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The Importance of Billet Microstructure in the Hot Extrusion of 2% Ti – Hastelloy N

D. N. Braski
R. E. McDonald

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THE IMPORTANCE OF BILLET MICROSTRUCTURE IN THE HOT EXTRUSION OF 2% Ti—HASTELLOY N

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THE IMPORTANCE OF BILLET MICROSTRUCTURE IN THE HOT EXTRUSION OF 2% Ti—HASTELLOY N

D. N. Braski and R. E. McDonald

ABSTRACT

The successful hot extrusion of billets of Ti-Hastelloy N (13% Mo, 7% Cr, 2% Ti, 0.06% C, 87.94% Ni) into tube shells and followed by cold drawing into 19-mm-OD (0.75-in.) tubing depends largely on the billet microstructure. It was imperative that the preheat annealing treatment given directly before hot extrusion be designed to uniformly dissolve most of the MC-type carbide particles. A 101.6-mm-diam × 304.8-mm-long (4 × 12-in.) billet required a preheat of 4 hr at 1533 K.

1. INTRODUCTION

Hastelloy N is a nickel-base alloy developed at this laboratory during the 1950s and 1960s for use as the primary containment material for a Molten Salt Reactor (MSR). More recently, the alloy was modified with a 2 wt % Ti addition (13% Mo, 7% Cr, 2% Ti, 0.06% C) and this new alloy showed improved resistance to radiation.[1] Development of 2% Ti—Hastelloy N required specimens of the alloy in the form of tubing. One method used to produce tubing was the hot extrusion of 101.6-mm-diam × 304.8-mm-long (4 × 12 in.) billets into tube shells that were then reduced to 19-mm-OD × 16.5-mm (0.750 × 0.65-in.) thick wall tubing by cold drawing.[2] During the initial fabrication runs, problems developed in both the extrusion and cold drawing operations. These problems were studied with modern microstructural analysis techniques including optical metallography, transmission electron microscopy, electrochemical precipitate extractions, and x-ray diffraction. The results of the investigation are included herein.

2. EXPERIMENTAL PROCEDURE

2.1 Hot Extrusion

The 2% Ti—Hastelloy N billets were heated to about 383 K; and then sprayed with a glass lubricant consisting of a mixture of 200 cm^3 7570 Corning glass, 200 cm^3 sodium tetraborate, 1 g sodium hydroxide, 2 g cellulose gum, and enough water to produce a slurry. Some billets were plasma-sprayed with molybdenum as a lubricant, and in this case, the hole through the billet was covered with molybdenum foil. The billets were preheated, before extrusion, by placing them vertically in a Lindberg furnace with a flowing argon atmosphere. Preheat temperature was measured by placing a Chromel-P—Alumel thermocouple between the billet and a steel hearth plate and measuring the thermocouple output with a Rubican potentiometer. The billets were hot-extruded in a 11.5 MN (1300-ton) Watson-Stillman horizontal extrusion press (Fig. 1).

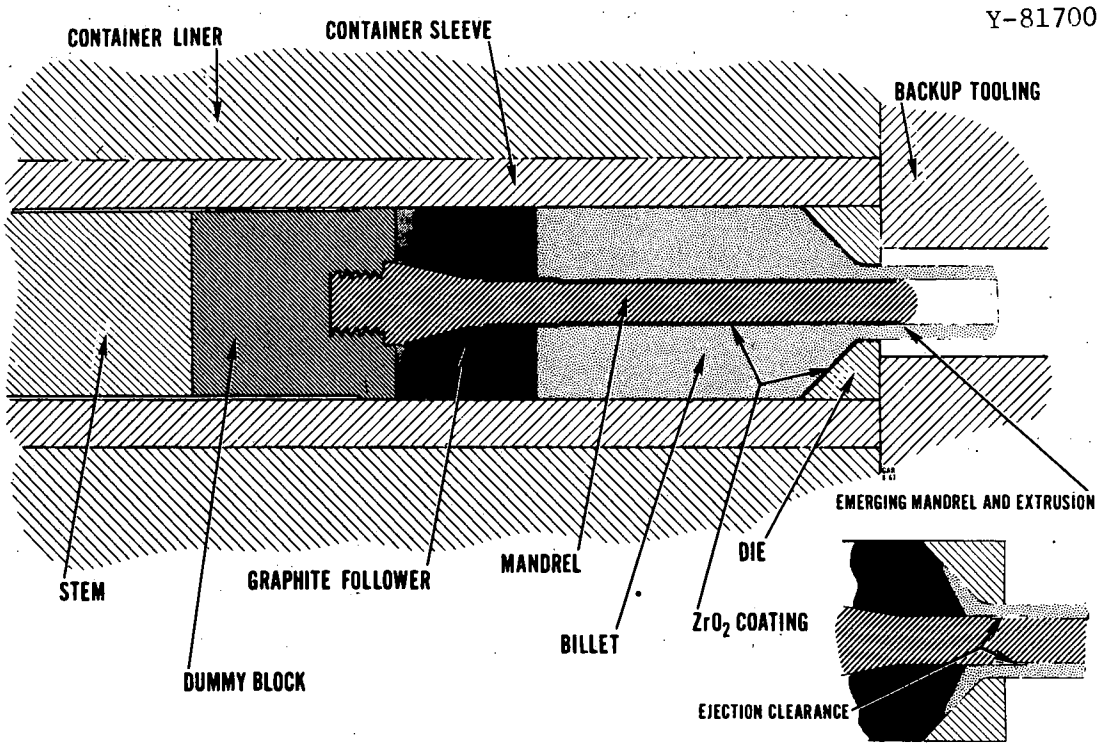


Fig. 1. A 1300-ton Horizontal Extrusion Press Set Up for Extrusion of tube shells.

