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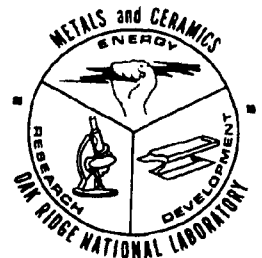
### Fabrication, Evaluation, and Inspection of Cold-Reduced and Cold-Drawn Tubes of Modified 9 Cr-1 Mo Steel

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FABRICATION, EVALUATION, AND INSPECTION OF COLD-REDUCED  
AND COLD-DRAWN TUBES OF MODIFIED 9 Cr-1 Mo STEEL

V. K. Sikka, R. E. McDonald, and J. H. Smith

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FABRICATION, EVALUATION, AND INSPECTION OF COLD-REDUCED  
AND COLD-DRAWN TUBES OF MODIFIED 9 Cr-1 Mo STEEL\*

V. K. Sikka, R. E. McDonald, and J. H. Smith

ABSTRACT

Modified 9 Cr-1 Mo steel is being developed and commercialized jointly by ORNL and Combustion Engineering (CE), Chattanooga, Tennessee, as an alternate steam generator material for breeder reactors. The improved elevated-temperature tensile, creep, fatigue, and creep-fatigue properties of this alloy also make it suitable for cladding and duct, upper internal, and large piping applications in breeder reactors. The alloy also has potential applications in fossil energy. The alloy is also being studied as a backup to HT9 for the fusion reactor first wall.

The alloy has been commercially melted by the argon-oxygen decarburization (AOD) process and refined by electroslag remelting (ESR). It has also been commercially fabricated into plate, bar, tube hollows, and centrifugally cast and cold-pilgered tubes. The purpose of this study was to develop procedures for fabricating tubes of various sizes by cold-reducing and drawing processes. Fabricated tubes were subjected to microstructural analysis, hardness measurements, and ultrasonic inspection. This study enabled the following conclusions:

1. The modified 9 Cr-1 Mo steel tube shells can be successfully cold reduced up to 78% in one step. This can be accomplished for tubes that were centrifugally cast and cold pilgered from air-melted stock or for hot-extruded tube from AOD-ESR stock.
2. The cold-reduced tubes could be cold drawn by using a 40% schedule following 800°C heat treatment for 15 min at each step.
3. A complete normalize and temper heat treatment is required to produce equiaxed grain structure in both circumferential and axial directions. This heat treatment also produces the same hardness for all tube sizes.
4. Ultrasonic inspection of cladding-size tubes showed that they should be normalized and tempered to reduce grain size and the corresponding background noise to render the tubes inspectable by this method.
5. The procedure developed in this study is useful for producing steam generator or cladding tubes of modified 9 Cr-1 Mo steel.

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\*Work performed under DOE/RRT AF 15 40 10.3, Task OR-1.7, "Advanced Alloy Technology."

## INTRODUCTION

Modified 9 Cr-1 Mo alloy is being developed and commercialized jointly by ORNL and CE, as an alternative steam generator material for breeder reactors. The improved elevated-temperature tensile, creep, and fatigue, and creep-fatigue properties of this alloy also make it suitable for cladding and duct, upper internal, and large piping applications in breeder reactors. It also has potential applications in the fossil energy area such as superheater and reheater tubing, main steam line, transfer lines, and large pressure vessels for synfuel plants. The alloy is also being studied as a backup to HT9 for the fusion reactor first wall application.

The alloy has been commercially melted by argon-oxygen decarburization (AOD) and refined by electroslag remelting (ESR). It has also been commercially fabricated into plate, bar, tube hollows, and centrifugally cast and cold-pilgered tubes. No problems have been encountered in melting the alloy within specifications and in fabricating it by conventional processes. The purpose of this study was to develop procedures for fabricating tubes of various sizes by cold-reducing and cold-drawing processes. Fabricated tubes were subjected to microstructural analysis, hardness measurements, and ultrasonic inspection.

## MATERIAL

The starting tube hollow used in the cold-reducing process studied at ORNL was 50.8 mm OD by 6.35 mm wall. Sections of this size were obtained from a centrifugally cast and cold-pilgered tube of heat XA3628 and a hot-extruded tube from argon-oxygen decarburization (AOD) electroslag-remelted (ESR) 14-Mg heat 30182. The chemical analyses of these tubes are listed in Table 1. Both tubes were normalized at 1040°C for 1 h and then tempered at 800°C for 15 min.

Table 1. Chemical analysis of heats used in tube fabrication by cold-reducing and cold-drawing processes

Element	Content, wt %		Element	Content, wt %	
	XA3628 <sup>a</sup>	30182 <sup>b</sup>		XA3628 <sup>a</sup>	30182 <sup>b</sup>
C	0.11	0.088	V	0.190	0.210
Mn	0.26	0.37	Nb	0.10	0.07
P	0.007	0.011	Ti	0.006	0.001
S	0.010	0.004	Co	0.017	0.017
Si	0.29	0.19	Cu	0.02	0.03
Ni	0.04	0.09	Al (Total)	0.031	0.009
Cr	8.47	8.47	N	0.044	0.054
Mo	0.94	0.88			

<sup>a</sup>Air induction melted, centrifugally cast, and cold pilgered.

<sup>b</sup>Argon-oxygen decarburization melted, electroslag remelted, and hot extruded.

#### FABRICATION

The AETNA-Standard 4-high tube reducer at ORNL was used in this study. Tube shells from each heat were reduced to 25.3 mm OD by 2.79 mm wall in one step, a reduction of 78%. At this point the cold-reduced tubes from heat XA3628 were cold drawn to many different sizes as described below.

The cold-reduced tube was tempered at 800°C in hydrogen for 15 min and then cold drawn to 13 different sizes according to a schedule calling for about 40% reduction between the anneals (Table 2). Rockwell hardness was used to follow various fabrication steps until the tubes became too thin. Figure 1 displays various cold-reduced and cold-drawn tubes of heat XA3628 of the modified 9 Cr-1 Mo alloy. A similar process was repeated on heat 30182 except that before pass 10 (Table 2) the intermediate tube was given a complete normalizing and tempering treatment (1038°C for 10 min and 760°C for 10 min). One piece each of this size tube (5.84 mm OD by 0.38 mm wall) was ultrasonically inspected both as fabricated and normalized and tempered. Results of this inspection are described in a later section.

Table 2. Schedule of cold-drawn tubes fabricated from heat XA3628 of modified 9 Cr-1 Mo steel

Pass	Tube size, mm			Diameter-to-wall ratio	Reduction, %		Condition
	OD	ID	Wall		Per pass	Accumulative	
Start <sup>a</sup>	25.3	19.7	2.79	9.05			1st anneal <sup>b</sup>
1	19.7	15.9	1.90	10.3	46	46	2nd anneal
2	15.5	12.7	1.40	11.1	42	69	3rd anneal
3	12.22	10.16	1.03	11.9	42	82	4th anneal
4	10.92	9.14	0.89	12.3	22	86	As drawn
5	9.78	8.23	0.77	12.6	22	89	5th anneal
6	8.94	7.62	0.66	13.5	22	91	As drawn
7	7.87	6.73	0.57	13.8	24	93	6th anneal
8	7.11	6.10	0.51	14.0	20	95	As drawn
9	6.43	5.56	0.43	14.9	23	96	7th anneal
10	5.84	5.08	0.38	15.3	20	96.7	As drawn
11	5.51	4.80	0.36	15.5	12	97.1	8th anneal
12	5.28	4.70	0.29	18.1	20	97.7	As drawn
13	4.75	4.22	0.27	17.8	18	98.1	As drawn

<sup>a</sup>This size was obtained by a one-step 78% cold reduction from 51 mm OD by 6.4 mm wall. The diameter-to-wall ratio for the starting tube was 8.0.

<sup>b</sup>Hydrogen anneal for 10 min at 800°C.

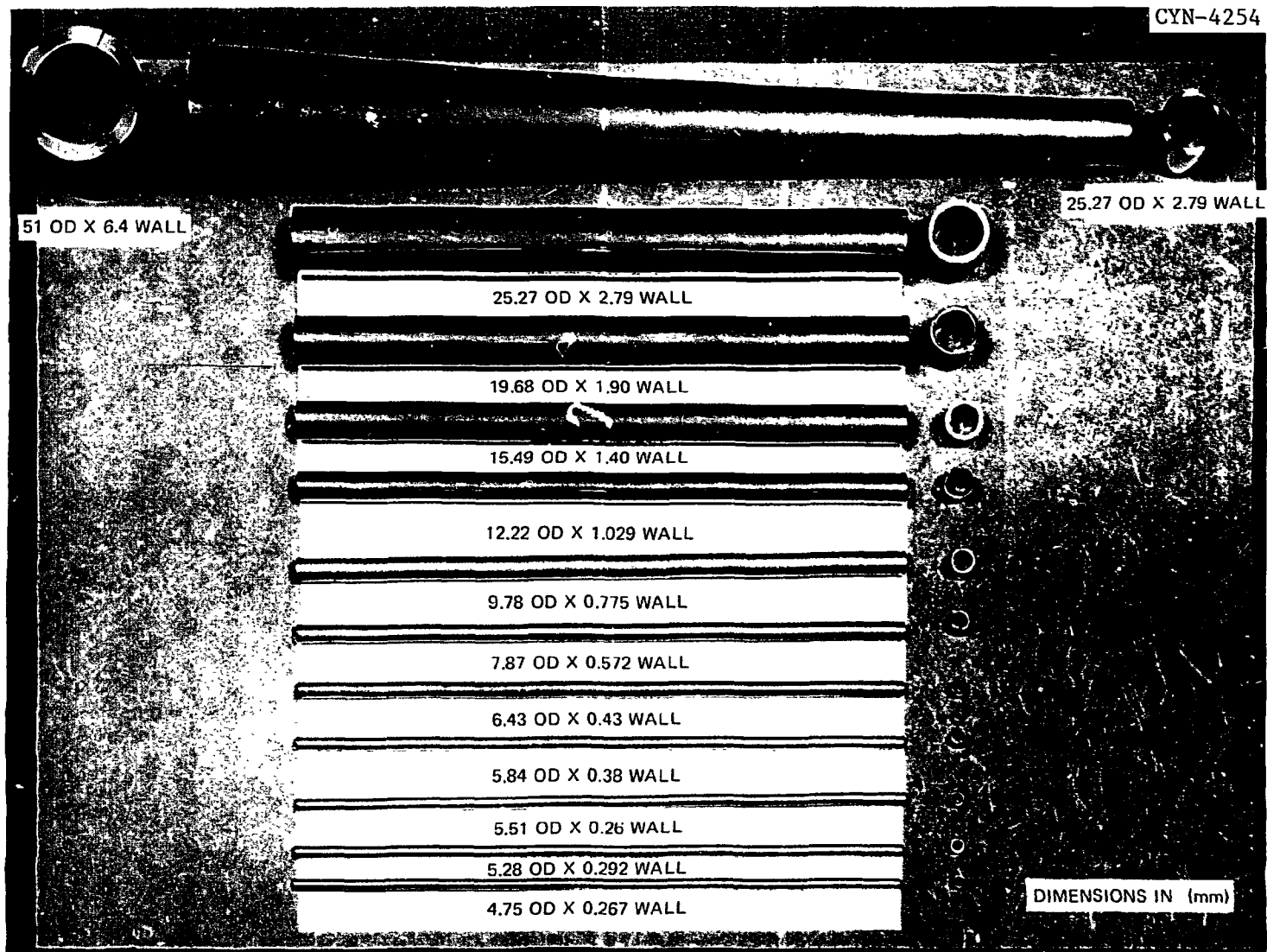


Fig. 1. Cold-reduced and cold-drawn tubes of modified 9 Cr-1 Mo steel (heat XA3628). The sizes are given in Table 2.

No problems were encountered during cold reducing or drawing on either the centrifugally cast and cold-pilgered or hot-extruded tubes.

#### HEAT TREATMENT, MICROSTRUCTURAL ANALYSIS, AND HARDNESS

Small sections (~10 mm long) were cut from the starting tube, the cold-reduced tube, and each tube size obtained after various drawing steps. In total 12 pieces were used to test heat treatment, microstructure, and hardness. The first batch of samples was as-fabricated tubes, designated as B0. The second batch, designated B1, was normalized for 10 min each at 1149 and 1040°C and then tempered 10 min at 760°C. The higher temperature normalize was given to simulate a hot working step frequently used in the fabrication of plate, bar, and tube. The third batch, designated B2, was normalized at 1038°C for 10 min and tempered at 760°C for 10 min. The fourth batch (B3) was only tempered at 760°C for 10 min. Batch B3 was used to check whether temper after fabrication was sufficient or a complete normalize and temper treatment was needed.

After heat treatment each tube was sectioned and mounted for microstructural examination in both the circumferential (transverse) and axial (longitudinal) directions. Specimens were mechanically polished and etched with Villala's reagent. After metallurgical examination, each section was used for microhardness measurements. The average hardness was obtained from four indentations on each section.

The optical microstructures of the starting tube (diameter/wall =  $d/w = 8$ ), the cold-reduced tube ( $d/w = 9$ , 78% cold reduced), a cold-drawn tube ( $d/w = 12$ , 40% cold reduction following an anneal), and small tubes ( $d/w = 15$  to 18, 20 to 40% cold reduction) are used here as typical examples for all four conditions (B0, B1, B2, and B3). The microstructures of the as-fabricated tubes are shown in Figs. 2 through 6. Hardness data for this batch are plotted as a function of tube diameter/wall ( $d/w$ ) in Fig. 7.

The first cold reduction (~78%) ( $d/w = 9$ ) increased the hardness significantly. The hardness of the as-fabricated tubes of all sizes was

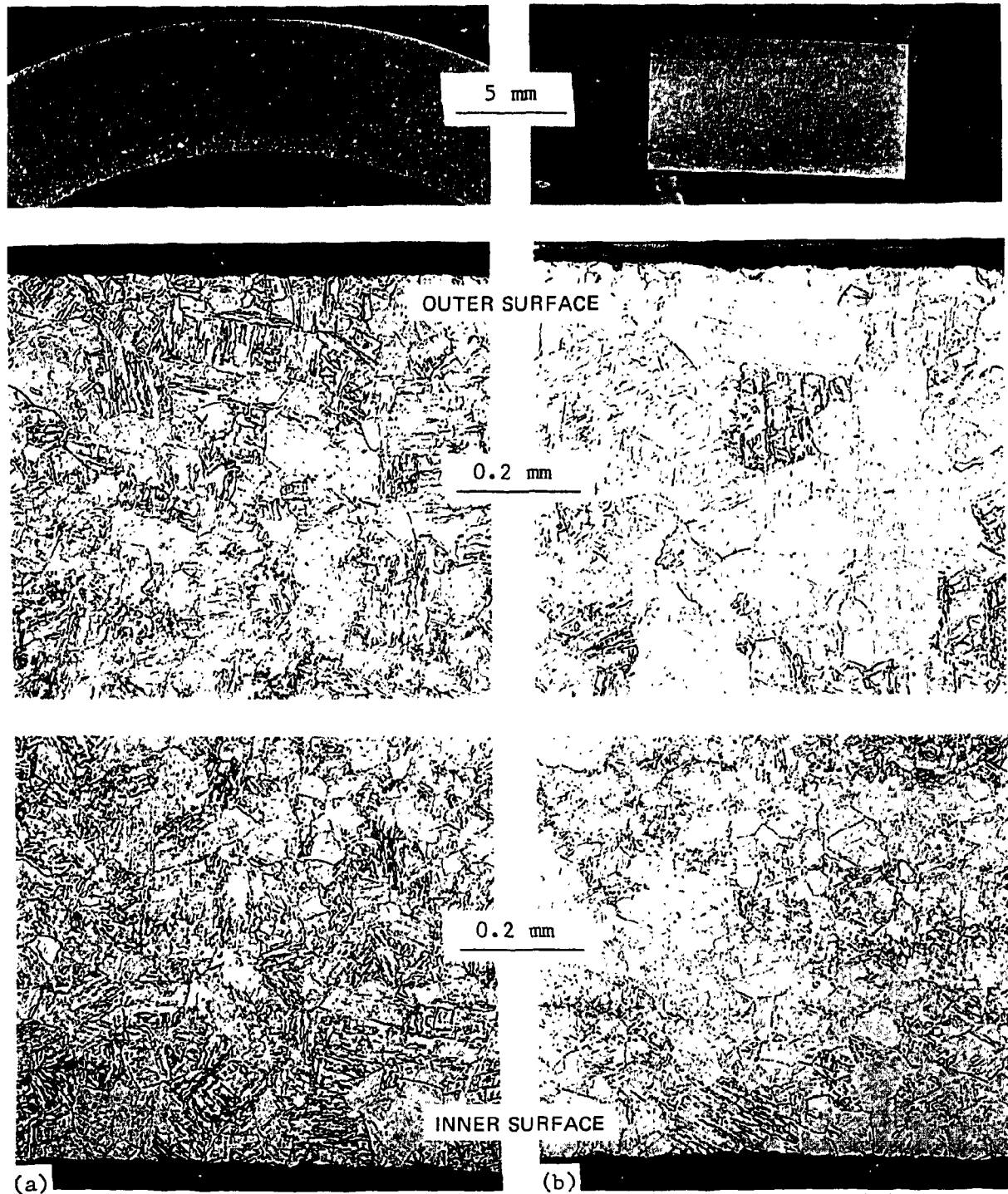


Fig. 2. Optical microstructure of the starting tube ( $d/w = 8$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was normalized and tempered (1038°C for 1 h, 760°C for 1 h). (a) Circumferential. (b) Longitudinal.

