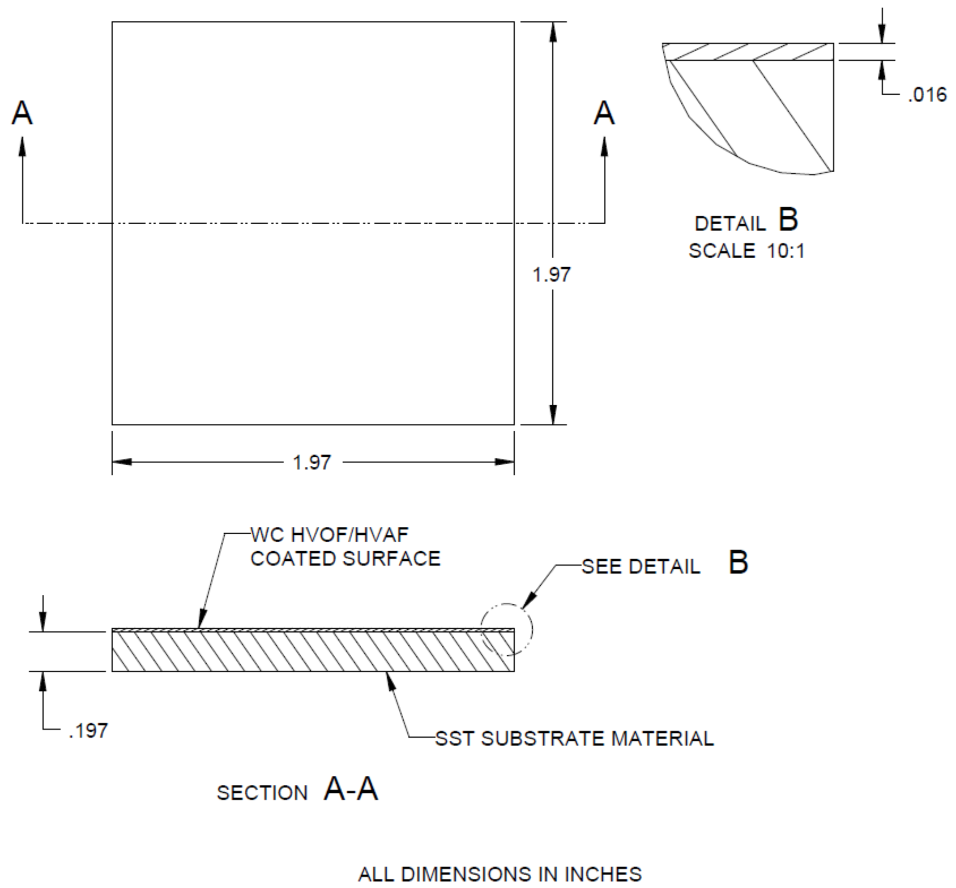


FIGURE 2 - SQUARE TEST COUPON DIMENSIONS

2. Test Objectives

2.1 Vickers Micro-hardness Tests

The purpose of this test is to determine the Vickers Micro-hardness number (HV) of several different Tungsten Carbide HVOF/HVAF coatings supplied by different coating vendors.

This test will be conducted in accordance to ASTM E384, which is the industry standard method for testing the Vickers micro-hardness of materials. The test is conducted by pressing a Vickers diamond indenter into the coated surface of the test sample at a known force, in this case 300gf (2.9421N), to form an indentation. Once the load is removed, the indentation diagonals are measured with a microscope, and the surface area of the indentation is calculated. For this test, it is assumed that the indentation does not undergo elastic recovery after the force is removed. The Vickers hardness number is then obtained by dividing the force applied through the Vickers indenter by the surface area of the indentation made by the indenter.

Each coating sample will have a minimum of five indentation made to it to establish the mean Vickers micro-hardness for each coating type. The standard deviation of the hardness values, for each coating type, will also be determined to assess the consistency of the hardness measurements.

The hardness measurements will be taken from the top surface of the test sample. This will provide more information as to how well the area exposed to fluid flow will perform under erosive conditions (i.e. bombarding particles and fluid flow).

2.2 Slurry Jet Erosion Tests

The purpose of this test is to determine the erosion rate of several different Tungsten Carbide HVOF/HVAF coatings supplied by different coating vendors.

This test is conducted using a slurry jet flow loop test rig, whereby a mixture of water and sand (CH-50 Silicate sand, 50 µm grain size) is jetted at a velocity of 24 m/s, at a particular impingement angle onto the coated surface of a test sample. The flow loop system is a closed-loop system meaning that the same fluid in continuously circulated and the sand to water ratio remains constant (approx. 2% sand content). The samples are weighed before and after testing, the recorded weight loss is then divided by the pumping time to determine the erosion rate of the coating. It is essential that the test sample is not eroded into the substrate in order to calculate the erosion rate of the coating alone.

The first set of flow tests will be carried out to determine the worst case impingement angle, after which all other tests will then be conducted using this worst case angle. The angles that will be tested are 20°, 60° and 90°, whereby one test and a potential re-test will be conducted for each angle.

Two tests will then be carried out for each coating type at the worst case impingement angle and a mean erosion rate will be calculated for each coating type. The erosion rates of the different coatings will be compared to find out which coating is the most erosion resistant under these particular flow test conditions.

2.3 **Micro-Structure Analysis**

The purpose of this test is to analyze the coating micro-structure of several different Tungsten Carbide HVOF/HVAF coatings, supplied by different coating vendors.

The test samples will first be cut in half and prepared so that the sectioned area is smooth and free of burrs. A Scanning Electron Microscope (SEM) will be used to determine the porosity, grain size and to identify the presence of any cracks or voids across the sectioned area of the coating.

The size of the 'grains' are measured by the height of the intersplats. The intersplat height of the coating will be determined from polished and etched cross-sections using the linear intercept method in accordance with ASTM E112-10.

The porosity level of the coating will be measured using the SEM's calibrated image analysis software.

3. **Results vs. Objectives**

All coating samples were visually inspected for obvious pits cracks or voids. None of the samples showed any obvious signs of damage. The thickness of the coating samples were measured before and after surface grinding/polishing.

All coated samples that were grinded (for hardness tests) had a coating thickness of 0.016 ± 0.003" (400 µm) before grinding and approximately 0.003" (75 µm) of coating was removed during grinding process, leaving a coating thickness of 0.013 ± 0.003" (330 µm) on samples at the time of testing.

Test Results

3.1 Vickers Micro-hardness Tests

3.1.1 Raw Measurements

Multiple Vickers micro-hardness measurements were taken per indentation. Raw values of the hardness measurements can been seen in Tables 3, 4, 5, 6, 7 & 8 in the Appendix. Measurements of the diagonal lengths measured by the test engineer are denoted by the letter "S", measurements made by the hardness test machine software are denoted by the letter "A" and measurements taken by the senior quality engineer are denoted by "H".

3.1.2 Measurement Quality

The quality of the Vickers hardness measurements primarily depended on the quality of the indentations left on the coated surface. The quality of the individual indentation could be analyzed by assessing the symmetry of the indentation and by comparing the difference between the diagonal lengths of the indentation. If the difference between the two diagonal lengths in an indentation was greater than 5%, then the measurement was discarded and a new indentation was made. This was also done when the indentation was asymmetric or when the end points of the diagonal could not be defined.

The quality of the indentation can be affected by the alignment of the indenter, but can also depend on the properties of the tungsten carbide coating. It was found that coatings which exhibited large pores tended to create poor indentations. Voids within a coating caused large areas of the coated surface to collapse once the indentation load was applied, resulting in less symmetric indents. Additionally, very brittle coatings caused cracks to from at the corners of the indentations, resulting in increased discrepancies between the two diagonal lengths.

Figures 6, 7, 8, 9, 10 and 11 in the appendix show the typical indentation made for each coating type. The quality of the indentations varies depending on the porosity of the coating, which can clearly be seen in the images.

We also tested to see if the Vickers hardness values were being affected by the substrate due to the depth of the indentation. If the indentation depth is larger than 10% of the coating thickness, than the substrate can contribute to lower hardness measurement values (due to the stress wave from the test being large enough to reach the substrate). Similarly, a reduction in the measured hardness level can also be contributed to the decohesion layer (at the coating/substrate interface). For this reason the indentation depth was calculated for each indentation using the mean diagonal length and the face angle of the indenter. It was found that the indentation depth for each indent lay in the region of 1% - 2% of the coating thickness (coating thickness of 330 µm). Therefore it can be confirmed that the substrate or decohesion layer did not have any effect on the measured Vickers hardness values recorded during this testing.

3.1.3 Hardness Results

The mean and standard deviation of the Vickers micro-hardness values were determined for each coating sample (i.e. vendor); these values can be seen on Figure 3.

