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by

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for

THE ROLE OF GRAIN BOUNDARY CHEMISTRY AND STRUCTURE IN THE
ENVIRONMENTALLY-ASSISTED INTERGRANULAR CRACKING
OF NICKEL-BASE ALLOYS

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SUMMARY

Progress during the current grant period (1990-93) focused on five main areas of research: 1) determination of the role of C and Cr on the deformation and IG cracking behavior of Ni-(5-30)Cr-9Fe, 2) determination of the effect of grain boundary misorientation on the IG cracking behavior, 3) construction of an electron backscattering pattern (EBSP) imaging system, 4) determination of the effect of the environment on creep and cracking and 5) characterization of the surface film. Results of the program showed that both C and Cr are potent solid solution strengtheners which can reduce the steady state creep rate at 360°C by several orders of magnitude. Intergranular cracking of 100 μm grain samples of high purity Ni-16Cr-9Fe at 360°C occurs by the formation of grain boundary voids and their interlinkage, driven by dislocation creep in the matrix. Creep experiments in primary water at 360°C and under an applied cathodic potential show that the creep rate is increased by an order of magnitude over that in Ar and the percent IG fracture increases as well. The oxide film composition and thickness is a sensitive function of the C content, increasing in thickness and Ni(OH)2 content with an increase in C or with a decrease in Cr to 5 wt%. Thermomechanical treatments along with electron channeling pattern (ECP) analysis were used to create and index, respectively, samples with enhanced fractions of coincident site lattice boundaries (CSLBs). Constant extension rate tensile (CERT) experiments on samples with a high percentage of CSLBs showed a reduction in the extent of IG cracking as compared with general high angle boundaries, GHABs. However, these experiments were conducted on samples with grain sizes of approximately 300 μm in size while commercial material is an order of magnitude smaller. As such, an electron backscattering pattern (EBSP) imaging system has been constructed for use in an environmental scanning electron microscope (ESEM) which can image grains below 5 μm. The system has been successfully benchmarked against results from ECP analysis.
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1. Introduction

The objective of this program is to determine the mechanism(s) of intergranular cracking of nickel-base alloys in high temperature, high purity water and primary water. Over the last three years, the proposed work, conducted under grant No. DE-FG02-85ER45184, has succeeded in identifying and isolating some important aspects of the deformation and cracking mechanism that were previously not understood or realized. This report summarizes the major findings of the current project over the period 1990-1993. Topics discussed include the effects of C and Cr on deformation and IG cracking in an argon environment, the role of grain boundary misorientation on IG cracking, the role of the environment on IG cracking and the characterization and probable role of oxide films on IG cracking.

2. Deformation and IG Cracking in Argon

This project has resulted in the determination of the mechanism of grain boundary cracking in high purity Ni-Cr-Fe at 360°C in an inert environment. The mechanism is dislocation-controlled creep leading to the nucleation, growth and linkage of grain boundary voids. [1,2] Determination was made by conducting constant extension rate tension (CERT) and constant load tension (CLT) experiments on Ni-xCr-Fe-yC alloys in 360°C argon in order to eliminate the influence of the environment. The presence of grain boundary voids in the cross-sectioned specimens, grain boundary sliding on the side surfaces, and fine dimples on the faceted fracture surface all indicate that creep is acting in conjunction with the imposed plastic deformation from the CERT experiment. Creep is dislocation-controlled and follows either power-law or power-law breakdown behavior in which the stress exponent in the eqn $\alpha \sigma^n$ is between 1 and 5, or greater than 5, respectively.

The link between the intergranular failure mode and the deformation mode of creep in CERT experiments is understood by considering the evolution of the grain boundary morphology with increasing stress or strain. At lower strains (corresponding to low stress), creep leads to the nucleation of grain boundary voids which begin to grow. At intermediate strains, the growing voids link up into larger voids and form cavities. At higher strains, cavity interlinkage forces ductile overload in the remainder of the cross section, producing intergranular facets covered with creep-induced voids on the order of 1-2 microns in size, Fig. 1. Failure is based on a type of 'damage accumulation' process in which the damage accumulates at a more rapid rate at higher loads where creep rates
Figure 1. Evolution of grain boundary damage accumulation process due to creep in a CERT experiment on the UHP6 alloy at 360°C showing the (a) presence of grain boundary voids at low strain, away from neck (b) void link up at intermediate strain in the necked region, and (c) complete failure in the highest strain region at the fracture surface.
are higher. The longer the time spent at these higher loads (i.e. a slower strain rate), the larger is the accumulated creep strain resulting in more extensive grain boundary cavity formation and more IG cracking.