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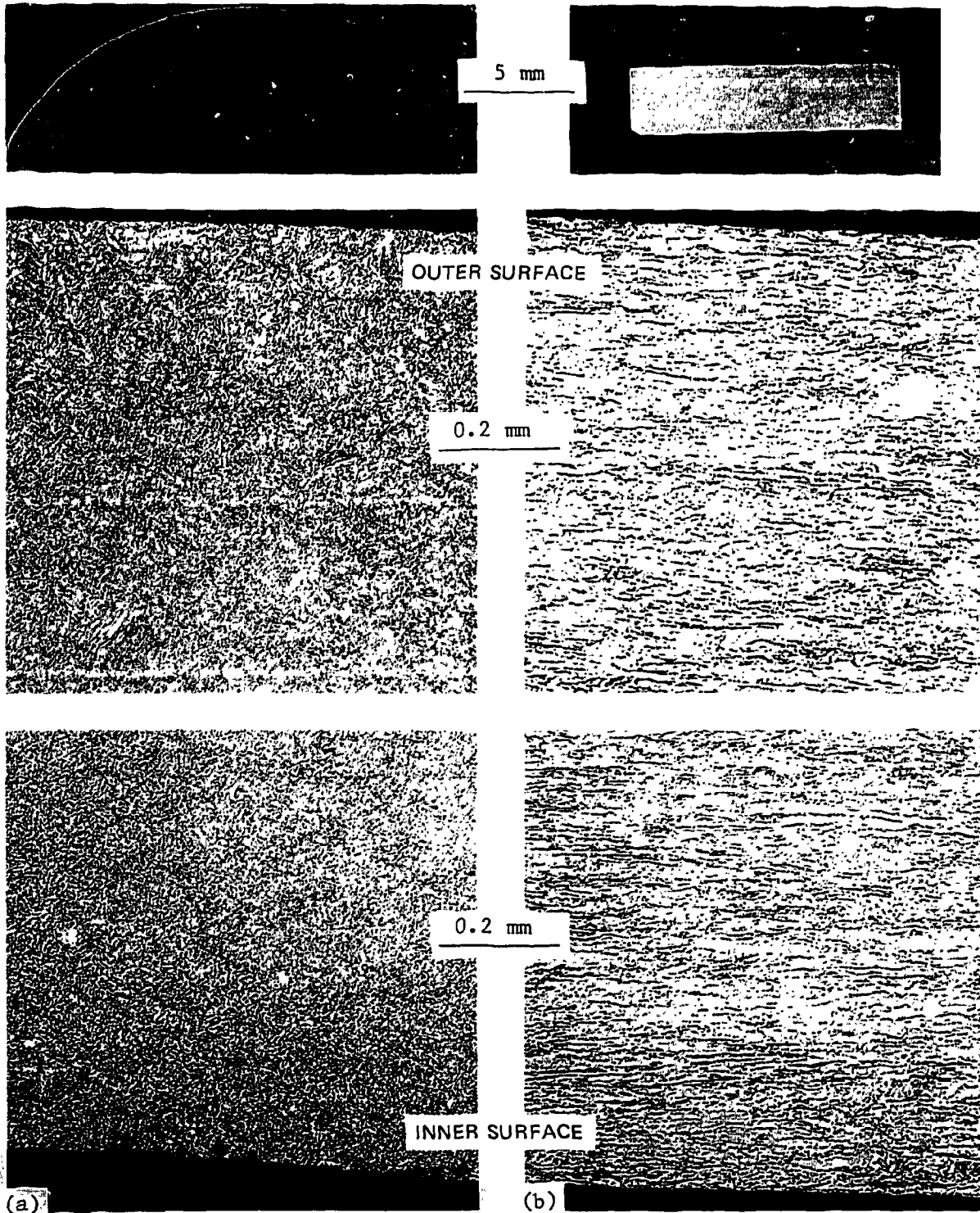


Fig. 3. Optical microstructure of the cold-reduced tube ( $d/w = 9$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube is as fabricated. (a) Circumferential. (b) Longitudinal.

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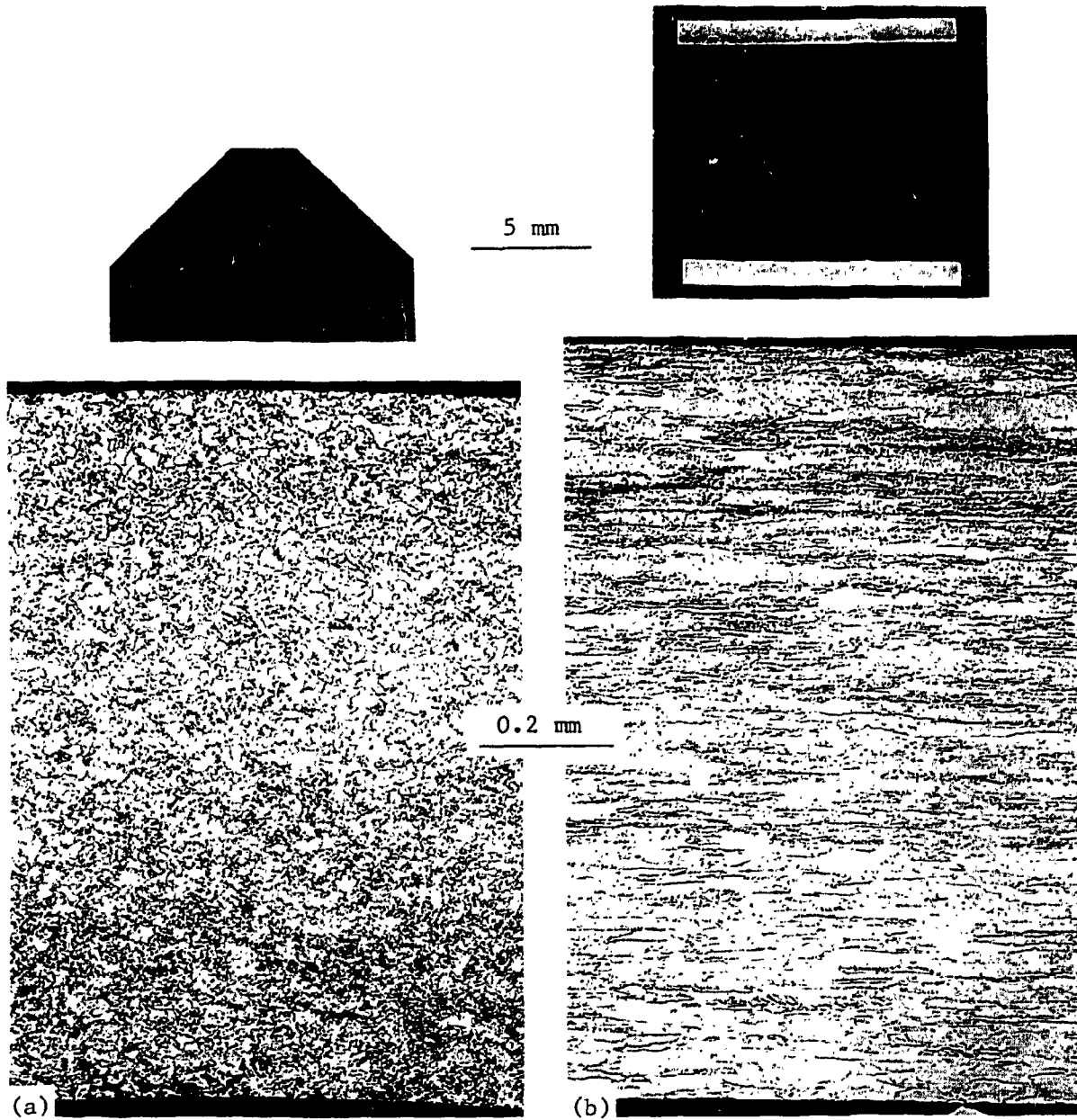


Fig. 4. Optical microstructure of the cold-reduced and cold-drawn tube ( $d/w = 12$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube is as fabricated. (a) Circumferential. (b) Longitudinal.

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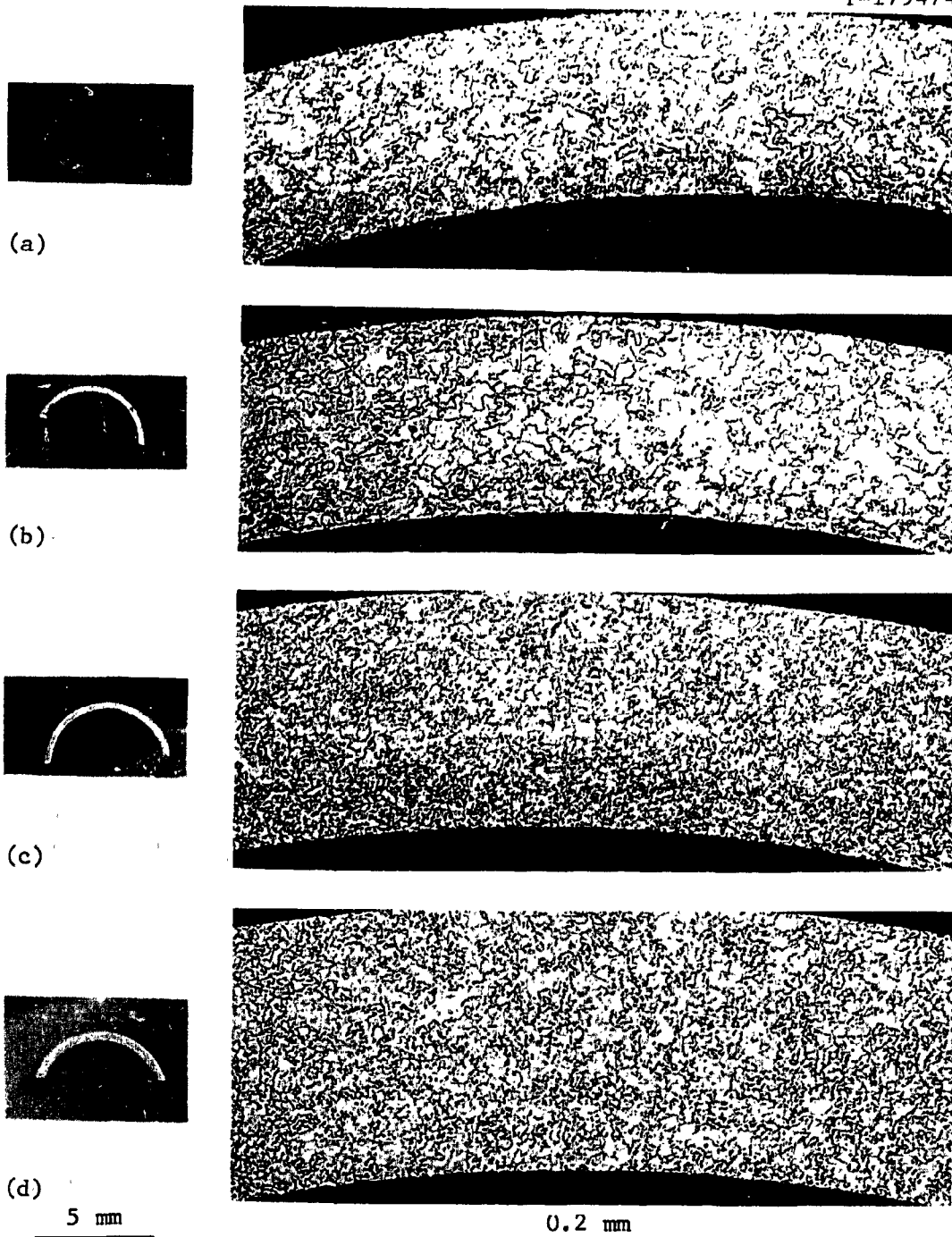


Fig. 5. Optical microstructure of cold-reduced and cold-drawn tubes of modified 9 Cr-1 Mo steel in the circumferential direction ( $d/w = 15$  to  $18$ ). As fabricated. (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

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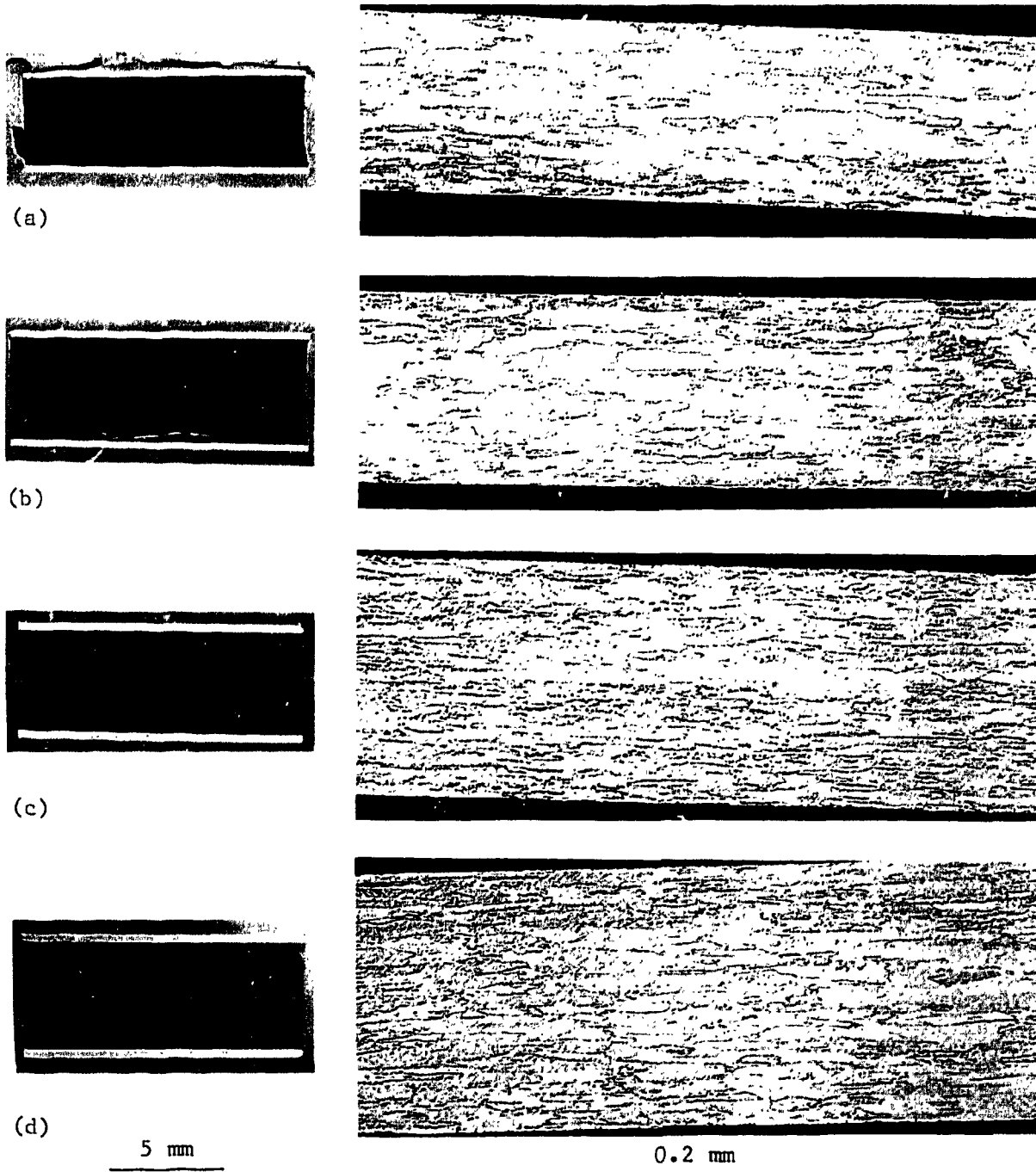


