THE EFFECT OF HYDROGEN ON THE DEFORMATION AND FRACTURE OF PWA 1480.

W.S. Walston, N.R. Moody‡, M. Dollar*, I.M. Bernstein* and J.C. Williams

Carnegie Mellon University, Pittsburgh, PA 15213
‡ Sandia National Laboratories, Livermore, CA 94550
* Illinois Institute of Technology, Chicago, IL 60616

ABSTRACT

The effect of internal hydrogen on mechanical properties and fracture behavior is being studied in the single crystal nickel-base superalloy PWA 1480. In particular, plane strain fracture toughness and tensile tests have been performed in hydrogen-free samples and hydrogen gas phase charged samples in two different sample orientations. The mechanical properties as well as the fracture process were found to be very dependent on orientation. Hydrogen did not affect the yield strength but reduced the uniform tensile elongation more than 75%. However, hydrogen reduced the tensile reduction-in-area and the fracture toughness by only 10%. Cleavage of the eutectic γ' as well as the role of hydrogen trapping will be discussed. These results will be analyzed in terms of the effects that orientation, microstructure and hydrogen have on the fracture process.

I. INTRODUCTION

The presence of a hydrogen-rich environment in the space shuttle main engine (SSME) provides a basis for the study of the effect of hydrogen on PWA 1480. PWA 1480 is a single crystal nickel-base superalloy and a candidate alloy for use as turbine blades in the high pressure fuel turbopump of the SSME. The effect of high pressure hydrogen and hydrogen-enriched steam on mechanical properties such as creep, low cycle fatigue and tensile properties has previously been studied for PWA 1480.[1-3] However, a detailed analysis of how hydrogen affects the deformation and fracture behavior has not been performed. This is the aim of the current study with a particular focus being to correlate observed hydrogen effects with the role of

Superalloys 1988
Edited by S. Reichman, D.N. Duhl,
G. Maurer, S. Antolovich and C. Lund
The Metallurgical Society, 1988

microstructural heterogeneities as trapping centers for hydrogen. PWA 1480 is being utilized as a model material to gain a better understanding of high γ' volume fraction superalloys in general. As a beginning to understanding the effect of hydrogen on the deformation and fracture behavior of PWA 1480, tensile tests and fracture toughness tests have been performed with samples containing a uniform concentration of internal hydrogen in two different orientations. The effect of hydrogen on the ductility and strength of this alloy was studied through the tensile tests, while the fracture toughness tests provided a means to study the crack growth behavior. This paper is concerned with the results of tests conducted at room temperature. The effect of higher temperatures on the deformation behavior has been previously reported[4] and the effect of internal hydrogen at these higher temperatures will be studied in the near future.

II. EXPERIMENTAL PROCEDURES

The composition of PWA 1480 is given in Table I. Single crystal bars were received from TRW Metals Division in the solutionized condition. The material was in the form of rectangular castings, 1.6 cm x 6.4 cm x 15.2 cm, with the solidification axis in the long direction. A standard heat treatment was performed which consists of a solutionizing treatment at 1288°C for 4 hours followed by water quenching, an aging treatment at 1080°C for 4 hours followed by air cooling and a final aging treatment at 875°C for 32 hours followed by air cooling.

Table I. Alloy Composition (wt. pct.)

Al	Co	Cr	Ta	Ti	W	Ni
4.8	5.3	10.4	11.9	1.3	4.1	bal.

Tensile tests and fracture toughness tests were performed with the loading direction parallel to the <001> orientation and a transverse orientation which was determined to be <130>. All hydrogen containing samples were gas phase charged at Sandia National Laboratories for 15 days at 350°C and a pressure of 103.5 MPa . Hydrogen content was analyzed by Luvak, Inc. by vacuum hot extraction at 900°C.

