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CONTAMINATION ANALYSES OF
TECHNOLOGY MIRROR ASSEMBLY
OPTICAL SURFACES

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CONTAMINATION ANALYSES OF TECHNOLOGY MIRROR ASSEMBLY OPTICAL SURFACES

INTRODUCTION

We received 24 tape lift samples. The samples consisted of 1/2" square adhesive tapes attached to 1/2" diameter aluminum pin-type SEM mounts. The 24 samples were shipped in six boxes, four samples per box. Each box was taped around the opening, and the samples were labeled 1 through 24 on the outside of each box. With the samples, we received a table entitled "Sample Priority Ranking" which listed the samples in order of analysis priority (Table I).

In your letter describing the sample analysis, you indicated that 16 of the tape samples contacted the TMA mirror surfaces at edges, thus only a fraction of the surface of these tape samples are actually tape lifts. In the case of these tape lift samples, you indicated, on the outside of the corresponding sample box, the portion of the tape that you believed had contacted the mirror surface.

You requested that we perform a size analysis of the particles on the surface of the tape lift samples. The goal of this work was to determine the area fraction of the mirror obscured by particle contamination. We analyzed the following seven samples: TMA-2, TMA-14, TMA-5, TMA-8, TMA-4, TMA-6, and TMA-1. All of the samples, except for TMA-6, were analyzed for particles larger than 0.5μm in diameter. In the case of sample TMA-6, only particles larger than 1.0μm in diameter were included in the analysis. These seven samples represent the first six samples in the priority ranking shown in Table I and priority sample 18 (TMA-1) which was used as a blank control sample. In this sample, less than 20% of the tape area had contacted the mirror surface. We used the remaining 80% that did not contact the mirror surface as a blank control.

ANALYSIS

The samples were analyzed using our JEOL JXA-8600 automated electron microprobe. Sample preparation consisted of coating the tape surface with a thin, 20nm thick, carbon film. Carbon coating is needed to prevent electron beam charging that can occur with uncoated insulating samples such as the tape lifts. The automated electron microprobe will automatically search the surface of the tape lift samples for particles larger than the specified minimum size. Particles are detected by an increase in their backscattered electron video signal above a preset threshold. The video threshold is usually set at a value slightly above the substrate (tape surface) video signal. Unfortunately, there is an artifact associated with the tape lift samples that precludes setting the video threshold at its lowest value. The surfaces of the tape lift samples have a crazed appearance when viewed in the electron microprobe. This is due to fine cracking of the carbon coat that is placed on top of the adhesive surface. Where the coating cracks, electron beam charging occurs and creates a brighter signal than the coated adhesive substrate.

Therefore, the video threshold had to be set at a higher level so as to omit these cracks from being included in the automated particle analysis. The size of a particle that can be found using the automated electron microprobe is a function of the video threshold setting, the average atomic number of the chemical elements in the particle, and the particle thickness. For example, it is easier to find smaller, high atomic number element-containing particles such as steels than it is to find lower atomic number element-containing particles such as aluminosilicates (dirt). The minimum particle size detection limit is estimated to be between 0.5 and 1.0μm for aluminosilicate particles and less than 0.5μm for particles heavier than titanium. The automated particle analysis programs provide data on the size and elemental composition of each particle found during the analysis. Particle size is determined by measuring eight diameters through the center of the particle. The program reports the minimum, maximum, and average diameter for each particle. Data are also provided for up to 27 chemical elements.