Fig. 6. Optical microstructure of cold-reduced and cold-drawn tubes of modified 9 Cr-1 Mo steel in the longitudinal direction ( $d/w = 15$  to 18). As fabricated. (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

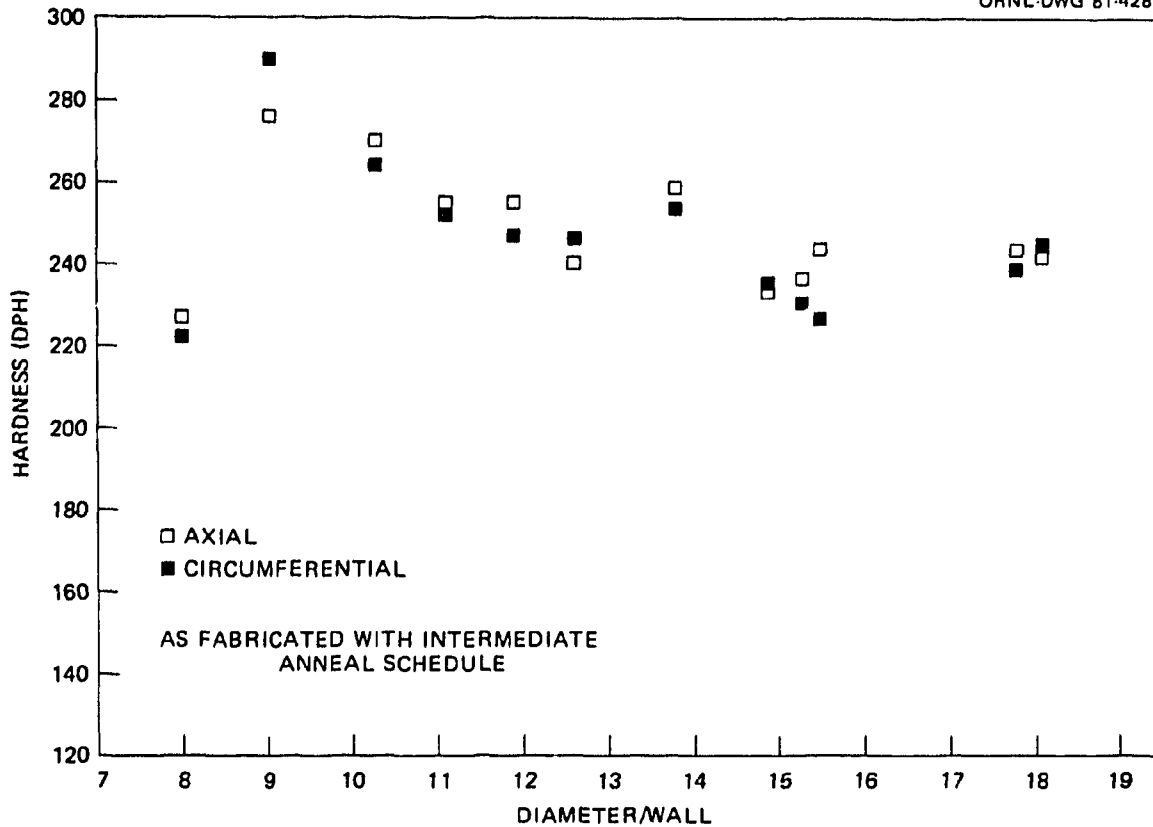


Fig. 7. Microhardness data as a function of diameter-to-wall ratio for various tubes of heat XA3628 of modified 9 Cr-1 Mo steel. The tubes are as fabricated with the intermediate anneal schedule.

higher than the starting hardness. The amount of hardness increase depended upon the extent of cold reduction during that pass (compare Fig. 7 with Table 2).

The same microstructures of tubes from batch B1, which were given a double normalize (1149°C for 10 min and 1038°C for 10 min) and a temper (760°C for 10 min), are shown in Figs. 8 through 12. The hardness data for this batch are plotted in Fig. 13. The heat treatment used for this batch produced grain refinement for the starting tube. All other tubes were recrystallized by this treatment, which produced equiaxed microstructure in both circumferential and axial directions. The hardness of all tubes was about the same after this heat treatment. Surprisingly, this treatment even increased the hardness of the starting tube, which did not

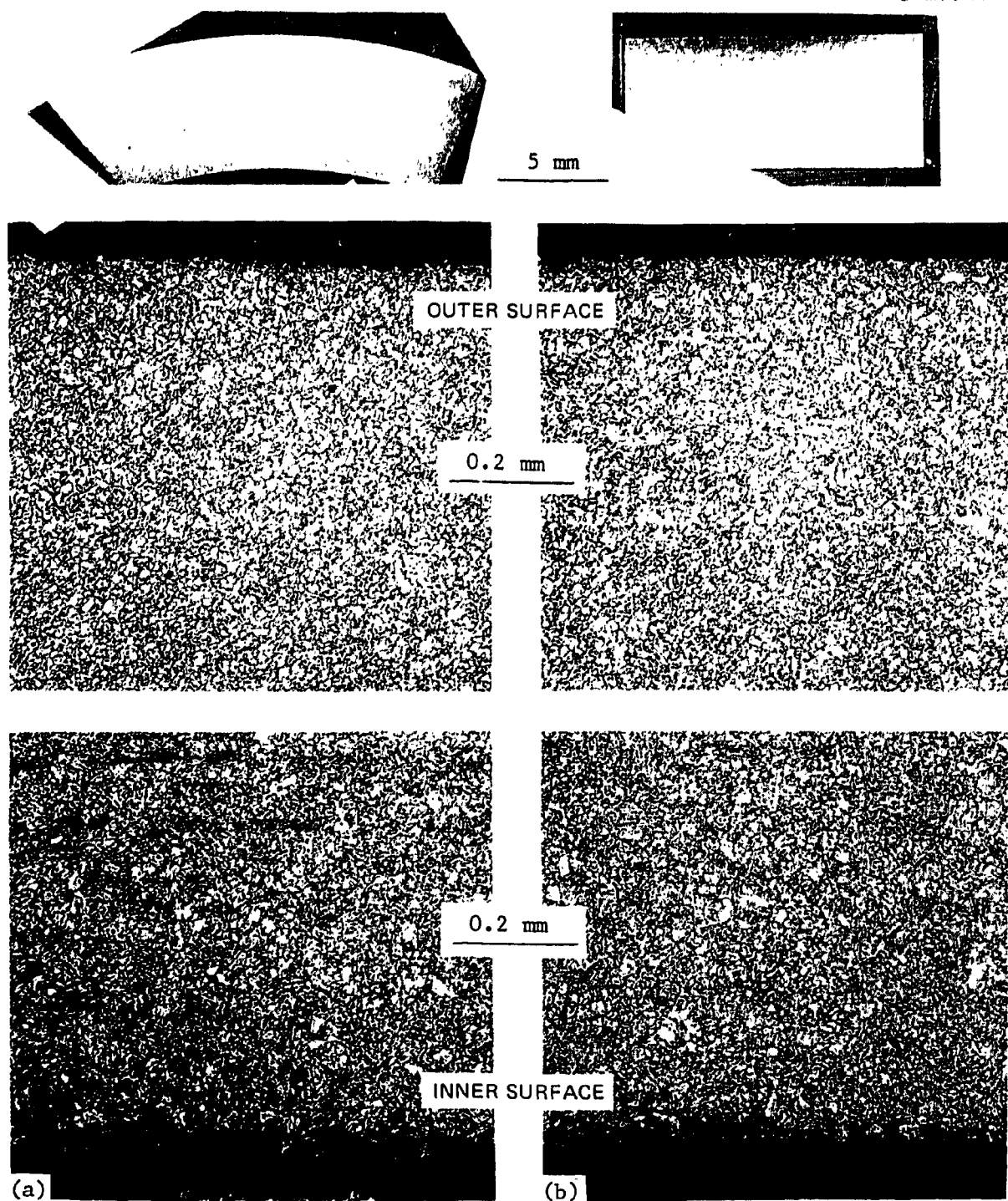


Fig. 8. Optical microstructure of the starting tube ( $d/w = 8$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was double normalized and tempered (1149°C for 10 min, 1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

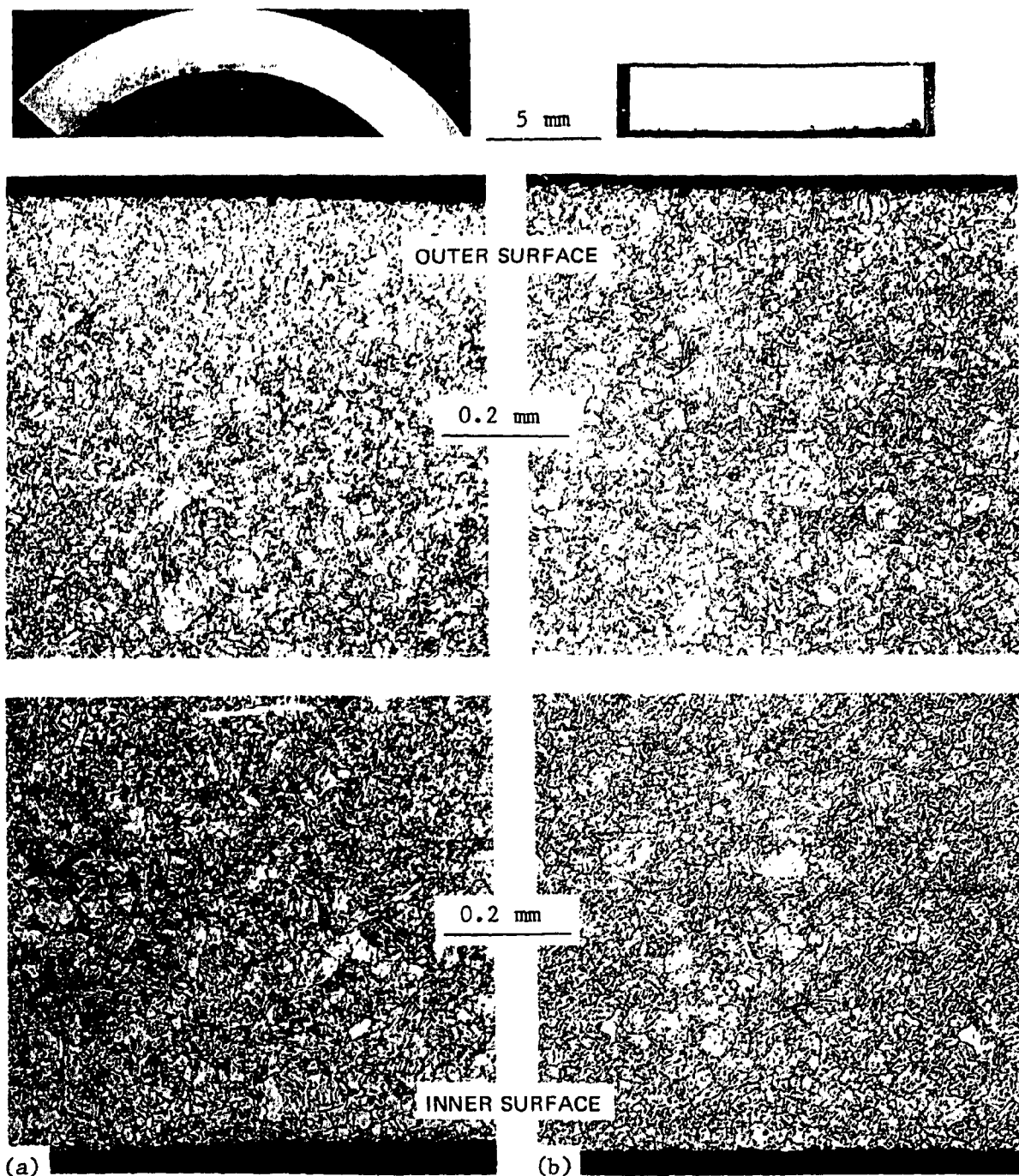


Fig. 9. Optical microstructure of the starting tube ( $d/w = 9$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was double normalized and tempered (1149°C for 10 min, 1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

Y-179478

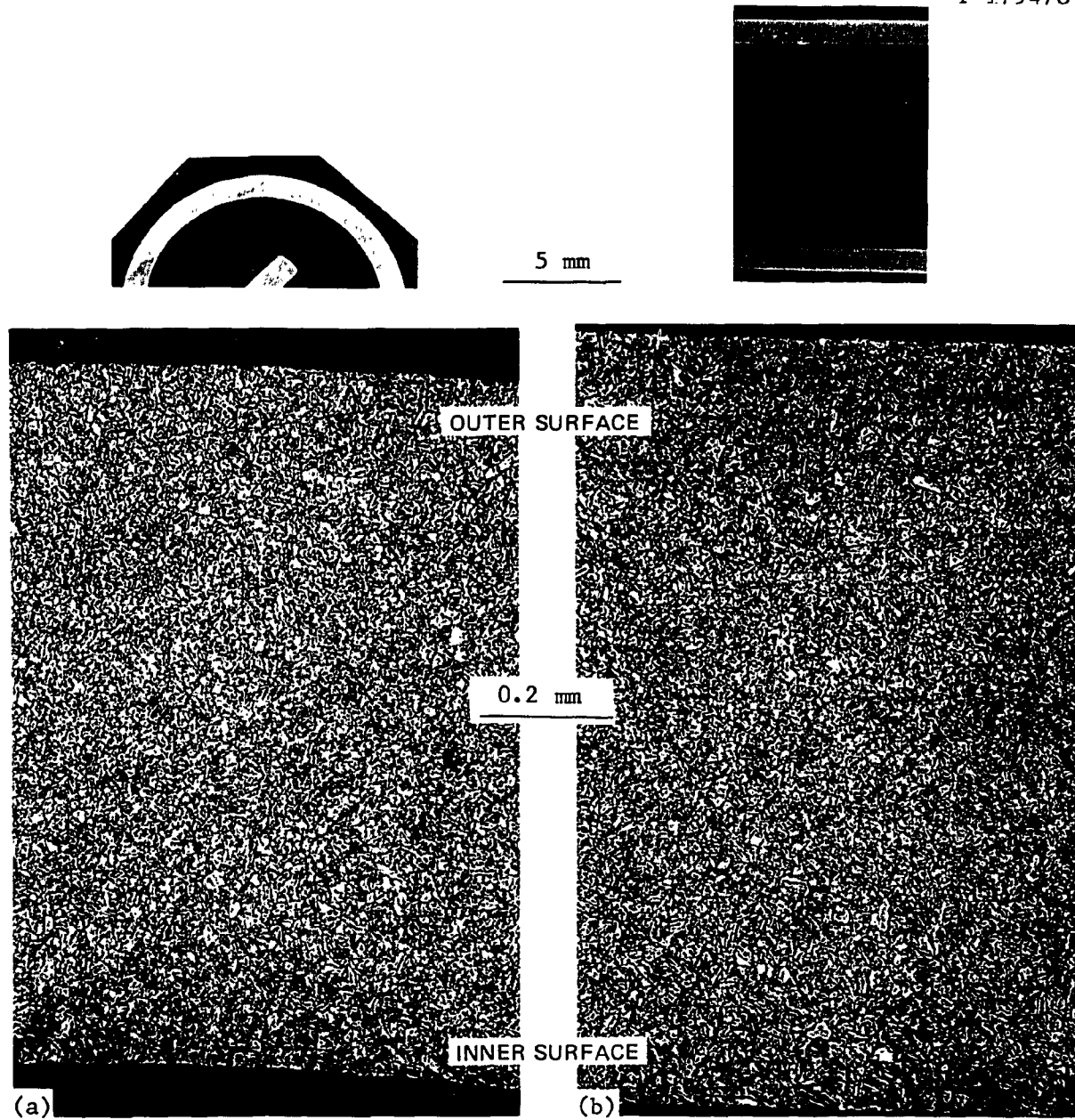


Fig. 10. Optical microstructure of the cold-reduced and cold-drawn tube ( $d/w = 9$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was double normalized and tempered (1149°C for 10 min, 1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