2.2 Metallography

Samples were cut on a water-cooled, high-speed, abrasive cut-off wheel and mounted in epoxy (Araldite). A small wire was spot-welded to the back of the sample to permit electrolytic etching. The samples were polished in vibratory units by alumina and diamond polishing compounds respectively. Samples were electrolytically etched for about 20 sec in a solution of 10 g oxalic acid in 100 g of water at room temperature, with a potential of 2 V dc.

2.3 Transmission Electron Microscopy

Material for Transmission Electron Microscopy was cut in 0.38-mm (0.015-in.) thick sheets using a water-cooled, high-speed abrasive cut-off sheel. Disks, 3 mm in diameter were then punched from the sheet with a steel punch and die. The disks were dimpled by jet polishing[3] to a thickness of about 0.076 mm (0.003 in.) in a solution of 66 cm^3 HNO_3:133 cm^3 methyl alcohol at room temperature. A potential of 200 V dc was used in jet-polishing. Finally the disks were electrochemically polished in a solution of 66 cm^3 HNO_3:133 cm^3 methyl alcohol: 10 cm^3 H_3PO_4 at 253.15 K until perforation occurred. A potential of about 50 V dc was used in final polishing.

2.4 Carbide Extractions

Carbide particles were extracted from samples by electrochemically dissolving the matrix material in a solution of 10 cm^3 HCL: 90 cm^3 ethyl alcohol at room temperature. A potential of 3.5 V dc was applied across the sample (anode) and a platinum cathode. The carbides were separated from the solution by centrifuging; then they were dried for at least 30 min. in a vacuum desiccator. The dried carbides were finally weighed with a precision of at least 0.06 mg.

2.5 X-Ray Analysis

The extracted carbides were analyzed by x-ray diffraction by first combining them with a drop of ethyl alcohol and depositing the slurry on a polished single crystal substrate of silicon. The silicon crystal was cut so that the surface normal was about 7° off the (111) pole and thus provided a low background substrate for x-ray diffraction analysis. The x-ray diffraction data were taken using a diffracted beam, graphite monochromator and collected on paper tapes via a teletype readout. The scan speed was 1/8°, 2θ/min. The information on the paper tapes was subsequently recorded on computer cards and the x-ray profiles plotted using a Calcomp plotter. This technique has been shown to provide excellent sensitivity when analyzing small amounts of material (1 mg or less) such as the carbides.[4]

3. RESULTS AND DISCUSSION

3.1 Importance of Carbides and Preheat Treatment to Extrusion

In the initial development period, 2% Ti—Hastelloy N billets 1603 through 1611 were extruded into tube shells according to varying parameters (Table 1.) During this period, the effects of varying the reduction ratios and extrusion speeds were investigated. Little attention was devoted to the preheat treatment and standard times and temperatures used in hot working Hastelloy N were used. At that time, heating of the billet was viewed simply as a means of lowering the yield strength of the material to permit its extrusion. Although reasonable yields of tube shell were obtained in these early runs, the breakthrough loads were very high and were nearly always close to the maximum limit of the 11.5 MN (1300-ton) extrusion press. Consequently, it was difficult to maintain adequate control over the process. In addition, the surface finish of the extruded tube shell was quite poor and a better lubricant was needed. Prior experience indicated that molybdenum would be an excellent lubricant.[5]

Table 1. Extrusion Parameters

Billet	Lubricant	Preheat Time (min)	Preheat Temperature (K)	Mandrel Size (mm)	Die Size (mm)	Reduction Ratio	Breakthrough Load (MN)	Extruded Shell Speed (mm/min)	Yield Length of Tube Shell (mm)
1603	Glass	15	1450	20.625	37.465	10.45:1	10.942626	508/762	762
1604	Glass	15	1450	25.4	41.275	9.45:1	10.3	508/762	1752.6
1605	Glass	30	1450	20.625	39.465	10.45:1	11.4	508/762	2082.8
1606	Glass	15	1450	25.4	41.275	9.45:1	14.9	508/762	25.4
1607	Glass	15	1450	25.4	41.275	9.45:1	11.4	1397	1752.6
1608	Glass	15	1450	25.4	41.275	9.45:1	14.9	1143	1776.0
1611	Glass	15	1523	25.4	41.275	9.45:1	88.9	1270	1397.0
1614	Mo	130	1473	25.4	41.275	9.45:1	14.9	1270	0
1644	Mo	75	1533	25.4	41.275	9.45:1	11.7	1270	0
1645	Mo	270	1533	25.4	41.275	9.45:1	5.3	5080	1549.4