III. RESULTS

A. *Microstructure*

The microstructure of PWA 1480 is similar to other high γ' volume fraction alloys[5-7] having a dendritic macrostructure with interdendritic porosity and eutectic γ' as shown in Figure 1. The dendritic structure has the following dimensions: core diameter ~150 µm; arm length

~400-500 μm; and spacing ~200 μm. The pores were fairly spherical with an average size of 5.9 μm and an average spacing of 49.7 μm. However, pores an order of magnitude larger have also been observed. The volume fraction of porosity is .0012 which corresponds with the results of Milligan and Antolovich.[8] Eutectic γ' is also found in the interdendritic region and has an irregular shape. The volume fraction of this phase is .023 with an average size of 13.5 μm. It is important to note that the amounts of porosity and eutectic γ' remain fairly constant within each single crystal bar but can vary from bar to bar. Figure 2 illustrates the γ/γ' microstructure for 65% volume fraction of γ' with an average size of about 0.4 μm. As previously reported,[8] there are isolated areas in the interdendritic regions where the γ' is about 1 μm. The average γ/γ' mismatch has been measured by Bowman[9] using extracted γ' and was found to be 0.28 percent.

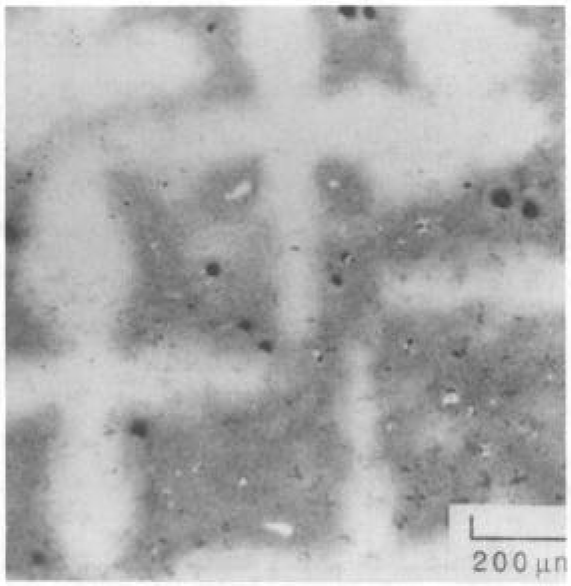


Figure 1. Dendritic macrostructure showing porosity and eutectic γ'.

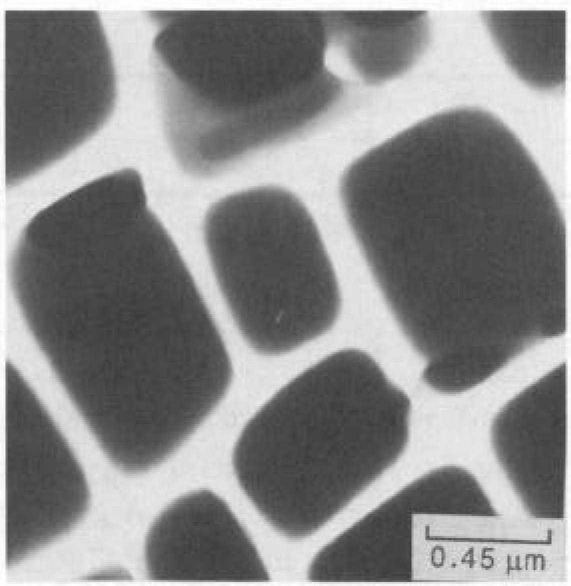


Figure 2. γ/γ' microstructure of PWA 1480.

B. *Hydrogen Trapping*

The measured hydrogen content was 30,000 appm (~3 at. pct.). This value is significantly higher than an estimated lattice concentration of 5,000 appm which was based upon data from a low γ' volume fraction alloy. In addition, after desorption for 5 hours at 300°C there is still a significant amount of hydrogen (17,000 appm) in the material. The high hydrogen concentration after charging and desorbing suggest the presence of strong trapping sites.

C. *Mechanical Behavior*

1. *Tensile Properties*

The tensile properties of PWA 1480 were found to be very dependent upon orientation as well as to the presence of hydrogen. Table II shows the tensile properties for both orientations with and without hydrogen. The <130> transverse orientation had about the same yield strength as the <001> orientation, but there was no work hardening. The major effect of orientation was seen in the ductility. The total elongation in the <001> direction was only 2.9%, while in the transverse direction it was 24.3%. The presence of internal hydrogen did not affect the strength or reduction-in-area, but dramatically reduced the elongation to failure for both orientations.

