The Initiation of Intergranular Failure in Inconel-600

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The nature of slip interaction with the grain boundaries was examined on the polished surface of Inconel-600 pulled in tension at 370°C and at the initial strain rate of 1.65×10^{-6} sec^{-1}. It was shown that the grain boundaries could not always accommodate slip taking place at the boundary regions. This led to the separation of grains at the boundary. Depending on the detailed morphology of the carbides, these intergranular cracks could form at axial strains as low as 10 pct. Since the cracking takes place in the inert atmosphere, it is concluded that the grain boundaries are inherently embrittled by the formation of carbides and/or by solute segregation. There was no evidence of grain boundary sliding, but when carbides were absent highly localized slip in the boundary region gave the appearance of grain boundary offsets. The implications of these observations to stress corrosion cracking are discussed.

STRESS corrosion cracking of Inconel-600 (a nickel-base alloy containing about 15 wt pct Cr, 8 wt pct Fe) is known to occur in a variety of aqueous solutions at temperatures from 100° to 350°C; cracking is invariably intergranular. Of special interest are papers by Copson and Economy,[1] who summarized the literature briefly, Copson and Dean,[2] and Coriou, Grall, Olivier, and Willermoy.[3] Coriou *et al.* reported susceptibility in pure water of stressed bend samples with 0.5 pct strain. They used commercial Inconel as well as a special alloy with very low C, P, and S and found both to be susceptible; the grain boundaries in the commercial alloy were decorated with carbides while those in the special alloy were clean. Coriou *et al.* did not comment specifically on the frequency or severity of cracking in the two types of materials, but the implication was that there was little difference.

Copson and Dean attempted to reproduce the results of Coriou *et al.* without much success, except that they did observe cracking in one alloy which had high carbon and low chromium and which had been given a sensitizing heat treatment (2 hr at 650°C). Evidently cracking in pure water is subject to some subtle influences, but there is a suggestion in the work of Copson and Dean that material which is sensitized is more susceptible. From the work of Copson and Economy, who used double U-bend specimens in oxygenated water, it is clear that Inconel-600 is more susceptible to cracking in the sensitized than in the annealed condition.

In addition to carbide precipitation, cold work is also of importance in determining the susceptibility of Inconel-600 to stress corrosion in high temperature water. Thus DePaul[4] and Hubner, DePourbaix, and Östberg[5] reported intergranular attack on Inconel springs.

The mechanism or mechanisms of the stress corrosion cracking of Inconel-600 in pure water and in oxygenated water is not known, but presumably involves the conjoint action of stress and the corrosive medium. We thought it might prove useful to study the role of stress separately in order to observe how deformation occurs in the absence of corrosion. Of particular interest was the possibility that grain boundary shear might occur in Inconel-600 at temperatures in the vicinity of 350°C. In order to accelerate creep deformation but not be very far above the temperature used by Coriou *et al.* we chose a temperature of 370°C (about 0.37 times the melting temperature), and used very slow strain rates in order to encourage grain boundary sliding.

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EXPERIMENTAL

Material

The commercial grade Inconel-600 used in this investigation had the following composition (wt pct): 16.0 Cr, 6.8 Fe, 0.037 C, 0.2 Mn, 0.31 Si, 0.007 P, 0.009 S, balance Ni. It was received as $\frac{1}{16}$ in. thick mill-annealed sheet. The details of subsequent heat treatment as well as the resulting structures are given in Fig. 1.

Carbides are preferentially precipitated along the grain boundaries in the mill-annealed, Fig. 1(*a*), and solution-annealed and slowly cooled conditions, Fig. 1(*c*). In the latter condition, the carbides were less uniformly distributed than in the mill-annealed condition with respect to a given boundary as well as different locations. After solutionizing plus heating at 850°C for 4 hr, Fig. 1(*d*), extensive carbide precipitation has occurred not only at the grain boundaries but also at what appear to be the locations of the former grains present in the mill-annealed condition. In most cases the grains are not nearly equiaxed, the boundaries are not smooth, and there are many annealing twins.

Test Specimens

Tension test specimens with 0.20 width and 2.0 in. gage length were cut perpendicular to the original rolling direction of the sheet. One side of the machined tension specimen surface was then given a metallographic polish. It was lightly etched in a solution of 4 parts HCl, 1 part HNO_3, and 5 parts water at about 70°C for 15 sec. After the light etching, a series of nearly straight lines was scribed along the length of gage section with 8 μm silicon carbide abrasive Mylar paper. This provided fine reference lines of about 0.5 μm in width on the polished surface of the tension specimens.

