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MEMORANDUM REPORT M70-10-1

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METALLURGICAL EXAMINATION OF THE FOULED GAS TUBE AND FLASH SUPPRESSOR FROM AN M16A1 RIFLE

by

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EDWARD H. HESS

February 1970

U.S. ARMY MUNITIONS COMMAND

DEPARTMENT OF THE ARMY
FRANKFORD ARSENAL
Philadelphia, Pa. 19137

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Pitman-Dunn Research Laboratories
FRANKFORD ARSENAL
Philadelphia, Pa. 19137

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ABSTRACT

The metallurgical investigation of the residue extracted from the fouled gas tube and the flash suppressor of an M16A1 rifle involved: (1) metallographic examination of the contact surface, which showed incipient cracks in the gas tube wall (which were of no significance); (2) electron microprobe identification and distribution of the particles, which showed copper-zinc particles randomly distributed in a calcite matrix; (3) electron microscope studies of particle size and shape, which showed the particles to be about 0.3 micron in size and irregular in shape; (4) x-ray examination of crystal structure and composition, which showed calcite with a diffraction pattern that coincided with that of hexagonal $CaCO_3$, and a copper-zinc face-centered cubic alloy with lattice constant corresponding to 90% Cu-10% Zn.

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INTRODUCTION

Operation of the cycling mechanism of the M16 rifle is critically dependent upon the proper transmission of a propellant gas pulse through the gas tube to the bolt mechanism. As deposits from continued firing build up in the tube, the passage becomes obstructed and the cyclic rate decreases. With increasing deposit, the blockage increases, thereby decreasing the pressure pulse to the point where the rifle begins to malfunction.

Not all cartridges contribute, to the same degree, to the deposit build-up phenomena. Some make little contribution; others, a great deal. Depositing appears to be related to propellant type and composition, cartridge component geometry, and rifle geometry. An understanding of gas tube fouling phenomena depends upon an accumulation and evaluation of a variety of types of input data. One input concerns that derived from metallurgical examination.

TEST PROGRAM

The metallurgical investigation of the residue included metallographic examination of the contact surface; electron microprobe identification and distribution of the particles; electron microscope studies of particle size and shape; and x-ray examination of crystal structure and composition.

Two highly localized residue locations were subjected to metallurgical examination - the forward end of the gas tube and the rear of the flash suppressor. All the data discussed in this report were gathered from studying a single gas tube and one flash suppressor. The gas tube came from a rifle through which 4310 rounds had been fired. The flash suppressor came from the same rifle after a total of 8423 rounds had been fired. Photomicrographs were taken of the polished and etched cross-sectioned surfaces.

One of the samples was then used as a specimen on the electron microprobe for labeling the constituents and their distribution. This same sample was then powdered and an x-ray diffraction pattern was taken for identifying the constituents and their crystalline structure. In addition, electron microscope examinations were made of the specimens from the gas tube and the flash suppressor for particle size and shape.

TEST RESULTS

A representative sample of a sectioned gas tube is shown in Figure 1 at a magnification of 500X and 1000X. Incipient cracks can be seen in the sidewall of the tube. The crack in the lower photograph is approximately 0.0005 inch long. A crack of this size is not significant in materials as ductile as that used for the gas tube. These photographs also show very clearly that there has been no penetration of the stainless steel tube wall by the residue. The bond is mechanical.

The residue in the gas tube was examined at each of six cross sections taken, respectively, at 0.5, 11/16, 1.2, 1.9, 2.6, and 3.4 inches from the inlet end. The locations are shown on the x-ray photographs in Figure 2. The surfaces were polished and etched with dichromate to bring the copper-bearing constituent into relief (Figures 3 through 7). The swirl pattern of the deposited residue is shown in Figure 3. The white area inside the tube is some of the plastic mounting material and should be ignored. The swirl pattern is again evident in Figures 5 and 6.

Figure 8 graphically shows the percent of decrease in cross-sectional area of the original tube and its distance from the orifice end. Figure 9 shows the variation of cyclic rate, gas tube weight, and flow meter change for the gas tube, with the number of rounds fired.

A small sample of the residue was cut from the 2.6 inch location for electron microprobe study. The small area under observation was not moved throughout the tests so that comparisons of the photomicrographs (Figures 10 through 14) could be made. The elements composing the residue are listed in Table I. The photomicrographs clearly show that the copper and calcium patterns are complementary. Trace elements of Ba, Sb, Zn, Al, and Fe are randomly dispersed, indicating no preferred combination with either copper or calcium. Lead and sulfur, however, tend toward the copper and calcium pattern, although it cannot be said with certainty that they are tied in with the propellant or combined within the residue.

The same sample used for the microprobe study was powdered, sifted through 325 mesh screen, and used for x-ray diffraction analysis. The x-ray diffraction pattern is shown in Figure 15 and the experimentally observed peak position, relative intensities, and calculated "d" spacings (distance between crystallographic planes) are given in Table II. For easy comparison with the experimental data, the intensities, "d" spacings, and peak positions of $CaCO_3$ and a Cu-Zn (face-centered cubic) alloy containing nine atomic percent Zn, are also given. The x-ray analysis performed by Franklin Institute Research Laboratories* on the flash suppressor residue (Table III) was in good agreement with that obtained in this analysis.

*Contract DAAA 25-69-M4349, with Franklin Institute Research Laboratories, Philadelphia, Pa.

TABLE I.
Microprobe X-Ray Analysis of Residue

Target Element	Atomic Number	Radiation Analyzed[a]
Aluminum	13	$K\alpha$
Sulfur	16	$K\alpha$
Calcium	20	$K\alpha$
Iron	26	$K\alpha$
Copper	29	$K\alpha$
Zinc	30	$K\alpha$
Antimony	51	$L\alpha_1$
Barium	56	$L\alpha_1$
Lead	82	$L\alpha_1$

[a] Probe voltage, 25 kilovolts

Use is made of these results in a subsequent analysis of electron diffraction data. The Cu-Zn alloy "d" spacings were computed for a face-centered cubic alloy having a lattice spacing of 3.63 Å. Extrapolation was used on the raw x-ray data in order to derive the precision lattice constant. According to the second edition of Pearson,* this lattice constant would be for a brass containing 10 percent zinc. It is therefore certain that the metallic particles in the residue are gilding metal; the nonmetallic portion is mostly $CaCO_3$. Two very faint lines (not covered by brass or $CaCO_3$) constitute the remaining constituents. An attempt to analyze them would be too time-consuming considering their somewhat doubtful importance.

Two cellulose acetate-silicon monoxide replicas were made for electron microscopy study, one for the gas tube and one for the flash suppressor. Both showed a sedimentary appearance at low magnification. The appearance of the particulate matter is seen at high magnification in Figures 16 and 17. Dichromate etching showed the presence of a number of dense black particles. These were identified as gilding metal through x-ray analysis. Samples were then etched in a way that would cause preferential attack of the carbonate so that replication could extract gilding metal particles.

Figure 18 shows a bright field image of the replica from the lightly etched flash suppressor. A distribution of fine particles can be seen against a generally continuous background. To confirm that metal particles had been extracted, an electron diffraction pattern (recorded from

*Wm. Burton Pearson, Handbook of Lattice Spacings and Structures of Metals and Alloys; New York, N. Y.: Pergamon Press; Vol 1, 2nd ed; 1958.

TABLE II.

Comparison between Experimental and Theoretical Data for $CaCO_3$ and 91% Cu-9% Zn

Line	Experimental Data			Theoretical Data				
	Intensity[a]	d Spacing[b]	Position	Position	d Spacing[b]	Intensity[c]	Identification	
1	W	3.8420	23.15	23.05	3.86	12	$CaCO_3$	(102)[d]
2	V.W.	3.1589	28.25					
3	S	3.0178	29.60	29.45	3.035	100	$CaCO_3$	(104)
4	W	2.8444	31.45	31.45	2.845	3	$CaCO_3$	(006)
5	M	2.478	36.26	36.00	2.495	14	$CaCO_3$	(110)
6	M	2.275	39.61	39.45	2.285	18	$CaCO_3$	(113)
7	V.S.	2.0873	43.31	43.14	2.095	18	$CaCO_3$	(202)
				43.07	2.098	~100	Cu-Zn	(111)
8	M	1.9159	47.41	47.12	1.927	5	$CaCO_3$	(204)
				47.48	1.913	17	$CaCO_3$	(108)
9	M	1.8732	48.56	48.51	1.875	17	$CaCO_3$	(116)
10	S	1.8067	50.47	50.13	1.818	~46	Cu-Zn	(200)
11	V.W.	1.7397	52.55					
12	W	1.6637	55.16					
13	W	1.5973	57.66	57.40	1.604	8	$CaCO_3$	(212)
14	W	1.5208	60.86	60.67	1.525	5	$CaCO_3$	(214)
				60.98	1.518	4	$CaCO_3$	(208)
15	W	1.4867	62.41					
16	W	1.4353	64.91	64.67	1.440	5	$CaCO_3$	(300)
				65.59	1.422	3	$CaCO_3$	(0.0.12)
17	S	1.2812	73.91	73.67	1.285	~20	Cu-Zn	(220)
18	V.W.	1.2406	76.76	76.30	1.247	1	$CaCO_3$	(220)
19	V.W.	1.1823	81.31	81.54	1.1795	3	$CaCO_3$	(2.1.10)
20	V.W.	1.1510	84.01	83.76	1.1538	3	$CaCO_3$	(314)
21	S	1.0930	89.61	89.33	1.0957	~17	Cu-Zn	(311)
22	M	1.0455	94.91	94.47	1.0492	~ 5	Cu-Zn	(222)
23	W	1.0155	98.66					
24	V.W.	.98176	103.36					
25	V.W.	.962813	106.26					
26	V.W.	.951358	108.12					
27	V.W.	.940761	109.92					
28	W	.907668	116.12	115.93	.90371	~ 3	Cu-Zn	(400)
29	W	.870471	124.47					
30	S	.833201	135.17	135.00	.93371	~ 9	Cu-Zn	(331)
31	S	.811938	143.12	142.83	.81263	~ 8	Cu-Zn	(420)

[a]Relative line intensities: V.W. - Very weak
W. - Weak
M. - Medium
S. - Strong
V.S. - Very strong

[b]d Spacings - distance between crystallographic planes.