RESULTS

The results of the analyses are listed in Tables II through VII. The amount of tape surface area analyzed varies from $1.343 \times 10^7 \mu m^2$ to $3.01 \times 10^7 \mu m^2$. The particles that were found on each sample were initially divided into two categories based on the total number of x-ray counts in the energy dispersive x-ray (EDX) spectrum. Those particles with less than 300 total x-ray counts (XT <300) are considered to be organic material or artifacts of the sample surface, (i.e., cracks that were mentioned earlier). Inorganic particles are those that have more than 300 total x-ray counts (XT >300). As we cannot distinguish whether the particles with total x-ray counts <300 are indeed organic particles on the surface of the tape or whether they are artifacts, the data listed in Tables II through VII are for the inorganic particles only. Each table lists the number and percent number of particles in each size range and the area and the percent area for the particles in each size range. In addition, the total particle area is given in each table. Please note that, if you add up all of the particles in the various size ranges, the total may not equal the number of particles with XT >300 listed at the top of each table. This is due to the fact that the number of particles in the smallest size range had to be interpolated from the histogram printout. However, the error in this interpolation should not make a significant difference in the overall analysis. Table VIII summarizes the results from Tables II through VII and lists the total particle area for each sample and the fractional area obscured by particles which range from 1.7×10^{-5} (TMA-4) to 7.7×10^{-5} (TMA-2).

It was observed, during the course of the particle size analysis, that many of the particles on samples TMA-2, TMA-14, TMA-5, and TMA-8 are submicrometer particles of stainless steel or stainless steel corrosion products. We relocated several submicrometer stainless steel particles in sample TMA-8 and performed a semi-quantitative elemental analysis to determine their composition. All of the particles contained major amounts of iron, chromium, and nickel. Figure 1 is a typical EDX spectrum of one of the stainless steel particles. In addition to iron, chromium, and nickel, the particles also contained a minor amount of silicon but did not contain molybdenum. The semi-quantitative analyses indicate that the particle composition was consistent with 304 stainless steel.

Sample TMA-4 contained very few inorganic particles (16), all of which were greater than 1.5μm in diameter. None were identified as stainless steel. Sample TMA-6 contained only 82 inorganic particles larger than 1μm in diameter. None of these particles were stainless steel. However, many of the particles contained aluminum and chlorine. Some also contained a minor amount of sulfur. It is possible that these particles are aluminum metal and aluminum metal corrosion products. Figures 2 and 3 are examples of EDX spectra taken from aluminum rich particles in sample TMA-6. Figure 2 shows a particle that contains major amounts of aluminum and a minor amount of copper, whereas Figure 3 shows a particle containing major amounts of aluminum with some chlorine and minor amounts of sulfur and copper. Figure 3b is simply the expanded vertical scale of the spectrum in Figure 3a.

We also analyzed the portion of sample TMA-1 that did not contact the mirror surface as a blank control for the other samples analyzed in this study. Sample TMA-1 was analyzed for particles larger than 0.5μm in diameter. The majority of the particles ranged in size from 1 to 30μm and only 6% of the particles were less than 1μm in diameter. Also, the composition of the particles was unlike most of the particles found on the other samples in this study (i.e., TMA-1 did not contain any significant amount of stainless steel or aluminum-rich particles).

SUMMARY

Based upon our automated analyses of the tape lifts from the TMA optical surfaces and the control blank, we can conclude that the particles identified on the actual samples were not a result of contamination due to the handling or sampling process itself and the particles reflect the actual contamination on the surface of the mirror.

TABLE I

PRIORITY	SAMPLE #	MIRROR	LOCATION	LIFT PROPORTION
1	2	P	Bottom-Middle	100%
2	14	H	Bottom-Middle	100%
3	5	P	Top-Middle	100%
4	8	P	Right-Middle	100%
5	4	P	Top-Front	50%
6	6	P	Top-Back	80%
7	17	H	Top-Middle	100%
8	20	H	Left-Middle	100%
9	7	P	Right-Front	25%
10	9	P	Right-Back	75%
11	16	H	Top-Front	40%
12	18	H	Top-Back	30%
13	19	H	Left-Front	45%
14	13	H	Bottom-Front	25%
15	15	H	Bottom-Back	70%
16	21	H	Left-Back	65%
17	3	P	Bottom-Back	80%
18	1	P	Bottom-Front	<20%
19	11	P	Left-Middle	100%
20	23	H	Right-Middle	100%
21	10	P	Left-Front	30%
22	22	H	Right-Front	30%
23	12	P	Left-Back	70%
24	24	H	Right-Back	40%

