

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963 A

12



**MORE PRODUCTS
and SERVICES**

ALLOY SURFACES

Company, Inc.

Wilmington, Delaware 19899
(302) 575-1555

ADA 112846

ALLOY SURFACES COMPANY, INC.
100 SOUTH JUSTISON STREET
WILMINGTON, DELAWARE 19801

DAVID McCALLISTER
PRINCIPAL INVESTIGATOR

A. L. BALDI
PROGRAM DIRECTOR

NRL CONTRACT NUMBER N00014-81-C-2514

FEBRUARY 2, 1982

DTIC
ELECTE
MAR 3 0 1982
D
H

DTIC FILE COPY

DISTRIBUTION STATEMENT A
Approved for public release;
Distribution Unlimited

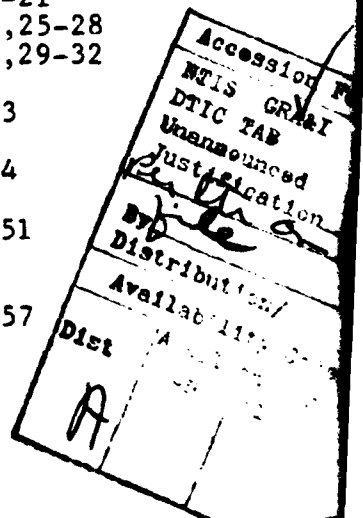
82 02 22 030

TABLE OF CONTENTS

	<u>PAGE</u>
I. Subject	1
II. Statement of Work	1,2
III. Raw Materials	2
IV. Fabrication of Cr-Fe Foils	3-7
A. Deposition and Post Diffusion of Cr	3-4
B. Simultaneous Deposition and Diffusion of Cr	3-5
C. Mechanism of Cr Diffusion	3
D. Energy Dispersive X-Ray Analysis	6
E. Atomic Absorption Analysis	6,7
V. M Diffusion and Activation of Cr-Fe Foils	7-11
VI. Performance of BD Activated Fe and Cr-Fe Elements	10-14
VII. Conclusions	15

Addendums

I. Energy Dispersive X-Ray Analysis for Specimens	16-32
BD 14-42 #35	16-21
BD 14-3 #3	22,23,25-28
BD 14-4 #4	22,24,29-32
II. Atomic Absorption Analysis	33
III. BD-4-57 Test Procedure	34
IV. Time-Temperature Profiles	35-51
V. Photomicrographs of One Specimen from Each Group	52-57



I. SUBJECT

Effect on thermal life of low chromium content in BD activated Chromium-Iron alloys.

II. STATEMENT OF WORK

The work performed under this contract is in support of an ongoing NRL program to develop special sources necessary for IR decoy development. The following tasks were required under this contract:

A. Fabrication of BD Activated Cr-Fe Foils

Cr-Fe foils should be fabricated with varying Cr concentration, preferably in the range of from about one (1) to five (5) percent. The chromium concentration shall be determined throughout the complete cross section of the foil. Such analyses shall be determined quantitatively by Energy Dispersive X Ray Analysis using the Scanning Electron Microscope (EDAX-SEM). The Cr-Fe foils will then be BD Activated and tested to determine the temperature-time profiles.

B. Fabrication of BD Activated Fe Foils to Act as a Control Base

Activated foils having approximately the same BD penetration as the Cr-Fe foils shall be fabricated to act as a control data base.

C. Determine the Percentage of Activation

The percentage of activation for each different element type shall be determined metallographically by measuring the depth of BD penetration.

D. Evaluate Performance of Activated Fe and Cr-Fe Foils

Evaluation shall consist of measuring the element thickness and the temperature-time profiles of each element. Using this information construction of J (infrared output in KW/Ster) versus time above J profiles shall be worked out for each varying Cr concentration element.

E. Provide Samples of Activated Foils

Deliver to NRL two sample lots A and B. Lot A should contain twenty (20) of the best Cr-Fe elements and Lot B, ten (10) of the corresponding Fe elements.

F. Report

Provide to the Scientific Officer a written final technical report including the results of all work performed under this contract.

III. RAW MATERIALS

A. AISI 1010 1 Mil Code Al Steel Shim Stock

Composition:

0.08 - 0.13% C
0.30 - 0.60% Mn
0.04 % MAX P
0.05 % MAX S
0.10 % MAX Si
Balance Fe

B. Chromizing Source Materials

These materials are proprietary to Alloy Surfaces Company, Inc.. They comprise as one constituent chromium. The chromium is of such activity that it can be transported as a vapor to the steel foil on which the Cr deposits and diffuses into the steel surface. The transporting material is a halogen.

IV. FABRICATION OF Cr-Fe FOILS

AISI 1010 1 mil steel foil designated as Fe was diffusion coated with chromium to provide a range of 0.25 to 8.5% Cr. It was obvious during our study that we had to investigate concentrations less than 1% as called for under Paragraph 1, Statement of Work. Two concentrations above the stated 5% level namely 6.3% Cr and 8.5% Cr were also included in the study.

Alloy Surfaces Company, Inc. has developed several proprietary high temperature chromium diffusion coating processes to deposit and diffuse chromium into ferrous and non-ferrous metals and their alloys. Two of our processes were successful in achieving the objective to fabricate Cr-Fe foil with a uniform Cr concentration throughout the foil thickness.

One of the two following procedures was used to accomplish this task:

A. Deposition and Post Diffusion of Cr

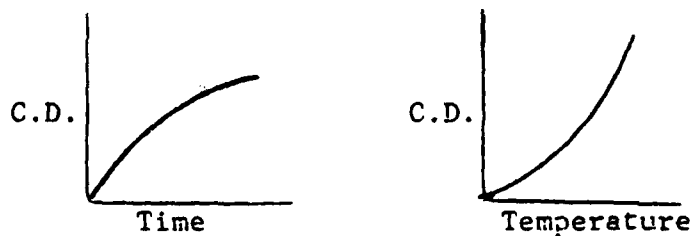
Deposition and diffusion of Cr into the foil surface (Step 1) followed by a post diffusion heat treatment to attain a uniform Cr concentration throughout the foil cross section (Step 2). The micrograph in Figure 1 shows a steel foil after Step 1. One can observe the rich chromium surface in which the Cr was diffused to about 0.2 mil into the surface. The micrograph in Figure 2 shows the same foil after Step 2 in which the Cr was post diffused to provide a uniform Cr concentration throughout the complete foil cross section.

B. Simultaneous Deposition and Diffusion of Cr

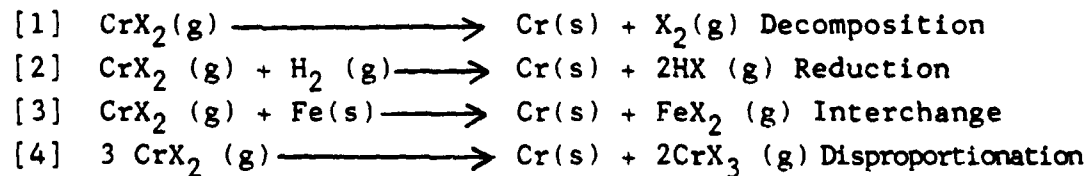
A one-step chromium and diffusion process to provide a uniform Cr concentration throughout the foil thickness is shown in Figure 3.

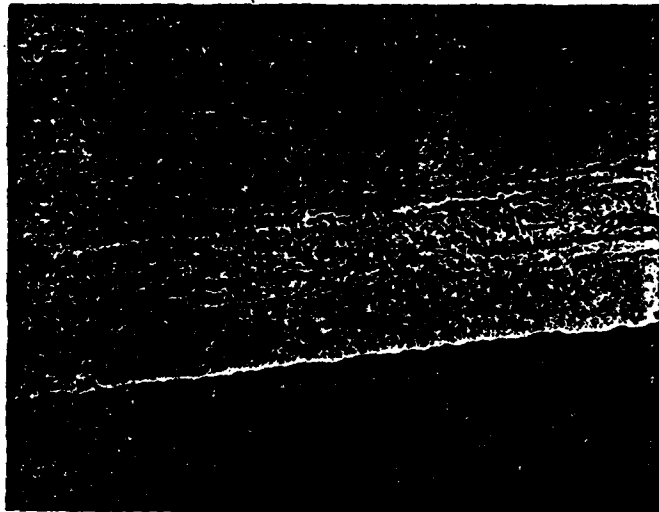
C. Mechanism of Chromium Diffusion

In both A and B processes, chromium was transported to the steel foil surface between 1400 and 2000°F for various times to provide the designated Cr concentration throughout the foil thickness. The case depth of chromium diffusion is a parabolic function of time and varies exponentially with temperature.



The Cr was transported as a gaseous halide from the Cr source to the steel surface. The reaction at the steel surfaces can be one of interchange or deposition or a combination of both. The equations depicting these reactions are:

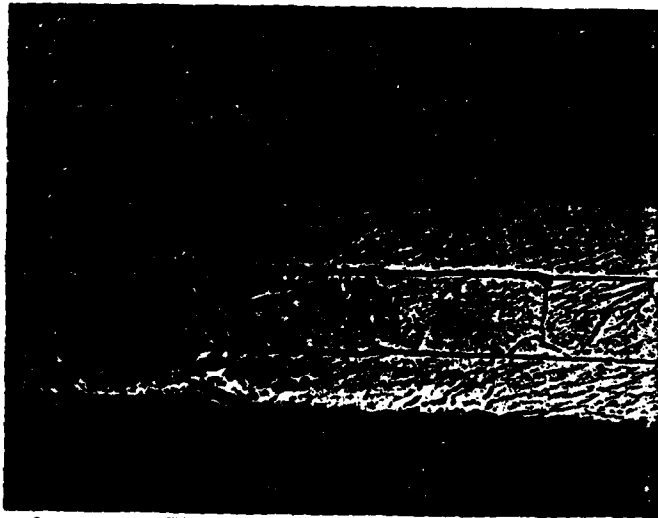




- Cr Rich Surface
- Fe
- Cr Rich Surface

450X 6801 DIL HNO₃ ETCH

FIG 1



) Cr-Fe Foil

450X 7314 DIL HNO₃ ETCH

FIG 2



Cr-Fe Foil

450X FIG3 N253

In all reactions atomic Cr(s) is deposited and diffused into the steel surface. During the inward diffusion of Cr, Fe also diffuses outwardly to bring about homogeneity. Reactions 1, 2 and 4 are deposition reactions and will result in an increase in weight of the original foil. Reaction 3 is an interchange reaction and since the atomic weight of Cr and Fe are close (52 vs 56) and one atom exchanges for the other, there will be little or no change in weight in this reaction. The composition of the steel and chromizing conditions will dictate the predominate reaction.

All elements in this study were weighed before and after chromizing. All resulted in a positive weight gain indicating that the deposition type reactions predominated. Several elements were analyzed for their Cr content to establish a relationship between weight pick-up (mg/cm^2) and Cr concentration.

D. Energy Dispersive X-Ray Analysis (EDAX) by the Scanning Electron Microscope (SEM)

Three (3) elements with varying weight pick-up were sent to Micron, Incorporated, Analytical Service Laboratory for EDAX-SEM quantitative analysis. Profiles for chromium show that the chromium concentration was uniform throughout the entire foil thickness. Addendum I includes these profiles as well as the quantitative analytical results for Cr and Fe in the Cr-Fe foils.

The quantitative analytical determinations of Cr are shown below. The corresponding weight gain after Cr diffusion in $\text{mg}/\text{sq cm}$ of total surface area are also shown.

<u>Element Identification</u>	<u>mg/cm^2</u>	<u>% Cr</u>
BD 14-42 #35	0.08	1.10
BD 14-3 # 3	0.27	3.20
BD 14-4 # 4	0.65	8.30

E. Atomic Absorption Analysis (AA)

Since the EDAX-SEM Analyses has shown conclusively that a uniform Cr concentration is provided throughout the entire foil thickness of the elements either by process A or B under IV, an in-house quantitative analysis procedure was used to determine the percent Cr in the elements. This method constitutes Atomic Absorption Analysis as described in Addendum II. Several elements were analyzed by this technique. The corresponding weight gain (mg/cm^2) and % Cr for each element were as follows:

<u>Element Identification</u>	<u>mg/cm²</u>	<u>% Cr</u>
BD 14-19 #18	0.02	0.30
BD 14-19 #57	0.10	1.30
BD 14- 3 # 2	0.25	3.30
BD 11-135 # 2	0.64	8.20
Untreated 1010	0	0.01

A plot of mg/cm² weight gain and percent chromium for each element included under Paragraph IV, D and IV, E are shown in the graph in Figure 4. It shows that the percent chromium varies directly with the weight pick-up after chromizing.

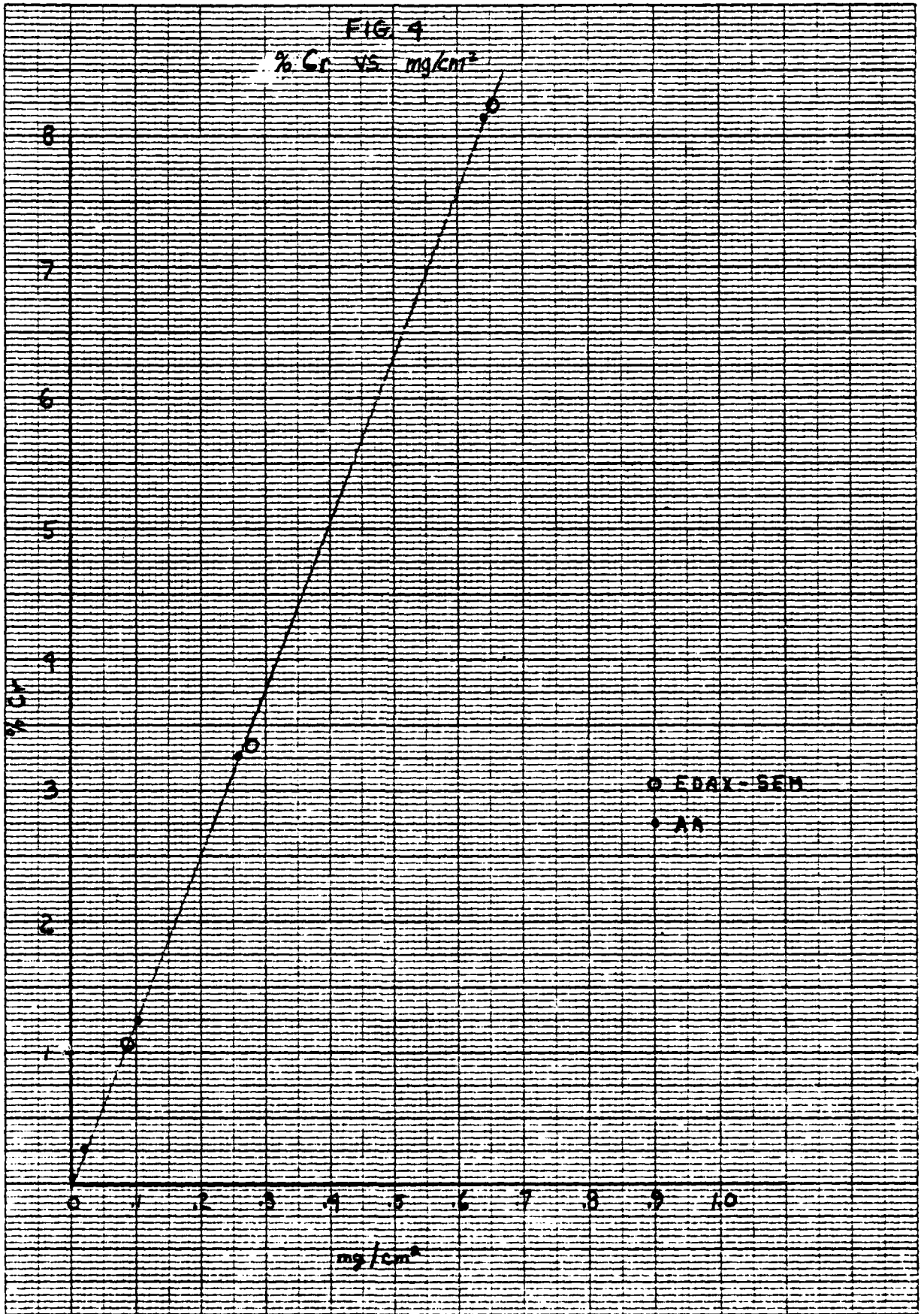
V. M DIFFUSION AND ACTIVATION OF Cr-Fe FOILS

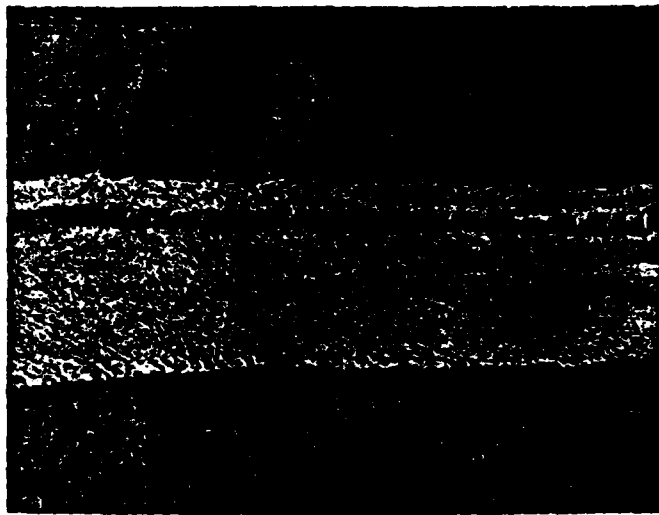
The BD process consists of diffusing one or more metals (M) into a base metal substrate and subsequent chemical leaching of this metal or metals out of the substrate to produce a very active surface with pyrophoric characteristics.