[c]Theoretical intensity base on scale of 100 for each substance

[d]Miller index.

TABLE III.

Comparison between Franklin Institute Experimental and Theoretical Data for $CaCO_3$ and Cu-Zn

Line No.	Measured d Spacing (experimental) (Å)	Theoretical Matched Values Cu-Zn (Å) (Computed)	Theoretical Matched Values $CaCO_3$ (Å) (ASTM-5-0586)
1	3.024		3.04
2	2.839		2.85
3	2.626		
4	2.462		2.50
5	2.086	2.100 (111)[a]	
6	1.919		1.91
7	1.867		1.88
8	1.812	1.819 (200)	
9	1.745		
10	1.514		1.52
11	1.489		1.47
12	1.426		1.44
13	1.283	1.286 (220)	
14	1.049	1.050 (222)	
15	0.953		
16	0.909	0.909 (400)	
17	0.834	0.834 (331)	
18	0.813	0.813 (420)	

[a]Miller index

the area of Figure 18) is shown in Figure 19. An analysis of Figure 19 is given in Table IV. Again, all but one line are matched by gilding metal or $CaCO_3$. The spotty nature of the Cu-Zn rings suggests that the discrete particles are metal; therefore, to identify the particles positively as metal, a dark field image was taken using one of the bright spots from the diffraction pattern. The micrograph shown in Figure 20 is of the same area of interest as is shown in Figure 19.

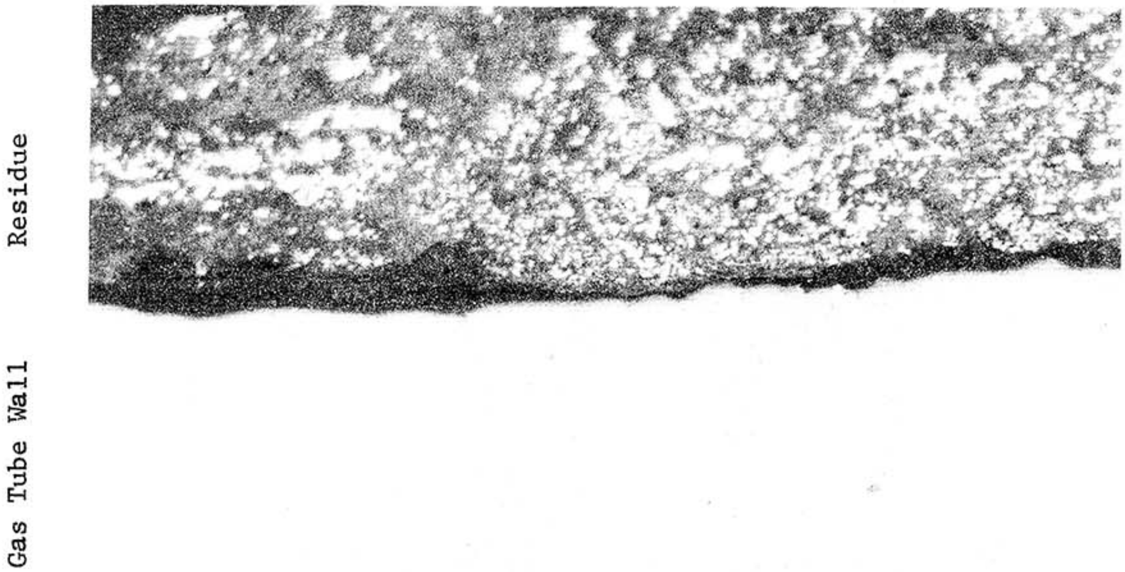
TABLE IV.

X-ray Diffraction Data Verifying $CaCO_3$ and Cu-Zn in Residue

Ring		d Spacing	Content (Å)	
Number	Type	(Å)	$CaCO_3$	Cu-Zn
1	Continuous	3.15	3.04	
2	Spotty	2.0		2.10
3	Spotty	1.88		1.82
4	Smooth	1.49	1.47	
5	Smooth	1.32		1.29
6	Spotty	1.17		
7	Spotty	1.10		1.10
8	Spotty	0.99		1.05

FINDINGS

1. The deposit is largely calcite ($CaCO_3$) and copper-zinc alloy.
2. The residue is a mechanically bonded mixture deposited in layers.
3. The calcite has a diffraction pattern which coincides with that of hexagonal $CaCO_3$, with lattice constants a_o = 4.989 Å and c_o = 17.062 Å.
4. The copper-zinc alloy is a face-centered cubic solid with a lattice constant of a_o = 3.63 Å corresponding to 90% Cu-10% Zn, with a particle size of 0.3 micron.



Mag: 500X

Mag: 1000X

Figure 1. Boundary between Residue and Tube Wall

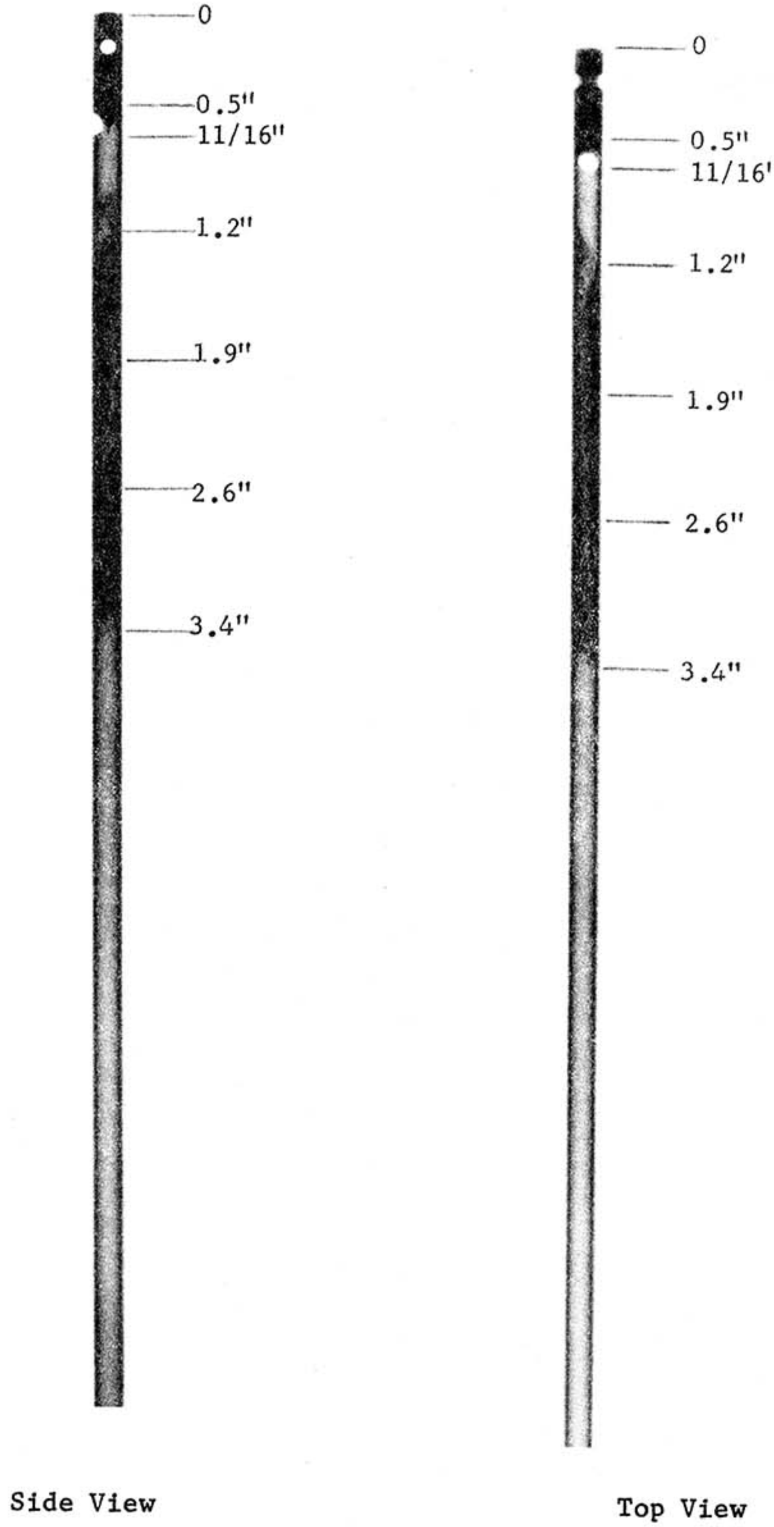
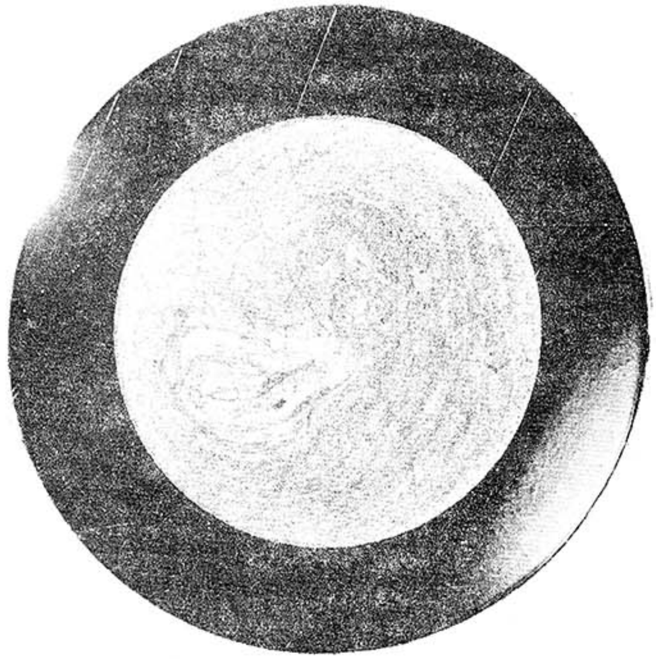


