SINGLE CRYSTAL PWA 1472

IN HIGH PRESSURE HYDROGEN

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Abstract

γ' strengthened Ni base single crystal alloys represent the state of the art for hydrogen fueled rocket engine turbopump airfoils. These alloys are however, susceptible to hydrogen embrittlement, degrading fatigue and fracture capability by orders of magnitude. Post test fractographic analysis of PWA 1490 (HIP Microcast INCO 718) tested in high pressure hydrogen has shown that the mechanisms that degrade the γ' superalloys are not operative in the INCO 718 class of alloys. This set of circumstances suggests that a high strength INCO 718 variant, PWA 1472 cast in single crystal form, might be superior to γ' strengthened single crystals in low temperature rocket turbopumps where safety and durability are principal considerations. To test this hypothesis PWA 1472 was cast in single crystal form and tested in air and hydrogen.

Superalloys 718, 625, 706 and Various Derivatives
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Introduction

Nickel base superalloys are the materials of choice for liquid hydrogen fueled rocket engine turbopumps. They are used extensively in current and planned (1) versions of the NASA Space Shuttle Main Engine (SSME). Two principal superalloy classes and their response to hydrogen are of concern in this paper.

Fundamental aspects of alloy structure play an important role when considering hydrogen embrittlement mechanisms. Cast γ' strengthened alloys such as single crystal (SC) PWA 1480 and equiaxed MAR-M-247 are used in turbopump hot section applications where temperatures approach 900°C. Columnar grain directionally solidified (DS) castings or (SC) forms are preferred for turbine blades. Equiaxed (EQ) castings are used in vane applications. The γ' strengthening precipitate in these alloys is composed of alloyed Ni_3Al with $L1_2$ order. It assumes a cuboidal morphology geometrically ordered in the γ matrix. The cube edges are aligned with the <001> directions. The precipitate structure of PWA 1480, a cast high volume fraction γ' (approximately 60 vol. %) strengthened alloy is shown in Figure 1.

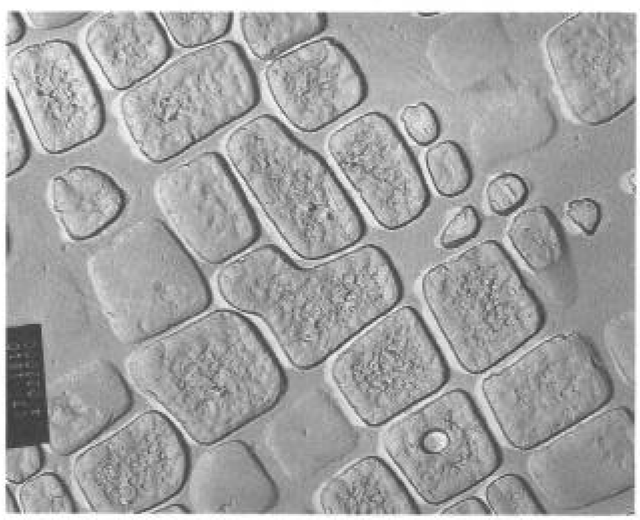


Figure 1. The cuboidal precipitates in a cast high volume fraction γ' strengthened alloy are geometrically ordered, coherent with the <001> directions. 22000X

The γ'' precipitation strengthened alloys, (INCO 718) find use in many structural applications such as pump housings and flanges. Their service temperature is generally limited to 650 °C. They are used exclusively in equiaxed form, wrought or cast. The primary strengthening precipitate in these alloys is γ'' (ordered Ni_3Cb) and assumes a lenticular morphology. γ'' exhibits both atomic ($D0_{22}$) and geometric order, coherent with the <001> directions. They are much finer than those found in cast γ' strengthened alloys and of a lower volume fraction. The γ'' structure of PWA 1490 is shown in Figure 2.