Several Fe and Fe-Cr foils after BD processing were tested in the BD-4-57 test rig to establish the time-temperature profiles. The BD-4-57 test procedure is described in Addendum III. A small section was cut from each foil to determine the depth of BD penetration. A fully activated BD foil element is one in which the leaching process has penetrated through the complete " M " diffused layer. This can be readily observed by metallographic examination of a mounted element cross section. The photomicrograph in Figure 5 shows an activated element in which the diffused metal " M " was only about 1/5 penetrated by the leaching solution and therefore only partially activated. The photomicrograph in Figure 6 shows a similar element in which the diffused metal was completely penetrated by more aggressive leaching conditions and therefore completely activated. Each element was weighed before and after chromizing to determine the mg/cm² pick-up. The corresponding percent Cr in the foil was established from graph in Figure 4. Metallographic study of each element showed the total depth of " M " diffusion and leaching penetration to be as follows:

46 1326

K•E 10 X 10 TO 14 INCH 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

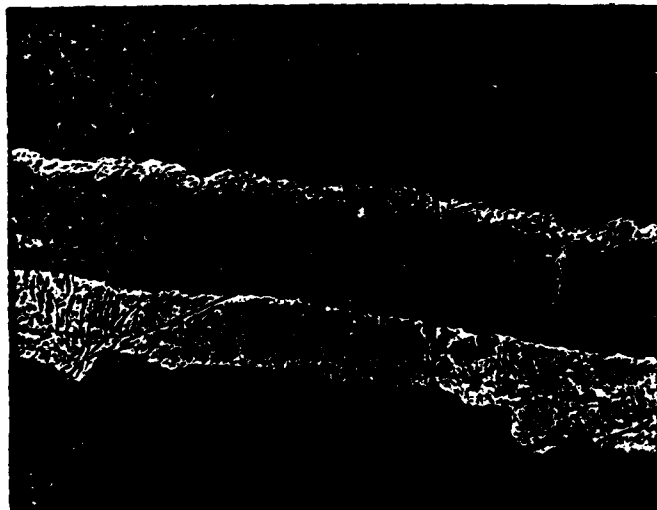




- ACTIVATED
- UNACTIVATED
- BASE FOIL

N268 450X

FIG 5



- > ACTIVATED
- BASE FOIL

N280 450X

FIG 6

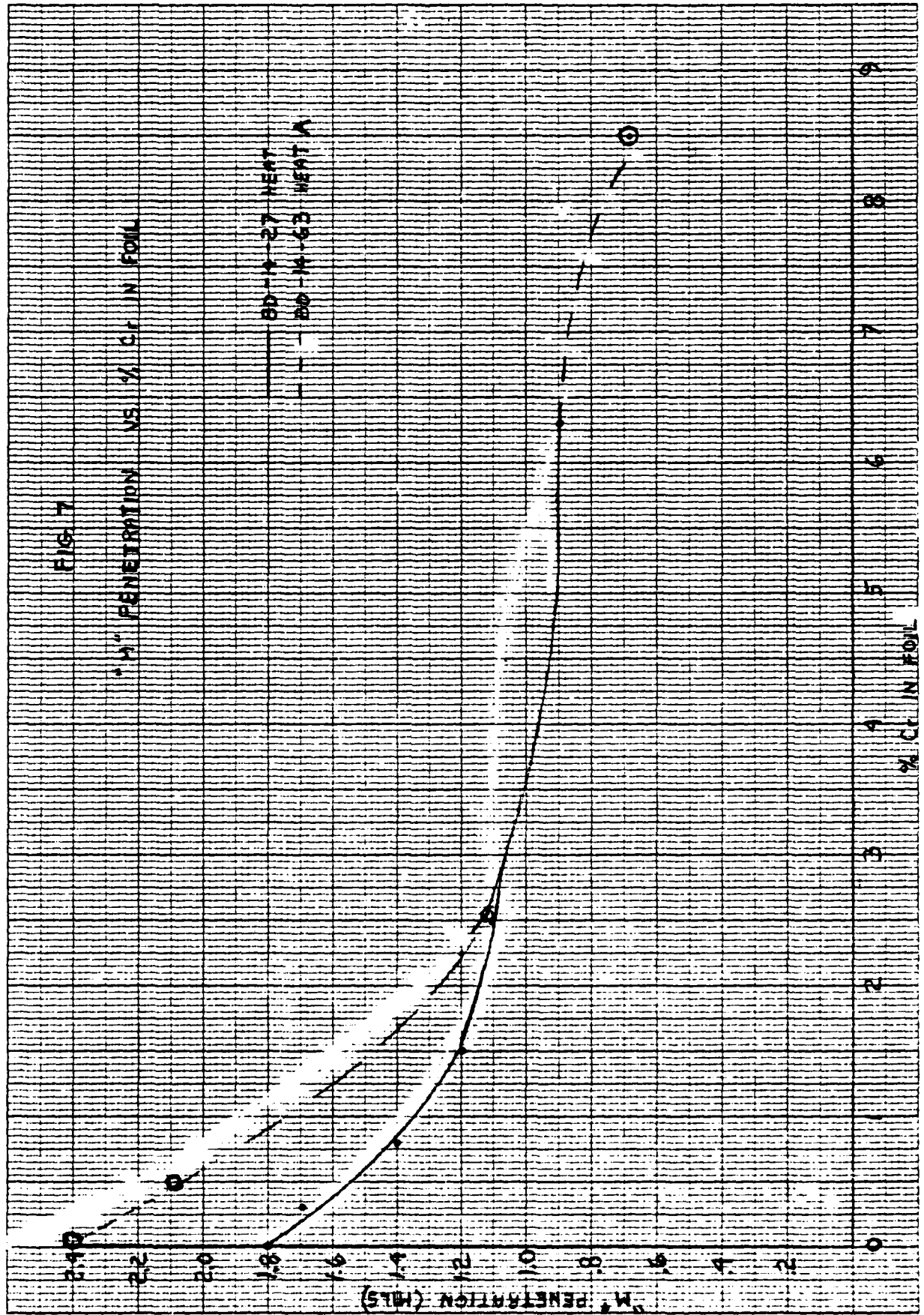
<u>BD Processing Heat</u>	<u>% Cr</u>	<u>BD Activation</u>	
		<u>M Penetration (mils)</u>	<u>Leaching Penetration (mils)</u>
BD-14-27	0.00	1.8	1.8
"	0.25	1.7	1.7
"	0.80	1.4	1.4
"	1.50	1.2	1.2
"	2.20	1.2	0.7
"	6.30	0.9	0.2
BD-14-63	0.00	2.6	2.6
"	0.70	2.1	2.1
"	2.50	1.1	0.2 (1.1)
"	8.50	0.7	0.1 (0.7)

() After more aggressive leaching at higher temperatures and longer times.

Graphs of % Cr versus M penetration is shown in Figure 7 for each BD processing heat. Diffusion heat BD-14-27 was run at a lower processing temperature than heat BD-14-63, hence the lower M penetration. In the case of elements containing up to about 2.5% Cr, the resistance of Cr to M penetration increases with the increase in Cr content. Above 2.5%, the M penetration for the BD-14-27 and BD-14-63 heats are comparable. An obvious wrinkling effect was noted for BD treated elements containing about 0.80 percent and greater Cr content. This resulted in erratic and greater δ values and drastically lowered the EPF values. This effect was not present in any element after chromium diffusion. This distortion was noted after M diffusion and chemical leaching.

VI. PERFORMANCE OF BD ACTIVATED Fe AND Cr-Fe ELEMENTS

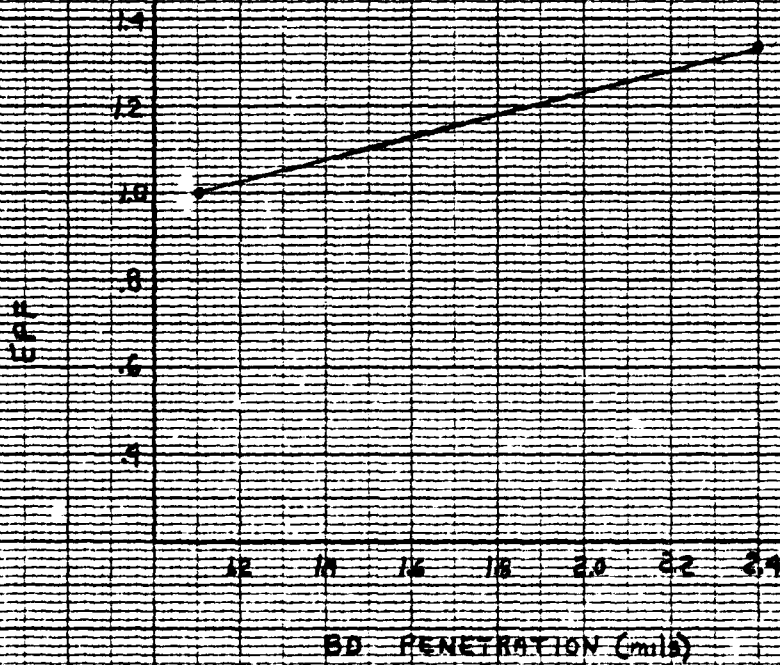
After leaching, the BD activated foils were thoroughly washed in water and while still wet with water were transferred to the specimen holder and tested according to the BD-14-57 test procedure as described in Addendum III. Time-temperature profiles of all elements are shown in Addendum IV. After testing at least one (1) element from each group, it was sectioned for metallographic examination of M and BD penetration. Micrographs of these specimens are shown in Addendums V. Energy and time performance factors were calculated for each element. Table I includes pertinent data and results identified as follows:



1. Element Identification
2. Percent of Cr in foil before BD activation (% Cr)
3. "M" penetration in mils (M_p)
4. BD penetration in mils (BD_p)
5. Element thickness in mils (t)
6. Energy performance factor (EPF)
7. Corresponding approximate EPF for BD Fe as determined from EPF vs BD penetration
Graph established in Task 2 of NRL Contract
NOO173-79-C-337 (EPF Fe)
(see Figure 8)
8. Time performance factor (TPF)
9. Maximum temperature attained by element in $^{\circ}F$ (T_{max})
10. Metallographic mount identification (Mount #)
11. Flatness of specimen

K28

ENERGY PERFORMANCE FACTOR
VS.
BD PENETRATION



K-E 10 X 10 TO 1/8 INCH 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

46 1326

Element Identification	% Cr	M _p	BD _p	δ	EPF	EPF (Fe)	TPF	T _{max}	Mount#	Flatness of Specimen	
BD-14-86	3858	0	1.3	1.3	2.2	1.1	1.1	0.30	760	N299	Flat
"	3859	0	1.3	1.3	2.2	1.1	1.1	0.26	840	-	Flat
"	3861	<1	1.2	1.2	4.0	nil	1.0	0.28	700	N302	Wrinkled
"	3862	<1	1.2	1.2	4.0	nil	1.0	0.16	640	-	Wrinkled
"	3863	2.5	1.0	1.0	3.0	nil	1.0	0.10	420	-	Wrinkled
"	3864	2.5	1.0	1.0	3.0	nil	1.0	0.10	420	N304	Wrinkled
BD-14-27	3782	0	1.8	1.8	2.8	1.0	1.2	0.39	1000	N283	Flat
"	3785	0	1.7	1.7	2.6	1.0	1.0	0.38	700	N284	Flat
"	3786	0	1.8	1.8	2.8	1.1	1.2	0.40	720	N277	Flat
"	3778	0.25	1.7	1.7	2.6	1.10	1.15	0.63	900	N282	Slight Wrinkle
"	3783	0.25	1.7	1.7	3.5	0.55	1.15	0.27	900	-	Slight Wrinkle
"	3787	0.25	1.8	1.8	2.6	1.30	1.20	0.43	780	N273	Slight Wrinkle
"	3788	0.27	1.7	1.7	2.8	0.75	1.15	0.27	750	-	Slight Wrinkle
"	3784	0.80	1.4	1.4	3.1	0.65	1.10	0.32	840	N275	Wrinkled
"	3789	0.80	1.4	1.4	2.7	0.50	1.10	0.23	660	N285	Wrinkled
"	3790	0.80	1.4	1.4	3.5	0.30	1.10	0.24	700	-	Wrinkled
"	3792	1.50	1.2	1.2	3.5	0.10	1.00	0.22	650	N274	Wrinkled
"	3793	1.50	1.2	1.2	4.5	nil	1.00	0.20	760	-	Wrinkled
"	3791	2.20	1.2	0.7	4.0	nil	1.00	0.17	560	N279	Wrinkled
"	3780	2.20	1.2	0.7	5.0	nil	1.00	0.05	440	-	Wrinkled
"	3779	6.3	0.9	0.2	18.0	nil	0.90	0.08	560	N276	Wrinkled
"	3795	6.3	1.0	0.3	12.0	nil	0.95	0.05	440	N293	Wrinkled
"	3796	6.3	1.0	0.3	12.0	nil	0.95	0.05	420	-	Wrinkled
BD-14-63	3830	0	2.4	2.4	3.0	1.1	1.3	0.45	780	N266	Flat
"	3831	0	2.4	2.4	3.0	1.1	1.3	0.43	740	N288	Flat
"	3840	0	2.4	2.4	2.9	1.2	1.3	0.40	960	N289	Flat
"	3832	.50	2.1	2.1	4.0	0.7	1.2	0.48	895	N267	Slight Wrinkle
"	3842	.50	2.2	1.7	3.5	0.6	1.3	0.30	800	N294	Slight Wrinkle
"	3833	1.7	1.4	1.4	3.0	0.8	1.1	0.30	910	N305	Slight Wrinkle
"	3843	1.7	1.4	1.4	3.5	0.30	1.1	0.20	1000	N306	Slight Wrinkle
"	3835	2.5	1.1	0.2	11.0	nil	1.0	0.00	300	N268	Wrinkled
"	3834	2.5	1.1	0.2	14	nil	1.0	0.00	300	-	Wrinkled
"	3845	2.5	1.1	0.2	6	nil	1.0	0.08	520	N295	Wrinkled
"	3844	2.5	1.1	0.2	11	nil	1.0	0.09	560	-	Wrinkled
"	3850*	2.5	1.1	1.1	10	nil	1.0	0.20	660	N280	Wrinkled
"	3848*	2.5	1.5	1.5	10	nil	1.0	0.15	660	N270	Wrinkled
"	3837	8.5	0.7	0.1	9	0	0.9	0	100	N269	Wrinkled
"	3836	8.5	0.7	0.1	9	0	0.9	0	100	-	Wrinkled
"	3846	8.5	0.7	0.1	3.5	0	0.9	0	100	N296	Wrinkled
"	3849*	8.5	0.7	0.7	3.5	0	0.9	0	220	N271	Wrinkled
"	3851*	8.5	0.7	0.7	3.5	0	0.9	0	220	N297	Wrinkled
"	3852*	0	2.3	2.3	3.0	1.2	1.3	0.43	780	N278	Flat
"	3854*	0	2.8	2.8	3.5	1.3	1.4	0.57	870	N272	Flat

* More aggressive leaching conditions

TABLE I

VII. CONCLUSIONS

→ Cr-Fe alloys were successfully produced by diffusing Cr into 1 mil 1010 steel foil stock. Uniform concentrations of Cr were provided throughout the complete foil thickness as determined by Electron Probe Analysis.