Figure 2. Locations of Gas Tube Samples



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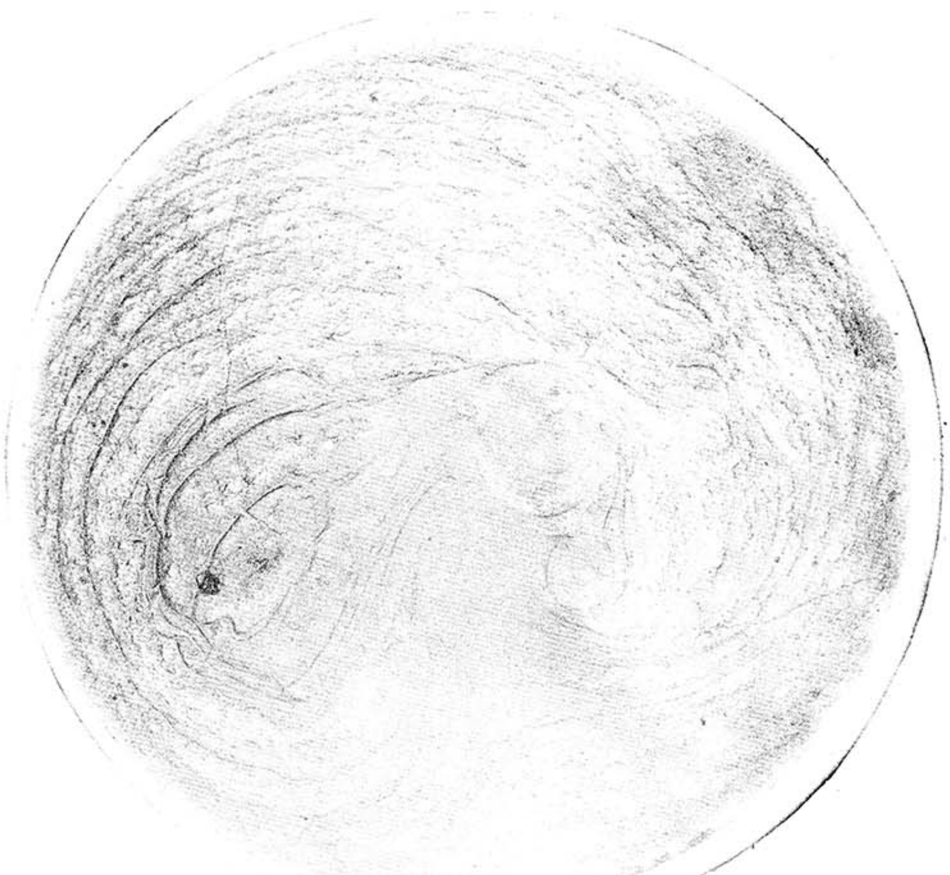
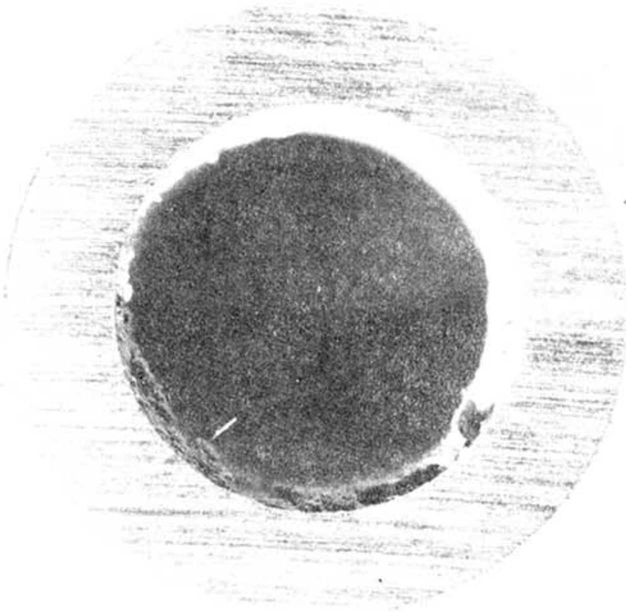


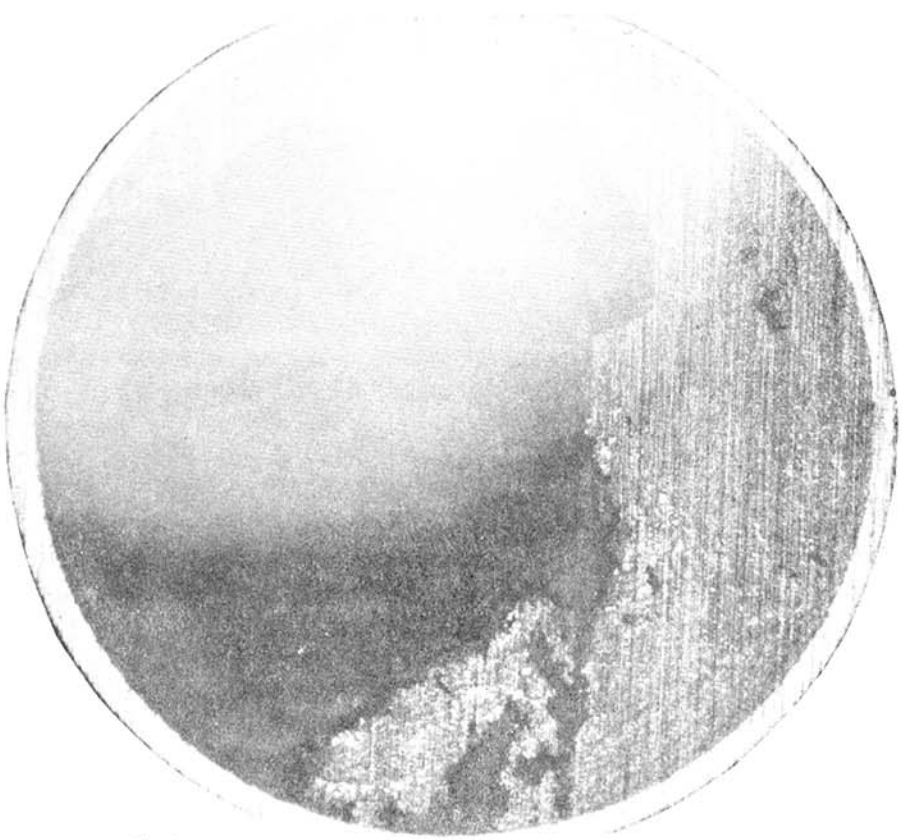
Mag: 35X

Figure 3. Residue at 0.5-in. Location (100% obstructed)



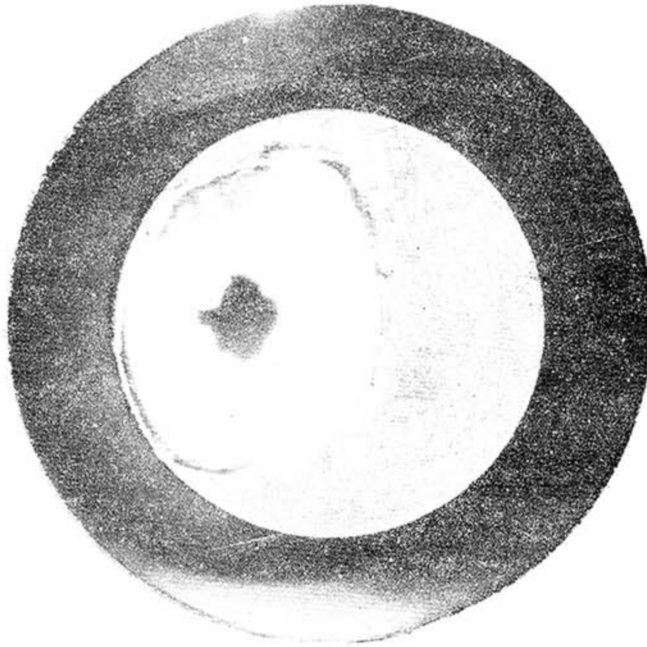
11/16 in. Mag: 17X

1.2 in. Mag: 17X



1.2 in. Mag: 35X

Figure 4. Residue at 11/16-in. (8% obstructed) and 1.2-in. (40% obstructed) Locations



