HIGH-PRESSURE HYDROGEN TESTING
OF SINGLE CRYSTAL SUPERALLOYS FOR ADVANCED
ROCKET ENGINE TURBOPUMP TURBINE BLADES

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A screening program determined the effects of high-pressure hydrogen on selected candidate materials for advanced single crystal turbine blade applications. The alloys chosen for the investigation were CM SX-2, CM SX-4C, and PWA 1480. Testing was carried out in hydrogen and helium at 34 MPa and room temperature, with both notched and unnotched single crystal specimens. Results show a significant variation in susceptibility to Hydrogen Environment Embrittlement (HEE) among the four alloys and a marked difference in fracture topography between hydrogen and helium environment specimens.
INTRODUCTION

The selection of the present SSME turbine blade alloy, MAR-M246(Hf)DS, was based to a great extent on its performance in material development programs for aircraft gas turbines. However, the operating environment of a rocket engine turbine requires material properties which differ in certain respects from those required for an aircraft gas turbine. Candidate materials for the next-generation SSME turbine blades are being evaluated by criteria more specific to their unique application.

One significant difference between rocket engine turbines and aircraft gas turbines is the use of hydrogen fuel rather than petroleum distillates. This creates an aggressive rocket engine operating environment in which high strength materials may exhibit hydrogen environment embrittlement (HEE). Until recently, however, little attention was given to the effect of a hydrogen environment on the mechanical properties of turbine blade materials. As part of the effort to select candidate materials best suited for the rocket engine, Marshall Space Flight Center initiated a contract with Oak Ridge National Laboratory to establish facilities for high pressure hydrogen testing.

OBJECTIVE

This program was designed strictly for screening purposes. Its goal is to rank the performance of candidate single crystal turbine blade materials under hydrogen conditions. Although researchers are attempting to understand the mechanism and the factors that influence hydrogen embrittlement of various alloys, as yet there is insufficient information to predict HEE susceptibility. Consequently, a hydrogen screening program such as this is indispensable.
EXPERIMENTAL PROCEDURE

The four materials chosen for the study, PWA 1480, CM SX-2, CM SX-4C and Rene N-4, are all single crystal superalloys which have demonstrated good mechanical performance in air. All were received in a heat-treated condition. The CM SX-2 and CM SX-4C material had received the standard heat treatment rather than the recently developed ONERA heat treatment. The PWA 1480 material had been heat treated according to Pratt and Whitney's modified regime, and the Rene N-4 received its normal heat treatment.

The test apparatus (Fig. 1) is located in an underground pit, with only the servo-hydraulic control instrumentation (a standard MTS system) and the hydrogen supply on the ground floor above. The system may be isolated, evacuated and purged with helium to flush out air and moisture. Then hydrogen or inert test gas is introduced and raised to 34 MPa within the autoclave by means of a booster pump. The specimen is mounted in the specimen grips; these, in turn, are mounted to the cap of the autoclave. A spherical bushing is used to maintain specimen alignment.

Both smooth and notched specimens of each alloy were tested (Fig. 2). The notched configuration was designed with a stress intensity factor of 8.0. All specimens were stress relieved at 850°C for 8 hours.

Three specimens of each alloy were tested in each of the categories in the test matrix (Table 1). Crosshead speed in all cases was $2.12 \, \mu \text{ms}^{-1}$, and the helium and hydrogen tests were conducted at 34 MPa and room temperature. The results which follow are the mean value of the three tests. In most cases, the coefficient of variation is less than 5%, indicating good consistency.
RESULTS

Smooth tensile testing shows only a small difference between the helium properties and those obtained in air for both ultimate and yield strength (Table 2). The difference between the UTS values and yield strength is an indicator of the alloy's ductile behavior in helium. Hydrogen yield strength is not significantly different from that in helium; but the impact of the hydrogen environment is seen in the ultimate tensile strength, which is virtually identical to yield strength in hydrogen. This represents a severe loss of ductility. As soon as the specimens begin to yield, they fail. In the smooth tests, all alloys performed comparably, showing no obvious ranking of performance among the four alloys.

Reduction in area measurements on the smooth specimens reveal the extent of the ductility loss in HEE: approximately 50% in most cases. But it is in the notched strength that the difference among the alloys is clearly seen (Table 3). When the results are expressed as a ratio, the degradation of performance in hydrogen is most apparent for standard heat treated CM SX-2, and the alloys are easily ranked, with PWA 1480 showing the least loss of ductility.

An inspection of the fracture surfaces supports this ranking (Fig. 3). The high-pressure helium notched fracture surfaces show very little difference among the alloys. They are all predominantly ductile, reflecting the similar notched strengths. However, the hydrogen surfaces clearly differ. CM SX-2 shows an almost entirely brittle fracture, representing nearly instantaneous failure. As the notched ratio increases, the fracture surfaces show increasing areas of ductility.
A closer look at the fracture surfaces by the scanning electron microscope shows some ductility at both the edge and the center of the CM SX-2 specimen in helium (Fig. 4). In hydrogen, the surface is cleavage fracture. Note the orthogonal cracking in the last-to-fail region, correlating to a crystallographic fracture plane. In helium, the fracture surface of CM SX-4C is similar to that of CM SX-2 (Fig. 5). All the samples tested in helium show similar characteristics. In the hydrogen specimen both cleavage and ductility can be seen, corresponding to this alloy's better notch ratio. The properties of Rene N-4 are better still; cleavage is apparent at the edge, with a larger ductile area in the center (Fig. 6). Cleavage is less pronounced in PWA 1480, even at the edge (Fig. 7). Thus, there is a direct correspondence between the notched ratio and fractography of a specimen, and material performance in HEE can be clearly ranked.

