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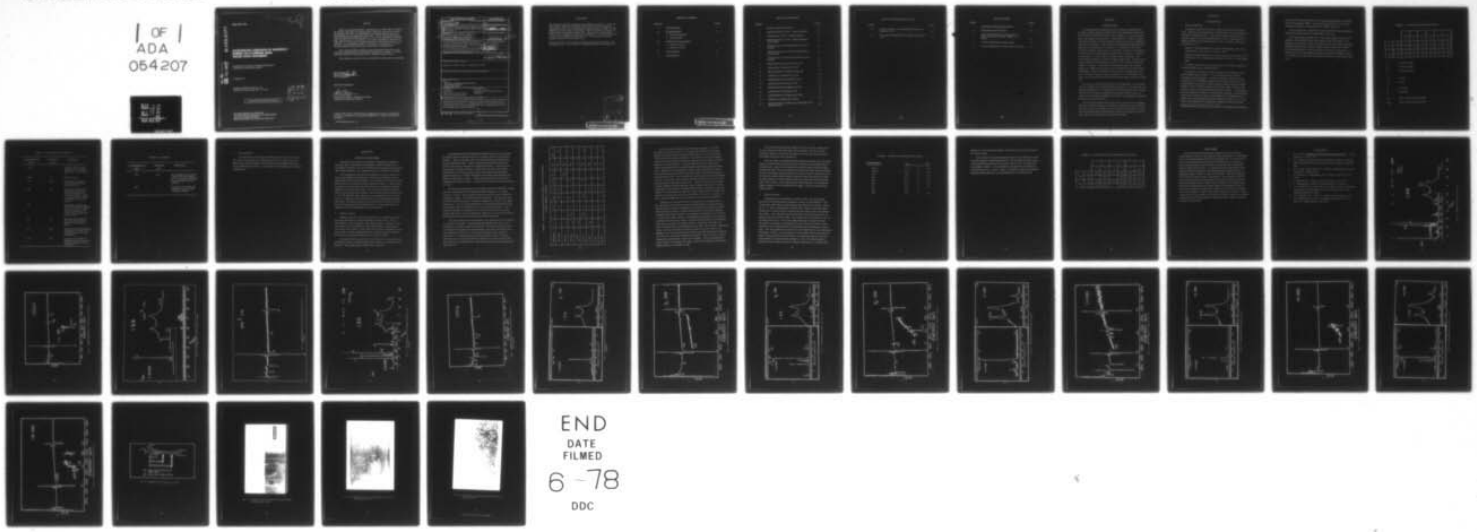

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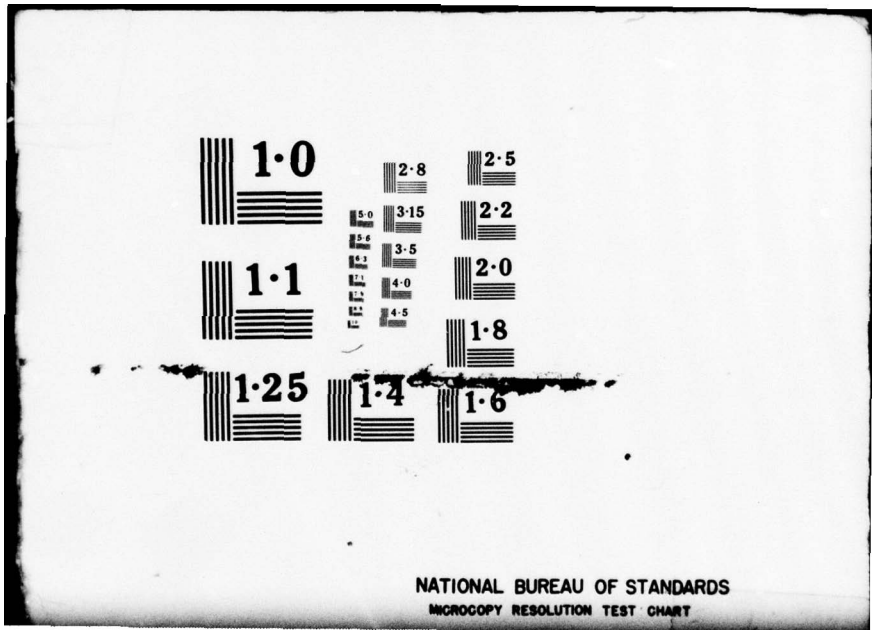
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AFML-TR-77-184

ACCELERATED CORROSION OF ADHESIVELY BONDED 7075 ALUMINUM USING WEDGE CRACK SPECIMENS

MECHANICS AND SURFACE INTERACTIONS BRANCH
NONMETALLIC MATERIALS DIVISION

OCTOBER 1977

TECHNICAL REPORT AFML-TR-77-184
Final Report for Period January 1976 – June 1977

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AIR FORCE MATERIALS LABORATORY
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This technical report has been reviewed and is approved for publication.

N. T. McDevitt

FOR THE DIRECTOR

S. W. Tsai, Chief
Mechanics & Surface Interactions Branch
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Accelerated corrosion of 7075 aluminum using the equal thickness adherend wedge test did not find flaws on the anodic oxide surface through the use of scotch tape contamination. It is apparent that the strength of the adhesive-oxide interface has to be much less than the center of the bond line in order for the wedge test to find the weakness.

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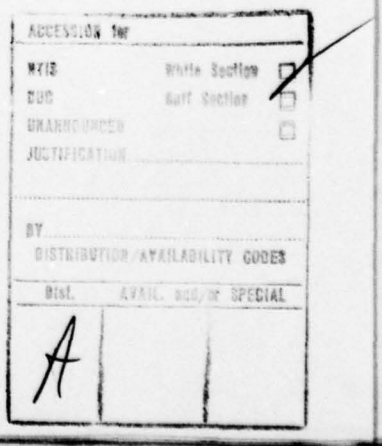
FOREWORD

This technical report was prepared by Neil McDevitt, William L. Baun and Gary Fugate, Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Air Force Materials Laboratory (AFML/MBM), Wright-Patterson Air Force Base, Ohio, and Mr. James S. Solomon, University of Dayton Research Institute, Dayton, Ohio. The work was initiated under Project 2419, "Nonmetallic and Composite Materials", and was administered by the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

This report covers work conducted inhouse during the period January 1976 through June 1977. The report was released by the author in October 1977.



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SECTION I

INTRODUCTION

The ultimately desired data base for all corrosion or durability tests on metal-to-metal adhesively bonded joints is the information obtained from these materials while used in actual service conditions. A data base generated in this manner may take years to reach maturity. In order for laboratory tests to generate a valid data base they must come close to reproducing actual service conditions. This requires the knowledge and measurement of many factors controlling service behavior: (1) metallurgy of the metal; (2) surface chemistry of the metal; (3) chemistry of the primer and adhesive; (4) chemistry of the condensates that collect at the interfaces; (5) temperature variations; (6) cyclic nature of wetting, drying, irradiation; and (7) the amount and types of stress the bondline experiences. This represents a large number of test parameters to control. This is usually difficult and expensive; therefore, accelerated test methods that deal with a smaller number of variables have become accepted as standards by both the military and industry. These test methods are too numerous to explain here; however, a number of extensive reviews (References 1-6) are available on the subject of corrosion and can provide the interested reader with more information.

The value of accelerated testing will not be misleading if the researcher realizes it applies only to precisely defined conditions, and it generates only qualitative information. The usefulness of the data depends on the researcher and directly on the types of conditions and variables that he chooses. If chosen correctly a good accelerated test program can highlight the important parameters and discount others.

This particular program was designed to fill a need for more complete data on the interface normally overlooked in a bonded joint, the oxide-metal interface. Data on this interface are quite limited in the literature, so several environments used in this study were chosen in an attempt to preferentially attack the chemistry of the oxide/metal interface.

SECTION II

EXPERIMENTAL

1. Surface Preparation

Rectangular panels were cut from a sheet of bare 7075-T6 aluminum alloy. Each panel was approximately 215-millimeters long, 100-millimeters wide, and 3.2-millimeters thick. The panels were acetone wiped, ultrasonically cleaned with carbon tetrachloride for 5 min., submerged in 0.1N sodium hydroxide at room temperature for 2 min., and then acid pickled with solution A or B.

Solution A: panel submerged in a solution of 300g. H_2SO_4, 40g. CrO_3 distilled water to one liter, for 10 min. at 60°C.

Solution B: panel submerged in a solution of 170 ml. nitric acid, 30 ml. hydrofluoric acid, distilled water to one liter for 2 min. at room temperature.

All panels were rinsed in running tap water for 15 minutes followed by a 5-minute standing rinse in deionized water.

The panels were anodized in pairs according to the conditions of each matrix number shown in Table 1. A 2-inch square control specimen accompanied each large panel. In order to introduce a known failure site at the crack tip of the wedge specimen (Figure 1) eight of the panels were contaminated in various ways. Pickle solution B usually causes a smutted* surface on 7075-T6 with the conditions used in this study. This smut was allowed to remain on the panels marked 13 and 14 in Table 1. These panels were then anodized according to their designated conditions. Panels marked 6 and 18 were allowed to remain in the phosphoric acid electrolyte for six minutes after the anodization voltage was turned off. Panels 2, 10, 14, and 22 were contaminated after anodization by applying a strip of scotch tape

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* With this alloy it is usually a form of cupric oxide replated on the surface.

across the 215-millimeter surface of the panel approximately in the middle of the 100-millimeter-width. It was then removed after several minutes. All of these panels are considered to have "doped" surfaces.

A pair of panels was bonded after each acid pickle with no anodization. The data from these panels are designated as P_A or P_B.

Each duplicate pair of panels was then bonded together with FM 123-2 adhesive, cured at 250°F and 25 psi, without the aid of a primer. The bonded panels were then cut into 25-millimeter wide specimens giving eight individual specimens for each test number shown in Table 1 for a total of 192 anodized specimens, plus 16 specimens for the P_A and P_B series. A description of each environmental test and its duration is outlined in Table 2. At least one specimen from each numbered matrix was subjected to these tests.

TABLE 1. Test Matrix Anodization Parameters

		C_1		C_2		C_3	
		V_1	V_2	V_1	V_2	V_1	V_2
P_A	t_1	1	3	5	7	9	11
	t_2	2	4	6	8	10	12
P_B	t_1	13	15	17	19	21	23
	t_2	14	16	18	20	22	24

C_1 = 0.5 Molar H_3PO_4

C_2 = 1.0 Molar H_3PO_4

C_3 = 2.0 Molar H_3PO_4

V_1 = 10 volts

V_2 = 20 volts

t_1 = 10 minutes

t_2 = 20 minutes

P_A = pickle solution A pretreatment

P_B = pickle solution B pretreatment

TABLE 2. Environmental Test Conditions

ENVIRONMENTAL TEST	DURATION (hrs)	CONDITIONS
168-A	5	Steam at 108°C, sealed container. Heat cycled, 15 minutes on, 30 minutes off.
168-B	10	Same as above.
169	960	Atmospheric conditions varied between 10-45°C, and 60-95% R.H.
208	86	Specimens from TEST 169 subjected to carbon tetrachloride atmosphere in enclosed container. Vapor in equilibrium with boiling liquid (75°C) in enclosed container.
218	3	Atmosphere consisting of carbon tetrachloride/methanol vapor in 2 to 1ratio. Heat cycled 15 minutes on, 45 minutes off in enclosed container, (70°C).
223	18	Specimens from TEST 218 placed in sealed container and subjected to steam at 108°C.
237	128	Carbon tetrachloride/methanol atmosphere, 5:1 ratio, in an enclosed container at room temperature.
243	192	Specimens from TEST 237 subjected to water vapor in an enclosed container at room temperature.

TABLE 2. (Continued)

ENVIRONMENTAL TEST	DURATION (hrs)	CONDITIONS
258	168	SO_2 atmosphere in enclosed container at room temperature. Atmosphere generated from saturated solution of $NaHSO_3$.
088	5	Specimens from TEST 258 subjected to steam (108°C) in sealed container.