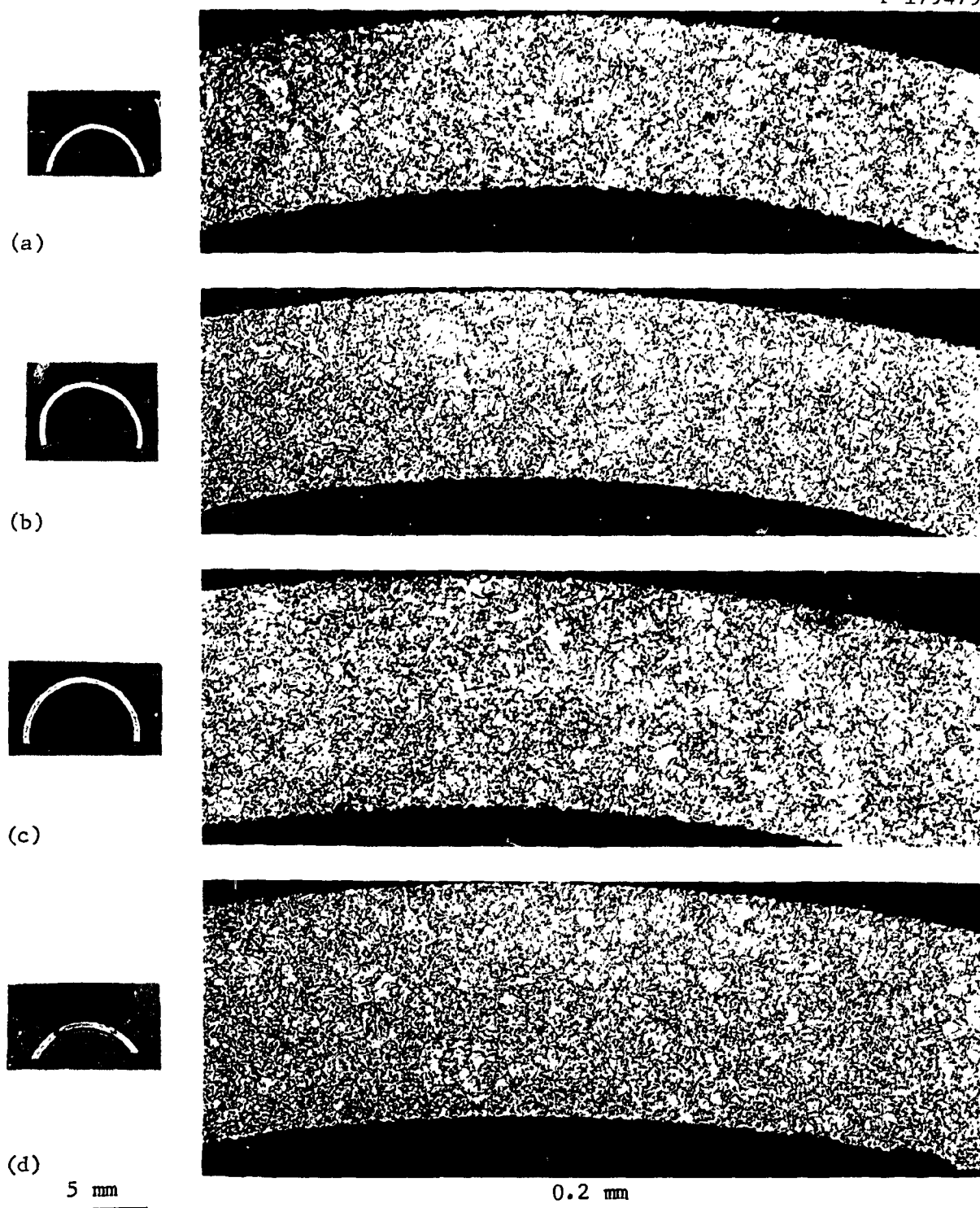


Fig. 11. Optical microstructure of cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the circumferential direction ( $d/w = 15$  to 18). The tubes were double normalized and tempered (1149°C for 10 min, 1038°C for 10 min, 700°C for 10 min). (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

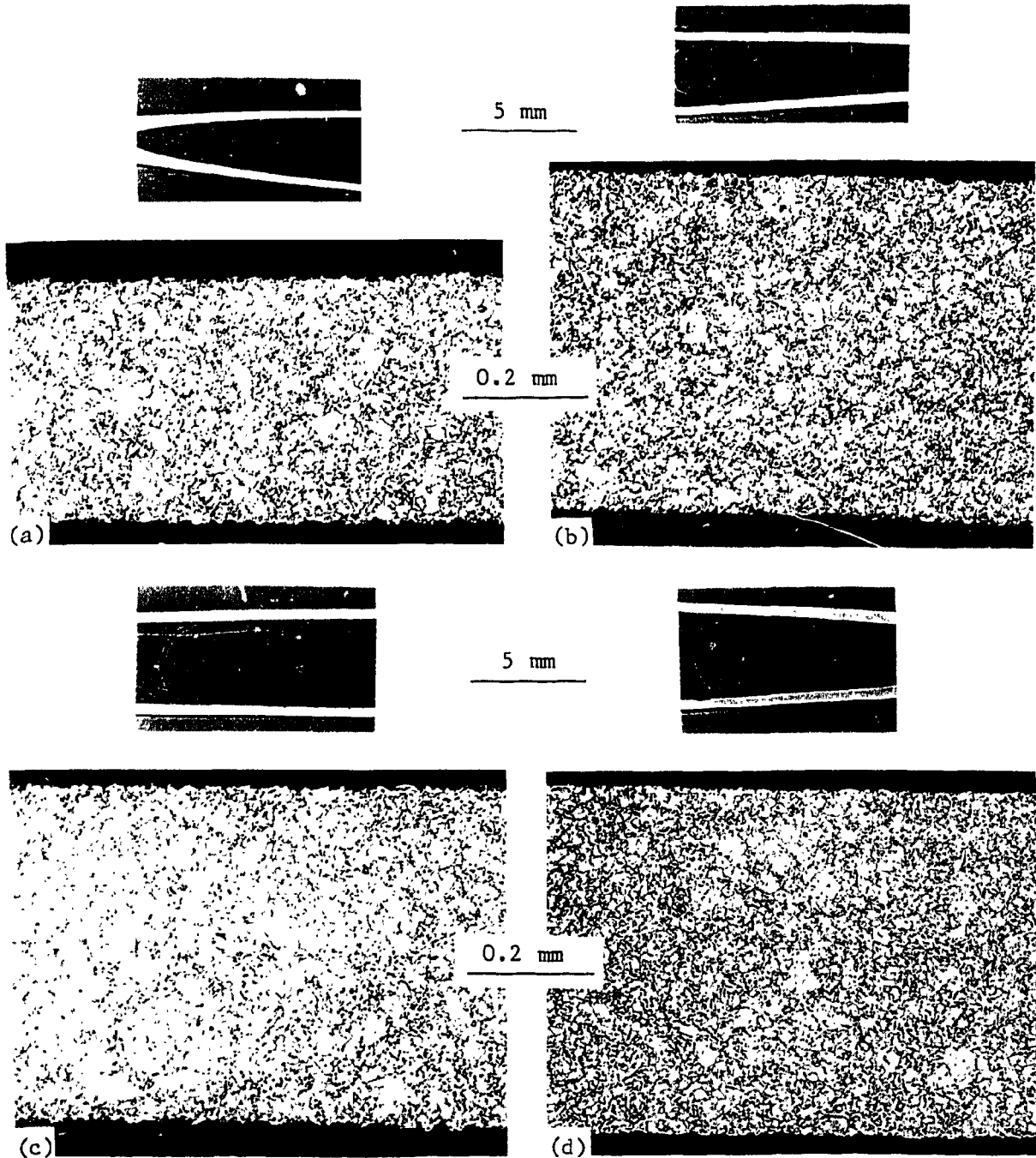


Fig. 12. Optical microstructure of cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the longitudinal direction ( $d/w = 15$  to 18). The tubes were double normalized and tempered ( $1149^{\circ}\text{C}$  for 10 min,  $1038^{\circ}\text{C}$  for 10 min,  $760^{\circ}\text{C}$  for 10 min). (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

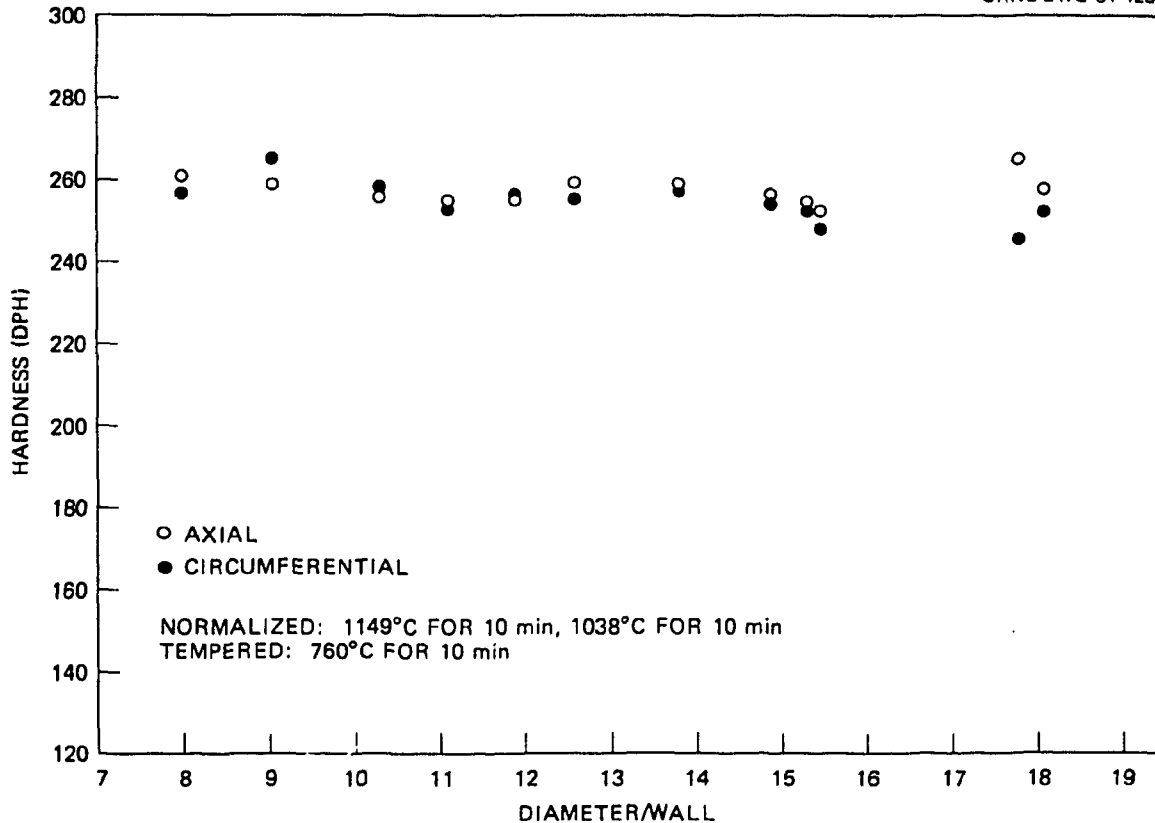


Fig. 13. Microhardness data as a function of diameter-to-wall ratio for various tubes of heat XA3628 of modified 9 Cr-1 Mo steel. The tubes were double normalized and tempered (1149°C for 10 min, 1038°C for 10 min, 760°C for 10 min).

have any cold work. The higher hardness of these tubes is possibly a consequence of the tempering time of 10 min used in this study. Work in progress on effects of the tempering time on hardness should help explain these results.

The microstructures of tubes with the batch B2 heat treatment are shown in Figs. 14 through 18, and hardness data are plotted in Fig. 19. Once again, this heat treatment produced complete recrystallization with equiaxed fine grain structure in both directions of all tubes. The hardness values for all tubes were nearly the same in both directions. However, these values were again much higher than the starting tube hardness and about the same as those observed for batch B1.

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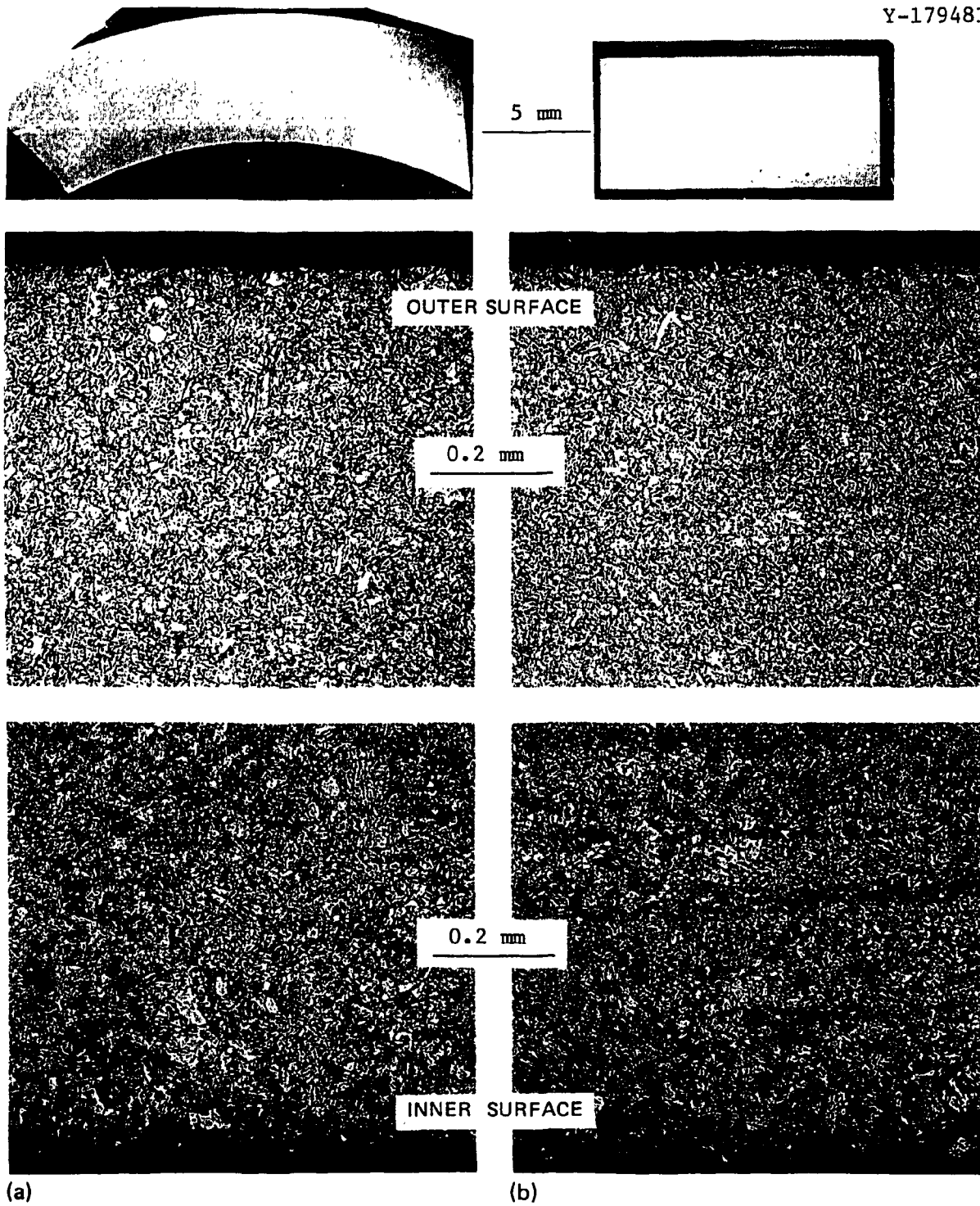


Fig. 14. Optical microstructure of the starting tube ( $d/w = 8$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was normalized and tempered (1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