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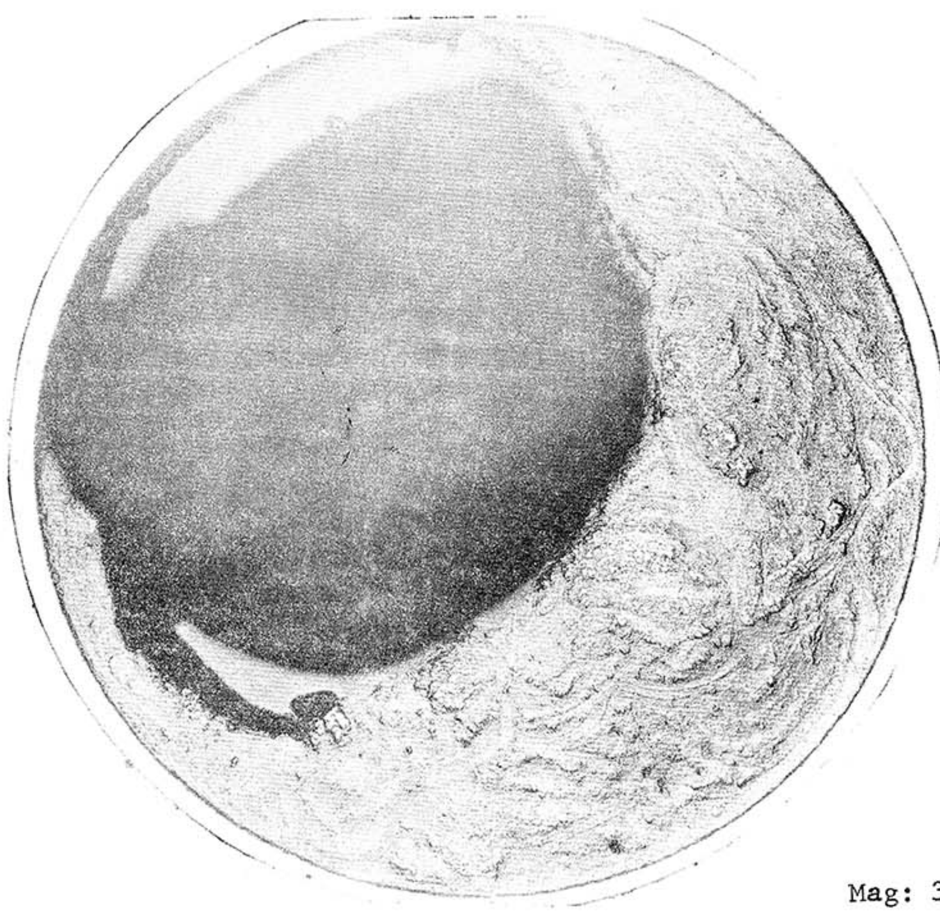
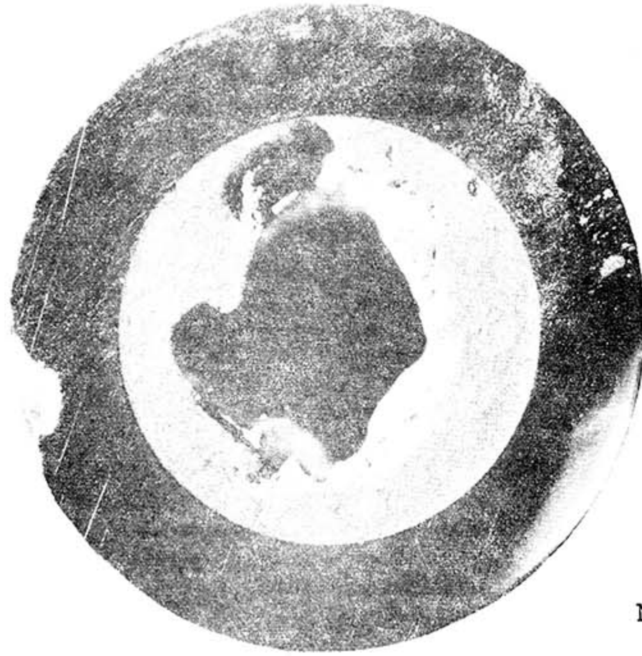


Mag: 35X

Figure 5. Residue at 1.9-in. Location (49% obstructed)



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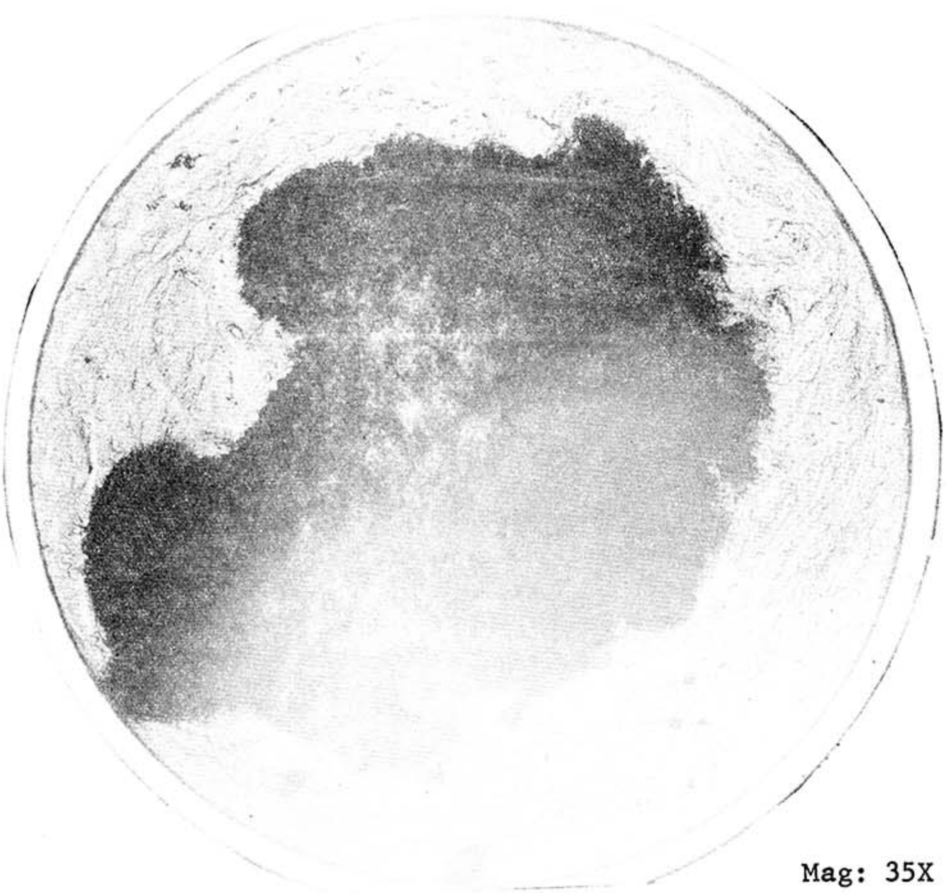
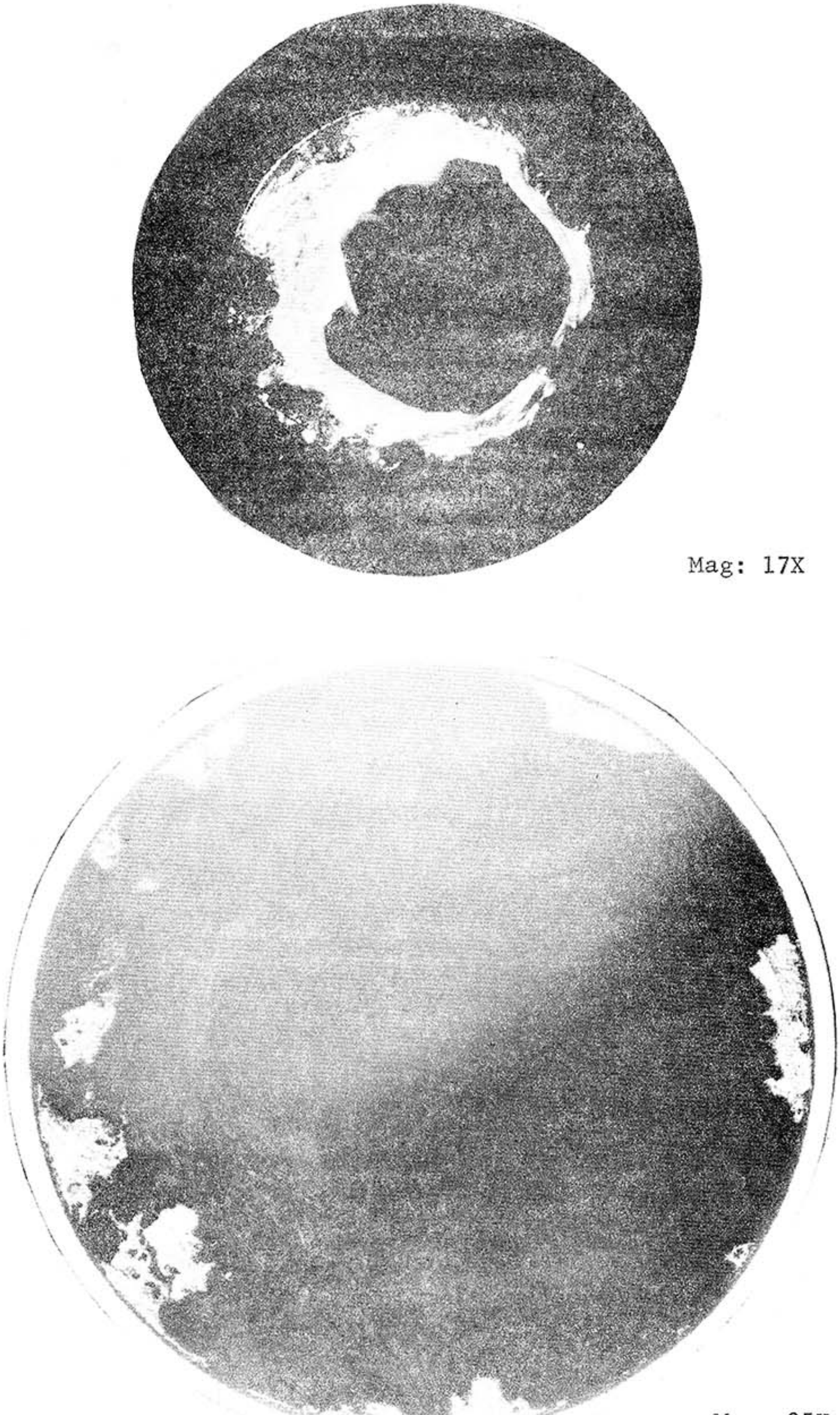


Mag: 35X

Figure 6. Residue at 2.6-in. Location (50% obstructed)



Mag: 17X

Mag: 35X

Figure 7. Residue at 3.4-in. Location (8% obstructed)