CONCLUSIONS

This screening program is not conclusive in that it is not yet possible to specify one recommended alloy for use in SSME turbine blades. Work by other researchers has indicated that the ONERA heat treatment may significantly improve hydrogen properties; therefore it would be premature to rule out the alloys in this study which showed poor performance. However, several conclusions can be drawn at this point. First, susceptibility to HEE differs dramatically among superalloys. Furthermore, mechanical testing in atmospheres other than hydrogen yields no useful information on predicting material performance under actual engine operating conditions. Therefore, hydrogen testing is essential in an SSME turbine blade super-alloy screening program.
REFERENCES


Fig. 1

FLOW SCHEMATIC FOR HIGH PRESSURE MECHANICAL PROPERTIES TEST FACILITY AT OAK RIDGE NATIONAL LABORATORY

(GROUND FLOOR)

(AUTOCLAVE PIT)

TEST GAS

SYSTEM ISOLATION

VENT

HIGH PRESSURE

PURGE

BOOSTER PUMP

VACUUM PUMP

AUToclAVE 5000 psi 75°F

EXHAUST

RUPTURE DISK

REMOTE OPERATED MANUAL VALVE
Fig. 2

TENSILE
SPECIMENS

NOTES
1. ALL DIAMETERS CONCENTRIC WITHIN .005 IN.
2. ALL DIMENSIONS IN INCHES
3. SLIGHT TAPER TO CENTER PERMITTED
4. DO NOT UNDERCUT RADIUS

NOTCH DETAIL - 10X

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Table 1
TEST MATRIX

<table>
<thead>
<tr>
<th>SPECIMEN</th>
<th>ENVIRONMENT</th>
<th>TEMPERATURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>NOTCHED</td>
<td>He</td>
<td>R. T.</td>
</tr>
<tr>
<td>NOTCHED</td>
<td>H₂</td>
<td>R. T.</td>
</tr>
<tr>
<td>SMOOTH</td>
<td>He</td>
<td>R. T.</td>
</tr>
<tr>
<td>SMOOTH</td>
<td>H₂</td>
<td>R. T.</td>
</tr>
<tr>
<td>SMOOTH</td>
<td>AIR</td>
<td>816°C</td>
</tr>
</tbody>
</table>

a CROSHHEAD SPEED = 2.12 μm·s⁻¹; NOTCH - Kₚ = 8; STRESS RELIEVED AT 850°C FOR 8 HOURS - VACUUM

b HELIUM AND HYDROGEN TESTS - 34 MPa; O₂ (H₂) < < 10 ppm
### Table 2
SMOOTH TENSILE TEST

<table>
<thead>
<tr>
<th>ALLOY</th>
<th>UTS (^b) (MPa)</th>
<th>Y. S. (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AIR 810(^o)C</td>
<td>He 20(^o)C</td>
</tr>
<tr>
<td>CM SX–2</td>
<td>980.9</td>
<td>1046.1</td>
</tr>
<tr>
<td>CM SX–4C</td>
<td>1004.2</td>
<td>1087.4</td>
</tr>
<tr>
<td>RENE N–4</td>
<td>969.0</td>
<td>1157.8</td>
</tr>
<tr>
<td>PWA 1480</td>
<td>995.4</td>
<td>1151.7</td>
</tr>
</tbody>
</table>

\(^a\) ALL SPECIMENS STRESS RELIEVED AT 850\(^o\)C FOR 8 HOURS/VACUUM  
\(^b\) CROSS HEAD SPEED OF 2.12 \(\mu\)m/s  
\(^c\) OXYGEN CONTENT \(< < 10\) ppm

### Table 3
NOTCHED TENSILE TEST

<table>
<thead>
<tr>
<th>ALLOY</th>
<th>R. A. (^b) (%)</th>
<th>NOTCHED STRENGTH (^c) (MPa)</th>
<th>NOTCHED RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>H(_2)</td>
<td>H(_2)</td>
<td>H(_2)/H(_s)</td>
</tr>
<tr>
<td>CM SX–2</td>
<td>14.3</td>
<td>1498.5</td>
<td>0.14</td>
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<tr>
<td>CM SX–4C</td>
<td>10.6</td>
<td>1656.6</td>
<td>0.36</td>
</tr>
<tr>
<td>RENE N–4</td>
<td>10.8</td>
<td>1472.9</td>
<td>0.46</td>
</tr>
<tr>
<td>PWA 1480</td>
<td>12.4</td>
<td>1618.0</td>
<td>0.40</td>
</tr>
</tbody>
</table>

\(^a\) ALL SPECIMENS STRESS RELIEVED AT 850\(^o\)C FOR 8 HOURS/VACUUM  
\(^b\) FINAL CROSS SECTIONAL AREA ASSUMED ELLIPTICAL  
\(^c\) NOTCH K\(_T\) = 8
Fig. 3

NOTCHED TENSILE FRACTURE SURFACES
2.5 X

CM SX-2  CM SX-4C  RENE N-4  PWA 1480

34 MPa HELIUM

34 MPa HYDROGEN