After BD activation, the thermal energy and to a lesser degree the thermal life was reduced by the Cr addition. Concentrations from 0.25% Cr to 8.5% Cr were included in this study. The higher the Cr content, the lower the EPF and TPF values. Cr concentration of only 0.5% reduced the EPF value to 50% of that of the Cr-free steel and 1% Cr and greater reduced the EPF close to 0.

Because of the higher negative free energy of formation of Chromium oxides over Iron oxides and because of the passive nature of Chromium, it was contemplated that small additions of Cr to steel would enhance both the thermal energy and thermal life properties.

One explanation of the reverse effect by the Cr addition might be attributed to an unexpected reaction of the leached or partially leached Cr-Fe alloy with the leaching media during removal of the BD diffusion metal. The Cr or Cr-Fe is converted to a compound during the leaching operation and is no longer available as an active metal lattice to participate in the pyrophoric air-oxidation reaction.

↳ One solution to the above problem might be to select another leaching media that would not react with the active Cr-Fe alloy. At the same time, however, the leaching bath must effectively remove most all of the BD diffusion metal.

ADDENDUM I

ENERGY DISPERSIVE X-RAY ANALYSIS



micron inc.
Analytical Service Laboratory

Report #R-5209

January 20, 1982

Mr. A. Baldi
ALLOY SURFACES COMPANY, INC.
100 South Justinson Street
Wilmington, DE 19899

Electron Probe Analysis of Sample BD-14-42

- Sample: * BD-14-42 #35
- Request:
- * Obtain semi-quantitative EPA profiles across a polished cross section for chromium and iron.
 - * Obtain data for one locus for sample BD-14-42.
 - * Compute quantitative concentration results from the profiles, for chromium and iron.

Results: Semi-quantitative profiles for iron and chromium are presented as Figures 1-3 for sample BD-14-42. Figure 3 is an expanded ordinate chromium profile.

The chromium level throughout the thickness of sample BD-14-42 was relatively uniform.

Quantitative concentrations are summarized in Table 1 and are detailed in the attached Tables.

Table 1

Quantitative Analysis Summary

<u>Sample</u>	<u>Weight Percent</u>	
	<u>Chromium</u>	<u>Iron</u>
BD-14-42	1.1	96.5

Table 1

BD-14-42

13-JAN-82

MICRON INC.

QUANTITATIVE ELECTRON MICROANALYSIS

	ELEMENT	K-RATIO	WT. CONC EST	WT. % CONC.
Cr	24	.014	.014	1.07632
Fe	26	.958	.958	<u>96.513</u>
				97.58

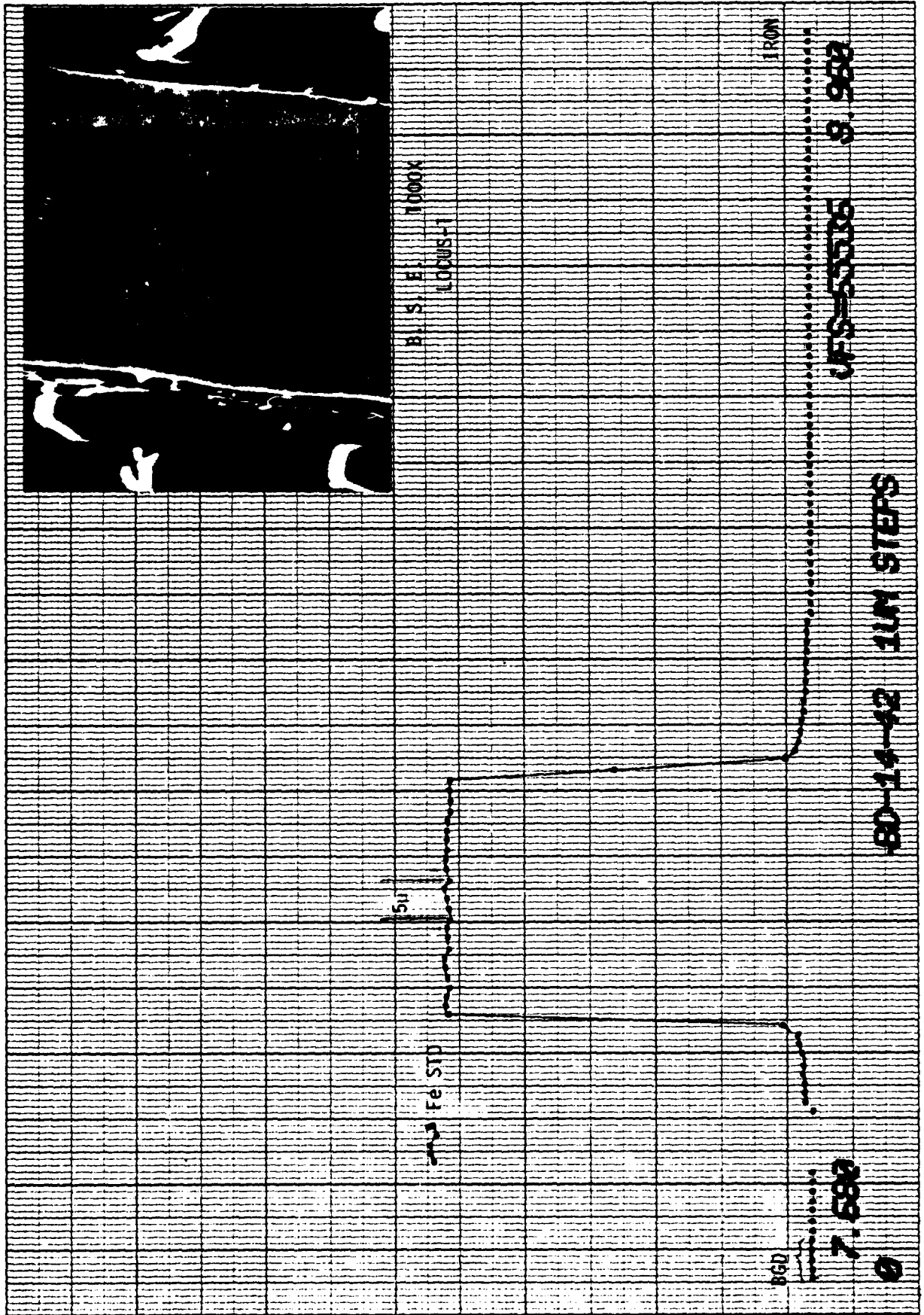


Figure 1

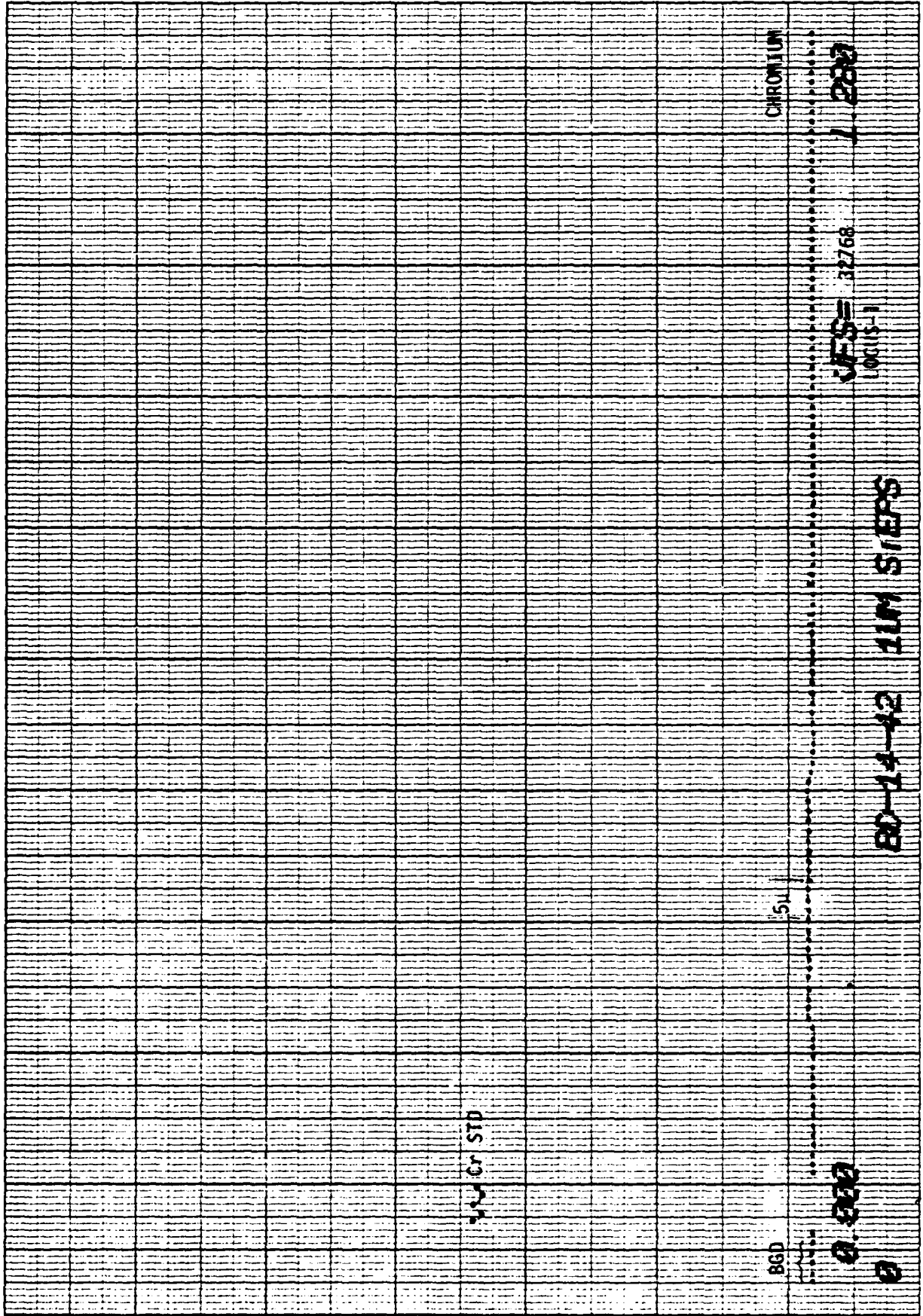
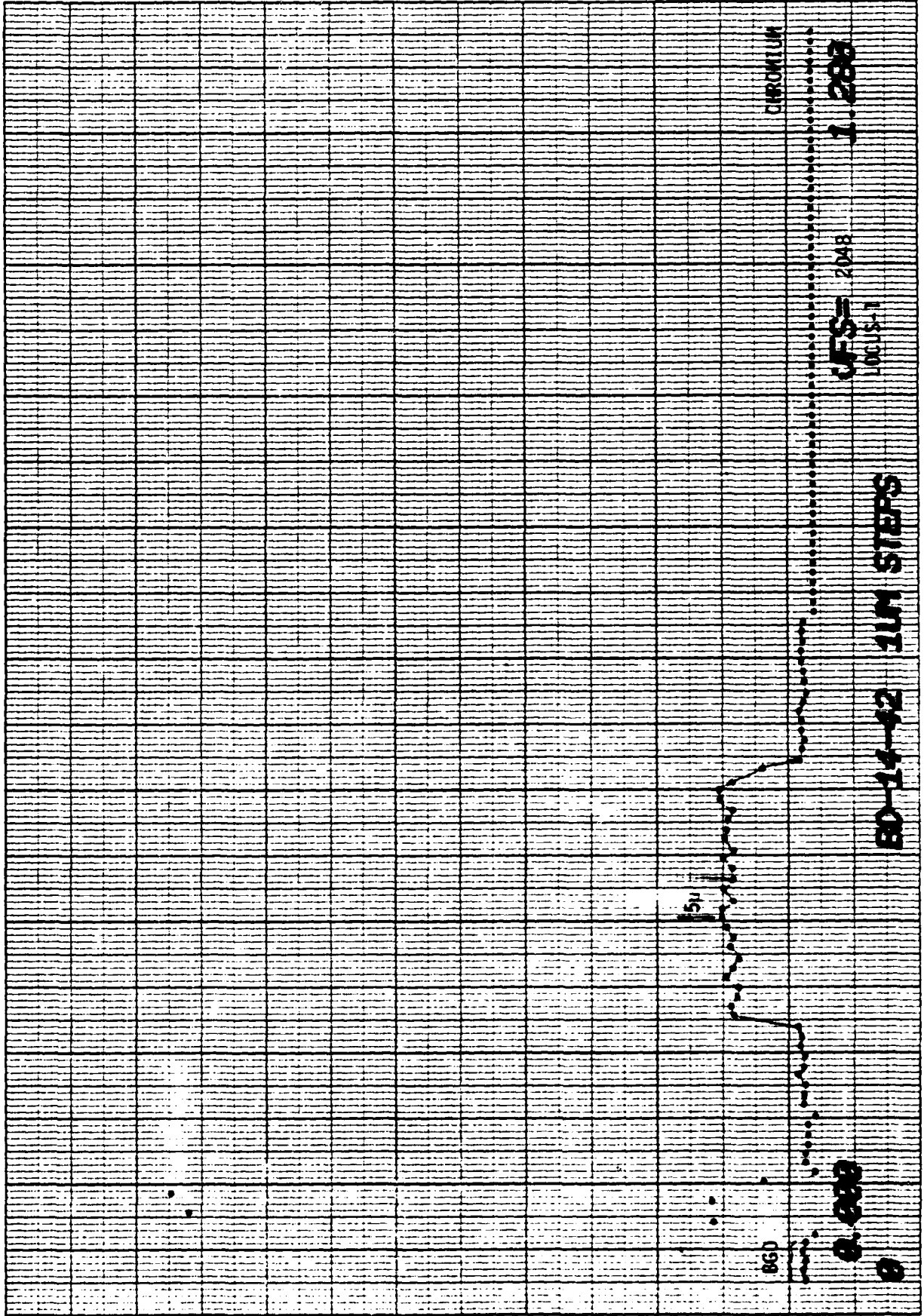


Figure 2

K•E 20 X 20 TO THE INCH • 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

46 1240





January 14, 1982

Report #R-5007

Mr. A. Baldi
ALLOY SURFACES COMPANY, INC.
100 South Justinson Street
Wilmington, DE 19899

Chromium-Coated 1010 Steel

Sample Code: #3 PD ONLY

#4 Cr + PD

Request:

- * Profiles for chromium and iron across the thickness of each sample.
- * Quantitative analysis for chromium and iron.

Results: The attached semi-quantitative profiles for chromium and iron show that chromium had diffused uniformly throughout the entire thickness of each sample.

Quantitative analysis results (weight percent) are:

<u>Sample</u>	<u>Cr</u>	<u>Fe</u>
#3	3.2	96.2
#4	8.3	91.6

(for details, see Tables 1-2).

The expanded scale profiles (Figs. 3-4, 7-8) show the chromium was relatively uniform throughout the entire thickness of each sample.

[22]

P.O. Box 3536 Wilmington DE 19807 ☐ (302) 998-1184

Table 1

#3 PD ONLY

13-JAN-82

MICRON INC.

QUANTITATIVE ELECTRON PROBE MICROANALYSIS

	ELEMENT	K-RATIO	INIT CONC EST	WT. % CONC.
Cr	24	.041	.041	3.21017
Fe	26	.954	.954	96.1925
				<hr/> 99.4

Table 2

#4 CR+ PD

13-JAN-82

MICRON INC.