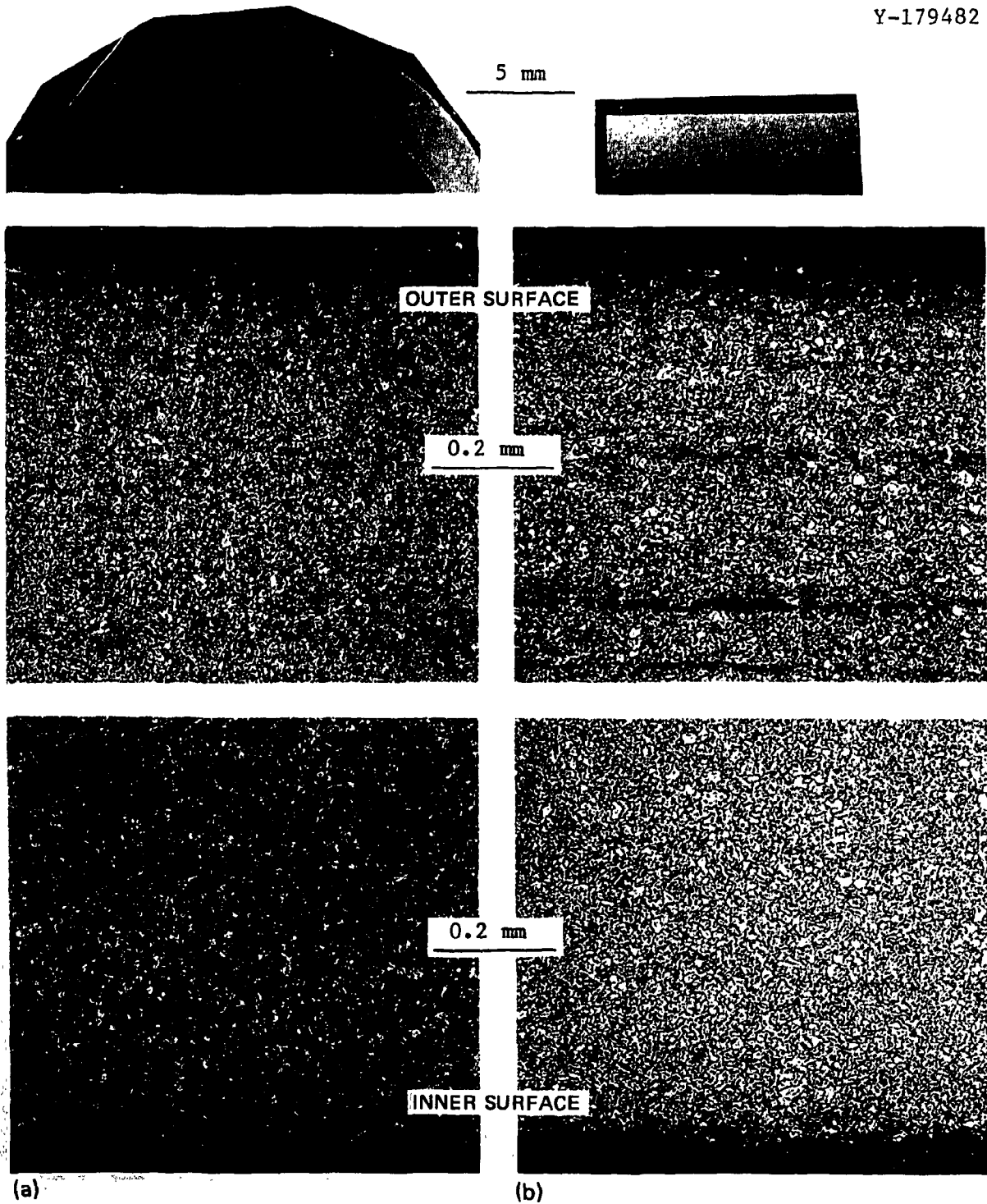


Fig. 15. Optical microstructure of the cold-reduced tube ( $d/w = 9$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was normalized and tempered (1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

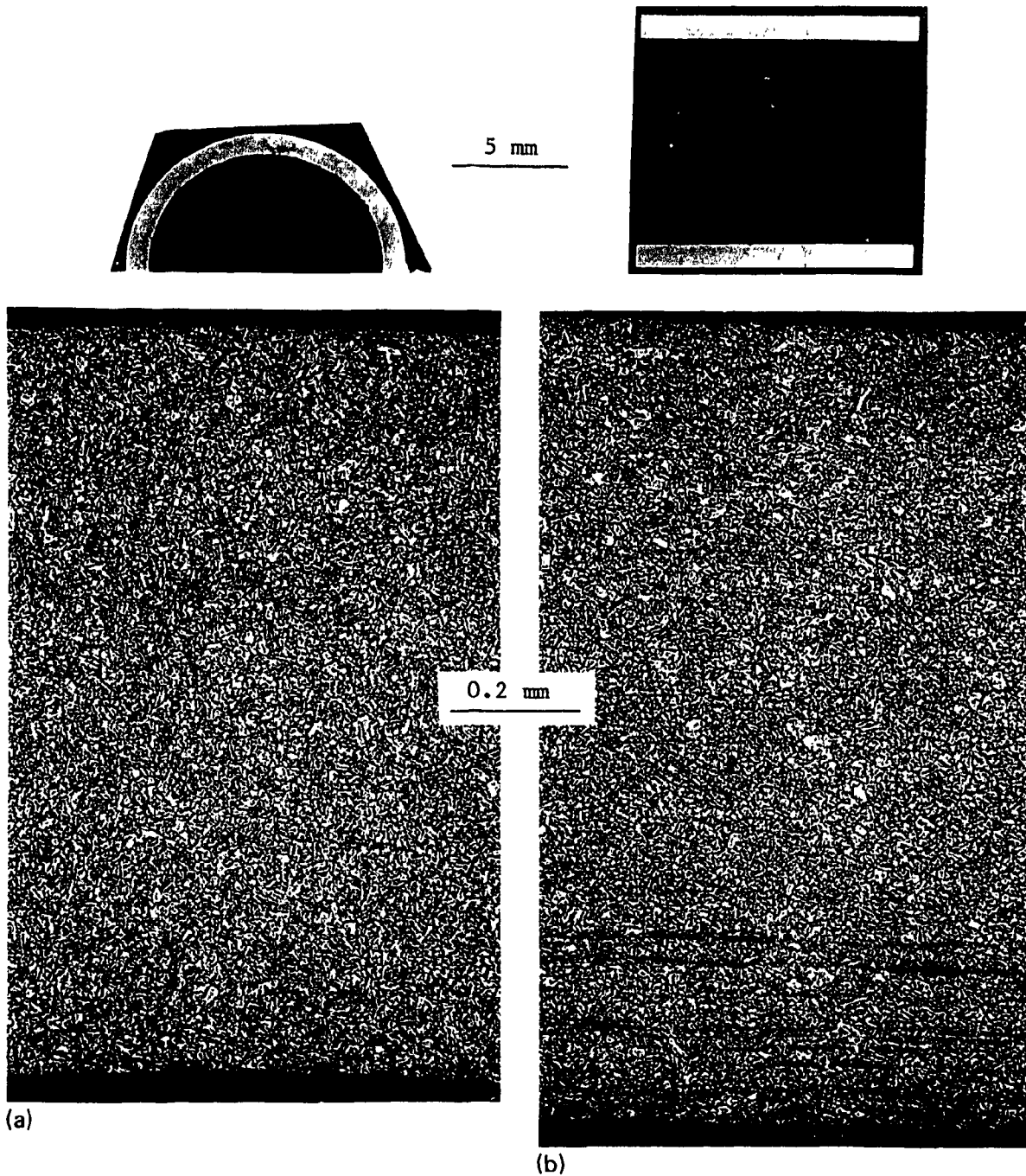


Fig. 16. Optical microstructure of the cold-reduced and cold-drawn tube ( $d/w = 12$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was normalized and tempered (1038°C for 10 min, 760°C for 10 min). (a) Circumferential. (b) Longitudinal.

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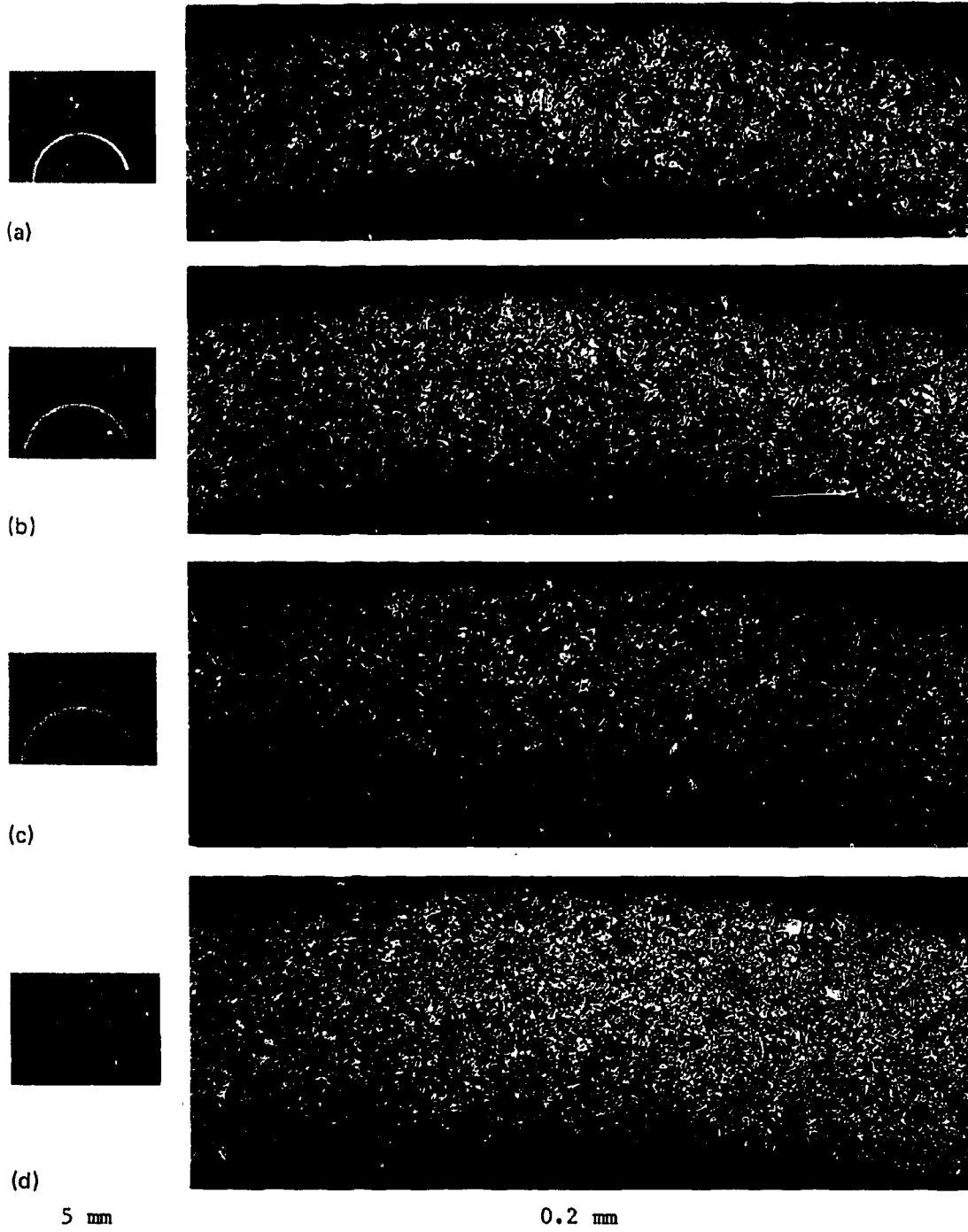


Fig. 17. Optical microstructure of the cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the circumferential direction ( $d/w = 15$  to  $18$ ). The tubes were normalized and tempered ( $1038^{\circ}\text{C}$  for 10 min,  $760^{\circ}\text{C}$  for 10 min). (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

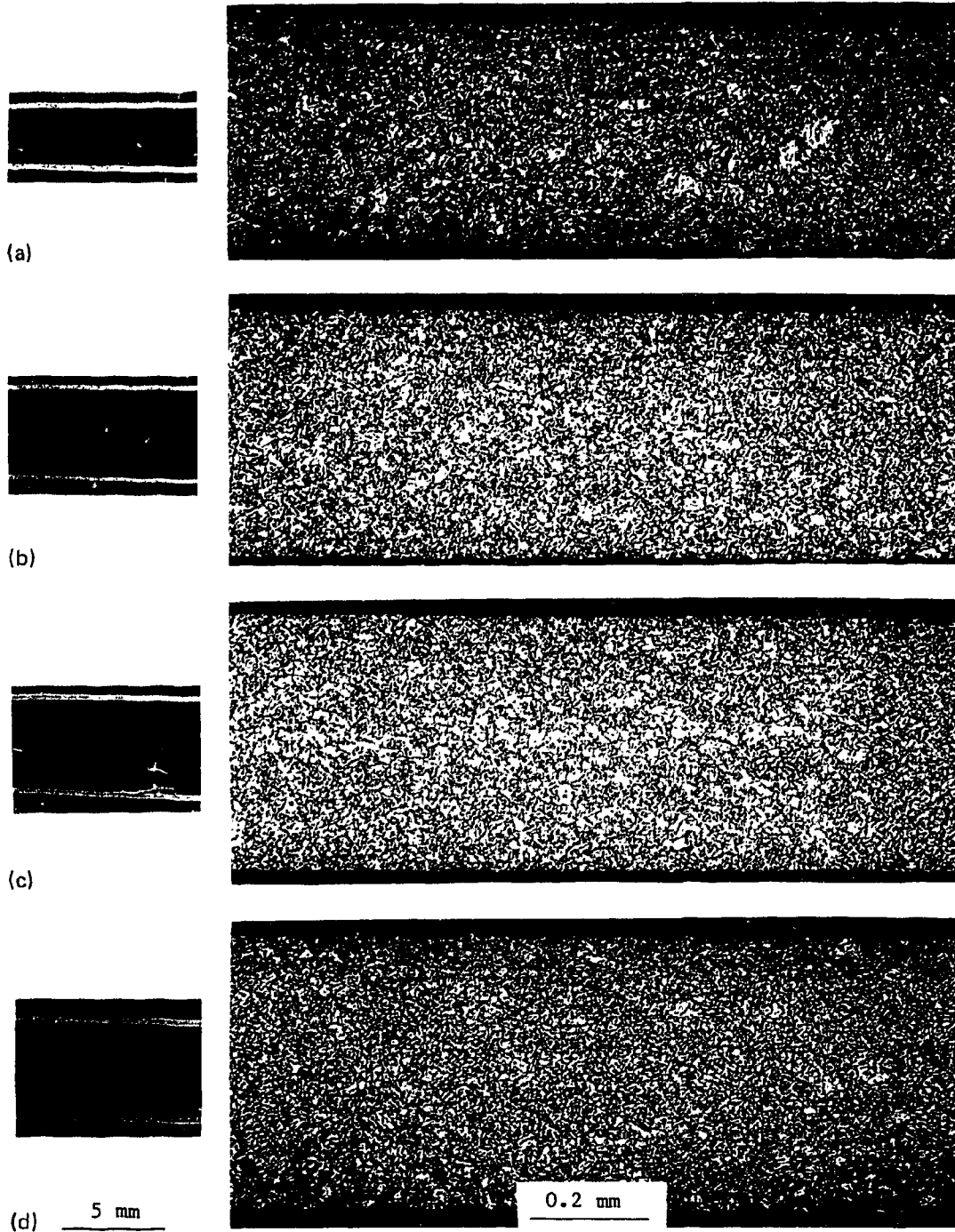


Fig. 18. Optical microstructure of the cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the longitudinal direction ( $d/w = 15$  to  $18$ ). The tubes were normalized and tempered (1038°C for 10 min, 760°C for 10 min). (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

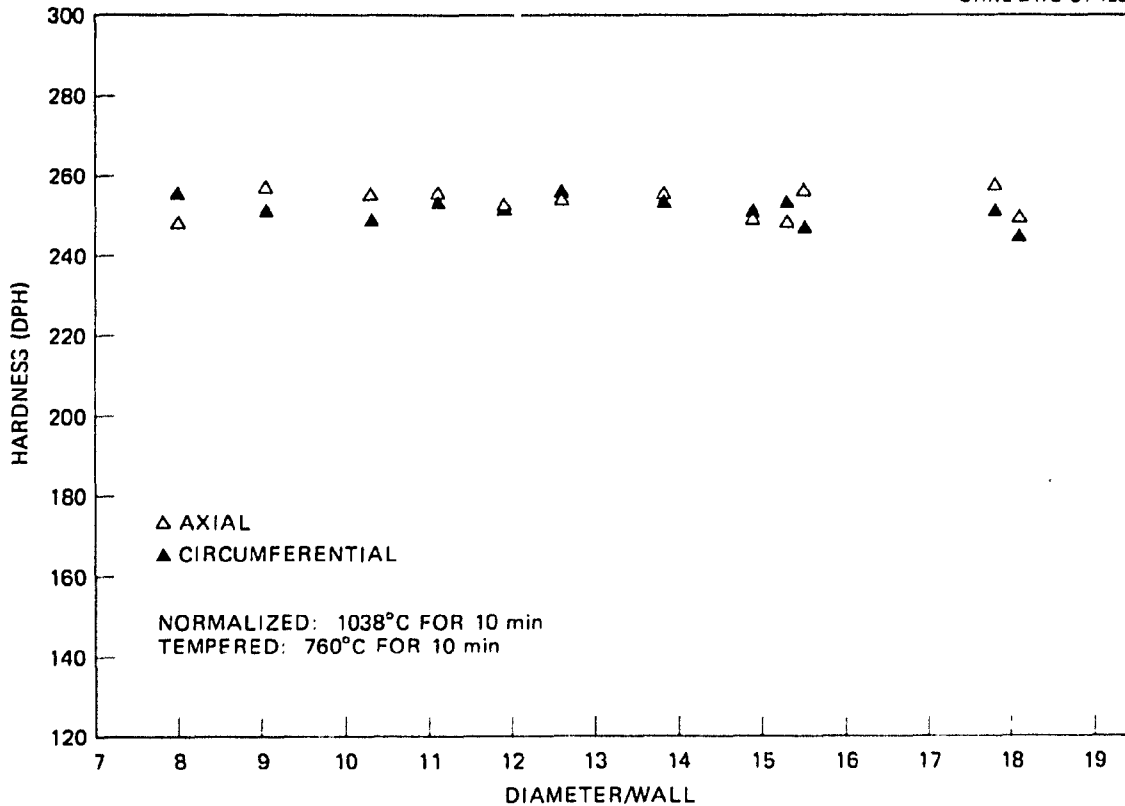


Fig. 19. Microhardness data as a function of diameter-to-wall ratio for various tubes of heat XA3628 of modified 9 Cr-1 Mo steel. The tubes were normalized and tempered (1038°C for 10 min, 760°C for 10 min).