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TABLE II

Sample	TMA-2
Area Analyzed (μm^2)	1.884×10^7
No. Particles (XT >300)	452 (56%)
No. Particles (XT <300)	361 (44%)

Size (μm)	No.	No. %	Area (μm^2)	Area %
0.5-1.0	288	64	127	9
1.0-1.5	132	29	162	11
1.5-2.0	21	5	51	4
2.0-2.5	9	2	35	2
2.5-3.0	2	0.4	12	0.8
3.0-5.5	-	-	-	-
5.5-6.0	1	0.2	26	2
6.0-7.5	-	-	-	-
7.5-8.0	1	0.2	47	3
8.0-35.0	-	-	-	-
35.0-36.0	1	0.2	990	68
TOTAL = 1450				

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TABLE III

Sample	TMA-14
Area Analyzed (μm^2)	1.343×10^7
No. Particles (XT >300)	277 (62%)
No. Particles (XT <300)	173 (38%)

Size (μm)	No.	No. %	Area (μm^2)	Area %
0.5-1.0	184	66	81	22
1.0-1.5	52	19	64	17
1.5-2.0	19	7	46	12
2.0-2.5	8	3	32	8
2.5-3.0	5	2	29	8
3.0-3.5	3	1	25	7
3.5-4.0	3	1	33	9
4.0-4.5	-	-	-	-
4.5-5.0	1	0.4	18	5
5.0-7.5	-	-	-	-
7.5-8.0	1	0.4	47	12
TOTAL = 375				

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TABLE IV

Sample TMA-5

Area Analyzed (μm^2) 2.83×10^7

No. Particles (XT >300) 380 (71%)

No. Particles (XT <300) 154 (29%)

Size (μm)	No.	No. %	Area (μm^2)	Area %
0.5-1.0	252	66	108	17
1.0-1.5	66	17	82	13
1.5-2.0	24	6	58	9
2.0-2.5	17	4	67	10
2.5-3.0	7	2	41	6
3.0-3.5	4	1	33	5
3.5-4.0	3	1	33	5
4.0-4.5	3	1	42	6
4.5-6.0	-	-	-	-
6.0-6.5	2	0.5	61	9
6.5-8.5	-	-	-	-
8.5-9.0	1	0.3	60	9
9.0-9.5	1	0.3	67	9
TOTAL = 652				

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TABLE V

Sample: TMA-8

Area Analyzed (μm^2): 1.67×10^7

No. Particles (XT >300): 345 (78%)

No. Particles (XT <300): 99 (22%)

Size (μm)	No.	No. %	Area (μm^2)	Area %
0.5-1.0	235	68	101	11
1.0-1.5	45	13	56	6
1.5-2.0	17	5	41	5
2.0-2.5	15	4	59	7
2.5-3.0	8	2	47	5
3.0-3.5	3	9	25	3
3.5-4.0	6	2	67	8
4.0-4.5	2	0.6	28	3
4.5-5.0	2	0.6	35	4
5.0-5.5	1	0.3	22	3
5.5-6.0	1	0.3	26	3
6.0-6.5	3	0.9	92	10
6.5-7.0	-	-	-	-
7.0-7.5	2	0.6	83	9
7.5-8.0	-	-	-	-
8.0-8.5	-	-	-	-
8.5-9.0	1	0.3	60	7
9.0-9.5	2	0.6	135	15
		TOTAL = 877		

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TABLE VI

Sample TMA-4

Area Analyzed (μm^2) 1.51×10^7

No. Particles (XT >300) 16 (89%)

No. Particles (XT <300) 2 (11%)

Size (μm)	No.	No. %	Area (μm^2)	Area %
0.5-1.0	-	-	-	-
1.0-1.5	-	-	-	-
1.5-2.0	2	13	5	2
2.0-2.5	3	19	12	5
2.5-3.0	1	6	6	2
3.0-3.5	1	6	8	3
3.5-4.0	3	19	33	13
4.0-4.5	1	6	14	5
4.5-5.0	2	13	35	14
5.0-5.5	1	6	22	9
5.5-6.0	-	-	-	-
6.0-6.5	-	-	-	-
6.5-7.0	-	-	-	-
7.0-7.5	1	6	41	16
7.5-10.0	-	-	-	-
10.0-10.5	1	6	82	32
TOTAL = 258				

