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EVALUATION OF HASTELLOY N TUBES FOR SNAP-8 APPLICATIONS
(Title Unclassified)

H. E. McCoy and J. R. Weir

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METALS AND CERAMICS DIVISION

EVALUATION OF HASTELLOY N TUBES FOR SNAP-8 APPLICATIONS

H. E. McCoy and J. R. Weir

APRIL 1967

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EVALUATION OF HASTELLOY N TUBES FOR SNAP-8 APPLICATIONS

H. E. McCoy J. R. Weir

ABSTRACT

The results of a brief study on Hastelloy N cladding material for SNAP-8 application are described. The alignment problem encountered in running axial tensile tests on tubes has been studied. A gripping technique was developed that reduced the bending stress to about 5% of the axial stress. Tensile tests on circular tube segments have been run, and the results compare favorably with those obtained in axial tube tests at 650°C and higher. Variations in the grain size of the chromized tubes and those tubes exposed to a thermal cycle duplicating that of the chromizing process have not been fully explained. However, the effect is thought to be largely associated with nonuniform working of the tubing during the final fabrication steps. Electron microprobe studies showed that chromium diffused into the Hastelloy N. The possibility of the chromium diffusing along grain boundaries to appreciable depths during service is pointed out.

INTRODUCTION

The purpose of this report is to relate the findings of a brief study directed toward an evaluation of the SNAP-8 cladding material. The material involved was Hastelloy N (Heat 281-4-0143), which was provided by Atomics International in the form of tubing. This study included the following: (1) developing a well-aligned gripping arrangement for testing tube specimens under an axial tensile stress; (2) evaluating a technique for testing rings of the same tubing with the stress being applied in the circumferential direction; (3) a microprobe study of the chromium- and silicon-rich layers put on the tubing during processing; (4) a metallographic study of the tubing before and after testing; (5) a brief investigation aimed at resolving the grain-size variations noted by ORNL and AI after the tubing had been given an identical heat treatment.

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DESCRIPTION OF TEST MATERIALS

The material used in this study was provided by AI in the form of tubing 0.540-ID × 0.0105-in. wall. The material was vacuum melted by Allvac Metals Company and was processed into tubing by Superior Tubing. The tubing was certified to be in the annealed condition, but the annealing temperature was not specified. The chemical analysis of the material is given in Table 1.

Table 1. Chemical Analysis of Test Material — Heat 281-4-0143

Element	Contents, wt %			
	As Received		ORNL	
	AI	ORNL	Chromized[a]	Coated[a]
Mo	16.88	16.9	15.7	15.3
Cr	7.0	6.9	12.4	11.1
Fe	0.28	0.58	0.52	0.47
C	0.05	0.033	0.054	0.081
Mn	0.50			
Si	0.28	0.30	0.24	0.98
B	0.0006			
S	0.009			
P	0.002			
Cu	0.03			
Co	0.01			
Al	0.20			
Ti	0.04			
W	0.01			
O	0.0006			
N	0.0025			
H	0.0004			

[a]Average values for a cross section of the tube.

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The tubing was also evaluated in the as-chromized condition. This treatment coated both the inside and outside surfaces with a thin layer of chromium. This process exposed the material 2 hr at a temperature of 1093 ± 14°C.

A third condition of the test material was the coated state. Preconditioning before coating involved chromizing, straightening (approx 2.6% strain), sandblasting, and resizing (approx 1.9% strain). The siliceous coating was applied through two cycles of 5 min each at 1150 ± 14°C.

RESULTS AND DISCUSSION

Tensile Tests on Tubing

An experimental problem in running uniaxial tensile tests is that of having the specimen properly aligned. If the force is not being applied along a line that coincides with the axis of the specimen, the test piece is subjected to a bending stress. If the material has low ductility, the strain from the bending stress may be sufficient to cause fracture before the test piece exhibits much overall strain. If the material is quite ductile, the test piece will deform so that its axis coincides with that of the applied stress and the initial misalignment does not pose a serious problem.

Hastelloy N has moderate ductility in the test range of interest (650 to 760°C); but with the specimen being a thin-walled tube (0.540-in. ID × 0.010-in. wall), it was anticipated that alignment would be quite critical. Hence, the first part of this study involved working out a gripping arrangement with reasonably good alignment. An Instron test machine was used and the load train is shown in Fig. 1. The most satisfactory method of gripping the specimen is shown in Fig. 2. We tried to use a 9/16-in. Swagelok, but found that this fitting did not have proper tolerances for this purpose. The male part of the fitting was machined on the end of the pull rod. The standard Swagelok nut and ferrules were used to hold the tube in place. The parts were put in place and hand-tightened. The assembly was laid in a piece of

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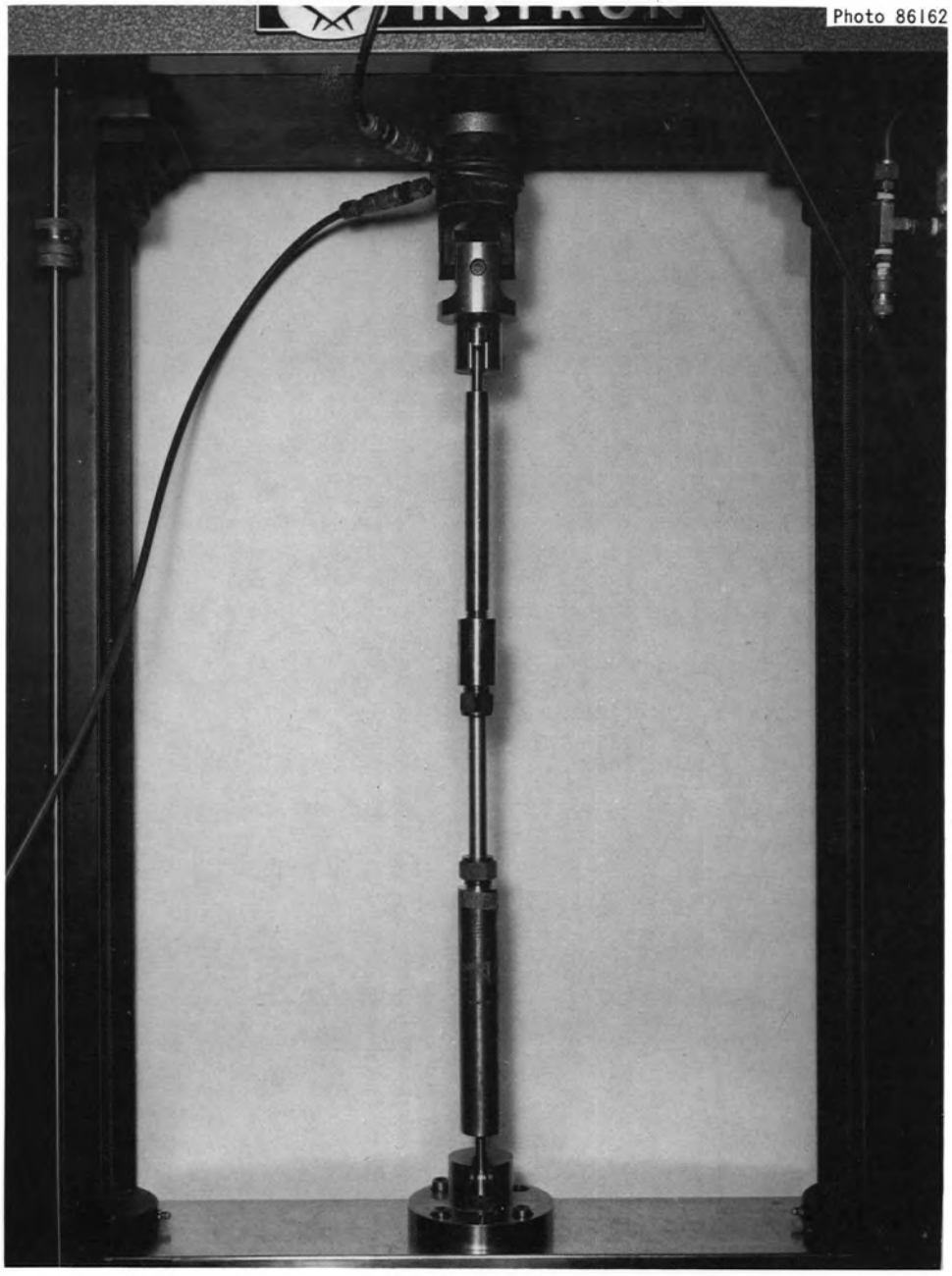

Fig. 1. Tensile Machine with Tube in Place for Testing.

Fig. 2. Photograph Showing the Method for Gripping the Tube Specimen. Fitting on the right is pulled back to show the components.

angle iron and each end of the pull rod was secured to the angle with a C-clamp. The nuts were then tightened.

Four strain gauges, spaced on 90° intervals, were placed on the center section of the specimen. The specimen was stressed in the elastic range, and the strains indicated by the four gauges were compared. By the gripping technique just described, the strains at 60,000 psi varied by 1×10^{-4}, or less. Assuming a modulus of 27×10^{6} psi, this corresponds to a maximum bending stress of 2700 psi at ambient temperature.

The specimen was heated by the small furnace shown in Fig. 3. The furnace had three zones with each controlled individually. A typical set of temperature readings at the beginning of a test is indicated. After-test measurements indicated that the effective gauge length of the tube was about 2 in. All tests were run at a crosshead speed of 0.002 in./min, so the strain rate was 0.001 min^{-1}.

The results obtained on the material at various stages of processing are given in Table 2. The uniform and total strains were taken from the Instron chart of crosshead travel versus load. The total strain was also determined by measuring the distance between gauge marks on the specimen after testing. The circumferential strain (ϵ_c) was determined by measuring the diameter near the fracture before and after testing. Where some degree of ovality was observed, maximum and minimum strain values are reported. If the material is isotropic and all of the strain is uniform, the axial strain measurements should be double the circumferential strain measurements. Within the accuracy of the data, this relationship seems to apply. Diameter measurements were made along

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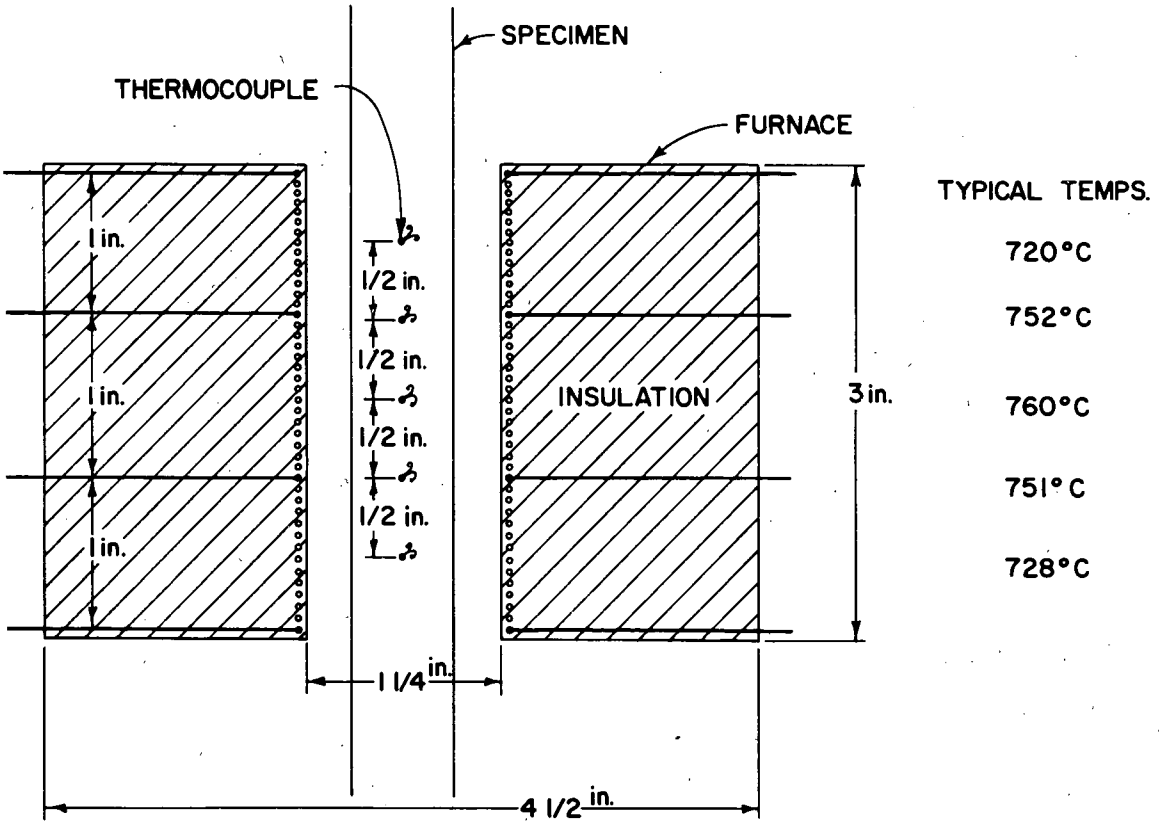

Fig. 3. Schematic Drawing of Furnace and Tube Configuration Used for Running Axial Tensile Tests on Tubes.

each tube after fracture. These diameter profiles are shown in Figs. 4 through 10. The stress-strain curves were noted to be serrated for the material in the chromized and coated conditions and almost smooth for the as-received material.

The ductility values obtained in these tests are compared with those reported by AI in Fig. 11. The ductilities obtained by both investigators in the as-received condition are in good agreement. However, our results indicate lower ductilities after both processing steps than those obtained by AI.