2. Instrumentation

The identification of the major elemental species present on the surface of the test specimens was obtained through the use of Ion Scattering Spectroscopy (ISS), Secondary Ion Mass Spectroscopy (SIMS), and Auger Electron Spectroscopy (AES). A description of these techniques is reported in Reference 7.

SECTION III

RESULTS AND DISCUSSION

Considerable care must be exercised in conducting and evaluating laboratory tests. Only after an accumulation of sufficient experience in correlating accelerated corrosion test data with actual performance behavior, under known conditions, can a significant degree of confidence be justified.

The following data represent the preliminary step in the pursuit of a better understanding of the chemistry of surfaces and interfaces and how they interact with harsh environments. The interaction of the surfaces in the bonded joints were evaluated using the wedge test (Figure 1). After driving the wedge into one end of the test specimen the crack length was allowed to stabilize over a period of two hours. The value for X was then measured (Figure 1) and the specimens exposed to the environments described in Table 2. The test specimens after exposure to an environment fall into two categories: (1) specimens that failed adhesively, and (2) specimens that failed cohesively. Instrumental analyses were performed on the metal oxide surface of all the specimens that failed adhesively. Crack growth data were obtained for all of the specimens that failed cohesively.

1. Adhesive Failures

Adhesive failure can best be described as the resin pulling away from the metal oxide-adhesive interface exposing the metal surface. This is determined by a visual inspection of the bonded surfaces. Analysis of these two interfaces by ion scattering or Auger Spectroscopy can usually detect resin on the oxide surface or oxide on the resin surface, indicating the resin wet the oxide. Strictly speaking the failure is usually interfacial; however, for the sake of discussion we shall call it an adhesive failure.

The panels marked P_A and P_B were not anodized. The P_A panels were treated with solution A and these panels failed adhesively in the majority of the tests. The P_B panels failed adhesively in every test.

Interpretation of the instrumental data obtained from the failed wedge specimens requires reference data on the cleaned, pickled, and anodized surfaces. Figures 2 through 7 show the instrumental data obtained from the pre-anodized surfaces. Table 3 summarizes the major elements detected on the surface of the various analyzed specimens. A description of the specimens in Table 3 follows: (1) 7075-Alk, data from surface after Alkaline etch; (2) 7075 P_A and P_B, data from surfaces after having the appropriate pickle; (3) 2-258, the number 2 represents the anodization parameters as described in Table 1, #258 represents the environmental test conditions described in Table 2. 6-258 and 18-258 follow the same description as 2-258.

The alkaline treatment leaves a detectable amount of magnesium, copper, and zinc on the surface of 7075 aluminum (alloy composition - 2.5% Mg, 1.6% Cu, 5.6% Zn). The P_A pickle solution removes the zinc but leaves the magnesium, copper, and a small amount of chromium. The P_B pickle solution leaves detectable amounts of fluorine, magnesium, calcium, and copper (Reference 7). We have shown (Reference 8) that chemical elements present in the surface layer can find their way into the anodic oxide layer. These elements could play an important role in the electrochemistry of the final surface film from a corrosion standpoint; therefore, it is necessary to know what elements are present on each surface before anodization.

The crack growth (Y) was measured for each bonded joint after exposure to a specific environment. The specimens failing adhesively were all examined with our surface analysis instruments. The data presented in Table 3 and Figures 8 through 17 are representative of the adhesively failed specimens obtained by exposure to environmental conditions described under #258 Table 2. The five bonded specimens annotated in Table 3 all failed adhesively under all the environmental conditions described in Table 2 with the exception of environment #169. Specimen P_B was the only failed specimen after test 169.

TABLE 3. Major Element Detected At the Surface of 7075-T6 Aluminum by ISS(I), SIMS(S), and AES(A)

SPECIMEN	C	F	Na	Mg	K	Ca	Cn	Cu	Zn
7075-ALK			S	$^{I}S_{A}$	S			$^{I}S_{A}$	A
7075-P_A	A			S			$^{I}S_{A}$	$^{I}S_{A}$	
7075-P_B	A	$^{I}S_{A}$	S	^{I}S		S		$^{I}S_{A}$	
P_A-258	A		S	A				A	
P_B-258		$^{I}S_{A}$	S	S_A		S		$^{I}S_{A}$	A
2-258	S_A		S		S			$^{I}S_{A}$	
6-258			S		S			S_A	
18-258			S	A	S			S_A	A

Figure 18 shows an adhesively failed wedge specimen. The wedge direction is shown by the arrow. This specimen (2-258) had scotch tape applied to the oxide surface as described previously. The area designated A shows where the tape caused failure. The initial stress from the wedge caused the crack to progress directly through this area and stop at B before exposure to the environment. An Auger analysis of spot A shows heavy carbon contamination and very small intensity peaks from oxygen and aluminum. Analysis of a spot opposite A (adhesive side) showed only carbon.) This indicates the resin never wet the oxide surface due to the carbon layer present from the tape. Once the adhesive (interfacial) failure was established in area A it remained at this interface and when exposed to the # 258 environment the failure continued through B to C. Figures 12 and 13 show the spectral data obtained from this specimen using a sample from area B. Carbon, copper and a normal amount of oxygen and aluminum were found in this area. Analysis of a spot opposite B (adhesive) shows a small amount of oxygen and aluminum indicating the resin did wet the oxide in this area. An analysis of spot C shows qualitatively the same elements as detected from sample B.

Samples were obtained from all of the adhesively failed specimens in the same manner as shown in Figure 18 and described in the previous text. The data shown in Table 3 qualitatively represents the average for all of the other specimens analyzed. Sodium and potassium are detected by SIMS on a number of specimens; however, it is difficult to determine its origin since it is prevalent in most solutions, and handling the specimens can be a source of sodium even when care is taken. Calcium is most notable in the SIMS data when there is fluoride in the pickle solution. Magnesium and copper are found in the Auger data for failed specimen P_A-258, while fluorine, magnesium, copper, and zinc are found on the failed surface of P_B-258. Matrix test specimens 2 and 6 were pretreated with pickle solution A and the failed surfaces of 2-258 and 6-258 both have detectable amounts of copper present; however, magnesium is not detected. Matrix test specimen 18 was pretreated with pickle solution B and magnesium copper, and zinc are detected, primarily by Auger analysis, on specimen 18-258.

Of all the elements observed copper is the only one that is detected on all of the adhesively failed specimens that we studied. We do not wish to imply that copper is the cause of the failure since the data presented here is not capable of giving us that information.

The above data was obtained from the adhesively bonded side of the joint. Figures 19 and 20 show the effect of the environment on the unbonded side of the bulk metal. Figure 19 was exposed to test environment 218 while Figure 20 shows the effect of test environment 258 on the bulk metal anodized surface. The 218 environment containing chloride ion shows extensive pitting corrosion. The 258 environment shows extensive large pits with a white powder growth. An Auger analysis on 18-258 (bulk) showed only oxygen and aluminum energy peaks, but no sulfur. This would indicate the white powder is an aluminum-oxygen compound. This compound did not appear to grow on the adhesively failed surfaces.

2. Cohesive Failure

Cohesive failure by definition is a failure in the resin and an equal amount (by visual observation) of adhesive remains on each adherend. This section deals only with the anodized panels that failed in this manner. Panels 10, 13, 14, and 22, although their surfaces were "doped" to fail adhesively, failed only in a cohesive manner and the data from these panels is included in this section. All of the data from the cohesively failed test specimens is reported as percent of crack growth. The average value for X (Figure 1) for all specimens was 50 mm. The crack growth data shown in Table 4 for each environment represents one specimen from each matrix box (Table 1) and is reported as the arithmetric mean of 24 anodized test specimens per environment. Since the population for each test is small these data are not statistically meaningful; however, it is of interest to see the effect of the various environments on these particular test specimens. Of particular interest is the zero crack growth in the environment annotated as # 258. This environment is apparently harsh enough to cause the "doped" specimens (2, 6, and 18) to fail

TABLE 4. % Crack Growth Data By Environment

ENVIRONMENT	A_M		S_D
168-A	9.7	±	2.3
168-B	32.3	±	3.6
169	2.6	±	1.1
208	22.0	±	3.4
218	21.7	±	4.7
223	56.7	±	7.5
237	18.6	±	4.5
258	0.0	±	
088	32.0	±	2.5

adhesively, but reacted only mildly to the specimens that normally failed in the cohesive mode.

Table 5 shows crack growth data (presented as the arithmetric mean) from the viewpoint of each test parameter. Environments that did not give crack growth data greater than 10 percent (#168-A, #169, and #258) were not included in Table 5. Also excluded was the data from the adhesively failed specimens (2, 6, and 18). Again it is difficult to apply any statistical meaning to these data due to the small population of test specimens.

TABLE 5. % Crack Growth Data For Each Matrix Parameter

		C_1		C_2		C_3	
		V_1	V_2	V_1	V_2	V_1	V_2
P_A	t_1	30.3	28.3	37.3	30.5	28.3	28.8
	t_2		29.0		24.4	28.2	31.2
P_B	t_1	29.0	34.5	39.5	42.5	30.3	27.2
	t_2	27.7	29.5		41.7	34.0	30.3

CONCLUSIONS

The only failures generated by this study were the ones induced by contaminating the surface or disturbing the anodic oxide surface. Surprisingly some of the contaminated surfaces did not fail adhesively. In fact, of the four surfaces contaminated with scotch tape, three did not fail. All of the surfaces prepared properly failed cohesively including the three contaminated surfaces mentioned previously. It is apparent from this study that the equal thickness adherend wedge test is not the appropriate testing procedure for studying the adhesive-oxide or oxide-metal interface. The stress generated by this test sees only the center of the bondline. We can speculate that the surface energy of the three contaminated surfaces that did not fail must have been large enough to form an integral bond. Therefore, the weakness introduced by the contaminant was not large enough for the wedge test to find it. This would indicate that strength of the interface has to be much less than the center of the bondline in order for the wedge test to point out the flaw. This is plausible since the geometry of the wedge test (Figure 1) generates primarily a Mode I failure and small differences between the center bondline and interface will not be observed.

