



Copper Tubing Evaluation in A Natural Gas Environment

**Project No.#: TS00-M036
Final Report, July 3, 2002**

Work performed jointly for
Copper Development Association Inc. and
Southern California Gas Company

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1.0 INTRODUCTION

This summary report documents test results and observations from exposing bare and tin-lined copper tubing to a natural gas environment that is typical of gas composition in the Southern California Gas Company territory. These samples were subjected to a flowing gas stream for a period of 52 weeks. The actual exposure period was between October 9, 2000 and November 19, 2001. During this period the test assembly received a total flow volume of 3,504,000 cubic feet of natural gas, which is equivalent to 15.96 years of average residential gas usage. The primary objective of this study was to evaluate and qualify the susceptibility of both bare copper tube and tin-lined copper tube to corrosion and hydrogen sulfide attack when conveying gas typically supplied to the Southern California Gas Company service areas. Comparison to the predetermined performance criteria shown in Section 2.5 would determine suitability for use of copper tubing in gas stream having similar chemistry.

2.0 SCOPE OF WORK

2.1 Design and Set-up of Copper Tubing Test Rack Assembly

The copper tubing test rack assembly was designed to simulate flow conditions that are representative of a typical full-scale residential house-line. It was configured to hold 4 sets of ½-in. copper tubing samples of 1-ft. length mounted on the test rack to be removed after specified exposure periods (**Figure 1**). These samples were exposed to flowing natural gas from Pico Rivera for periods of 4, 12, 24 and 52 weeks. The projected consumption of natural gas flow through a typical house-line was estimated to be: 4 weeks = 268,800 cubic feet; 12 weeks = 806,400 cubic feet; 24 weeks = 1,612,800 cubic feet and 52 weeks = 3,504,000 cubic feet. Using an average house-line usage of 219,000 cubic feet/year, the accelerated testing at specified exposure period was equivalent to the following usage in years; 4 weeks = 1.23 years, 12 weeks = 3.68 years; 24 weeks = 7.36 years and 52 weeks = 15.96 years. This is equivalent to a continuous gas flow of 400 cfh. Testing was interrupted 16 weeks after start-up due to repairs on the test rack air compressor. The test samples were not exposed to flowing gas during the repair and its interior surface was kept isolated from exposure to atmosphere. Continuous testing resumed 6 weeks later on April 4, 2001.

2.2 Assessment of Bare Copper Tubing and Tin-lined Copper Tubing

After each exposure period, the bare and tin-lined tubing samples were removed and evaluated for general appearance, tubing material loss, corrosion product and internal corrosion deposit. Each sample was weighed to determine material loss due to corrosion before and after the exposure period. Corrosion deposits in the filter were removed for further analysis and identification. Once the sample had been removed, it was replaced with 0.25-in. outside diameter steel pipe with similar inside diameter as the copper samples. This was intended to maintain the flow characteristic of the assembly during the exposure period.

2.3 Corrosion Products Identification

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The corrosion products collected were analyzed after each exposure period. Elemental testing was performed using a scanning electronic microscope with energy dispersive x-ray spectroscopy (SEM/EDX) capabilities.

2.4 Natural Gas Composition Analysis

Sampling of natural gas quality was conducted for the duration of the project to monitor the varying gas composition in the Pico Rivera distribution system. Natural gas composition was analyzed to identify potentially corrosive constituents in the gas. As a minimum, sulfur, hydrocarbon, water content, other corrosive solids and BTU content of the natural gas were identified for correlation to corrosion or corrosion byproduct creation. Sampling of the gas consisted of 2 Tedlar bags to measure sulfur content and 2 stainless steel cylinders to measure the BTU and hydrocarbon content. Water content in the gas was tested at 300 psig or above to satisfy the analyzer's range of accuracy.

2.5 Performance Criteria

Performance criteria in evaluating the corrosion susceptibility of both bare and tin-lined copper tubing for use in gas streams having similar chemical compositions were based on three criteria: 1) useful service life of the tubing, 2) deposit buildup, and 3) deposit mechanism. Since criteria 2 and 3 are assumed to be direct contributors to criterion 1 and will be quantified to determine criterion 1, the calculation of the useful service life will use the quantified values of deposit and buildup for its determination. The acceptable service life will be 20 years and defined as having a wall thickness of greater than 80%. The deposit mechanism will be determined by the chemical composition, color, size and shape of the deposit. This accelerated testing will project the useful life of the tubing material to 16 years. Any additional time beyond 16 years will be extrapolated based on the best curve fit from the generated data points gathered.

3.0 TEST RESULTS

3.1 General Appearance of Bare and Tin-lined Copper Samples

52-Week Sample

The exterior appearance of the samples changed from shiny reddish brown to rusty dark brown color due to oxidation as exposure time increased. The reddish brown color had totally faded on the 52-week tubing sample and turned rusty dark brown. There were no signs of corrosion on the exterior surface of the samples.

The bare copper tubing was split in half to reveal the extent of internal corrosion as a result of natural gas flow during the 52-week exposure period. The interior surface of the sample exhibited a coating of black corrosion products distributed across the entire interior surface of the sample (**Figures 2 and 3**). The copper tube wall beneath this corrosion product layer exhibited some roughening of the surface as a result of shallow, indistinct surface pitting. This general surface pitting was not measurable by the available instruments. No distinct, deep/active corrosion pitting sites indicative of aggressive localized pitting corrosion were identifiable on the interior surface of the tube.

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The tin-lined copper tubing sample was visually inspected after being split lengthwise. The tin coating had discolored to charcoal black color with isolated pits. There were two areas where the protective tin coating exfoliated exposing the bare copper surface material underneath (**Figures 4 and 5**).

24-Week Sample

The exterior of the samples showed signs of oxidation and discoloration from weathering. When removed the filter appeared clean. The weight of the corrosion deposit in the filter assembly was determined by the difference of the pre- and post-exposure filter weight. Upon sectioning the samples horizontally, the interior of the bare copper tubing had a dark corrosion byproduct/deposit layer, coincident with numerous shallow pits. This pitting represented an increase in general surface roughness but did not appear to indicate any deep or otherwise aggressive pitting behavior. The pits were too shallow to be measured with the available instrumentation.

The interior surface of the tin-lined copper tubing sample showed some isolated, shallow pitting and some flaking of the tin coating.