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Table 2. Tensile Data at 760°C[a]

Specimen Number	Condition	Stress, psi		Strain, %			Strain, %		Comments
		Yield	Ultimate	Axial[b]	Circumferential		Uniform[c]	Total[c]	
					Maximum	Minimum			
3	As received	41,000	41,300	17.2	13.3	12.1	17.0	17.5	
6	As received	40,800	41,000	21.7		12.5(av)	20.0	25.0	Tube tore badly.
1	Chromized	37,500	45,100	8.5	4.7	3.2	6.1	9.3	
7	Chromized	37,000	40,600	20.0	9.8	8.9	19.6	21.1	
5	Chromized	36,000	39,700	22.1	8.7	7.7	23.1	24.8	Temperature dropped, test restarted.
2	Coated		38,800	7.4	2.2	2.0	4.6	6.0	
4	Coated	35,500	39,800	13.7	4.6	3.9	13.5	13.8	

[a]Strain rate, 0.001 min^{-1}

[b]Measured from the test specimen after fracture.

[c]Taken from Instron chart based on a 2-in. gauge length.

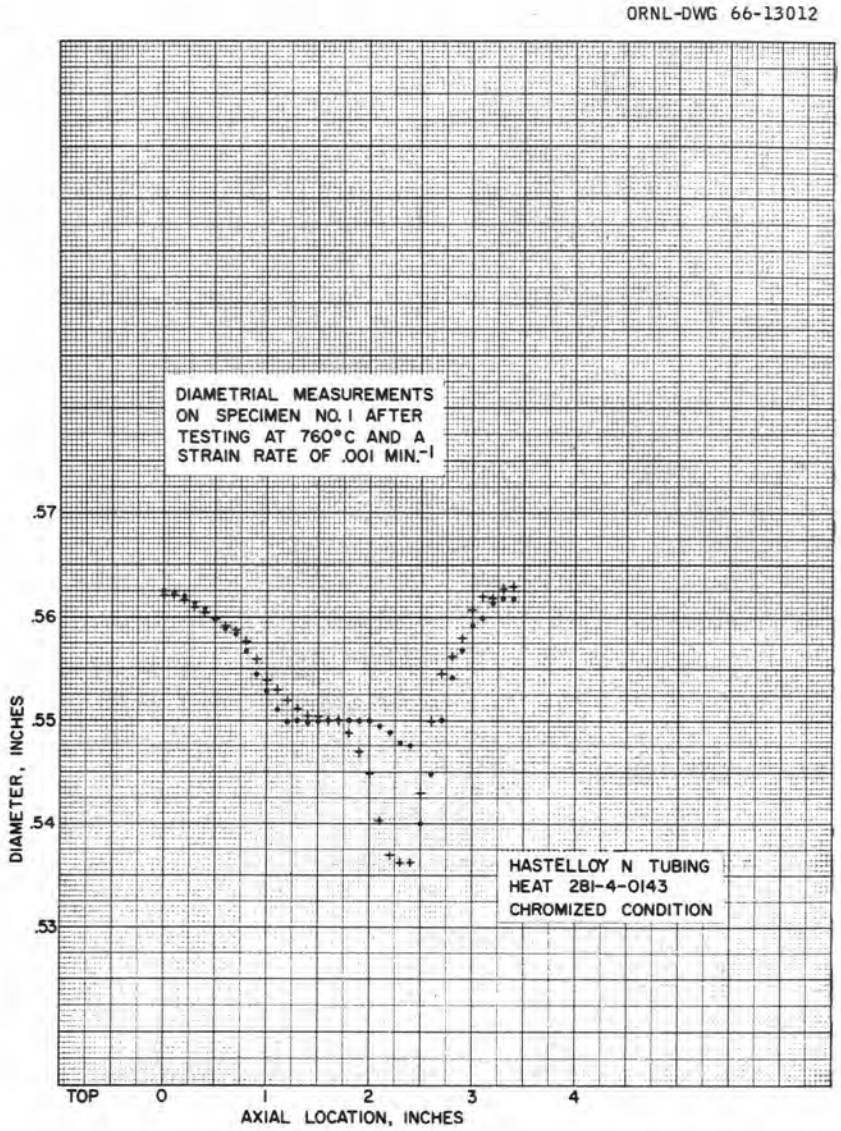


Fig. 4. Test Specimen and Diameter Measurements on Specimen 1.

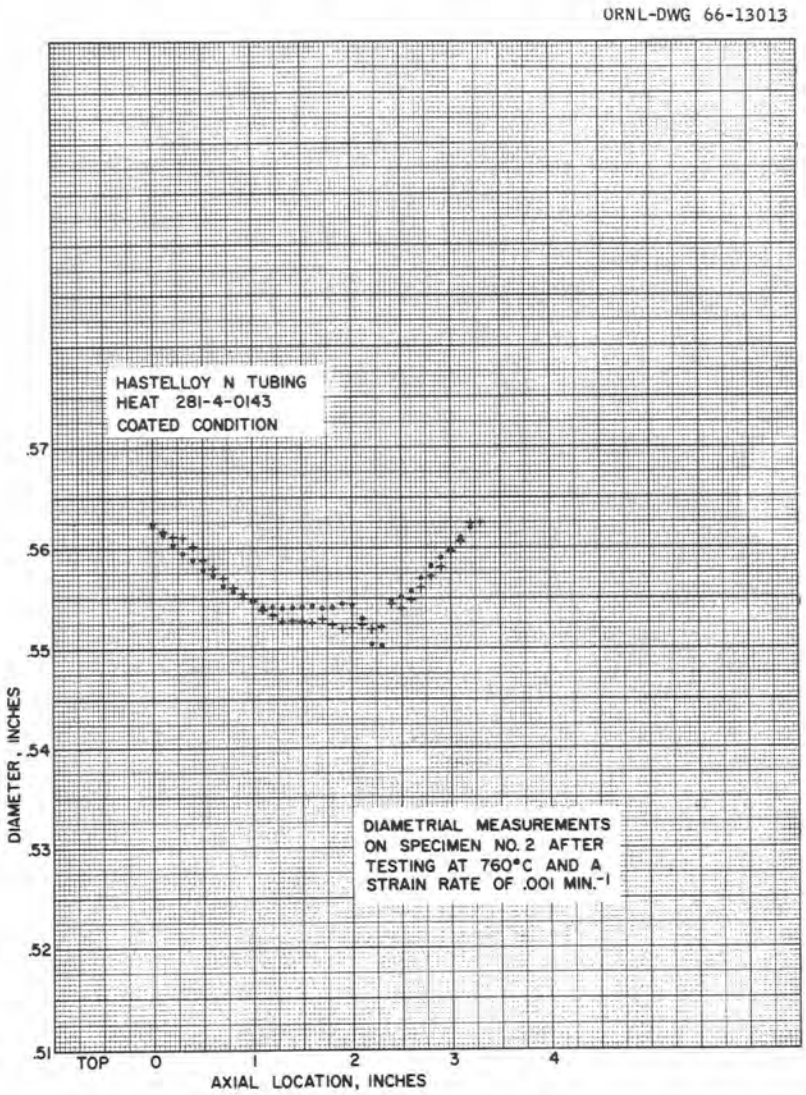
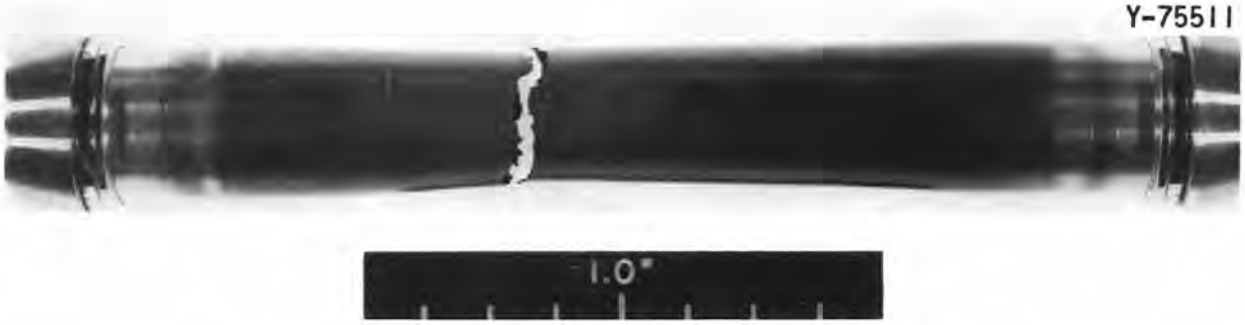


Fig. 5. Test Specimen and Diameter Measurements on Specimen 2.



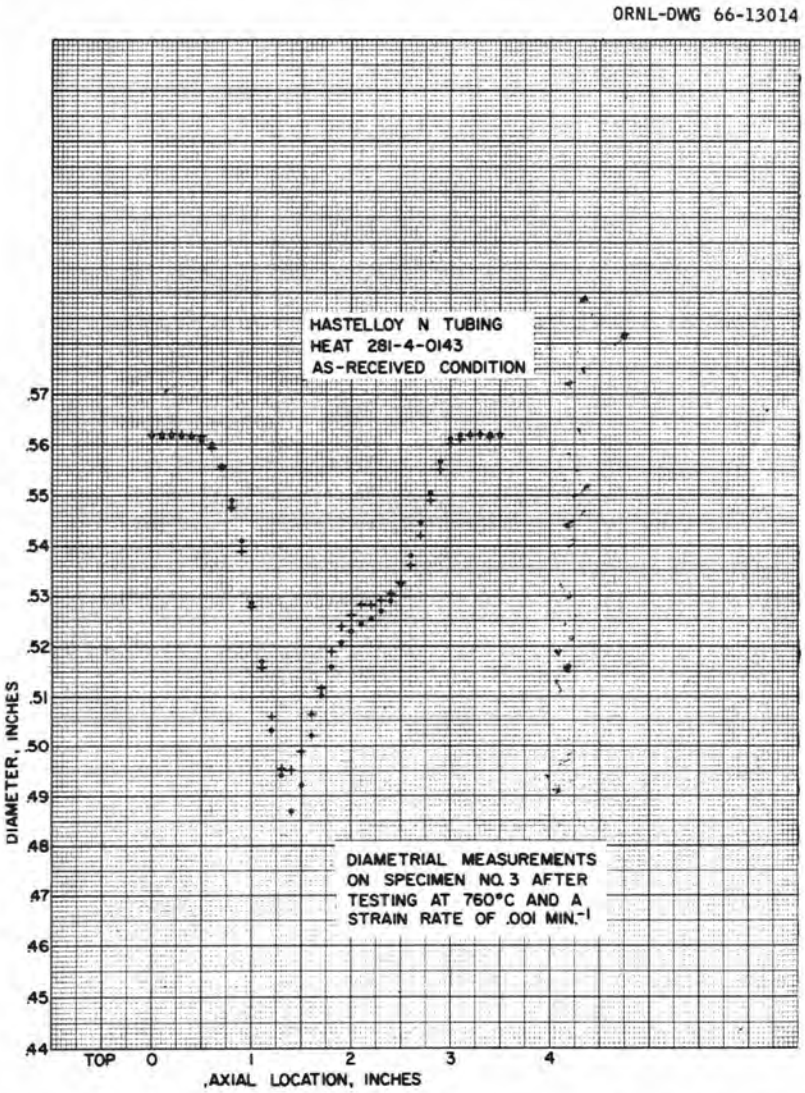
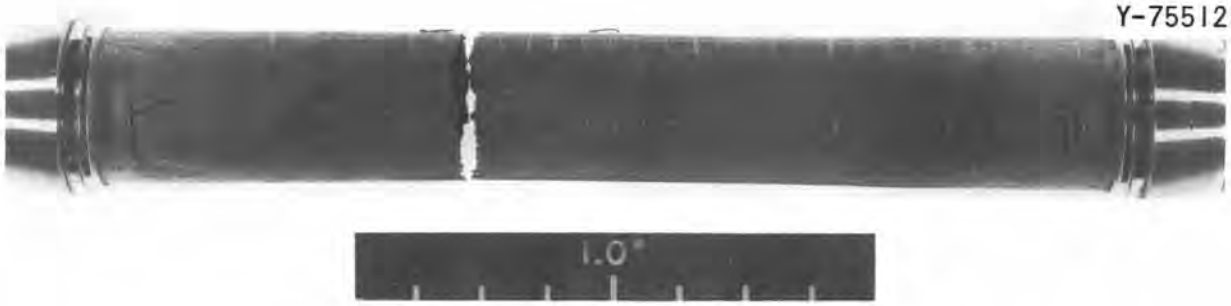


Fig. 6. Test Specimen and Diameter Measurements on Specimen 3.



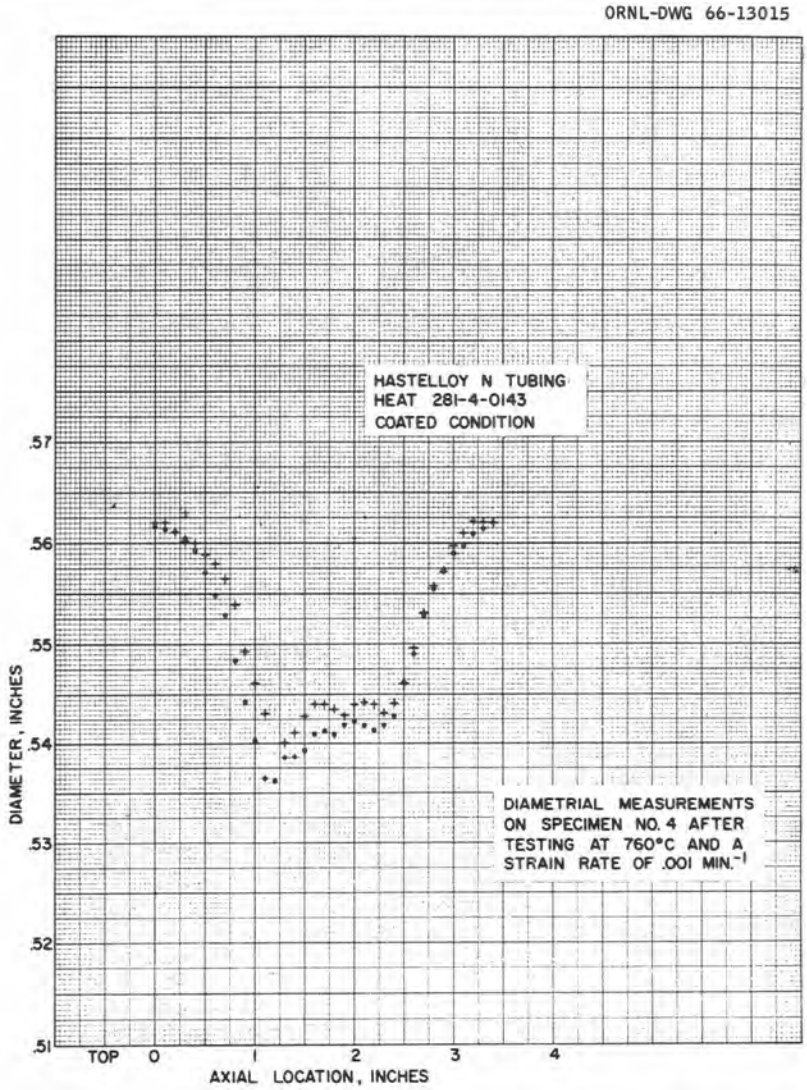


Fig. 7. Test Specimen and Diameter Measurements on Specimen 4.