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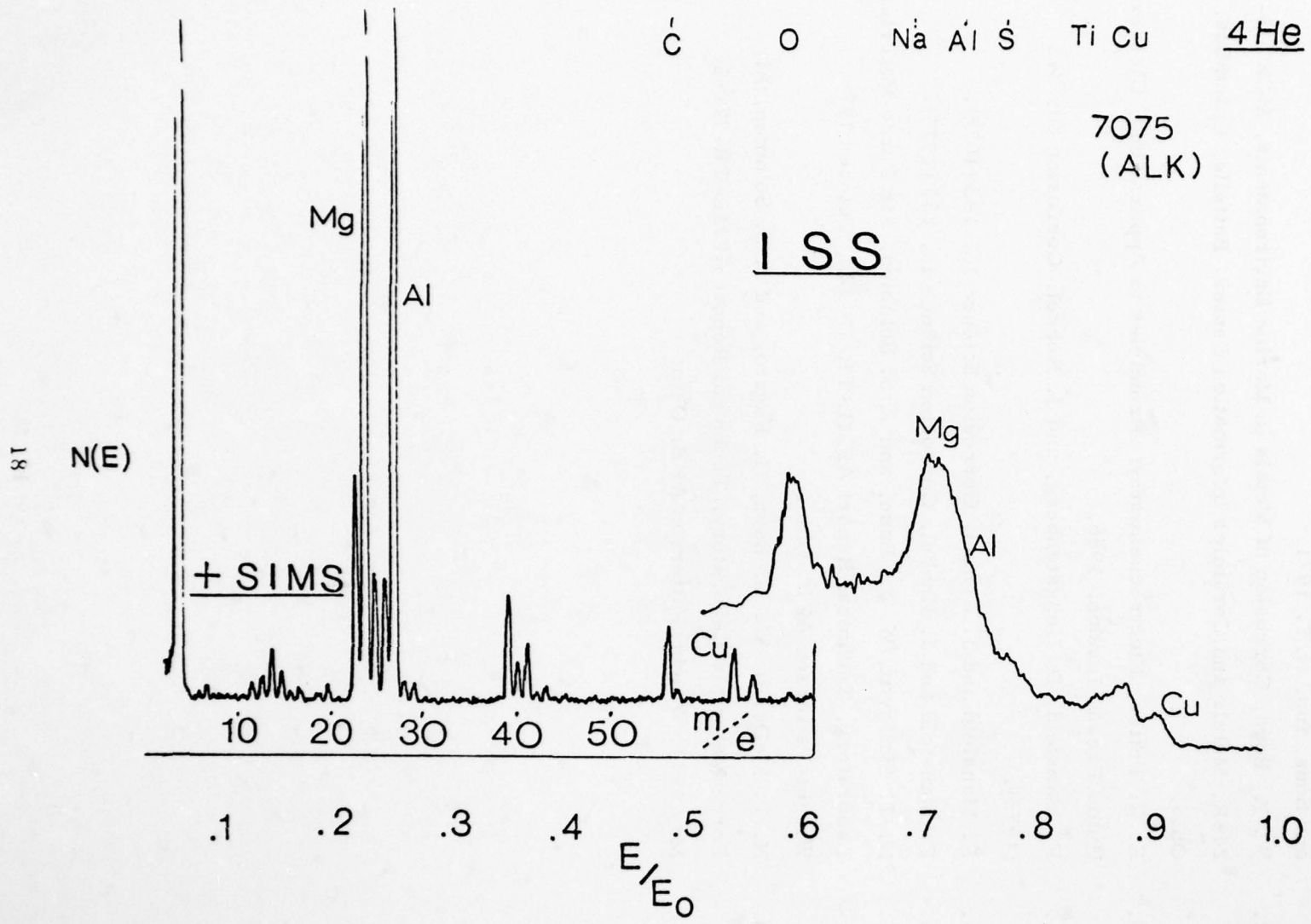

Fig. 1. Wedge Test Specimen Details.

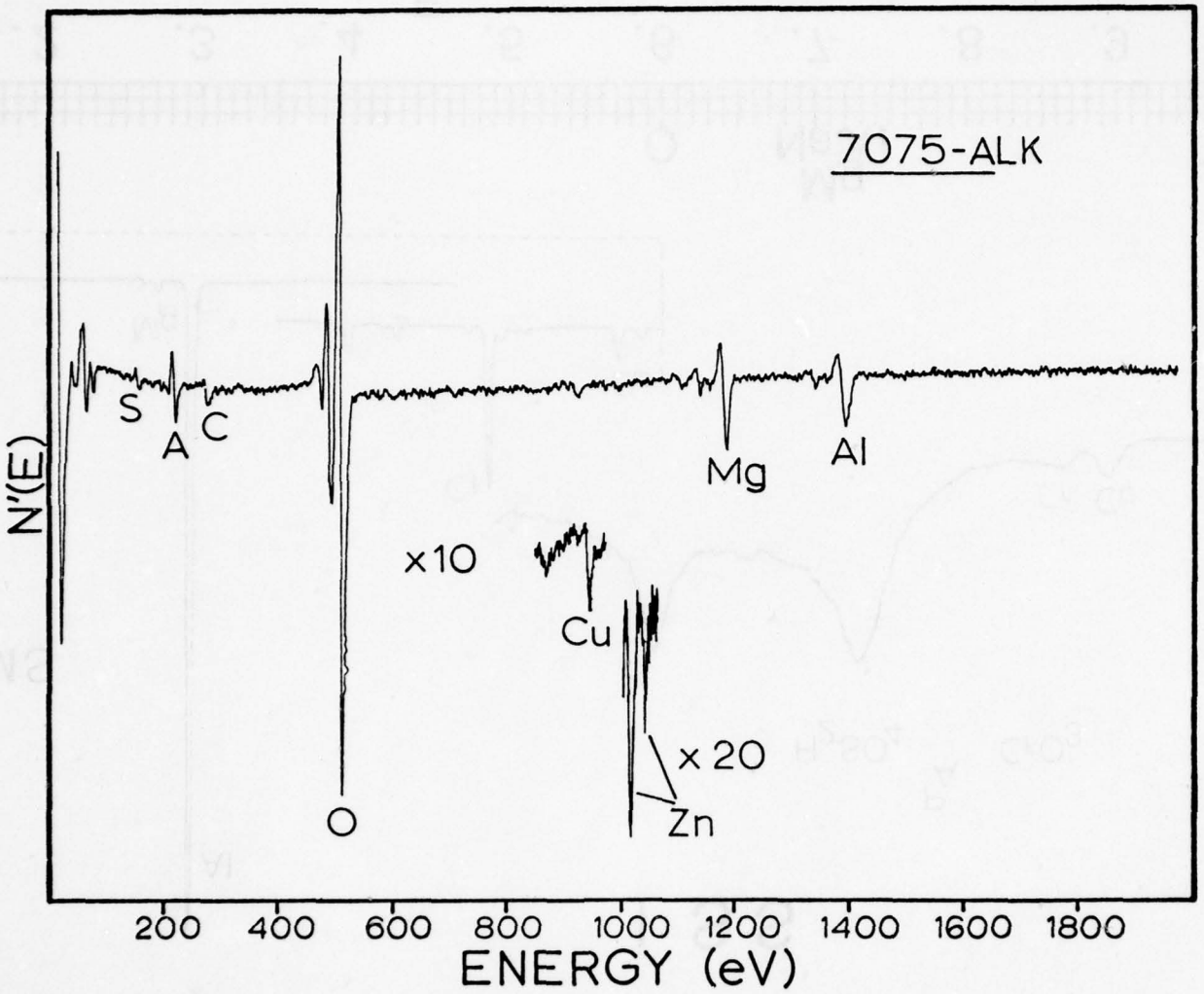

Fig. 2. SIMS-ISS Spectrum of 7075-Alkaline Cleaned.

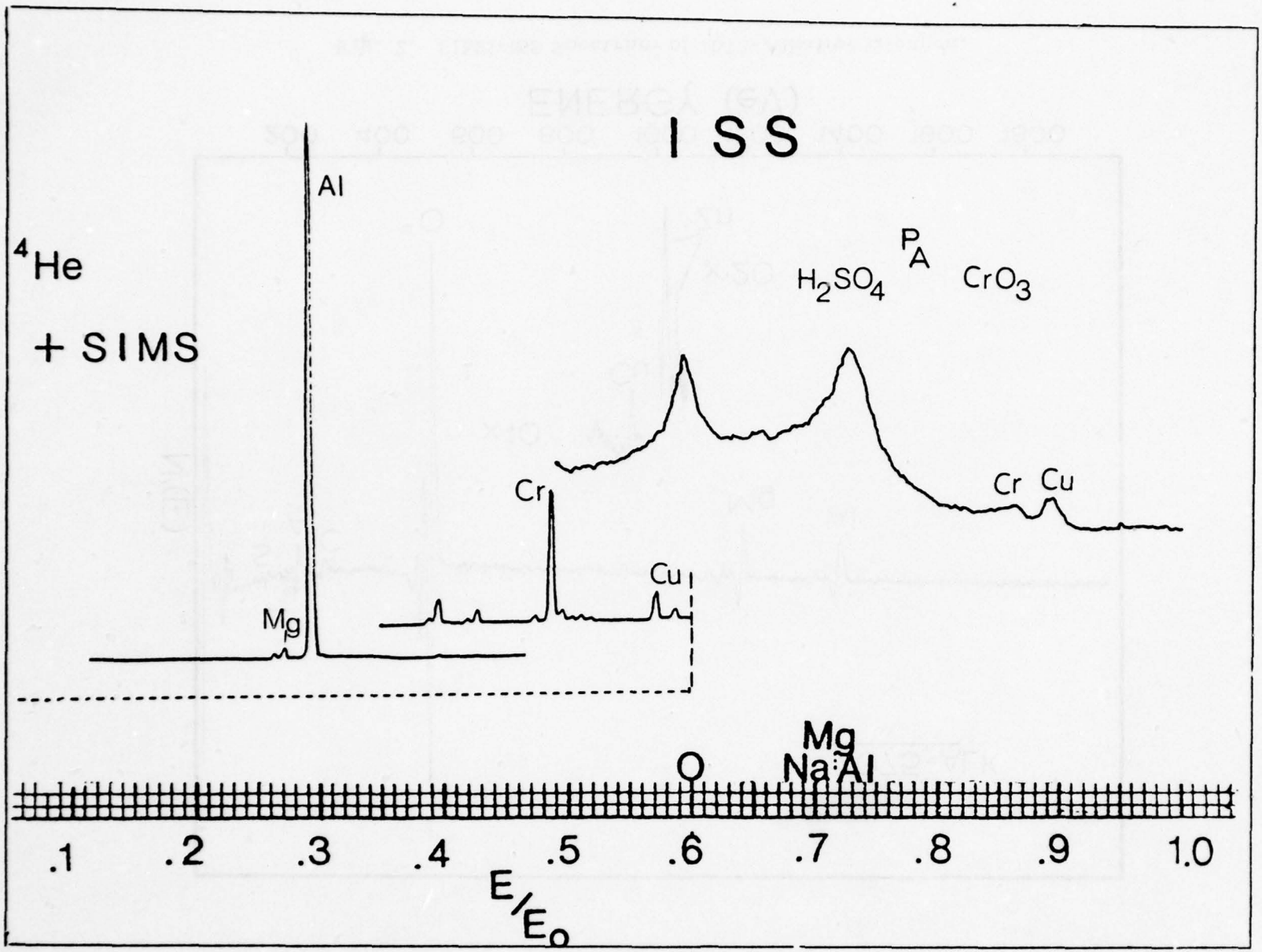

Fig. 3. AES Spectrum of 7075-Alkaline Cleaned.