Table II. Tensile Properties

[001]	σ_{ys} (MPa)	σ_{UTS} (MPa)	e_t (%)	R.A. (%)
uncharged	1001	1120	2.9	3.1
charged	1014	1060	0.38	2.7
<u>transverse</u>				
uncharged	929	929	24.3	25.3
charged	906	906	6.9	21.2

2. *Tensile Fractography*

The differences in these properties as a function of orientation is reflected in the associated fractography, as shown in Figure 3. In the <001> tensile samples with and without hydrogen, 10-20 μm diameter cleavage regions were surrounded by smaller ductile voids around a micron in size. These large cleavage regions were often associated with pores, but more importantly there were many cases where the cleavage regions were not next to pores. Plateau etching revealed that these cleavage regions were the eutectic γ' as illustrated in Figure 4. In some cases the pores seemed to act as initiation sites, however in many cases there was no identifiable microstructural feature with the initiation site. In all cases, the river markings led back to the edge of a cleavage facet and never to the interior.

The entire transverse fracture surface consists of the small ductile voids which were seen in the <001> samples. Thus, the only difference between the two orientations is the presence of the cleavage facets in the <001> orientation. Plateau etching also shows that there is a relationship between these voids and the γ/γ' microstructure. The voids on the fracture surface usually consist of two or three γ' particles with the edge of the void representing a tear ridge along various γ/γ' interfaces.

