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DETERMINATION OF CLEAVAGE PLANES AND FRACTURE CHARACTERIZATION OF Ni-BASED SINGLE CRYSTAL SUPERALLOYS

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ABSTRACT

The room temperature fracture behavior of the Ge Rene N-4, CMSX-2, and CMSX-4C single crystal Ni-based superalloys was studied. All crystals were grown along the [001] direction and tensile tested in both helium and hydrogen atmospheres. A stereoscopic technique developed for use with a scanning electron microscope was applied to determine cleavage planes. Planar γ' morphologies also were examined to help determine cleavage planes. Helium charged specimens failed on a number of planes including the (111), (110), and (320). In most cases planes of the (111)-type initiated at the notch region and became smaller and smaller as they moved in radially. Tensile strengths in helium averaged 1000 MPa higher than that of the hydrogen charged specimens. Specimens tested in hydrogen generally failed on (100)-type planes originating from the notch region. This (100) region comprised 60 to 80% of the total fracture surface on most samples and appeared as large flat planes perpendicular to the growth direction of the crystal. The interior regions contained (100)-type planes as well as (321),(320),(210), and (111)-types. Hydrogen charged specimens also showed a high percentage of large cracks oriented at 90° to one another, indicative of the (100)-type fracture. The Ge Rene N4 and the CMSX-4C samples contained 3-5% γ/γ' eutectic, while the CMSX-2 samples had little or no γ/γ' eutectic. The relationship between γ/γ' eutectic and the fracture surface has not been fully determined, but it is thought that the γ/γ' eutectic may serve as a possible trapping site for hydrogen.

TABLE OF CONTENTS

Tables and Figures

INTRODUCTION

Single crystal superalloys are being investigated for use as turbine blades in hydrogen fueled advanced jet or rocket engines. For this purpose they must possess favorable high temperature properties, such as creep resistance, thermal fatigue resistance, and oxidation resistance.[1] Mechanical properties, at both high and low temperatures, have been continually improved by controlling the composition and microstructure. Nickel-base superalloys consist of a nickel-rich fcc γ phase and an ordered fcc γ' phase based on Ni_3AL. The γ' phase is usually coherent with the γ matrix, and makes up 60-70% of the volume fraction. γ' precipitates can more than double the strength of the single crystal with temperatures up to 700° C.[1]

The absence of a grain boundary in single crystals eliminates the problem of segregation of elements along the grain boundary, thus resisting grain boundary embrittlement. Single crystals exhibit anisotropic behavior, allowing the orientation to be maximized for superior properties over that of polycrystalline microstructures. However, these materials when introduced to a hydrogen environment may suffer hydrogen embrittlement.[2] Little is known about the relationship between this embrittlement and crystal orientation. Therefore, the purpose of this research was to characterize the fracture behavior and the microstructure of four different compositions of nickel-based superalloys and to evaluate the effects of hydrogen on the fracture as compared with non-hydrogen tested specimens. The presence of γ/γ' eutectic and its relationship with the fracture behavior was also considered. Specimens were mechanically tested in both high pressure helium and hydrogen environments at room temperature. Scanning electron microscopy was used to study the microstructure and determine orientations in the four different superalloys all grown along the [001] direction.

LITERATURE REVIEW

General

Superalloys in service as turbine blades must withstand extreme conditions. They must retain their properties in instances of high temperature, corrosive gases, vibrations, mechanical loads due to centrifugal force, and thermal and mechanical fatigue due to engine starts and stops.[2] Nickel-base superalloys with high γ' percentage, generally become stronger at higher temperatures. They can retain their usefulness at temperatures up to 1000° C. These heat resistant alloys are used in gas turbine engines to make up the turbine blades and the compressor, near the combustion chamber, where the air is at it's highest temperature and pressure. The actual combustion chamber is made of Cobalt-based alloys, which are not as strong as nickel-base alloys, but retain their strength at higher temperatures.[2] The nickel-base superalloy is made up of a γ matrix with coherent γ' precipitates, the γ' phase being more ordered.

The single crystal nickel-base superalloy is made using directional solidification with a helical restrictor between the chill plate and the upper part of the mold. As the columns grow, the helical restrictor allows only one grain to grow through, so there are no grain boundaries. The amount of γ' is controlled by the composition, while the size of the γ' particle is controlled by the cooling rate. Optimally, a superalloy should have 60-70% small γ' particles coherent within the γ matrix. The high temperature strength stems from the growth of these small γ' particles during exposure to high temperature.[3]

γ/γ' Eutectic

During solidification, the γ' phase forms first, leaving molten metal in the interdendritic regions. As the temperature drops, the γ/γ' eutectic is formed. The γ/γ' eutectic can be one of two types, that formed at higher temperature which is finer and lacelike, and that which forms at lower temperature which is more uniform and consists mostly of γ'.[1] Walston et al.[4] have reported that the γ/γ' eutectic had a significant effect on the ductility, increasing the strain to failure. However, it did not affect the yield strength. Furthermore they observed a dislocation buildup at the γ/γ' eutectic interface with little activity inside the γ/γ' eutectic. This would imply that the interface is an effective barrier to dislocation motion. While the presence of γ/γ' eutectic did not significantly affect the appearance of the fracture surface, Walston et al.[4] believed that crack initiation began in or at the interface of the γ/γ' eutectic. A build up of dislocations at the interface of the γ/γ' eutectic suggested that large stresses could develop there and initiate cleavage. Similarly, in samples without γ/γ' eutectic present, dislocations built up in the γ matrix at the γ' interface and caused shearing of the γ' phase. The γ/γ' eutectic can have a significant effect on the fatigue properties of superalloys, serving as initiation sites for fatigue cracks to develop.[5]

One of the reasons for high strength in nickel-base superalloys is the difficulty in dislocation movement.[2] Impedement of dislocation motion is primarily caused by the highly ordered γ' precipitates. Because of this order, nickel and aluminum atoms occupy specific sites within the crystal. When a half plane is shifted by dislocation movement, the sites that were occupied by an atom of one type are now occupied by the other type atom. This arrangement causes an increase in the energy of the system and makes dislocation movement difficult. A second dislocation would restore the crystals order and would allow the two to move as a pair with a high energy antiphase boundary between them. The movement of a dislocation creates an antiphase boundary, so the energy required to create an antiphase boundary opposes the dislocation movement. A single dislocation can move relatively easily through the γ phase, but becomes pinned by the γ' cuboids making deformation difficult.