Unfortunately, the first billet (1614) processed using molybdenum as a lubricant failed to extrude, even after a 130 min at 1473 K preheat and a 11.5 MN (1300-ton) load. Metallographic examination of the billet nose showed that it consisted of irregularly shaped grains of γ solid solution with numerous carbide stringers [Fig. 2(a)]. The distribution of carbides was inhomogeneous and a smaller grain size was observed in areas of high carbide concentration. This microstructure is typical of hot-worked 2% Ti—Hastelloy N and the effect of carbide particles in retarding grain growth has been noted previously by others.[6,7] The tail portion of the billet contained fewer carbides and the grains were more equiaxed [Fig. 2(b)]. Most of the carbides in the tail portion were again in a stringer morphology. The amount of carbides in both the nose and tail sections of 1614 were determined quantitatively by electrochemical extraction. The nose section contained 0.45 wt % carbides while the tail portion contained only 0.22 wt %. X-ray diffraction analysis showed that the carbides in both areas were of the fcc, MC-type. The microstructure in the nose section of 1614 [Fig. 3(a)] consists of many subgrains of γ with a relatively high dislocation density. Only a few MC particles were visible in this particular area, but were generally difficult to discern anyway, because of the large number of dislocations. A high dislocation density might be expected in this area because a small portion of the nose of the billet was actually extruded and

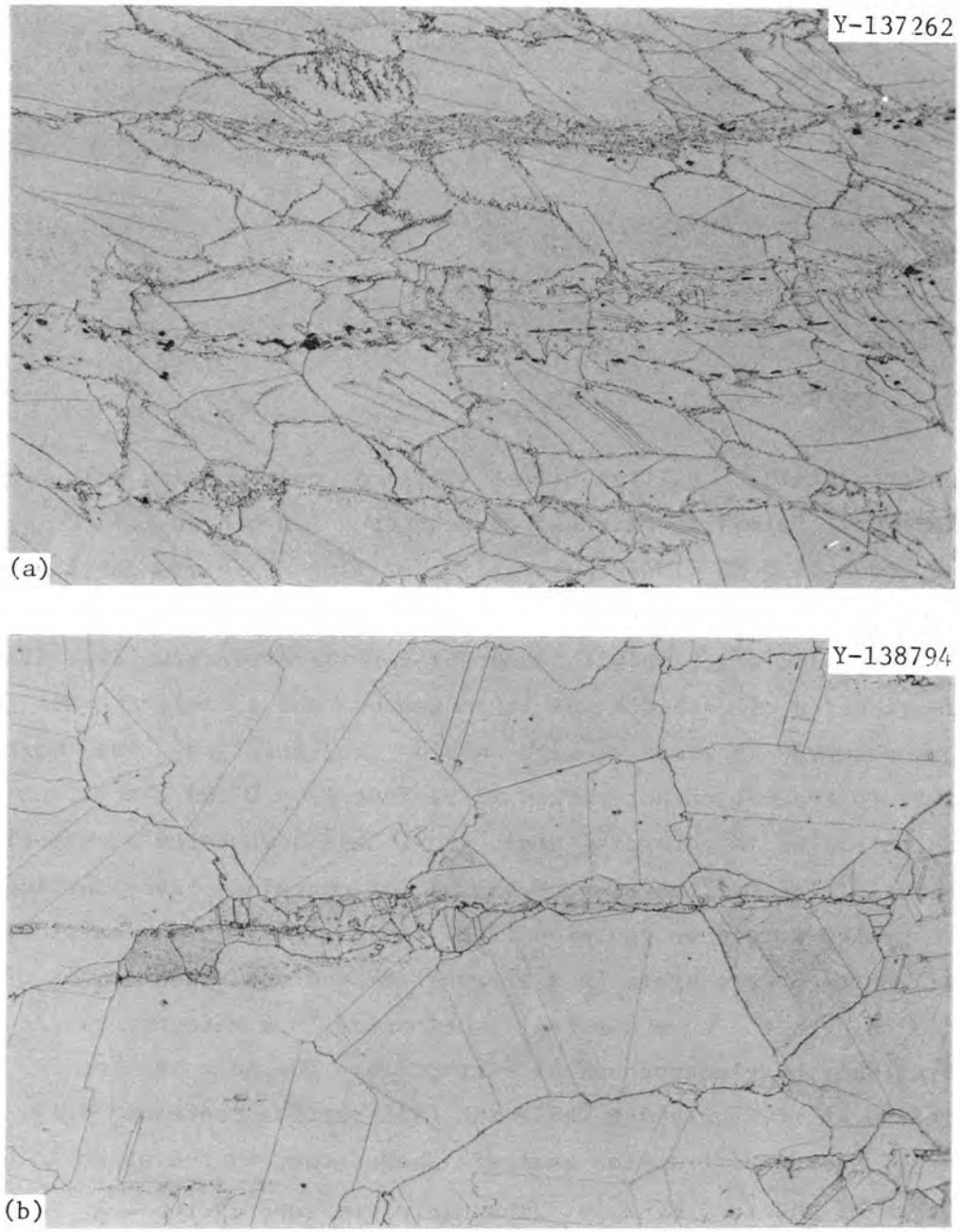
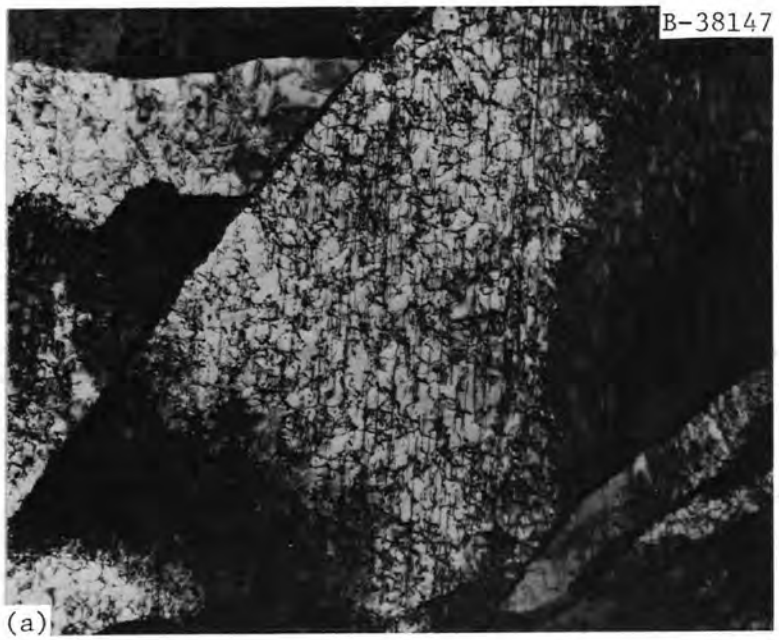