12-Week Sample

The exterior of the samples showed some oxidation and water marks from weathering. When removed from service, the filter appeared clean and had an odor that would be expected from a mercaptan odorant. The brass fittings also showed a weathered appearance. Once the tubing was split in half lengthwise, the interior surface of the bare copper tubing exhibited a discontinuous layer of dark corrosion products. These products were a dark greenish black color and coincided with widespread, shallow pitting. The incipient pits were too shallow to be measured with available instruments and only resulted in overall surface roughness. No deep, active pits were identified.

4-Week Sample

The exterior of the samples showed some oxidation and water marks from precipitation. When removed, the filter appeared clean and relatively new. There was an odor from the filter that matched that expected from mercaptan odorant. Upon sectioning the tubing, the interior surface of the bare copper sample exhibited a black color. The interior surface of the tin-lined tube had a dark black color as well but had a dusting of a blue-green color on the tin coating.

3.2 Wall Loss Measurement

52-Week Sample

Corrosion products formed on the surface of the copper tubing, primarily copper sulfide, were friable and broke easily from tubing wall; light tapping on the tubing wall was usually sufficient to dislodge the corrosion products from the tubing wall. It is possible that once a corrosion layer was removed to expose bare copper, sulfides in the gas stream could continue to react with the exposed copper to form another layer. This process gradually resulted in general tube wall loss over time, however the wall losses did not indicate a significant reduction in wall thickness for the samples exposed for 52 weeks.

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The amount of wall loss was estimated by tubing weight changes before and after exposure period, assuming that wall loss was uniform throughout. Weight loss from the bare copper sample was measured to be 3.06 grams and weight loss from the tin-lined sample was 0.71 grams. The friable flakes transported downstream were collected in the filter. A mass balance analysis of the copper tube sample and the resulting corrosion products could not be used to verify or positively account for weight loss from the tubing sample since some corrosion products were lost while removing the filter from the test rack assembly.

24-Week Sample

There was a slight weight change in the before and after weights of the bare copper tubing sample. The weight of the sample was reduced from 309.25 grams to 308.31 grams, a net reduction of 0.94 grams. The weight loss was accounted for in the weight gain exhibited by the filter assembly and the weight of the corrosion product mechanically removed from the sample for analysis. The weight of the filter increased from 17.89 grams to 18.55 grams, a net gain of 0.66 grams.

There was no appreciable weight change in the tin-lined sample.

12-Week Sample

There was a measurable change in the before and after weights of both the bare and tin-lined copper tubing samples. The bare copper tubing sample changed from 310.04 to 308.40 grams, a net reduction of 1.64 grams. However this value may be suspect due to the measurement being made with the couplings.

The filter on the tin-lined copper sample changed from 18.06 grams to 18.23 grams, a net gain of 0.17 grams. The pitting observed cannot be explained by the weight loss measurements. The pitting was isolated, small and would not account for the weight change measured.

4-Week Sample

There was no measurable weight change for either the bare copper tubing sample or the tin-lined copper tubing sample. The pre- and post-weights were 309.16 grams for the bare copper sample and 307.09 grams for the tin-lined sample. The scale used for measurement was limited to 2 decimal places.

3.3 Corrosion Deposit Composition Analysis

52-Week Sample

Elemental mapping of the deposit was determined by using a scanning electron microscope with energy dispersive x-ray spectroscopy (SEM/EDX). The analysis of corrosion deposits revealed that it contained predominantly copper (Cu) and sulfur (S), which most likely were in the form of copper sulfide. A spectrum showing the results is attached in **Appendix A**. Close-up views of the surface morphology revealed that corrosion products formed on the bare copper sample was friable with cracks that could easily break away from the tubing wall (**Figure 6**). **Figures 7 and 8** showed where the

protective tin coating exfoliated and allowed formation of similar corrosion byproduct deposits in those areas and on the bare copper tubing.

24-Week Sample

Corrosion products on the inside of the bare copper tubing sample were wiped with a nylon brush to remove deposits and measure the quantity. The corrosion deposits were weighed at 0.2432 grams. Wiping on the tin-lined tubing sample did not yield a measurable amount of deposit.

The elemental composition of the deposit was determined using SEM/EDX testing which revealed that the deposits were composed mainly of copper and sulfur with a trace of oxygen present.

12-Week Sample

Corrosion products on the interior surfaces of the sample were removed with a nylon brush to determine quantity. Wiping of both the bare copper and the tin-lined copper tubing samples did not yield a measurable amount. The elemental composition (determined by SEM/EDX) of the adherent corrosion products on the bare copper tubing wall surface contained copper (Cu), sulfur (S), and oxygen (O). In addition to the aforementioned elements, the deposits on the tin-lined copper tubing sample also contained tin (Sn).

4-Week Sample

Corrosion products on the interior of the samples were removed using a nylon brush to measure quantity. There was not a measurable amount of deposits removed by wiping in this manner although there were some scratches on the deposit layer. There was some resulting dust that was not heavy enough to land in the collection tray, but floated in the air.

The elemental composition using SEM/EDX was determined for the products removed from both the bare copper and tin-lined copper tubing samples. The analysis of both revealed that the deposits contained copper (Cu), sulfur (S), oxygen (O), and silicon (Si).

3.4 Natural Gas Composition

Total sulfur, H₂S, water, CO₂ and oxygen in natural gas are known to influence corrosion in copper lines. Their specific concentrations were monitored throughout the different test phases. In all, sampling for natural gas composition was conducted eight times to determine if the gas in contact with copper tubing exceeded So Cal Gas Company's gas quality specification. The natural gas was sampled and analyzed on October 10, November 7, December 7, 2000, January 5, February 5, July 18, August 28, and October 29, 2001. These results are summarized in **Table 1** and **Table 2** below. The variations in gas composition are deemed within expected ranges for the Southern California Gas Company service area.