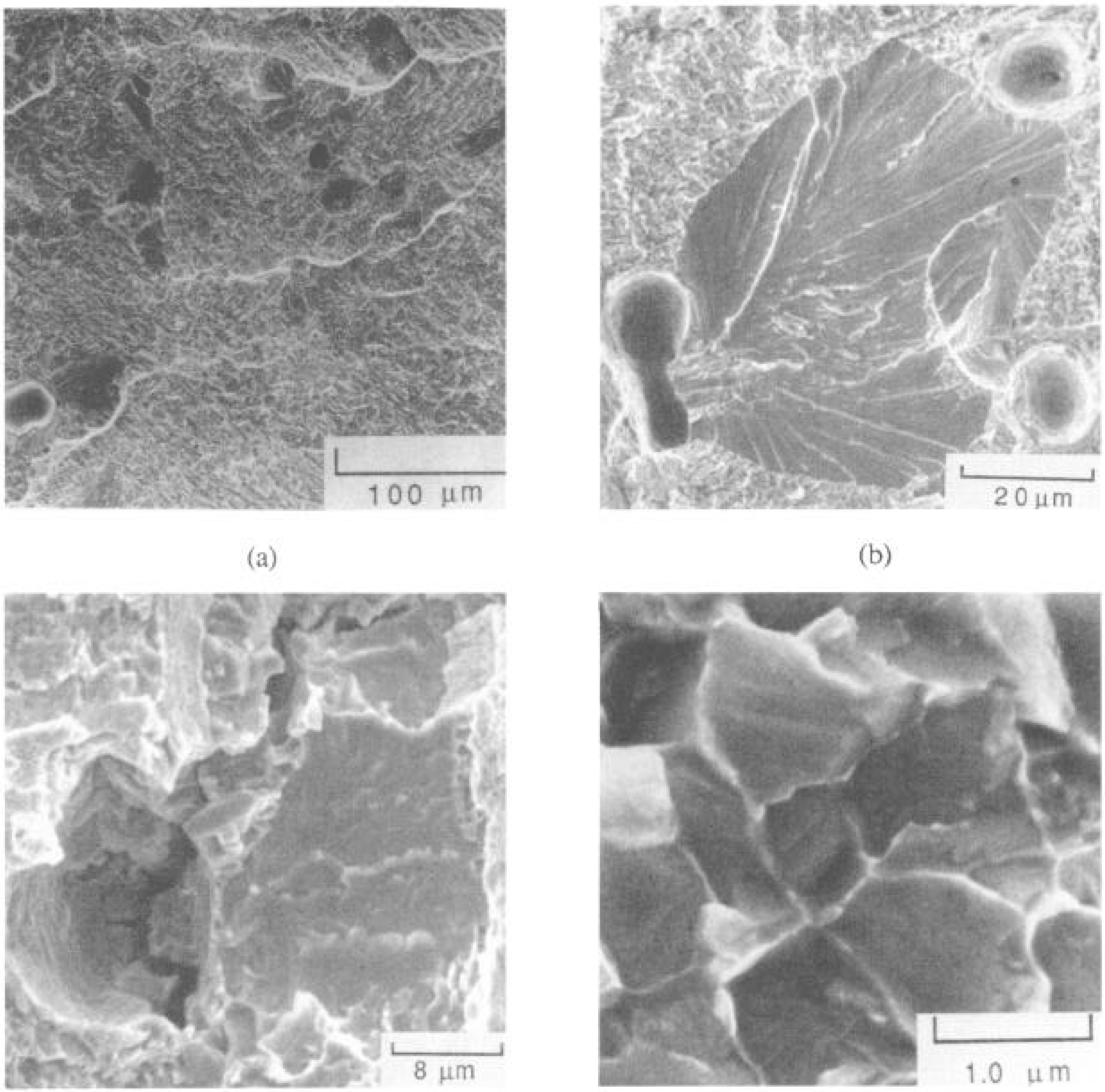


Figure 3. Tensile fractography. (a) Overall view in <001> showing porosity and cleavage. (b) <001> cleavage facet. (c) <130> with hydrogen showing brittle area. (d) Small ductile voids present in all specimens.

The presence of internal hydrogen had little effect on the tensile fractography. The only difference observed between the uncharged and charged samples showed up in the cleavage facets. In the [001] orientation, cracking within the cleavage regions was seen in the hydrogen charged samples only. Furthermore, there were small, flat regions in the charged transverse samples as seen in Figure 3(c) which were about the same size as the cleavage facets seen in the <001> samples. These regions were not seen in the transverse samples without hydrogen. They weren't the classic cleavage facets with river markings as were seen in the <001> samples, but appeared to be a more brittle type of fracture than the surrounding material. The presence of hydrogen did not seem to affect the size or shape of the small ductile voids.

Microcracks have also been observed in both orientations which change directions at 90° about every 0.5 μm suggesting that the crack is following the γ/γ' interface.