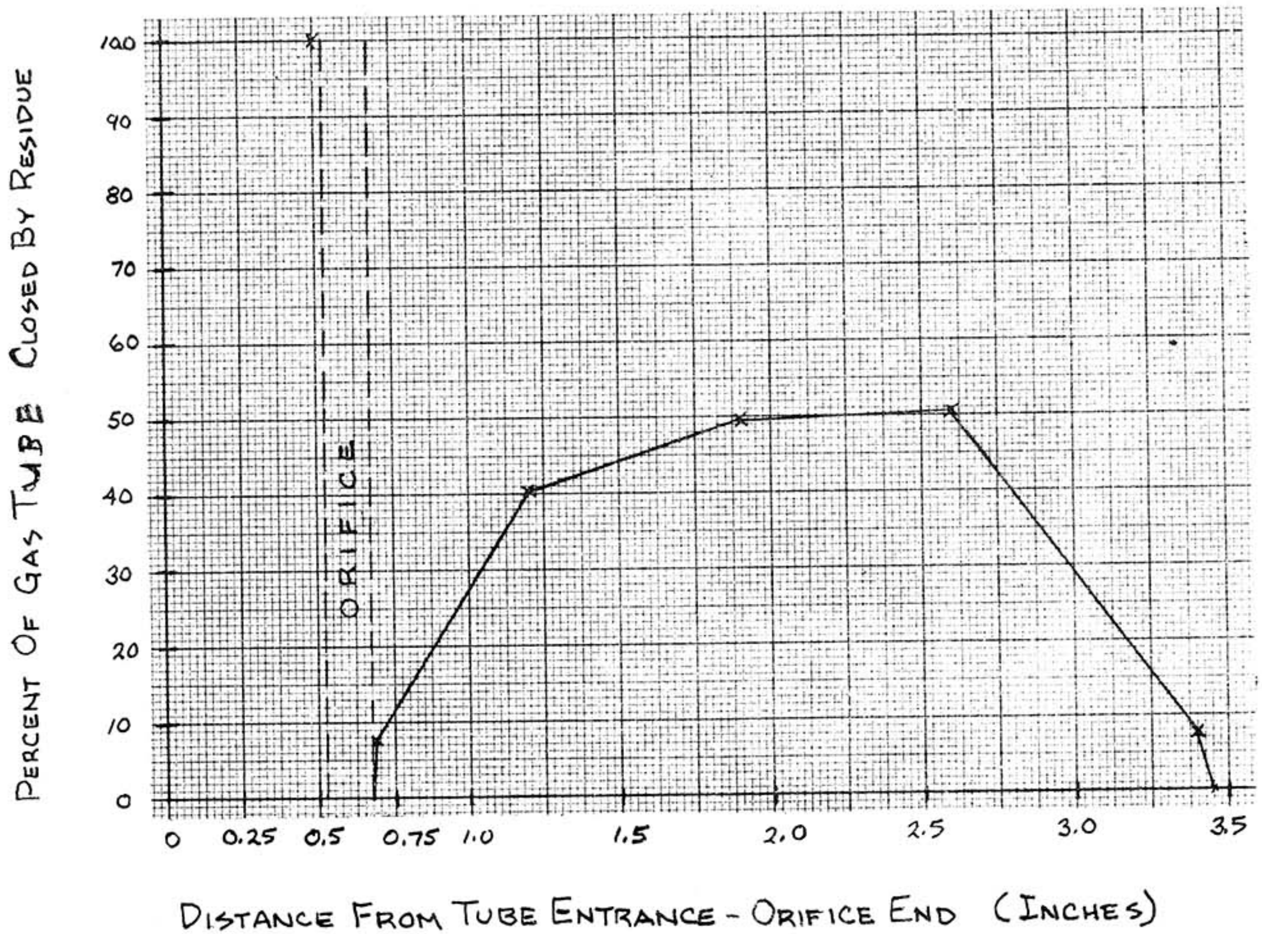


Figure 8. Variation of Residue in Gas Tube with Distance from Orifice End

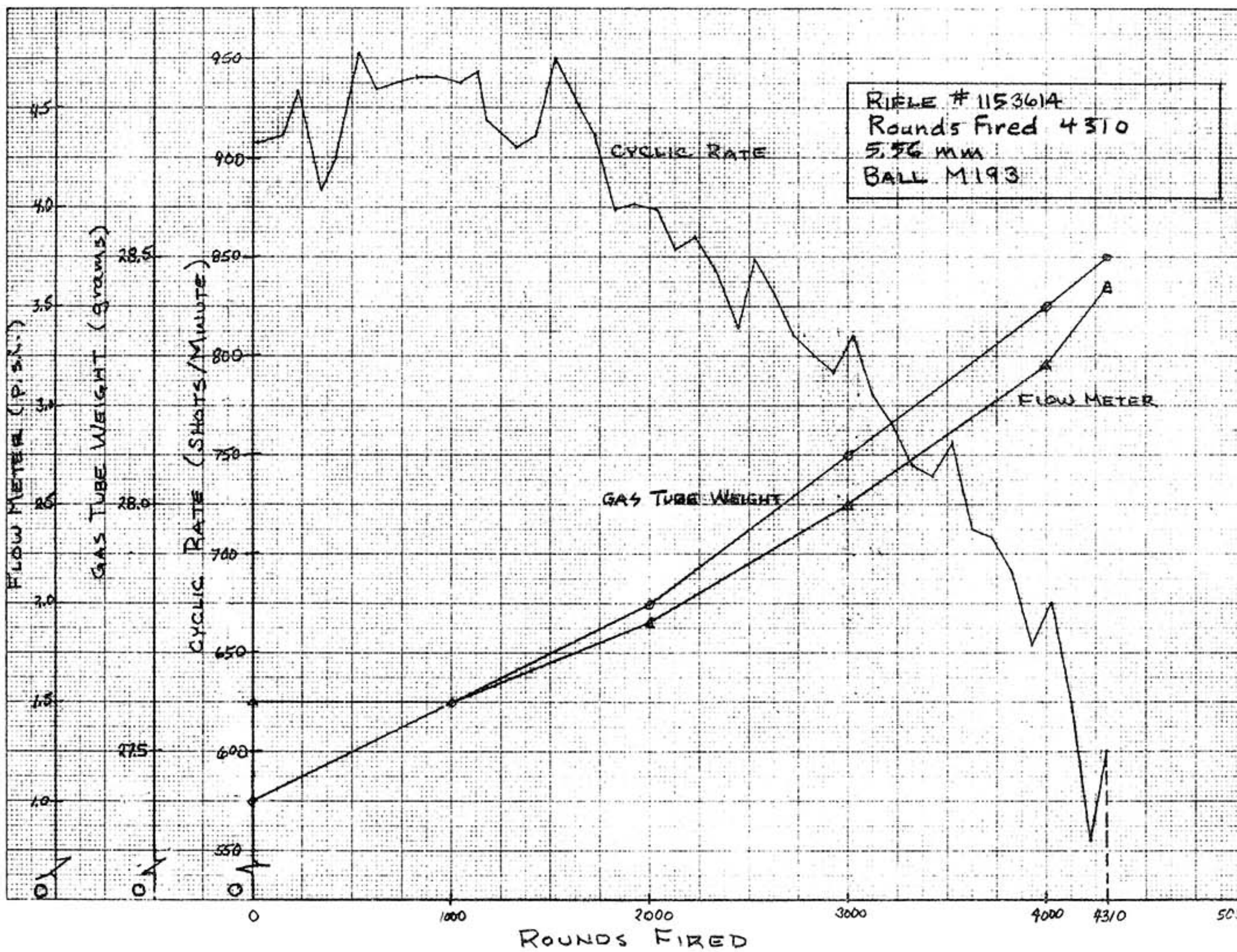
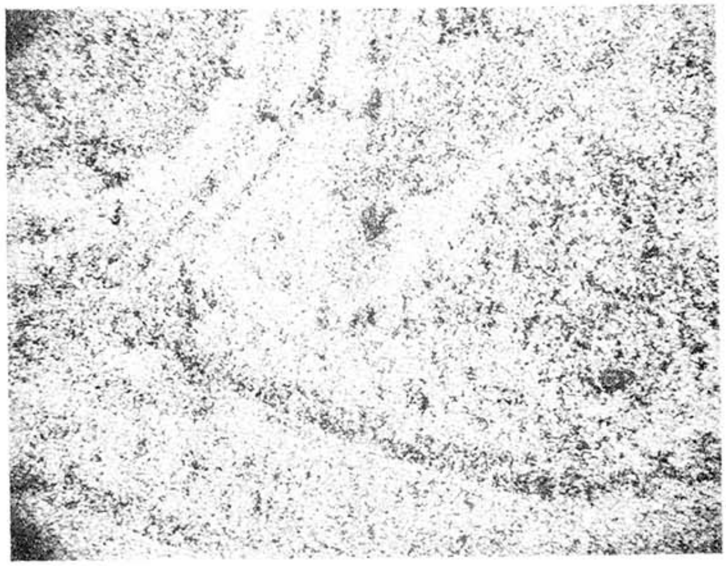


Figure 9. Performance Characteristics, Gas Tube



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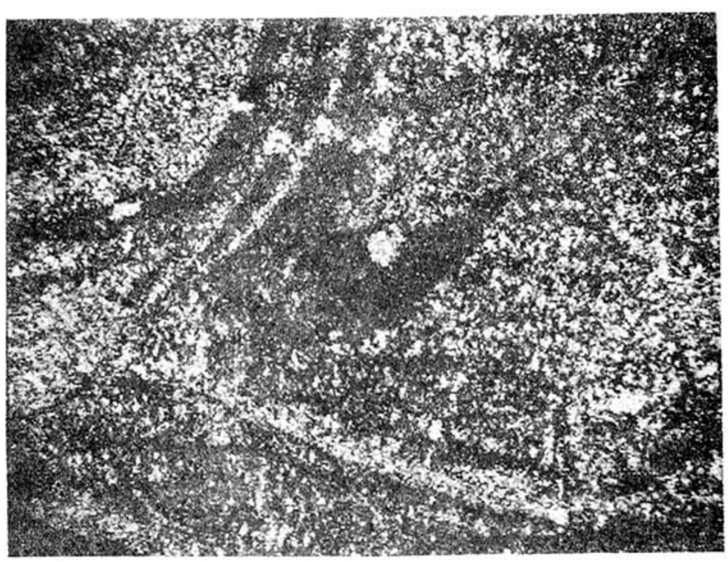
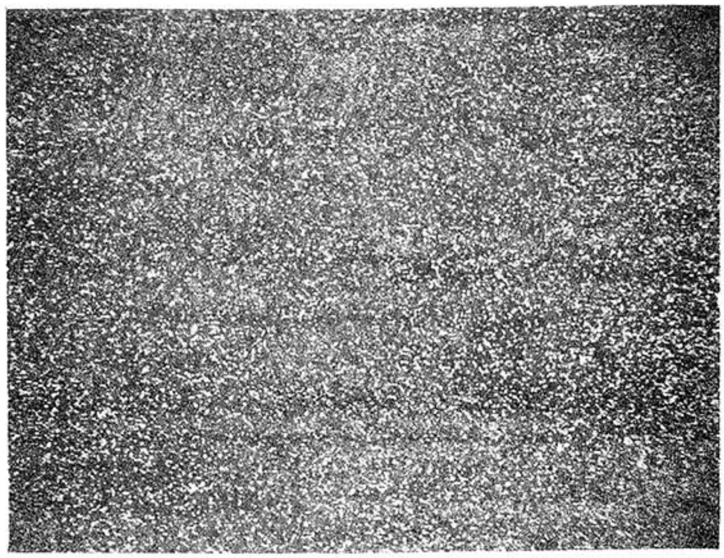
Calcium