Fig. 2. Microstructure of Billet 1614 that Failed to Extrude. (a) Nose section. (b) Tail section. 100×.



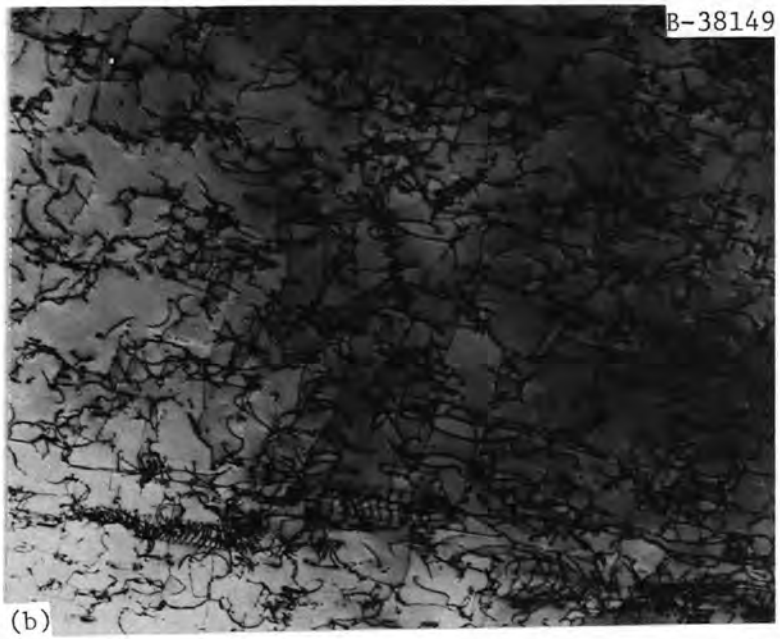


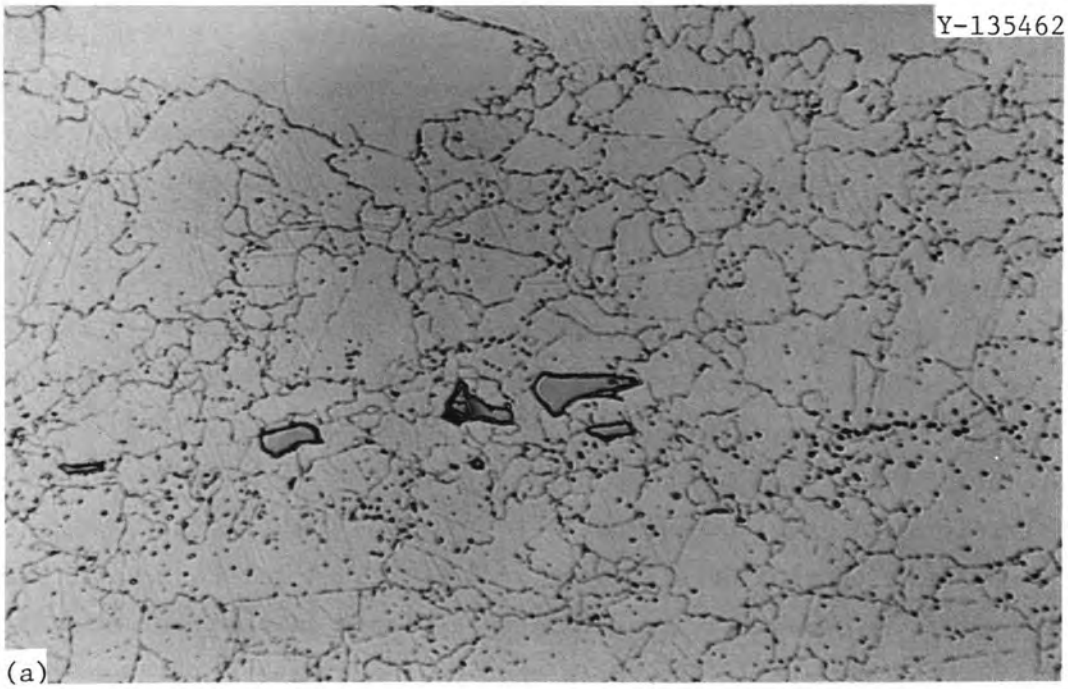
Fig. 3. Transmission Electron Micrograph of Foil Taken from Billet 1614. (a) From the nose portion. Plane of foil: $(0\bar{1}1)$. Dislocation density: 2×10^{10} cm/cm^3. (b) From the tail portion. Plane of foil: (200). Dislocation density: 6×10^9 cm/cm^3.