These results suggest that there is no difference between the double and single normalize treatments, so the latter will be sufficient to produce equiaxed microstructure and the same hardness for all tubes.

The microstructures of tubes from the B3 heat treatment are shown in Figs. 20 through 24. The hardness data for this batch are plotted in Fig. 25. These figures show that the tempering treatment of 760°C for 10 min was not sufficient to produce a recrystallized equiaxed grain structure in both directions of various tubes. The microstructure of the starting tube was not altered by this treatment. A comparison of Figs. 7, 13, 19, and 25 shows that the tempering treatment of 10 min at 760°C reduced the hardness of all tubes. In fact, the hardness values now fall below that of the starting tube with the exception of the

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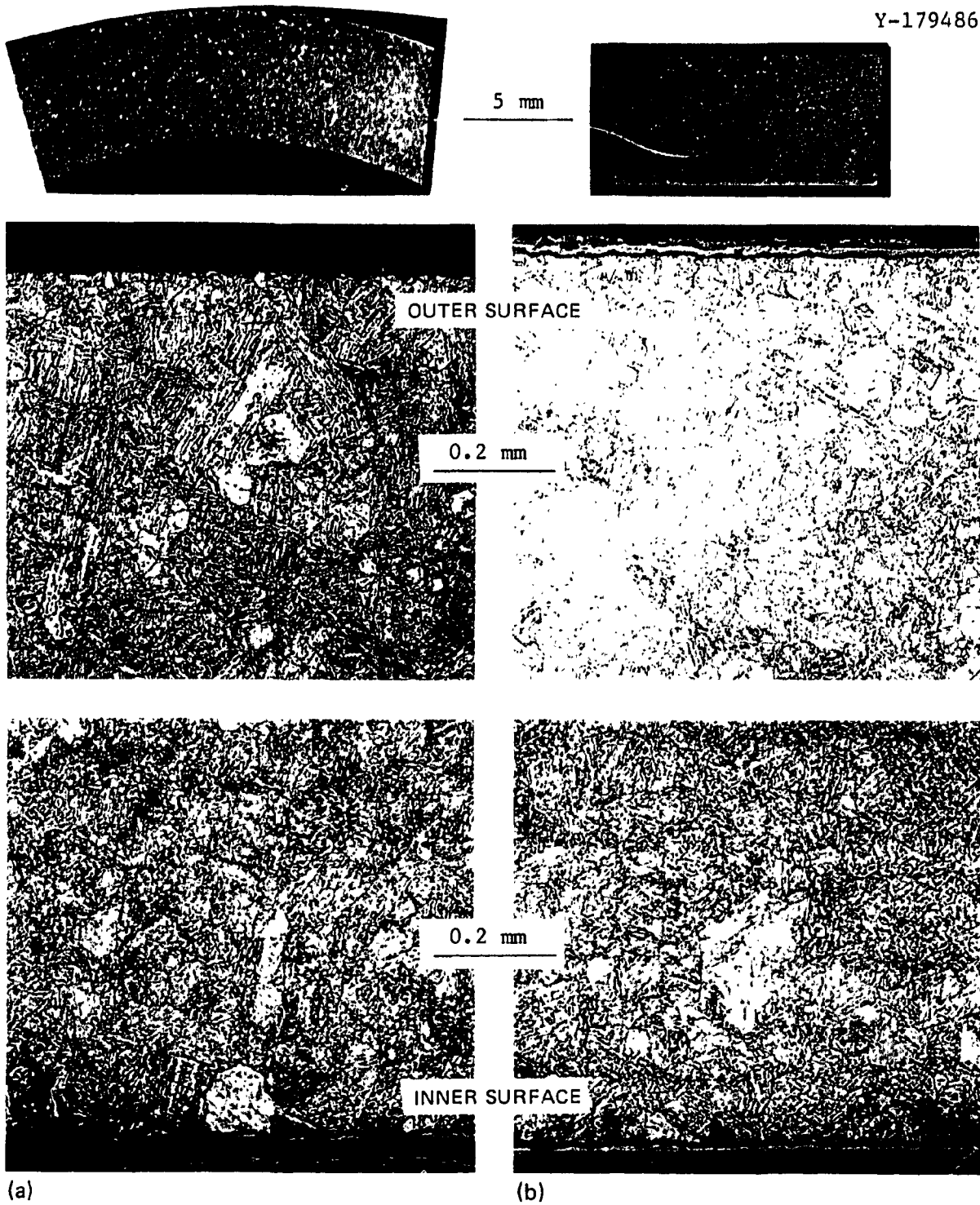


Fig. 20. Optical microstructure of the starting tube ( $d/w = 8$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was only tempered ( $760^{\circ}\text{C}$  for 10 min). (a) Circumferential. (b) Longitudinal.

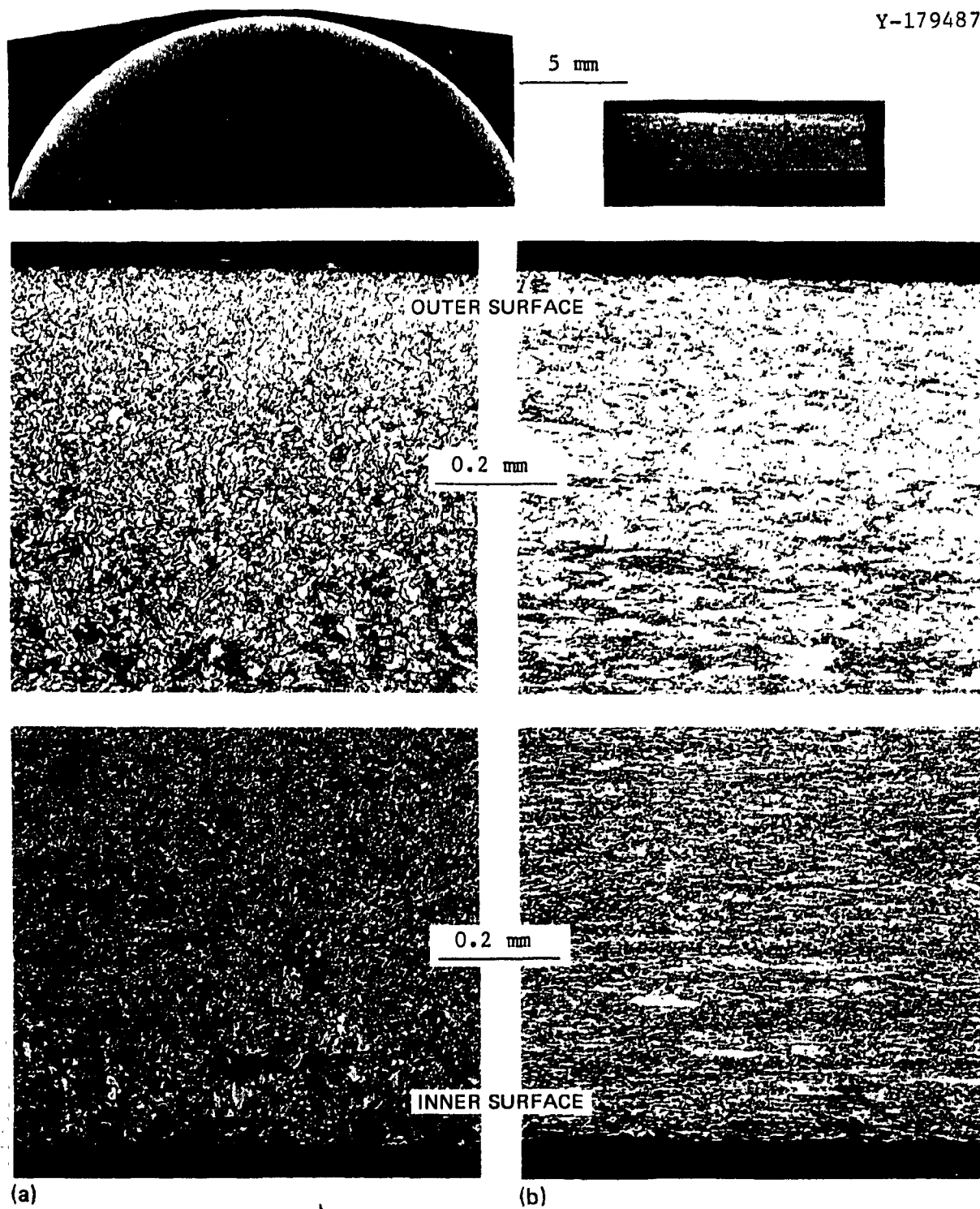


Fig. 21. Optical microstructure of the cold-reduced tube ( $d/w = 9$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was only tempered (760°C for 10 min). (a) Circumferential. (b) Longitudinal.

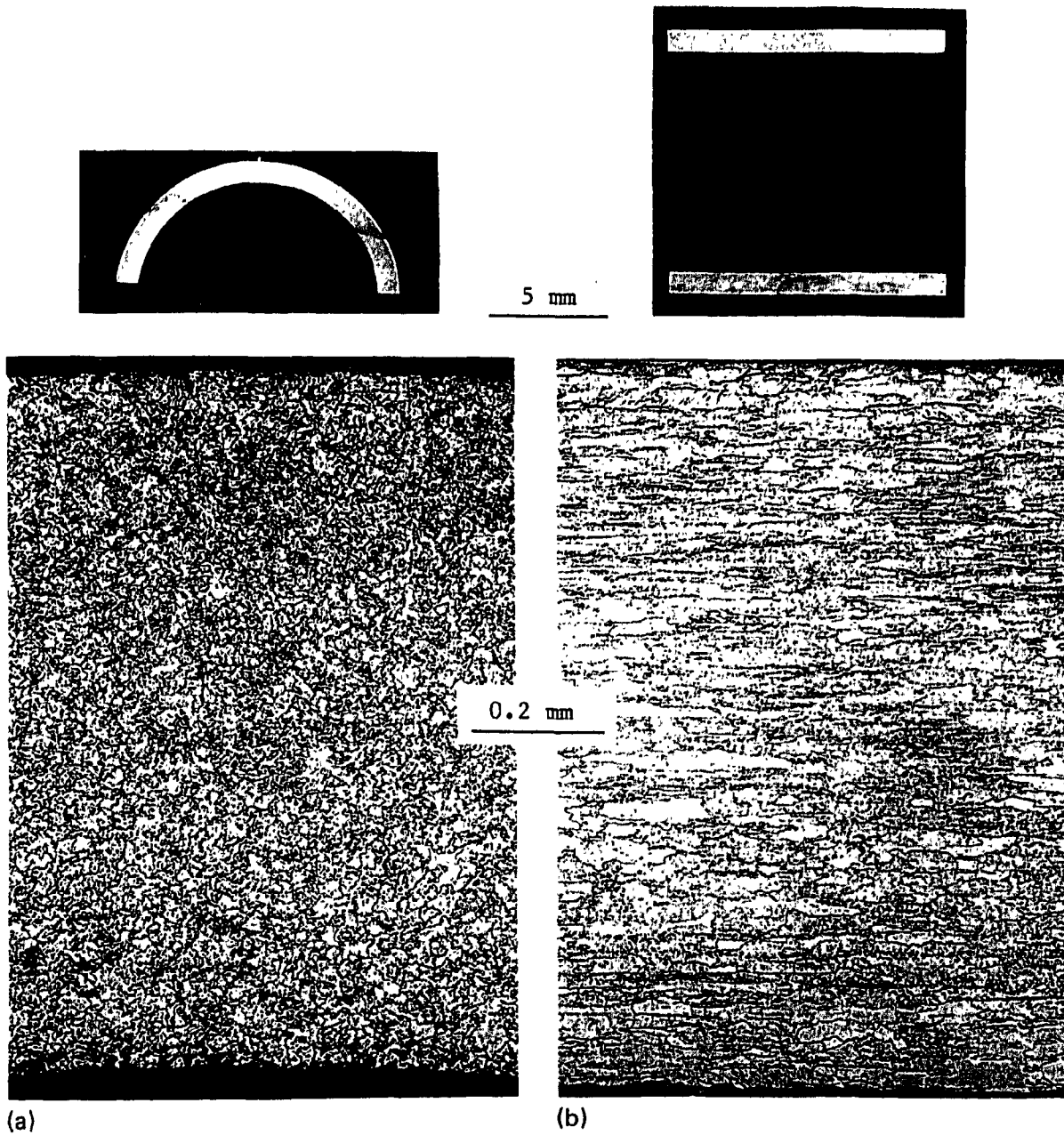


Fig. 22. Optical microstructure of the cold-reduced and cold-drawn tube ( $d/w = 12$ ) of heat XA3628 of modified 9 Cr-1 Mo steel. The tube was only tempered (760°C for 10 min). (a) Circumferential. (b) Longitudinal.

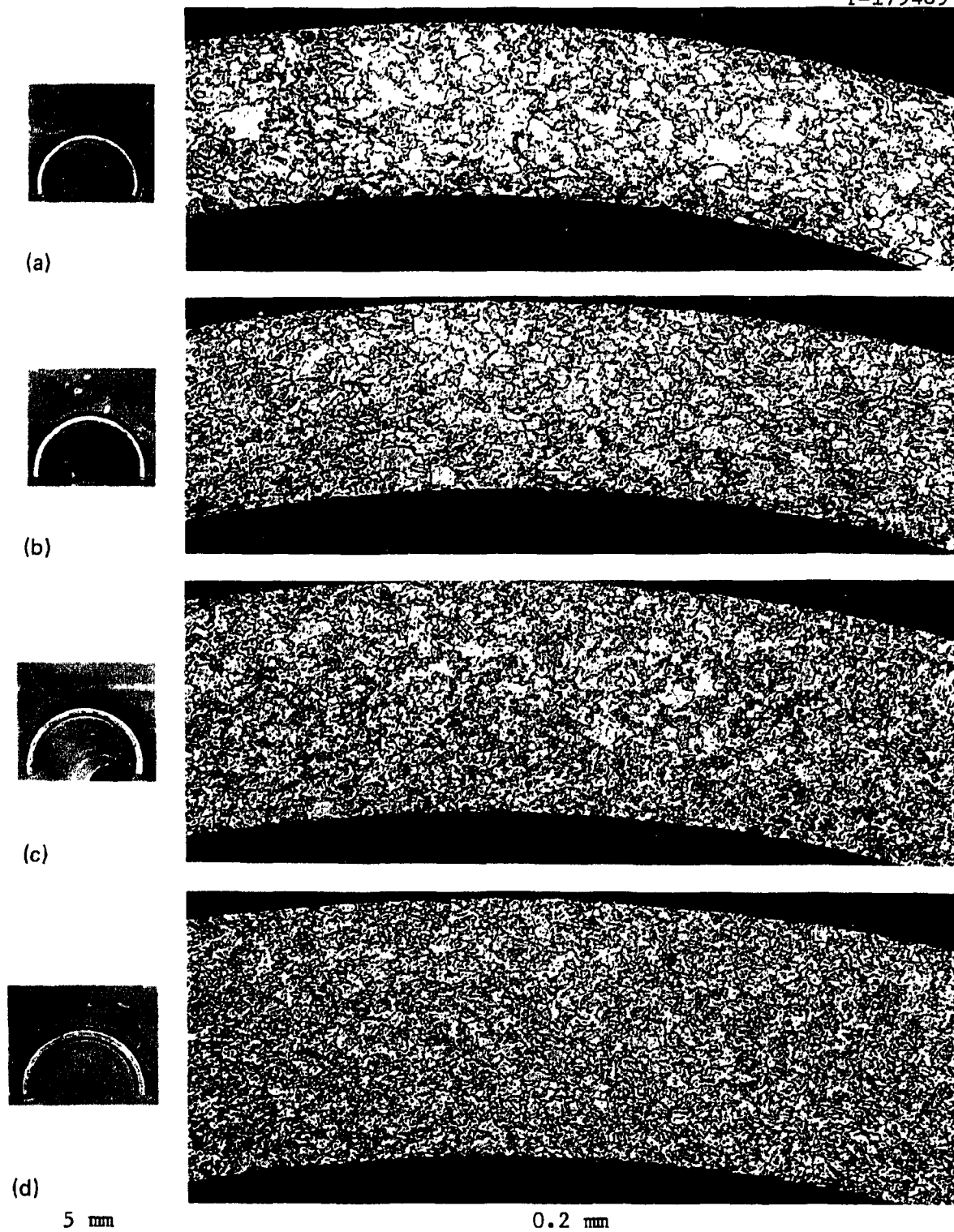


Fig. 23. Optical microstructure of the cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the circumferential direction ( $d/w = 15$  to  $18$ ). The tubes were only tempered ( $760^{\circ}\text{C}$  for  $10$  min). (a)  $4.75$  mm OD by  $0.267$  mm wall. (b)  $5.28$  mm OD by  $0.292$  mm wall. (c)  $5.51$  mm OD by  $0.356$  mm wall. (d)  $5.84$  mm OD by  $0.381$  mm wall.