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TABLE VII

Sample	**TMA-6**
Area Analyzed (μm^2)	**3.01×10^7**
No. Particles (XT >300)	**82 (34%)**
No. Particles (XT <300)	**158 (66%)**

Size (μm)	**No.**	**No. %**	**Area (μm^2)**	**Area %**
1.0-2.0	45	55	79.7	10.1
2.0-3.0	18	22	88.4	11.2
3.0-4.0	8	10	96.2	12.1
4.0-5.0	3	4	63.6	8.0
5.0-6.0	5	6	143.0	18.1
6.0-7.0	1	1	33.2	4.2
7.0-8.0	-	-	-	-
8.0-9.0	-	-	-	-
9.0-10.0	-	-	-	-
10.0-11.0	-	-	-	-
11.0-12.0	-	-	-	-
12.0-13.0	1	1	123	15.5
13.0-14.0	-	-	-	-
14.0-15.0	1	1	165	20.8
TOTAL = 792				

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TABLE VIII

Fractional Area Obscured by Particles >0.5μm in Diameter on TMA Tape Lift Samples

SAMPLE	TOTAL PARTICLE AREA (μm^2)	FRACTIONAL AREA OBSCURED BY PARTICLES
TMA-2	1450	7.7×10^{-5}
TMA-14	375	2.8×10^{-5}
TMA-5	652	2.3×10^{-5}
TMA-8	876	5.2×10^{-5}
TMA-4	258	1.7×10^{-5}
TMA-6*	792	2.6×10^{-5}

* Particles >1μm in diameter.