Tension Testing

All the tests were made in the Instron machine at a cross-head speed of 0.002 in. per min. The test spec-

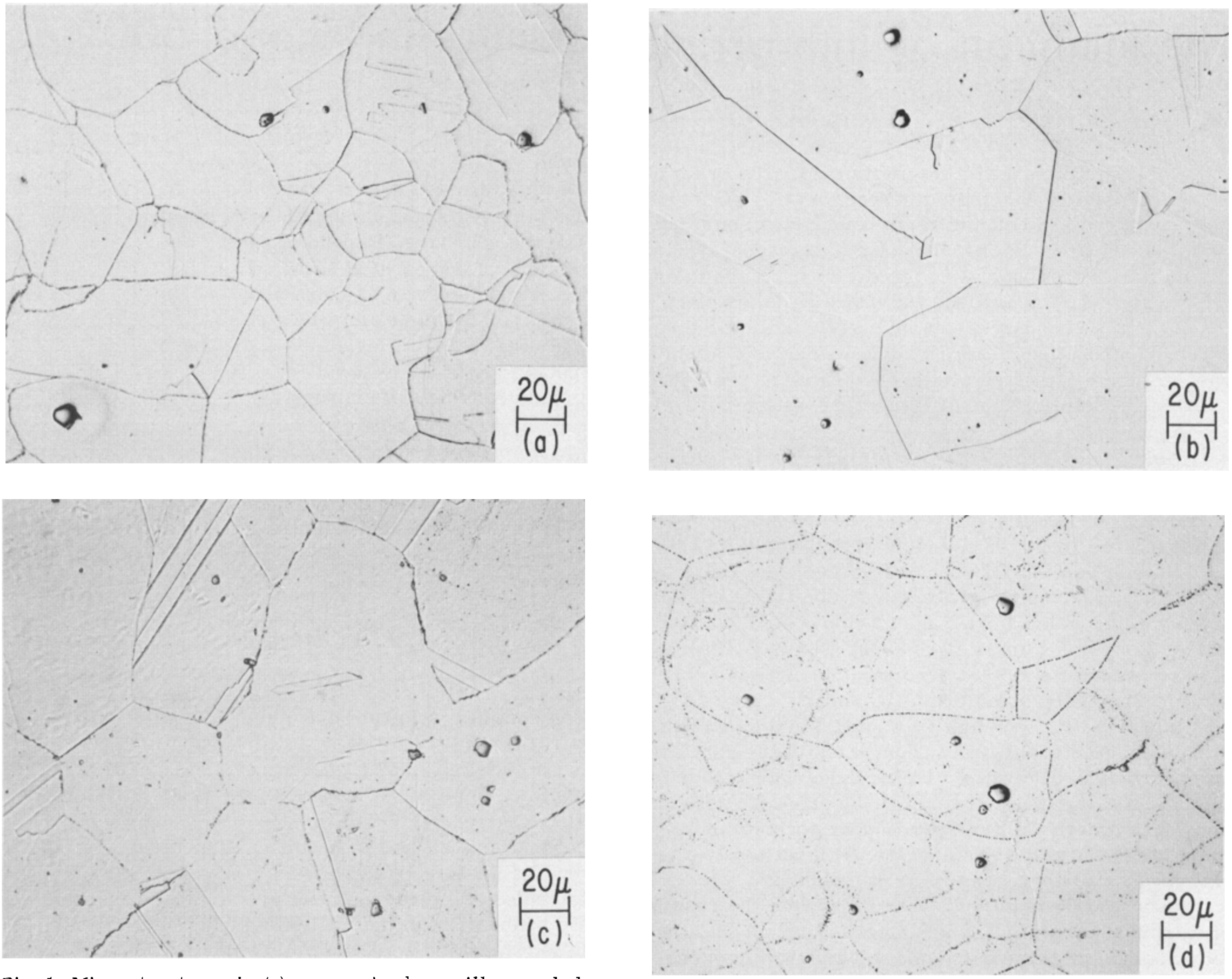

Fig. 1—Microstructures in (*a*) as-received or mill-annealed condition; (*b*) 1100°C for 10 min (solution-anneal) and water quenched; (*c*) 1100°C for 10 min and cooled to room temperature at the rate of 25°C per hr; and (*d*) 1100°C for 10 min, water quenched and 850°C for 4 hr followed by water quench. Etched in a solution of 4 parts HCl, 1 part HNO_3, and 5 parts water at about 70°C. Longitudinal cross-section.

imen was completely enclosed in a stainless steel cylinder through which purified helium gas was circulated. The cylinder was heated by a resistance wire furnace; the variation of specimen temperature was less than ±3° at 370°C.

Metallography

In addition to optical metallography, all the specimens were examined in the scanning electron microscope after they were pulled. The specimen surface was tilted by 50 ~ 60 deg with respect to the primary electron beam to bring out the details of surface irregularities.

RESULTS

A) Stress-Strain Behavior

An important feature of the tensile behavior was that of large strain-hardening capacity in all the heat-treated conditions. Since the strain-hardening exponent was estimated to be approximately 0.4, the strain at load instability could be correspondingly large. There was indeed no indication of necking in the gage section of specimens deformed to ~40 pct in axial strain.