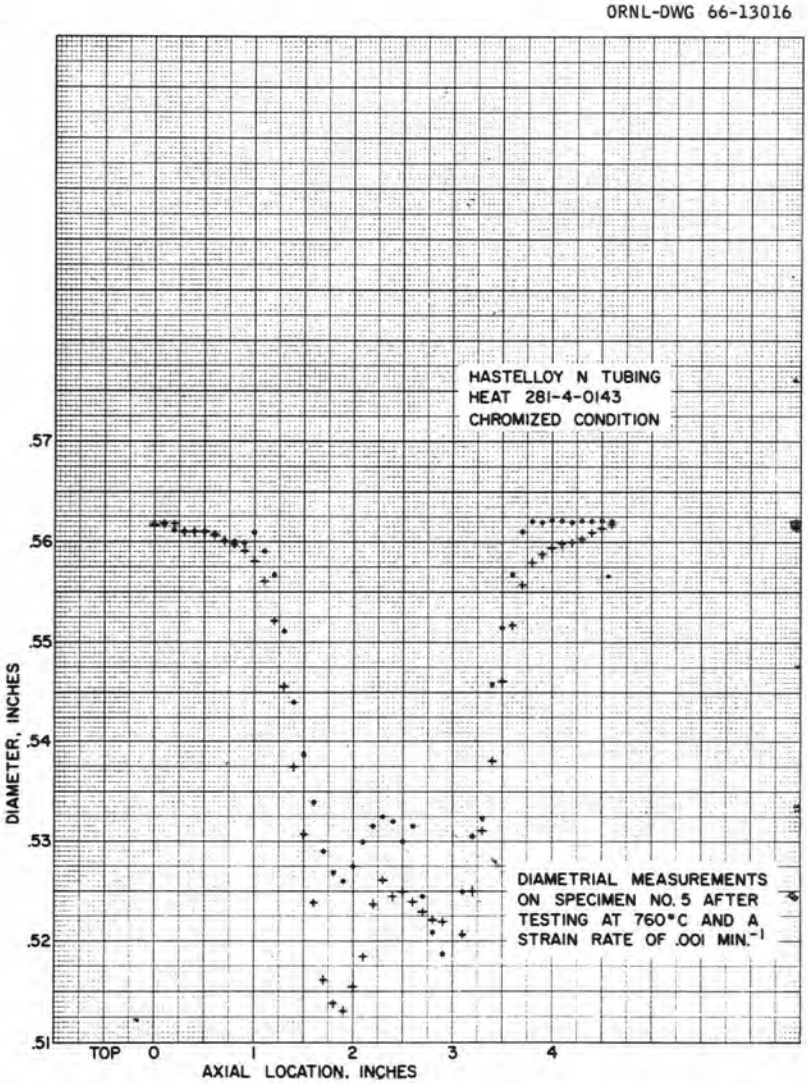
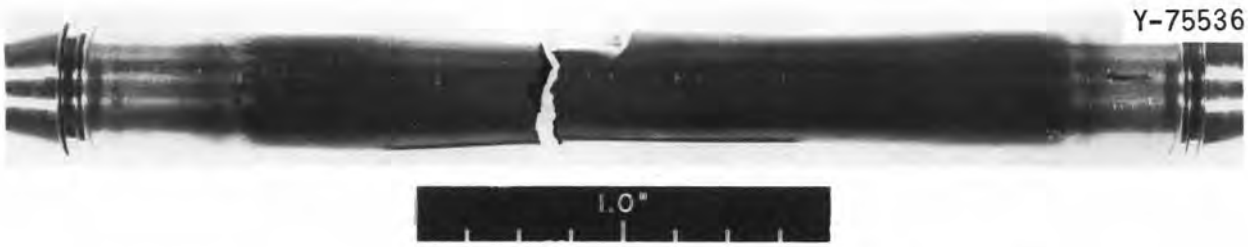


Fig. 8. Test Specimen and Diameter Measurements on Specimen 5.



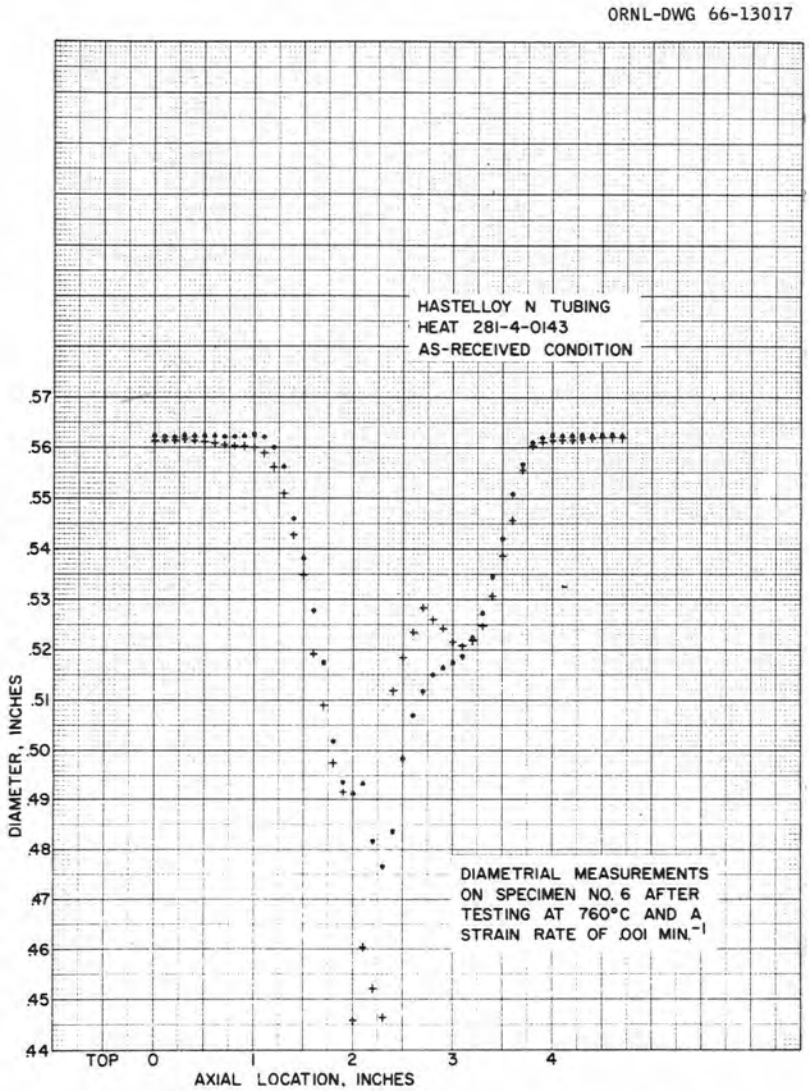


Fig. 9. Test Specimen and Diameter Measurements on Specimen 6.

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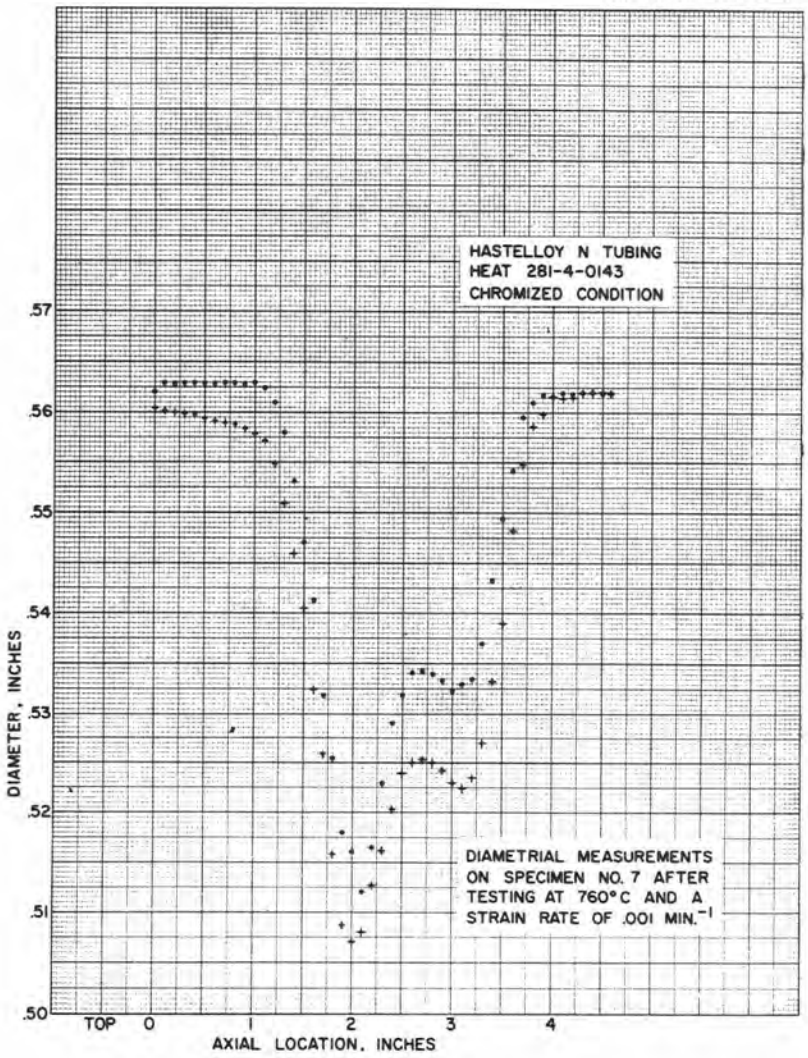

Fig. 10. Test Specimen and Diameter Measurements on Specimen 7.

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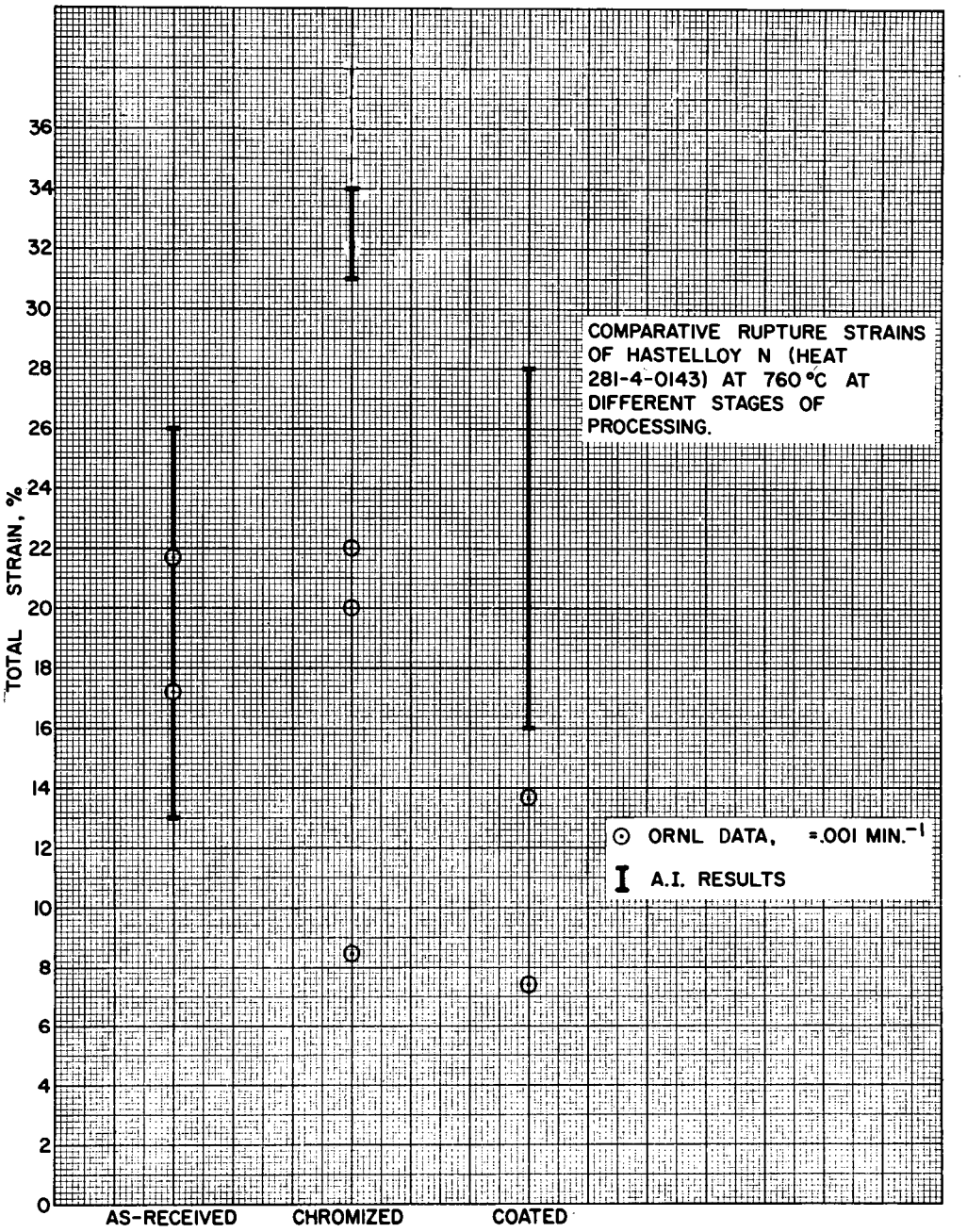

Fig. 11. Comparative Rupture Strains of Hastelloy N at Various Stages of Processing.