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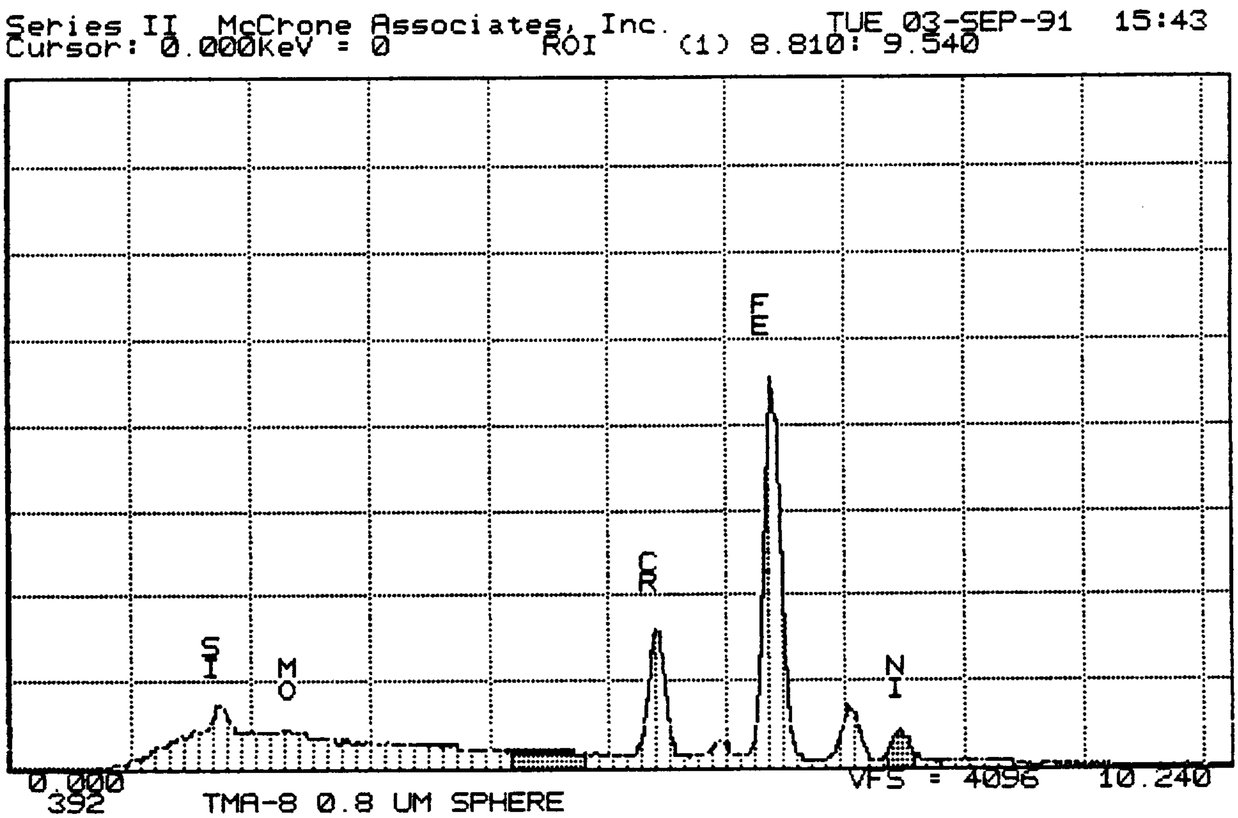
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CR
FE
NI
0.000
392
TMA-8 0.8 UM SPHERE
VFS = 4096
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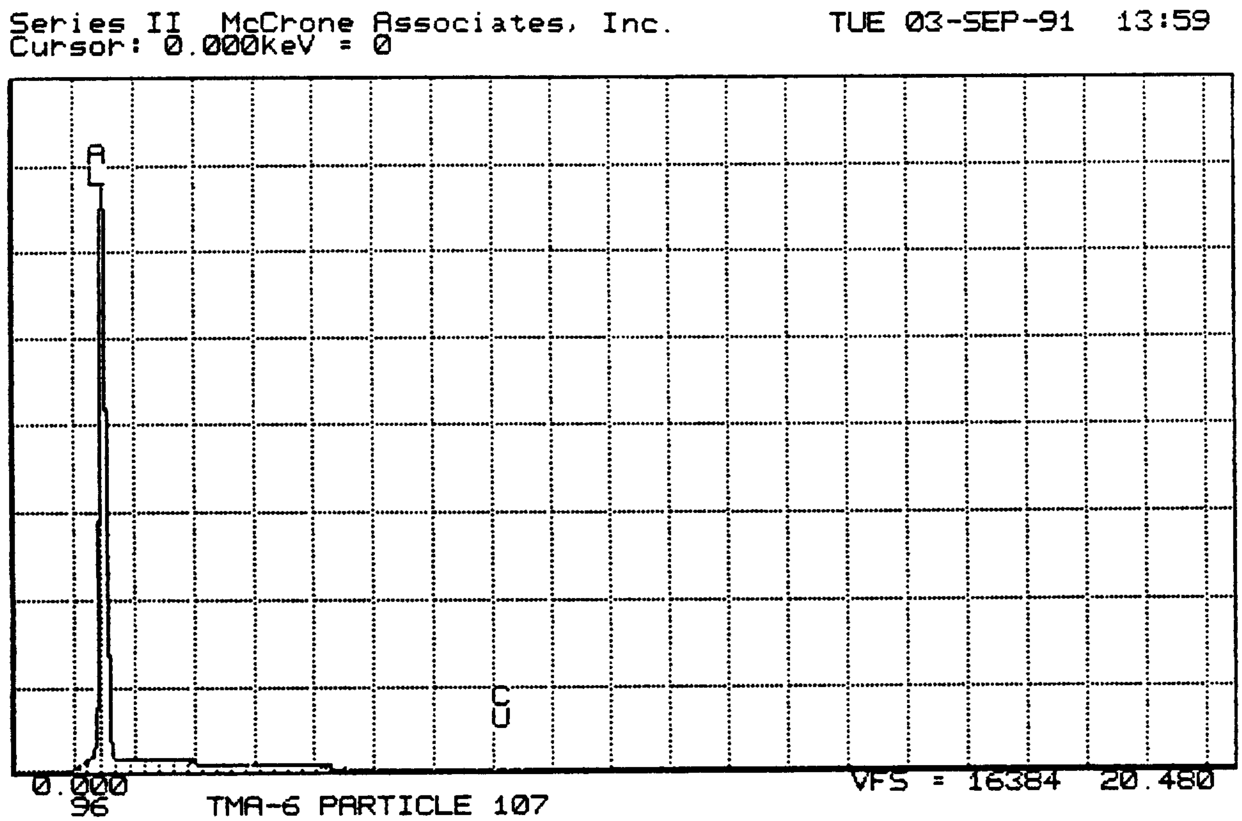
FIGURE 1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TMA-6 PARTICLE 107
VFS = 16384
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FIGURE 2