QUANTITATIVE ELECTRON PROBE MICROANALYSIS

ELEMENT	K-RATIO	INIT CONC EST	WT. % CONC.
Cr 24	.102	.102	8.28229
Fe 26	.899	.899	91.5636
			<u>99.84</u>

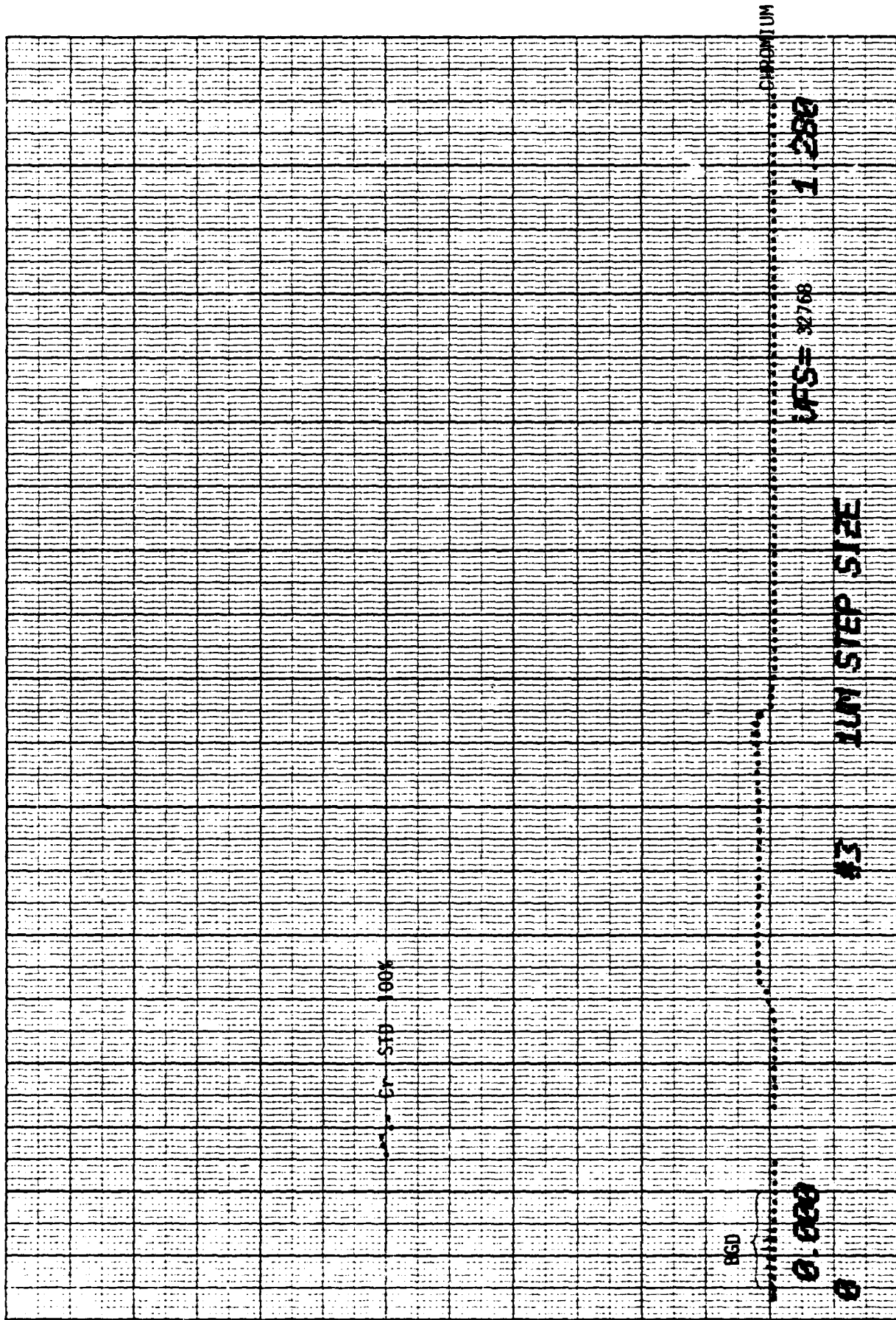


Figure 1

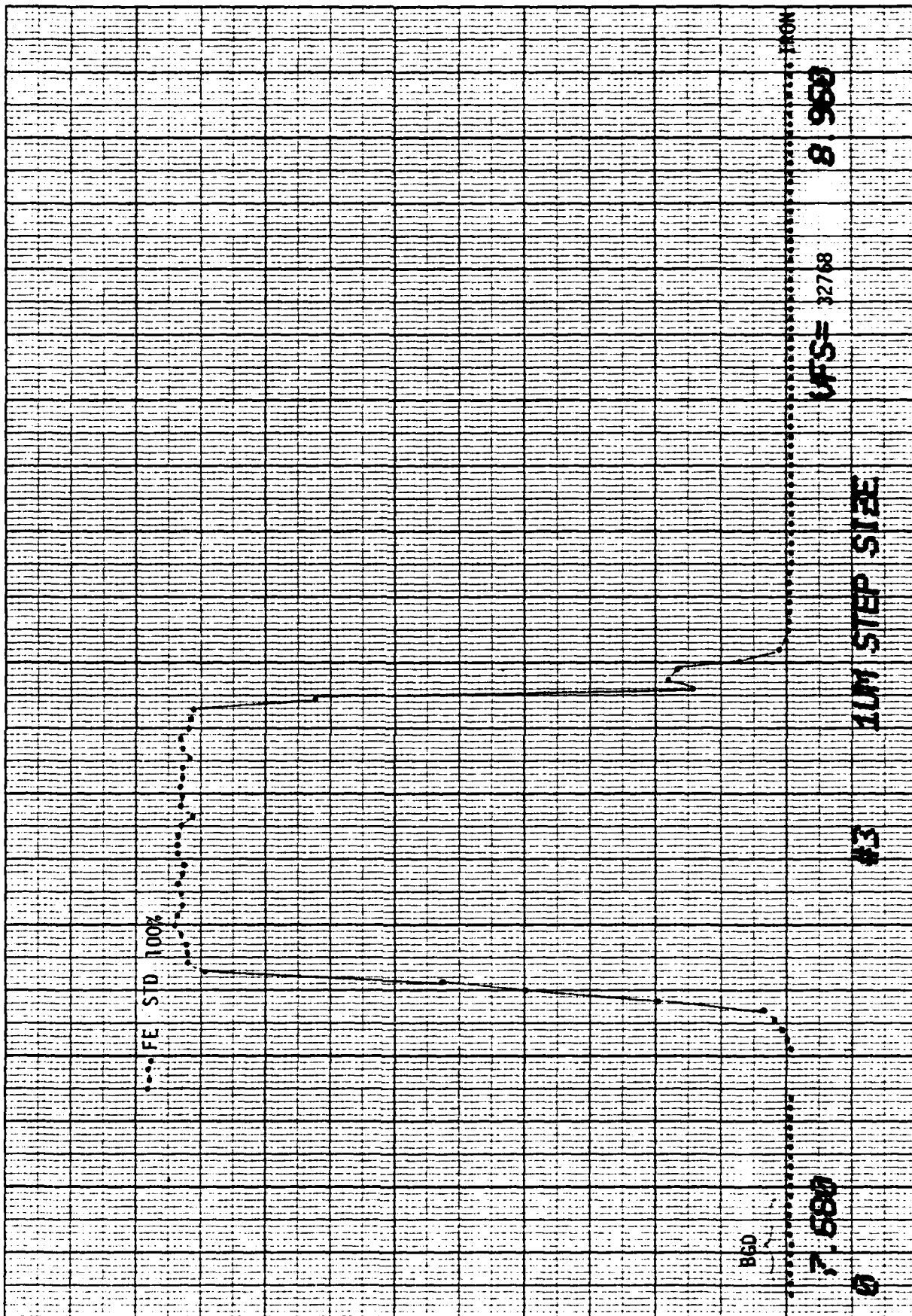
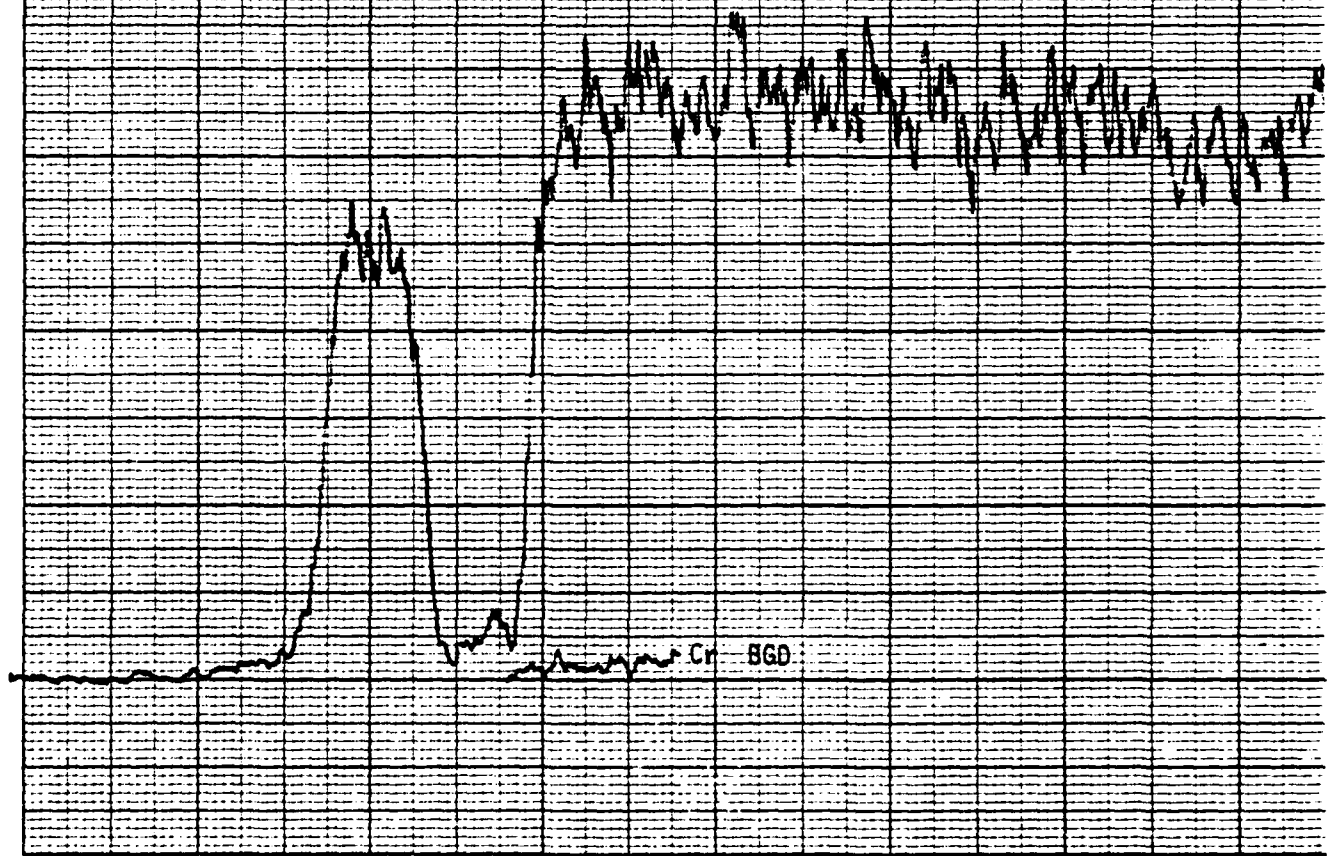


Figure 2

OPER. COND. 20KV: 0.015A: 3000X: PROBE SCAN

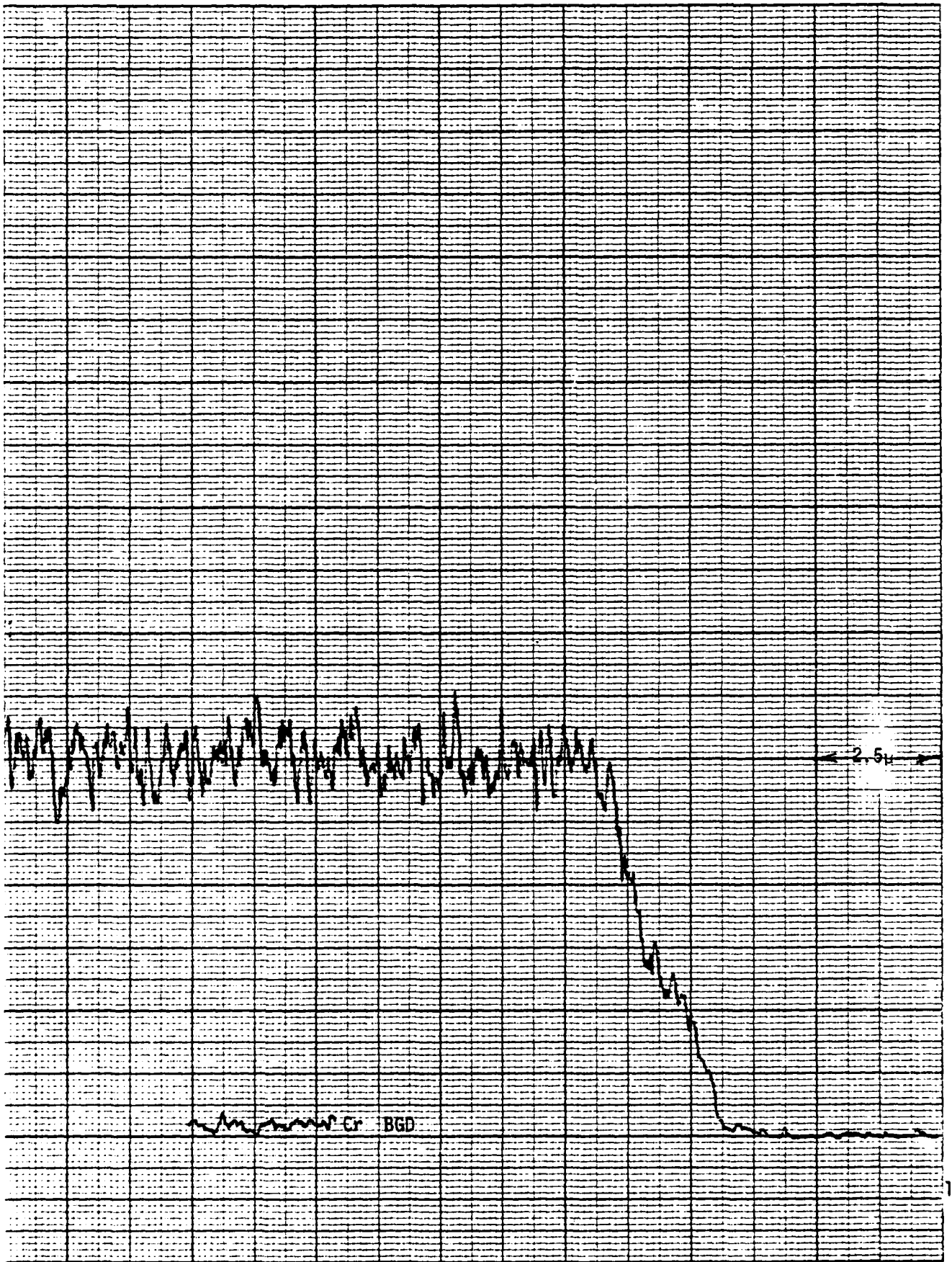
SAMPLE #3 - CHROMIUM PROFILE



47 1240

K-E 20 x 20 TO THE INCH • 10 x 15 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

Figure 3



Cr

103

OPER. COND. 20KV; 0.015uA; 3000X; PROBE SCAN

SAMPLE #3 - IRON PROFILE

47 1240

20 x 20 TO THE INCH • 10 x 15 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