Table 1. Sulfur and Water Content

Component	Oct 10	Nov 7	Dec 7	Jan 5	Feb 5	July 18	Aug 28	Oct 29	Maximum Allowed
Total Sulfur (ppm)	1.19	2.54	1.99	2.273	2.724	1.795	1.253	1.439	12.00
Hydrogen sulfide H₂S, (ppm)	0.060	0.100	0.100	0.034	0.060	0.090	0.044	0.035	4.00
Hydrogen sulfide H₂S, (grainsH₂S/100scf)	0.0038	0.0063	0.0063	0.0021	0.0038	0.0057	0.0028	0.0022	0.25
Water content (lbs/MMscf)	~ 6	5.1	3.4	5.3	13.0	7.9	32.0	7.4	7.0
Water content (ppmv)	~ 126	107	71	111	273	170	600	130	147

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Table 2. Hydrocarbon and BTU Content

Component	Oct 10	Nov 7	Dec 7	Jan 5	Feb 5	July 18	Aug 28	Oct 29
Higher Heating Value, HHV (BTU/ ft³)	1012.5	1018.2	1023.6	1033.2	969	1018	1017	1011
Lower Heating Value, LHV (BTU/ ft³)	912.3	917.3	922.1	929.9	873	918	917	911
Specific Gravity	0.5806	0.5853	0.5884	0.6035	0.5872	0.5816	0.5833	0.5790
Methane (mol%)	96.091	95.442	95.492	93.607	92.870	95.925	95.693	96.792
Ethane (mol%)	1.598	1.932	1.919	2.780	1.496	1.780	1.889	1.177
Propane (mol%)	0.230	0.252	0.293	0.040	0.321	0.291	0.281	0.180
iso-Butane (mol%)	0.029	0.033	0.040	0.046	0.038	0.036	0.034	0.030
n-Butane (mol%)	0.046	0.047	0.064	0.077	0.075	0.061	0.056	.0035
iso-Pentane (mol%)	0.014	0.012	0.017	0.018	0.020	0.019	0.018	0.012
n-Pentane (mol%)	0.014	0.039	0.049	0.045	0.026	0.017	0.016	0.009
C6 plus (mol%)	0.001	0.090	0.14	0.51	0.013	0.035	.0028	0.023
Carbon Dioxide (mol%)	1.027	0.898	1.151	0.972	1.315	0.795	0.967	1.316
Oxygen (mol%)	0.017	0.010	0.012	0.245	0.229	0.007	0.004	0.008
Nitrogen (mol%)	0.934	1.240	0.822	1.662	3.606	1.035	1.013	0.420

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4.0 CONCLUSIONS

4.1 “Useful Service Life” of Copper Tubing

The acceptable “useful service life” of the copper tubing is defined in **Section 2.5** as having a remaining wall thickness of greater than 80% of the nominal at the end of 20 years in service. For the standard ½-in. outside diameter, 0.05-in. thick wall copper tubing samples, the allowable wall loss is 10 mils. This is based on the assumption that corrosion wear was uniform throughout the samples. As such, the wall loss is calculated on the basis of tubing sample weight measurements before and after the specified exposure period (**Table 3**). Any localized corrosion resulting in pitting other than general surface wear would have to be assessed as to the resulting penetration into the tube wall. The following graph (**Figure A**) shows a plot of the amount of wall loss versus exposure time. The projected wall loss at 20 years of service life for bare copper tubing is well below the 10 mils limit. Based on a uniform wall loss assumption, the data suggest that bare copper tubing will maintain structural integrity for the duration of its “service life.”

However, examination of the interior surfaces of the bare copper tubing revealed that corrosion wall losses were not uniform. Several sites of localized pitting extending deeper into the tube wall were visible. The presence of these pits and their resultant effect on wall loss had to be accounted for to ensure that these localized areas of pitting would not jeopardize the tubing wall and result in wall thickness less than that specified in the performance criteria. Examination of these pitting sites indicated that while the pits were deeper than the surrounding general surface corrosion, they were still too shallow to be measured with available instruments. Thus they were not significant enough to exhibit a measurable difference in the expected service life and are not a cause for failure of the performance criteria. Also, the average wall loss after 20 years of projected usage is so small that the preferential corrosion sites would have had to exhibit an increased corrosion rate approximately 35 times higher than the average rate to actually develop a pinhole in the accepted service life. If these sites had been corroding at this extreme rate, then the visible pits would have exhibited a measurably greater depth, which is not corroborated by the observed results.

Tin-lined copper tubing did not exhibit susceptibility to sulfide corrosion while the tin coating was in tact. It was clearly more resistant to corrosion than bare copper tubing, although there were isolated corrosion pits formed on the surface but no indication of deep penetration due to corrosion. However, two areas of tin coating failure were observed in which the unprotected copper tubing reacted in the gas stream to form copper sulfide. Regardless, the results indicate that the tin-lined copper is well within the performance criteria and can be expected to have a useful life much longer than the requisite 20 years indicated in this test. Also, the results indicate that the tin-lined copper tubing should not exhibit any significant potential for blockage of orifices, valves, or burners due to the release of significant amounts of friable corrosion products.

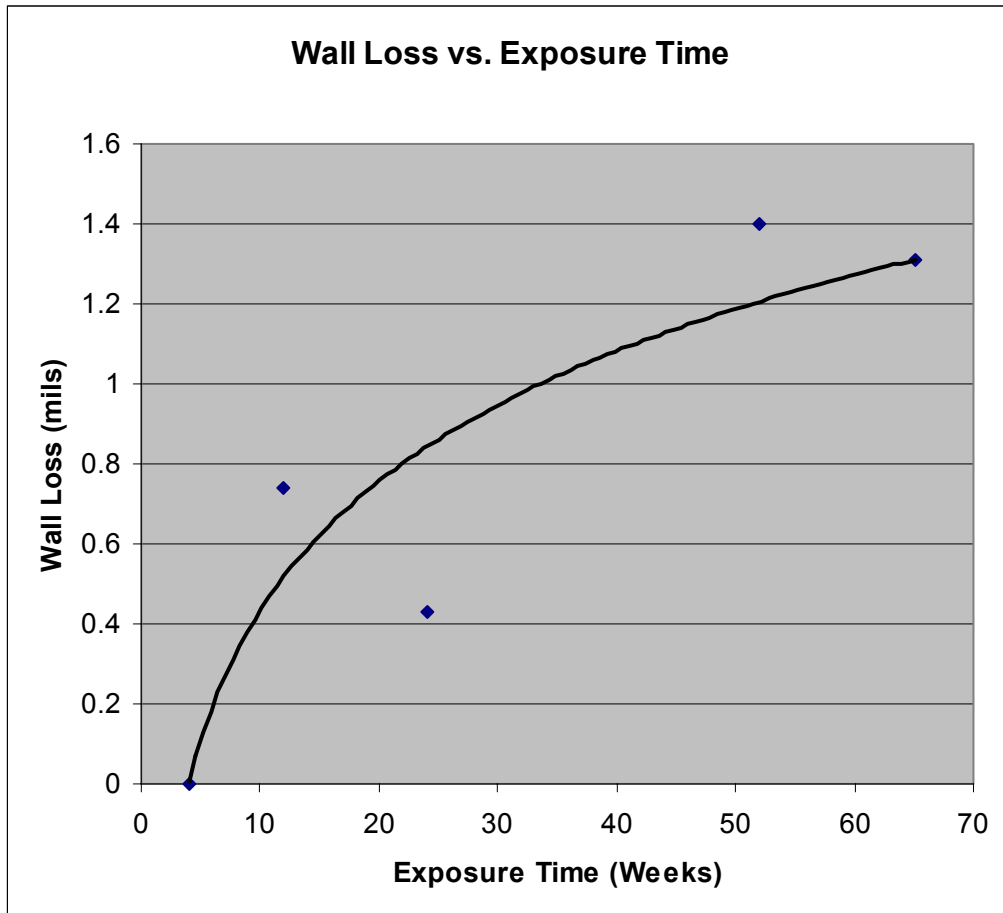
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Table 3. Weight and Wall Loss vs. Exposure Time