underwent considerable reduction, and the presence of numerous carbide particles would be expected to inhibit the motion of dislocation during slip. In contrast, the tail section of 1614 (Fig. 3b) contained a substantially lower dislocation density and few subgrains. Less MC particles were also observed in this section compared with the nose. Other work[7] with 2% Ti—Hastelloy N has shown that the amount of MC present in the microstructure decreases with increasing annealing times and temperatures.[4] Therefore the higher quantity of MC in the nose section of 1614 was probably caused by inadequate solution annealing. The tail section, which rested directly on the furnace hearth, was more fully annealed and more MC was dissolved into the matrix. Therefore, the reason that the billet could not be extruded was apparently because of a strengthening of the billet nose due to the presence of MC-type carbides in that region. The significance of the microstructural analysis was that it pointed out the importance of the billet preheat treatment. No longer could the preheat be regarded as merely heating the material to a hot-working temperature. For a successful extrusion, it is necessary to heat the billet at that temperature and for those times which will dissolve most of the MC particles in to the matrix. Furthermore, the analysis revealed that the billet must be preheated uniformly or otherwise carbides might precipitate in one portion of the billet but stay in solution in another.

The next experiment used a 609.6-mm-long (24-in.) billet, which was cut in half and machined to produce two extrusion billets of similar composition and history. Using these billets, numbered 1644 and 1645 (Table 1), enabled us to vary the preheat treatment twice while keeping essentially all other variables constant. The 1644 billet was preheated for 75 min at 1533 K but still failed to extrude, even under a load of 11.7 MN (1320 tons). However, the 1645 billet, preheated for 270 min at 1533 K was extruded rapidly under a relatively low load of approximately 5.3 MN (600 tons). The microstructures of samples cut from 1644 and 1645 were examined by optical metallography and found to differ considerably. They can be compared with the microstructure of a thin slice cut from the center of the starting billet for both

1644 and 1645 [Fig. 4(a)]. The hot-worked structure has a moderately small grain size with numerous small MC precipitates. Several large particles, presumable MC, were also present. Again, areas containing more carbide particles had a smaller grain size. The amount of MC precipitate in the starting billets was 0.49 wt %, as measured by electrochemical extraction. Figure 4(b) is a micrograph from the tube shell made from billet 1645. Rather large equiaxed grains, characteristic of a well-annealed material, were evident with only a few carbides. Extraction from the 1645 extrusion produced only 0.12 wt % of MC precipitates. A well-annealed structure and low percentage of MC indicated that the 270 min. at 1533 K preheat had uniformly dissolved most of the MC before extrusion.

The nose section of billet 1644 failed to extrude. Two different areas (Fig. 5) demonstrate the relatively large quantity and nonuniform distribution of carbides found in that region. Some areas near the outer surface of the billet contained less carbides possibly due to decarburization. These areas displayed a duplex structure dominated by large grains outlined by smaller grains in [Fig. 5(a)]. Higher magnification [Fig. 5(b)] showed that the smaller grains contained MC-type carbide in their grain boundaries. As mentioned, the carbide inhibits grain growth at elevated temperatures. These areas near the outer surfaces were only partially annealed by the preheat because only a few carbide particles were dissolved. The interior areas of the nose section of 1644 contained a larger number of small grains and MC particles [Fig. 5(c)]. This microstructure closely resembles that of the starting billet [Fig. 4(a)] and, therefore, suggests that these regions of the billet were unaffected by the preheat treatment at 1533 K. Higher magnification of the same area [Fig. 5(d)] showed again that the grain boundaries of the small grains were lined with MC. Extraction of MC precipitates from the nose area of 1644 showed that it contained 0.33 wt % — more than twice the amount extracted from 1645. The tail portion of 1644 had an annealed structure with equiaxed grain and numerous twins [Fig. 6(a)].