K•E

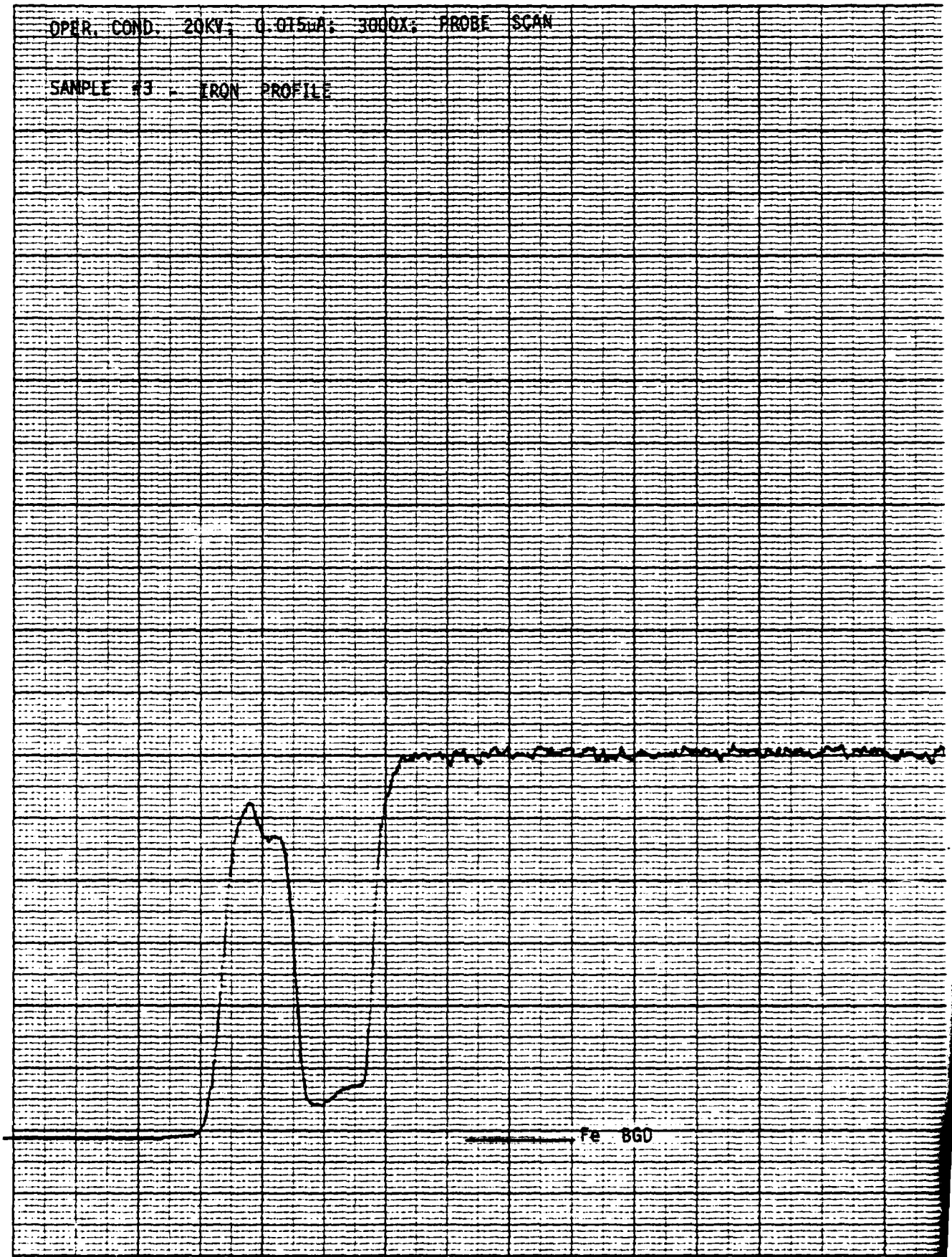
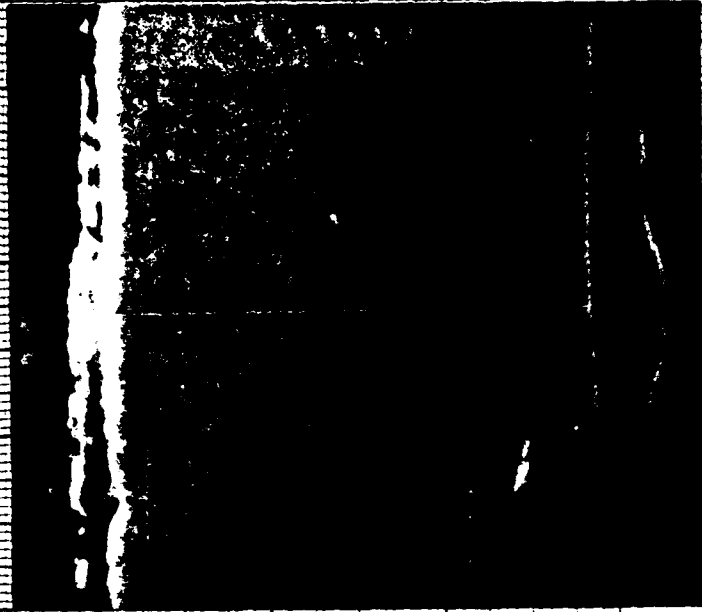
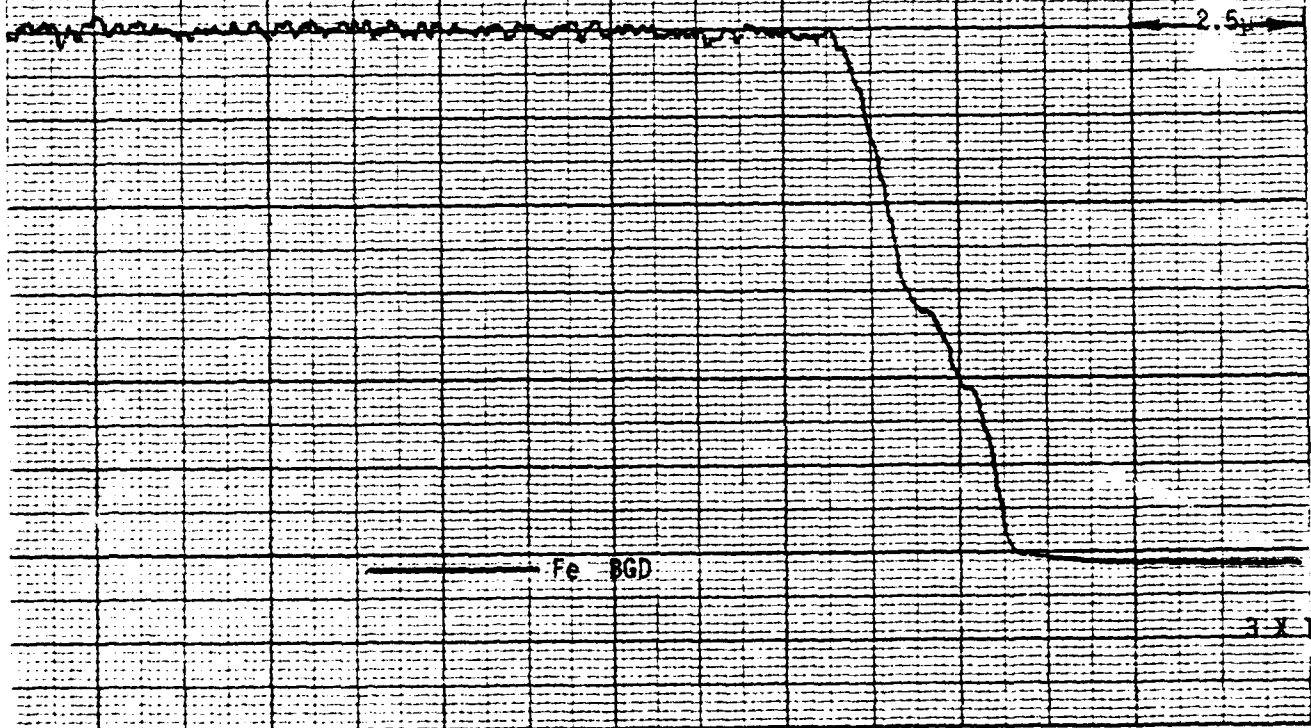


Figure 4



B. S. E. 3000X
LOCUS-1



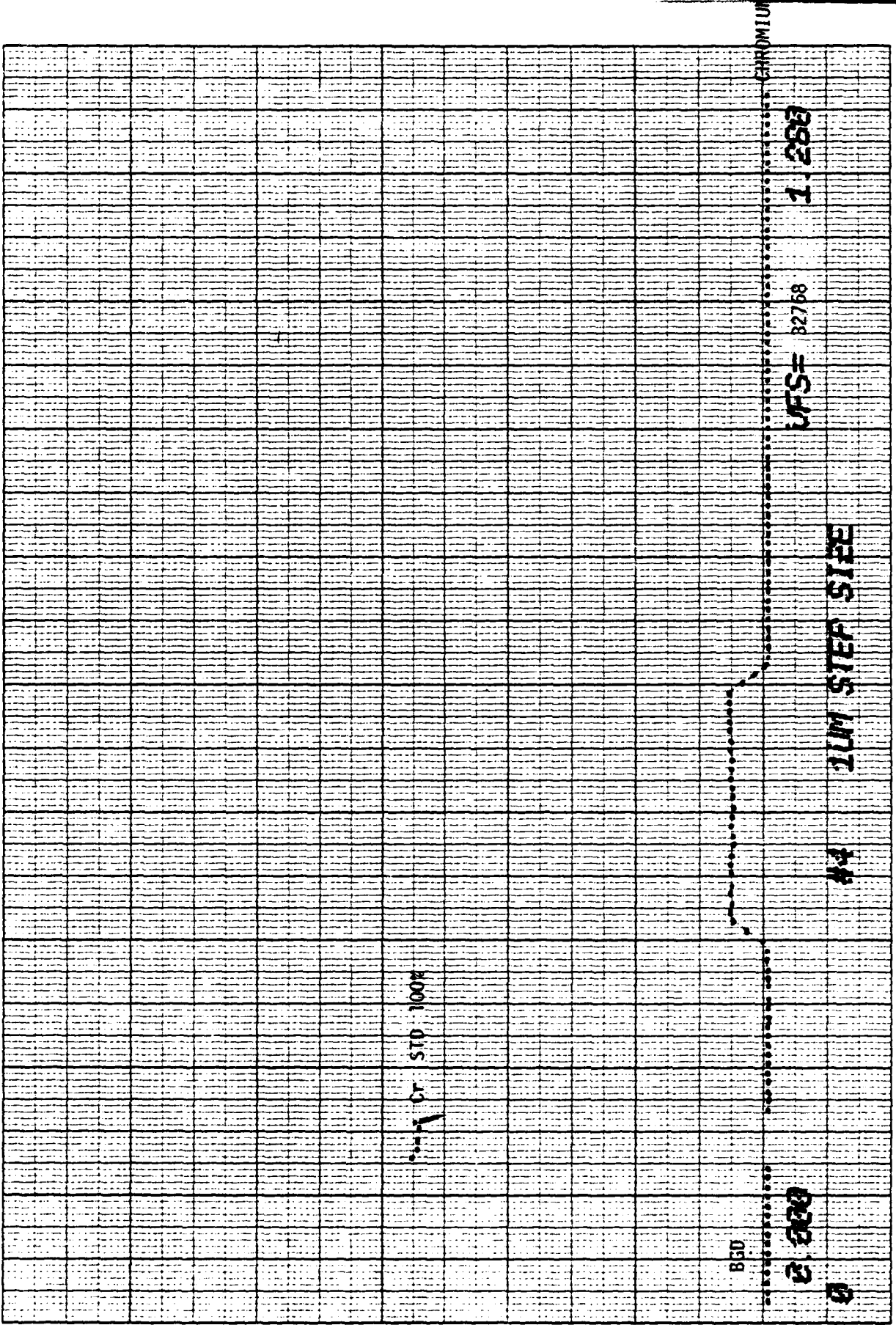


Figure 5

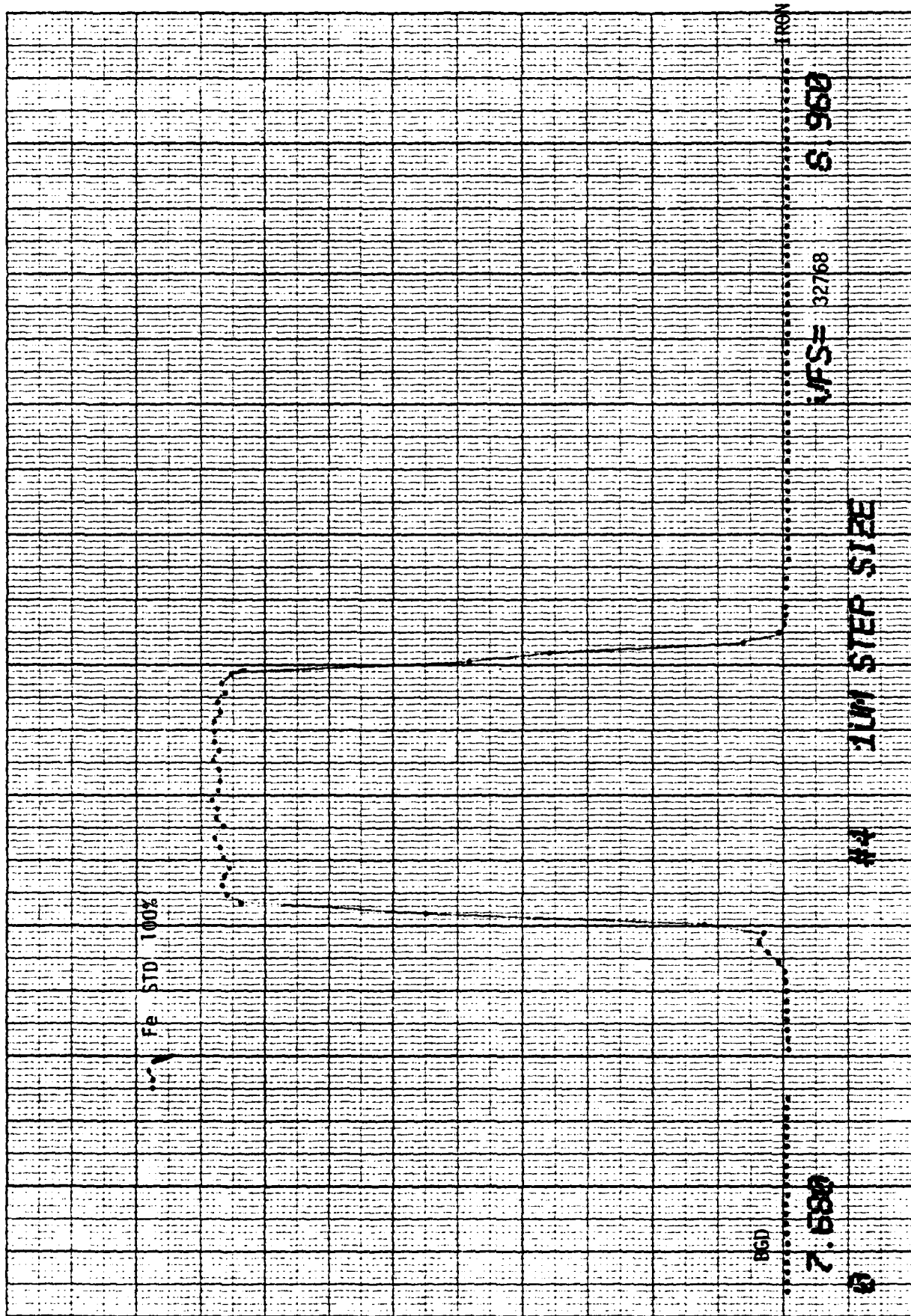
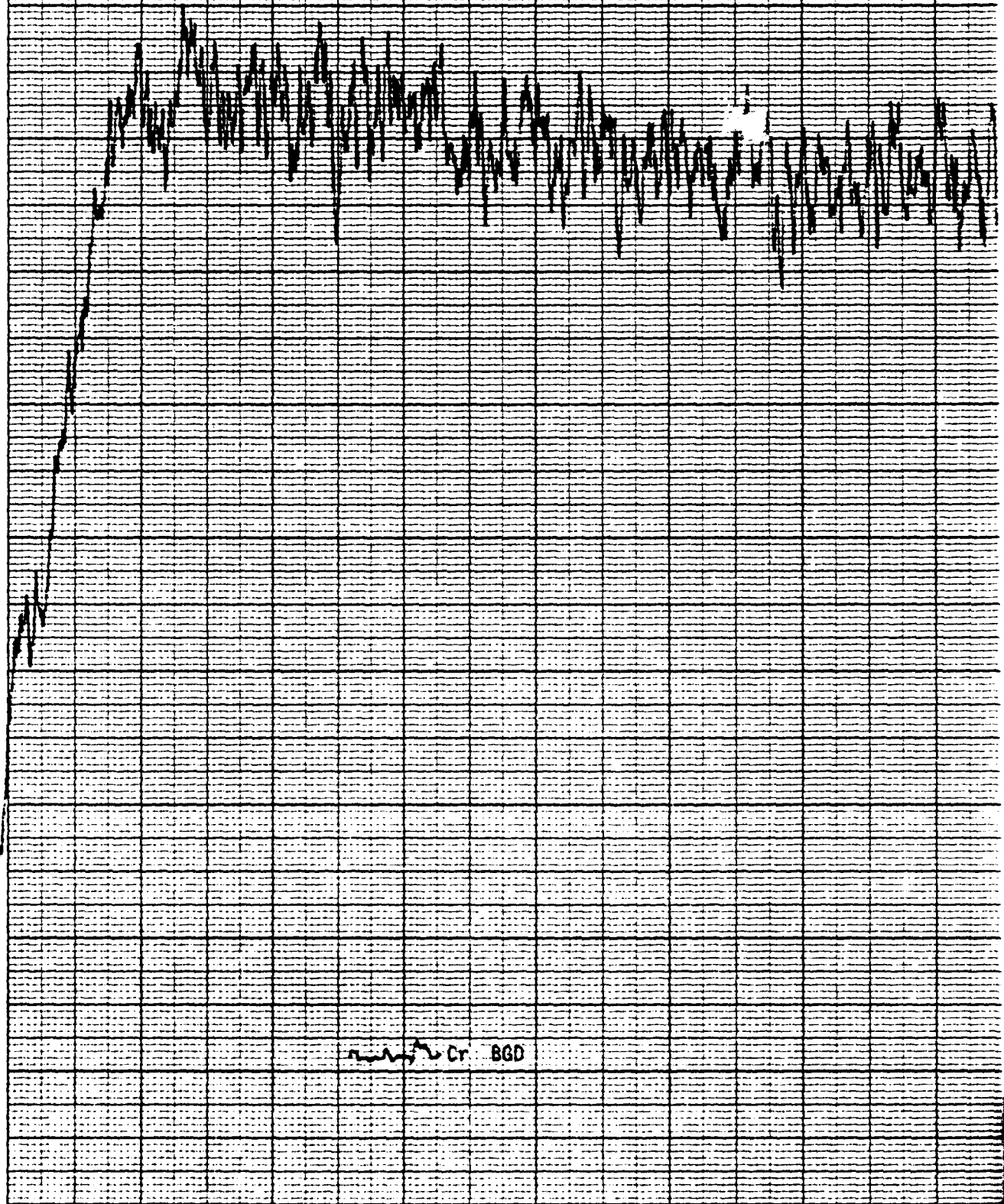