Exposure Time (Weeks /Years)	Bare Copper Tubing Weight w/Fittings (Before) (Grams)	Bare Copper Tubing Weight w/Fittings (After) (Grams)	Net Weight Loss ¹ (Grams)	Wall Loss R _o - R _i (mils)
4 / 1.23	309.16	309.16	0.00	0.00
12 / 3.68	310.04	308.40	1.64	0.74
24 / 7.36	309.25	308.31	0.94	0.43
52 / 15.96	308.39	305.33	3.06	1.4
65.16 / 20.0	n/a	n/a	n/a	1.31 ²

- (1) Assume uniform wear: net weight loss = $\pi\rho L (R_o^2 - R_i^2)$
Where ρ = density of copper L = length of copper tubing sample
- (2) Projected wall loss at 20 years of service life

Figure A. Copper Tube Wall Loss vs. Exposure Time



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4.2 Corrosion Products

It was clear that bare copper tubing was susceptible to corrosion when exposed to distribution gas in the Pico Rivera service area. Corrosion products formed on the surface were friable and easily broke away from tubing wall. The amount of corrosion products trapped in the filter downstream did not clog or restrict gas flow. Nonetheless, the quantity of corrosion products generated in the foot-long sample did not represent the actual amount that could form in a typical house line. With longer lengths of bare copper tubing, as would be installed in a typical system, it is likely that a greater quantity of corrosion products would form and be available for transport downstream through the piping system. Based on the scope and results of this testing, it is not possible to project the quantity of corrosion products and the potential to cause blockage at orifices in valves, regulators, etc. It is inconclusive that increases in the production of corrosion products due to longer lengths would actually lead to downstream blockage problems because the average size, consistency, and strength of these friable products were not determined. In most cases, the friable products were brittle and tended to break down into smaller pieces as it dislodged from the tubing wall.

In the case of tin-lined copper tubing, very minimal corrosion was observed, and in most cases the corrosion products generated were so minimal as to not be measured. This indicates that there is less likelihood that a system using tin-lined copper tubing will generate sufficient corrosion products to cause potential problems downstream.

4.3 Effects of Natural Gas Composition

Natural gas constituents identified as corrosive to copper are sulfur, H₂S, water, CO₂ and oxygen. In **Tables 1** and **2**, sampling results showed that most were within the recommended concentration limits specified in the Gas Company specifications except for the water moisture content sampled in February and August 2001, which exceeded the 7 lbs/MMscf limit. The source of higher water content was believed to coincide with storage withdrawal operations at Montebello, which could elevate moisture if the gas was not fully dehydrated during processing. In addition, the oxygen level in the gas increased significantly in the months of January and February. These could have affected the corrosion rate in the copper.

4.4 Overall Conclusion

Both the bare copper tubing and the tin-lined copper tubing evaluated in this test met the performance criteria to indicate that they will maintain structural integrity even though there were clear signs of sulfide corrosion. The extent of the observed corrosion was not severe enough to cause concern with through-the-wall type corrosion in the context of a 20-year useful life. However, a suggested area for further study is the evaluation of the copper sulfide corrosion products to determine if they are of sufficient size or consistency to present the possibility of creating blockage in system components.

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For purposes of accelerated testing, the gas flow for the test was set at a larger value than the assumed typical houseline gas flow such that 4 weeks of continuous flow at the testing conditions were equivalent to 1.23 years of usage at the houseline rates, and so forth. The higher than normal gas flow rate of these test conditions may not have reproduced the operating conditions that a typical gas system might be exposed to in an actual installation. While the samples were continuously exposed to the gas stream, the test conditions may have resulted in exposures that resulted in dwell times of the gas that are shorter than normal and gas flow velocities that are higher than normal. A recommended area for further study would be whether these factors have any effect on Cu_2S production and release.