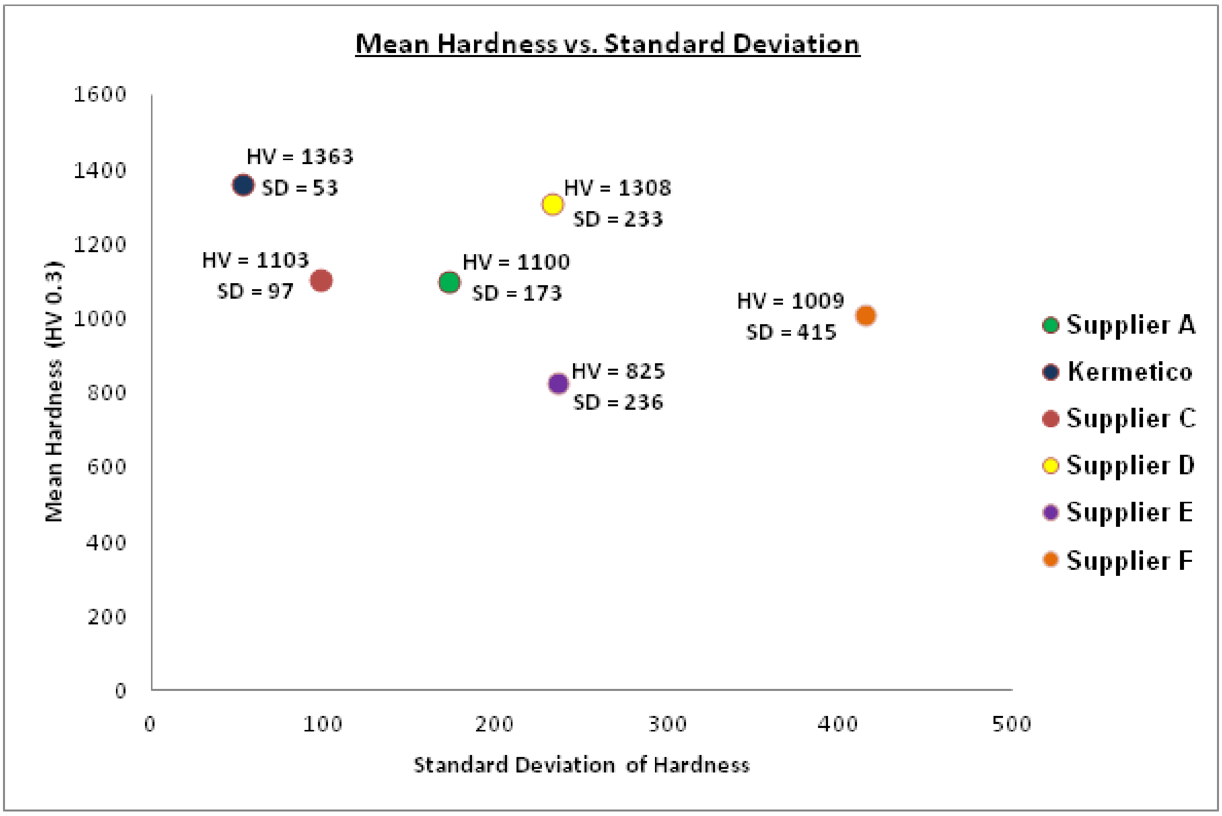


FIGURE 3 - MEAN COATING HARDNESS VALUES AND STANDAD DEVIATION

From Figure 3 it is possible to see that the mean Vickers Micro-hardness varies quite significantly between the different coating types, with mean Vickers hardness values ranging from 825 – 1363 HV 0.3. The standard deviation of the hardness depicts the degree of variation between the measured hardness values. Figure 3 illustrates that for some of the coatings, the standard deviation is very high meaning that measured hardness values vary significantly. Large variations in hardness may be a result of large pores in the coating which may be due to large grain sizes or inclusions in specific areas of the coating.

3.2 Slurry Jet Erosion Tests

3.2.1 Critical Angle Determination

Test samples with Supplier "A" coating were tested at three different impingement angles (20°, 60°, 90°) to determine which angle produced the highest mass loss rate (i.e. erosion rate). Results from the test can be seen in Figure 4 below, for raw values of this data please refer to Table 9 in the Appendix.

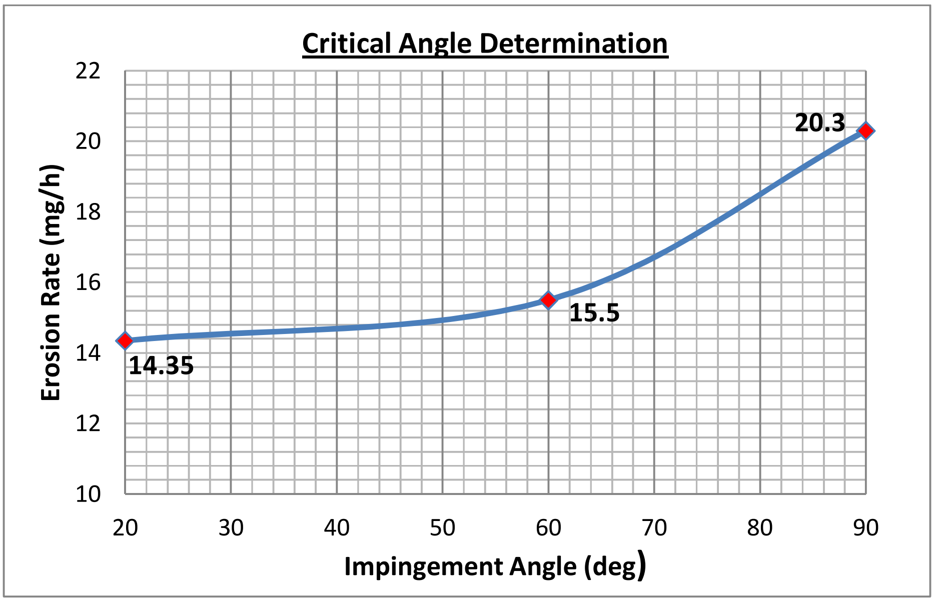


FIGURE 4 - EROSION RATE AT VARIED IMPINGEMENT ANGLES (SUPPLIER "A" COATING)

It can be concluded from Figure 4 that the highest level of erosion (mass loss per hour) occurs at high impingement angles, particularly at angles close to 90 deg.

Due to the similar nature of the different tested coatings (i.e. all Tungsten Carbide thermal spray coatings) it can be assumed that all the coatings will perform the worst at very high impingement angles. Therefore the remaining slurry jet tests will be carried out using an impingement angle of 90 degrees.

3.2.2 Erosion Rate for Different Coating Types

The different coating types were tested under the determined worst case impingement angle (90°) and a repeat test was performed for each coating type. Visual examination of the tested samples indicate that none of the coatings were eroded to the substrate after 2 pumping hours. The erosion rate of the coating supplied by each vendor can be seen in Figure 5, for the raw values refer Table 10 in the Appendix.

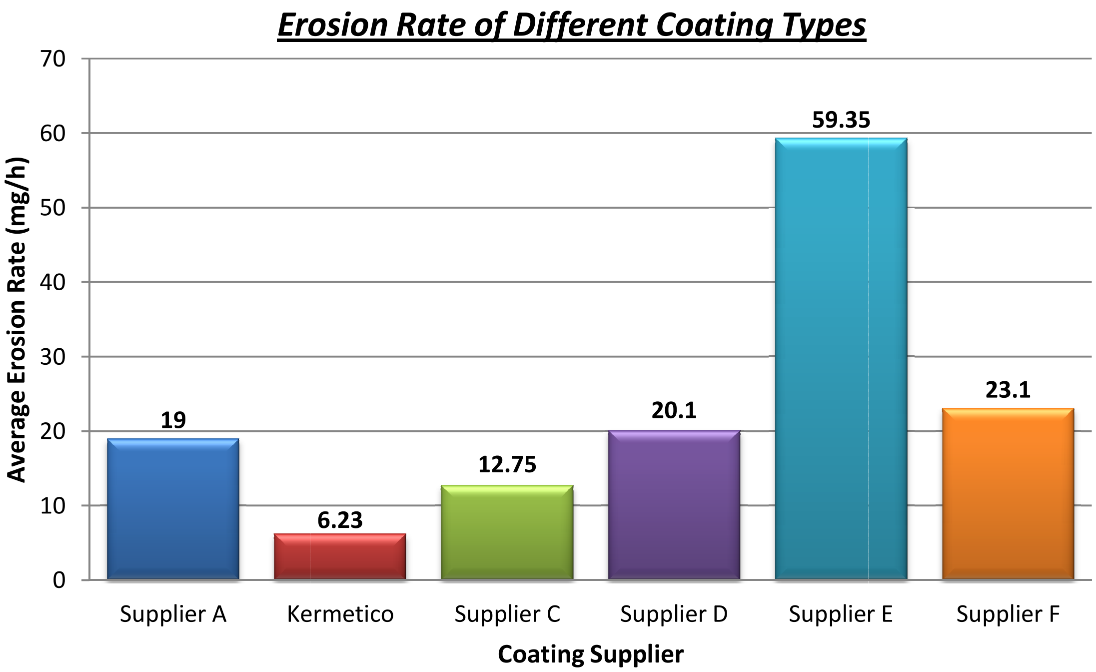


FIGURE 5 - EROSION RATE OF DIFFERENT SUPPLIER COATINGS

3.3 Micro-Structure Analysis

Due to HVOF coating not exhibiting an equiaxed grain structure, the size of the grains were measured by the height of the intersplats. The intersplat height of the coating was determined from polished and etched cross-sections using the linear intercept method. Refer to Table 2 for the measured grain size values.