The effect of heat treatment on the level of flow stress is summarized in Fig. 2. The strength after all our heat treatments was about equal and was substantially below that in the mill-annealed condition.

Another notable feature of the tensile behavior was that the load-elongation curve for all specimens was very jagged and had the appearance of saw-teeth. The maximum amplitude of the load fluctuation was about 2 pct of the load, and the period, which appeared to be quite regular, was about 3 min. It is speculated that the material is undergoing strain aging at this low strain rate.

B) Grain Boundary Shear (GBS) vs Concentrated Slip

For metals in general at low homologous temperature, deformation across the grain boundaries takes place by activating slip in the adjacent grains. The normal strain across the boundary is therefore discontinuous, but the shear strain *within* the boundary is

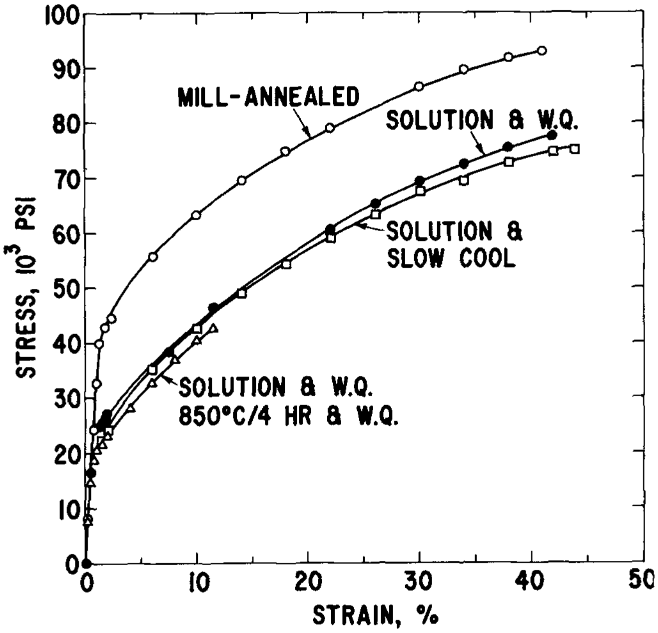

Fig. 2—Engineering stress-strain curves based on the load-displacement charts. Each test was stopped at the terminal data point shown in the curves. Test temperature 370°C.

always zero. This is not true if GBS takes place. Whether or not GBS takes place at the boundary can be determined by examining the offsets in the scribed lines. This is illustrated with materials in the mill-annealed and solution and quenched conditions.

In the mill-annealed condition, Fig. 3(*a*), there was no indication of offsets in the scribed lines across the grain boundaries. On the other hand, apparent offsets were observed in the solution and quenched condition, as shown in Fig. 3(*b*). However, when the same specimen was examined in the scanning electron microscope it was found that the offsets were not really due to GBS but were the result of concentrated slip near the grain boundaries. This is best illustrated in Fig. 4, a clear demonstration of the capability of grain boundary regions in the solution-annealed specimen to accommodate high localized strains. In the mill-annealed condition, however, the boundary region was not capable of accommodating localized strain; instead, a premature failure occurred by the separation of grains at the boundaries.