Mag: 200X

Copper

Figure 10. Microprobe of Ca and Cu Content of Residue in Gas Tube



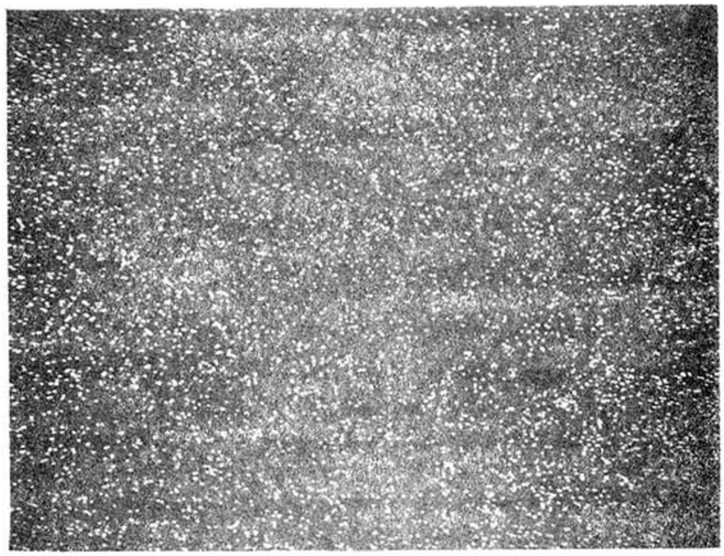
Mag: 200X

Barium

Mag: 200X

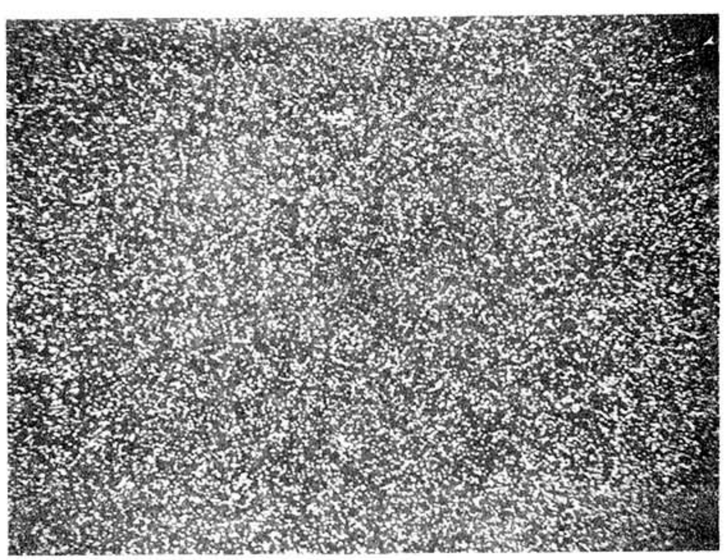
Antimony

Figure 11. Microprobe of Ba and Sb Content of Residue in Gas Tube



Mag: 200X

Zinc



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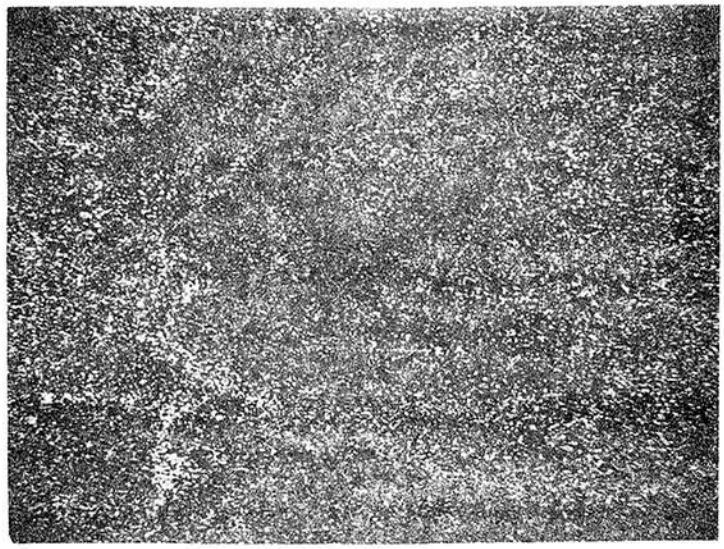
Aluminum

Figure 12. Microprobe of Zn and Al Content of Residue **in Gas Tube**



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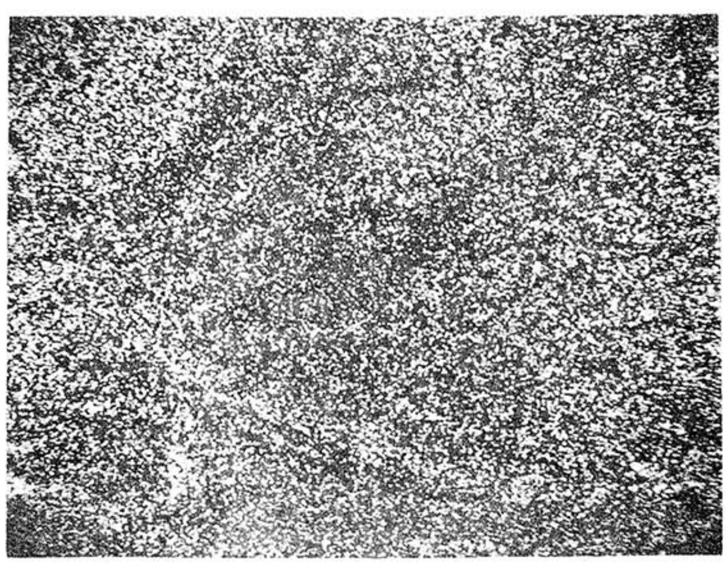
Lead



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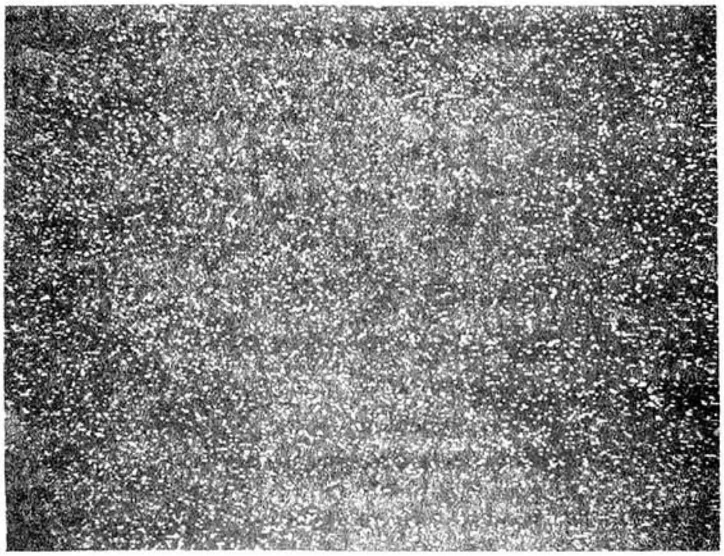
Sulfur

Figure 13. Microprobe of Pb and S Content of Residue in Gas Tube



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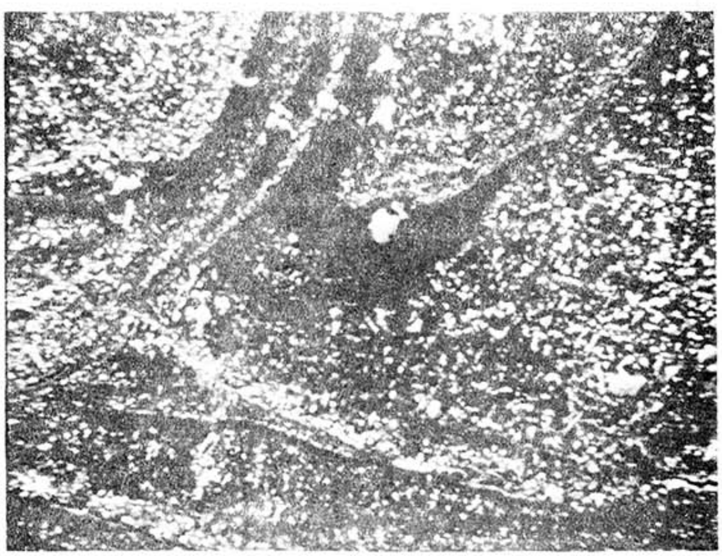
Iron