The porosity level of each coating was measured using calibrated image analysis software. Several areas were assessed and the average values of the individual coatings are provided in Table 2.

Test Results

TABLE 2 - COATING PROPERTIES

Supplier and process	Thickness after polishing, µm (in.)	Grain size, µm	Porosity level, %
Supplier A (HVOF)	387±14 (0.015)	10 – 20	5.53 (±0.8)
Kermetico (HVAF)	430±14 (0.017)	5 – 10	0.89 (±0.05)
Supplier C (Special Process)	259±2 (0.010)	5 – 10	3.85 (±0.2)
Supplier D (HVOF)	364±20 (0.014)	12 – 20	3.75 (±0.9)
Supplier E (HVOF)	471±16 (0.019)	15 – 25	3.98 (±0.8)
Supplier F (HVOF)	326±14 (0.013)	15 – 25	9.01 (±0.4)

3.4 SEM Optical Microscopy Images

The sectioned samples were mounted using metallographical techniques and polished to a one micron finish. Optical microscopy was then used to image the coating interface and coating morphology of each sample. The images can be seen in Figures 12 to 35 in the Appendix.

4. **Appendix**

TABLE 3 – SUPPLIER "A" RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(S)	24.3	24.2	0.41	24.25	317.12	943	962
1(A)	23.6	24	1.69	23.8	305.46	981	
2(S)	21.6	22.3	3.24	21.95	259.82	1152	1155.5
2(A)	22.1	21.7	1.81	21.9	258.64	1159	
3(S)	24.3	24	1.23	24.15	314.51	952	976.5
3(A)	23.9	23.3	2.51	23.6	300.35	1001	
4(S)	19.8	20.4	3.03	20.1	217.87	1373	1406.5
4(A)	19.7	19.6	0.51	19.65	208.22	1440	
5(S)	21.7	22.6	4.15	22.15	264.58	1134	1122
5(A)	22.4	22.3	0.45	22.35	269.38	1110	
6(S)	25.5	25.5	0.00	25.5	350.66	853	853

TABLE 4– KERMETICO RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(S)	20.5	20.5	0.00	20.5	226.63	1318	1349.5
1(A)	20.1	20.1	0.00	20.1	217.87	1381	
2(S)	20.2	19.8	1.98	20	215.71	1389	1423.5
2(A)	19.5	19.6	0.51	19.55	206.11	1458	
3(S)	20	20.1	0.50	20.05	216.79	1389	1377
3(A)	20.2	20.2	0.00	20.2	220.04	1365	
4(S)	20.7	21.3	2.90	21	237.82	1266	1280.5
4(A)	20.7	20.8	0.48	20.75	232.19	1295	
5(S)	20	20.2	1.00	20.1	217.87	1381	1385
5(A)	20.1	20	0.50	20.05	216.79	1389	

Test Results

TABLE 5 – SUPPLIER "C" RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(A)	20.4	20.7	1.47	20.55	227.73	1318	1318
2(S)	22	22.4	1.82	22.2	265.77	1128	
2(H)	21	21.3	1.43	21.15	241.23	1245	1201.33
2(A)	21.1	21.4	1.42	21.25	243.51	1231	
3(S)	22.8	23	0.88	22.9	282.80	1059	
3(H)	23.5	23.2	1.28	23.35	294.02	1021	1032
3(A)	23.5	23.3	0.85	23.4	295.28	1016	
4(S)	22.8	23.6	3.51	23.2	290.26	1032	
4(H)	23.2	24	3.45	23.6	300.35	1001	1016.33
4(A)	23.2	23.6	1.72	23.4	295.28	1016	
5(S)	22.3	22.8	2.24	22.55	274.22	1093	
5(H)	22.7	22.80	0.44	22.75	279.10	1076	1091
5(A)	22.6	22.3	1.33	22.45	271.79	1104	

TABLE 6 – SUPPLIER "D" RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(S)	20.10	20.20	0.50	20.15	218.95	1373	
1(H)	20.50	20.70	0.98	20.60	228.84	1310	1341.33
1(A)	20.30	20.40	0.49	20.35	223.32	1341	
2(S)	19.50	19.70	1.03	19.60	207.16	1449	1399.00
2(A)	20.40	20.20	0.98	20.30	222.23	1349	
3(S)	18.50	19.10	3.24	18.80	190.60	1570	1560.00
3(A)	18.90	19.00	0.53	18.95	193.65	1550	
4(S)	25.40	25.70	1.18	25.55	352.04	853	843.50
4(A)	25.30	26.40	4.35	25.85	360.35	834	
5(S)	20.20	20.10	0.50	20.15	218.95	1373	1377.00
5(A)	20.00	20.20	1.00	20.10	217.87	1381	

Test Results

TABLE 7 – SUPPLIER "E" RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(S)	28.3	29.3	3.53	28.8	447.29	671	672.00
1(A)	29.3	28.1	4.10	28.7	444.19	673	
2(S)	21	21.6	2.86	21.3	244.66	1224	1238.00
2(A)	20.8	21.4	2.88	21.1	240.09	1252	
3(S)	30	29.9	0.33	29.95	483.72	619	622.50
3(A)	30	29.6	1.33	29.8	478.89	626	
4(S)	28.6	29.1	1.75	28.85	448.84	668	652.00
4(A)	29.5	29.7	0.68	29.6	472.48	636	
5(S)	24.2	24.5	1.24	24.35	319.74	939	941.00
5(A)	24.1	24.5	1.66	24.3	318.43	943	

TABLE 8 – SUPPLIER "F" RESULTS

Measurement Number	Dx (µm)	Dy (µm)	Normalized Diagonal error (%)	Mean Diagonal Length (µm)	Indentation surface area (µm²)	Measured Hardness (HV 0.3)	Mean Hardness Measurement - Per Indent (HV 0.3)
1(S)	28.7	28.9	0.70	28.8	447.29	671	658.50
1(A)	29.1	29.6	1.72	29.35	464.54	646	
2(S)	19.4	19.5	0.52	19.45	204.01	1475	1512.50
2(A)	18.9	19	0.53	18.95	193.65	1550	
3(S)	19.4	19.6	1.03	19.5	205.06	1466	1475.00
3(A)	19.1	19.6	2.62	19.35	201.91	1484	
4(S)	31.1	30.3	2.57	30.7	508.25	593	518.00
4(A)	35.6	35.3	0.84	35.45	677.70	443	
5(S)	25.8	25.5	1.16	25.65	354.80	849	880.50
5(A)	24.8	24.6	0.81	24.7	329.00	912	

Test Results

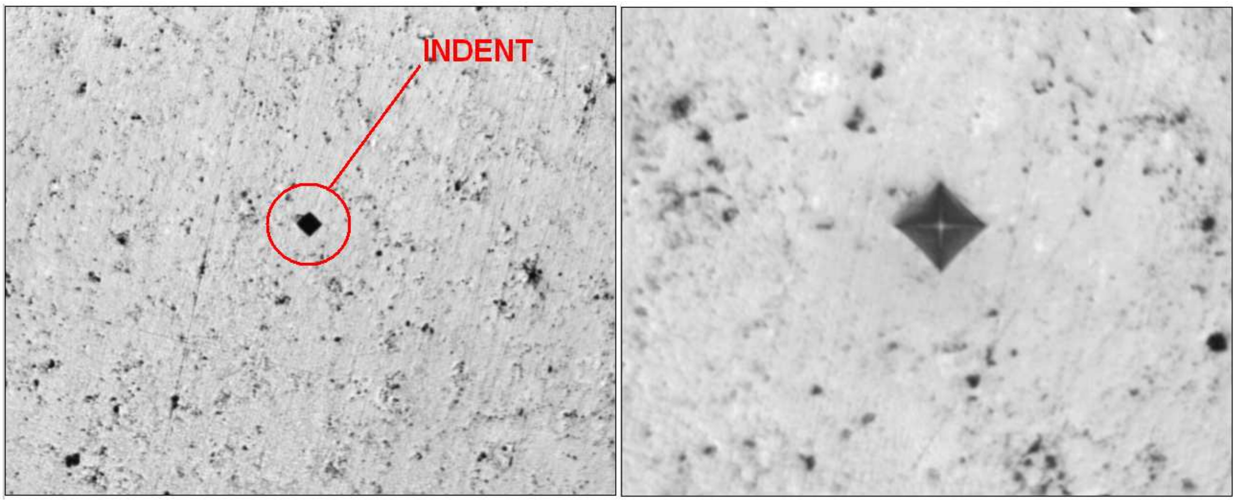


FIGURE 6 – **SUPPLIER "A" TEST SAMPLE**: 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