<u>Hydrogen Effects</u>

Environmental conditions present in the Space Shuttle Main Engine initialized study of the effects of hydrogen on superalloys many years ago.[1] Several useful mechanisms have been developed to explain the hydrogen embrittlement process. Among these, the most relevant to this study are listed below.

1 Hydride precipitation

2 Trap theory

3 Hydrogen enhanced local plasticity

Borowiecka et al. [6] state that while the existence of the hydride is a necessary, but not sufficient condition for hydrogen embrittlement, it is doubtful that hydride formation is responsible for embrittlement. This statement stemmed from hydride tests on HASTELLOY Alloy C-267 in which their most susceptible forms (cold worked and aged) of the alloy showed less damage than others (cold worked or annealed). Therefore hydride precipitation is not considered in this work.

Trap Theory and Hydrogen Induced Plasticity

When hydrogen is trapped at a site, and it's concentration is sufficient, a crack will initiate.[7,8] A number of microstructural features can serve as trapping sites, and have been observed acting as trapping sites in superalloys, making trapping a relevant mechanism to this research. Among the trapping sites are voids, inclusions, dislocations, and interstitial sites. Hydrogen has been observed to accumulate in the γ/γ' eutectic, either by being trapped or because of increased solubility in the γ/γ' eutectic.[9,10] Studies done by Baker et al. [10] in CMSX-2 nickel-based superalloys found that voids acted as strong hydrogen trapping sites and affected both crack initiation and growth, supplying a source of hydrogen to the crack tip. They also found that cleavage planes were of the (100)-type, likely caused by hydrogen induced localized strain in the γ phase regions. This is in agreement with the hydrogen enhanced localized plasticity model. In this later model, hydrogen is believed to reduce the stress necessary to generate and move dislocations and thus there is high dislocation activity in a small area, leading to local strain. Dislocations have also been observed as trapping sites and also dislocation sweep-in of hydrogen has been observed to allow hydrogen concentration profiles to penetrate much deeper than $\sqrt{Dt}$ diffusion would allow.[7] Extensive hydrogen induced cross slip onto (100)-type planes was also observed in CMSX-2 superalloys.[11] Within uncharged samples, fracture occurred primarily on (111)-type planes by ductile shearing of the matrix.

EXPERIMENTAL PROCEDURE

Hydrogen and helium charged tensile specimens were provided by NASA, Marshall Space Flight Center. Specimens of four different compositions were tensile tested by the Materials and Processes Laboratory at Marshall Space Flight Center. All specimens were grown along the [001] direction. The single crystals had been machined into tensile specimens, 8.5 mm in diameter, 50 mm long with a 6mm notch diameter. Tensile testing was performed at room temperature in both helium and hydrogen at 34 MPa to simulate Space Shuttle Main Engine conditions. Helium was considered to be a hydrogen free environment. Specimen compositions are given in table 1. The deviation of each specimen from the exact [001] direction is given in table 2.

Table 1. Chemical compositions and specimen material.

	Cr	Mo	Co	W	Ta	Ti	Al	Ni
Ge Rene N-4	9.0	1.5	7.5	6.0	4.0	4.2	3.7	balance
CMSX-2	8.0	0.6	5.0	8.0	6.0	1.0	5.5	balance
CMSX-4C*	-	-	-	-	-	-	-	unknown
CMSX-2(ONERA)*	-	-	-	-	-	-	-	unknown

* *The compositions of these samples are not yet known.*

All specimens were reported to have been stress relieved at 850° C for 8 hours in a vacuum. Specimens were prepared for SEM (scanning electron microscope) analysis, including ultrasonic cleaning in acetone and methanol. A Joel 840 scanning electron microscope operating at 20 kv was used for SEM analysis. A stereographic technique developed by Chen[1] was used to determine the angle between the electron beam and the cleavage plane, therefore defining the cleavage plane. A Noran Industries image processing unit in conjunction with the SEM was used to determine relative percentages of primary and secondary cleavage planes, as well as to determine the γ' volume fraction.

Cross-sections were taken below the fracture surface and analyzed for morphology and other microstructural features. Cross-sections were cut using an EDM (Energy Dispersion Mechanism), polished and electrolytically etched using chromatic acid and 5-6 volts for about 6 seconds. After cross-sections showed the existence of a γ/γ' eutectic phase, a Microcomp Image

Analysis System was used to determine the volume fraction of this phase. For this volume fraction and the γ' volume fraction the equation:

$$A_A = V_V$$

was utilized, where A_A represents the area fraction of the desired feature, and V_V represents the volume fraction of the desired feature.

X-ray analysis and Vickor's Microhardness tests were done on the γ/γ' eutectic to verify the γ/γ' eutectic. The fracture surface itself was then cross-sectioned, polished, and etched to try to determine what effects the microstructural features below the fracture surface had on the fracture surface.

RESULTS AND DISCUSSION

Hydrogen Induced cleavage

Table 2 below shows the samples used in this research. Figures 1, 2, 3, 4, 5 and 6 show the fracture surfaces of the specimens tested in helium and in hydrogen. Figures 1, 3 and 5 are those tested in helium, while figures 2, 4 and 6 are the ones tested in hydrogen. The appearance of the fracture surface of the hydrogen charged samples differ greatly from those of the helium samples.

Table 2. Specimen Properties.

GE Rene N-4		CMSX-2		CMSX-4C	
helium	hydrogen	helium	hydrogen	helium	hydrogen
LO63	BF062	#3	#5	AE60	AW60
Deviation from [001] 1.0°	Deviation from [001] 1.5°	Deviation from [001] 5.0°	Deviation from [001] 5.0°	Deviation from [001] 0.0°	Deviation from [001] 0.0°