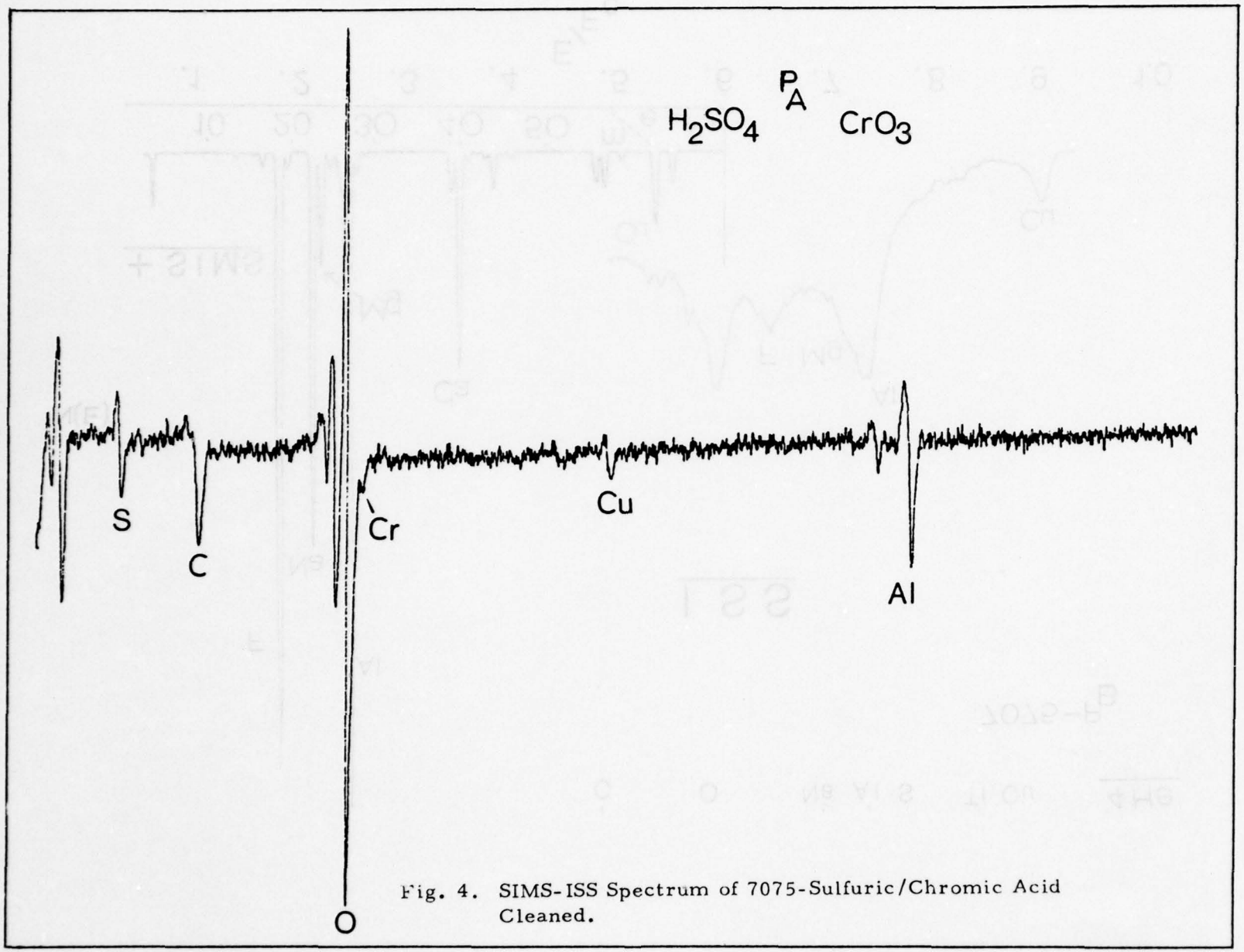

Fig. 4. SIMS-ISS Spectrum of 7075-Sulfuric/Chromic Acid Cleaned.

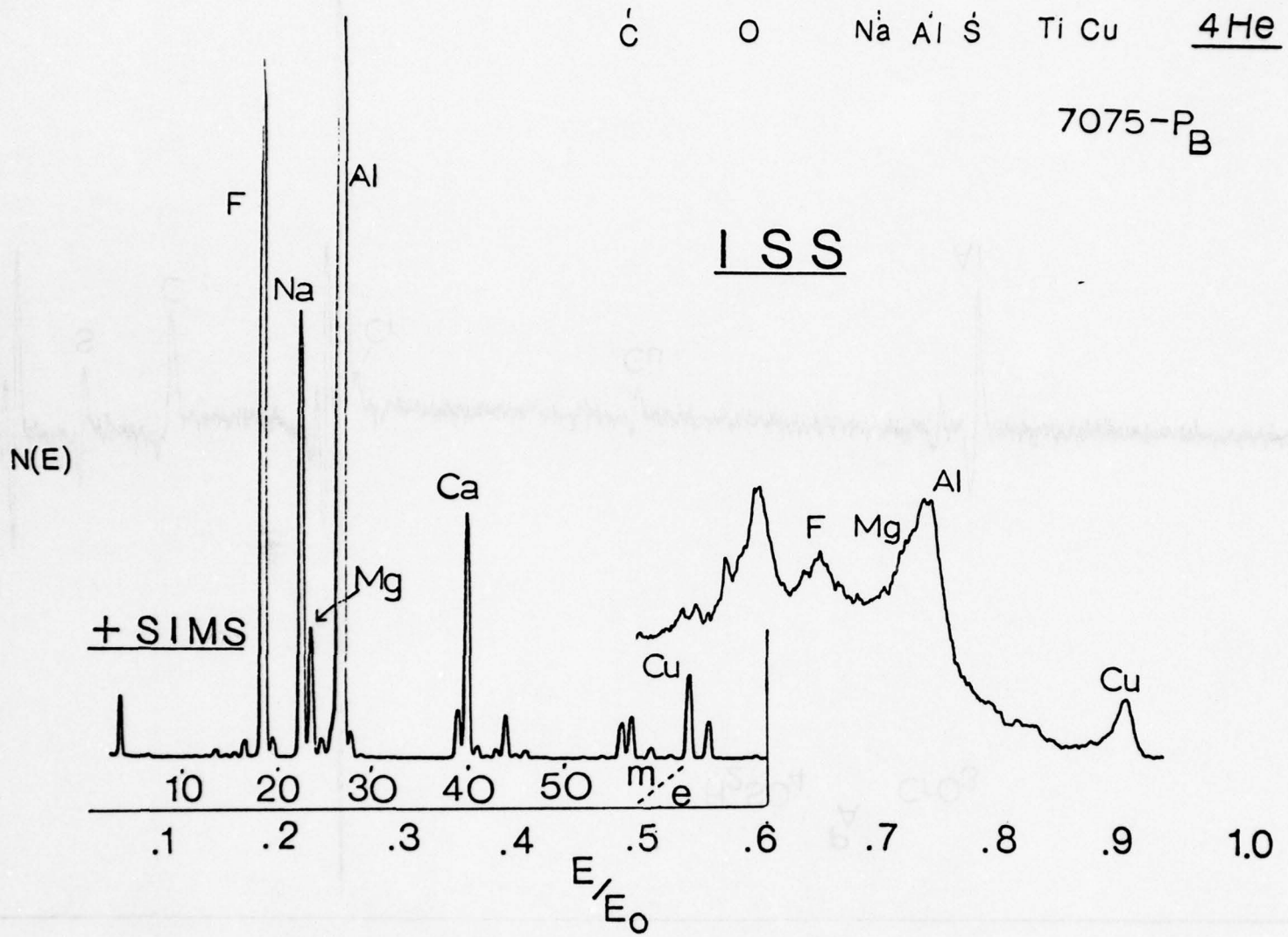

Fig. 5. AES Spectrum of 7075-Sulfuric/Chromic Acid Cleaned.

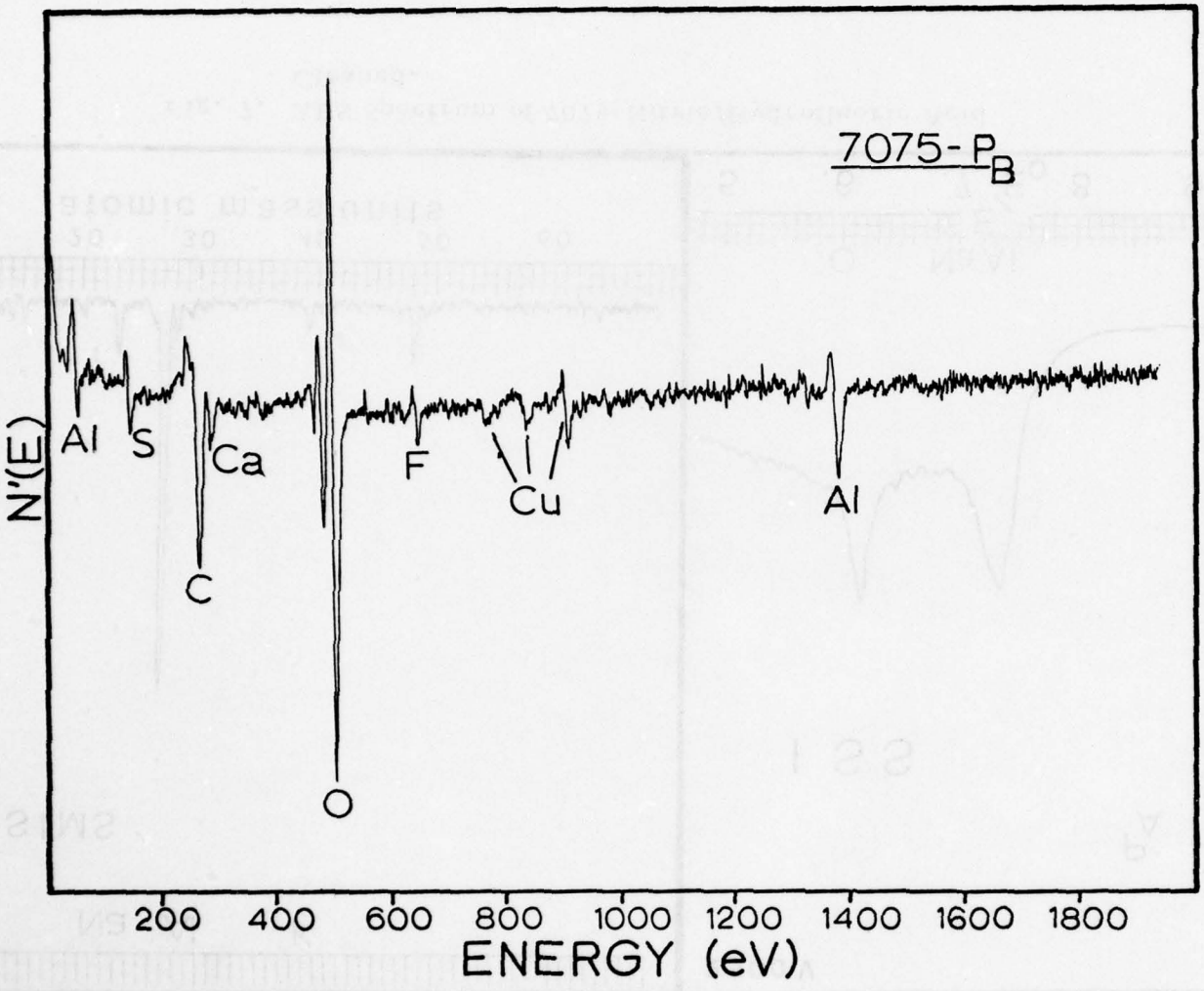

Fig. 6. SIMS-ISS Spectrum of 7075-Nitric/Hydrofluoric Acid Cleaned.

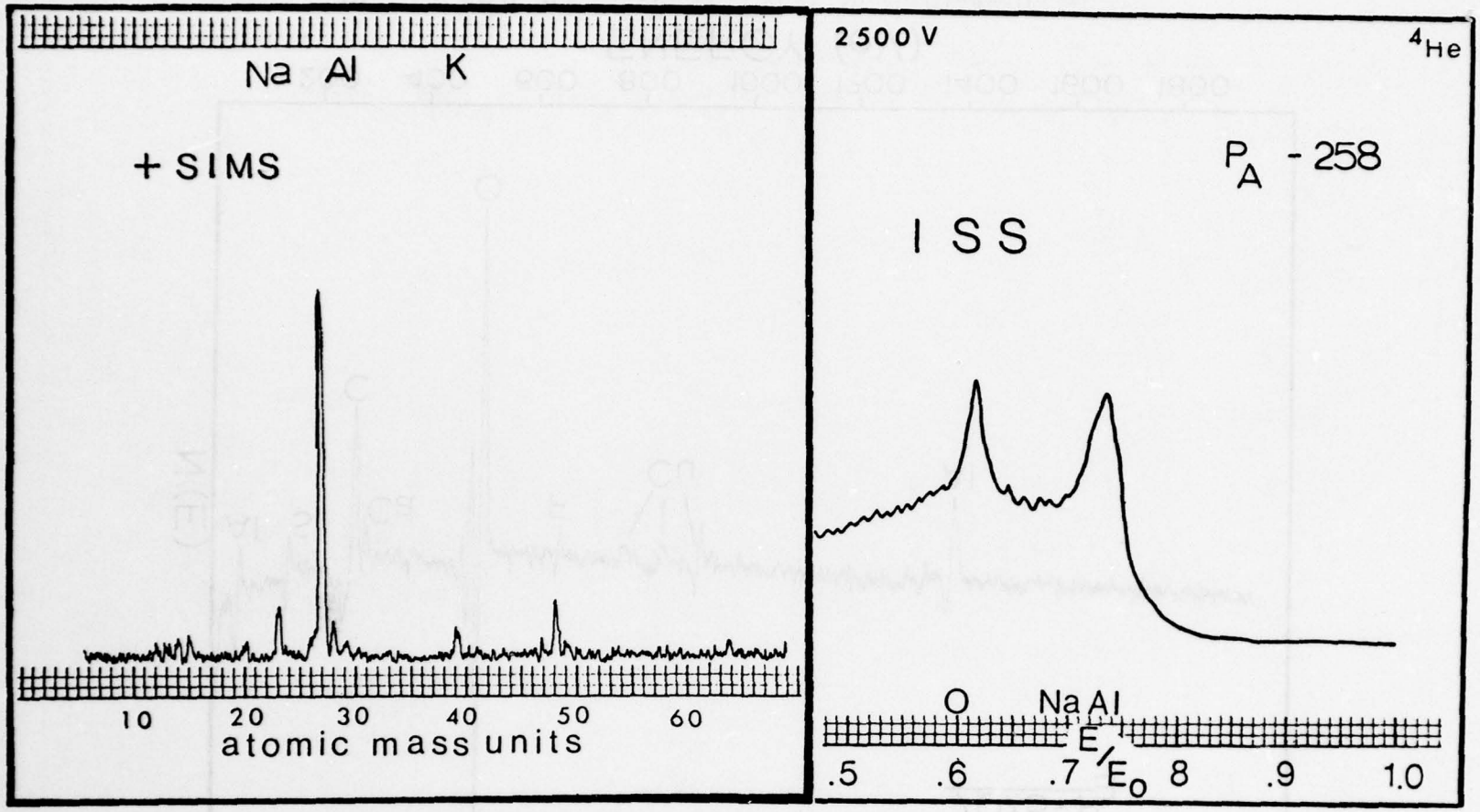

Fig. 7. AES Spectrum of 7075-Nitric/Hydrofluoric Acid Cleaned.