FIGURE 7 - **KERMETICO TEST SAMPLE**: 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

Test Results

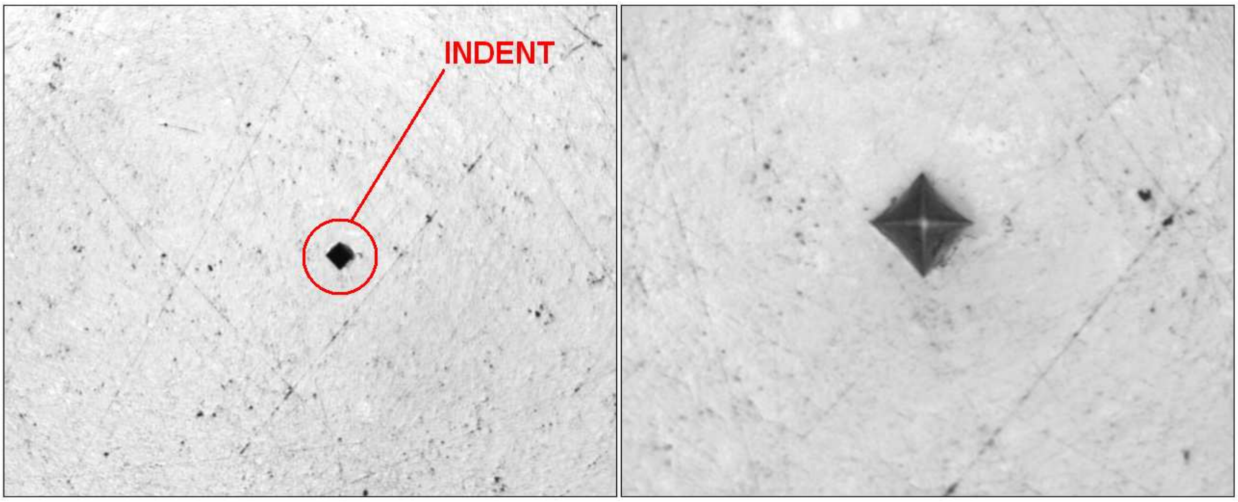


FIGURE 8 – **SUPPLIER "C" TEST SAMPLE:** 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

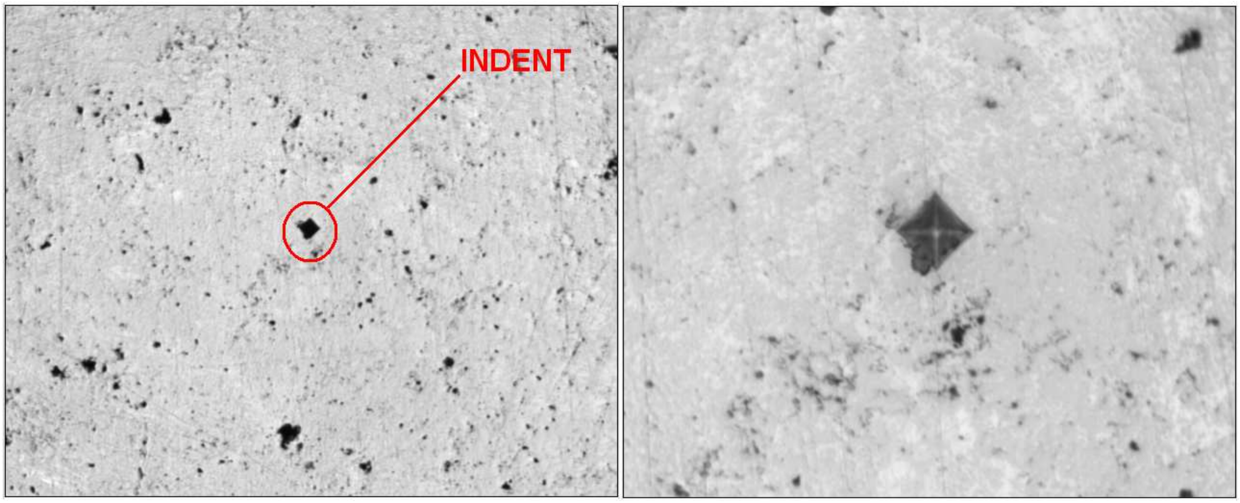


FIGURE 9 – **SUPPLIER "D" TEST SAMPLE:** 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

Test Results

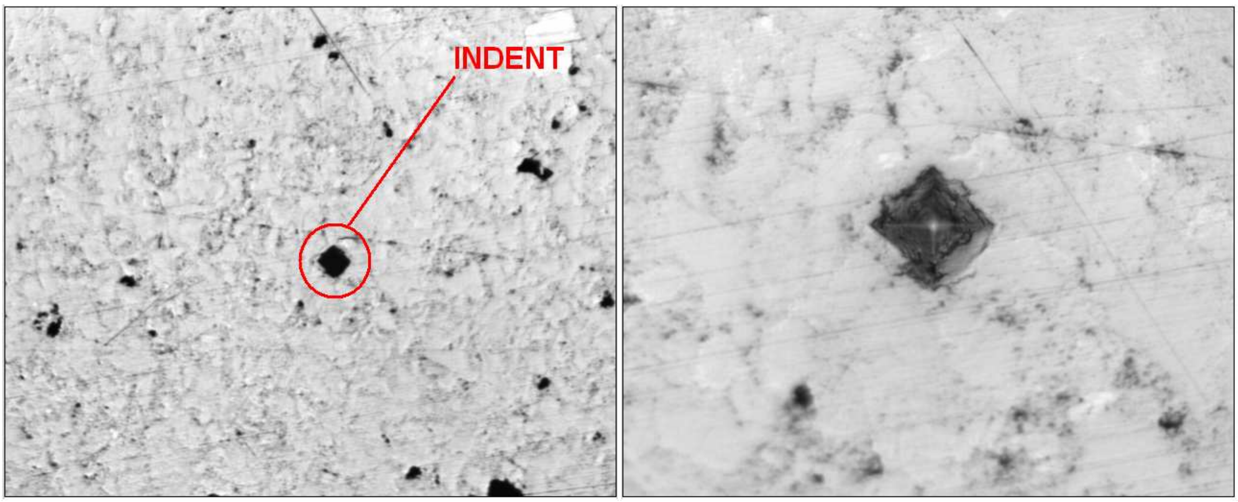


FIGURE 10 – **SUPPLIER "E" TEST SAMPLE**: 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

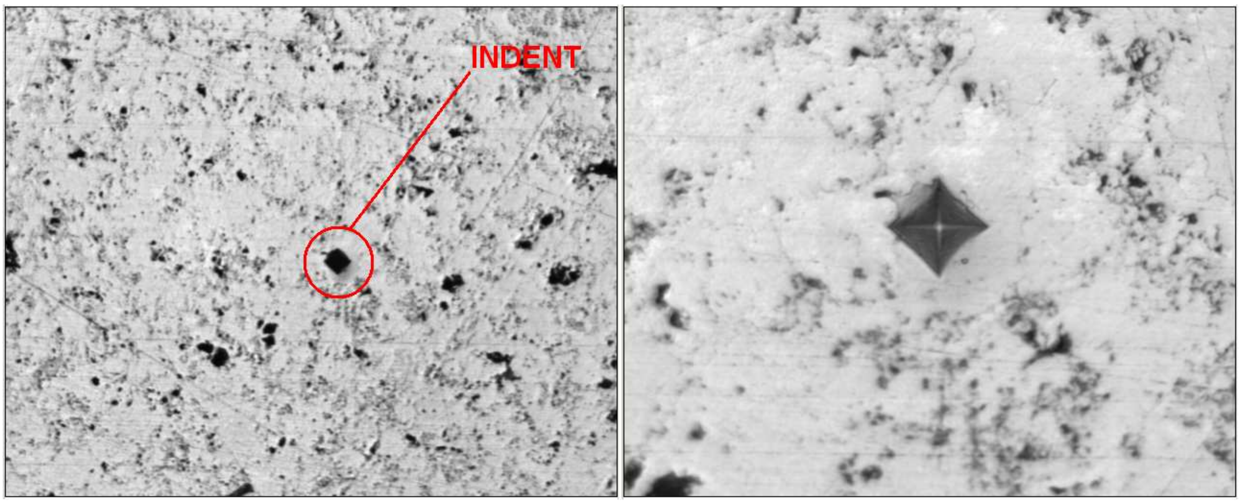


FIGURE 11 – **SUPPLIER "F" TEST SAMPLE**: 10 X MAGNIFICATION (LEFT) & 40 X MAGNIFICATION (RIGHT)