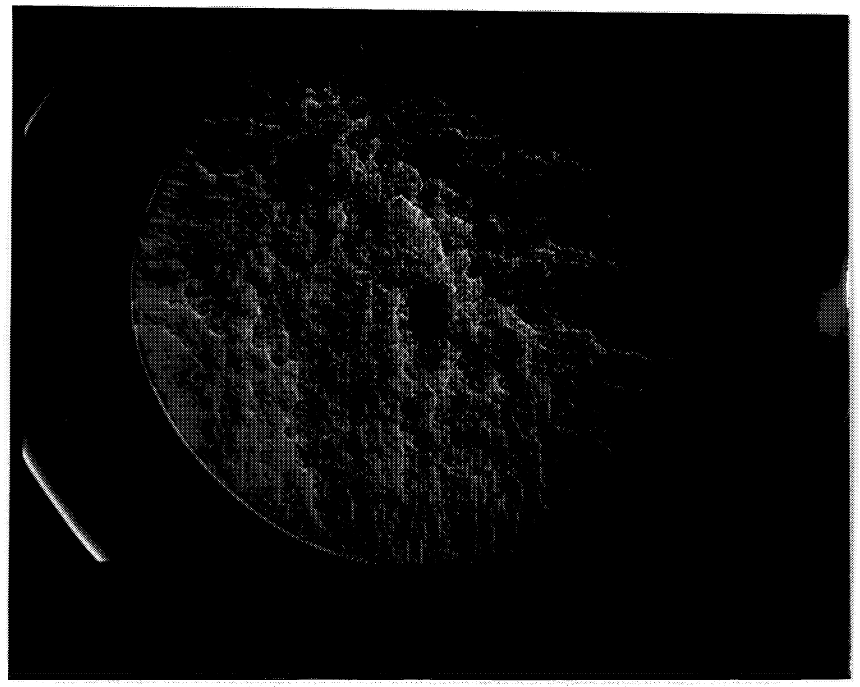
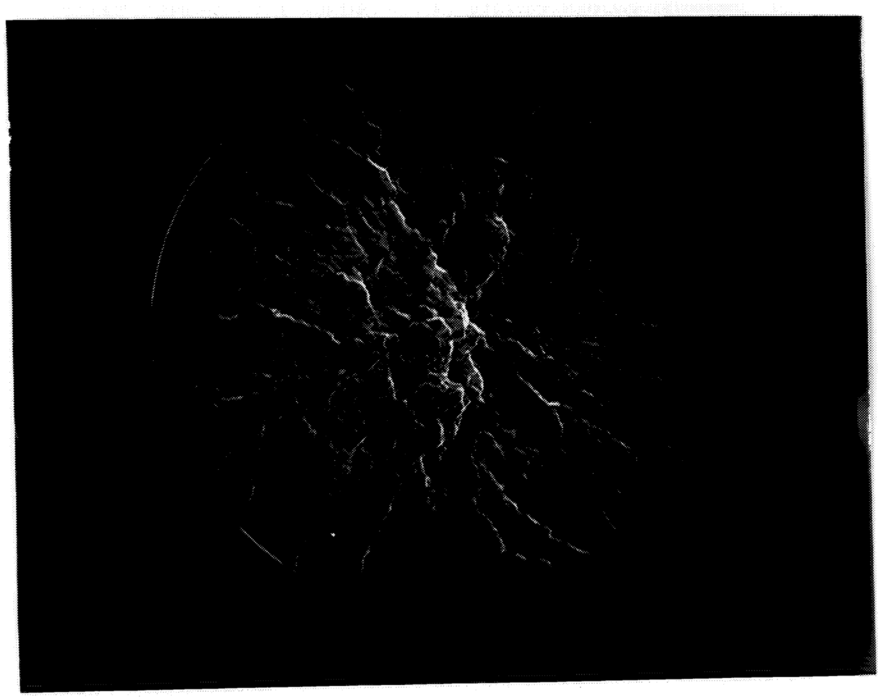


fig 1

⇑ LO63 fractured in helium GE Rene N-4 BF062 fractured in hydrogen ⇓

fig 2



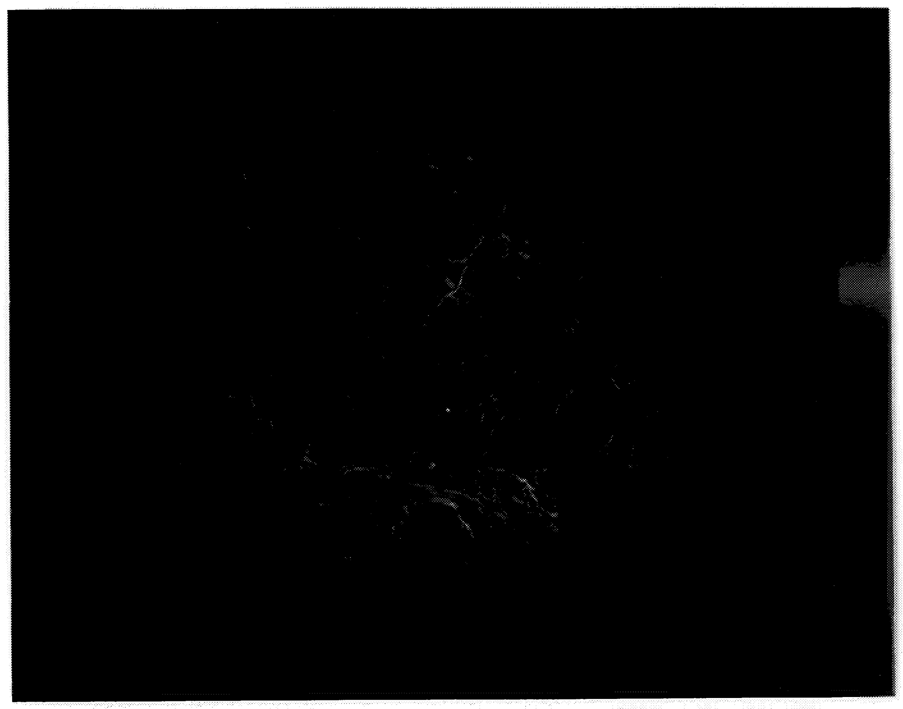
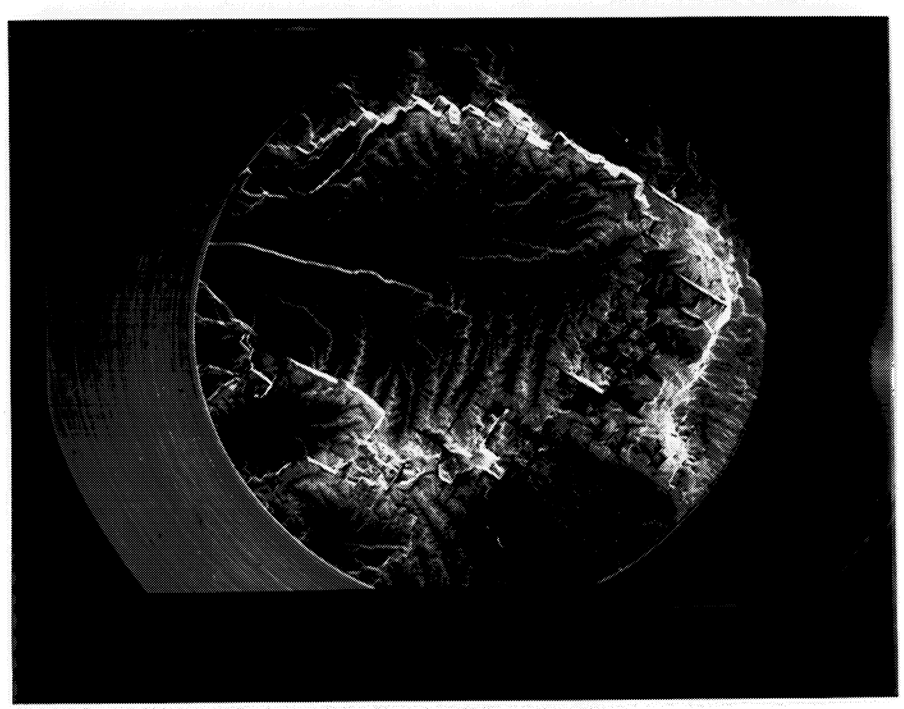