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Tensile Tests on Tube Rings

A test was investigated for testing rings cut from the tubing so that the tensile stress was applied in the circumferential direction. The test fixture used in this test is shown in Fig. 12. Since a gauge length is not defined in this type of specimen, one can only obtain the fracture strain and the ultimate tensile stress. The fracture strain is determined from the reduction in the width of the ring. Since this strain is 90° from the maximum principal strain, the assumptions of an isotropic material and uniform straining are used to predict the magnitude of the principal strain to be twice the percent reduction in width. If considerable necking occurs at the fracture, the predicted maximum principal strain will be too high.

The results of tests on material in the as-received and chromized conditions are shown in Table 3. The strains observed below 650°C are lower than would be expected. At 650°C and above, the calculated strains are only slightly lower than those measured by axial tests on the tubes (Table 2). For example, chromized rings at 760°C (crosshead speed equals 0.05 in./min) have a calculated circumferential strain of 14% whereas the axial strain for tubes varies from 8.5 to 22% ($\dot{\epsilon}$ = 0.001 min^{-1}).

The ultimate tensile strength obtained by the ring test agrees reasonably well with data available on a heat of wrought Hastelloy N (5065). This is demonstrated by Fig. 13. The lower strength of the chromized material is probably due to the larger grain size.

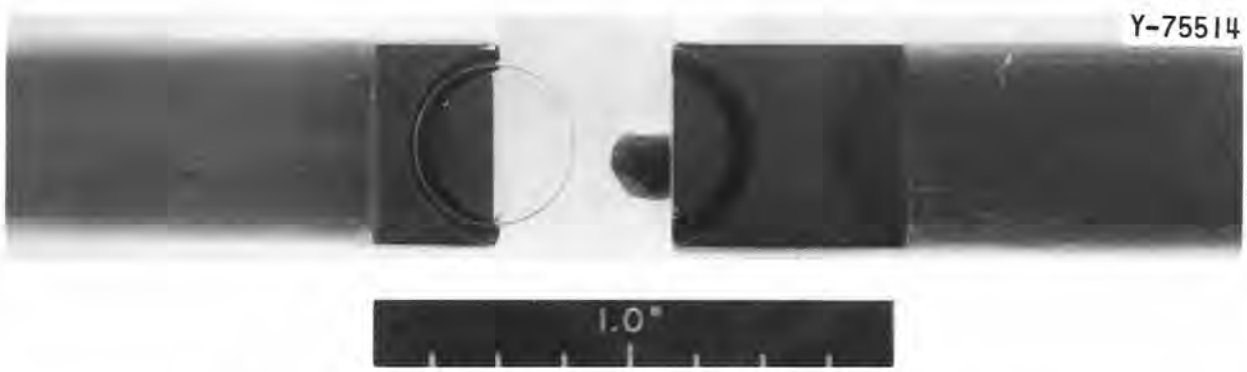

Fig. 12. Fixture for Testing Ring Specimens.

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Table 3. Summary of Tests on Hastelloy N Rings[a]

Test Temperature (°C)	Ultimate Stress (psi)	Strain in Width (%)	Crosshead Speed (min^{-1})	Calculated Circumferential Strain (%)
		As-Received Tubing		
25	132,700	16.0	0.05	32.0
400	107,600	15.1	0.05	31.2
650	66,800	4.0	0.05	8.0
650	31,700	2.0	0.002	4.0
760	58,500	3.6	0.05	7.2
850	39,800	15.3	0.05	30.6
		Chromized		
25	101,500	14.2	0.05	28.2
400	83,400	11.5	0.05	23.0
650	71,100	5.0	0.05	10.0
650	41,100	4.0	0.002	8.0
760	56,700	7.0	0.05	14.0
850	43,100	14.0	0.05	28.0

[a]Nominal tube dimensions: 0.540-in. ID × 0.010-in. wall × 0.250-in. width, Heat 281-4-0143.

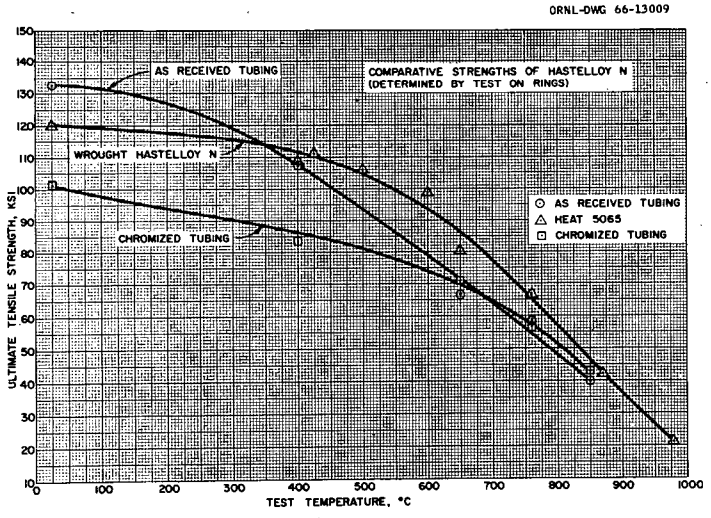

Fig. 13. Comparative Tensile Strengths of Hastelloy N Determined by Tests on Rings and on Standard Tensile Bars.

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The possible effects that the width of the specimen might have on the properties of the rings were investigated briefly at 650°C. The results of these tests are shown in Fig. 14. There does not seem to be any appreciable effect of width over the range of 0.074 to 0.253 in. Since duplicate tests were run at several conditions, these results give some indication of the reproducibility of the test.

Metallographic Studies

The tubing was examined metallographically in several conditions. The tubing in the as-received condition is shown in Fig. 15. The grain size is quite small (average grain diameter = 0.02 mm) and the

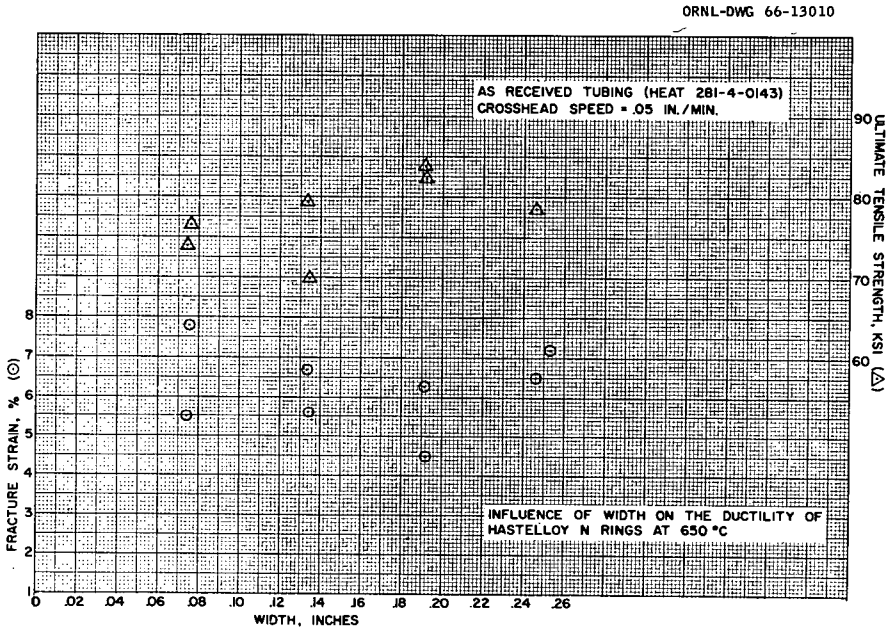

Fig. 14. Influence of Width on the Tensile Properties of Hastelloy N Rings at 650°C.

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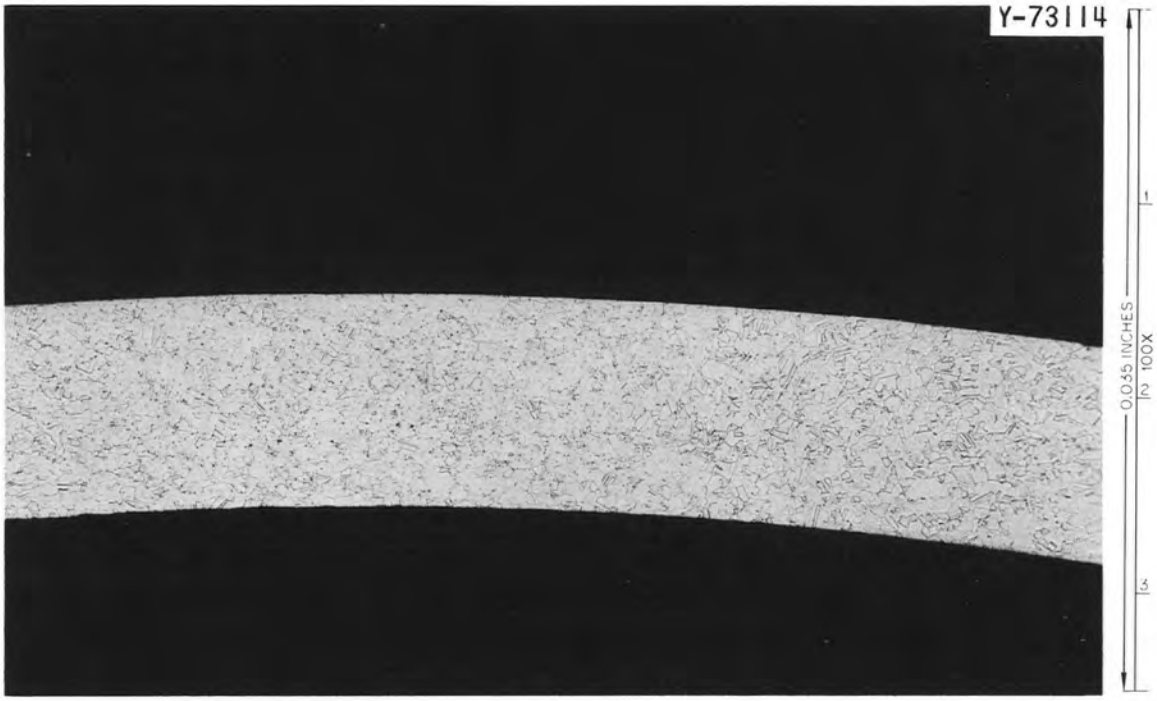

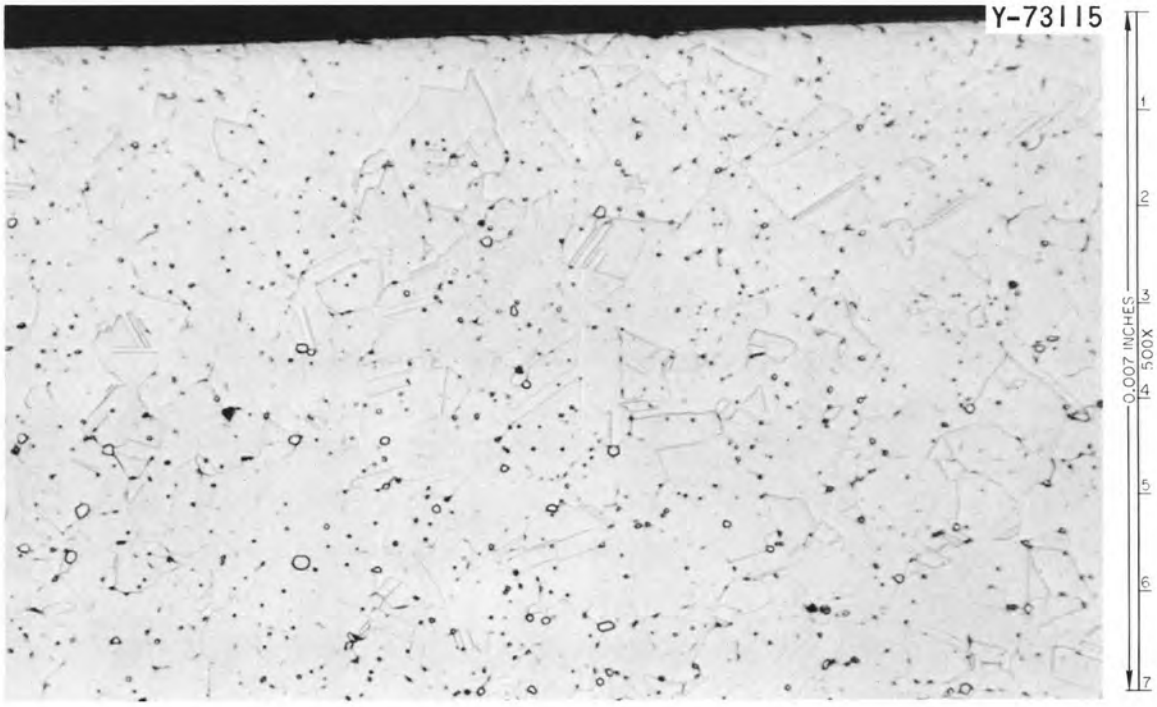

Fig. 15. Microstructure of the As-Received Tubing. Etchant: glyceria regia.