Test Results

TABLE 9 - WORST CASE ANGLE DETERMINATION

Sample Number	*Sample Type*	*Impingement angle (deg)*	*M1 (g)*	*M2 (g)*	*Erosion Rate R_E (mg/h)*
1	Supplier “A”	20	111.1755	111.1468	14.35
2	Supplier “A”	60	111.4722	111.4412	15.5
3	Supplier “A”	90	111.876	111.8354	20.3

TABLE 10 - EROSION RATE OF DIFFERENT COATING TYPES

Sample Number	*Coating Type*	*M1 (g)*	*M2 (g)*	*ΔM (mg)*	*Erosion Rate R_E (mg/h)*	*Average Erosion Rate R_E (mg/h)*
1	Supplier “A”	111.876	111.8354	40.6	20.3	19.00
2	Supplier “A”	111.7134	111.678	35.4	17.7	
1	Kermetico	106.7706	106.7586	12	6	6.23
2	Kermetico	107.0637	107.0508	12.9	6.45	
1	Supplier “C”	124.3742	124.3467	27.5	13.75	12.75
2	Supplier “C”	122.6693	122.6458	23.5	11.75	
1	Supplier “D”	109.1972	109.1534	43.8	21.9	20.10
2	Supplier “D”	110.5468	110.5102	36.6	18.3	
1	Supplier “E”	108.6549	108.5332	121.7	60.85	59.35
2	Supplier “E”	106.157	106.0413	115.7	57.85	
1	Supplier “F”	109.1802	109.1311	49.1	24.55	23.10
2	Supplier “F”	110.2698	110.2265	43.3	21.65	

FIGURE 12 - OVERVIEW OF SUPPLIER "A" COATING

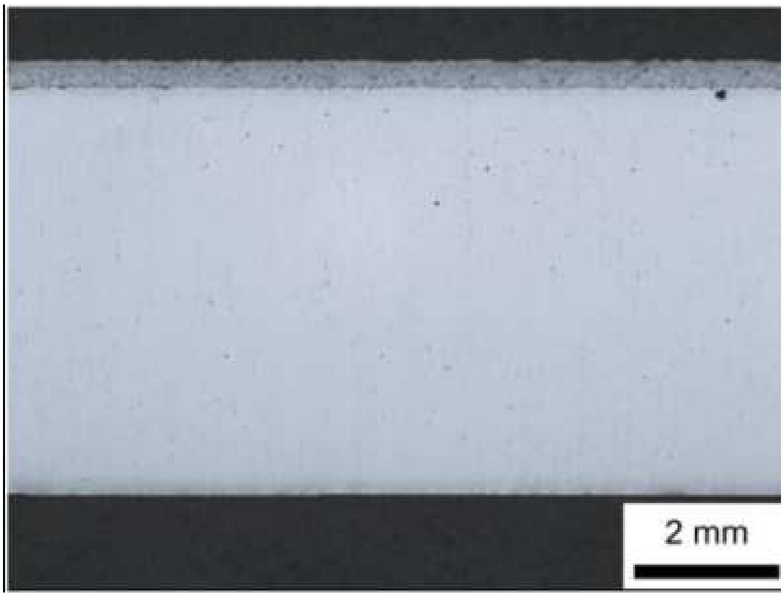


FIGURE 13 - OVERVIEW OF SUPPLIER "A" COATING

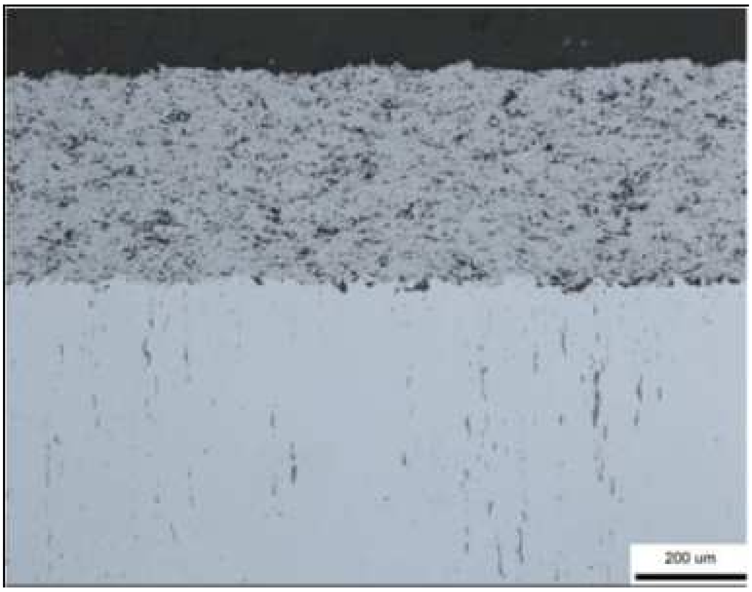


FIGURE 14 - DETAIL OF SUPPLIER "A" COATING

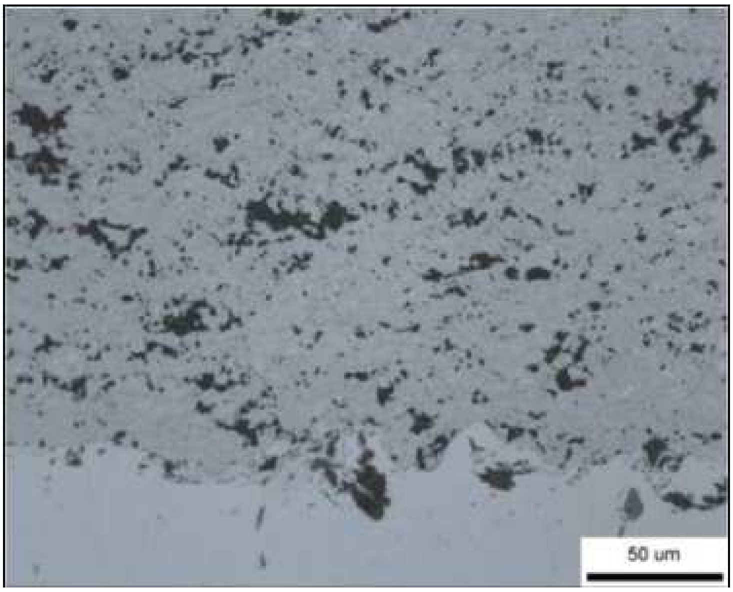


FIGURE 3 - MAGNIFIED IMAGE OF SUPPLIER "A" COATING (ETCHED)