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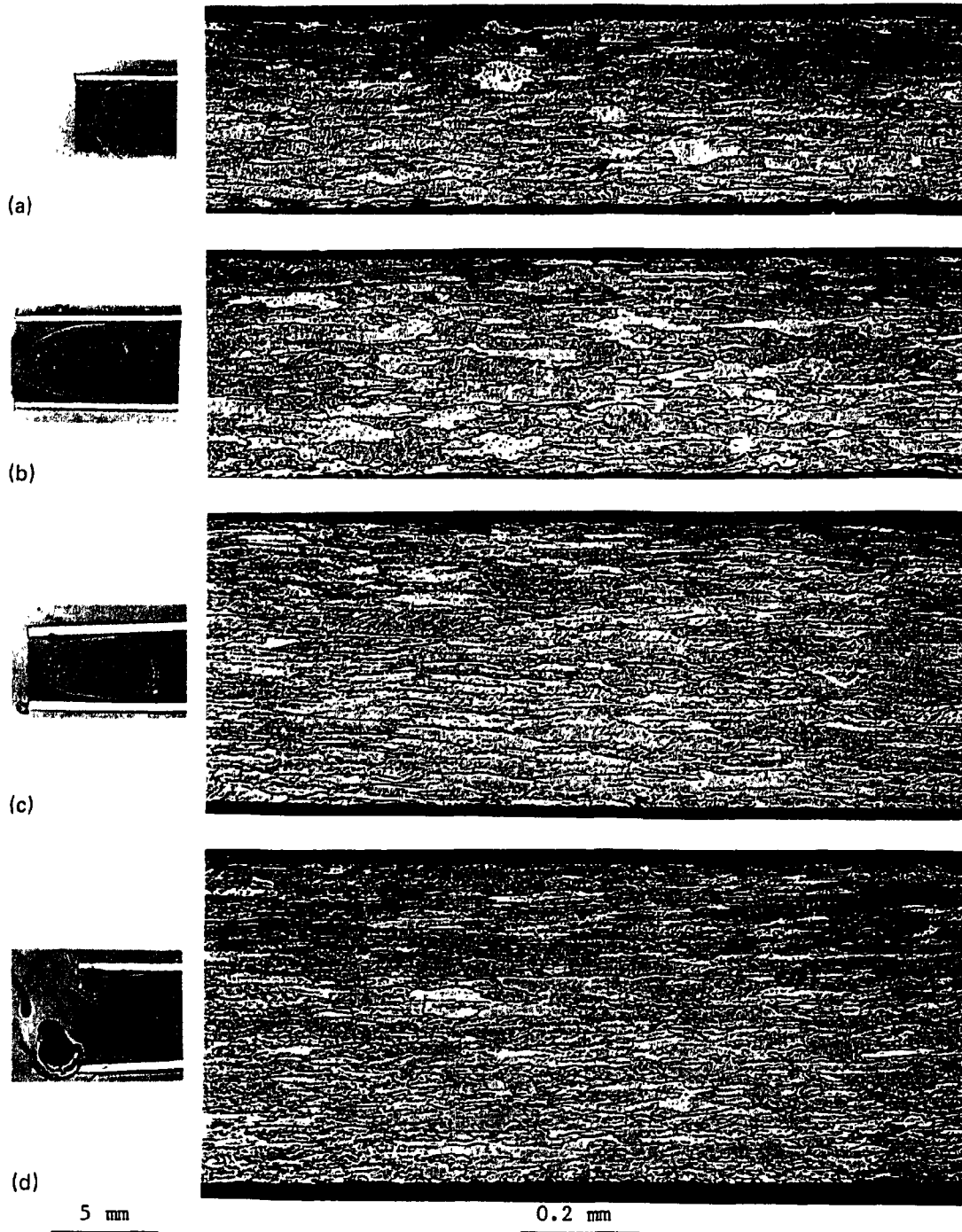


Fig. 24. Optical microstructure of the cold-reduced and cold-drawn tubes of heat XA3628 of modified 9 Cr-1 Mo steel in the longitudinal direction ( $d/w = 15$  to  $18$ ). The tubes were only tempered ( $760^{\circ}\text{C}$  for 10 min). (a) 4.75 mm OD by 0.267 mm wall. (b) 5.28 mm OD by 0.292 mm wall. (c) 5.51 mm OD by 0.356 mm wall. (d) 5.84 mm OD by 0.381 mm wall.

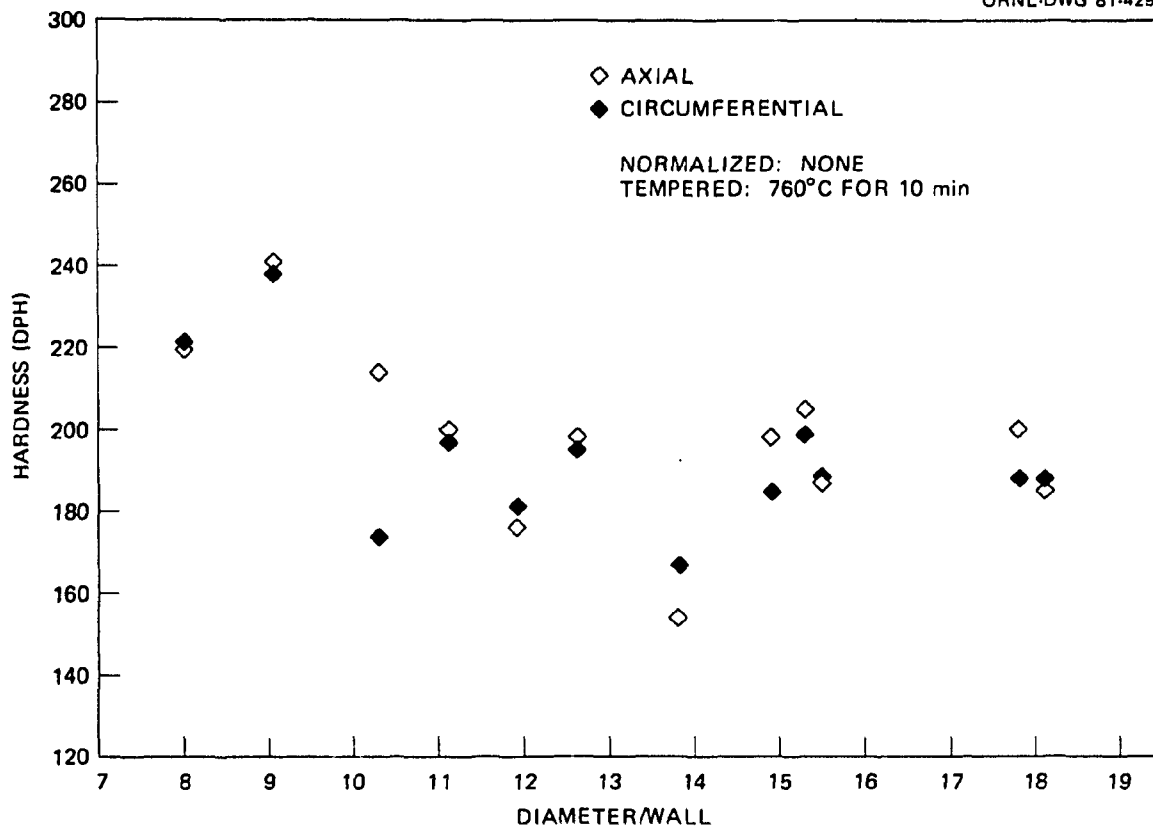


Fig. 25. Microhardness data as a function of diameter-to-wall ratio for various tubes of heat XA3628 of modified 9 Cr-1 Mo steel. The tubes were tempered (760°C for 10 min).

cold-reduced tube, which had seen the most work. The lower hardness after tempering is possibly caused by any or all of the processes:

1. recovery of cold work,
2. cleaning out of substructure<sup>1</sup> and its growth, assisted by cold work, and
3. growth of carbide particles.

In the case of the as-reduced tube (~78% work), the higher hardness after a 10-min temper at 760°C than the starting tube hardness is possibly a result of only partial recovery. Processes 2 and 3 suggested above were probably not operative.

The fabrication response of the commercial heat (30182) tubing to heat treatment was similar to that presented above for the centrifugally cast and cold-pilgered tube heat (XA3628).

## ULTRASONIC INSPECTION

Several samples of modified 9 Cr-1 Mo steel tubing were ultrasonically inspected to evaluate the material conditions and to determine if major discontinuities had developed during fabrication. The tubing was inspected after pass 10 in the fabrication cycle, see Table 2 and Fig. 1. At this stage the tubing had received approximately 97% accumulative reduction in size. The test samples discussed in this report were taken from materials heats XA3628 and 30182. The nominal diameter of the tubes inspected was 5.84 mm (0.230 in.) and their wall thickness was 0.38 mm (0.015 in.). All test samples were 0.9 m (35 in.) long.

The tests were conducted in a water-immersion setup using a Tektran model 725 Immerscope and a 12.7-mm-diam 10-MHz focused transducer. Sound energy was directed circumferentially around the tube wall as the transducer scanned along the axis of the rotating tube, thus producing a helical scanning motion, refer to Fig. 26. Output data were recorded on a Brush model 220 strip-chart recorder.

A type 316 stainless steel tube having similar dimensions [6.35 by 0.38 mm (0.250 by 0.015 in.)] and containing five well-defined electrical-discharge-machined (EDM) notches was used as the reference standard. This tube contains three notches on the inner surface and two on the outer surface. All notches are oriented in the longitudinal (axial) direction and are approximately 0.04 mm deep, 0.76 mm long, and 0.04 mm wide (0.0015 by 0.030 by 0.0015 in.). This sample was chosen because of its availability and because its dimensions matched those of the test samples. Also there is no significant difference (<5%) between the sound velocities in type 316 stainless steel and 9 Cr-1 Mo steel.

The test procedure was to calibrate the ultrasonic system using the reference tube, to inspect the test pieces, and then rerun the reference tube to make sure that no changes had occurred in the test system during the inspection. The inspection angle was determined experimentally to be the angle that produced optimum response for the five notches in the stainless steel reference tube.

Figure 27 shows the typical response obtained when the sound beam was scanned over the five notches in the type 316 stainless steel reference



sample. The outer and inner surfaces of the tube where the identified notches are located are indicated by OD and ID. The indications from inner surface notches have lower amplitudes because the sound energy is attenuated more as it travels through the tube wall. Notches 2 and 3 are located in the same plane along the axis of the tube. Notch 2 is located on the inner surface and notch 3 is located on the outer surface similar to the sample in Fig. 26. The difference in the sound paths required to detect these two notches is too small to be detected by the instrumentation used, therefore the two indications coincide on the recorder chart.

The first modified 9 Cr-1 Mo steel tube ultrasonically inspected was sample 3628, and the resulting data are shown in Fig. 28. The sample was in the as-fabricated condition, which results in an axially elongated grain structure (see Fig. 29). Many of these grains have lengths that are comparable to the wavelength of shear sound waves in steel, about 0.30 mm (0.012 in.). When the length of the reflecting surface of a grain approximates the wavelength of the impinging sound energy, the grain will act as a singular reflector. If a large number of grains have this length the result will be many reflections, resulting in the noise pattern shown in Fig. 28.

This sample was returned to the shop where it received a normalizing and tempering treatment, which resulted in a more nearly equiaxed grain structure (see Fig. 28). There is also evidence of decarburization in the sample. The sample was reinspected ultrasonically by the same techniques, and the results are shown in Fig. 31. The background noise, although not eliminated, has been significantly reduced. Referring to Fig. 30, we see that the larger grains in the decarburized area have dimensions of 0.03 to 0.04 mm (0.001 to 0.0015 in.), which are significantly smaller than the wavelength of the inspecting sound beam.

We also detected a crack in the outer surface of the tubing during this ultrasonic inspection; note the large indication in Fig. 31. It was confirmed visually later with an optical microscope (see Fig. 32). The crack is about 13 mm (0.5 in.) long and is oriented about 30° with respect to the tube axis. We are uncertain as to whether the crack was present on the first inspection and was masked by the noise signals or whether it developed during handling or normalization and tempering. The fact that

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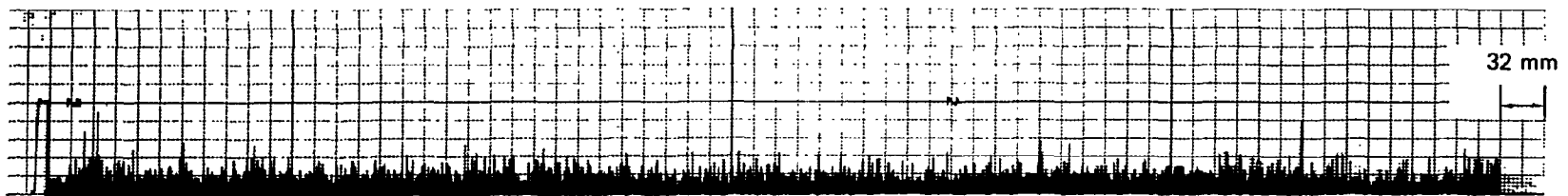


Fig. 28. Results of ultrasonic inspection of modified 9 Cr-1 Mo steel tube 3628 as fabricated.

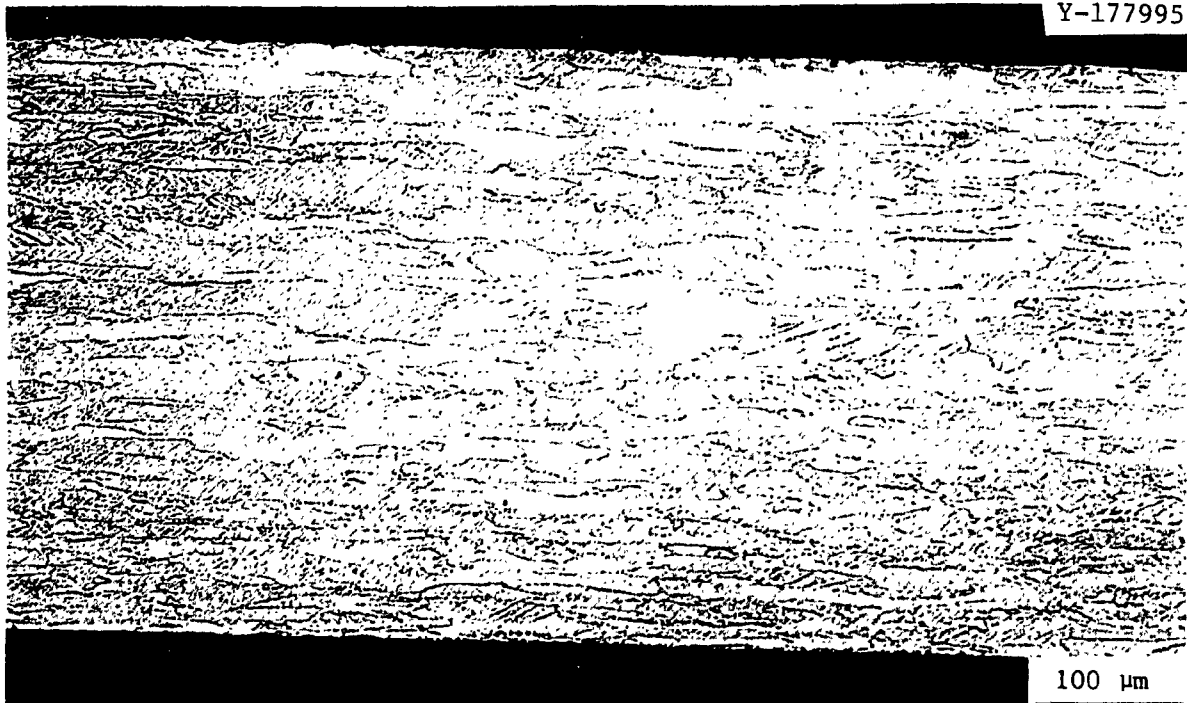


Fig. 29. Grain structure in the axial direction for as-fabricated modified 9 Cr-1 Mo steel tube sample 3628.