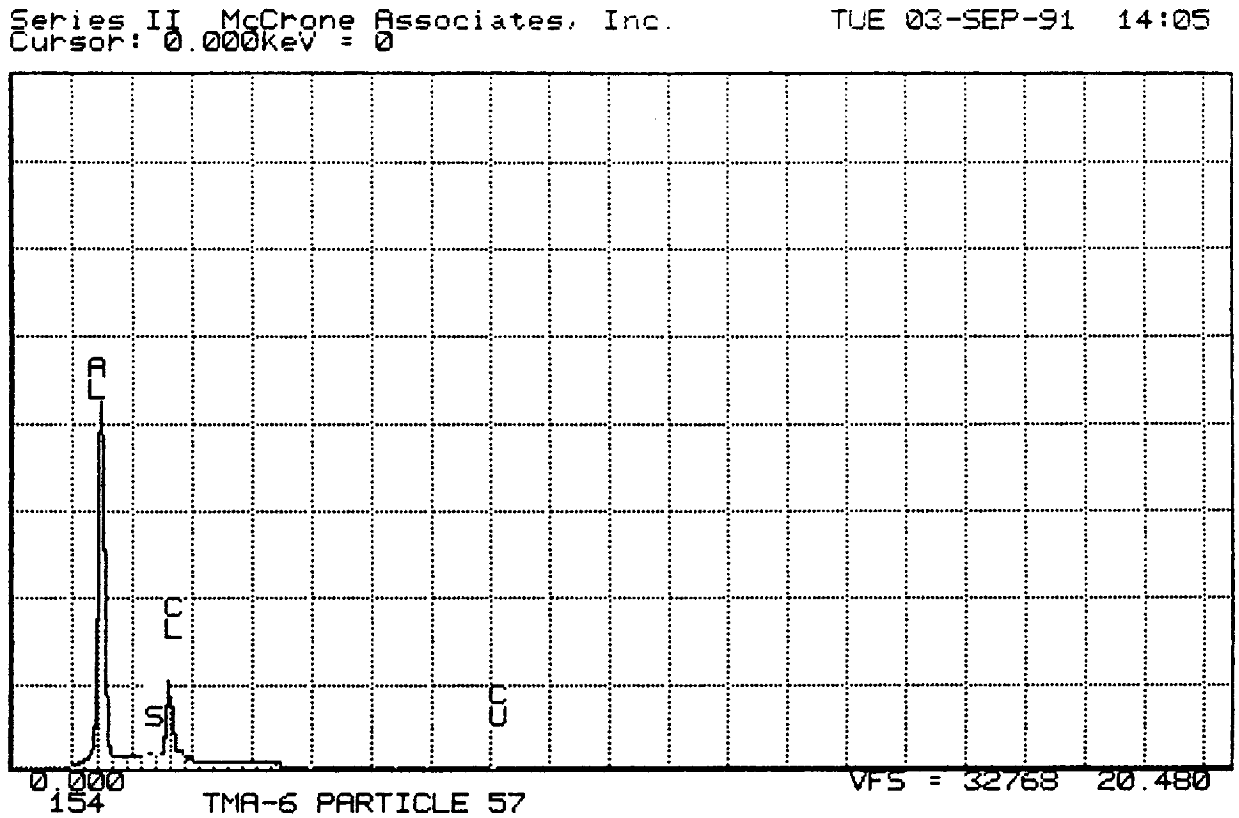

FIGURE 3a

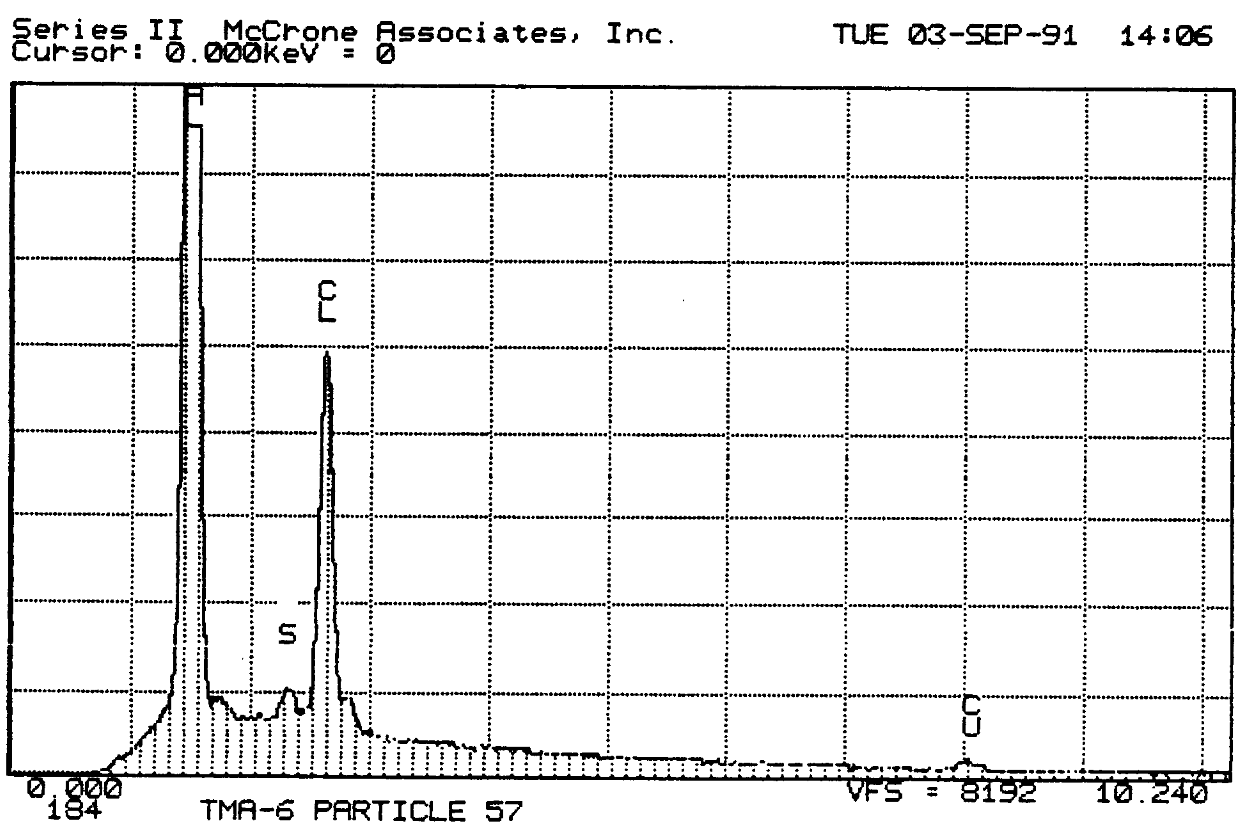

FIGURE 3b

NASA

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16. Abstract

Automated electron microprobe analyses were performed on tape lift samples from the Technology Mirror Assembly Optical Surfaces. This report details these analyses and identifies the contamination on the Mirror surfaces.

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