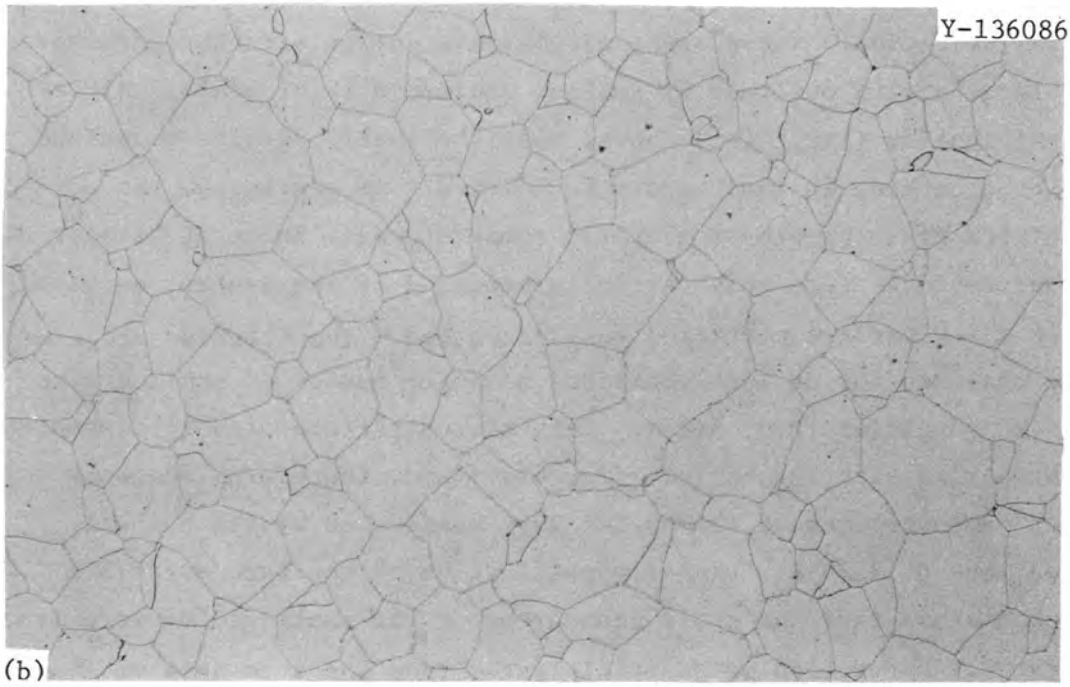


Fig. 4. Representative Microstructure of Billets 1644 and 1645 Before Preheating and Extrusion. (a) From center of the starting billet for both 1644 and 1645. 500×. (b) Section from tube shell made from billet 1645. 100×.

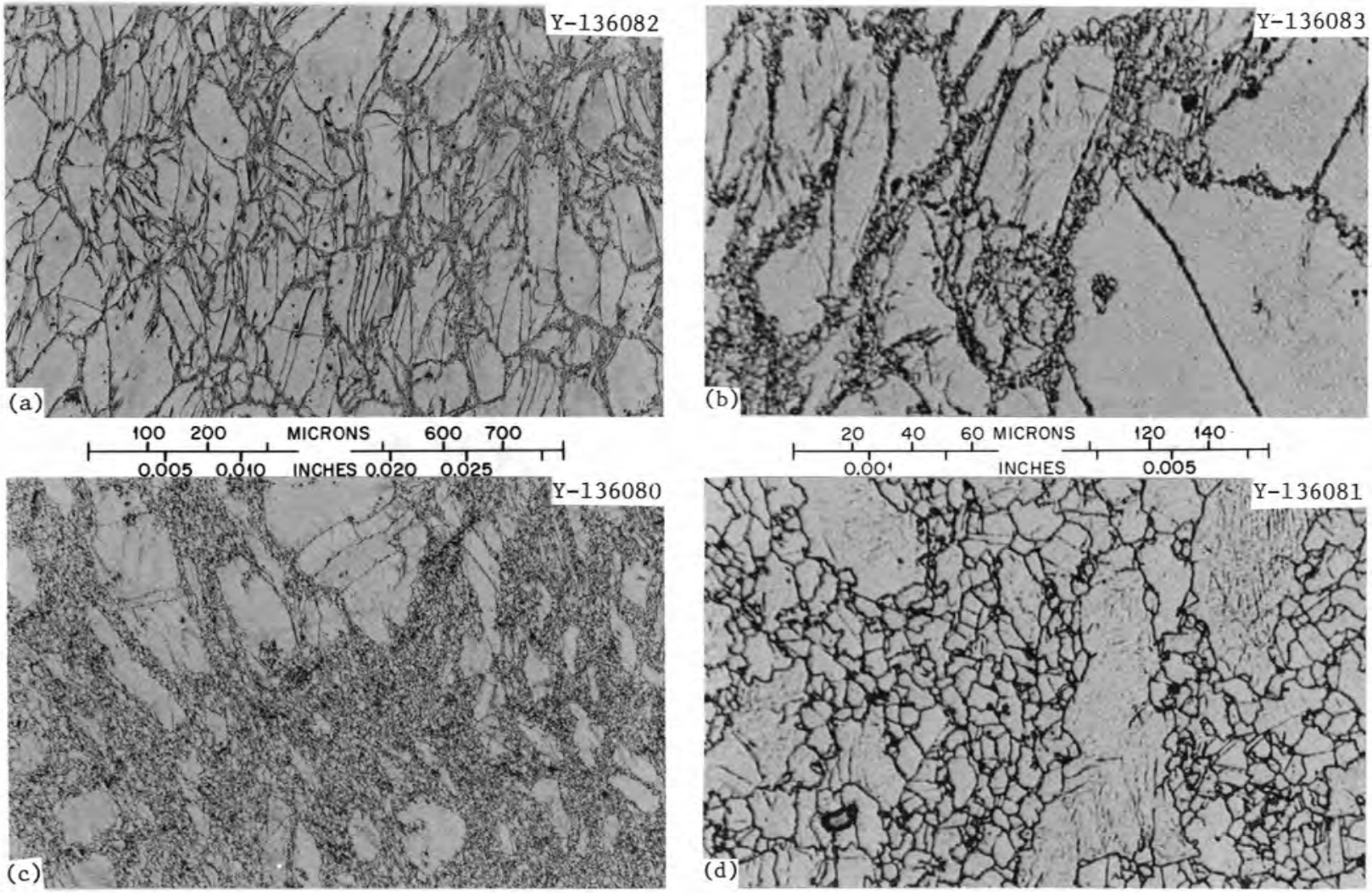


Fig. 5. Microstructure of Nose Section of Billet 1644. (a) and (b) Near the outer surface. (c) and (d) Interior area.