The additions of chromium and carbon to nickel-base alloys improve their resistance to IG cracking in argon at 360°C. Carbon is an extremely potent solid solution strengthener, resulting in a decrease in the creep rate by over 4 orders of magnitude as the carbon level is increased from 20 to 300 wppm. [3] The very potent effect of small concentrations of carbon on creep behavior indicates that dislocation-carbon interaction is occurring during creep with the probable result that originally mobile dislocations are being pinned, as evidenced by serrated yielding during deformation, Fig 2a. Chromium also strongly affects creep by a solid solution strengthening mechanism, Fig. 2b. [2] The creep rate (in stop-load experiments) is reduced by several orders of magnitude as the chromium content is increased from 5 to 30 wt%. Hence, both C and Cr influence the amount of IG cracking through their influence on the creep rate of the alloy.

![Fig. 2a. Strain rate dependence of serration behavior as a function of carbon in solution at 400°C.](image1)

![Fig. 2b. Summary of step-load test results showing the effect of chromium on the steady state creep rate of Ni-xCr-9Fe-C alloys in argon at 360°C.](image2)

3. Role of Grain Boundary Misorientation

Our recent work on this topic has revolved about three areas; 1) the effect of grain boundary misorientation on IG cracking in high purity water, 2) the determination of the effect of grain boundary character distribution on creep deformation, and 2) development of an electron backscattering pattern imaging system.
3.1 Misorientation effects on IG cracking

The effect of grain boundary misorientation on the intergranular cracking behavior of pure Ni-16Cr-9Fe was assessed by determining if low angle boundaries (LABs) or coincident site lattice boundaries (CSLBs) are more resistant to cracking than general high angle boundaries (GHABs) in argon and water. [4] Cracking susceptibility of boundary types was determined using CERT experiments in 360°C argon and deaerated water. IG cracking susceptibility was determined using two measures: the % of characterized grain boundaries that cracked, and the average cracked boundary fraction. Samples were either in the annealed condition (typically 4% LAB, 18% CSLB, 78% GHAB) or the CSLB-enhanced condition (4% LAB, 36% CSLB, 60% GHAB).

Cracked boundary fractions for CSLB-enhanced samples tested in either environment ranged from 0.01 to 0.08, while those for annealed samples ranged from 0.07 to 0.10, indicating that samples with increased proportions of CSLBs are more crack resistant. No LABs cracked in either environment. In annealed samples, the proportion of CSLBs that cracked in water was 6.7% compared to 1.5% in argon; the proportion of GHABs that cracked in water was 9.3% compared to 6.6% for argon. Thus, CSLBs are more crack resistant than GHABs in either environment, and both are more crack resistant in argon than in water. The higher amounts of cracking and the higher CSLB cracking susceptibility in high purity water indicate the presence of an environmental effect on cracking behavior. Results are summarized in table 1 and Fig. 3a.

<table>
<thead>
<tr>
<th></th>
<th>high purity water</th>
<th>argon</th>
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<tbody>
<tr>
<td></td>
<td># of % of</td>
<td># of % of</td>
</tr>
<tr>
<td></td>
<td>type total 1</td>
<td>cracked type 2</td>
</tr>
<tr>
<td>CSLBs</td>
<td>119 18%</td>
<td>8 6.7%</td>
</tr>
<tr>
<td>LABs</td>
<td>29 4%</td>
<td>0 0</td>
</tr>
<tr>
<td>GHABs</td>
<td>526 78%</td>
<td>49 9.3%</td>
</tr>
<tr>
<td>Total</td>
<td>674 57%</td>
<td>85%</td>
</tr>
<tr>
<td>CSLB-enhanced samples</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CSLBs</td>
<td>228 37%</td>
<td>4 1.8%</td>
</tr>
<tr>
<td>LABs</td>
<td>33 5%</td>
<td>0 0</td>
</tr>
<tr>
<td>GHABs</td>
<td>356 58%</td>
<td>19 5.3%</td>
</tr>
<tr>
<td>Total</td>
<td>617 23%</td>
<td>3.7%</td>
</tr>
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</table>