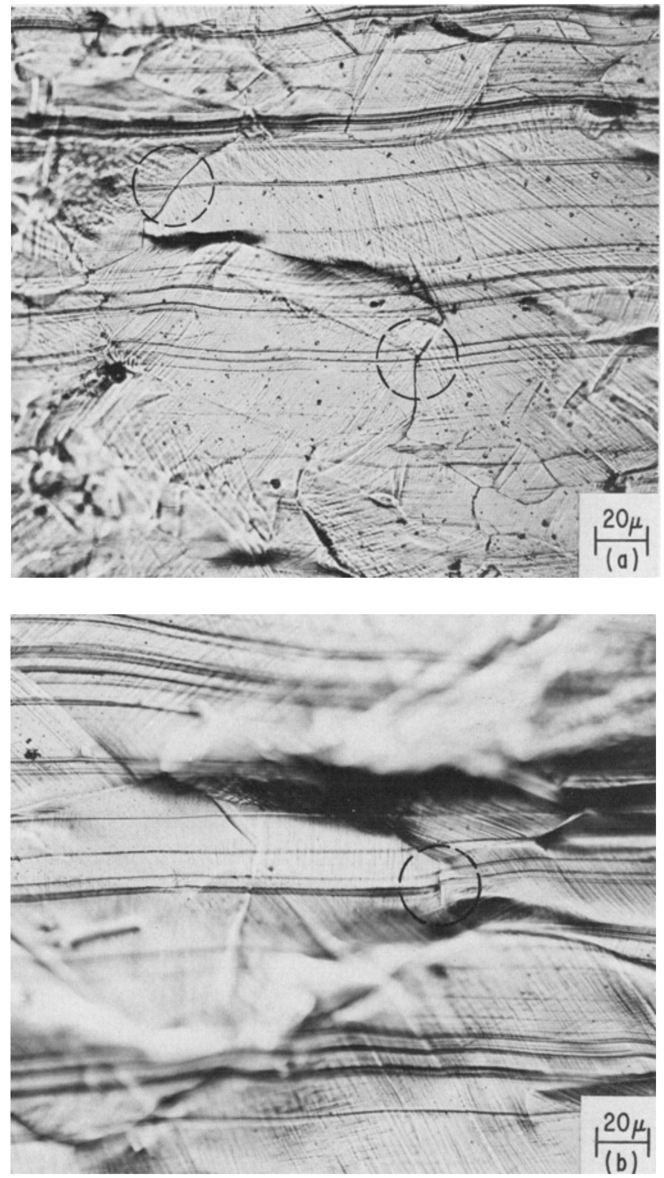

Fig. 3—Microstructures after the completion of testing: (*a*) mill-annealed pulled to a total strain of 41 pct; and (*b*) solution and water quenched pulled to a total strain of 42 pct. The horizontal lines represent the reference lines scribed on the surface before the test. The tension axis is horizontal. Note the grain boundaries marked by dashed circles.

Grain Boundary Cracking

Under our conditions of low strain rate grain boundary failure was observed in all except the solution annealed and quenched condition. For convenience, each heat-treated condition will be examined separately.

1) MILL-ANNEALED

Evidence for grain-boundary failure in the mill-annealed condition is given in Fig. 5. It is obvious from Fig. 5(*a*) that little plastic deformation has taken place near the boundary; the scribed lines remain undistorted at the boundary regions. Some shearing has taken place in the upper grain in Fig. 5(*b*), however.

The ease with which boundary failure occurs would depend on the relative orientation of slip planes in the adjoining grains, the angle of the boundary with respect to the tension axis, and so forth. For these reasons, more constraint will be imposed on those boundaries unfavorably oriented to accommodate slip across the boundaries. It is probably for these reasons that the fraction of boundaries that had openings as shown in Fig. 5 was not more than 30 pct. The initiation of intergranular failure was also observed in the specimen that had only 10 pct total axial strain.

2) SOLUTION-ANNEALED

There has been highly localized deformation near the grain boundaries but no grain boundary shear and no cracking.

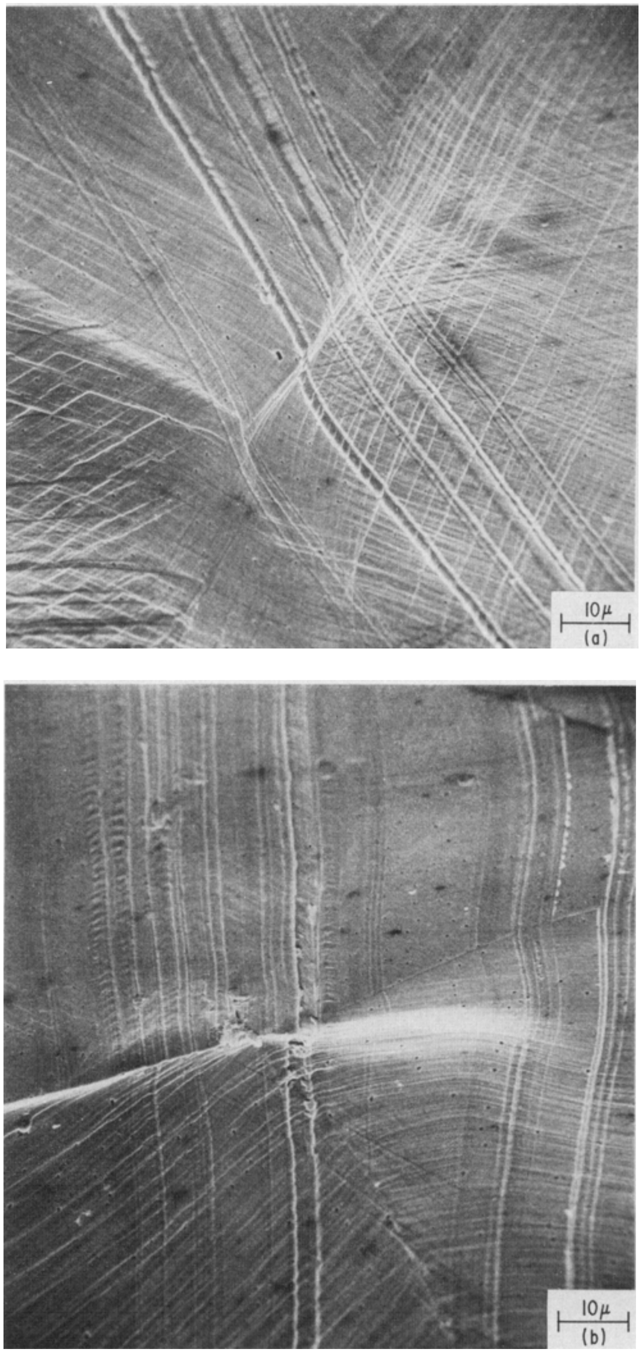

Fig. 4—Electron scanning micrographs of the deformed surface for the solution-annealed and quenched condition pulled to 42 pct axial strain. The loading direction is parallel to the scribed lines.