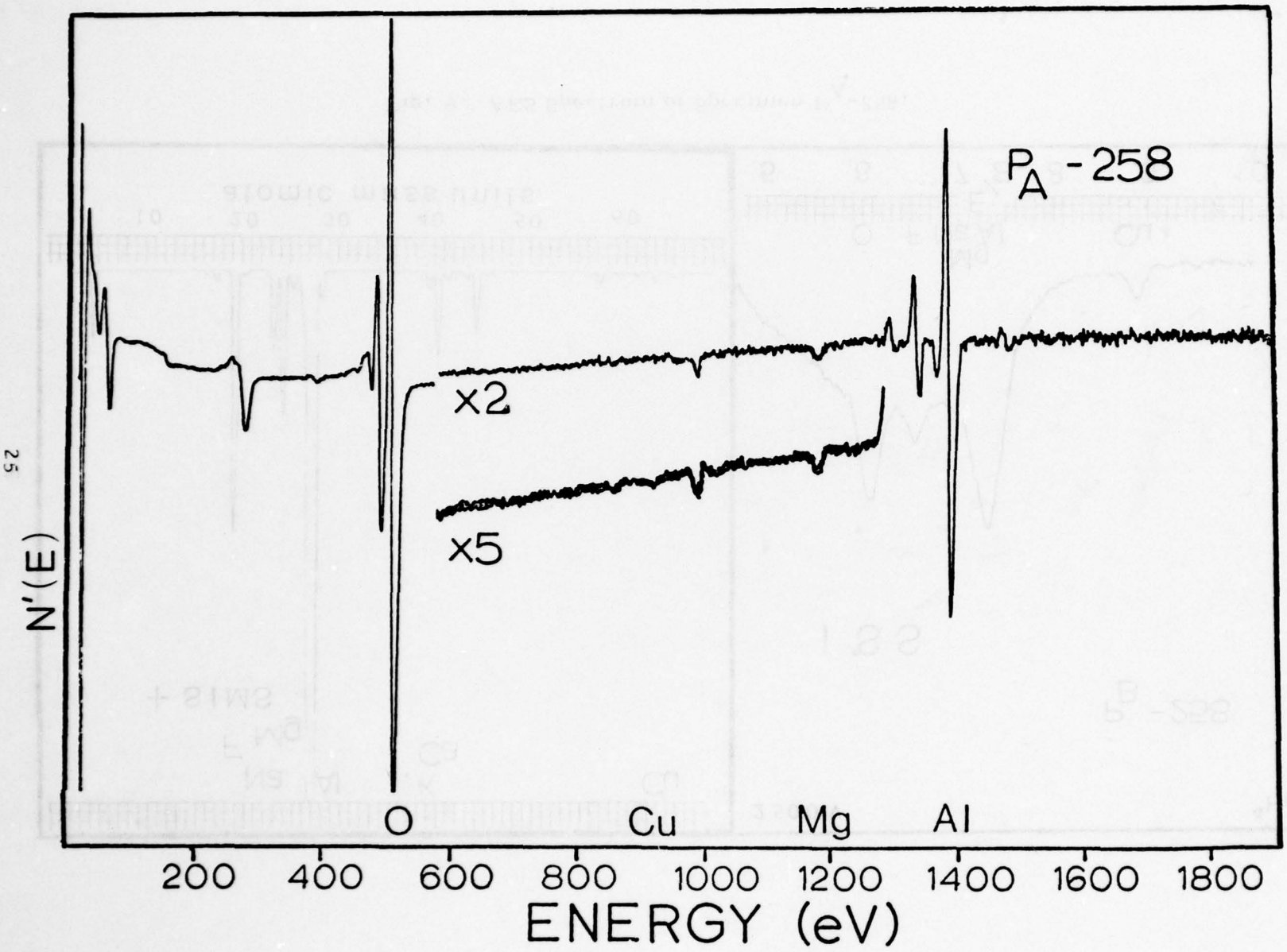

Fig. 8. SIMS-ISS Spectrum of Specimen P_A-258.

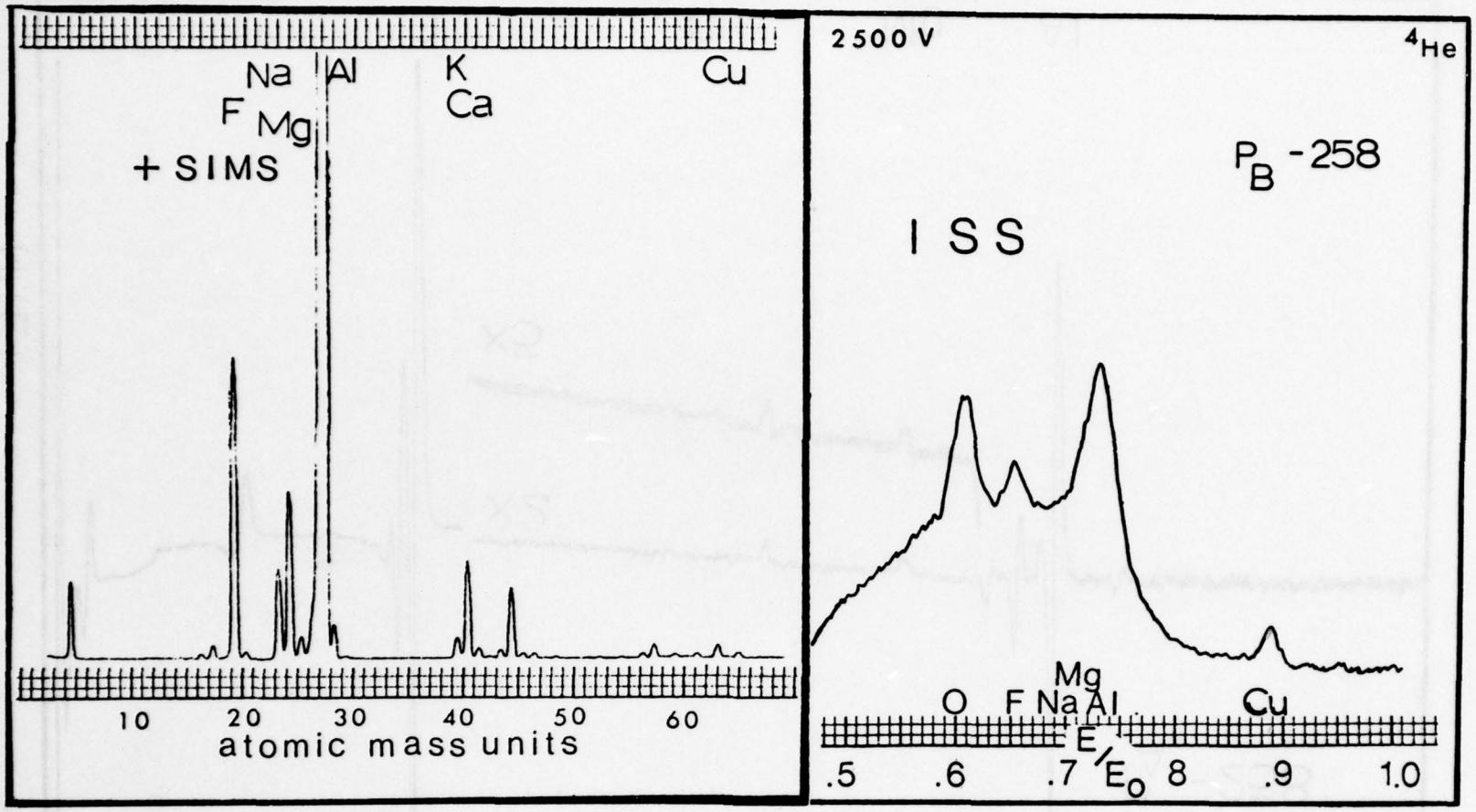

Fig. 9. AES Spectrum of Specimen P_A-258.

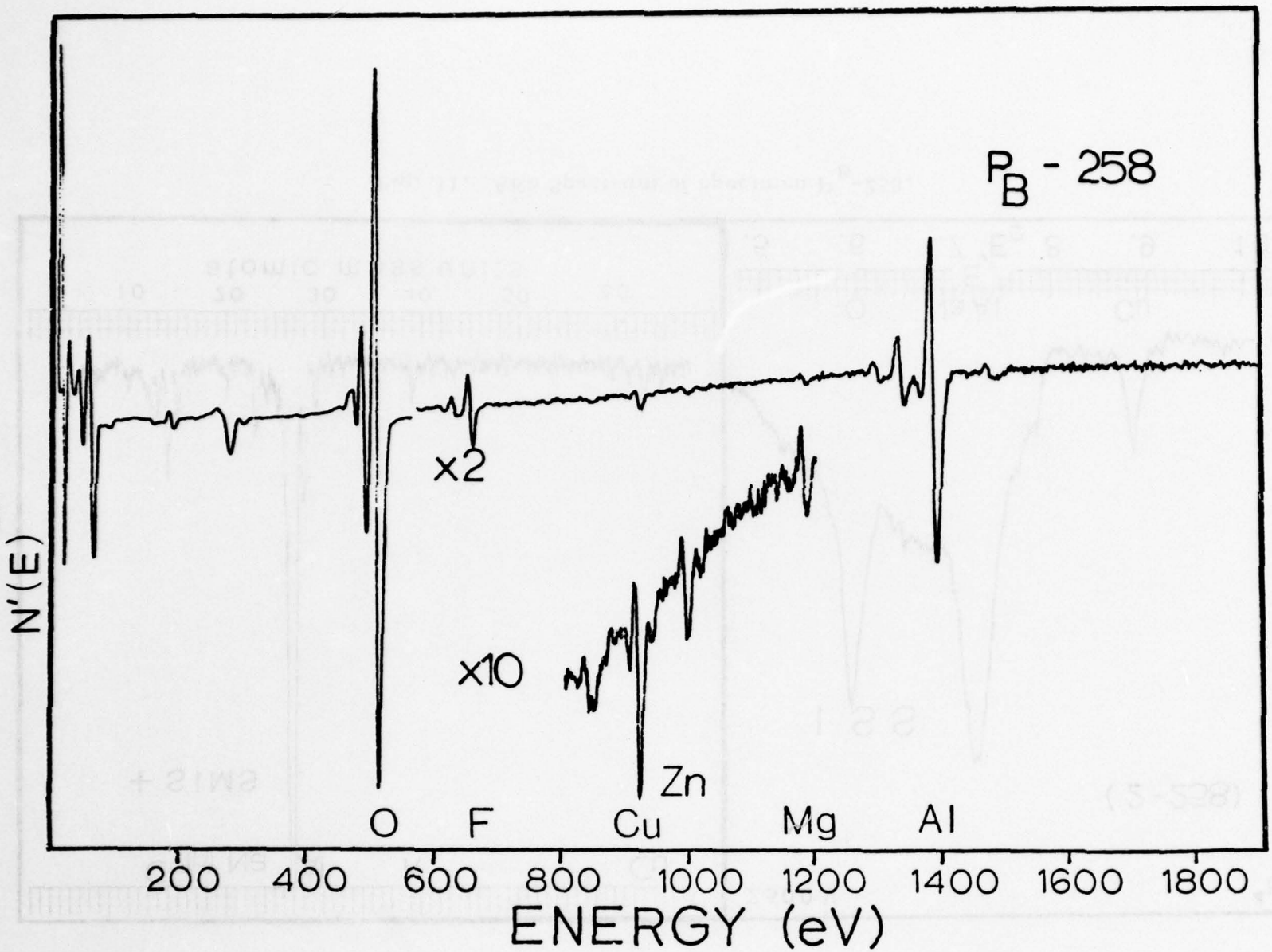

Fig. 10. SIMS-ISS Spectrum of Specimen P_B-258.