Figure 6

OPER. COND. 20KV, 0.015uA, 4000X, PROBE SCAN

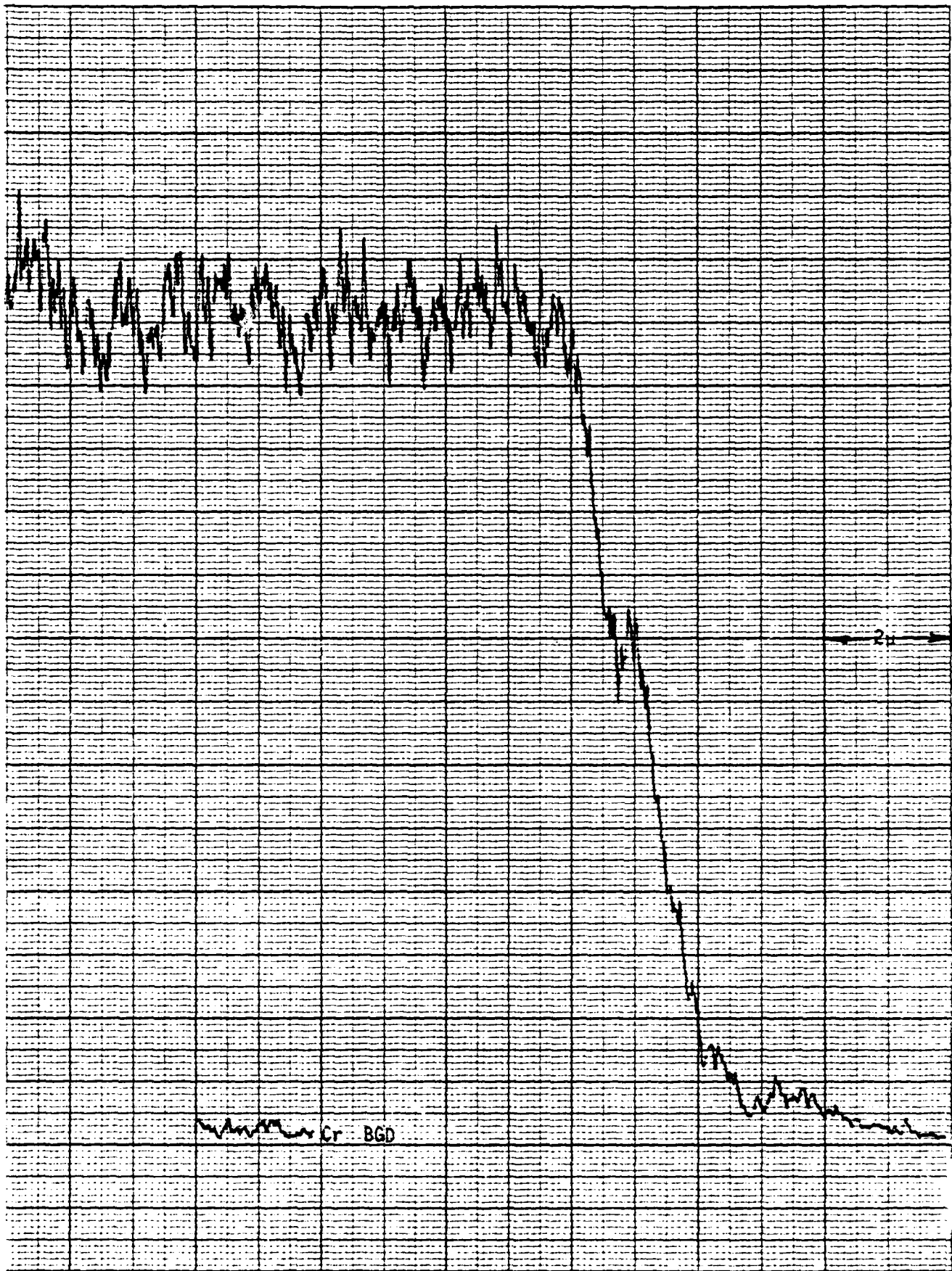
SAMPLE #4 - CHROMIUM PROFILE



47 1240

K•E 20 x 20 TO THE INCH • 10 x 15 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

Figure 7



OPER. COND. 20KV; 0.015A; 400X; PROBE SCAN

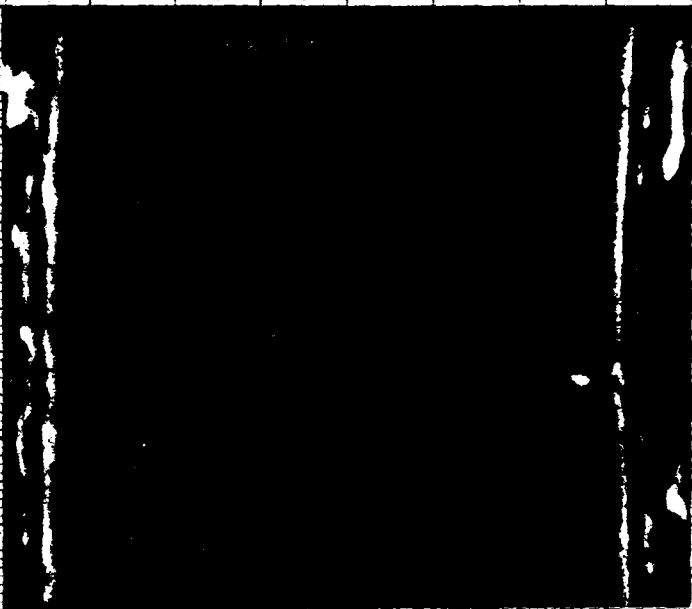
SAMPLE #4 - IRON PROFILE



47 1240

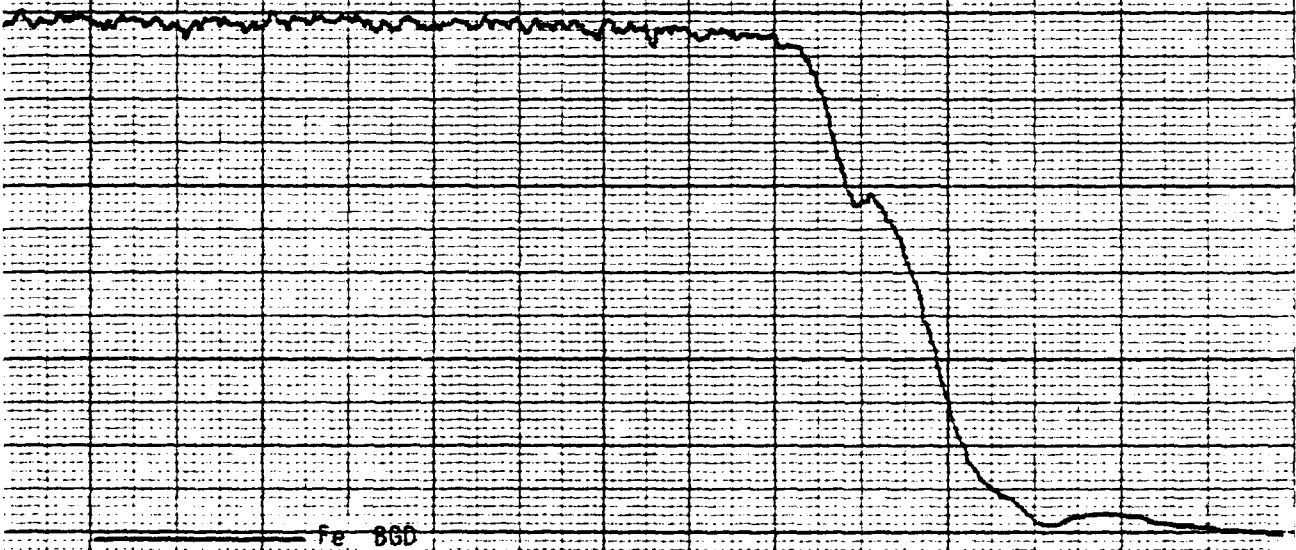
K α 20 X 20 TO THE INCH • 10 X 15 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

Figure 8



B. S. E. 4000X
LOCUS-1

2



Fe BGD

Fe

3×10^4

ADDENDUM II

Procedure used for Atomic Absorption Analysis of Fe-Cr Foils

PURPOSE: To analyze foils for % Cr

METHOD:

1. Cut the foil to be analyzed to a size which weighs approximately 0.060 gms. Place in a 125-ml Erlenmeyer flask.
2. Weigh 0.060 gms of Fe-Cr standard powder into a 125-ml Erlenmeyer flask. (This is a mixture of 2% by weight Cr powder and 98% by weight Fe powder).
3. Add 30cc of 50% HCl (v/v water) to each flask.
4. Heat for 15-20 minutes to reduce the volume.
5. Quantitatively filter the solution through Whatman No. 2 filter paper into a 200 ml volumetric flask.
6. Dilute to the mark with distilled water.
7. The solutions are now ready to analyze on the A.A. spectrophotometer.

A.A. Instrument Information

Model: Perkin-Elmer Model 305
Wavelength Setting: 357.9 nm (U.V.)
Slit Setting: 4 (0.7 nm)
Light Source: Hollow Cathode Lamp
Flame Type: Air-Acetylene Flame (reducing; rich, yellow)

8. Calculation for % Cr

$$\% \text{ Cr} = \frac{\text{ppm Cr} \times 0.2}{\text{sample weight (mg)}} \times 100$$

ADDENDUM III

BD-4-57 Test Procedure

A. MATERIALS

1. Leeds & Northrup Speedomax with recorder utilizing speed -10" min.
2. Pt-Pt-10Rh thermocouple 0.002" diameter wire with 0.005" junction.
3. Make up of test rig.
 - a. Platform with clamps
 - b. Line for Argon injection
 - c. Plastic snorkel to hold thermocouple
 - d. Lever to bring specimen in contact with thermocouple junction
 - e. Specimen holder
 - f. Plastic bell
 - g. Fan with 6" sec. flow capacity

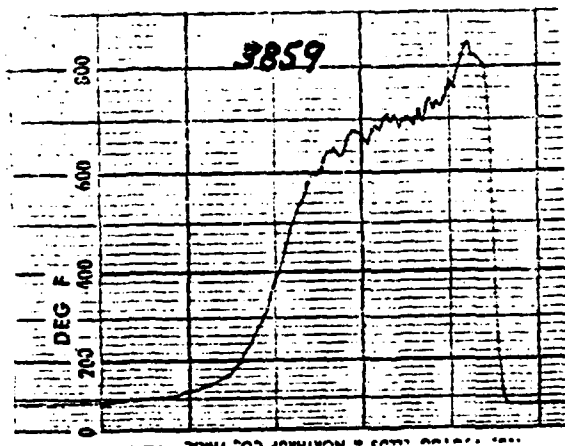
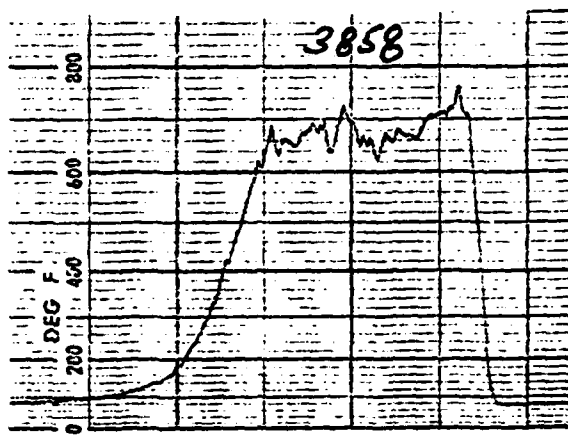
B. PROCEDURE

1. Thoroughly rinse BD test specimen in water.
2. Transfer test specimen to specimen holder.
3. Immediately place bell over specimen and clamp bell to base. Start argon flow and turn fan on.
4. Continue argon flow until specimen is dry.
5. Move lever until BD specimen just contacts thermocouple junction.
6. Turn recorder on.
7. Unclamp bell from base.
8. Permit fan to run and 6'/sec. air flow will be delivered to specimen.
9. Turn argon off.
10. Raise bell from base.
11. Record until indicator on recorder is below threshold temperature.
12. Measure thermal life from time-temperature profile.

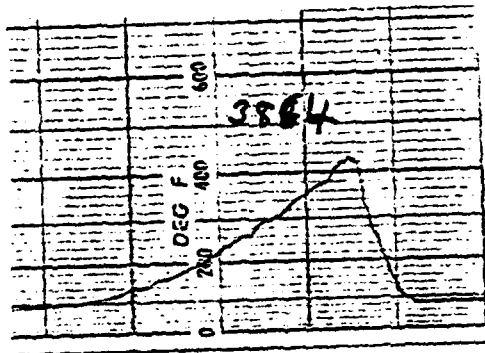
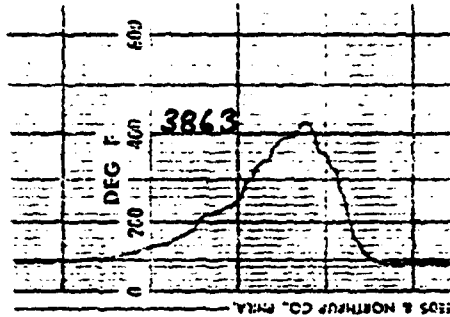
ADDENDUM IV

TIME-TEMPERATURE PROFILES

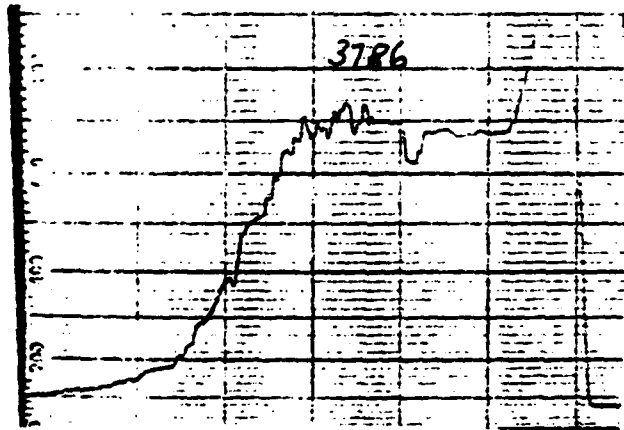
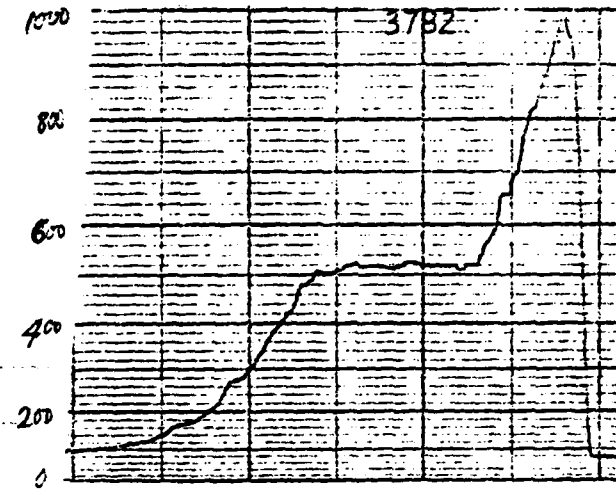
0% Cr
BP-14-63