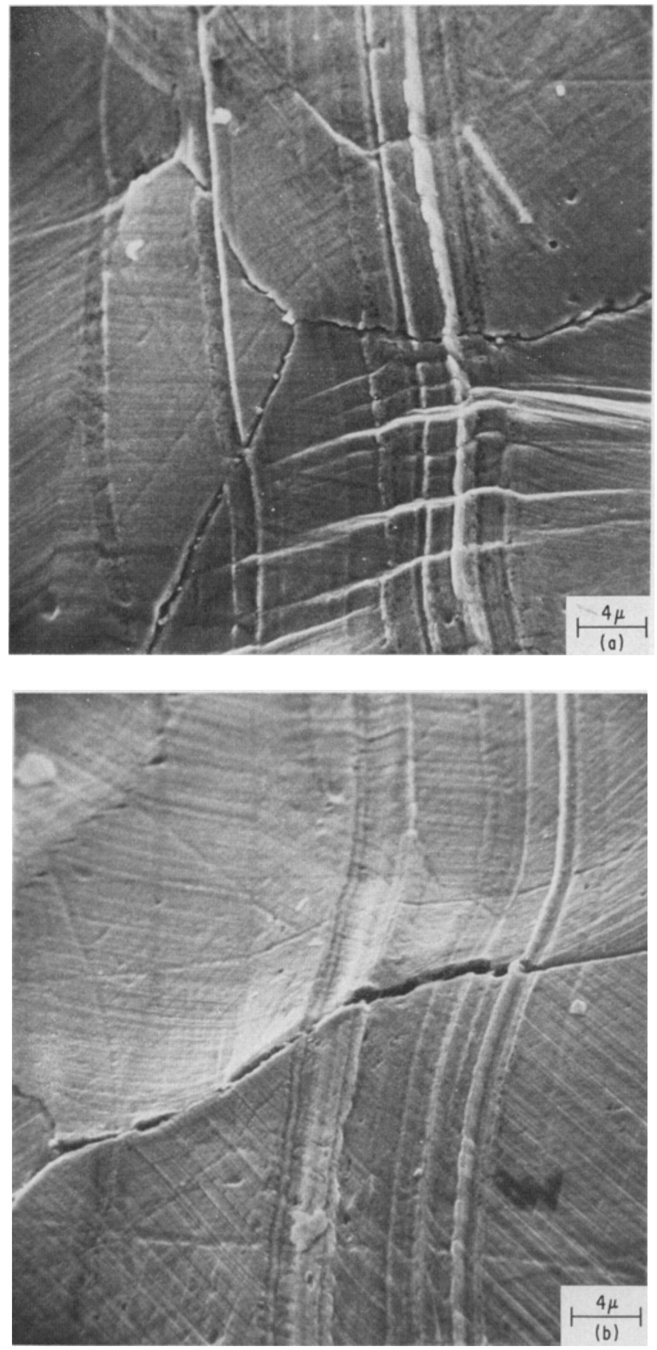

Fig. 5—Electron scanning micrographs of the deformed surface for the mill-annealed condition pulled to 41 pct axial strain. The loading direction is parallel to the scribed lines.

3) SOLUTION-ANNEALED AND SLOW COOLED

Some of the cracking that took place in this specimen was more severe than for the mill-annealed specimen, as shown in Fig. 6. The crack in (*a*) has serrated appearance, but in (*b*) it appears to be fairly flat. There is also an indication that the crack has formed by the intersection of slip planes in (*b*). Here again, there is little evidence that the boundary region has sheared by any appreciable amount.

Fine cracks were also detected in specimens given this heat treatment at only 10 pct total strain; an example is shown in Fig. 7.

4) SOLUTION-ANNEALED AND QUENCHED; 850°C/4 HR

In the presence of coarse carbide precipitates the frequency of intergranular failure was somewhat less than in cases (1) and (3). Cracking seems to occur at the boundary between the carbides, as shown in Fig. 8(*a*). Extensive slipping is seen to take place near the boundary, as shown in Fig. 8(*b*).