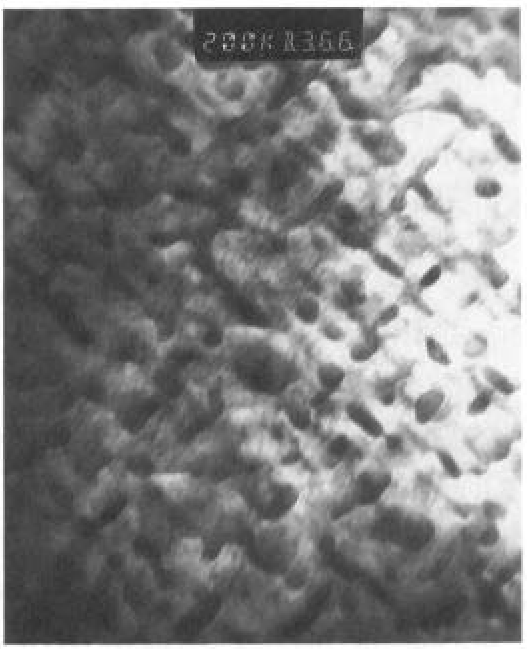


Figure 2. The lenticular Ni_3Cb precipitates in a cast γ'' strengthened alloy are also geometrically ordered, coherent with the <001> directions. 200,000X

Many turbopump components are exposed to high pressure gaseous hydrogen during operation and the mechanical properties of nearly all of the materials employed, are substantially degraded by this exposure. A description of some of the degradation mechanisms affecting these materials is provided in the following section.

Materials and Degradation Mechanisms

The effects of hydrogen on the fatigue and fracture characteristics of Ni base superalloys have been the subject of intensive research for decades. (2-4). The mechanisms of hydrogen degradation vary depending on, among other things, the alloy class (γ', γ''), casting form (DS, SC, EQ) and the particular mechanical property in question (fatigue, crack growth). Frequently degradation is the result of a microscopic fracture mode transition resulting from hydrogen induced changes in dislocation mobility (5-6).

Recent studies (7) conducted at Pratt & Whitney examined hydrogen degradation mechanisms in several cast γ' and γ'' strengthened alloys. One of these investigations focused on PWA 1489 and PWA 1490. Low cycle fatigue (LCF) and fatigue crack growth (FCG) specimens tested in a high pressure (34.5 MPa) hydrogen environment were compared to specimen fractures produced in air. Test results indicated that the maximum hydrogen to air debit for these materials occurs at 26°C. Notched LCF tests were the most sensitive indicator of degradation so these specimens were chosen for review in order to obviate any differences between air and hydrogen fracture details.

The γ' strengthened equiaxed alloy PWA 1489, a HIP'd Microcast version of Mar-M-247, was found to experience a fracture mode transition from Stage I transgranular fracture in air to predominantly intergranular fracture in hydrogen. In addition, isolated areas of γ - γ' decohesion were observed at fatigue origins.

γ-γ' decohesion is a fracture mode that occurs in the near threshold region of fatigue crack growth when testing in room temperature air (8). It is also observed in specimen fractures produced at room temperature in hydrogen environment testing where it remains operative at much higher stress intensities (7). It is associated with large increases in FCG in single crystal and columnar grain turbine blade alloys when tested in hydrogen. A high magnification view of γ-γ' interfacial failure (decohesion) in a single crystal alloy is provided in Figure 3.