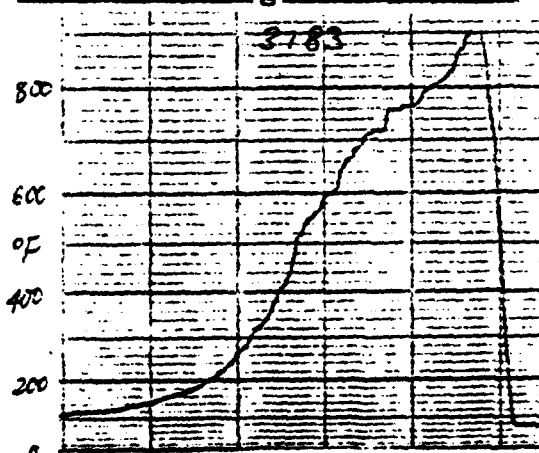
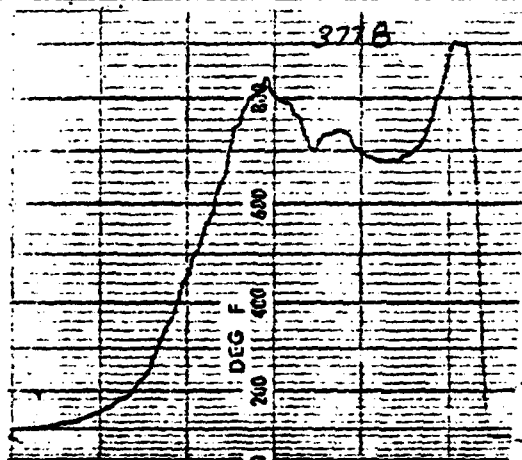
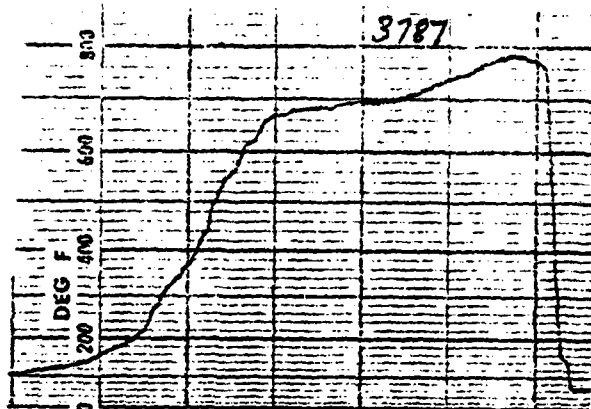
2.5% Cr
BD-15-15



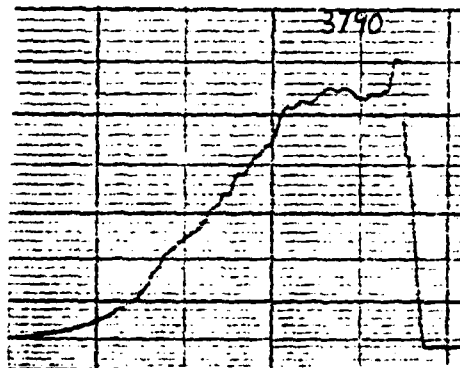
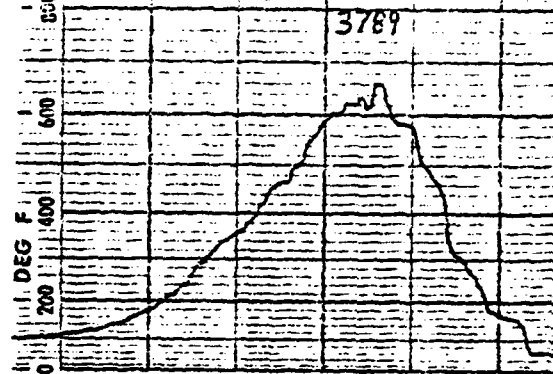
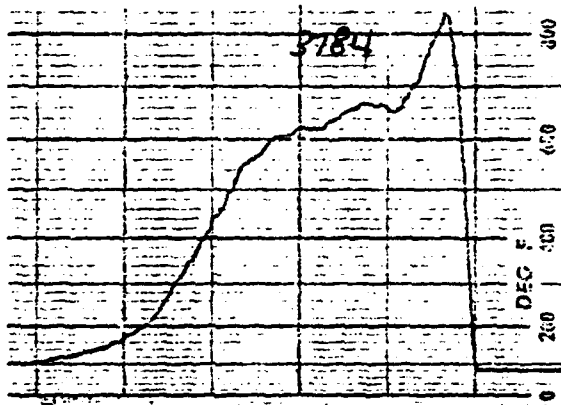
0% Cr
BD-14-27



0.25% Cr BD-14-27

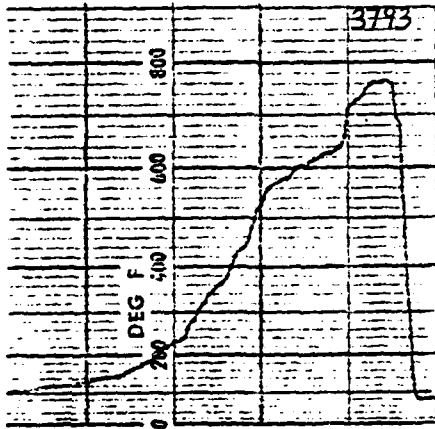
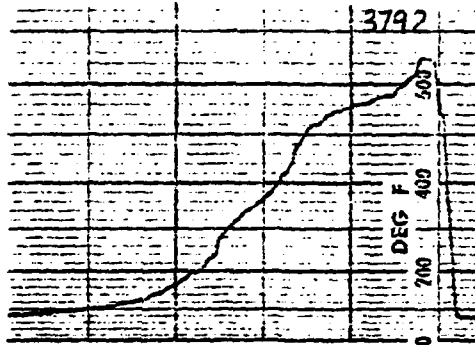


0.80% Cr
BD-14-27

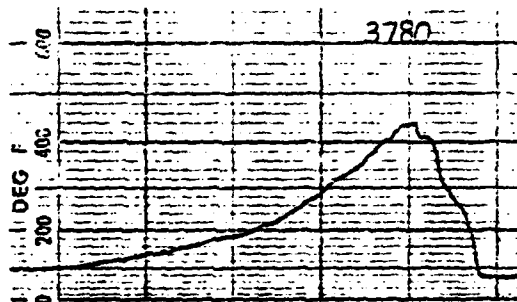
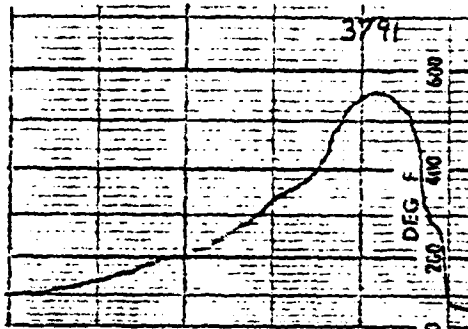


1.50% Cr

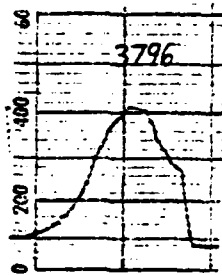
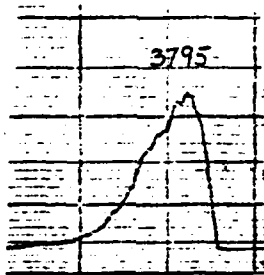
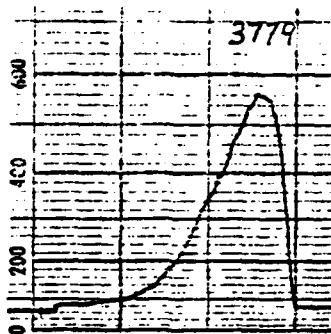
BD-14-27



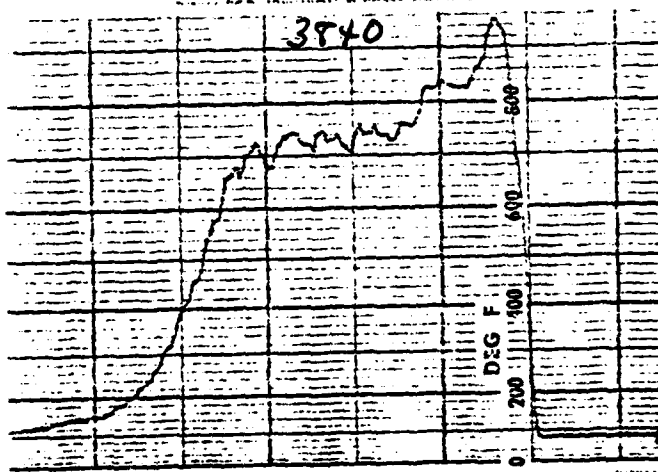
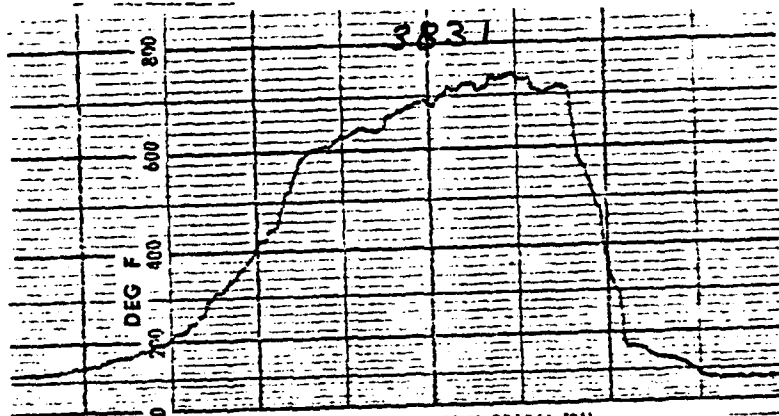
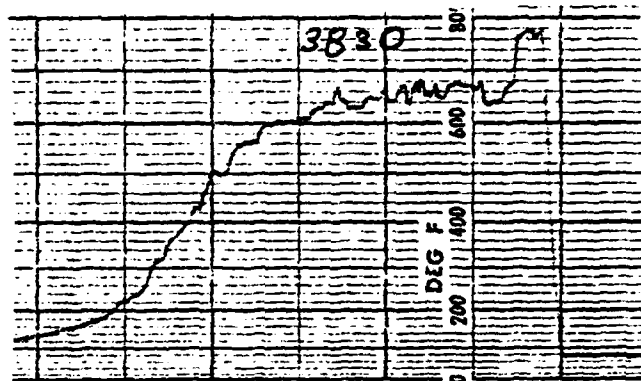
2.20% Cr
BD-14-27



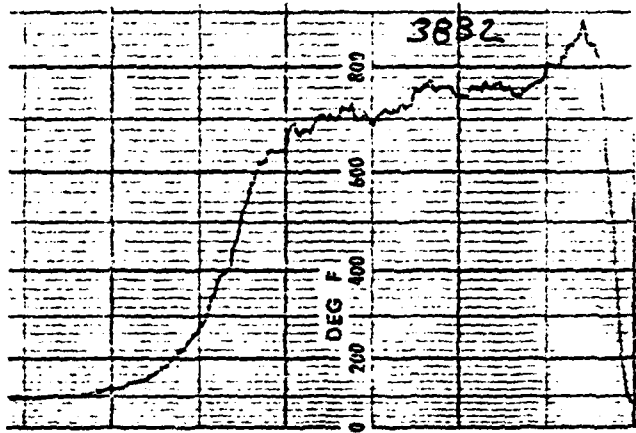
6.3% Cr
BD-14-27



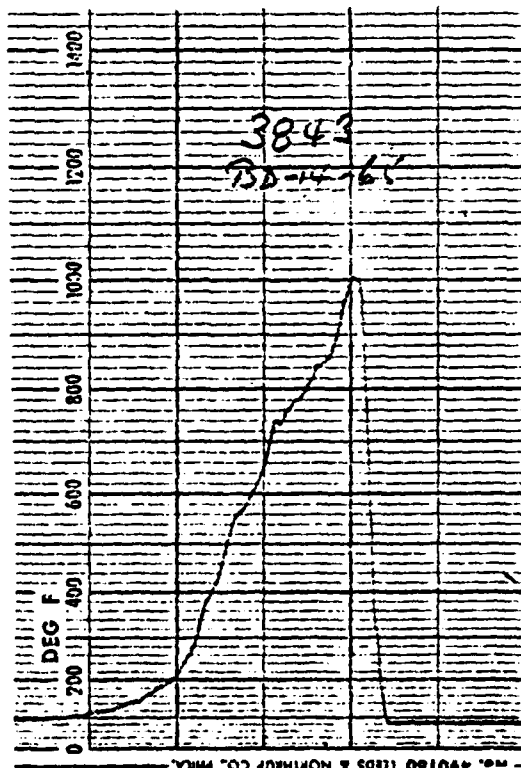
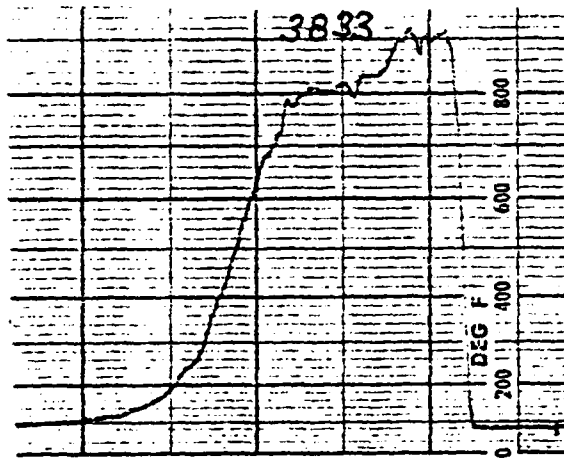
0% Cr
BD-14-63



0.50% Cr
BD-14-63

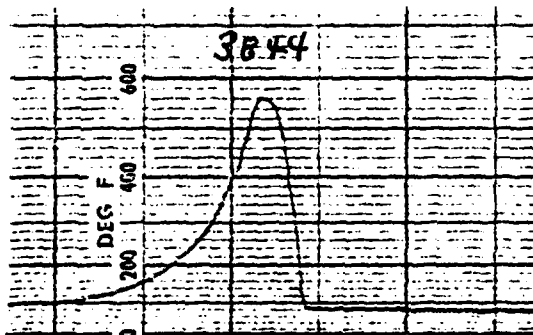
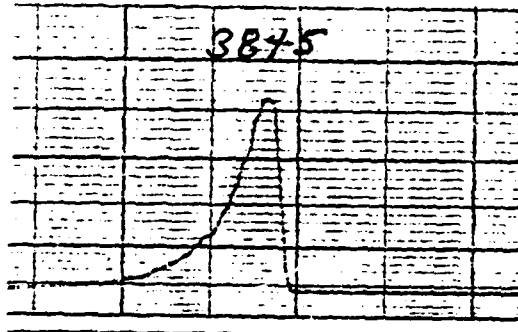
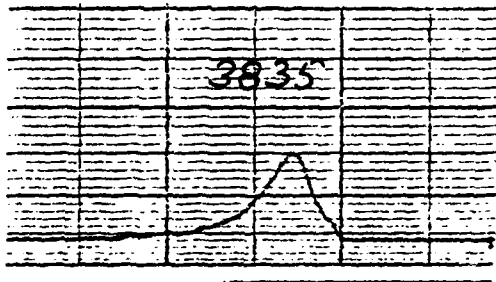
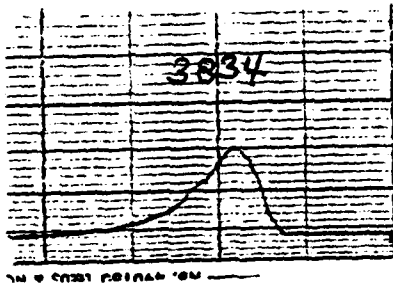


1.7% Cr
BD-14-63

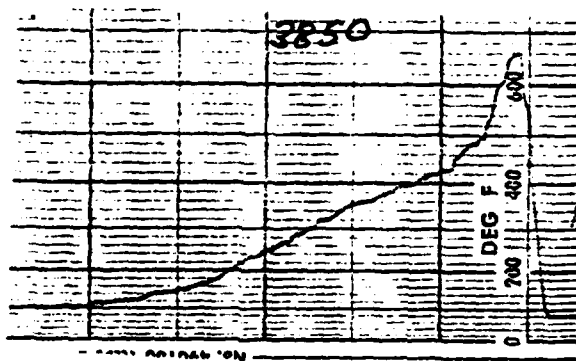
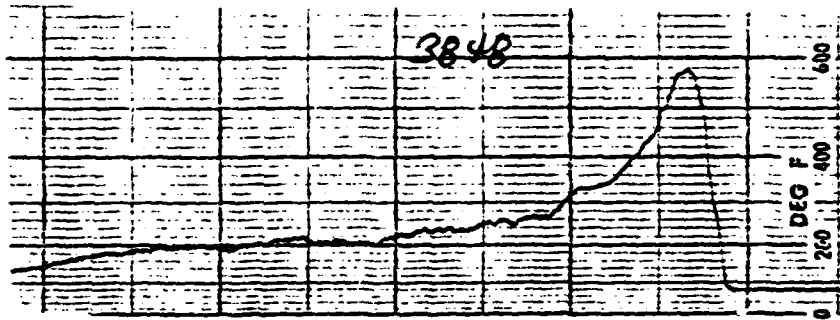


— MO. AVIATION LEADS & NORTHROP CO. PHA.