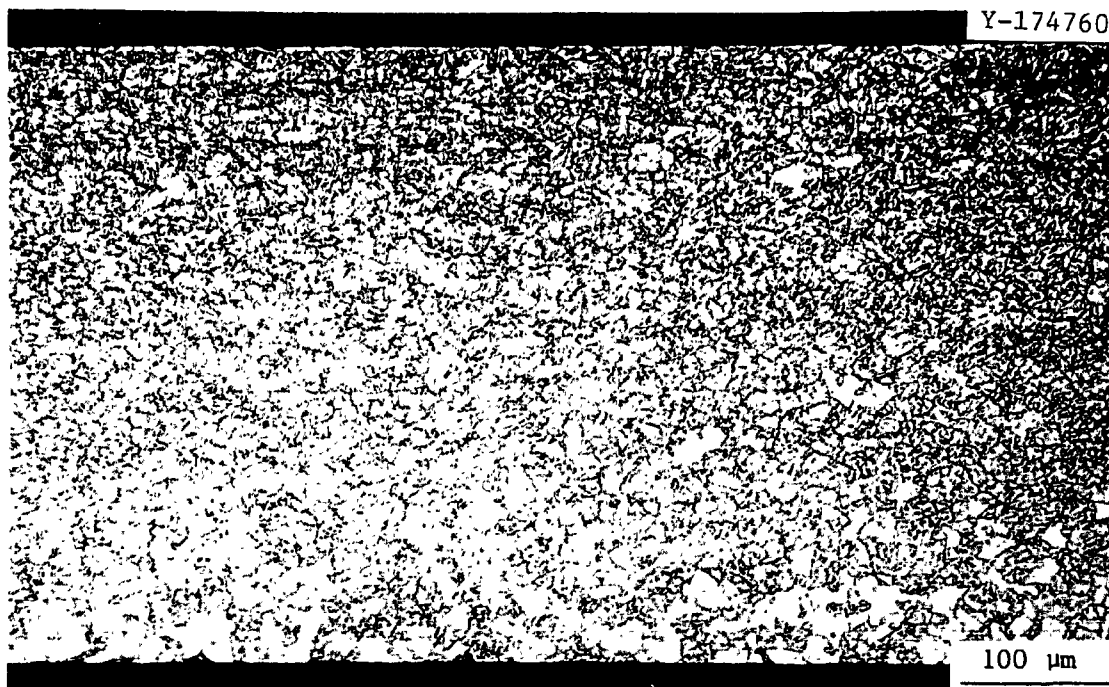


Fig. 30. Grain structure in the axial direction for modified 9 Cr-1 Mo steel tube sample 3628 after normalizing and tempering.

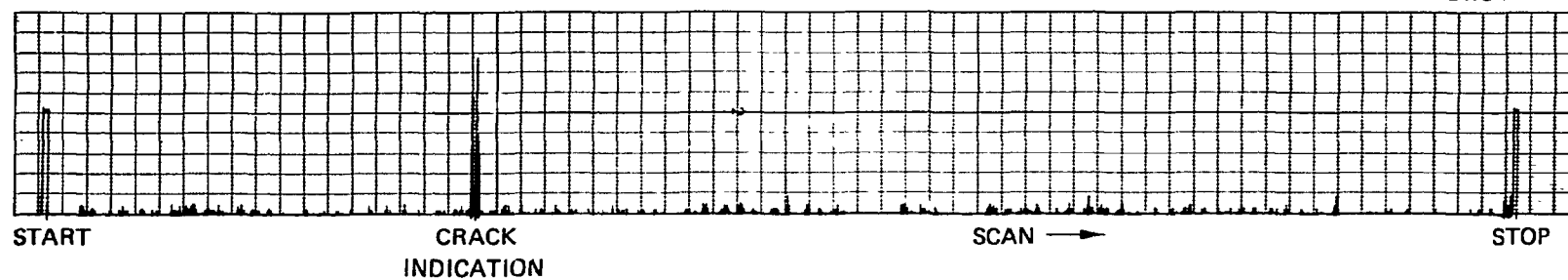


Fig. 31. Results of ultrasonic inspection of modified 9 Cr-1 Mo steel tube 3628 after normalizing and tempering.

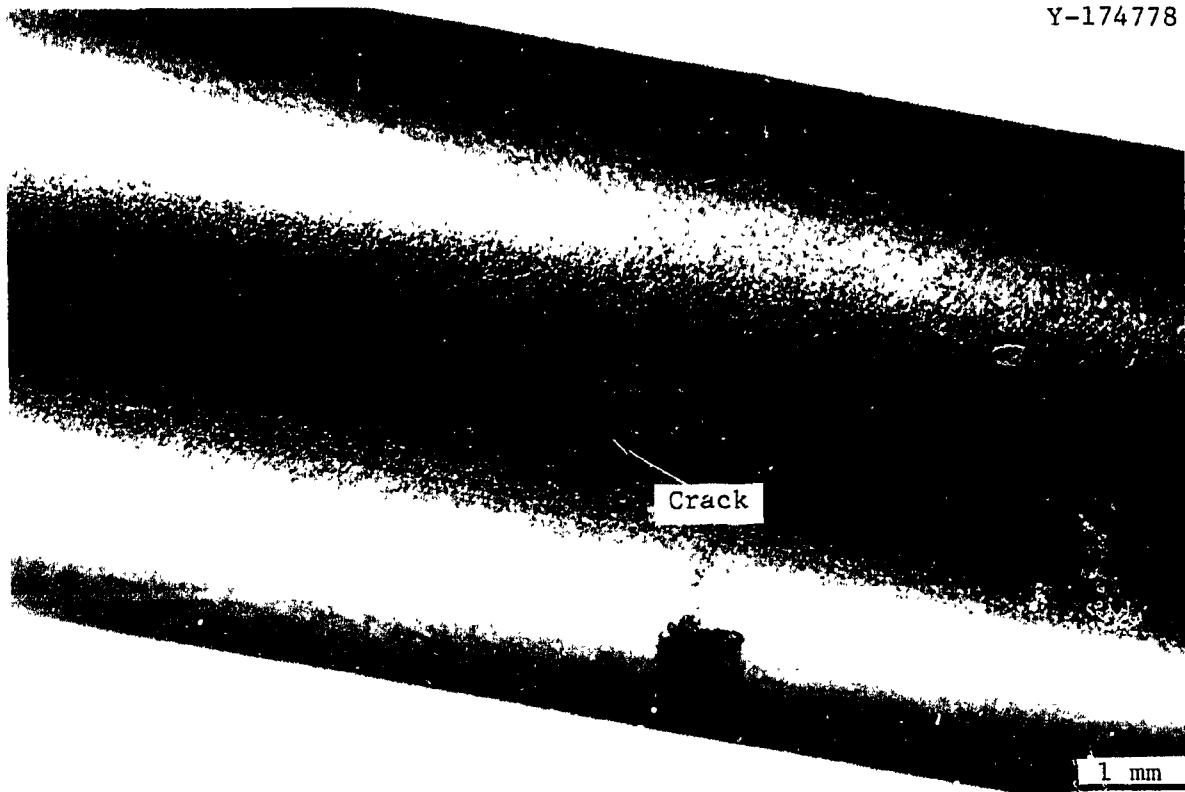


Fig. 32. Surface view of crack detected in modified 9 Cr-1 Mo steel tube sample during ultrasonic inspection.

the crack was detected indicates a good test sensitivity, and it verifies that the use of the stainless steel tube for a reference sample was adequate to attain this sensitivity.

Two additional 9 Cr-1 Mo tubes were inspected. Sample 30182 was fabricated similarly to sample 3628 except that an in-process anneal (1038°C for 10 min and 800°C for 10 min) was added after pass 10. The sample was then cut in half, and the resulting two tubes were labeled A and B. Sample A received no further treatment, but sample B was normalized and tempered similarly to sample 3628 for comparison. The two samples were ultrasonically inspected, and the results are shown in Fig. 33. No defect indications or noise signals were detected in either sample. The grain structures of samples A and B are shown in Fig. 34.

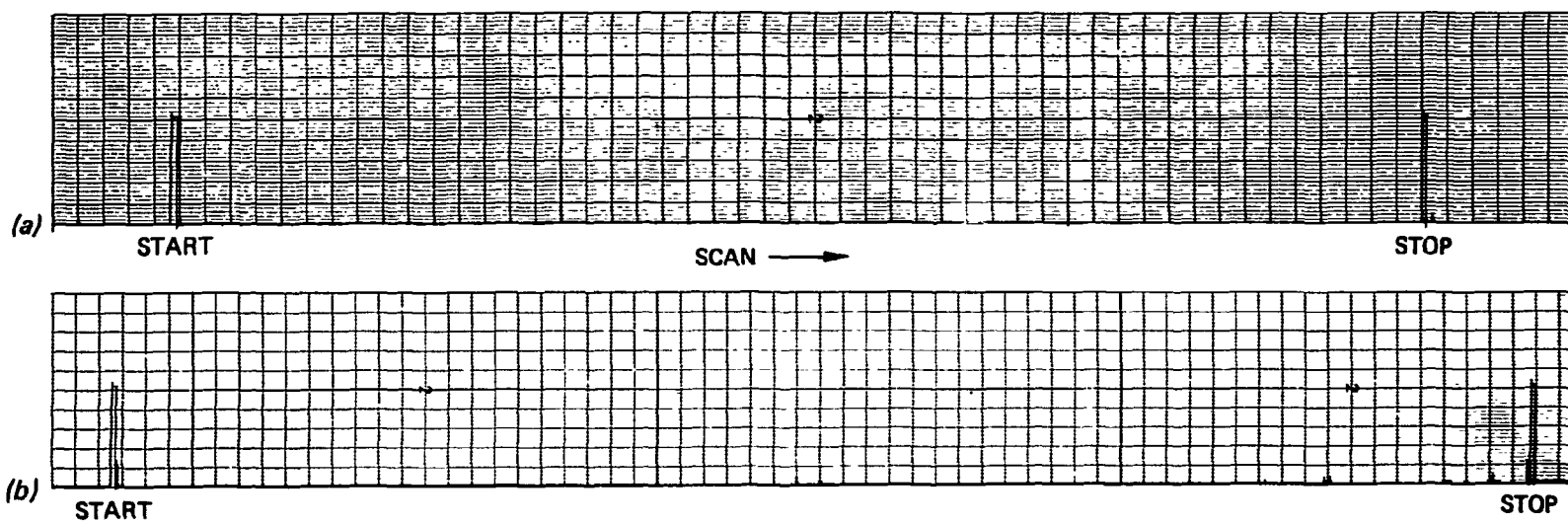


Fig. 33. Results of ultrasonic inspections on modified 9 Cr-1 Mo steel tube sample 30182. (a) Sample A (as fabricated plus anneal). (b) Sample B (normalized and tempered).

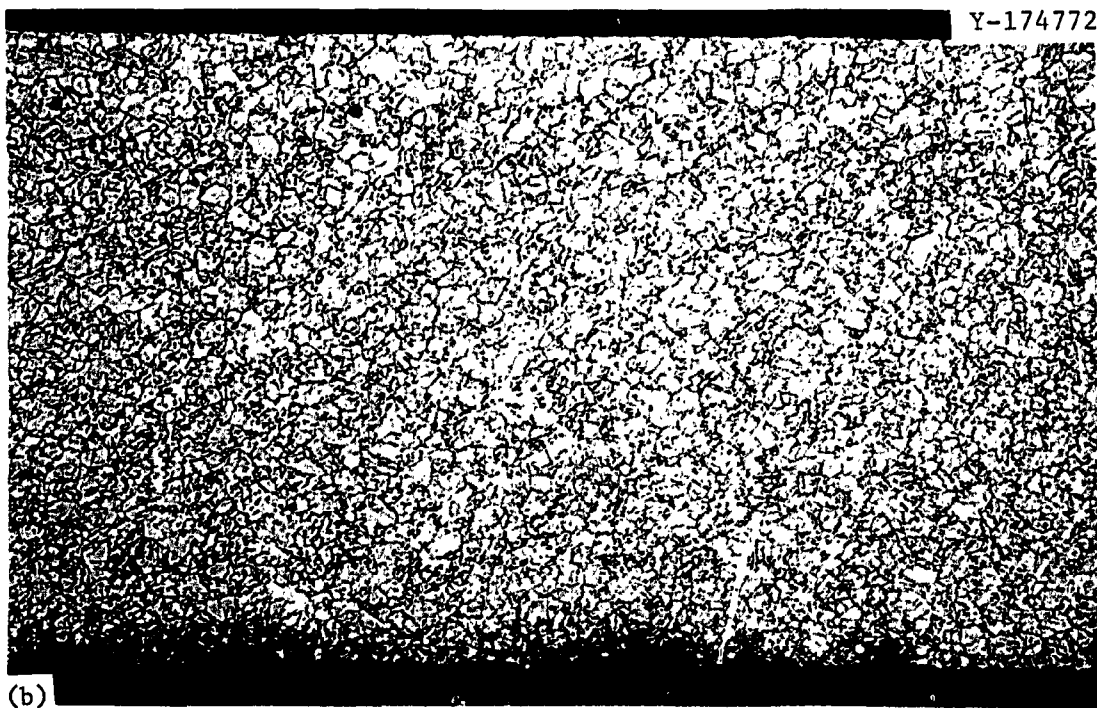


Fig. 34. Grain structure in the axial direction modified 9 Cr-1 Mo steel tube sample 30182. (a) Sample A (as fabricated plus anneal). (b) Sample B (normalized and tempered).

Some grain directionality is obvious in sample A, but the largest grain dimensions are still 0.03 to 0.05 mm (0.001 to 0.002 in.), which is apparently small enough to avoid significant background noise.

#### SUMMARY AND CONCLUSIONS

Tube shells from two different heats of modified 9 Cr-1 Mo steel were cold reduced and cold drawn to many different sizes. The cold-reducing step introduced approximately 78% cold reduction without any indication of cracking or tearing. The cold-reduced tube was subsequently given a 15-min treatment at 800°C followed by a schedule of 40% reductions between anneals to make a variety of sizes. Sections of these tubes were subjected to three different heat treatments; B1, normalize at 1149°C for 10 min and 1038°C for 10 min and temper at 760°C for 10 min; B2, normalize at 1038°C for 10 min and temper at 760°C for 10 min; and B3, temper at 760°C for 10 min. As-fabricated and heat-treated tube sections were microstructurally examined and hardness was measured. Pieces of the cladding tube size (5.84 mm OD by 0.38 mm wall) were ultrasonically inspected in both as fabricated and normalized and tempered (1038°C for 10 min and 760°C for 10 min).

The following conclusions can be drawn from this work:

1. The modified 9 Cr-1 Mo steel tube shells can be successfully cold reduced up to 78% in one step. This was accomplished for tubes centrifugally cast and cold pilgered from an air-melted stock and for a hot-extruded tube from an AOD-ESR stock.

2. The cold-reduced tubes could be cold drawn by use of a 40% schedule following 800°C heat treatment for 15 min at each step.

3. A complete normalize and temper is required to produce equiaxed grain structure in both circumferential and axial directions. This heat treatment also produces the same hardness for all tube sizes. The normalizing and tempering treatment used in this study was 1038°C for 10 min and 760°C for 10 min. The hardness produced by this treatment was about 40 DPH higher than that of the starting material. To obtain the desired hardness level, a correlation between hardness and tempering time is needed. This work is currently under way.

4. Ultrasonic inspection of the cladding-size tubes showed that they should be normalized and tempered to reduce grain size and its concomitant background noise to render the tubes inspectable by this method.

5. According to the ultrasonic data obtained, the in-process anneal used for sample 30182A appeared to be sufficient to reduce the ultrasonic background noise. But only one sample was examined, and its photomicrographs still showed grain directionality. We do not assume that the in-process anneal alone would be sufficient to eliminate background noise during ultrasonic inspection until more samples are examined.

6. The procedure developed in this study is useful to produce steam generator or cladding tubes of modified 9 Cr-1 Mo steel.

#### ACKNOWLEDGMENTS

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#### REFERENCE

1. P. S. Sklad and V. K. Sikka, "Microstructural Observations of Strain Softening in Ferritic 9 Cr-1 Mo Alloys," paper presented at ALME meeting, Chicago, Feb. 22-26, 1981.