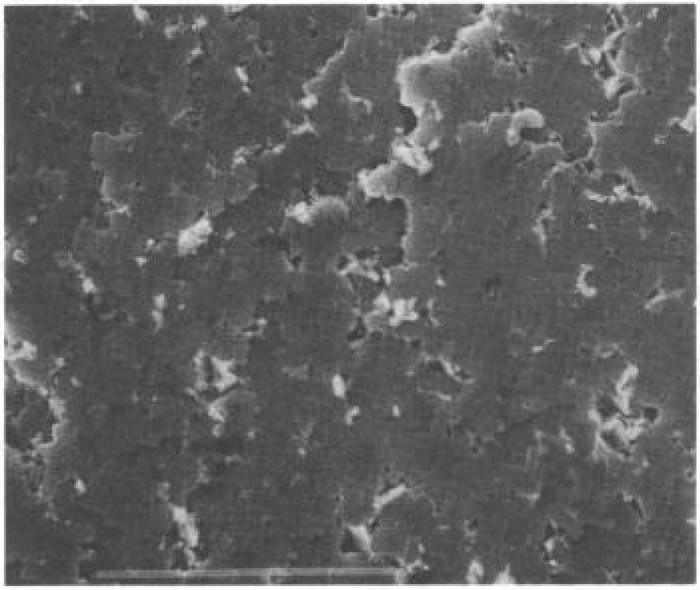
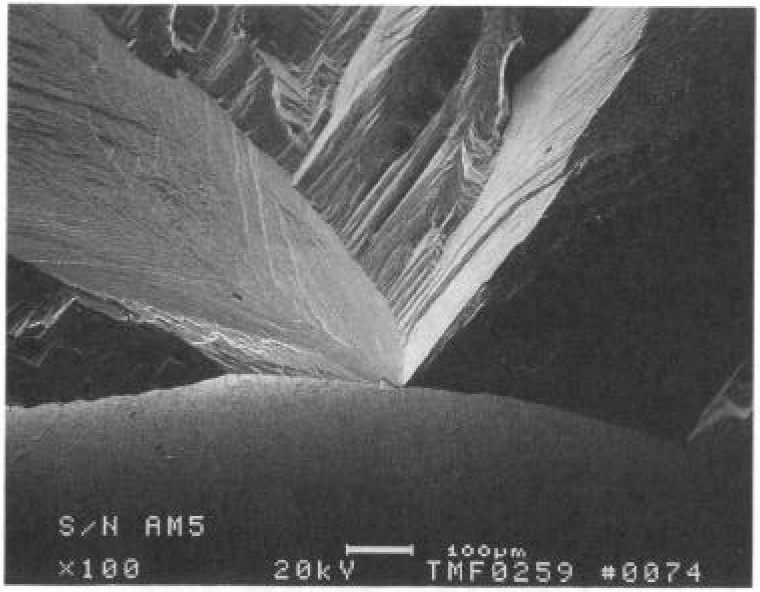


Figure 3. A high magnification view of γ-γ' interfacial failure (decohesion) in a single crystal alloy tested in 34.5 MPa hydrogen. 4400X

Decohesion has been shown to be sub microscopic (111) fracture confined to the γ matrix phase (6). The result is a separation at the γ-γ' interface. The normal fracture mode observed in air is by shearing of γ' precipitates on (111) planes. A macroscopic comparison of the two modes is seen in Figure 4.

The γ'' alloy , PWA 1490, also experienced the transition to intergranular fracture in the presence of hydrogen but did not show a tendency to fail by matrix / precipitate decohesion.

This observation suggested that in the absence of transverse grain boundaries (i.e. cast as a single crystal) INCO 718 would not be susceptible to one of the principal forms of degradation that γ' blade alloys suffer.



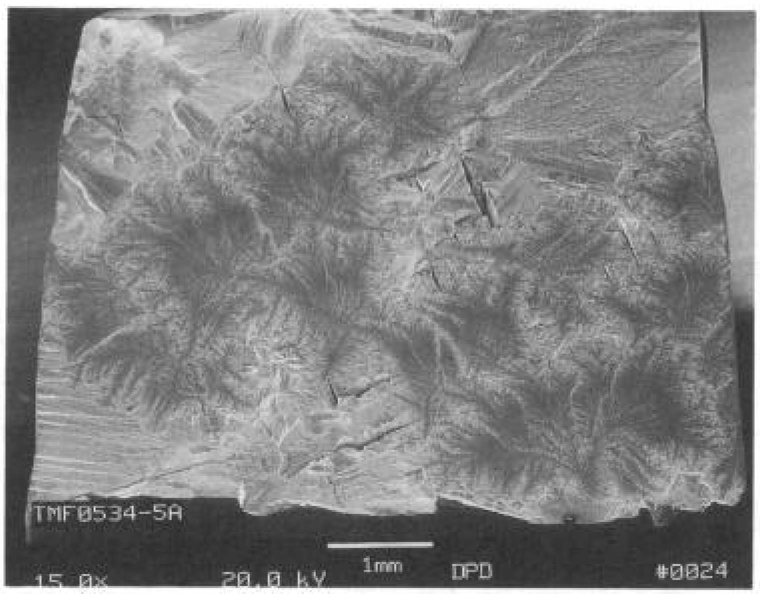


Figure 4. A macroscopic view of fatigue fracture surfaces from γ' strengthened single crystal specimens tested in air (at top) versus 34.5 MPa hydrogen (bottom).