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precipitates present are typical of those normally present in Hastelloy N. The extensive working of the material into tubing has broken up the precipitate stringers (would be evident as patches in this cross-sectional view), so that the precipitates are randomly located. Some question arose concerning the use of etchants other than glyceria regia. The specimen shown in Fig. 15 was etched with glyceria regia. Figures 16 and 17 show the same specimen etched with 10% oxalic acid and Marble's Reagent, respectively. The oxalic acid attacks the precipitate very heavily and gives very good grain boundary delineation. However, the basic microstructure revealed by each etchant is identical. The material is shown in the chromized condition in Fig. 18. The chromized layer is apparent with a total thickness ranging from 0.010 to 0.016 mm. The reaction layer between the chromium and the Hastelloy N is about 0.004 mm thick. Extensive grain growth has occurred and the average grain diameter is now about 0.1 mm. Several of the larger precipitates have gone into solution. Figure 19 shows the material after coating. The coating is 0.46 mm thick, and the microstructure of the Hastelloy N appeared unchanged by the additional processing.

We were quite concerned over the large amount of grain growth that occurred during the chromizing treatment. This treatment was supposed to have exposed the material to a temperature of 1093 ± 14°C for 2 hr. The resulting grain size was much larger than we would anticipate for this treatment. Rings were cut from the piece of as-received tubing and were annealed by various people at ORNL. Metallographic examination showed very large variations in grain size, even in different tubes annealed together. The smallest grain size observed is shown in Fig. 20, and the largest is shown in Fig. 21. However, Fig. 22 shows one of many typical areas in which the grain size varied considerably. Hence, we feel that nonuniform working of the tubing during some of the final stages of processing is responsible for the large grain size variation. This may be a final sizing or straightening step, and the problem could be minimized by setting the processing steps so that the tubing is cold worked (> 10%) before these final strains are introduced.

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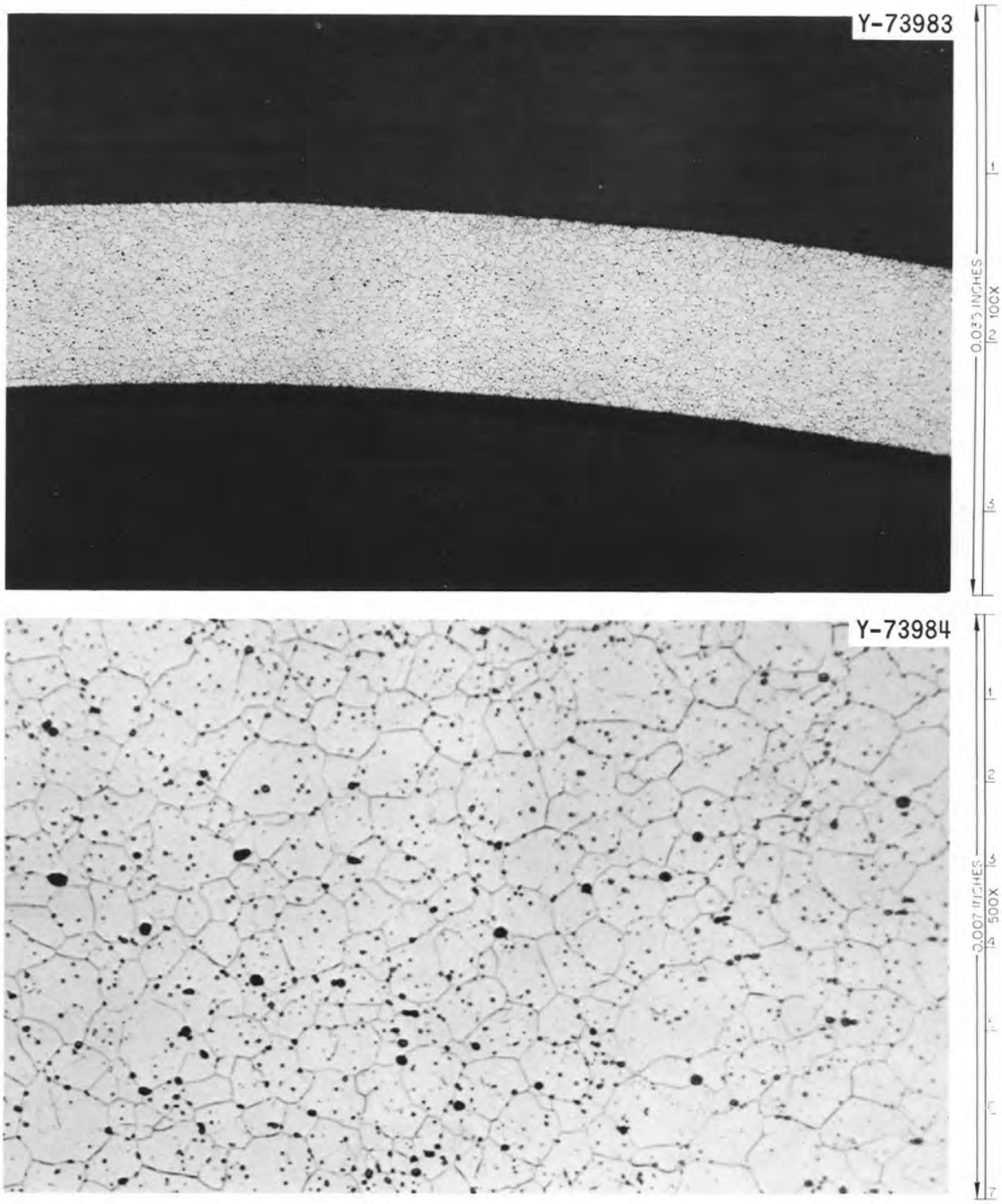

Fig. 16. Microstructure of the As-Received Tubing. Etchant: 10% oxalic acid.

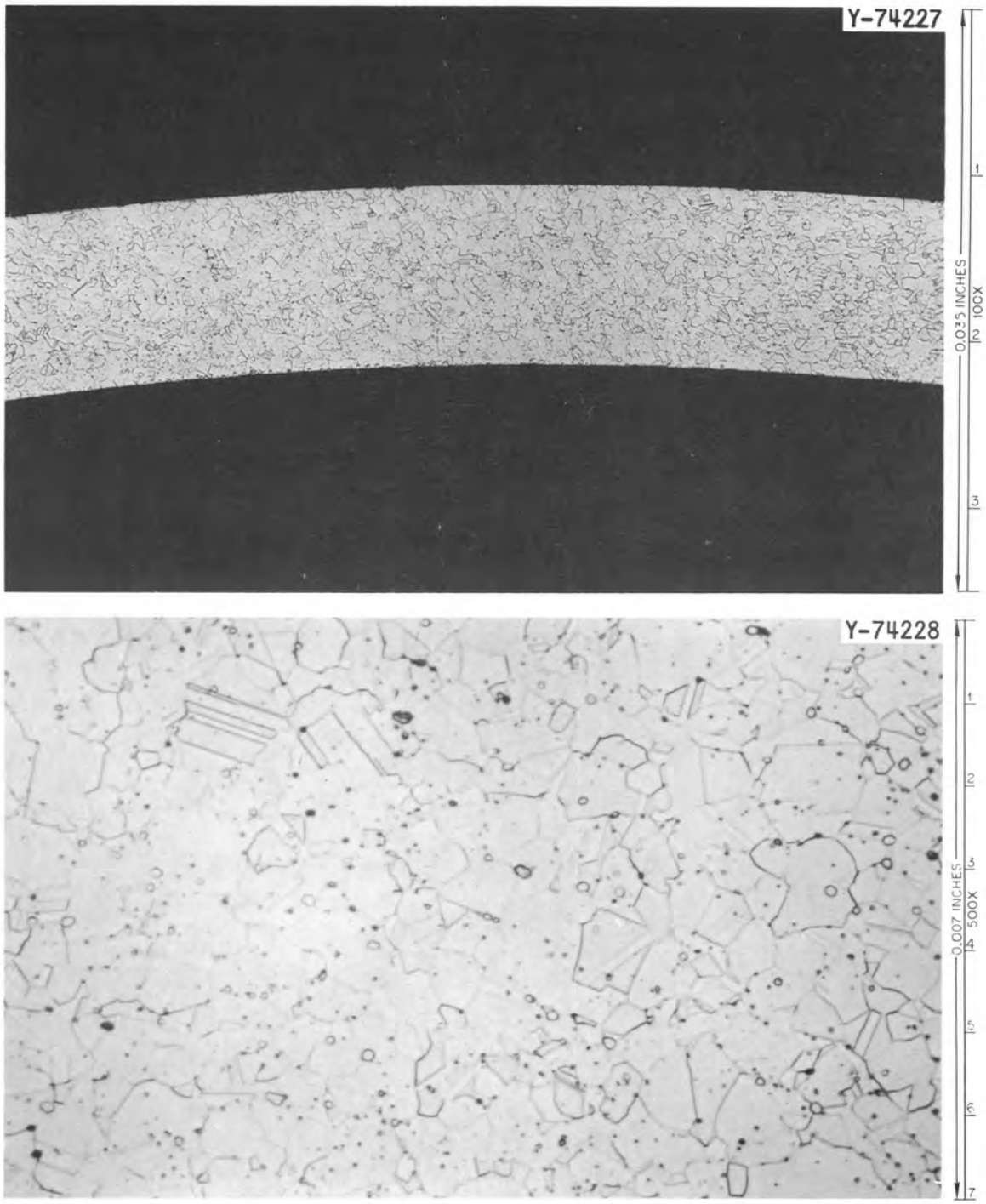

Fig. 17. Microstructure of the As-Received Tubing. Etchant: Marble's reagent.

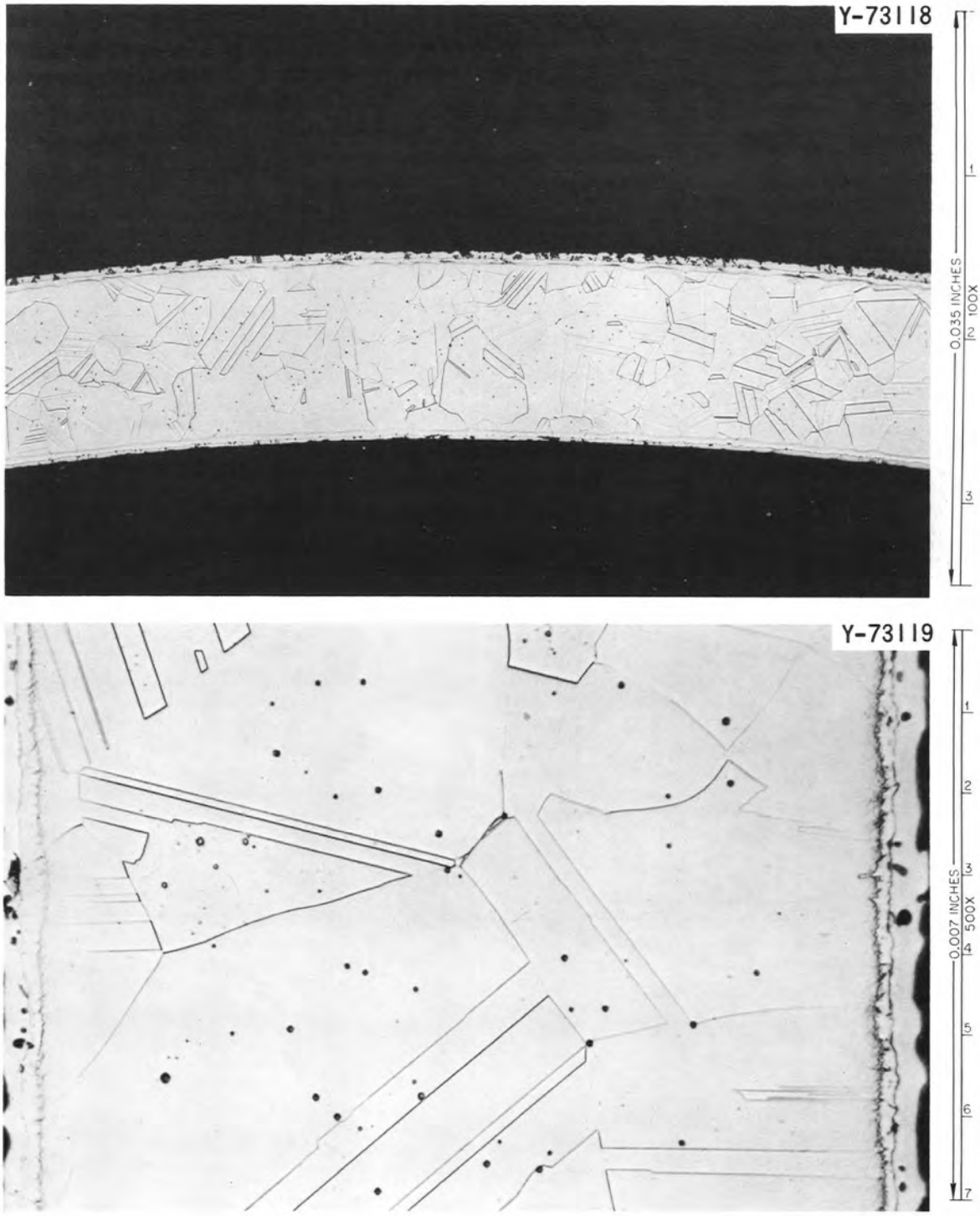

Fig. 18. Microstructure of the Tubing in the Chromized Condition. Etchant: glyceria regia.