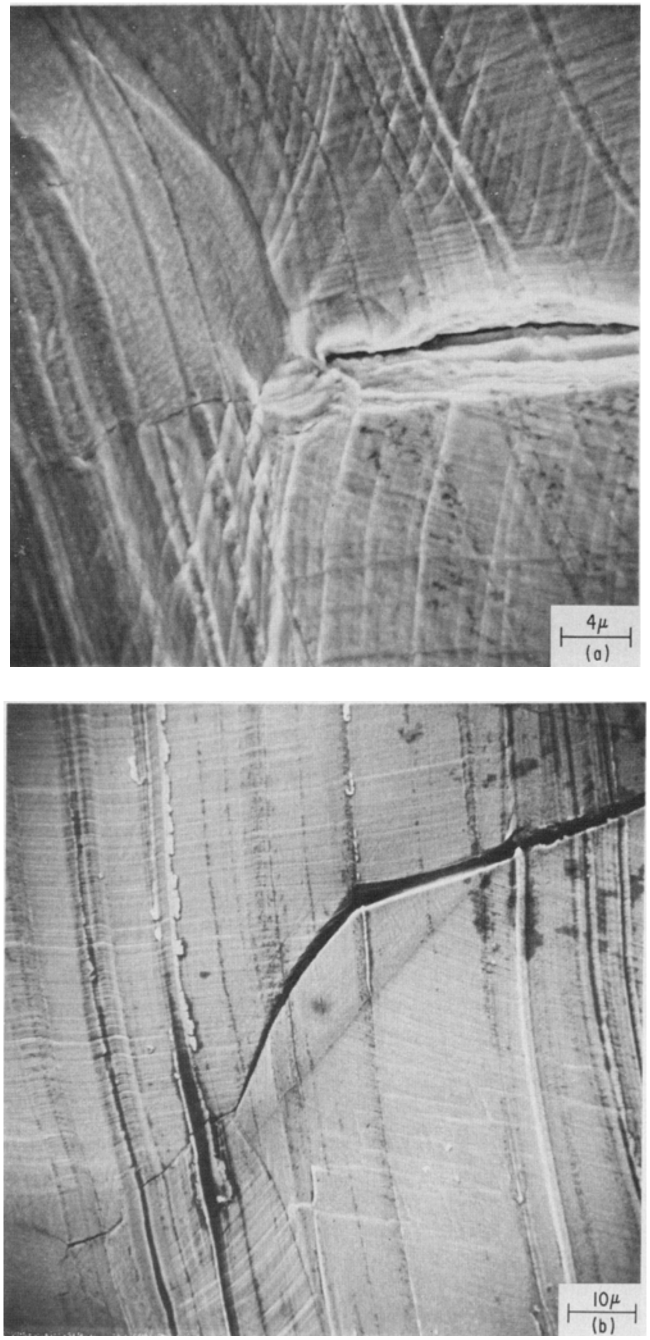

Fig. 6—Electron scanning micrographs of the deformed surface for the solution-annealed and slow-cooled condition pulled to 44 pct axial strain. The loading direction is parallel to the scribed lines.

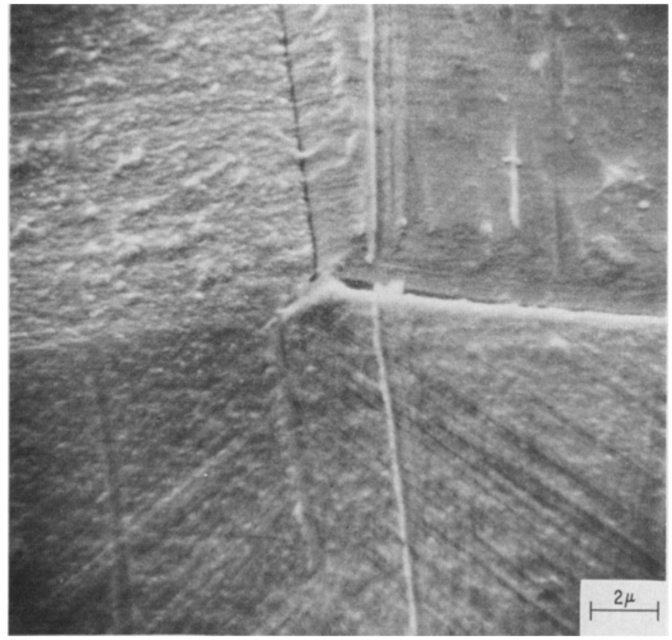

Fig. 7—Electron scanning micrograph of the deformed surface for the solution-annealed and slow-cooled condition pulled to 10 pct axial strain. The loading direction is parallel to the scribed lines.

Concurrently, some of the concentrated slip at the twin boundary formed a steep slip step, thus acting as a site for crack initiation, as shown in Fig. 9. Twin boundaries therefore also serve as barriers for slip.