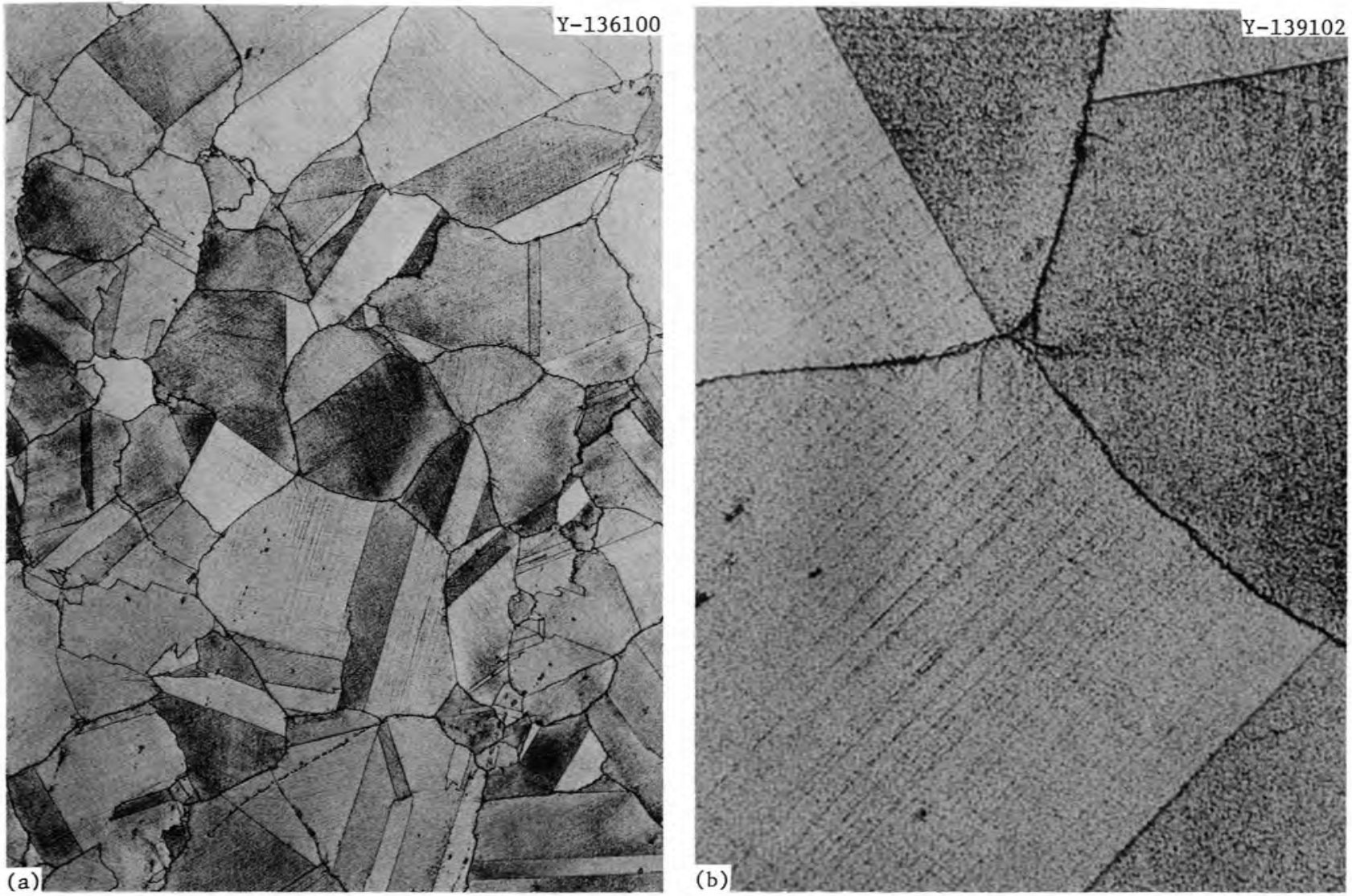


Fig. 6. Microstructure of Tail Portion of Billet 1644 at (a) Low, 100×, and (b) High, 500×, Magnification.

Faint arrays of small particles, presumably MC-type carbide, were also visible within the grains. This feature was more obvious at higher magnification [Fig. 6(b)]. This carbide morphology has been observed previously[4] in 2% Ti—Hastelloy N where thin platelets or stacking fault precipitates of MC lie epitaxially on [111] matrix planes. In that earlier work, this particular morphology appeared only in aged samples that had been previously solution annealed at temperatures from 1533—1573 K to dissolve the MC stringers; therefore, the implication from the microstructure in the tail section of 1644 is that most of the MC was indeed dissolved during the preheat and a small amount reprecipitated during the slow cooling of the billet after the attempted extrusion run. The difference between the tail and nose microstructures again indicates that the billets were not preheated uniformly by vertical placement in the furnace. The preheat dissolved the carbides in the billet tail, which rested on the furnace hearth plate, but only partially dissolved them in the nose section. Probably these undissolved MC-type carbides in the nose section provided sufficient yield strength in that area at 1533 K to prevent the billet from extruding even at the maximum press load. When the carbides in both the nose and tail regions were dissolved by using a longer preheat as in the case of 1645, extrusion took place with a load of only about 5.3 MN (600 tons).