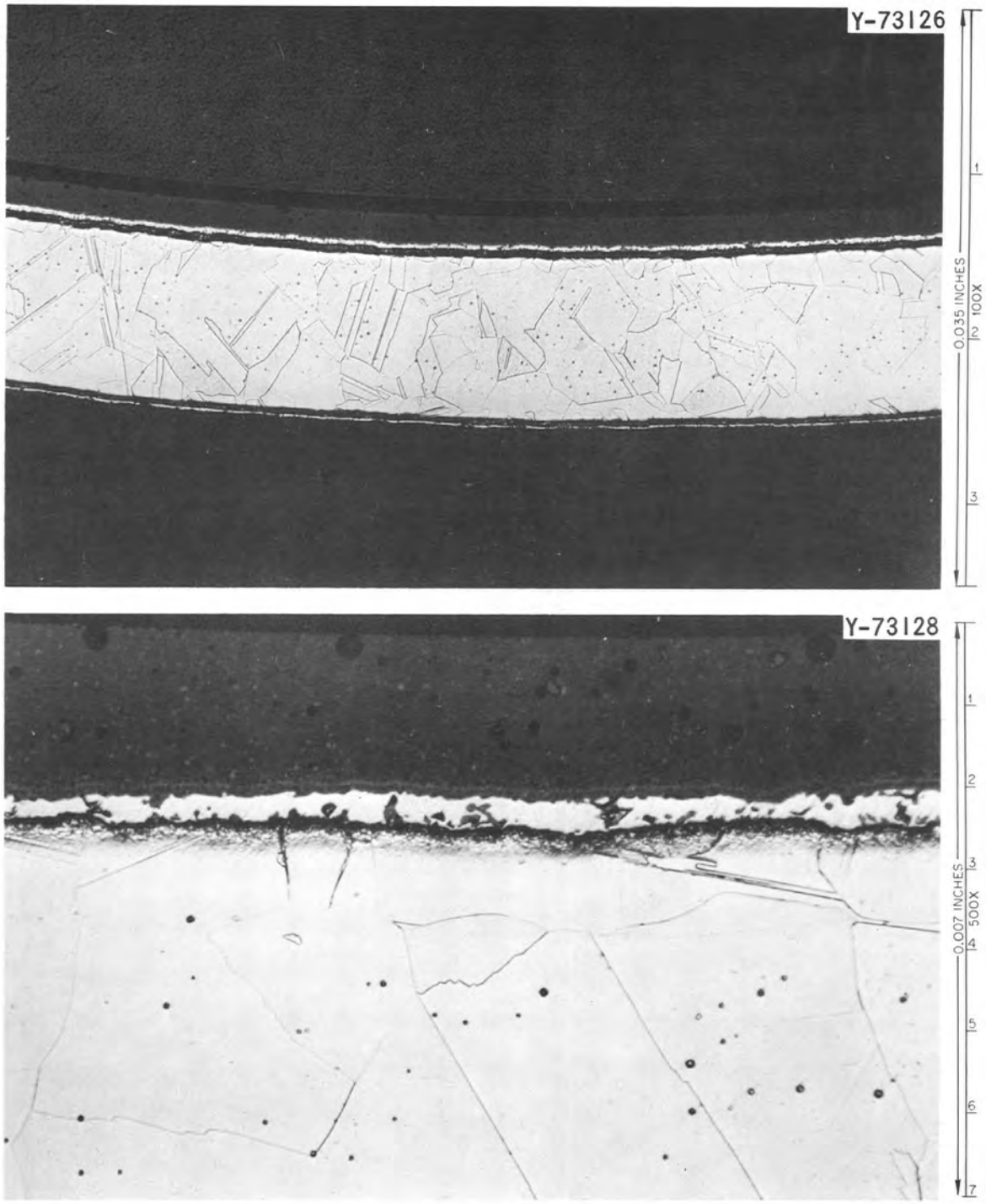

Fig. 19. Microstructure of the Tubing in the Coated Condition. Etchant: glyceria regia.

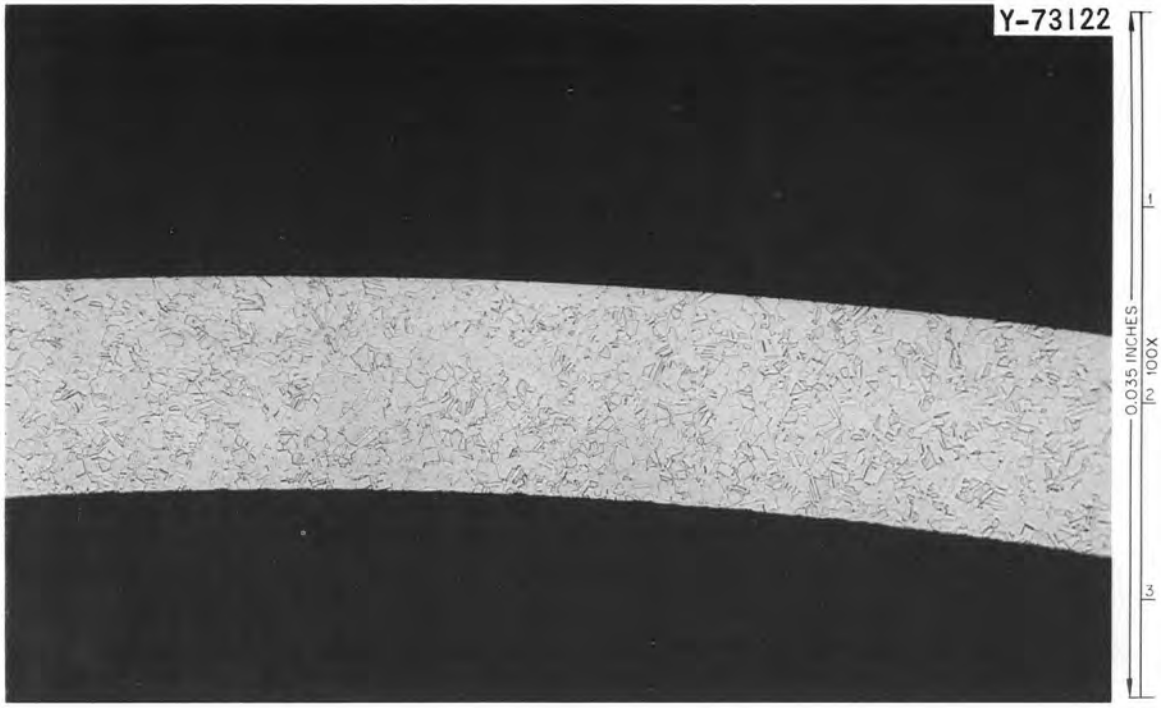

Fig. 20. Smallest Grain Size Noted in As-Received Tubing that had Been Annealed for 2 hr at 1093°C. Etchant: glyceria regia.

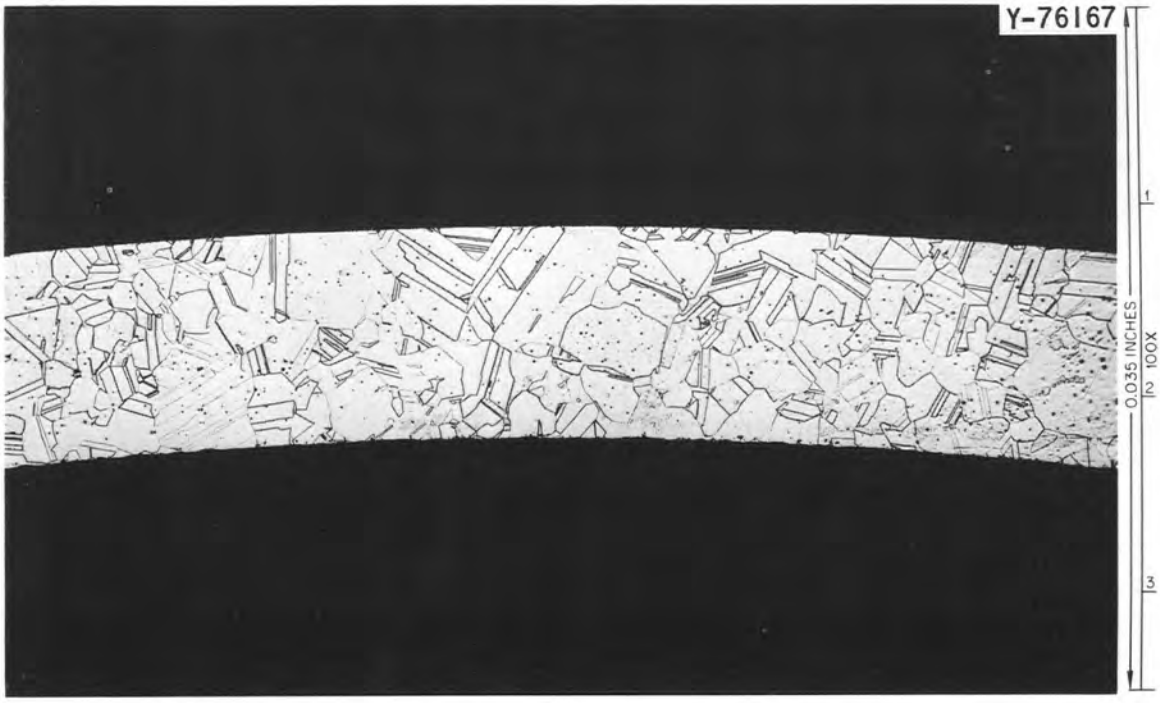

Fig. 21. Largest Grain Size Noted in As-Received Tubing that had Been Annealed for 2 hr at 1093°C. Etchant: glyceria regia.

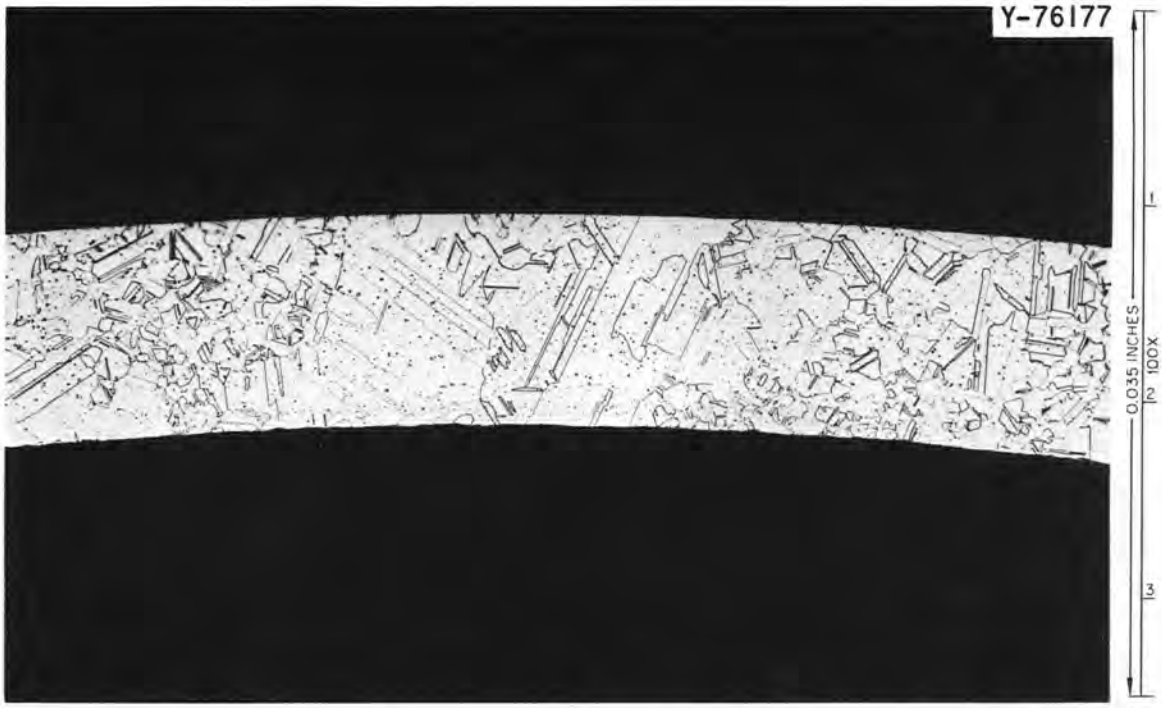

Fig. 22. Typical Area Showing Mixed Grain Size that was Noted in Several Sections of As-Received Tubing after a 2-hr Anneal at 1093°C. Etchant: glyceria regia.