APPENDIX A

Copper Tubing Evaluation in A Natural Gas Environment

**Project No. TS00-M036
Final Report, July 3, 2002**

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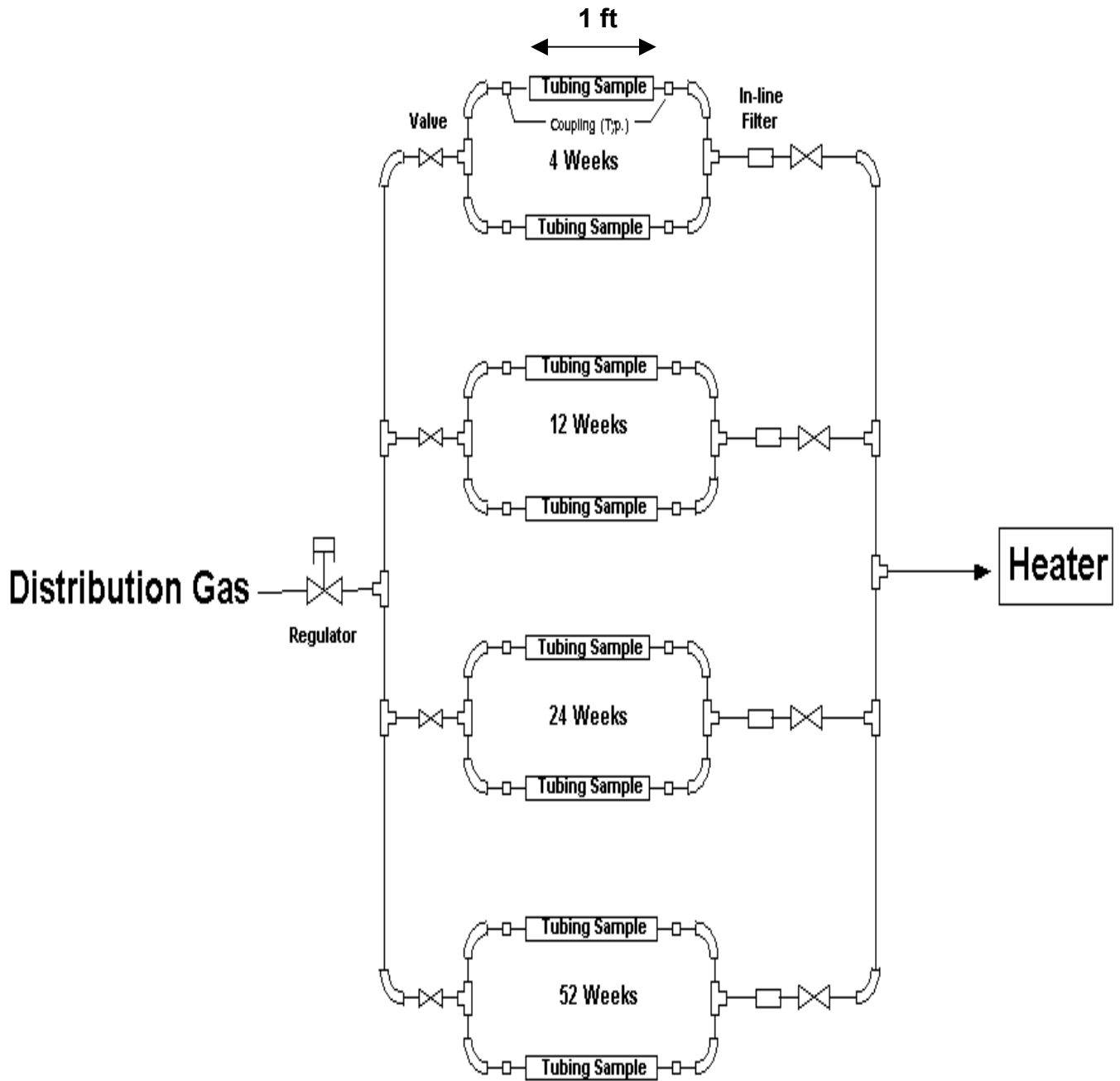


Figure 1. Test-rack assembly.

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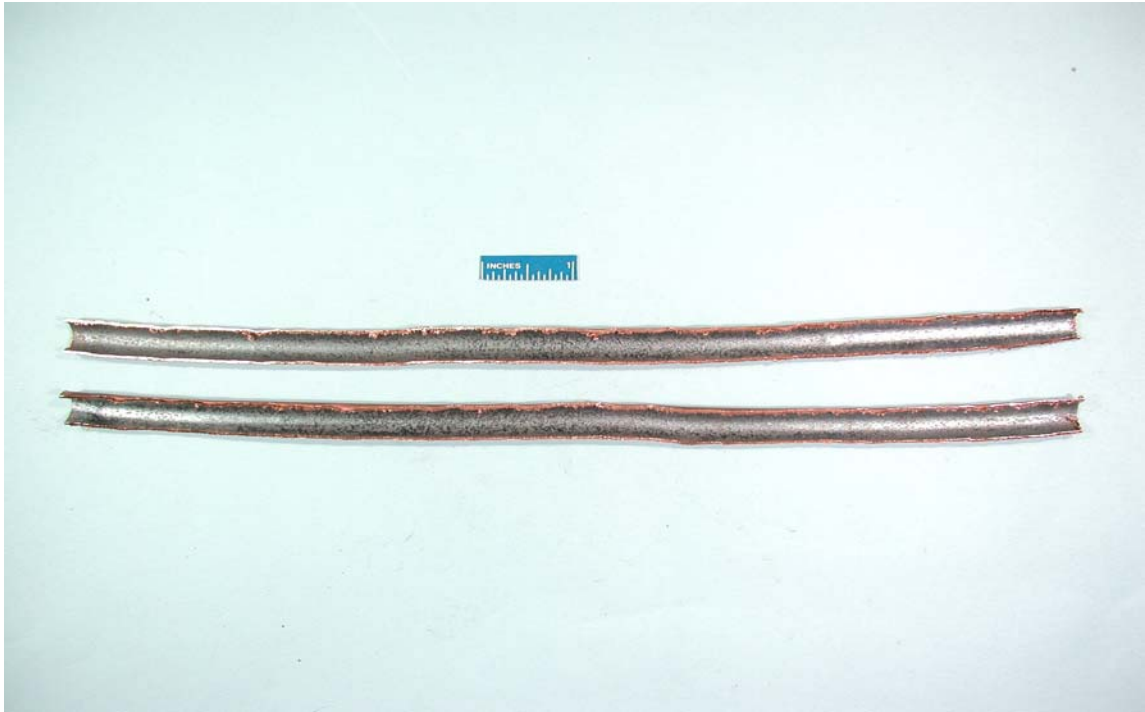


Figure 2. Bare copper sample split in half after 52-weeks' exposure.



Figure 3. Close-up view of corrosion deposit on the inside of the bare copper sample after 52- weeks' exposure.