fig 3

⇑ #3 fractured in helium CMSX-2 #5 fractured in hydrogen ⇓

fig 4



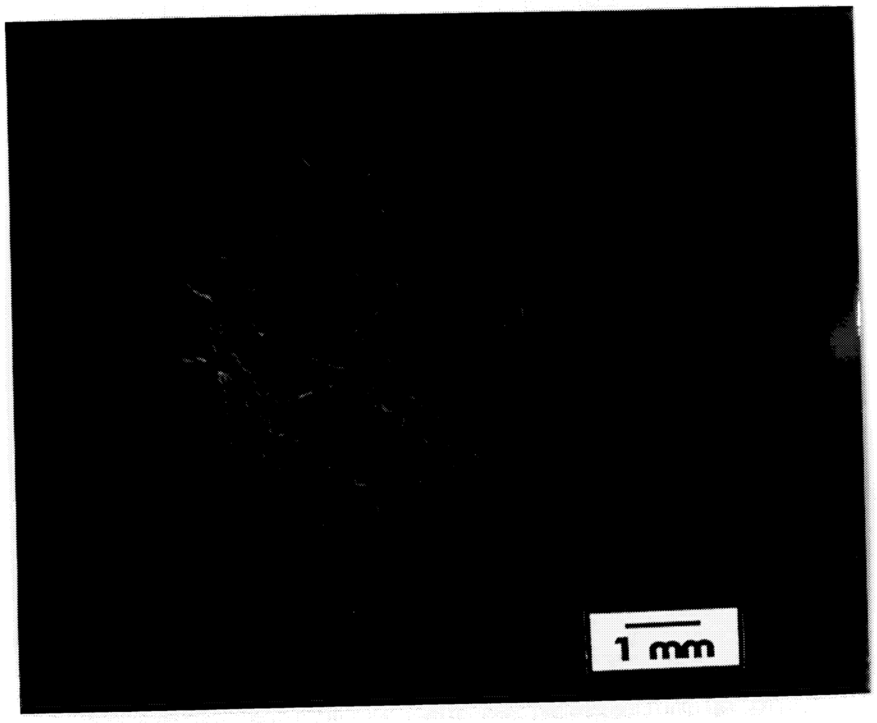


fig 5

⇑ AE60 fractured in helium CMSX-4C AW60 fractured in hydrogen ⇓

fig 6

Helium samples

All crystals fractured in helium showed large cleavage planes at the notch. These cleavage planes became smaller and smaller as they moved towards the crystal axis. Planes greater than 5 μm were analyzed using the stereographic technique for quantitative analysis of cleavage plane orientation developed by Chen.[2] The larger analyzable planes were mainly restricted to the notch region. Primary cleavage planes were found to be of the (111)-type which is consistent with the literature. However, there were planes found in the notch area and in the interior that were of the (110), (210) and (320)-types. This differs somewhat from Chen's[2] PWA 1480E samples tested in helium, which were predominantly (111)-type. Table 3 shows the angle between these planes and the [001] direction and is the angle given when applying the stereographic technique developed by Chen.[2] corresponding to these planes. Figure 7 shows planes of these types in the GE Rene N-4 LO63 sample, initiating at the notch and becoming smaller as they move toward the crystal axis. Plane A is of the (111)-type, planes B and D are of the (110)-type, and plane C is of the (320)-type. Figure 8 shows the triangular morphology of a (111)-type plane (plane A) in figure 7. This is in agreement with the literature which indicates that non-hydrogen charged samples fracture primarily on (111)-type planes by ductile shearing of the matrix.[4,10,11]

PLANES	ANGLE
(100)	0°
(110)	45°
(111)	54.74°
(210)	26.56° or 63.43°
(320)	33.69° or 56.31°

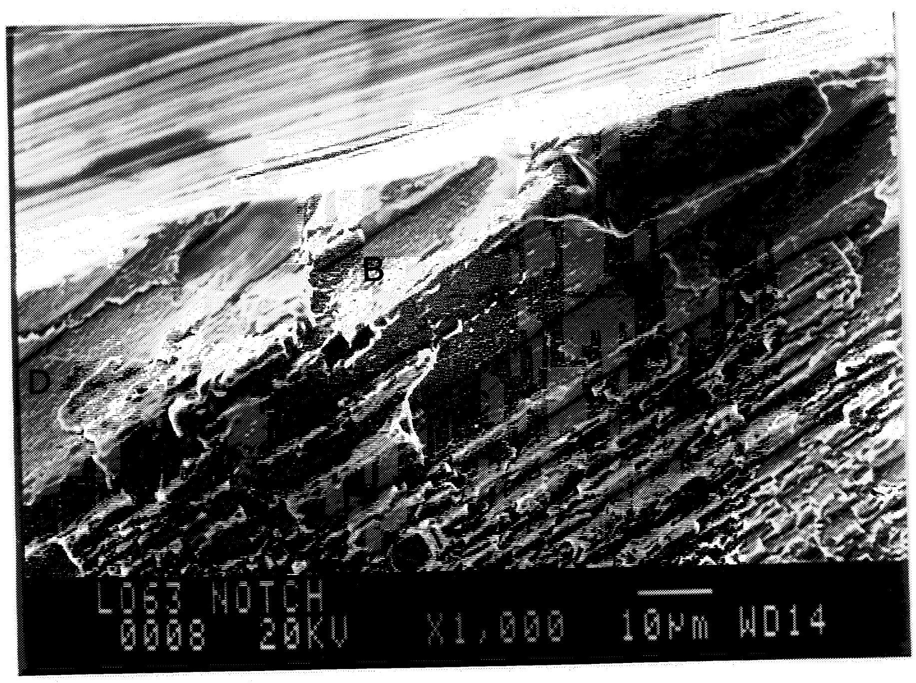
Table 3. Angles between planes and the [001] direction.