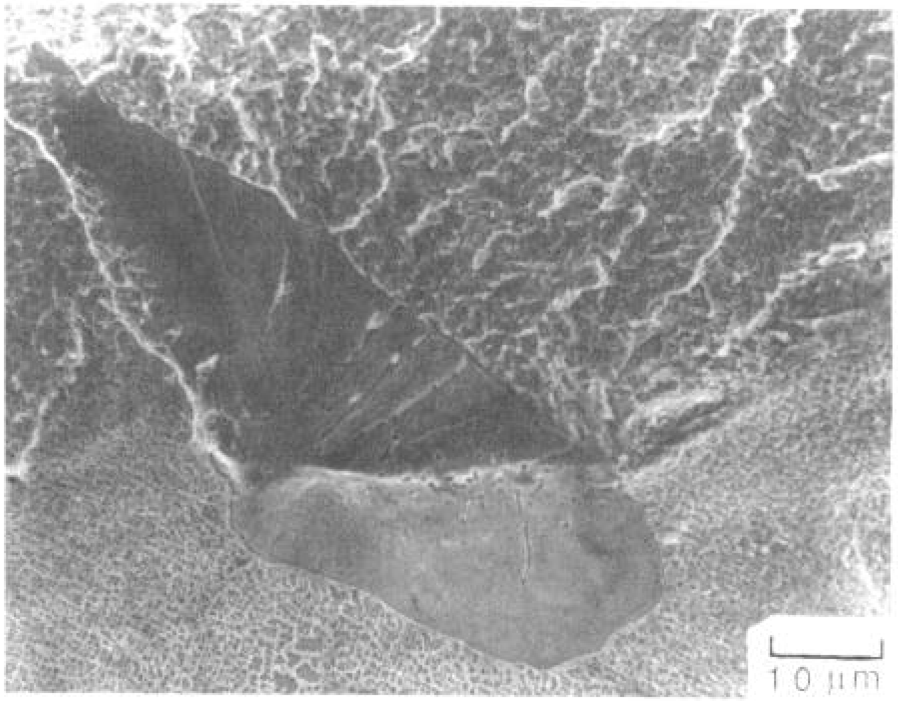


Figure 4. Plateau etch showing that eutectic γ' are the cleavage facets

3. *Fracture Toughness Properties*

Fracture toughness tests have been performed in the <130> orientation in the uncharged and charged conditions and in the <001> orientation in the charged condition. Testing is currently underway on the <001> samples without hydrogen. PWA 1480 exhibited very good fracture toughness (K_Q) in both orientations. In the <130> transverse orientation, hydrogen decreased the values of K_Q from 133 to 101 MPa·m$^{1/2}$. The value of K_Q in the charged <001> sample was 94 MPa·m$^{1/2}$. As a result of these high values and the morphology of the single crystal bars, valid toughness data could not be obtained.

4. *Fracture Toughness Fractography*

Fractographic analysis has only been performed on the transverse samples to date. The uncharged samples fractured predominantly along crystallographic planes while hydrogen causes a tortuous, less crystallographic crack path. In contrast to the tensile specimens, there were no cleavage regions observed in any samples. It was also noted that in all cases pores acted as initiation sites. Most of the pores had cracks either emanating from them or passing through them linking them to other pores. There were no other microstructural features which seemed to act as crack initiation sites.

IV. DISCUSSION

A. *Hydrogen Trapping*

The large amount of hydrogen in the material after the initial gas phase charging indicates the presence of at least weak traps. Even after desorption for several hours at temperatures of 150°C and 300°C, the concentration of hydrogen was an order of magnitude greater than has been found in nickel and austenitic stainless steels.[10,11]. It appears that the presence of internal hydrogen embrittled the eutectic γ' and weakened the γ/γ' interface and both can be considered trap sites, but the most obvious site for strong trapping is the pores.

More can be learned from the above observations by comparing them with a previous study on a similar alloy CMSX-2.[5] CMSX-2 is a single crystal nickel-base superalloy with approximately the same volume fraction porosity as PWA 1480. CMSX-2 contained some eutectic γ', but not to the same extent as PWA 1480. Similar concentrations of hydrogen were found in CMSX-2 after cathodically charging thin disks. Desorption experiments showed that the pores was a strong trapping site which the authors estimated to have a binding energy of 0.7 to 0.8 eV. Further desorption experiments are necessary to determine an accurate binding energy for the porosity in PWA 1480.

In CMSX-2, after desorbing for a short time at 300°C there was very little hydrogen left whereas there was still a large amount of hydrogen in PWA 1480 after desorbing for several hours at 300°C. Based upon microstructural comparison given it doesn't seem that the trapping in PWA 1480 should be that much stronger than in CMSX-2. Both alloys contain similar trapping sites which should have similar trap binding energies. The γ/γ' mismatch is larger in PWA 1480 so the trapping strength of this interface may be slightly greater. The trapping characteristics of the eutectic γ' are unknown and cannot be compared. There may be more trap sites in PWA 1480 due to the much higher volume fraction of eutectic γ' and the widely varying porosity distribution. The average volume fraction of porosity in these single crystals is known to vary widely from bar to bar. A possible explanation for the differences could be that the bars selected for desorption actually had quite different volume fractions of porosity. A further explanation may be that PWA

1480 simply has more available sites for trapping due to the larger γ/γ' misfit and the higher eutectic γ' volume fraction.

B. *Tensile Specimens*

The major difference in the tensile properties of the two orientations was manifested in the tensile elongations, however at this point there is no explanation for this large difference. The determination of active slip systems is currently underway as are TEM studies which may explain the observed differences.