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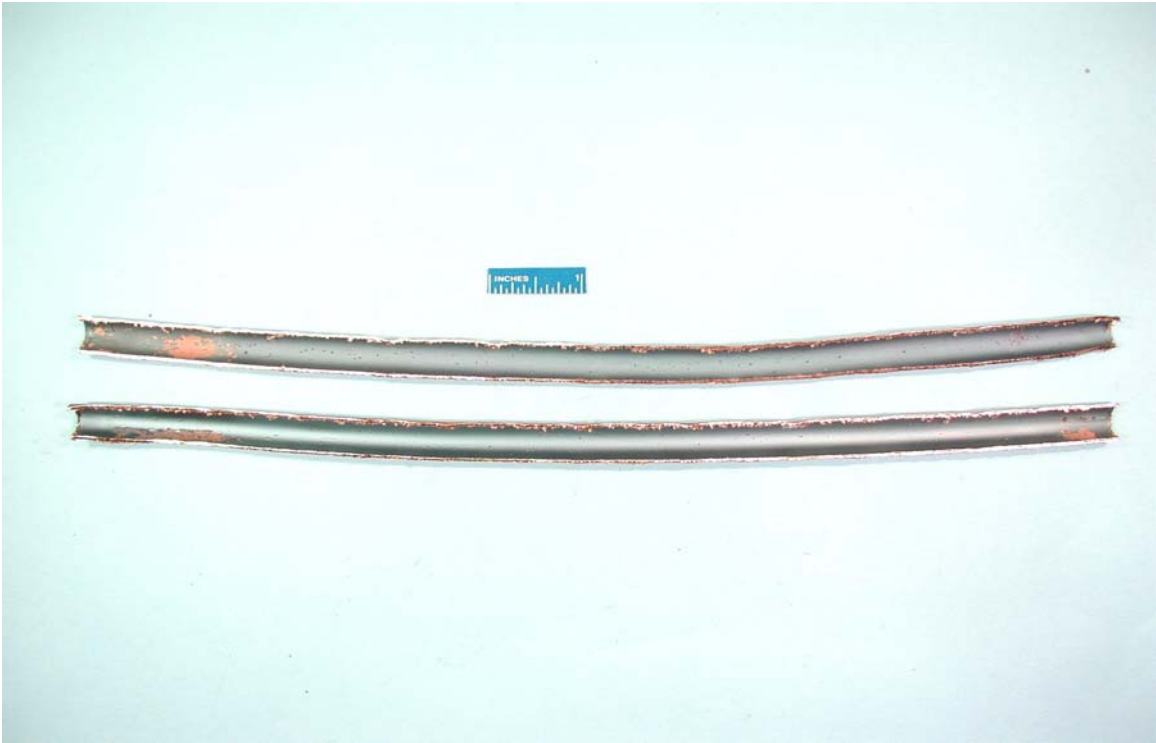


Figure 4. Tin-lined copper sample split in half after 52-weeks' exposure.

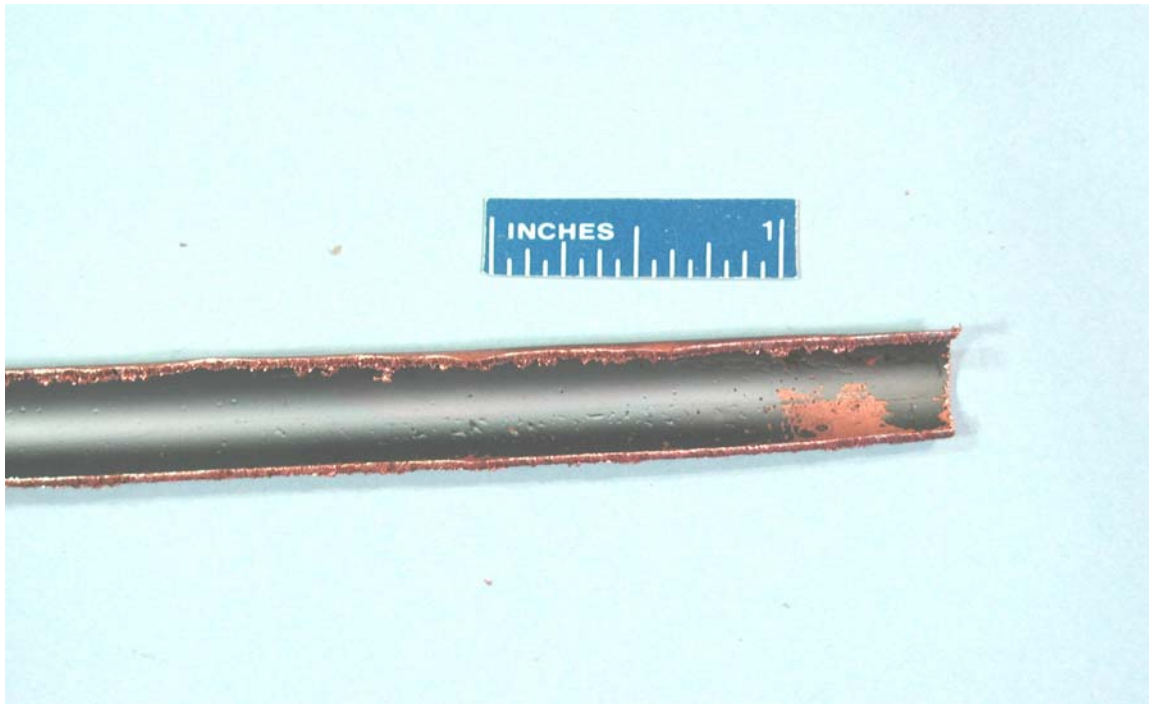
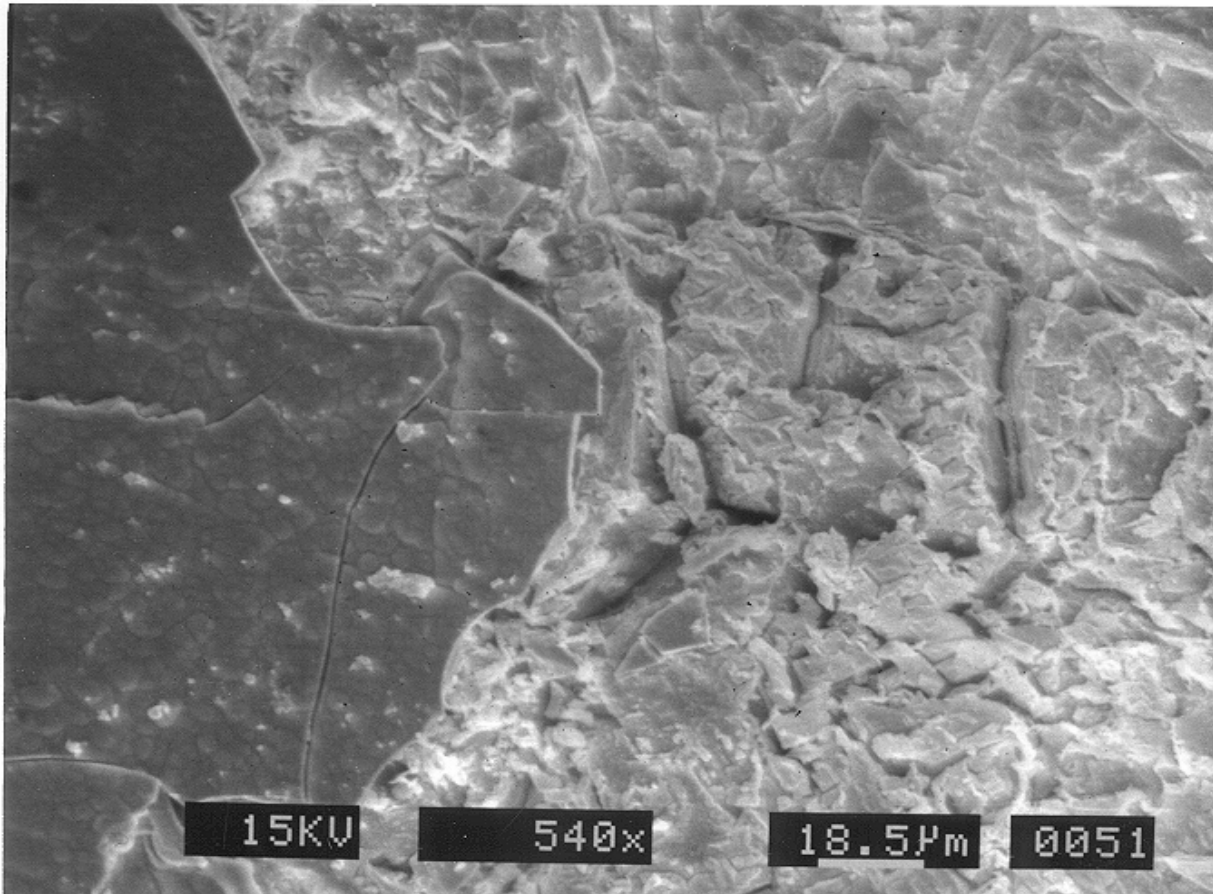