Mag: 200X

Backscatter Image

Figure 14. Microprobe of Fe Content and Backscatter Image of Residue in Gas Tube

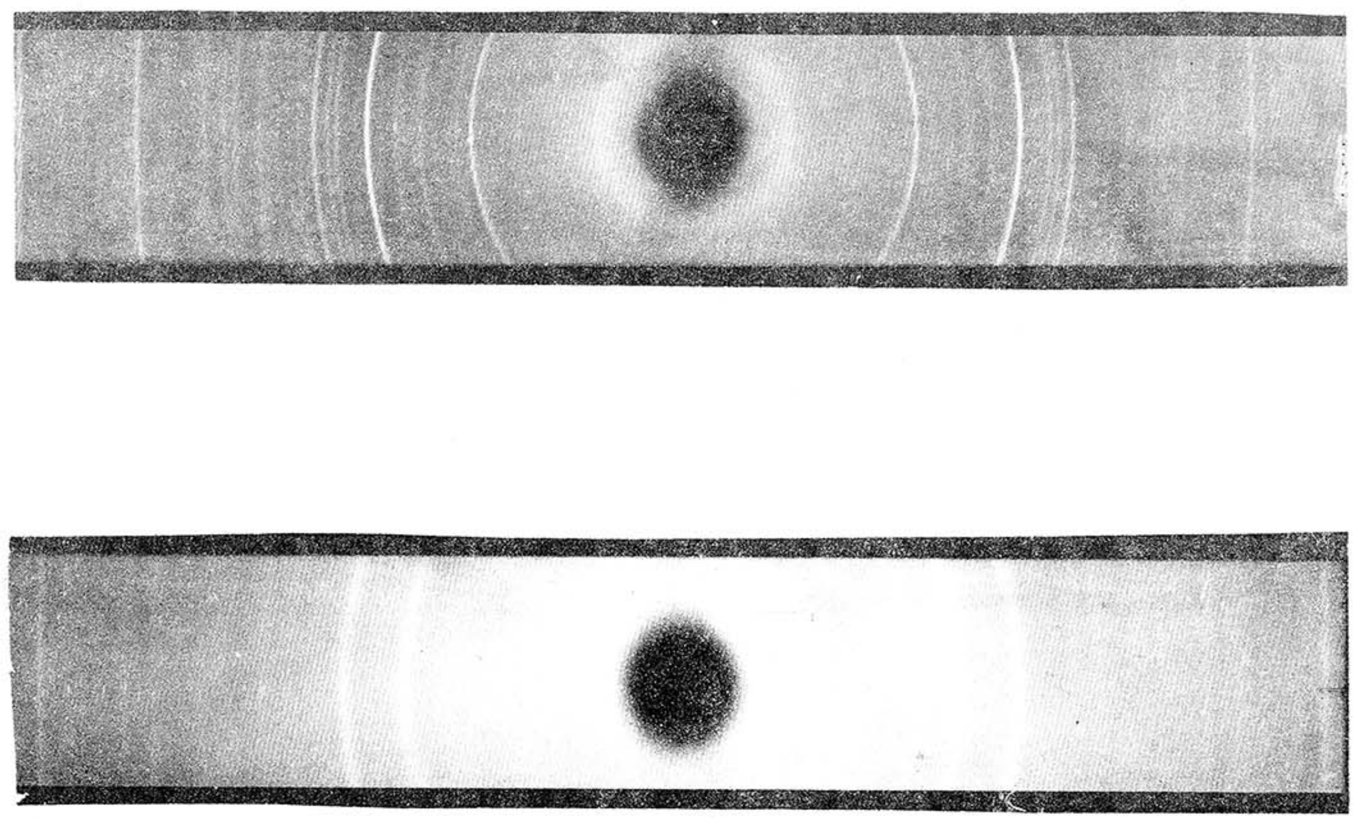


Figure 15. X-ray Diffraction Pattern of Powder Residue from the 2.6-in. Location of Gas Tube