Hydrogen samples

Hydrogen samples all had a more brittle appearance than the helium samples. This is especially true for the CMSX-2 #5 and CMSX-4C AW60 samples which had large, shiny areas perpendicular to the growth direction. These two crystals also had a feathery appearance as seen in figures 4 and 6. The feathery appearance begins at the notch and extends into the interior regions of the crystal. This differed greatly from Chen's[2] work in that none of his samples had the large feathery areas shown in figures 4 and 6. The feathery appearance was caused by an inter-network of (210)-type planes. The Ge Rene

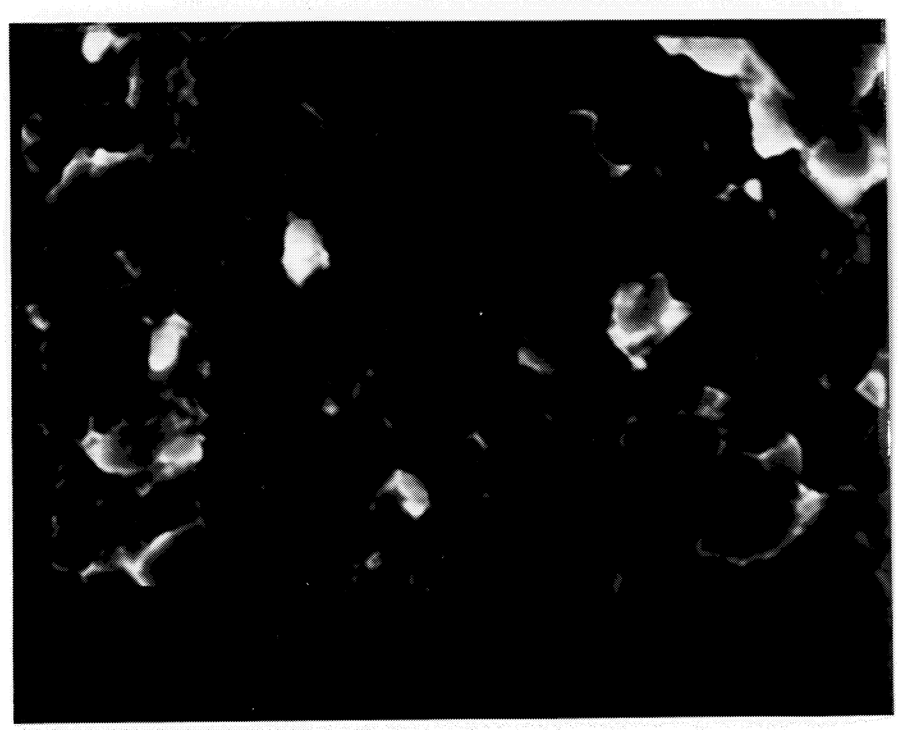
N-4 (BF 062) crystal had (100)-type cleavage planes starting at the notch and moving toward the crystal axis around the whole circumference. This region of (100)-type planes extended 1.5-1.8 mm from the notch, toward the crystal axis. The primary plane of cleavage in all three samples was the (100)-type, with some (111), (210) and (320) present. Figure 9 shows the morphology of (100)-type planes in the Ge Rene N-4 (BF 062) crystal. In all three samples, 70-80% of the fracture surface was comprised of cleavage on (100)-type planes. All three samples showed numerous large cracks, all oriented 90° to each other, caused by hydrogen embrittlement. Figures 10 and 11 show some of these cracks. The (100) type cleavage is consistent with cross-slip reported in the literature.[4,10,11]