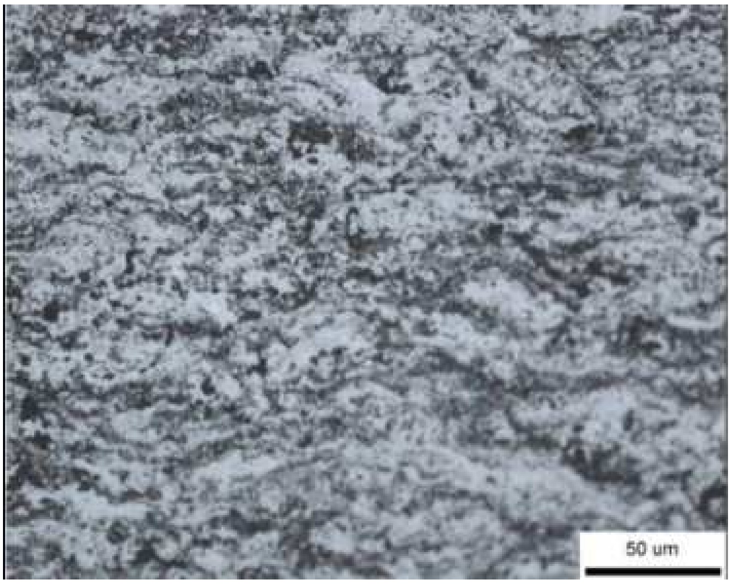


FIGURE 4 - OVERVIEW OF KERMETICO COATING

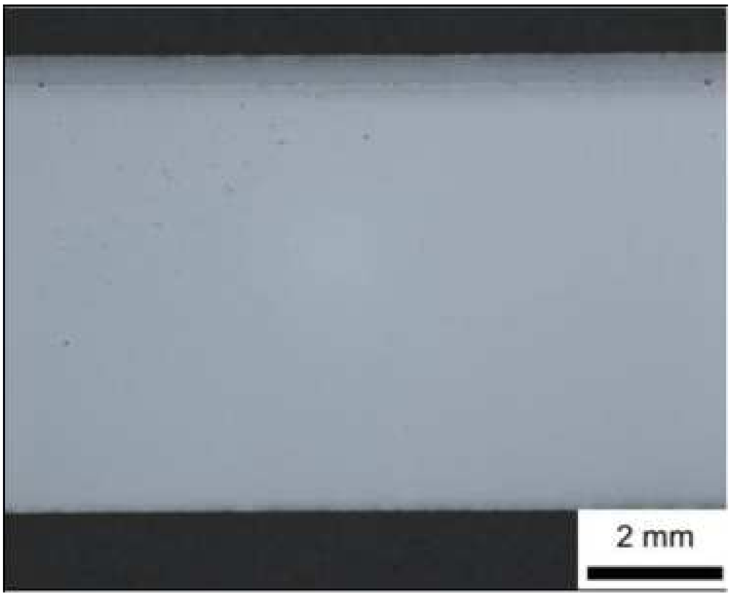
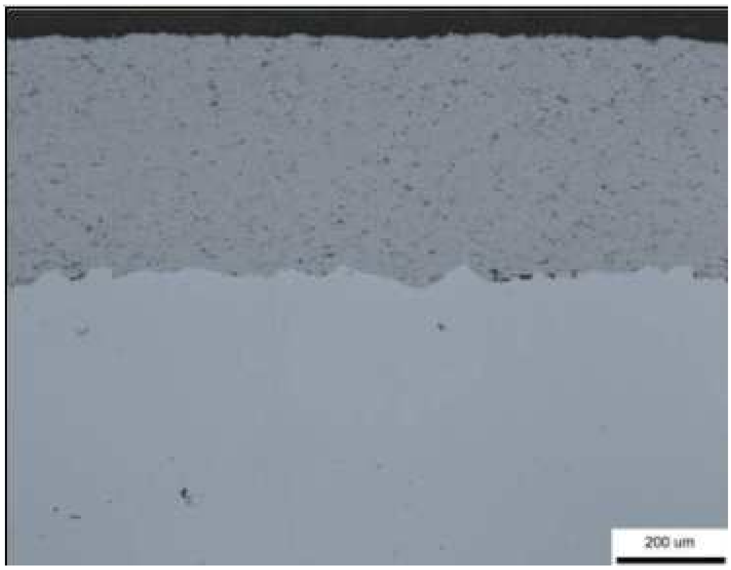


FIGURE 17 - OVERVIEW OF KERMETICO COATING



Test Results

FIGURE 18 - DETAIL OF KERMETICO COATING

FIGURE 19 - MAGNIFIED IMAGE OF KERMETICO COATING (ETCHED)

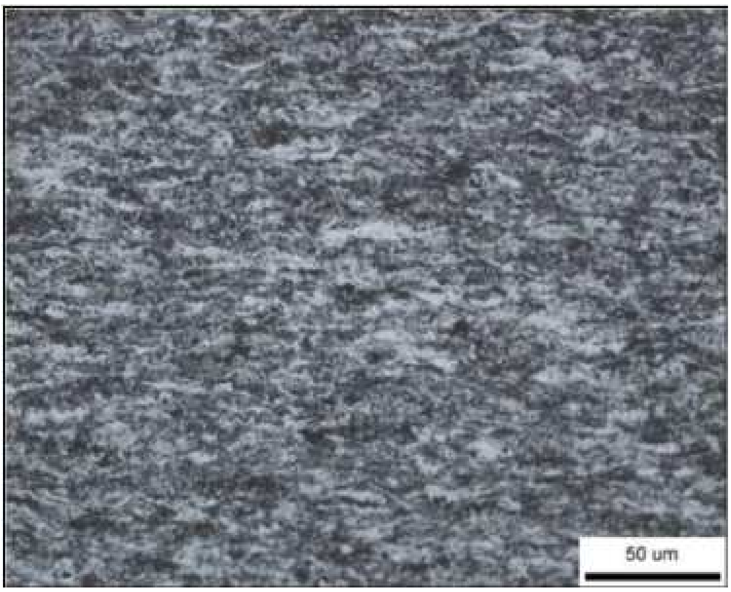


FIGURE 20 - OVERVIEW OF SUPPLIER "C" COATING

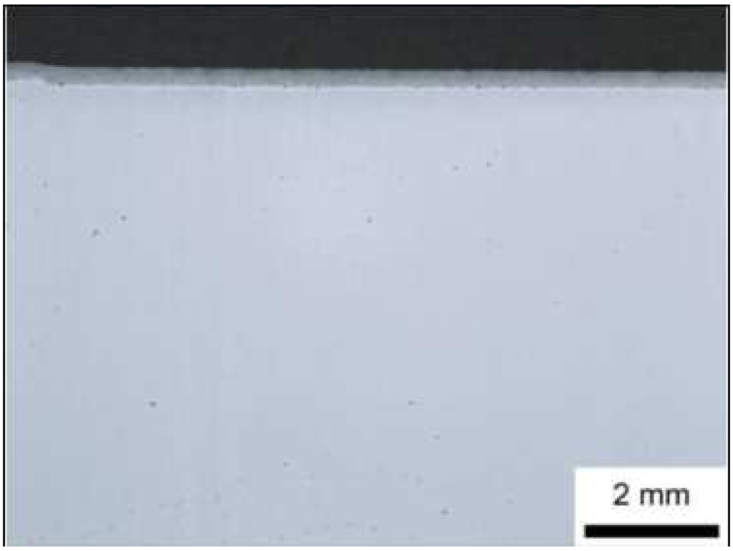


FIGURE 21 - OVERVIEW OF SUPPLIER "C" COATING

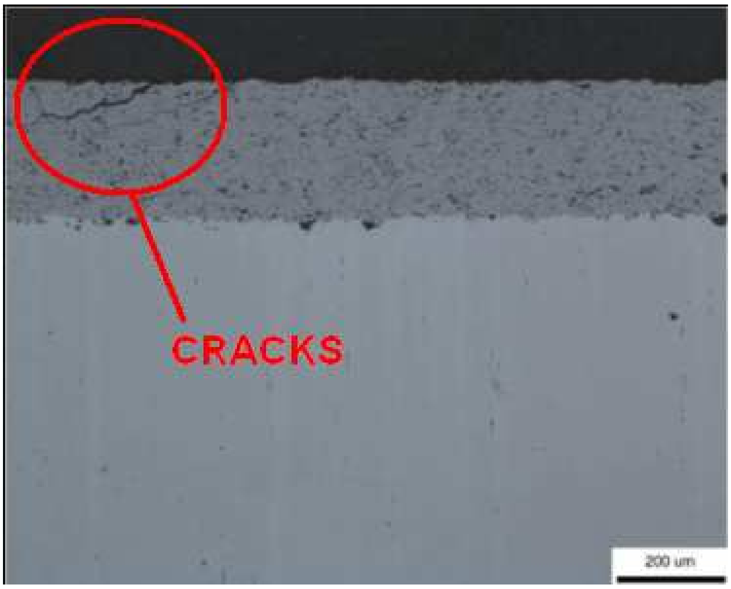


FIGURE 22 - DETAIL OF SUPPLIER “C” COATING

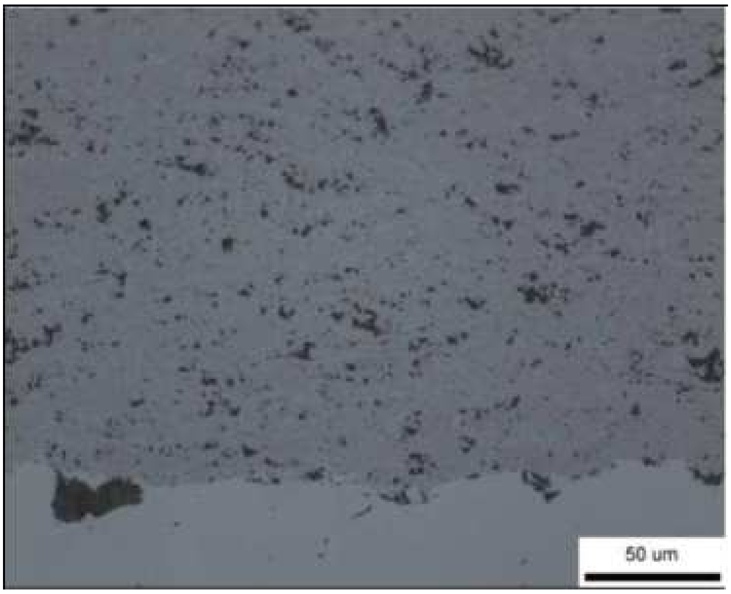


FIGURE 23 - MAGNIFIED IMAGE OF SUPPLIER “C” COATING (ETCHED)