DISCUSSION

Plastic deformation of a polycrystalline metal usually produces highly stressed regions near grain boundaries, twin boundaries, inclusions, and other regions which act as barriers to the motion of dislocations. Such high local stresses could be relieved by localized shear in the vicinity of the boundary, or by cracking. Cavities can also form at the grain boundaries from stress concentrations associated with grain boundary sliding, such as by Zener and other mechanisms.[6] In these studies we found no evidence of grain boundary shear, a fact which is not surprising in view of the relatively low homologous temperature.[7]

We did find evidence for localized deformation and for cracking, and discovered that the ability of the boundary region to undergo shear was greatly reduced in specimens which had been heat treated so as to cause grain-boundary carbide precipitation. Thus for solution-annealed and quenched specimens the high localized stresses near the grain boundary were relieved almost exclusively by plastic deformation in regions near the boundary. In all of the specimens containing carbides, however, there was little evidence for plastic deformation near the boundary and the high stresses were relieved by grain boundary rupture. Carbides may contribute in at least two ways to the cracking of grain boundaries. One is that they act as additional barriers to the motion of dislocations, thus forming potentially weak interfaces. The other is that carbides help to form grain boundaries with somewhat jagged appearance, as shown in Fig. 5. This would have a consequence similar to the formation of grain boundary cavities by the penetration of slip bands into the boundary.

The fact that grain boundaries act as weak interfaces may also be in part due to the segregation of chemical species yet unidentified. Evidence for this hypothesis comes directly from the jagged stress-strain curves which is indicative of strain aging-type behavior. The postulate that some form of dynamic strain aging occurs by solute segregation to the grain boundary during the slow strain-rate test can indeed explain many of these observations.[8]

These results suggest that sensitized specimens

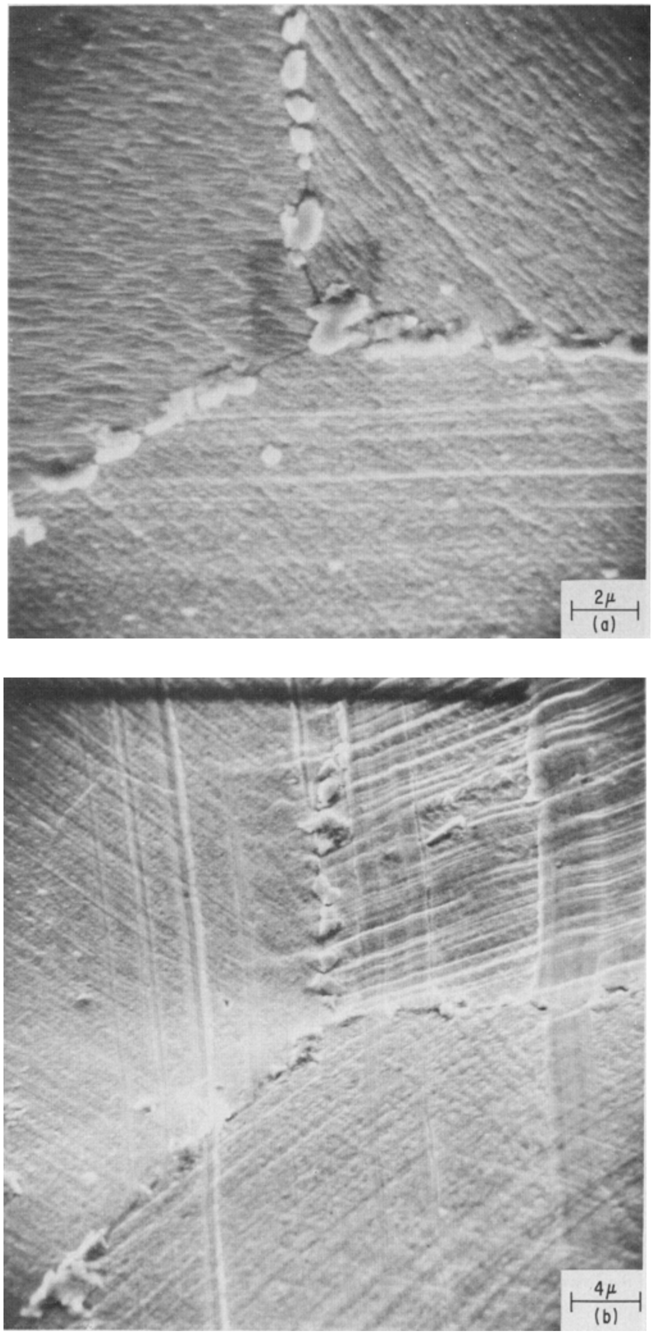

Fig. 8—Electron scanning micrographs of the deformed surface for the solution-annealed, water quenched, 850°C/4 hr and water-quenched condition pulled to 11.5 pct axial strain. The loading direction is parallel to the scribed lines.

should be more susceptible to intergranular stress-corrosion cracking than solution-annealed and quenched specimens. In the latter specimens localized deformation within a region roughly 5μ wide on each side of the boundary allows the relief of high stresses, so that there is no highly concentrated deformation at the boundary and the stress level at the boundary is relatively low. In the specimens containing grain boundary carbides such deformation does not occur with the result that the grain boundary stresses are much higher. In our experiments under conditions of continued deformation these high stresses caused cracks to develop at the grain boundaries. In stress corrosion presumably the high grain boundary stresses would enhance whatever mechanisms account for the cracking. Thus while both solution-annealed and sensitized specimens may be susceptible to stress corrosion, the cracking rate should be greater in the sensitized material. It should be noted that plastic strains of 10 to 40 pct are to be expected in the material near the tip of a growing stress-corrosion crack so that our observations are directly applicable to stress-corrosion cracking.