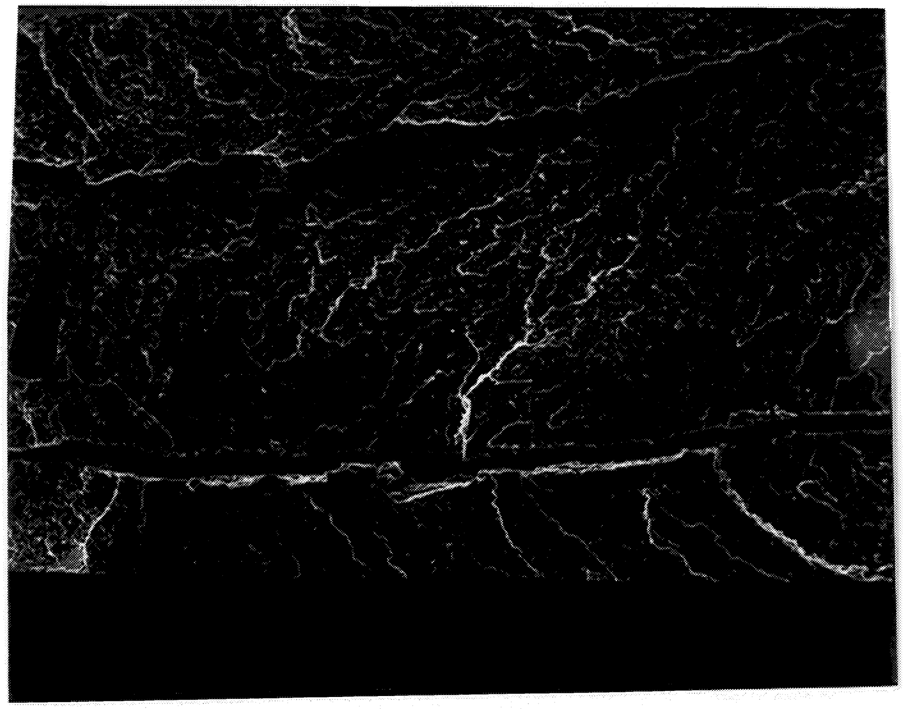
fig 7 LO63, planes near notch region ⇓



⇑ fig 8 LO63 morphology of plane A above

fig 9 morphology of BF062 fractured in hydrogen ⇓





⇑ fig 10 cracks in BF062 fractured in hydrogen

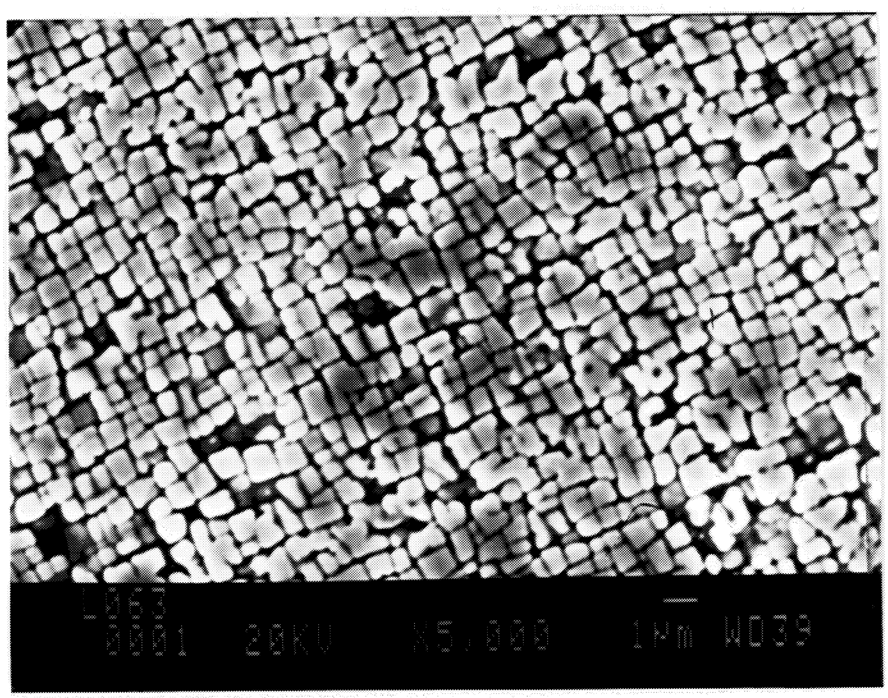
γ/γ' Eutectic

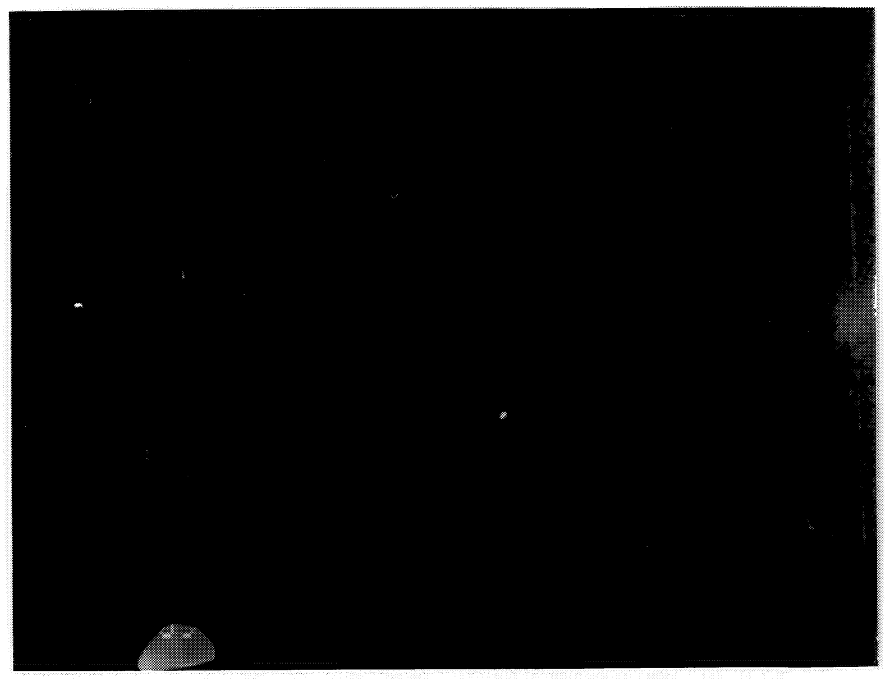
On viewing the etched cross-sections (figure 12) of various samples, an irregular phase was found to be present in the microstructure (figure 13). After searching the literature this phase was thought to be γ/γ' eutectic. X-ray analysis revealed a single peak, shown in figure 14. This single peak was evidence that the composition of the irregular phase was similar to the γ' and γ matrix. Microhardness tests revealed that the phase had relatively the same hardness as the rest of the microstructure. From the literature[1], γ/γ' eutectic can be one of two different forms; that which forms at high temperature and has a lacelike appearance, and that which forms at a lower temperature and is more uniform, consisting mainly of γ'. Figure 13 shows that the irregular phase is uniform and resembles the γ' phase. The x-ray analysis and the microhardness tests also point to this irregular phase as being γ/γ' eutectic.

While the purpose of this research is not to compare fractures of specimens containing γ/γ' eutectic with those not containing γ/γ' eutectic, it is clear from the literature[4,5,9] that this constituent will affect the fracture behavior of the material.