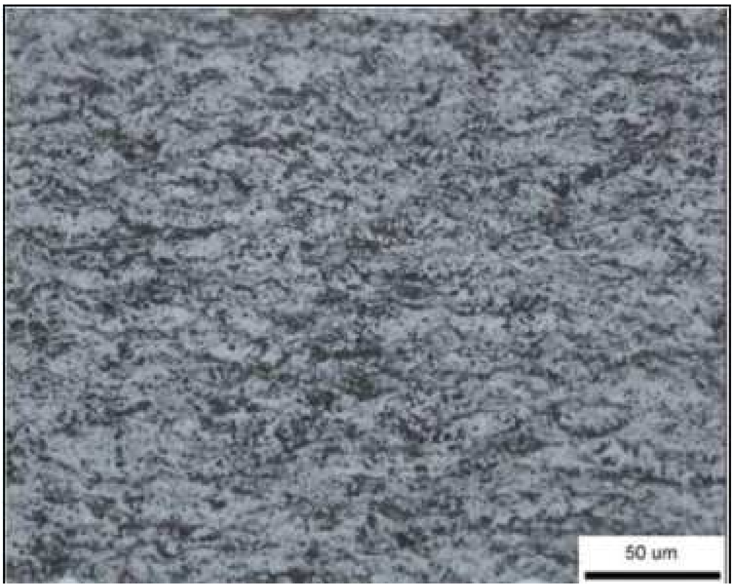


FIGURE 24 - OVERVIEW OF SUPPLIER “D” COATING

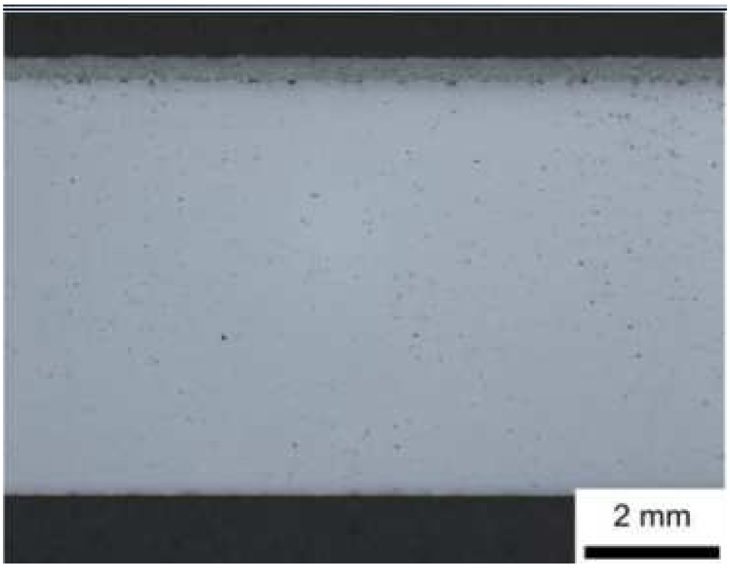


FIGURE 25 - OVERVIEW OF SUPPLIER “D” COATING

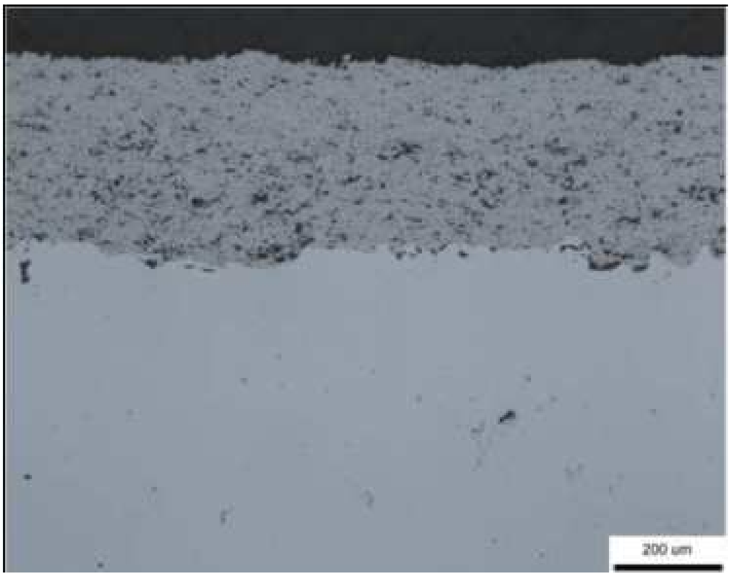


FIGURE 26 - DETAIL OF SUPPLIER “D” COATING

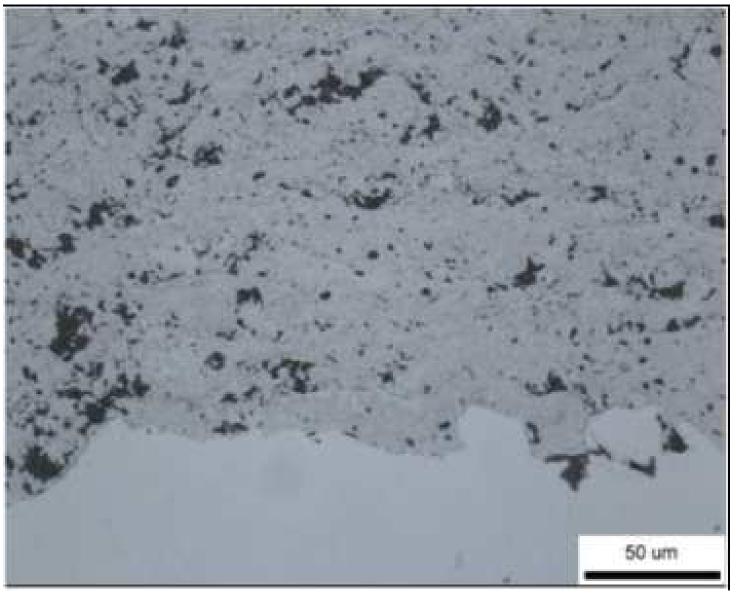


FIGURE 27 - MAGNIFIED IMAGE OF SUPPLIER “D” COATING (ETCHED)