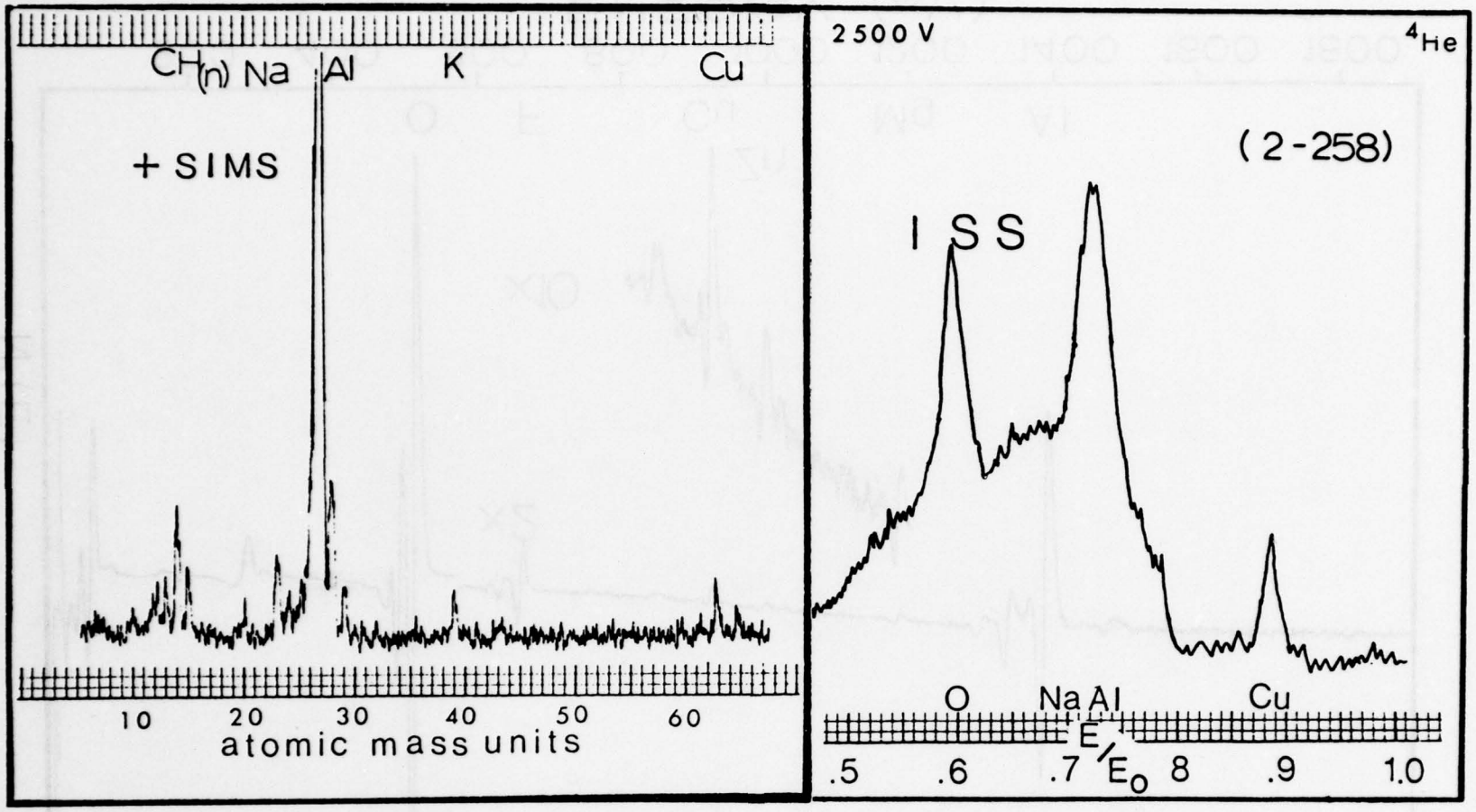

Fig. 11. AES Spectrum of Specimen P_B-258.

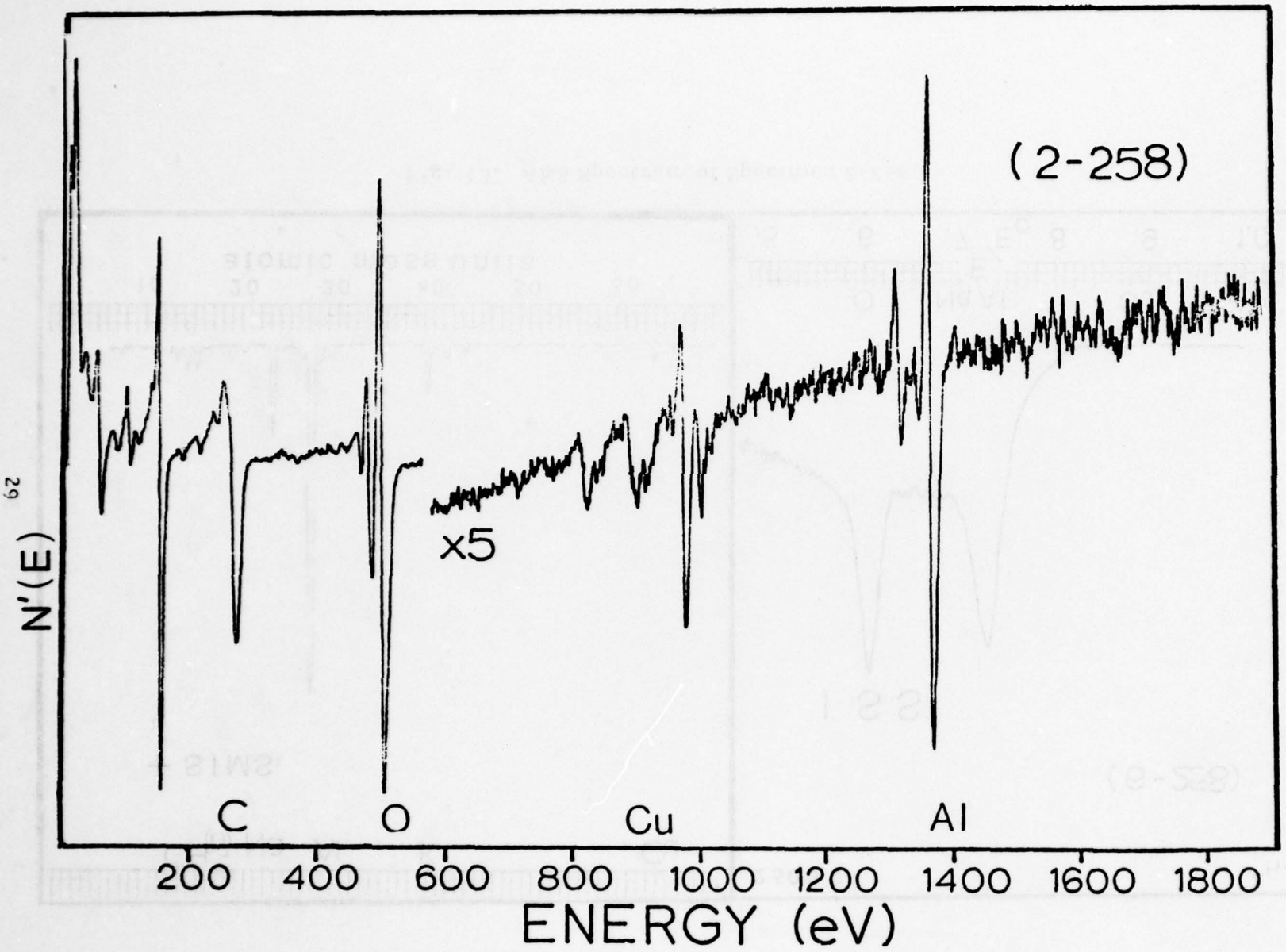

Fig. 12. SIMS-ISS Spectrum of Specimen 2-258.

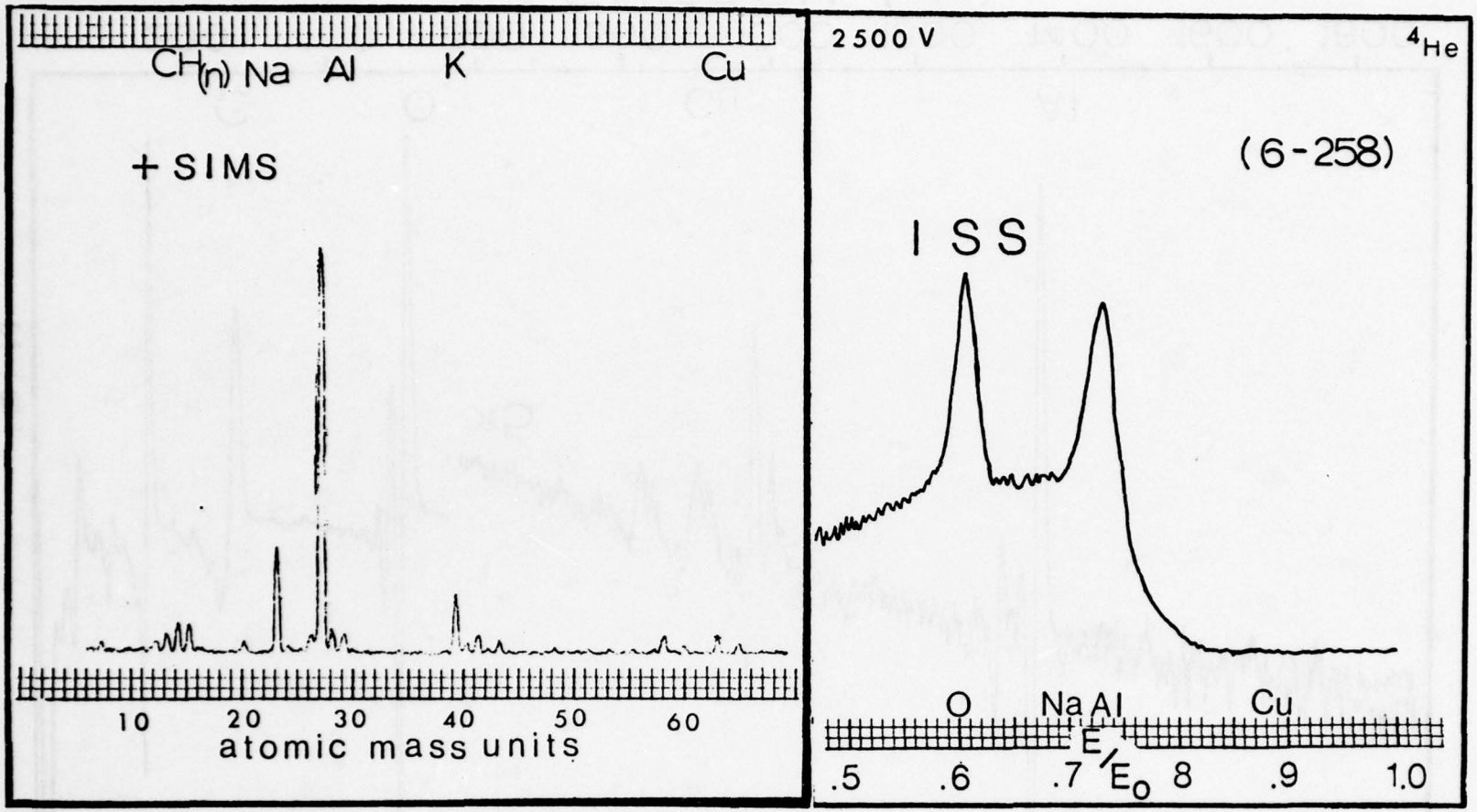

Fig. 13. AES Spectrum of Specimen 2-258.