All of the test specimens were examined metallographically. Figure 23 shows the fracture of specimen 6 that is typical of the tubes tested in the as-received condition. Numerous intergranular cracks were formed with a large number apparently originating at the surface. The deformation is quite uniform with little evidence of necking at the fracture. Figure 24 shows the fracture of chromized tube No. 1 that exhibited a rather low ductility. The fracture is intergranular, and the individual grains are not greatly distorted. Figure 25 shows the microstructure of specimen 5 (also typical of No. 7). The failure is again intergranular, but the individual grains show signs of extensive deformation. Although specimen 1 appears somewhat atypical, there seems to be nothing abnormal about the test conditions, and one must conclude that there probably are sections in the tubing having ductilities this low. The coated tubes showed much the same behavior as the chromized tubes. Figure 26 shows typical microstructures of tube 4. In Fig. 26a there are numerous intergranular cracks, considerable deformation near the fracture, and some signs of transgranular shearing at

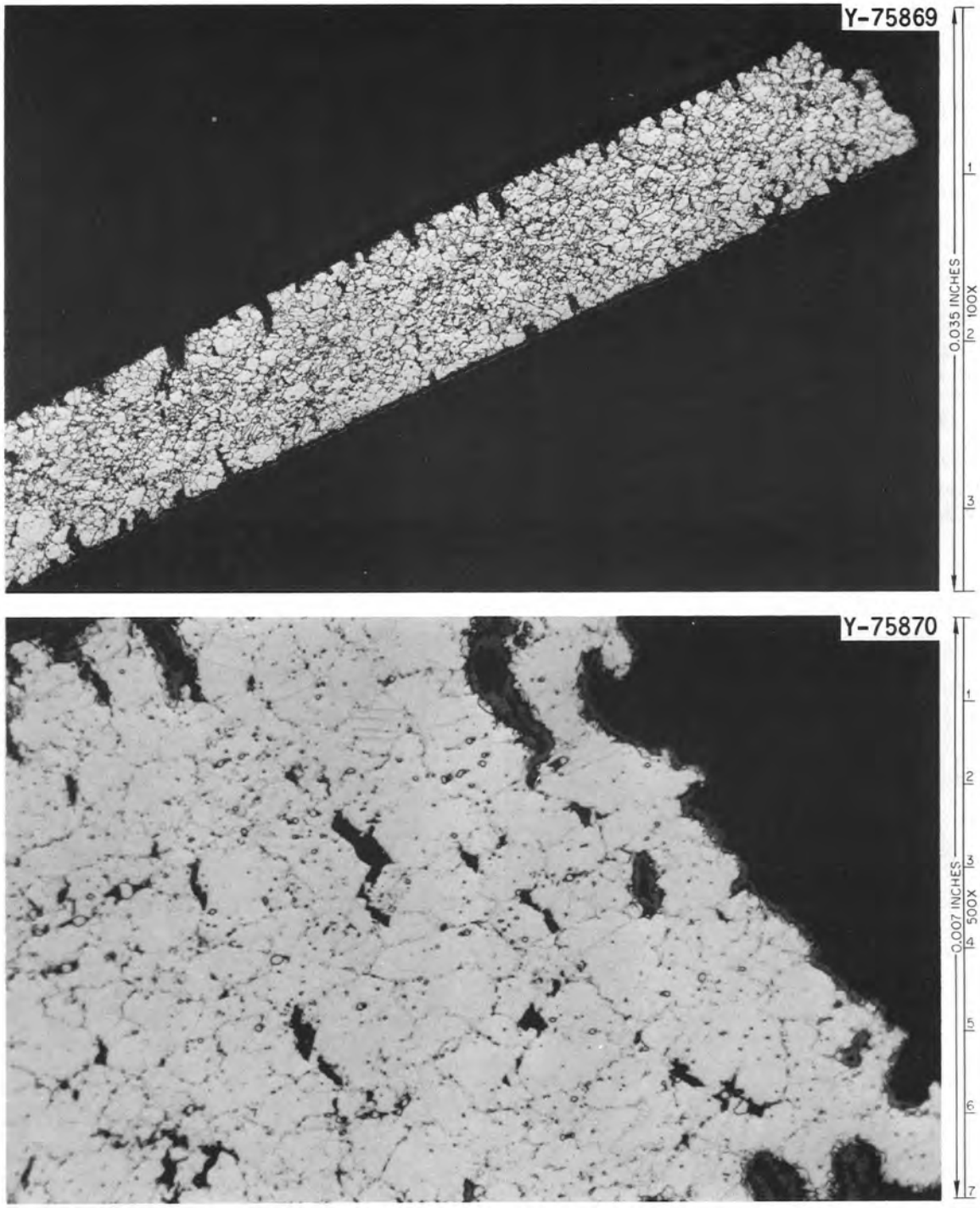

Fig. 23. Longitudinal View of the Fracture in Specimen 6. Tested in the as-received condition at 760°C and at a strain rate of 0.001 min^{-1}. Etchant: glyceria regia.

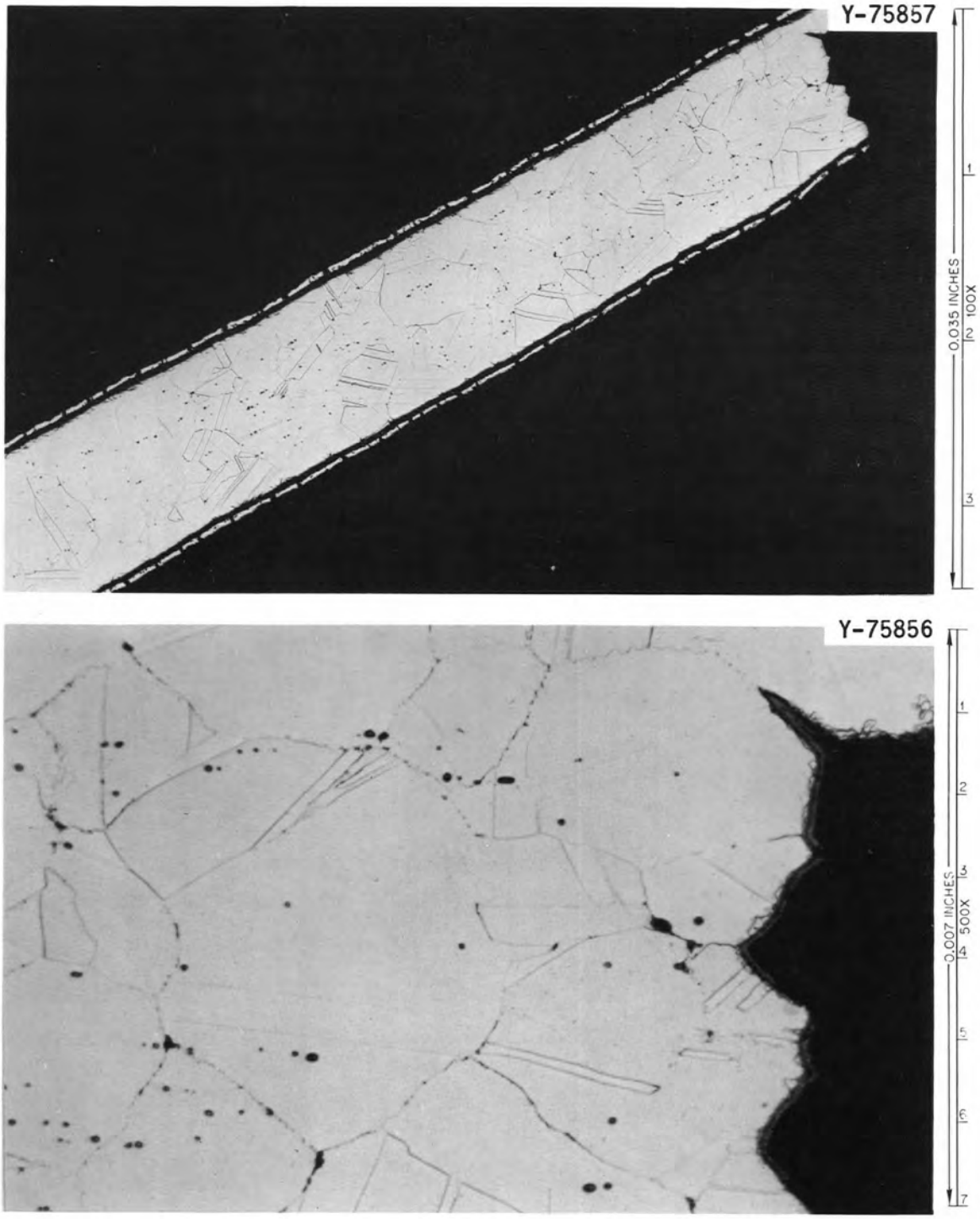

Fig. 24. Longitudinal View of the Fracture of Specimen 1. Tested in the chromized condition at 760°C and at a strain rate of 0.001 min^{-1}. Etchant: glyceria regia.

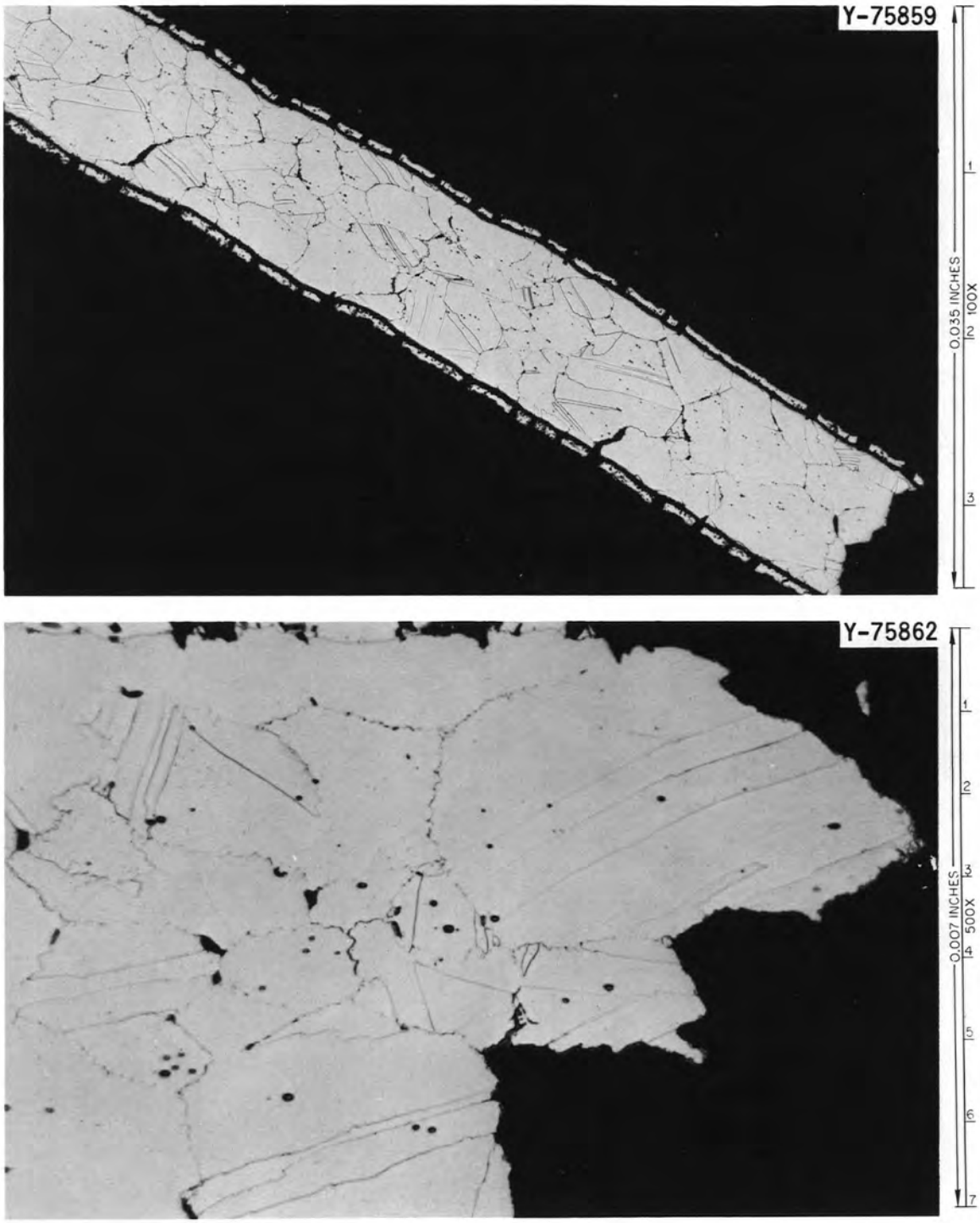

Fig. 25. Longitudinal View of the Fracture of Specimen 5. Tested in the chromized condition at 760°C and at a strain rate of 0.001 min^{-1}. Etchant: glyceria regia.

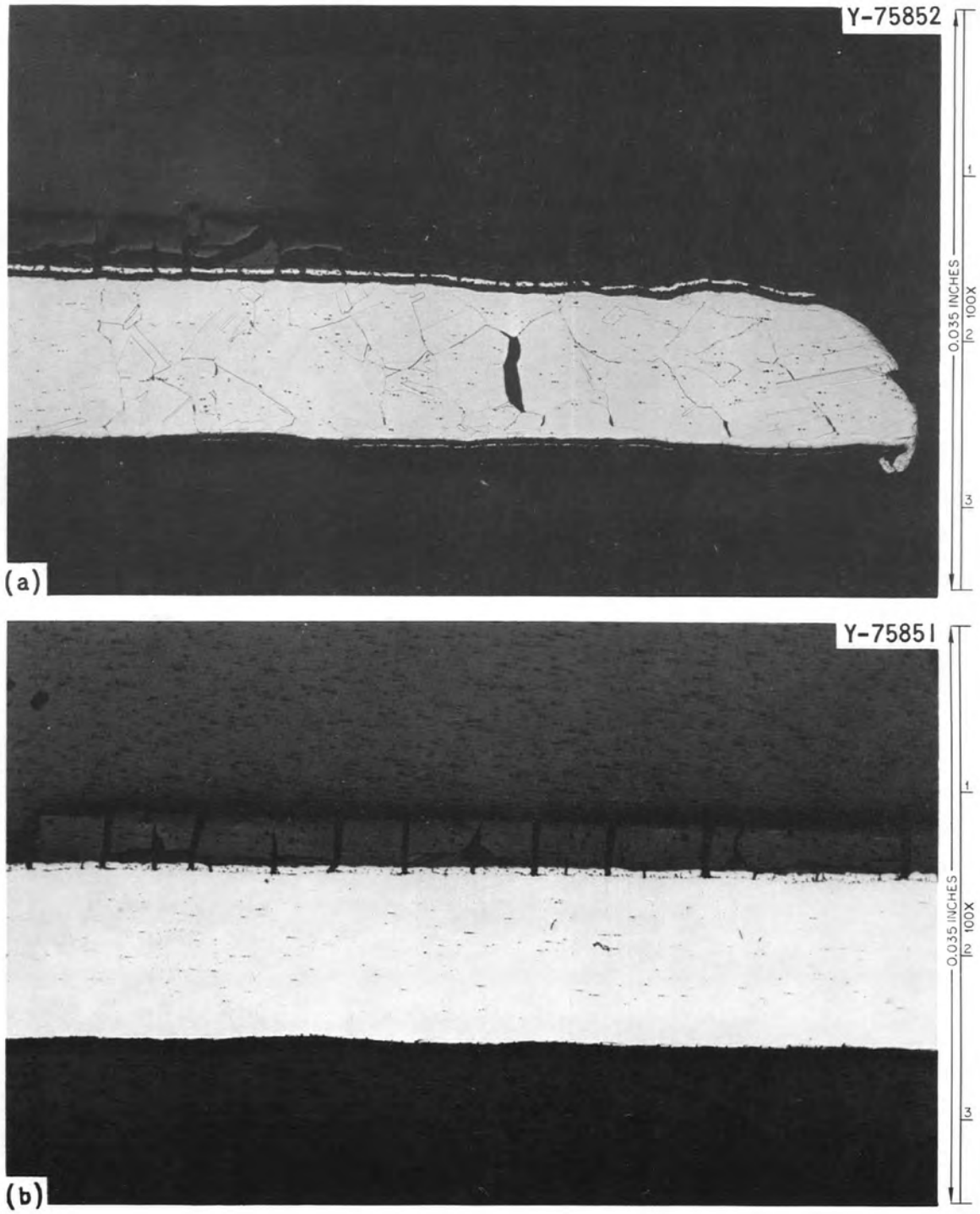

Fig. 26. Longitudinal View of Specimen 4 Tested in the Coated Condition at 760°C and at a Strain Rate of 0.001 min^{-1}. (a) Fracture. Etchant: glyceria regia. (b) About 1/2 in. from fracture. As polished.

the fracture. The low ductility of the coating is shown in Fig. 26b. This photograph was taken about 1/2 in. from the fracture where the axial strain was about 3%. Figure 27 shows the fracture of specimen 2 that had only about one-half the ductility of specimen 4 (Fig. 26). The intergranular cracking is very limited, and there is little evidence of deformation in the grains near the fracture. Thus, there seems to be a good correlation between the observed ductilities and the microstructure. However, this limited study has not revealed why the ductility is lower in some areas.