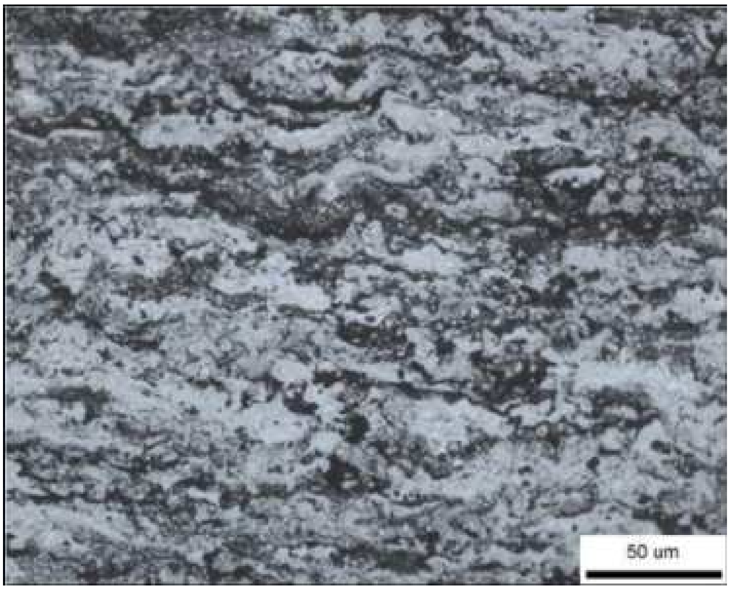


FIGURE 28 - OVERVIEW OF SUPPLIER “E” COATING

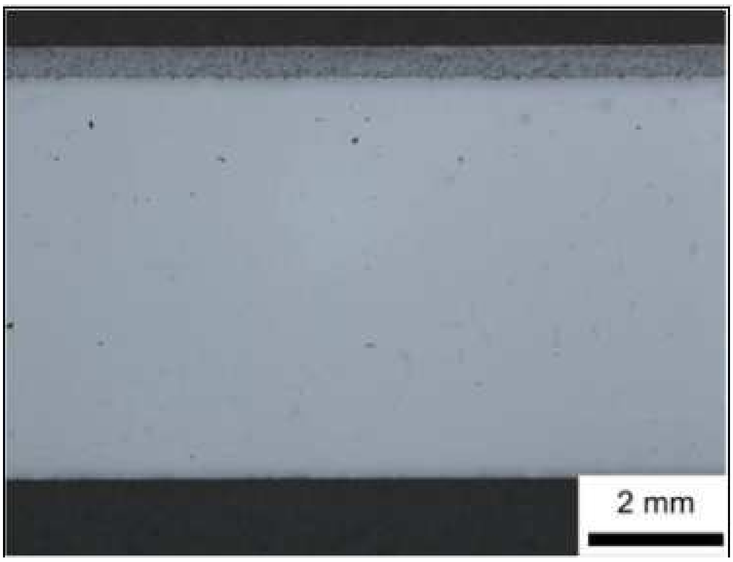


FIGURE 29 - OVERVIEW OF SUPPLIER “E” COATING

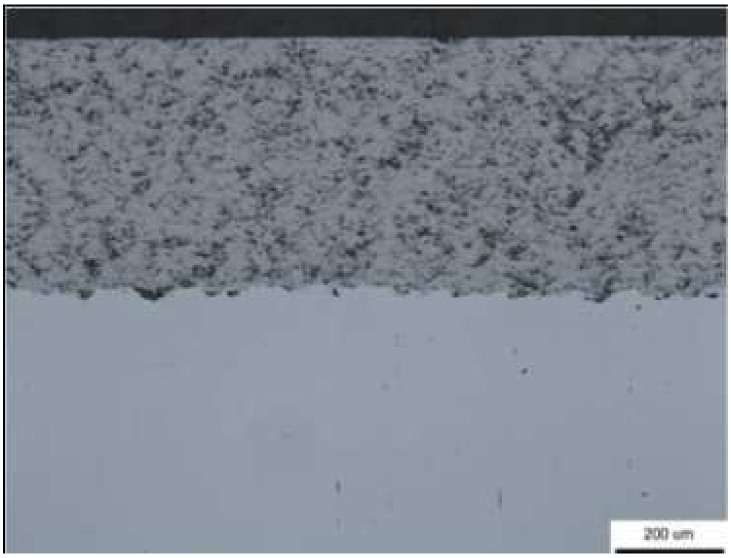


FIGURE 30 - DETAIL OF SUPPLIER “E” COATING

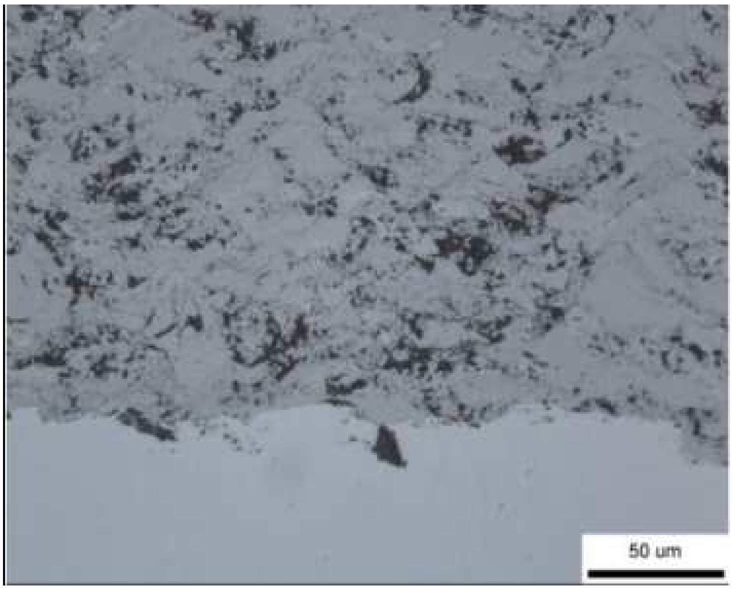


FIGURE 31 - MAGNIFIED IMAGE OF SUPPLIER “E” COATING (ETCHED)

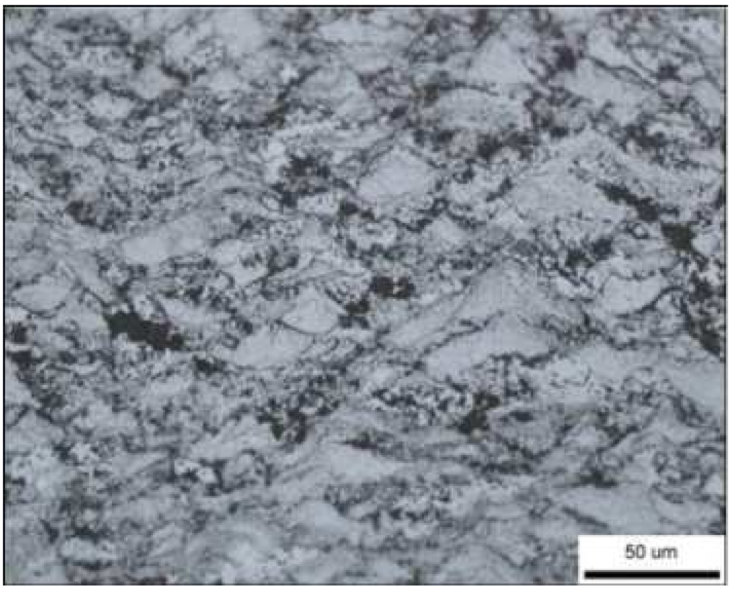


FIGURE 32 - OVERVIEW OF SUPPLIER “F” COATING

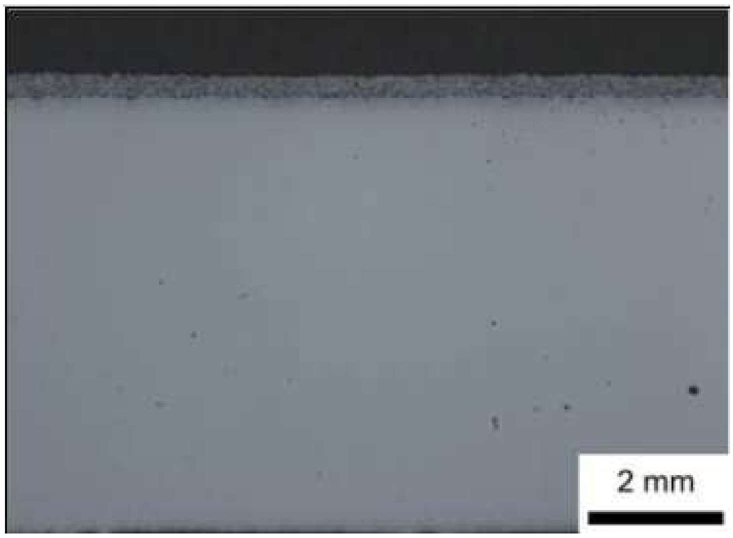


FIGURE 33 - OVERVIEW OF SUPPLIER “F” COATING

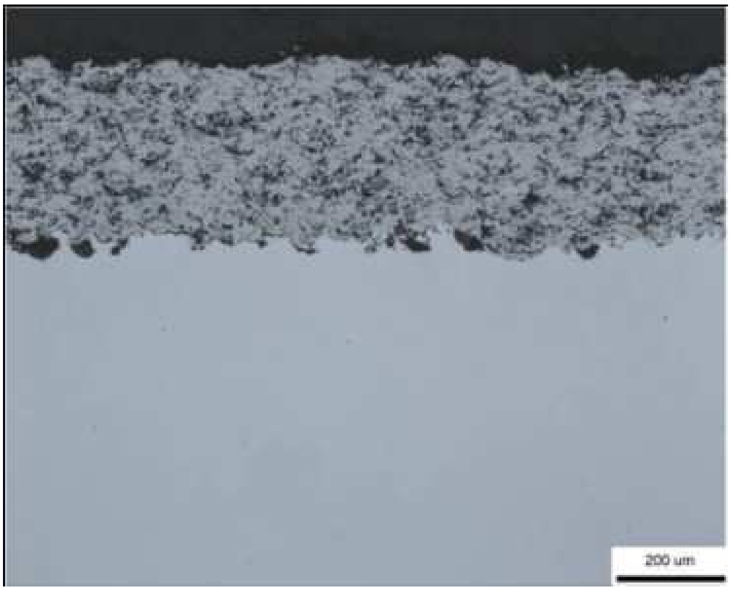


FIGURE 34 - DETAIL OF SUPPLIER “F” COATING

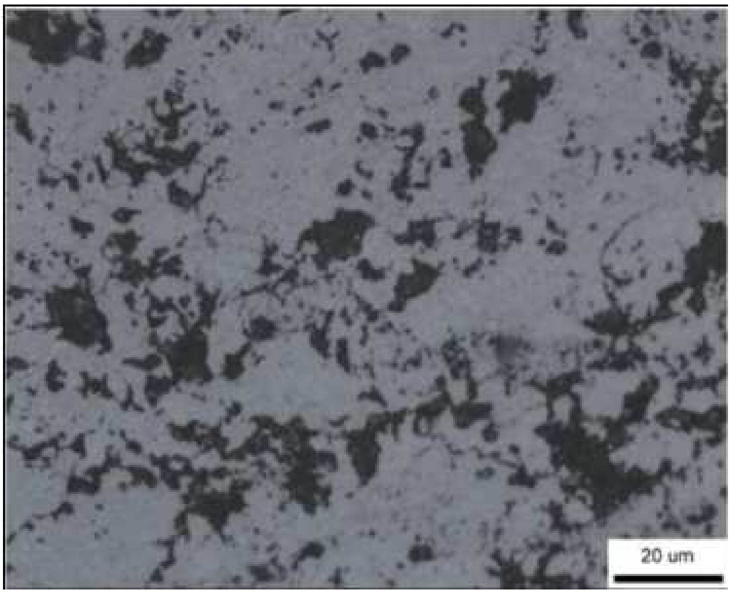


FIGURE 35 - MAGNIFIED IMAGE OF SUPPLIER “F” COATING (ETCHED)