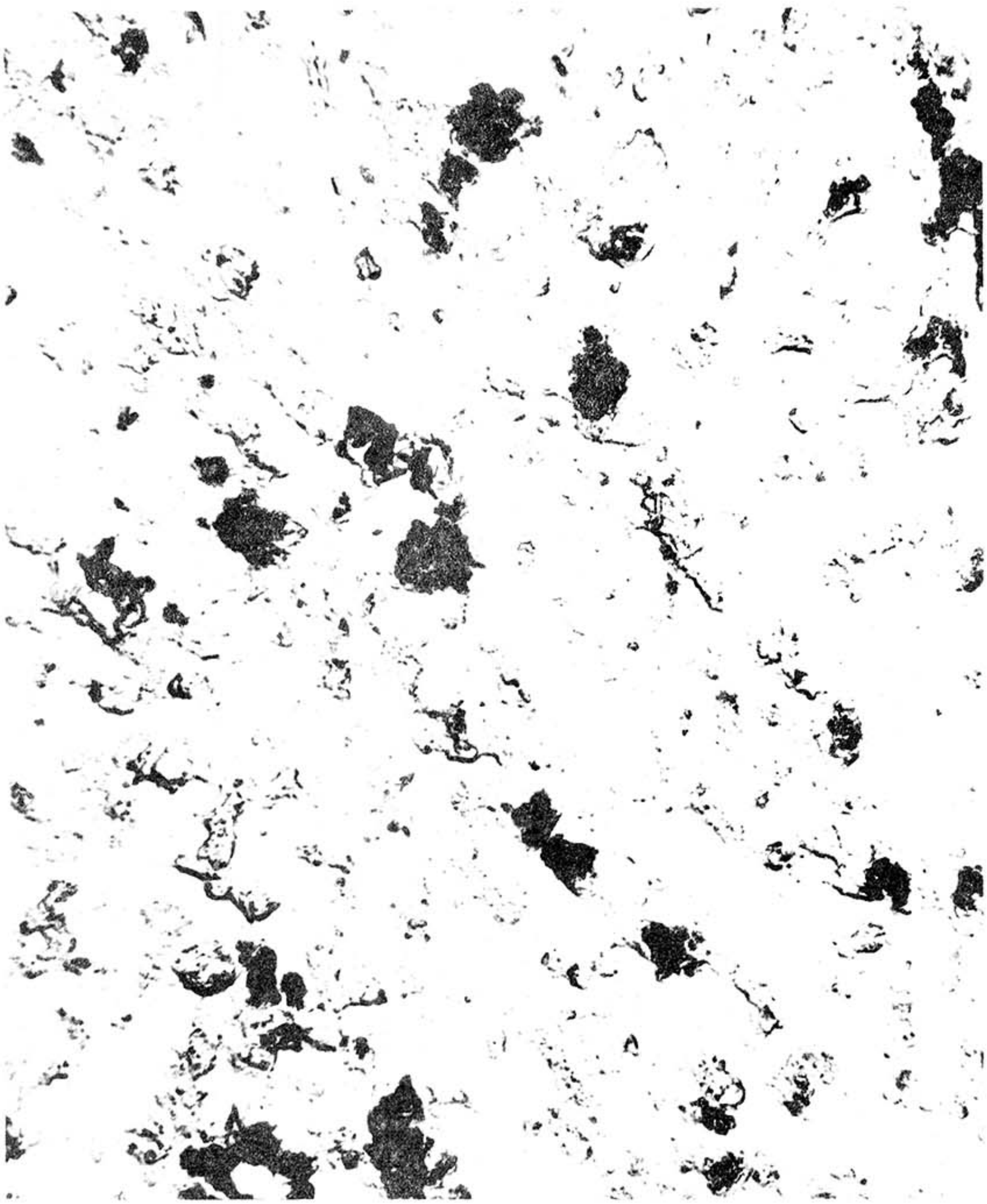


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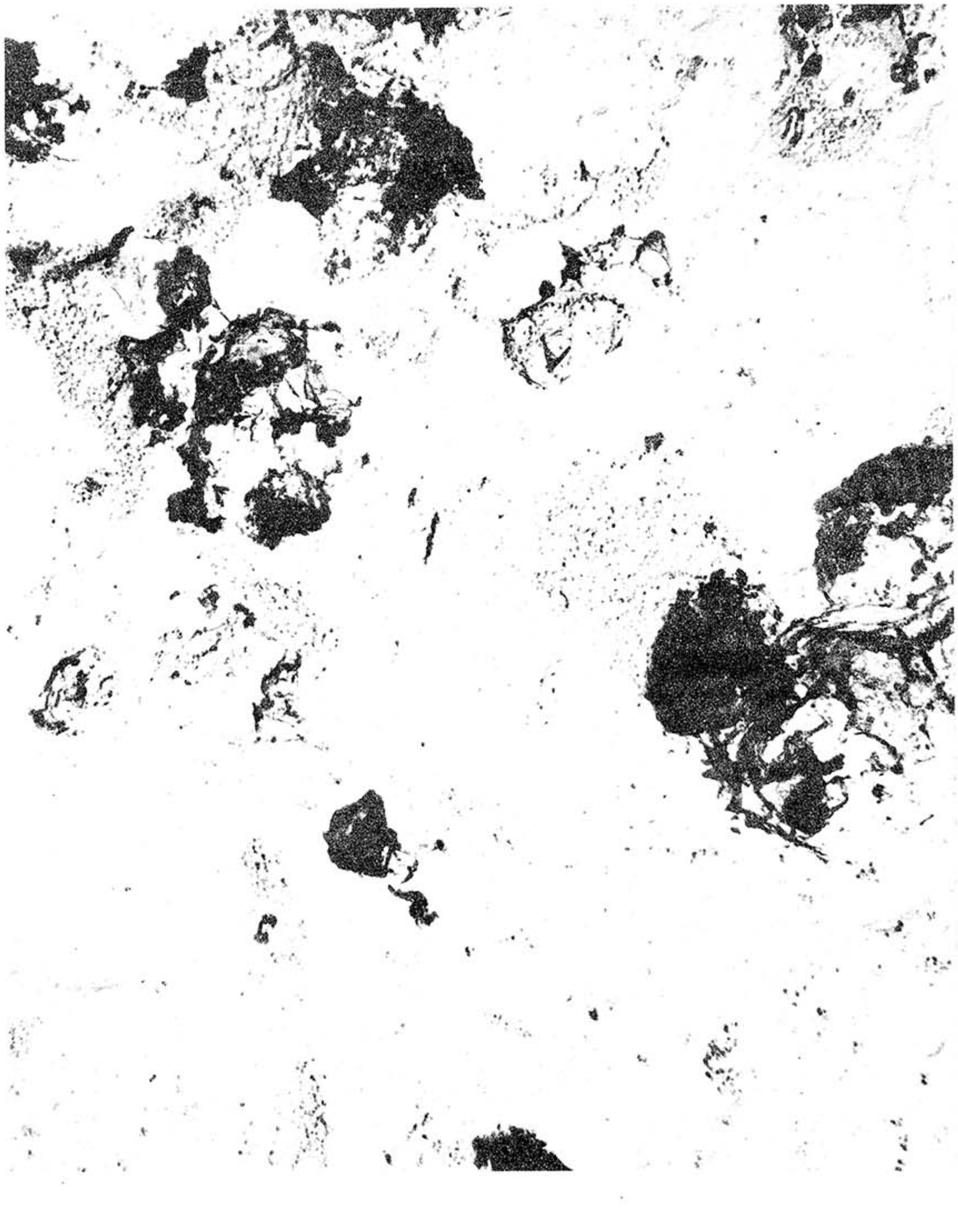
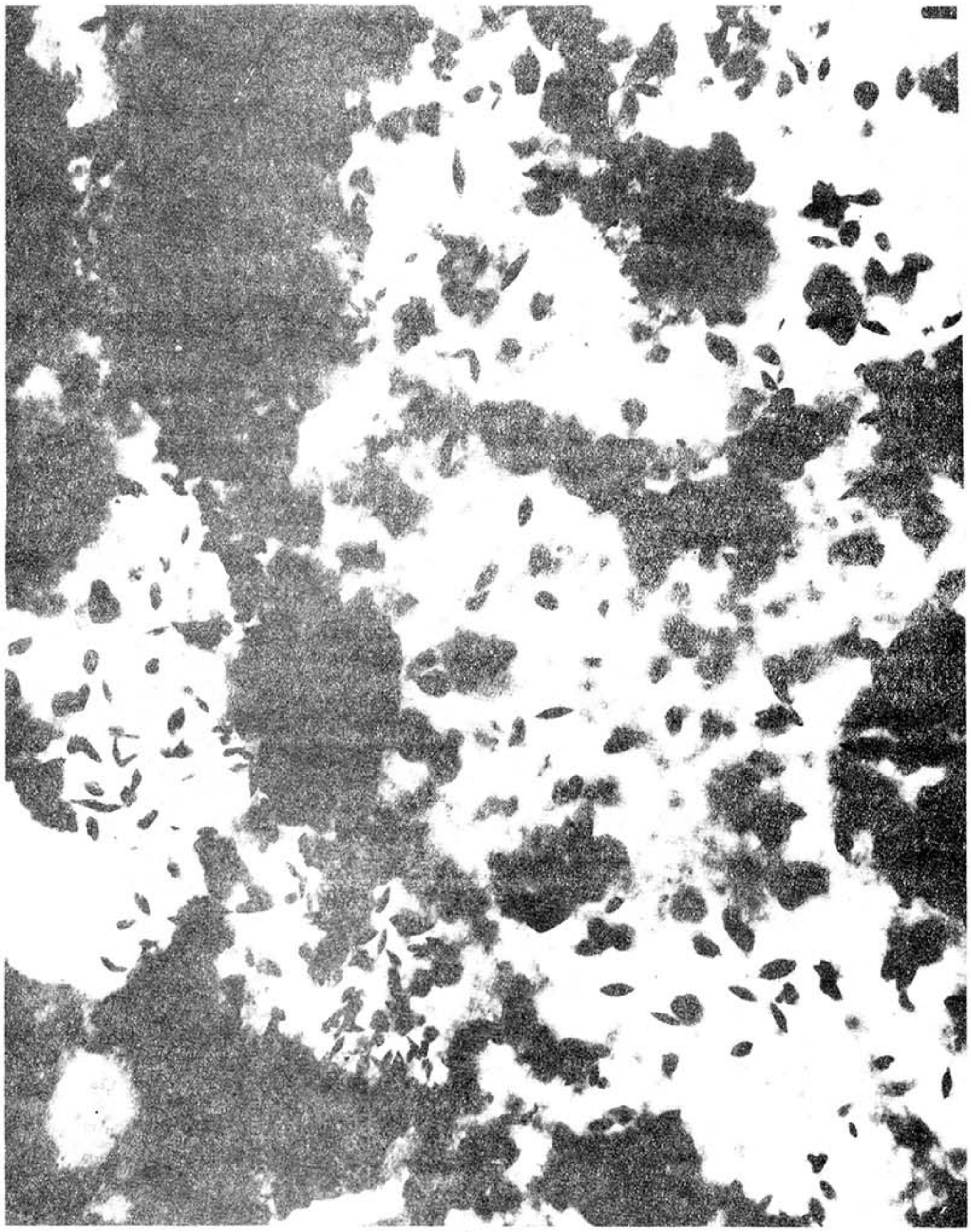
Figure 16. Electron photomicrograph of Deposit in Flash Suppressor



Mag: 20,000X

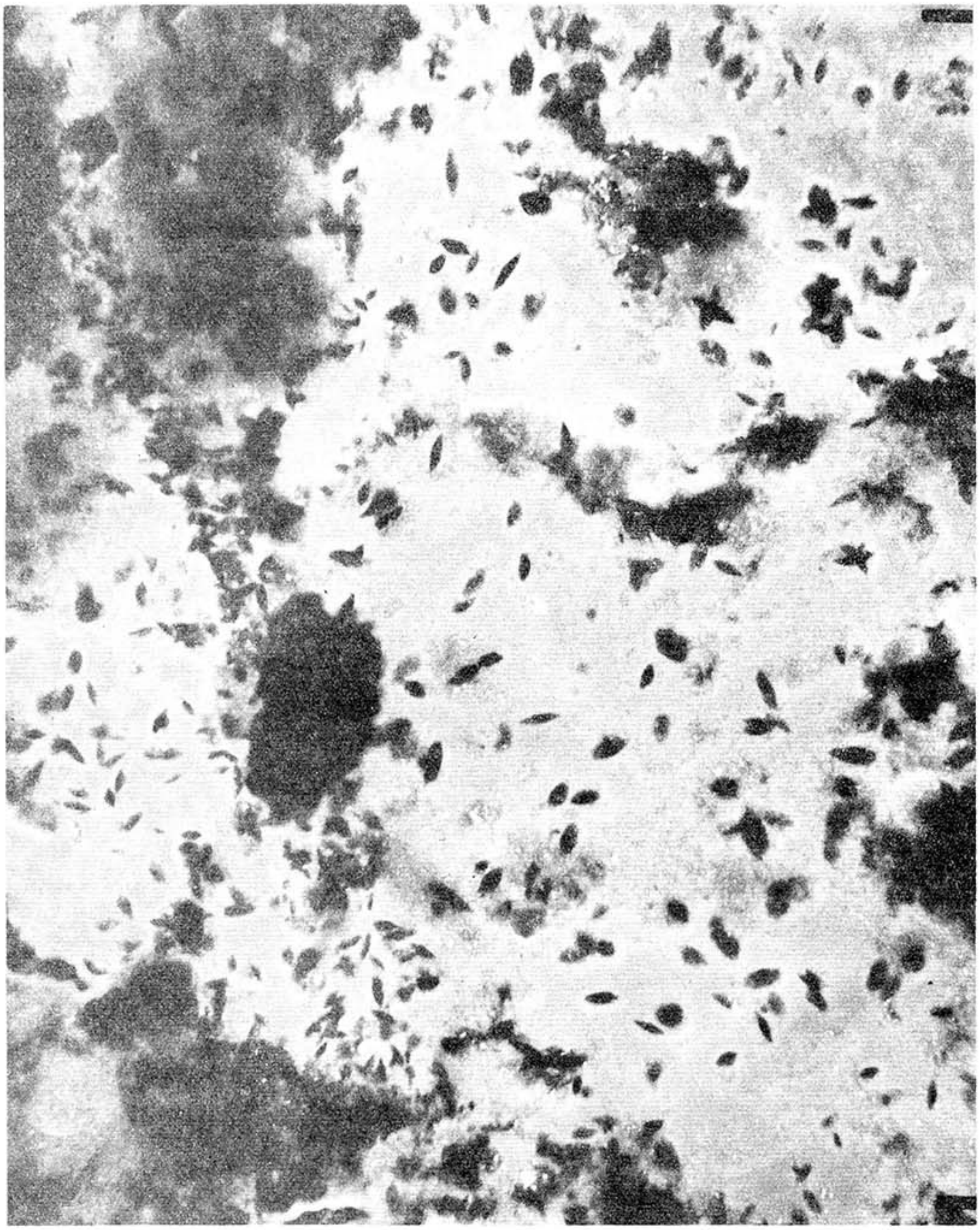
Figure 17. Electron photomicrograph of Deposit in Gas Tube



Mag: 20,000X

Figure 18. Electron photomicrograph of Deposit in Flash Suppressor after Repolishing and Etching in Dilute HCl

Figure 19. Diffraction Pattern of Residue in Gas Tube



Mag: 20,000X

Figure 20. Dark Field Image of Area in Figure 18

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13. ABSTRACT

The metallurgical investigation of the residue extracted from the fouled gas tube and the flash suppressor of an M16A1 rifle involved: (1) metallographic examination of the contact surface, which showed incipient cracks in the gas tube wall (which were of no significance); (2) electron microprobe identification and distribution of the particles, which showed copper-zinc particles randomly distributed in a calcite matrix; (3) electron microscope studies of particle size and shape, which showed the particles to be about 0.3 micron in size and irregular in shape; (4) x-ray examination of crystal structure and composition, which showed calcite with a diffraction pattern that coincided with that of hexagonal $CaCO_3$, and a copper-zinc face-centered cubic alloy with lattice constant corresponding to 90% Cu-10% Zn.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Rifle Fouling Gas Tube Residue Flash Suppressor Residue Residue in M16A1 Rifle M16A1 Rifle						