Experimental Program

PWA 1472 is a high strength variant of INCO 718. Room temperature strength (in equiaxed form) compares favorably with (SC) PWA 1480, a candidate material for the NASA SSME liquid hydrogen fueled rocket engine.

Based on the hypothesis that a single crystal γ'' strengthened alloy would not experience a hydrogen induced fracture mode transition, PWA 1472 was cast in single crystal form to study the effect of hydrogen on fracture behavior.

A mold of cast test bars of PWA l472 was produced in single crystal from. The bars were 0.5 cm in diameter and approximately 10 cm long. The material was homogenized at 1225 °C for 4 hours followed by a fan cool to room temperature. Precipitation was carried out at 760 °C for 8 hours. The resultant precipitate structure was examined via replica transition electron microscopy and will be discussed in later.

Notched LCF tests were selected to test the hypothesis for reasons cited earlier and because the air to hydrogen debit for this property is known for a variety of superalloys. LCF tests were conducted at 26 °C with a stress ratio of 0.05 ($\sigma_{min}/\sigma_{max}$) at 0.17 Hz. Cylindrical gage notched LCF specimens (K_t =2.16) were tested at a net section stress of 620.5 MPa in air and in 34.5 MPa gaseous hydrogen.
Life (cycles) to failure was determined in air and hydrogen and the ratio of air to hydrogen fatigue life was determined. Post test fractographic analysis included optical and scanning electron microscopy.

Results and Discussion

Single crystal PWA 1472 did not exhibit a hydrogen induced fracture mode transition. The microscopic fracture mode in both air and hydrogen was crystallographic along (111) octahedral planes. A comparison of fatigue fracture surfaces from single crystal PWA 1472 specimens tested in air and in 34.5 MPa hydrogen is shown in Figure 5. The air to hydrogen life ratio was found to be 5X, significantly lower than the 100X ratio observed in PWA 1480.