Figure 5. Close-up view of the isolated pits and an area of failed tin coating.

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**Figure 6. Close-up view of the surface morphology on the bare copper tubing.
Note the corrosion products on the left were brittle with fractured lines.**

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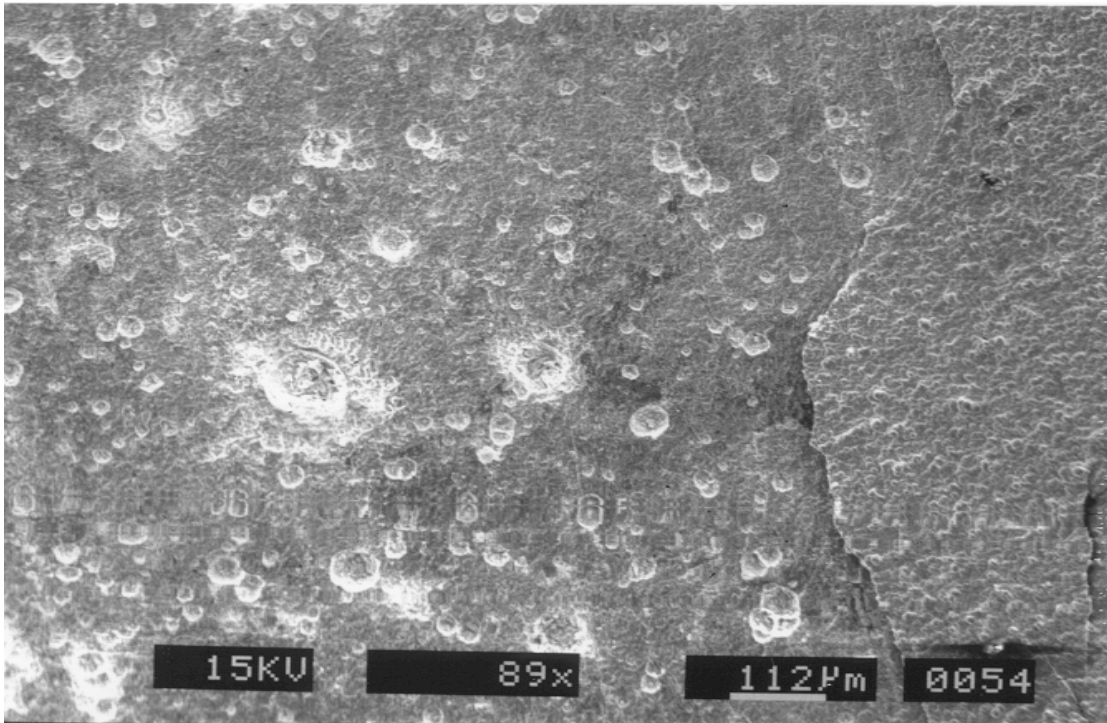


Figure 7. Surface morphology of the tin-lined copper tubing.