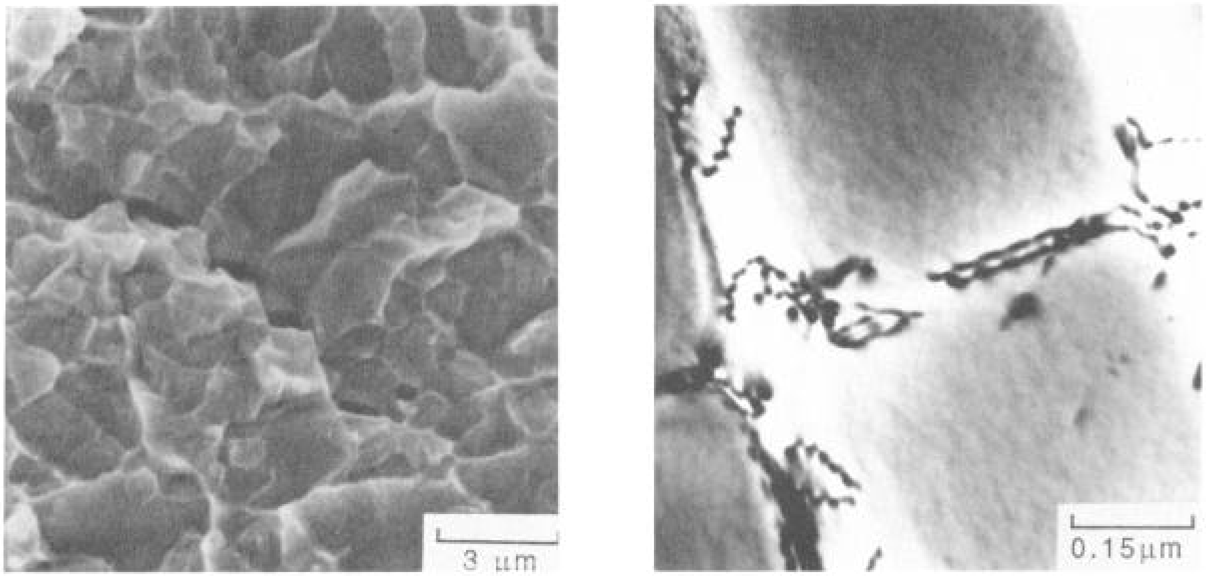
Internal hydrogen had no effect on the strength of PWA 1480 however hydrogen slightly decreased the reduction-in-area and markedly decreased the total elongation in both orientations. The hydrogen charged tensile samples exhibited less elongation of the gage length, but as much reduction in cross-section diameter as the uncharged samples. These observations suggest that hydrogen localized the plastic flow.

It was observed that only some cleavage facets initiated pores while other facets didn't have any obvious microstructural initiation site. These observations suggest that the stress field surrounding the pores do not cause cleavage of the eutectic γ'. If it was the stress field surrounding the pore, then every cleavage facet would be initiated at a pore. It is not known exactly what is causing the cleavage initiation. One observation that may help is that in every instance the river markings led back to the edge of the facet suggesting a nucleation site at the interface of the eutectic γ'.

It should also be noted that the cleavage facets were only observed in the tensile specimens and never in the fracture toughness specimens. Taking into account that the tensile specimen undergoes a combination of stress and strain prior to failure and that the fracture toughness specimen principally experiences only a stress state in the crack tip region prior to failure, one may conclude something about the requirements for cleavage of the eutectic γ'. Orowan's[12] original proposal that a critical value of tensile stress is required to produce cleavage fracture doesn't explain the observations in this study. It seems that some critical strain or combination of stress and strain is necessary to produce cleavage as found by Lewandowski and Thompson[13,14] in pearlitic 1080 steel.

Plateau etching revealed that there is a relationship between the ductile voids on the fracture surface and the γ/γ' microstructure. The ductile voids are on the order of 1 micron, while the size of the γ' is about 0.4 μm. Close observation of the plateau etching micrographs suggests that the fracture occurs along the γ/γ' interface. The actual void consists of two or three γ' particles with the edge of the void representing a tear ridge along various γ/γ' interfaces. This observation can be correlated with the occurrence of microcracks and with the development of the dislocation structure during deformation of PWA 1480[4]. The microcracks appear to be occurring along the γ/γ' interface. This hypothesis is supported by the deformation observations shown in

Figure 5. In particular, the dislocation density in the matrix was found to be significantly higher than that in the precipitates for all strains. This promotes strain localization in the matrix and may result in premature plastic failure due to strain exhaustion, as manifested by the occurrence of microcracks following γ/γ' interfaces.