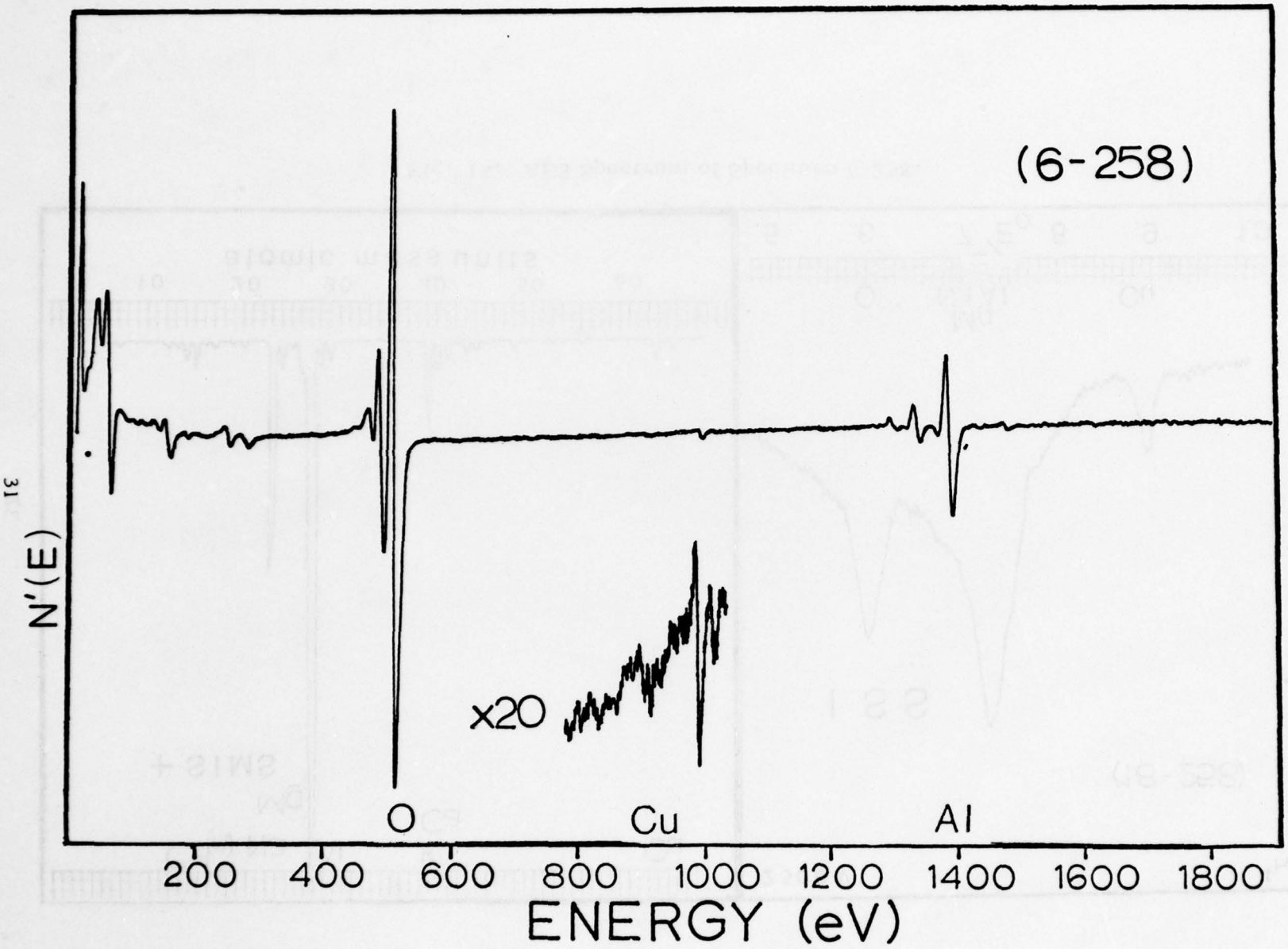

Fig. 14. SIMS-ISS Spectrum of Specimen 6-258.

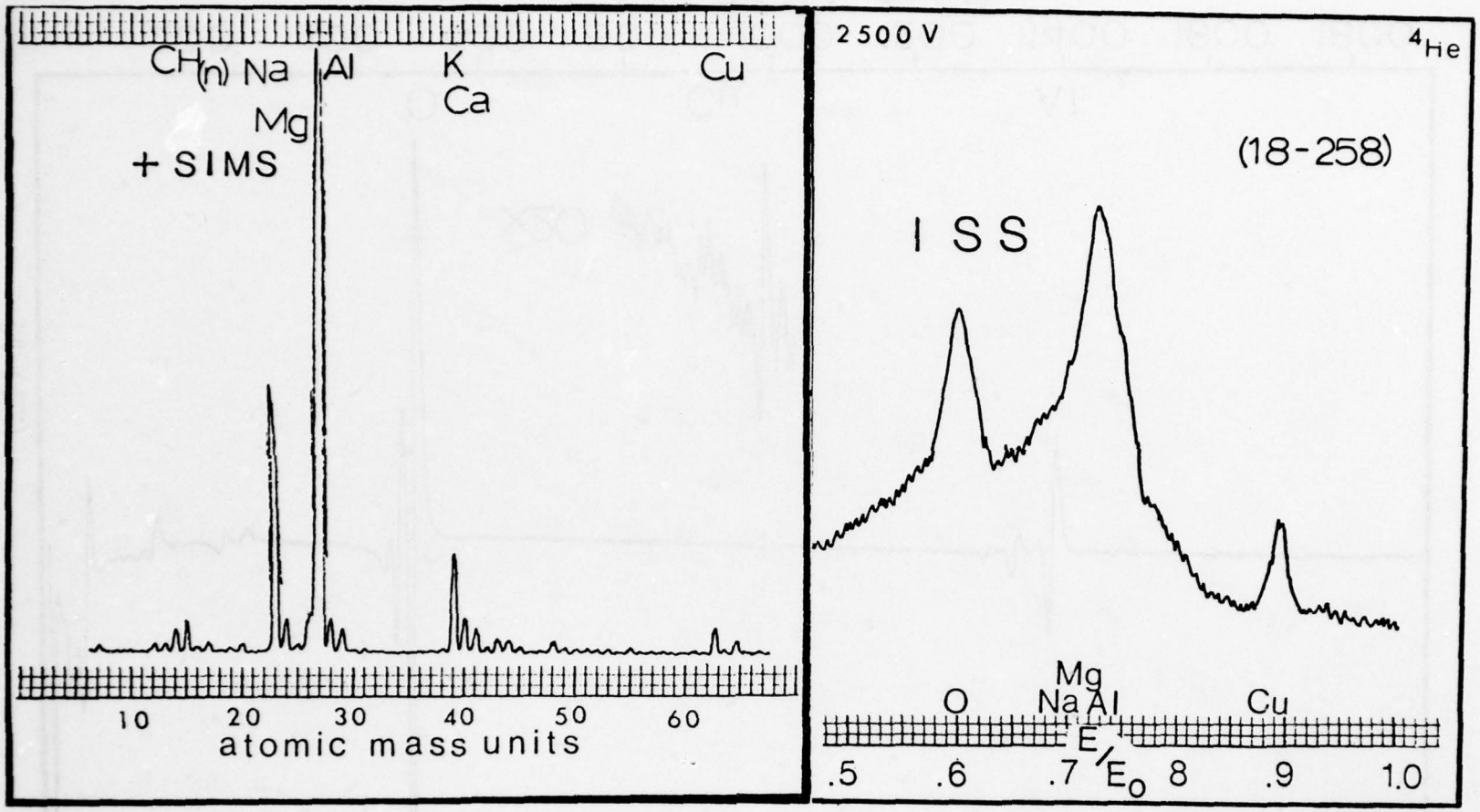

Fig. 15. AES Spectrum of Specimen 6-258.

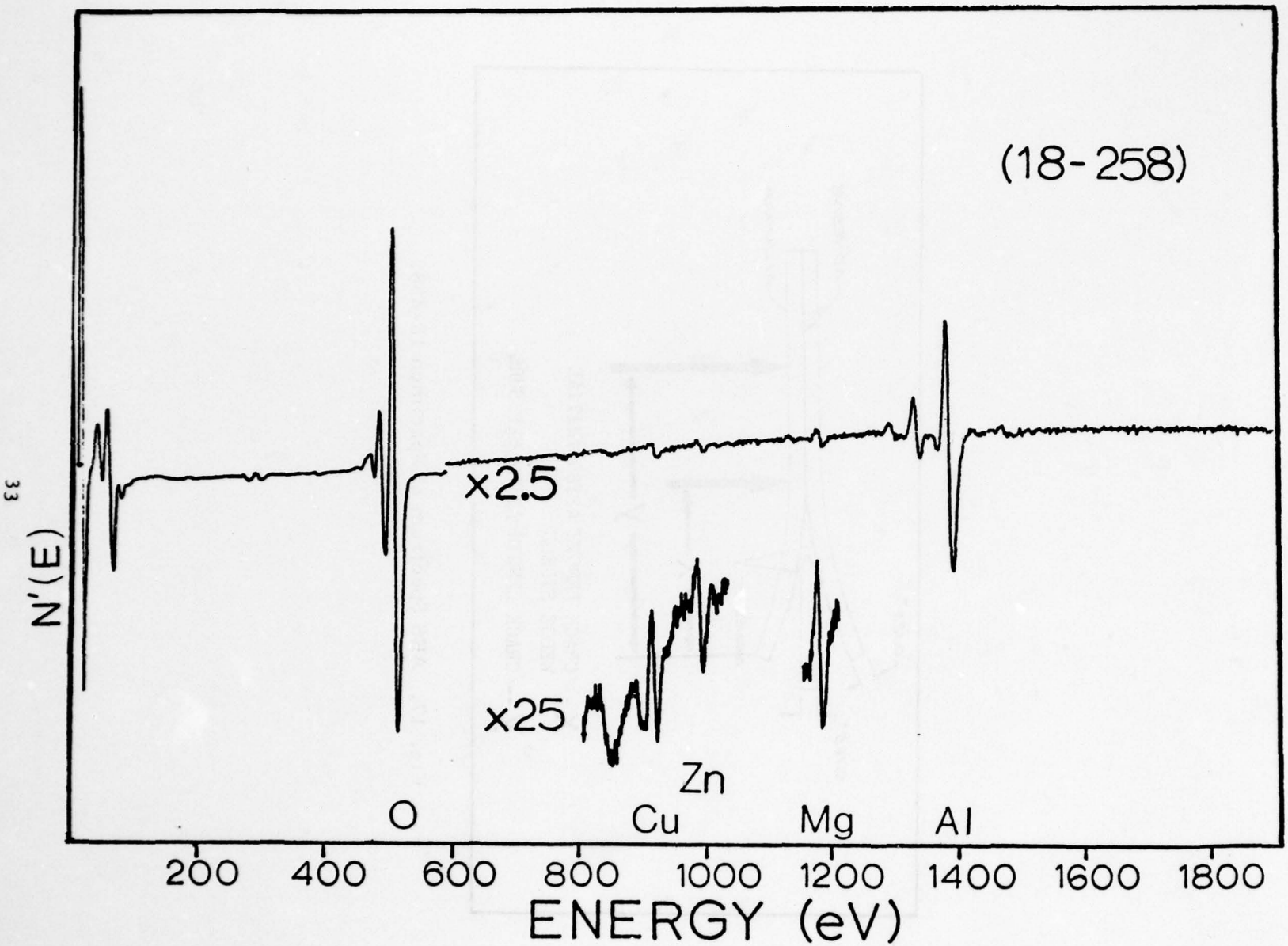

Fig. 16. SIMS-ISS Spectrum of Specimen 18-258.