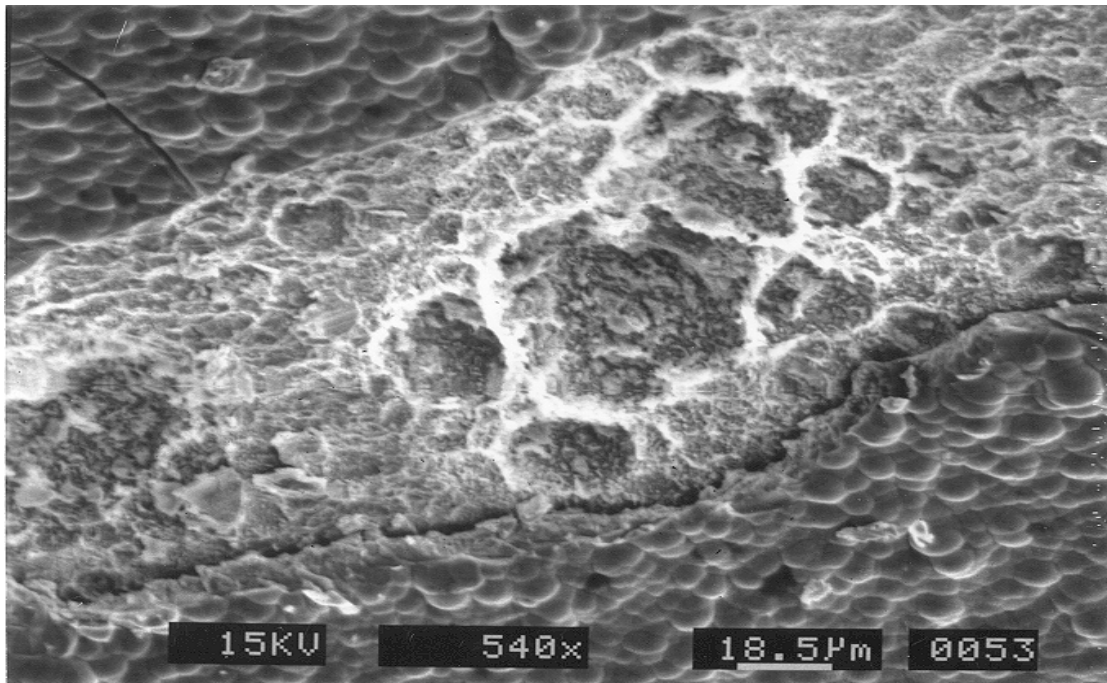
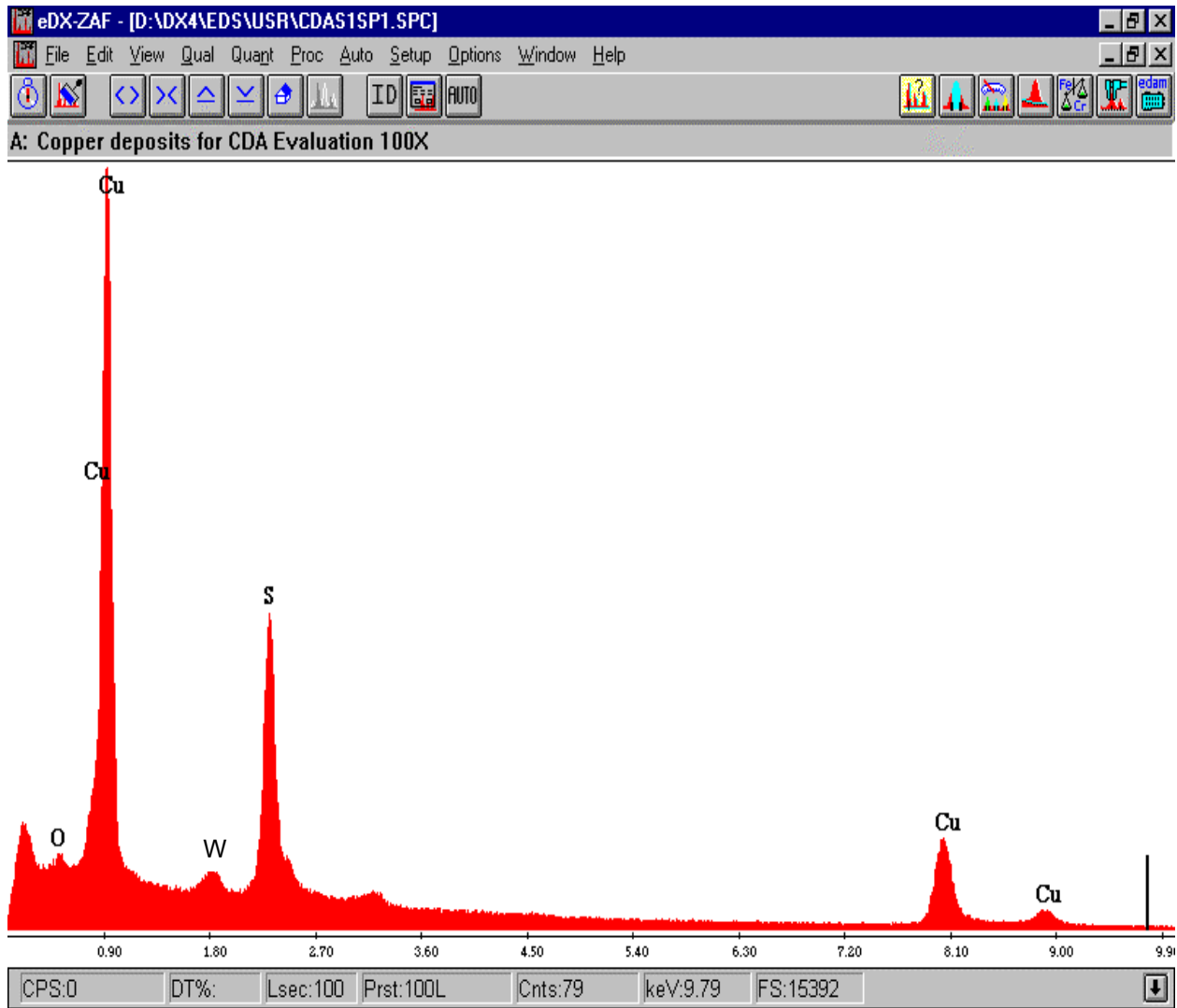


Figure 8. Close-up of an area where tin coating failed at the surface exposing copper tubing to a flowing gas stream.

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CDA- EDX ANALYSIS

D:\DX4\EDS\CDAS1SP1.SPC Label: Copper deposits for CDA Evaluation 100X

Acquisition Time: 14:19:04 Date: 15-Nov-2000

EDAX ZAF Quantification (Standardless)

Element Normalized

SEC Table: Default

Elem	Wt %	At %	K-Ratio	Z	A	F
W M	9.99	2.63	0.0581	0.8301	0.6999	1.0010
S K	38.73	58.38	0.2174	1.1024	0.5090	1.0000
Cu K	51.28	39.00	0.4803	0.9539	0.9820	1.0000
Total	100.00	100.00				

kV: 15.00 Tilt: 0.00 Take-off: 20.00 Tc: 40

Det Type:SUTW, Sapphire Res: 134.47 Lsec: 100

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