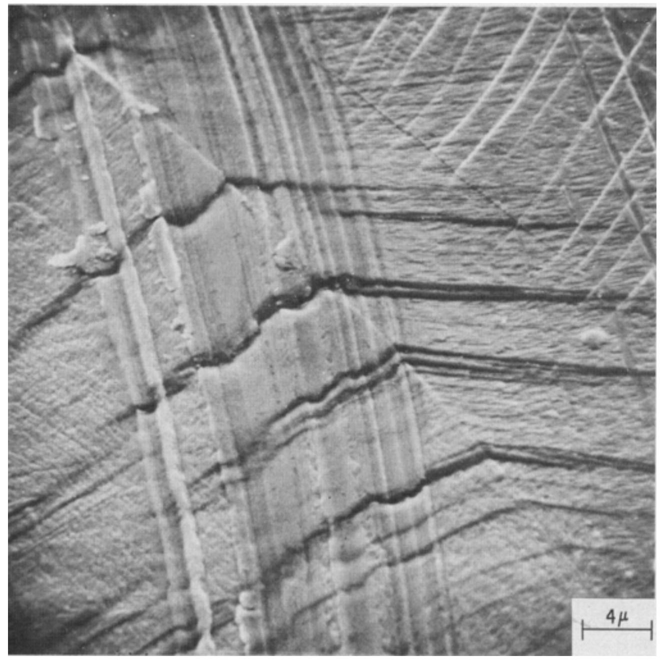

Fig. 9—Electron scanning micrograph of the deformed surface for the solution-annealed, water-quenched, 850°C/4 hr and water-quenched condition pulled to 11.5 pct axial strain. Note the slip steps forming at a twin boundary.

SUMMARY AND CONCLUSIONS

The conditions leading to the initiation of intergranular failure were examined in Inconel-600 at 370°C. The material was tested in different conditions of heat treatment so that the slip characteristics at the grain boundary region could be related to the presence of grain boundary carbides. All the tension tests were made in an inert atmosphere to study the role of stress alone, in the absence of corrosion. Some of the conclusions that can be drawn from this investigation are as follows:

1) In the solution-annealed and quenched condition where the grain boundary is free of carbides, it was shown that the boundary region is capable of accommodating all the concentrated slip at axial strains as large as 40 pct.

2) There was no evidence of grain boundary sliding in any of the specimens. High magnification and resolution were required to distinguish between highly localized slip in the boundary region and grain boundary shear.

3) Boundary regions in sensitized materials could not always accommodate deformation occurring near the boundary. This led to the formation of cavities at the boundary at axial strains as low as 10 pct.

4) It was shown that grain boundaries in the sensi-

tized condition could become embrittled not only by the presence of carbides but also by another mechanism, which may involve the segregation of solute elements during the dynamic strain-aging process.

5) The initiation of intergranular failure could occur by localized strain alone in the sensitized condition. Therefore, the rate of crack propagation during stress-corrosion cracking should be greater when the material is in the sensitized condition.

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REFERENCES

1. H. R. Copson and G. Economy: *Corrosion,* 1968, vol. 24, p. 55.
2. H. R. Copson and S. W. Dean: *Corrosion,* 1965, vol. 21, p. 1.
3. H. Coriou, G. Grall, P. Olivier, and H. Willermoz: *Proc. Conf. on Fundamentals of Stress Corrosion Cracking,* p. 352, NACE, Houston, Texas, 1969.
4. *Corrosion and Wear Handbook,* DePaul, ed., McGraw-Hill, New York, 1957.
5. W. Hubner, M. DePourbaix, and G. Östberg: *Ext. Abs. 4th Intern. Congr. on Metallic Corrosion,* Amsterdam, 1969, p. 5.
6. H. Conrad: *Mechanical Behavior of Materials at Elevated Temperatures,* J. E. Dorn, ed., p. 263, McGraw-Hill, New York, 1961.
7. R. C. Gifkins and T. G. Langdon: *J. Inst. Metals,* 1964, vol. 93, p. 347.
8. D. Lee, E. F. Koch, and H. C. Rogers: *Met. Trans.,* 1970, vol. 1, p. 3125.