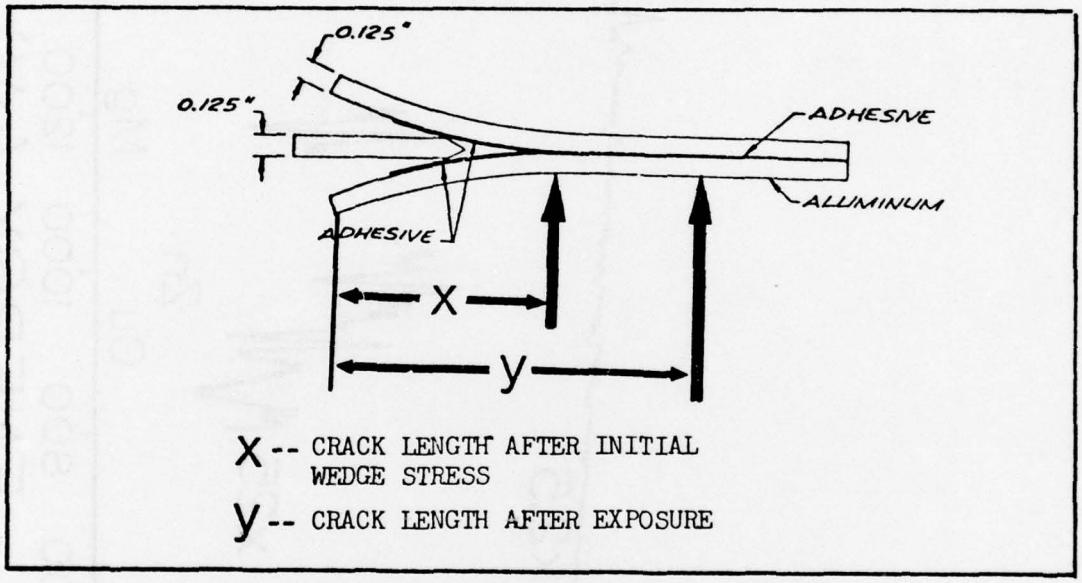

Fig. 17. AES Spectrum of Specimen 18-258.

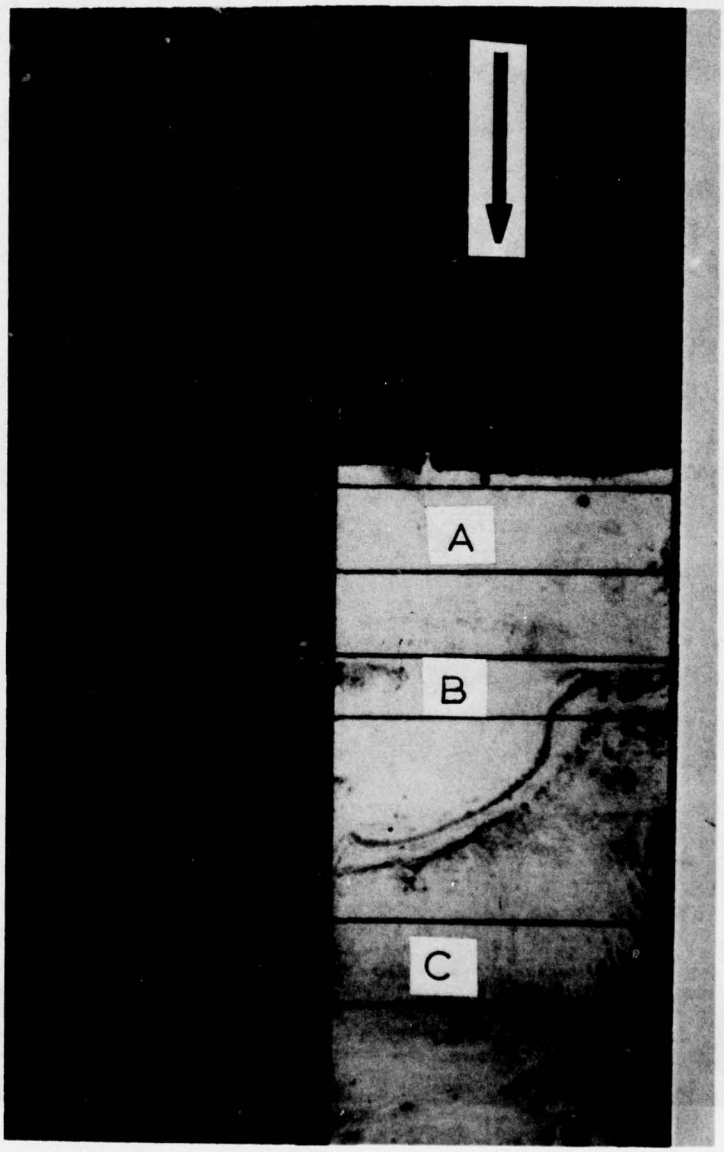

Fig. 18. Sampling Details of Adhesively Failed Wedge Test Specimen 2-258.

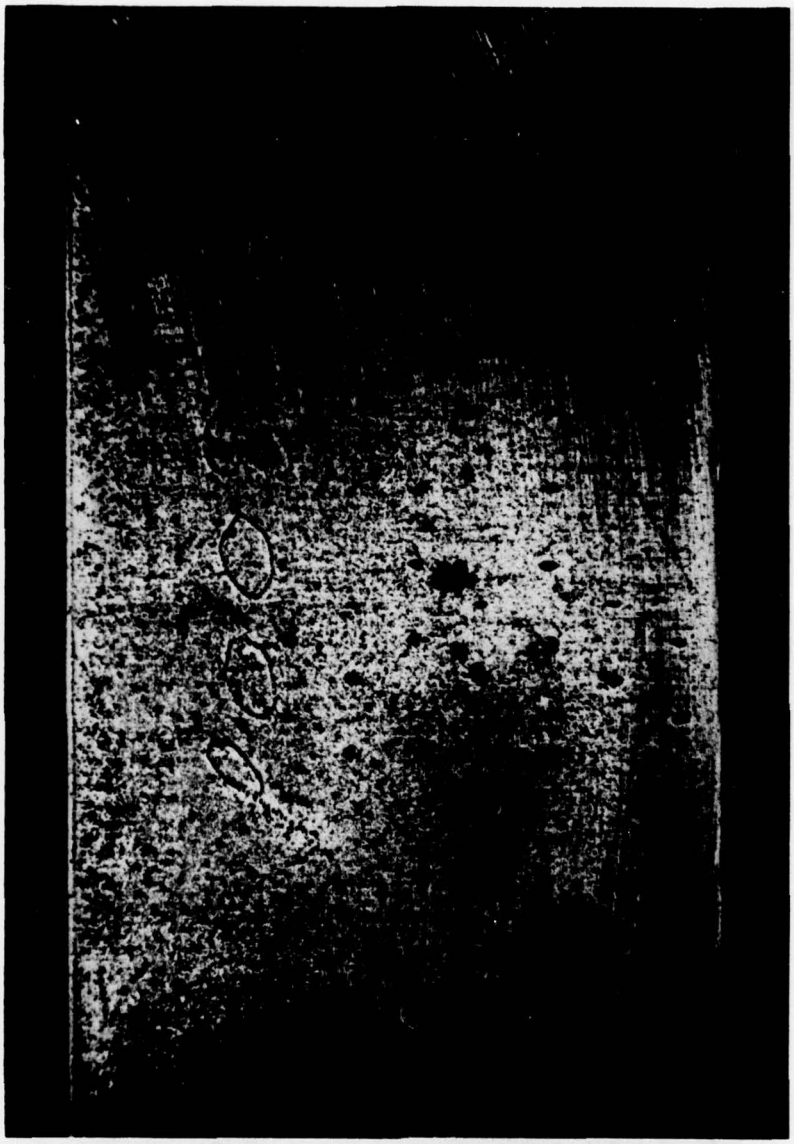

Fig. 19. Pitting Corrosion on Unbonded Side of Wedge Test Specimen 6-218.

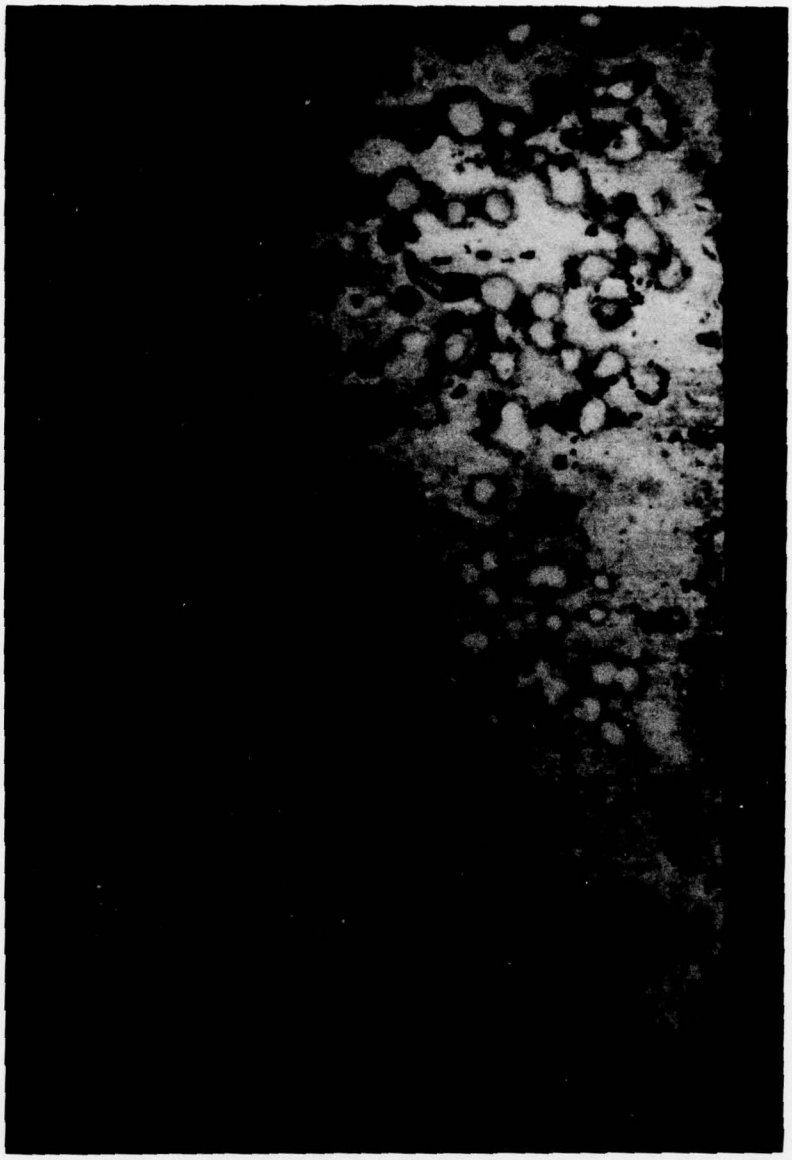

Fig. 20. Corrosion on Unbonded Side of Wedge Test Specimen 8-258.

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