2.5% Cr
BD-14-63



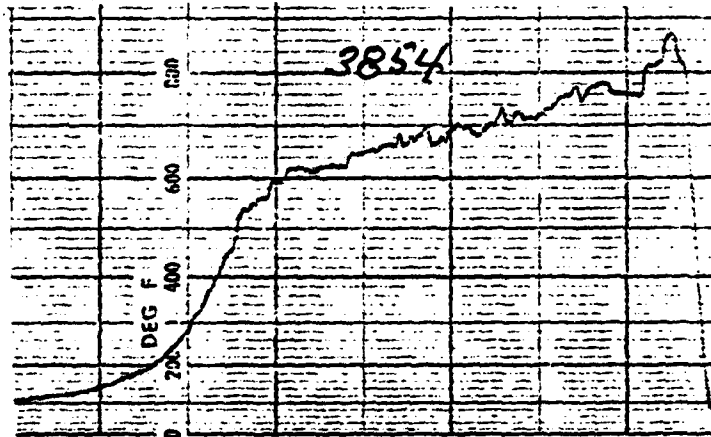
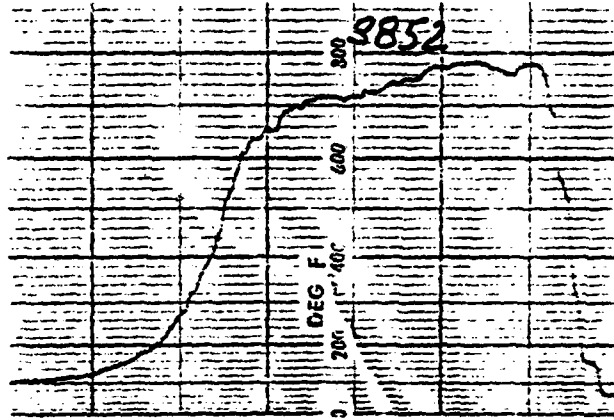
2.5% Cr
BD-14-63



3.5% Cr
BD-14-63

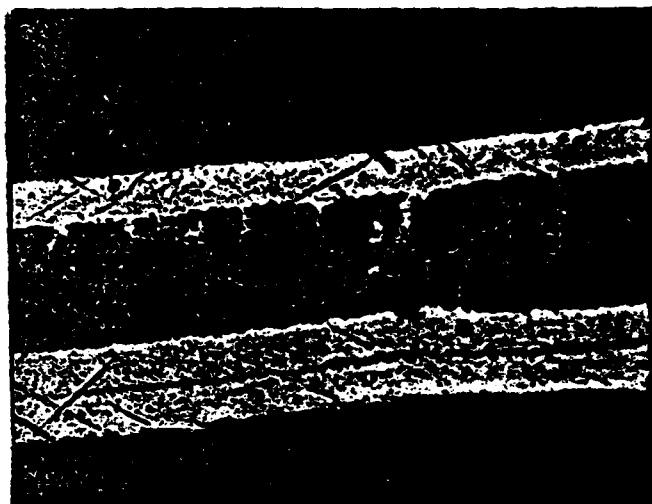


0% Cr
BD-14-63

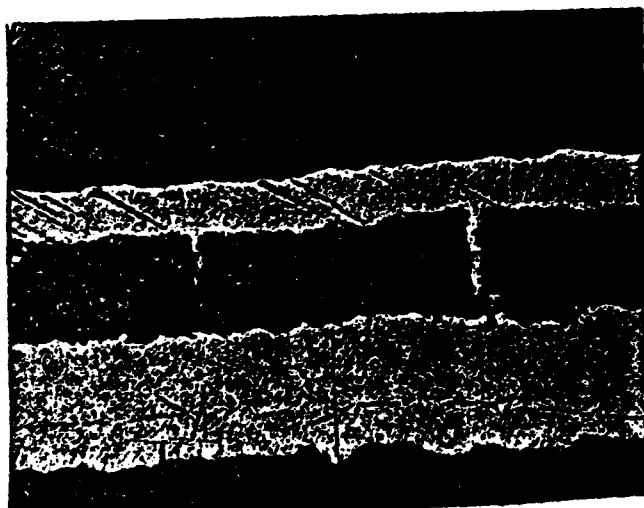


ADDENDUM V

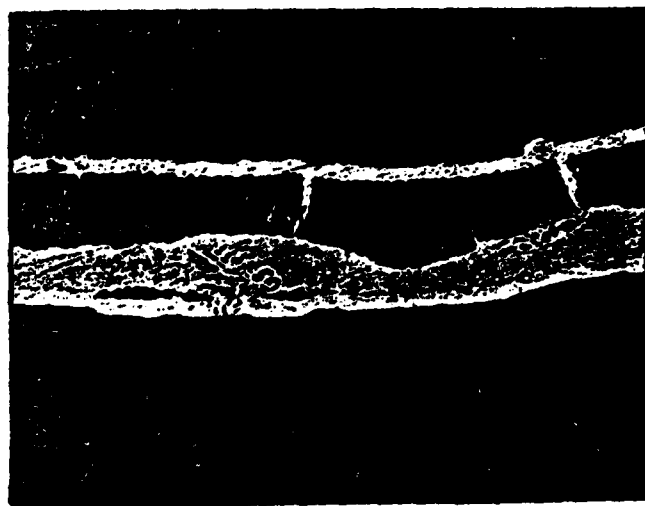
PHOTOMICROGRAPHS OF
ONE SPECIMEN FROM
EACH GROUP TESTED



N299
450X



N302
450X

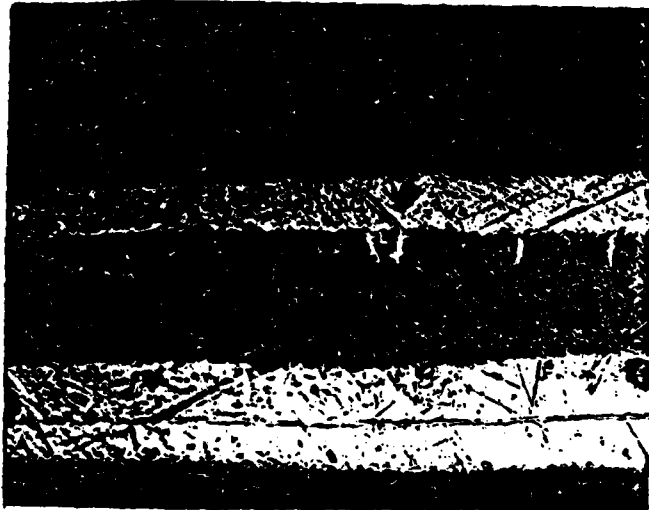


N304
450X



N 277

450X



N 273

450X

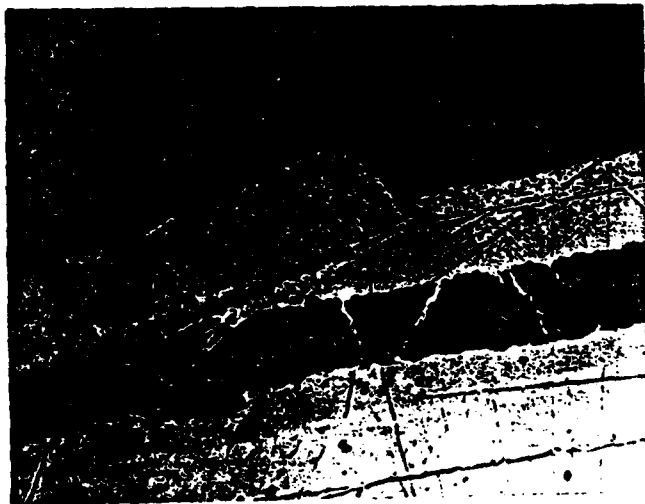


N 275

450X



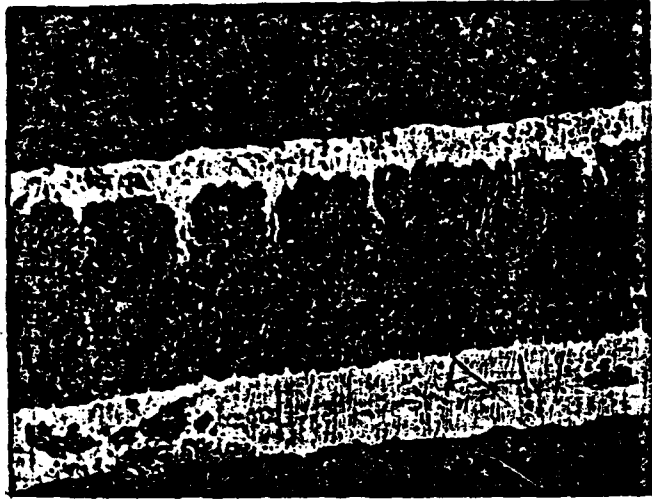
274
450X



279
450X

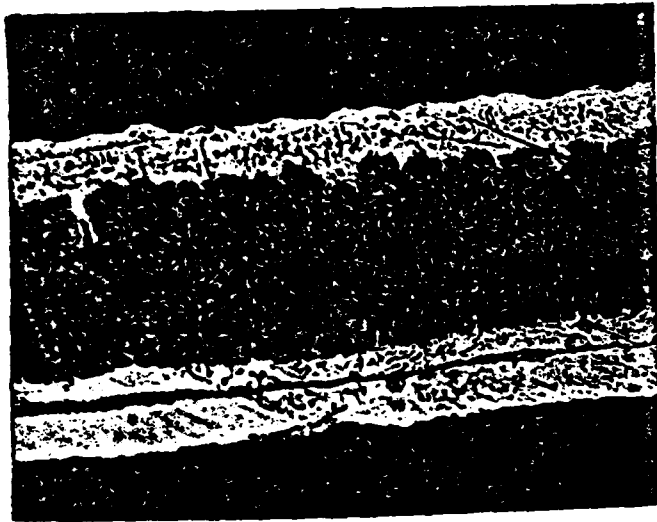


276
450X



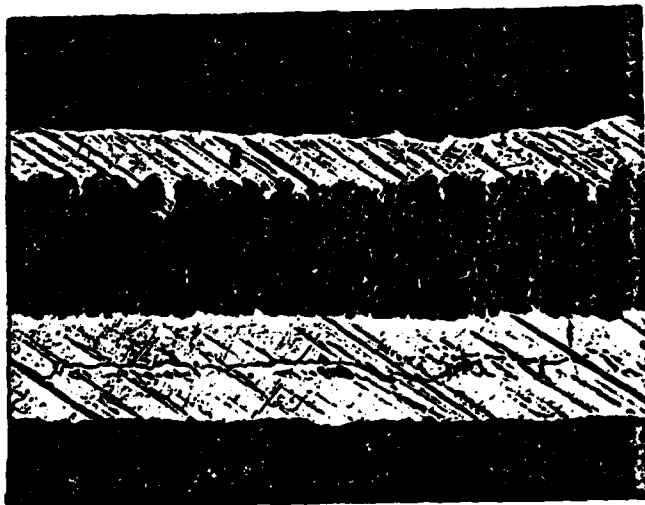
N289

450X



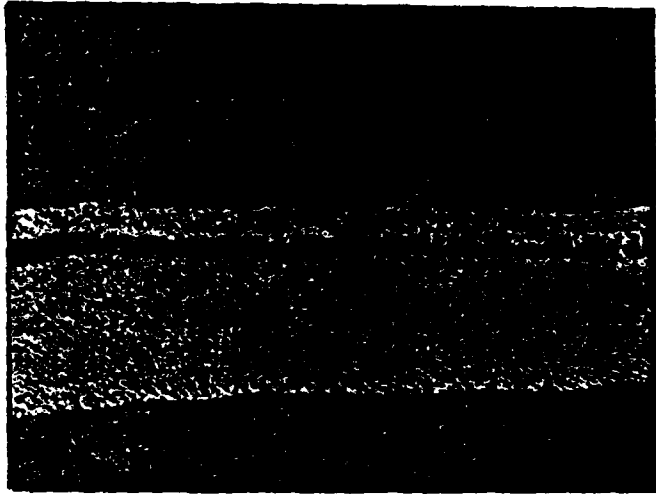
N267

450X

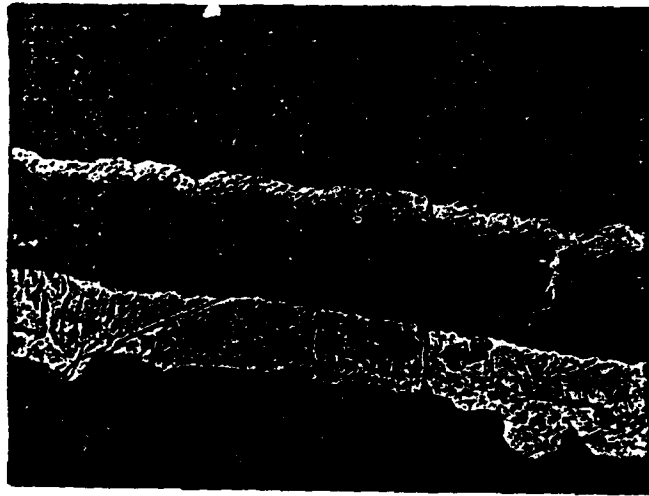


N305

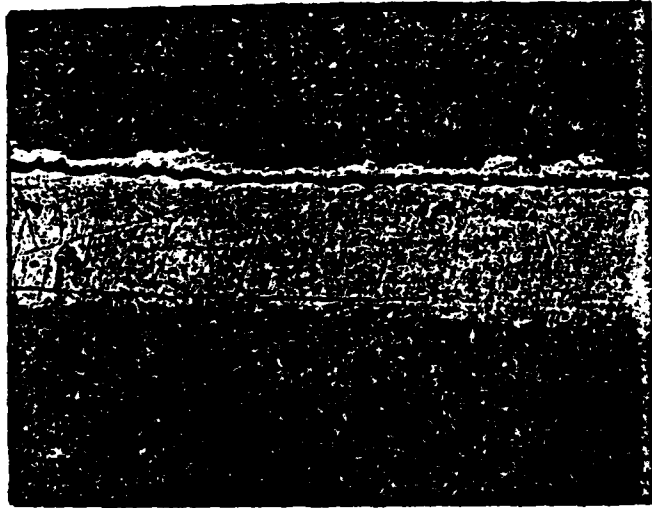
450X



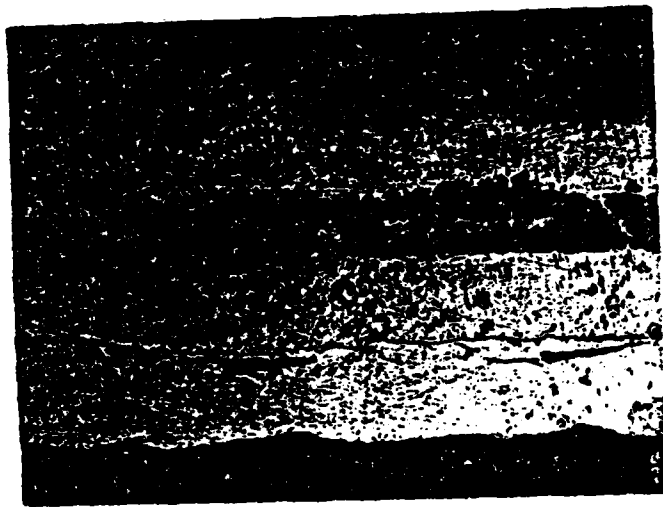
N268
450X



N280
450X



N269
450X



N271
450X