(a) (b)

Figure 5. (a) Microcracks in tensile specimen. (b) Accumulation of dislocations in γ matrix at small strain, $\varepsilon_p = 0.24\%$ (from Dollar and Bernstein[4]).

C. *Fracture Toughness*

Preliminary test results indicate that PWA 1480 has very good fracture toughness. There does not appear to be any effect of orientation on the fracture toughness values, at least in the two orientations tested. Although the tests on the <001> samples without hydrogen have not been completed, it may be assumed that this value will be close to the uncharged <130> value. This is based upon similar values in the two orientations for the charged samples and also the results of short rod fracture toughness tests.[15] If this is the case, then hydrogen decreases the fracture toughness value by about 25%. The presence of internal hydrogen also affected the crack growth behavior in the transverse samples. It will be interesting to see if hydrogen has the same effect on the <001> orientation. In contrast to the tensile samples, the pores seem to act as fracture initiation sites in all fracture surfaces examined to date. Completion of the fractography and J integral tests to determine valid fracture toughness values are part of the future research.

V. CONCLUSIONS

1. The pores present in PWA 1480 act as very strong trapping centers for hydrogen while the eutectic γ' and the γ/γ' interface may serve as weaker traps.
2. The presence of internal hydrogen localized the plastic flow in the tensile samples as evidenced by a large change in elongation and a correspondingly small change in reduction-in-area.
3. The cleavage facets seen on the tensile fracture surfaces are the eutectic γ' in the microstructure. The stress field surrounding each pore is not responsible for initiating these cleavage facets, however it is not known what is initiating the cleavage.
4. It appears that a critical value of tensile stress is not sufficient to initiate cleavage, but that a critical strain or combination of stress and strain may be necessary.
5. Tensile fracture is occurring along the γ/γ' interface or more likely through the γ phase. This is supported by plateau etching, the observation of microcracks and the study on the development of the deformation structure.
6. PWA 1480 exhibited very good fracture toughness values with and without hydrogen.
7. The fracture initiation site in all of the fracture toughness samples was the porosity.

VII. REFERENCES

1. D.P. Deluca and B.A. Cowles, *AFWAL-TR-84-4167*, Feb. 1985.
2. Cowles et. al., *Adv. Earth-to-Orbit Propulsion Tech. 1986*, Huntsville, AL, NASA CP 2437, pp. 727-748.
3. N.L. Weeks and J. Mucci, *NASA FR 19269*, June 1986.
4. M. Dollar and I.M. Bernstein, *Acta Met.*, in press.
5. C.L. Baker, J. Chene, I.M. Bernstein and J.C. Williams, *Met. Trans.*, **19A**, 73, (1988)
6. B.J. Piearcey, B.H. Kear and R.W. Smashey, *Trans. ASM,* **60**, 634, (1967)
7. M.V. Nathal, R.D. Maier and L.J. Ebert, *Met. Trans,* **13A**, 1767, (1982)
8. W.W. Milligan and S.D. Antolovich, *Met. Trans.*, **18A**, 85, (1987)
9. R.R. Bowman, *M.S. Thesis*, Georgia Inst. of Tech., Atlanta, GA, 1986.
10. W.M. Robertson, *Met. Trans.,* **8A,** 1709, (1977)
11. Y. Shehu, P. Menut, J. Chene and M. Aucouturier, *Proc. 3rd Int. Congress on Hydrogen and Materials,* Paris, 1982, p. 889.
12. E. Orowan, *Trans. Inst. Engrs. Shipbuilders Scot.,* **89**, 165, (1945)
13. J.J. Lewandowski and A.W. Thompson, *Met. Trans.,* **17A**, 1769, (1986)
14. J.J. Lewandowski and A. W. Thompson, *Acta Met.,* **35**, 1453, (1987)

ACKNOWLEDGEMENTS

The authors are grateful to A.W. Thompson for many fruitful discussions. This research was supported by NASA-Lewis Research Center under the technical direction of Dr. R. Dreschfield.