⇑ fig 11 cracks in #5 fractured in hydrogen

fig 12 regular cross-section showing γ and γ' ⇓



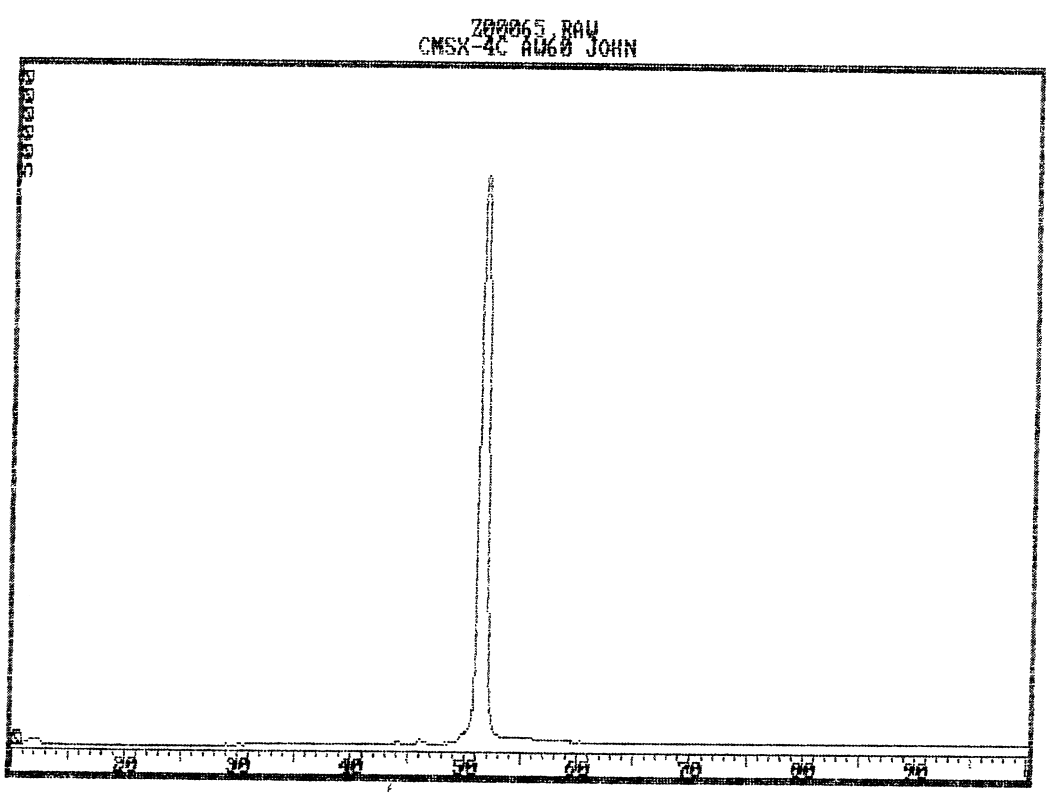


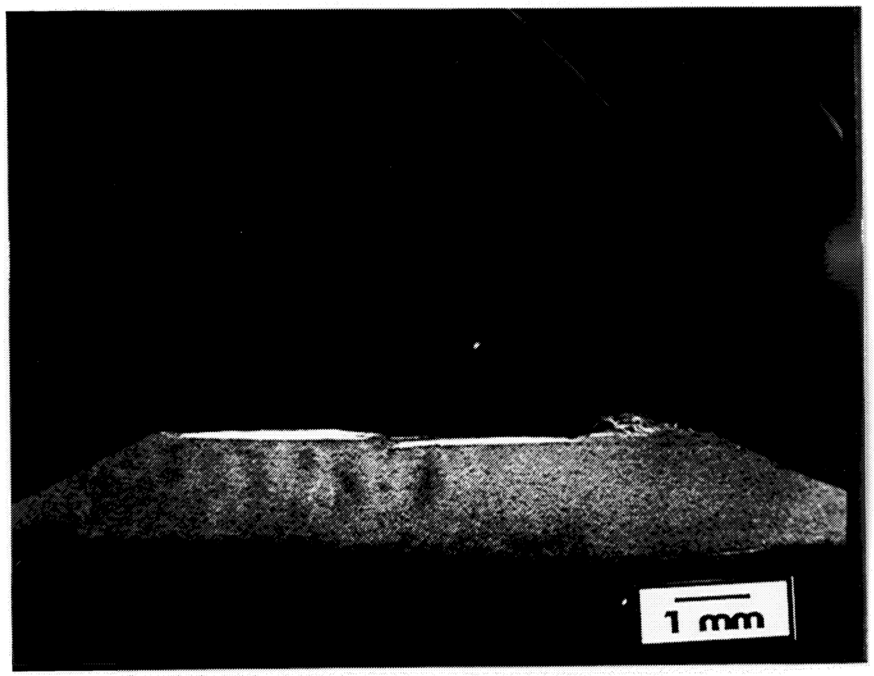
⇑ fig 13 showing γ/γ' eutectic

Quantitative analysis of the cross-sections revealed the GE Rene N-4 samples (table 2) contained 3-5% volume fraction of γ/γ' eutectic as did the CMSX-4C samples. The CMSX-2 #3 sample (fractured in helium) contained no γ/γ' eutectic, while the CMSX-2 #5 sample (fractured in hydrogen) contained an almost undetectable amount. The question of why the GE-Rene N-4 and CMSX-4C samples contain γ/γ' eutectic and CMSX-2 samples do not, could possibly be explained by composition differences. However, it is also noted that these specimens were stress relieved at 850° C for 8 hours, while Walston[4] used a 20 hour heat treatment at 1288° C to remove the γ/γ' eutectic from his PWA 1480 Superalloy.

On analyzing the cross-section of the fracture surface (figure 15), γ/γ' eutectic was found at the fracture surface (figure 16 and 17). From the literature[4] this region of γ/γ' eutectic probably was a source of crack initiation. Undoubtedly, the interface is a source of dislocation pile up, but transmission electron microscopy work would be required to prove that dislocations piled up at this interface and initiated cracking. Figure 18 shows a crack running perpendicular from the fracture surface, and another crack running perpendicular to the initial one. Figure 19 was taken at the end of the second crack in figure 18 and shows that the crack ends at a γ/γ' eutectic interface. While not relevant to the fracture surface itself, the γ/γ' eutectic in figure 19 showed evidence of cracks within itself. Furthermore, the second crack likely acted as a source of relief for the stesses built up around the γ/γ' eutectic interface by dislocation pileup. The mechanism for fracture in the γ/γ' phase is believed to be very similar to that of the shearing of the γ' phase. Walston[4] noted that both the γ' phase and the γ/γ' eutectic fracture in a brittle manner, and that the dislocation densities were higher at the γ/γ' eutectic interface, similar to the dislocation buildup that occurs in the γ matrix around the γ'. This similarity is feasible because the γ/γ' eutectic is mostly γ' and the eutectic should thus behave similarly to γ'.

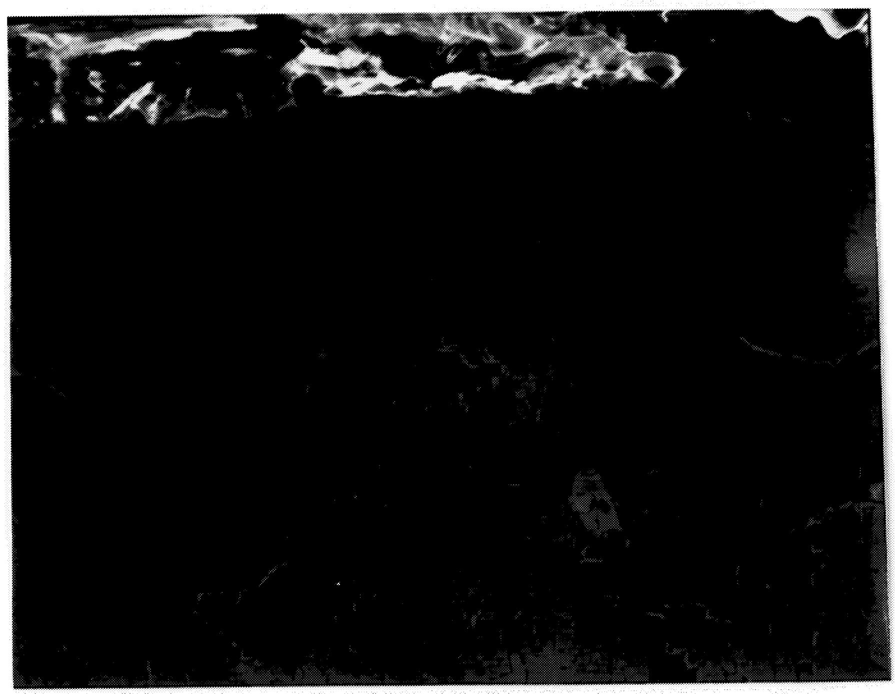
fig 14 x-ray analysis of Ni-based single crystal superalloy ⇓