1. Percentage of total characterized boundaries.
2. Percentage of boundary type that is cracked.

Table 1. Numbers of Characterized and Cracked Boundaries by Type for Annealed and CSLB-Enhanced Samples Tested in High Purity Water and Argon.
The beneficial effect of LABs and CSLBs is likely due to the ability of these boundaries to induce slip in neighboring grains by either transmitting or absorbing and re-emitting lattice dislocations, thereby reducing grain boundary stresses and the propensity for crack initiation. The results indicate that control of grain boundary proportions can improve the intergranular stress corrosion cracking susceptibility of pure Ni-16Cr-9Fe.

3.2 Misorientation effects on creep deformation

Results from constant load tests of annealed (SA) and CSLB-enhanced samples with a comparable grain size indicate that the creep behavior of this alloy can be significantly influenced by altering the grain boundary character distribution. Using a grain size of 330 μm, two constant load tests on annealed samples reached the tertiary stage at 165 and 115 h at total strains of 1.8% and 1.35%, respectively, Fig. 3b. A marked difference in creep resistance of the CSLB-enhanced sample as compared to the annealed samples was observed. The CSLB-enhanced sample did not reach the tertiary stage until after 250 h at a total creep strain of 0.8%. However, the most striking difference was in the distribution of creep strain between the creep regimes. In the SA samples, primary creep accounted for 80% of the total strain as compared to <40% in the CSLB-enhanced sample. This latter sample spent the bulk of its life in the secondary regime whereas steady state creep

![Fig. 3a. Comparison between cracked boundary fractions and the percentages of characterized boundaries that cracked.](image)

![Fig. 3b. Constant load creep curves of SA/CSL Ni-16Cr-9Fe alloys at 300 MPa in 360°C argon.](image)
was reached only momentarily in the SA samples. It is interesting to note that a CSLB-enhanced sample with a 405 μm grain size also exhibited superior creep resistance, implying that the influence of grain size may play a secondary role in creep behavior compared to grain boundary character distribution.

Scanning electron microscopy revealed that most of the cracks in the annealed samples are wedge type whereas the CSLB-enhanced samples do not show any preferential crack types. The wedge geometry was shown by Chang and Grant [5] to be due to grain boundary sliding which is known to be an important source of creep deformation. Watanabe [6] demonstrated that the amount of sliding strongly depends on the type and misorientation of grain boundaries and it occurs more easily in GHABs. Kokawa et al. [7] reported that GHABs exhibited more sliding than CSLBs. Since grain boundary sliding has been shown to be proportional to the amount of total creep strain [8], a sample with more CSLBs would be expected to exhibit less total strain. Thus, the observed difference in the creep behavior between annealed samples and a CSLB-enhanced sample may be interpreted in terms of the effect of grain boundary misorientation on the rate of grain boundary sliding.

3.3 Development of an electron backscattering pattern (EBSP) imaging system

A second project involves the development of an electron backscattering pattern (EBSP) imaging system for use in an environmental scanning electron microscope (ESEM). This system was developed to allow us to address grain boundary character distributions in small grain samples typical of commercial material (15-25 μm) as compared to the restriction of the electron channeling pattern method which requires nominal grain sizes >200 μm. [9] To facilitate EBSP imaging, the standard specimen stage in the ESEM has been replaced with a special specimen mount that holds the sample at an angle of 19.4° to the electron beam. EBSPs are formed when the electron beam is focused on a crystal or grain as a stationary probe; as a result, the spatial resolution of the technique is only limited by the electron beam size (= 1 μm). In this technique, EBSPs are imaged directly on a phosphor screen mounted on a stainless steel tube that is attached to the leaded glass viewing port in the side of the microscope's sample chamber. EBSPs are recorded by digitizing the image from a Dage-MTI CCD-72 TV camera coupled with a Nikon 60mm macro lens and a 1.4x Nikon teleconverter and sent to a Scion LG-3 frame capture board installed in an Apple Macintosh Iici personal computer. The pattern quality is improved significantly by incorporating a small program to subtract the background level semi-automatically. In order to generate accurate
orientation information, the stage of the microscope is controlled by a small program
written in the National Instrument LabVIEW2 environment to assure the same working
height as those from the calibration specimen while the sample is being translated. On-
line analysis of EBSPs is achieved by incorporating software into the public domain
image processing program, Image1.45. Thus orientations can be calculated while EBSPs
are being imaged. Fig. 4a illustrates a diagram of the system constructed for this work.