The possibility of using the brittle coating as a means of measuring strain seems quite feasible. If one compares the total crack width with the specimen length in Fig. 26b, the ratio is 0.066. This compares favorably with the measured circumferential strain of 0.03 (0.06 axial strain). A photomicrograph of the fracture on the same specimen had a crack width-to-total length ratio of 0.10. This compares very well with the measured circumferential strain of 0.046 (0.092 axial strain). Hence, it may be possible to measure localized strains by this technique.

Electron Microprobe Studies

Since we were asked to do microprobe studies on the chromized and coated specimens but were not given the constituents of the coating, we had to do some very unnecessary "detective" work. The electron microprobe data are semiquantitative in nature. No corrections were made for x-ray absorption or fluorescence. The samples were initially investigated with respect to nickel, chromium, molybdenum, and silicon. These elements were found to be distributed uniformly in the as-received material with the following semiquantitative percents of each present: 76% Ni, 7.2% Cr, 12.4% Mo, and 0.1 to 0.2% Si. The concentration gradients in the chromized and coated specimens were very similar to each other. These concentration profiles are shown in Fig. 28. There appears to be considerable interdiffusion between the chromium layer and Hastelloy N substrate, with appreciable quantities of molybdenum in the chromium layer and penetration of the chromium to a depth of about 60 μ

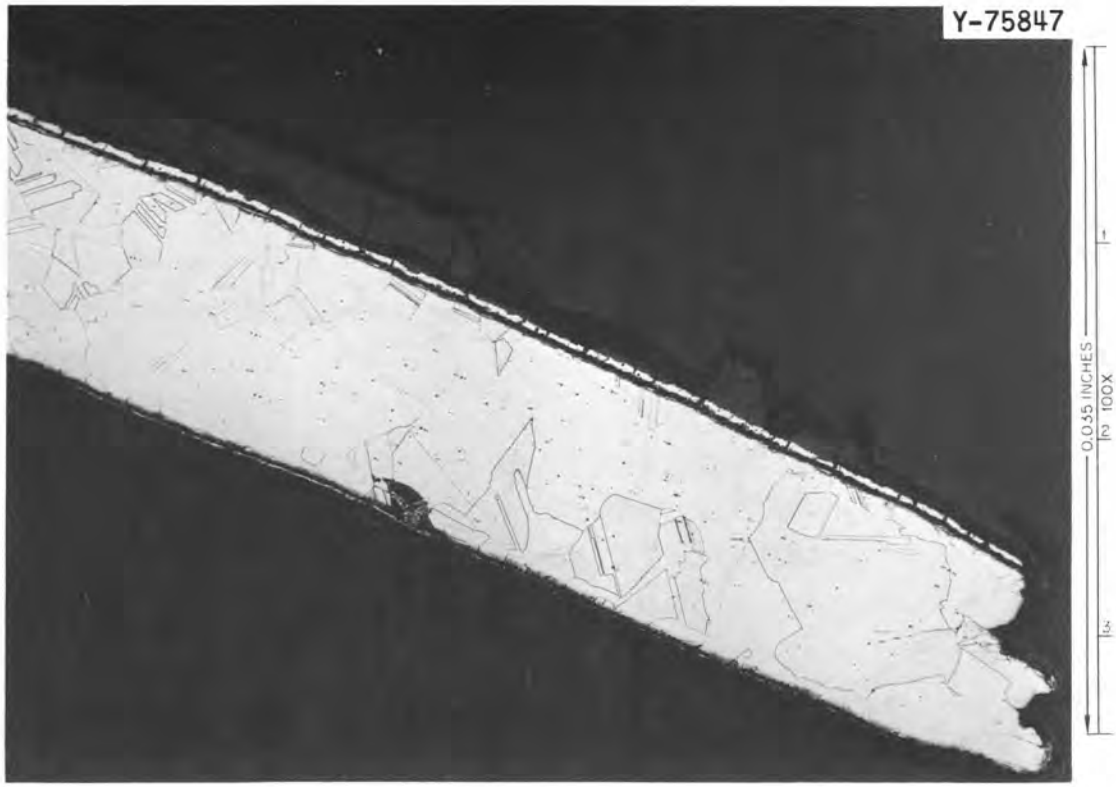

Fig. 27. Longitudinal View of the Fracture of Specimen 2. Tested in the coated condition at 760°C and a strain rate of 0.001 min^{-1}. Etchant: glyceria regia.

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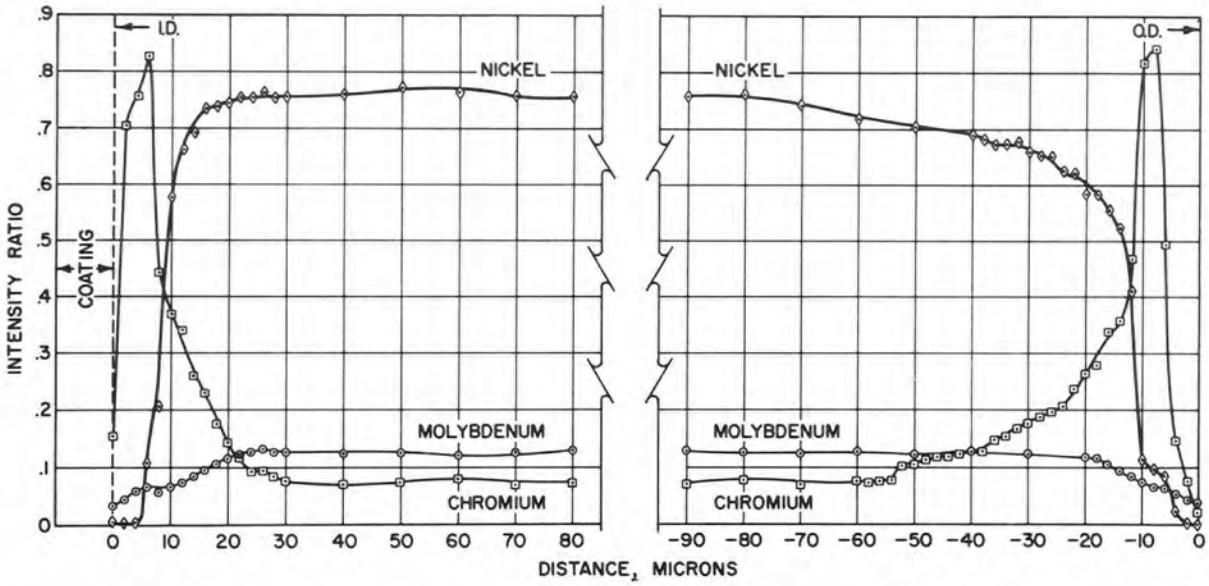

Fig. 28. Concentration Profiles of Nickel, Chromium, and Molybdenum Across a Coated Hastelloy N Tube. Determined by electron microprobe techniques.

[redacted]

(approx 0.002 in.). The reason for the greater depth of chromium penetration from the outside diameter of the tube is not known. There appeared to be very little diffusion of silicon into the specimen from the silicon-rich surface layer with only 0.1 to 0.2% Si detected in the chromium layer.

The microprobe results indicated that the coating was not pure SiO_2, and some analytical work was done on the coating. The coating was separated from the Hastelloy N by electrochemical dissolution in a methanol–10% HCl solution. Spectrographic analysis on the layer gave the results indicated in Table 4. Several of these elements were then checked by the microprobe, and the estimated concentrations are given in Table 4.

Table 4. Analytical Work on the Coating

Element	Chemical Composition, wt %	
	Spectrographic Analysis Concentration	Microprobe Estimated Concentration
Al	3.7	
Ba	18.2	a
Ca	0.25	
Cr	1.6	0.5
Fe	0.05	
Mn	0.01	
Si	19.4	20
Sr	0.02	
Ti	4.5	7

[a]Detected by microprobe, but no standards were available for estimating the concentration.

[redacted]

[redacted]

These studies indicated that the surface chromium diffuses into the metal. The potential degradation of properties due to this process deserves some attention. The diffusion rate of chromium in Hastelloy N has been determined.[1] These data and the grain-boundary diffusion model of Turnbull[2] have been used to predict the chromium concentration as a function of distance into the specimen (Fig. 29). The ratio of the grain-boundary diffusion coefficient to the bulk diffusion coefficient is of the order of 10^6 for many metals. If this is the case for Hastelloy N at 760°C, the grain-boundary chromium concentration at the center of the specimen may approach 20%. The effects of such high chromium concentrations on the properties are not known. However, tests on specimens aged for at least 10,000 hr at 760°C should define whether a problem exists.

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[1]R. B. Evans, J. H. DeVan, and G. M. Watson, Self-Diffusion of Chromium in Nickel-Base Alloys, ORNL-2982 (January 1961).

[2]D. Turnbull, "Grain Boundary and Surface Diffusion," p. 129 in Atom Movements, American Society for Metals, Cleveland, Ohio, 1951.

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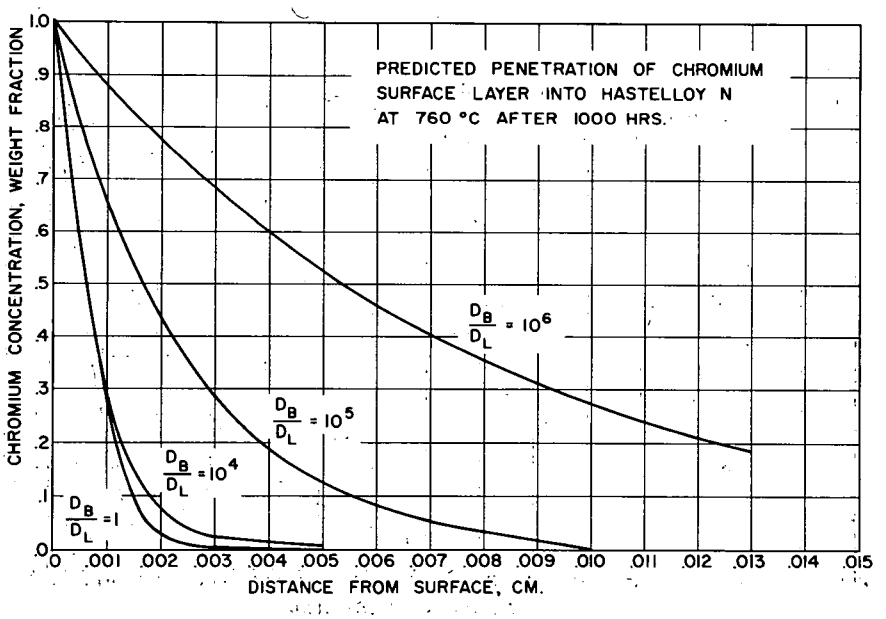

Fig. 29. Predicted Penetration of Chromium from the Surface Layer into the Hastelloy N after 1000 hr at 760°C.

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SUMMARY

This study has demonstrated that bending stresses are present in tubes that are being stressed axially. The best "reasonable" techniques that we could develop resulted in a bending stress of about 5% of the axial stress. Tensile properties were determined at 760°C and at a strain rate of 0.001 min^{-1} for the tubing in the as-received, chromized, and coated conditions. The minimum strains observed were 7%, although the values normally were in the range of 15 to 25%. In those cases where the fracture ductilities were low, the microstructure also reflected the lack of plastic deformation.

A tube-ring test was investigated in which rings of the tube were pulled, so that the maximum principal stress was in the circumferential direction. These tests gave ultimate tensile strengths and fracture ductilities at temperatures of 650°C, and above, that were in good agreement with those obtained by axial tests on the tubes. These tests offer no particular advantage over the axial tube test except under conditions where sufficient material may not be available for performing the axial test.

The large grain size of the chromized tubing does not appear to be totally explained. Much of the variation in grain sizes observed can probably be explained in terms of nonuniform working of the tubing prior to the chromizing process. This effect can be minimized by fabricating the tubing by a schedule that places the tubing in a cold-worked state before its final sizing and straightening. The lack of knowledge concerning the fabrication schedule used in preparing the tubing seems to be a shortcoming of the program.

Electron microprobe studies have shown that the chromium from the surface layer diffuses into the specimen. Predictions have been made concerning the depth of penetration of chromium along the grain boundaries during service at 760°C.

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