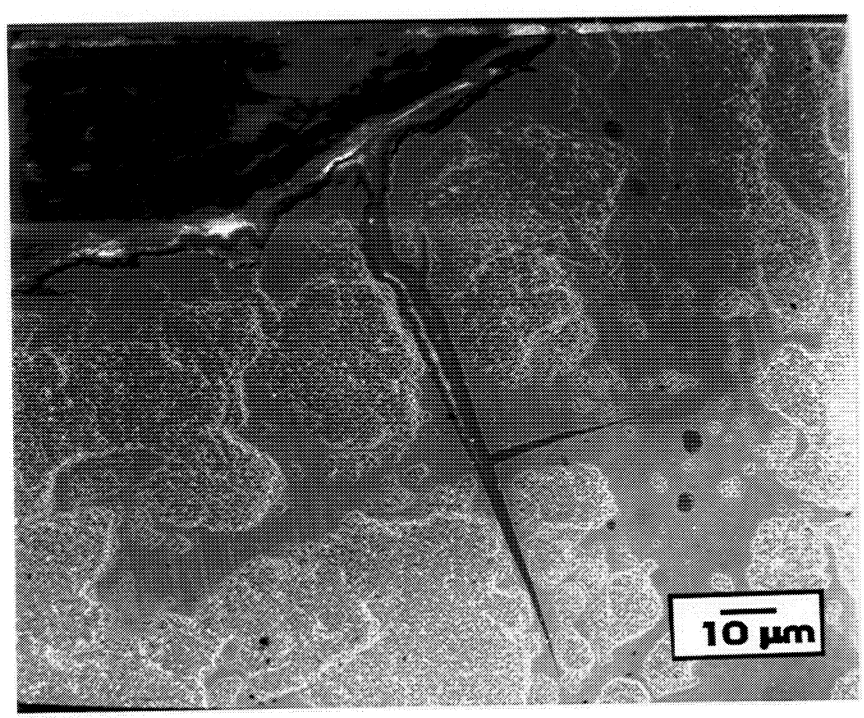
⇑ fig 15 cross-sections of fracture surface of AW60

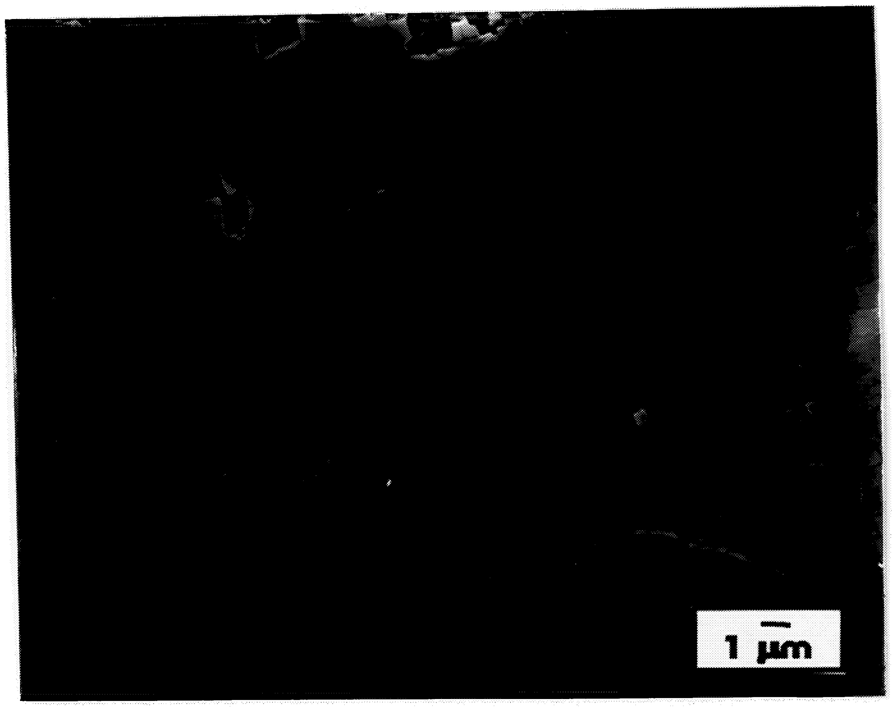
fig 16 shows γ'/γ' eutectic at fracture surface ⇓



⇑ fig 17 shows γ'/γ' eutectic at fracture surface

fig 18 shows cracks running down from fracture surface ⇓





⇑ fig 19 γ/γ' eutectic at tip of crack in fig 18

CONCLUSIONS

- From comparisons of the specimens fractured in hydrogen to those in helium, the mechanisms involved in fracture are very different.
    - ⇒ Helium samples fractured mainly by (111)-type cleavage. Cleavage was believed to be induced by shearing of the γ' precipitate after dislocation build up in the surrounding γ matrix. Planes were larger at the notch region, growing smaller moving toward the crystal axis.
    - ⇒ Hydrogen samples fractured mainly on (100)-type cleavage planes. Cleavage was believed to be induced by γ' shearing, and cross-slip was believed to have allowed dislocations to move on (100)-type planes. Large areas of (100)-type planes, perpendicular to the growth direction and appearing feathery, were present. Heavy cracking along [100]-type directions occurred. Microstructural features were thought to act as trapping sites for hydrogen, thus supplying crack initiation sights.

- The γ/γ' eutectic interface is thought to be an area of dislocation pile-up, leading to stresses and a possible initiation site for cracks. γ/γ' eutectic was found quite often intersecting the fracture surface of the GE Rene N-4 samples and the CMSX-4C samples.

Future Work

More samples of the same compositions will be analyzed. The samples analyzed so far are ones that are reported to have been the closest to the [001] growth direction. Also samples from the CMSX-2 (onera) group will be analyzed.

In the future work, transmission electron microscopy should be utilized to study the relationships of the γ/γ' eutectic to the fracture surfaces by looking at dislocation interactions at the interface. Proof of the mechanisms thought to be involved in the fracture behavior needs to be obtained through transmission electron microscopy work.

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