In order to evaluate differences in results obtained with the EBSP and SACP
techniques, grain boundaries in an ultra high purity Ni-16Cr-9Fe alloy were characterized
with both techniques. Due to the limitation in spatial resolution in the SACP technique, a
sample of the alloy was prepared by solution annealing at 1200°C/1.5 hr. to produce a
mean grain size of 320 μm. [10] Prior to pattern imaging, the sample surface was
mapped out with backscattered electron images and 70 grain boundaries were assigned.
Fig. 4b shows the distribution of grain boundary misorientation angle determined by
SACPs and EBSPs. The distribution shows excellent agreement between the techniques.

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Fig. 4a. Schematic diagram of the EBSP camera on the Electroscope ESEM.

Fig. 4b. Comparison of grain boundary misorientation distribution of SA
Ni-16Cr-9Fe after 1.5 h at 1200°C using SCAP and EBSP techniques.
It was found that the average difference in grain boundary misorientation angle between the two techniques is only 0.98°. The difference in grain boundary misorientation angle may be due to a larger distortion introduced by gnomonic projection in EBSPs which is a direct result of the angular view of diffraction space of EBSPs that extends over 90° whereas it covers no more than 20° in SACPs. A major difference between the two techniques used in the present work was found to be the time required to completely characterize 70 grain boundaries. Owing to the capability to perform on-line analysis of EBSPs, the EBSP technique required only half the time as the SACP technique.

4. Effect of the Environment on Deformation and Cracking

The most recent accomplishment has been the determination that hydrogen is the likely cause of the increase in intergranular cracking in high temperature water. [11] This conclusion is based on both CERT and CLT experiments conducted in high purity water and primary water at controlled potentials. One of the most significant observations is the large increase in creep rate in primary water over that in argon. In primary water, the creep rate of ultra-high purity Ni-16Cr-Fe at 360°C and 500 MPa at a potential of -1150 mV\text{SHE} is an order of magnitude greater than that in water, Fig. 5a. The time to failure is reduced by a factor of three and the % IG fracture is increased by 50%. A detailed examination of both the side surfaces and the cross-section of the samples revealed that while dislocation plasticity accounted for 84% of the strain in the sample tested in argon, the percentage increased to 96% of the strain in water. This was due to a drop in the fractional strain due to grain boundary sliding and grain boundary cavitation. Although dislocation plasticity is the dominant deformation mechanism in both cases, grain boundary sliding and cavitation play an even smaller role during creep in water.

Constant extension rate tensile (CERT) tests of alloys with varying levels of Cr and C revealed a consistent increase in IG cracking percentage on the fracture surface in high purity water as compared with argon. However, two observations should be noted. First, the amount of cracking in both environments decreases with increasing Cr or C level. Second, the difference between the amount of cracking in the two environments decreases as the amount of Cr or C increases. For example, although the difference in %IG cracking in water and argon is 42% vs. 27%, respectively, for the LCr (5 wt% Cr, low C) alloy, the difference drops to 2% for the high Cr alloy (30 wt%) and zero for the 300 wppm C alloys. CERT results in primary water with an applied potential are even more significant as they show that a cathodic potential (~230 mV below OCP) results in
the greatest amount of IG cracking with progressively less IG cracking at OCP and less
still at anodic potentials, ~250 mV above OCP, Fig. 5b. Both sets of results point toward
hydrogen as the likely species accelerating the creep rate and enhancing IG fracture.