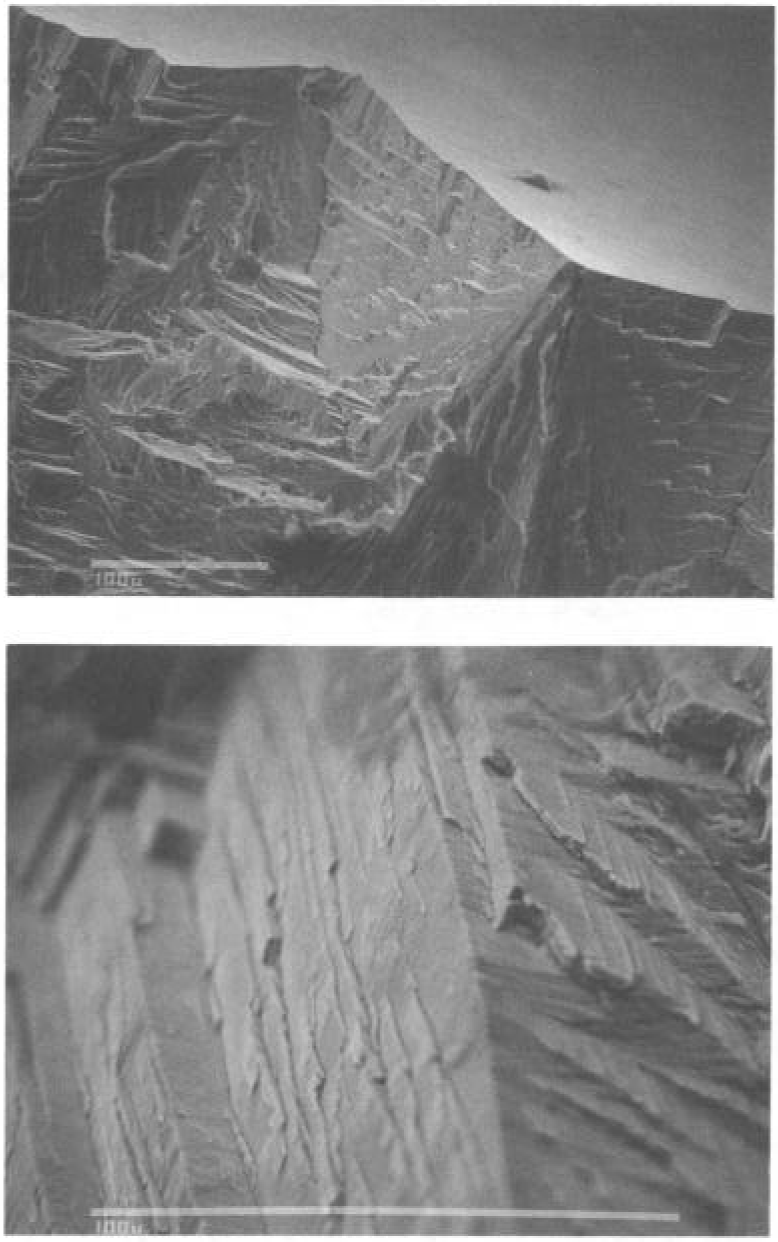


Figure 5. Fatigue fracture surfaces from single crystal PWA 1472 specimens tested in air , 260X (top) versus 34.5 MPa hydrogen, 860X (bottom).

Notched LCF life for SC PWA 1472 in hydrogen was greater than that of PWA 1480. This finding supported the original hypothesis, that in the absence of a fracture mode transition there would be little or no life debit between air and hydrogen. Unexpectedly, when tested in air the LCF life of SC PWA 1472 was lower than PWA 1480. This finding appears to violate assumption that no appreciable strength difference would exist between PWA 1480 and SC PWA 1472. As such, SC PWA 1472 LCF life in air would be comparable to PWA 1480. That assumption was based on previous equiaxed PWA 1472 experience. It is likely that the two step heat treatment described earlier did not produce the strength expected. The heat treatment cycle was chosen partly based on PWA 1490 experience and was expected to produce a γ'' size and volume fraction of uniformly fine γ'' similar to PWA 1490 for maximum strength.

The cycle was also based on experience gained with grain boundary precipitation of δ phase during the development of EQ PWA 1472. The heat treat cycle was chosen to minimize the precipitation of δ phase and other potential interdendritic segregants. Although δ phase is desirable in an equiaxed alloy for strengthening grain boundaries, it was felt to be unnecessary in a monograin casting. δ phase could also become a preferential fatigue crack initiator in hydrogen (if not in air).

Tensile testing is planned and should resolve this question. Pending those results it is assumed that the apparent low γ'' volume % and coarse γ'' size affected strength and therefore fatigue life in air. If so, volume fraction and precipitate size can be optimized through further heat treatment development.

Summary Conclusions

PWA 1472 did not undergo a fracture mode change in hydrogen and the hydrogen to air fatigue debit was much lower for PWA 1472 than PWA 1480.

PWA 1472 LCF was superior to PWA 1480 when compared in hydrogen but PWA 1480 LCF was superior to PWA 1472 when compared in air.

Potential applications for a single crystal γ'' strengthened alloy would be in hydrogen fueled rocket engines. γ' strengthened blade alloys like PWA 1480 were developed for much higher (sustained) temperatures than those present in rocket turbine applications but they are susceptible to preferential matrix failure in hydrogen. This appears not to be the case with PWA 1490 and SC PWA 1472 however more specimen testing is needed to investigate elevated temperature exposure effects.

It is also likely that an alloy with a similar composition to SC PWA 1472 could be tailored for SC form by the elimination of grain boundary strengtheners.

This and heat treatment optimization are logical next steps toward a more capable alloy with a higher service temperature limit.

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