3.2 Importance of Billet Homogeneity

If inadequate solution anneals are given billets, inhomogeneities in the carbide distribution (Figs. 4 and 5) may result. Even if the billet is successfully extruded, subsequent processing is apt to develop problems. This point can be illustrated by describing the processing of billet 1608 (Table 1). About 1778-mm (70 in.) of 1608 was extruded into tube shell using a 15-min preheat at 1450 K, glass lubricant, and a load of 11.5 MN (1300 tons). However, serious cracking along the inner diameter surfaces developed during subsequent cold drawing operations. Metallographic examination of sections cut from the tube shell revealed an approximately 0.3-mm (0.012-in.) layer of

carbides near the inner surface. Cracks extended through the thickness of this layer (Fig. 7). Chemical analysis of the carbide layer showed that it contained 0.46 wt % C — about six times greater than the nominal billet value of 0.07 wt %. The large number of carbides substantially increased the strength of the material in the layer as evidenced by the increase in microhardness from 389 DPH in the matrix to 448 DPH in the layer. With this increase in strength, there was undoubtably an accompanying loss in ductility. This reduction in ductility would account for the development of cracks during the cold drawing process. This observation again demonstrates in a different way the necessity of keeping the MC-type carbides in solution during all phases of fabrication. Improved mechanical properties can be achieved in the alloy after fabrication by utilizing suitable aging treatments.

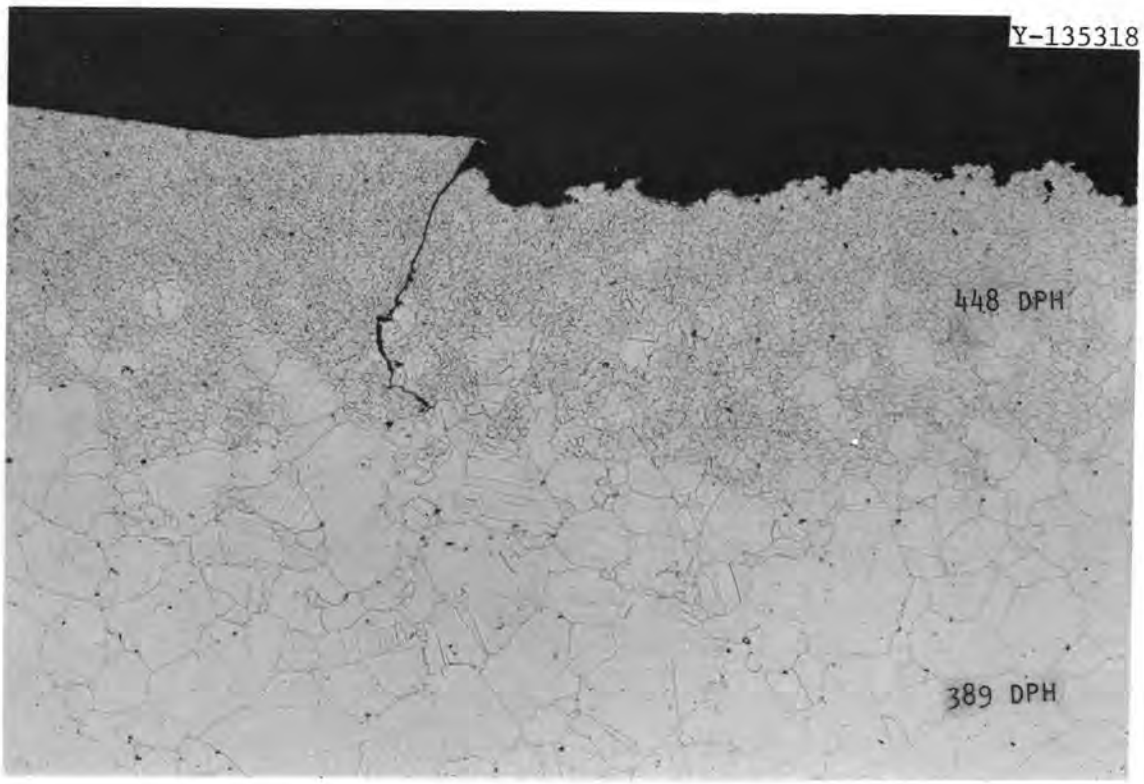


Fig. 7. Crack in Cold-Drawn Tube Fabricated from Billet 1608. Note that crack extends only through layer that contains a large amount of MC-type carbide particles. Microhardness values for the layer and tube matrix are shown. 100×.

4. CONCLUDING REMARKS

The results of the investigation demonstrate the importance of billet microstructure in the extrusion and cold drawing of 2 Ti-Hastelloy N. The critical step governing the billet microstructure was the preheat treatment given just prior to extrusion. It was found that the time and temperature of the preheat must be sufficient to uniformly dissolve most of the MC-type carbides into the matrix. The 101.6-mm-diam × 304.8-mm-long (4 × 12 in.) billet required a 4 hr preheat at 1533 K. Larger billets would require more time. Billets that were inadequately preheated contained MC-type carbides in the nose region of the billet that provided moderate yield strength to the material at the extrusion temperature. In such cases the billet could not be extruded even when maximum press loads were applied. Billets that were extruded using lower preheat temperatures and glass lubricant produced tube shells with inhomogeneous carbide distributions. Consequently, surface regions of the tube shell, which contained high carbide concentrations, were severely cracked during subsequent cold drawing operations.

5. ACKNOWLEDGMENTS

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