Fig. 5a. CLT test results of the UHP7 alloy at an effective stress of 500 MPa in Ar and
deaerated, hydrogenated, 0.001M LiOH + 0.01M H₃BO₃ at -1150 mV SHE at 360°C and an initial strain rate of 3 x 10⁻⁷ s⁻¹.

Fig. 5b. CERT test results as a function of chromium, grain size, environment and potential in 0.001M LiOH + 0.01M H₃BO₃ at 360°C at an initial strain rate of 3x10⁻⁷ s⁻¹.

The relationship between the deformation mode of dislocation-controlled creep and
the fracture mode of IG cracking is believed to be as follows. Time dependent
deformation in the matrix causes a strain at the grain boundary that must be
accommodated, usually by the processes of cavitation and sliding. Without these
processes, the strain must be accounted for by another manner such as slip transfer across
the grain boundary. Fracture in argon indicates that grain boundaries are inherently weak
and prone to failure. Therefore, accommodation of the matrix deformation occurs by the
formation of grain boundary cracks. It follows that as the amount of creep damage
increases, the amount of IG cracking should also increase. This is verified by the results
which show that the amount of IG cracking increases as the strain rate in a CERT test
decreases. However, it still needs to be determined whether the amount of matrix creep
deformation or the rate of time dependent deformation is responsible for the IG cracking
behavior. Most likely it is a combination of the two. For the UHP7 alloy crept at 360°C
and 500 MPa, it was shown that the amount of side surface IG cracking increased by a
factor of 3 as the matrix strain doubled and the creep rate increased by a factor of 10 in water over that of argon, preventing any clear conclusion about the effect of the rate and amount of strain. It may be that the strain rate is more important in the water environment than argon, where a synergistic effect between hydrogen and dislocation motion may occur. Further experimentation is necessary to separate the effects of creep strain rate and total creep strain on the IG cracking behavior of these alloys at 360°C.

5. Oxide Films and Their Relation to Intergranular Cracking

Since the surface film has been implicated in several models of intergranular stress corrosion cracking (IGSCC) of nickel-base alloys, this study was initiated to provide a foundation from which to determine the possible link between the nature of the surface film and IG cracking susceptibility. The influence of chromium, carbon and yttrium on the nature of the surface film formed on nickel-base alloys was investigated after 100 hours of exposure in high purity, deaerated, hydrogenated water at 360°C. The chemical composition of an ultra-high purity (UHP) alloy of Ni-17Cr-9Fe-0.0030C was altered by varying the Cr content, doping with C, or ion implanting Y. X-ray photoelectron spectroscopy and scanning transmission electron microscopy provided information on the chemical composition, structure, morphology and thickness of the surface film. Increasing the chromium content of a Ni-xCr-9Fe-low C alloy from 5 to 17 wt % dramatically changes the surface film from predominantly Ni(OH)₂ to Cr₂O₃. An additional increase in Cr from 17 to 30 wt. % does not significantly alter the type, distribution or thickness of the oxide phases formed. This suggests that there is a critical chromium concentration that controls the formation of Cr₂O₃ after 100 hours of exposure to high purity, deaerated, hydrogenated water at 360°C. The presence of Cr₂O₃ in these alloys is attributed to the high affinity of Cr for oxygen, and the increased stability of Cr₂O₃ over many other oxides in reducing environments. The addition of 300 wppm carbon to a Ni-17Cr-9Fe-0.0030C alloy increases the film thickness while promoting the formation of Ni(OH)₂, possibly by an increase in the diffusion of Ni or by an enhancement of the surface activity of the Ni atoms. The addition of 300 wppm carbon to a Ni-30Cr-9Fe-0.0020C alloy increases the film thickness without changing Cr₂O₃ as the dominant surface species. Yttrium implantation to 2.4 at. % at a depth of 70 nm in Ni-17Cr-9Fe-0.0030 and 0.030 C alloys produces a similar film thickness and similar composition profiles containing slightly more Ni(OH)₂ than Cr₂O₃. Yttrium is known for forming Y₂O₃ which most likely promotes the formation of Cr₂O₃ by acting as nucleation sites for the similarly structured chromia.
In primary water, the film thickness remains unchanged but the nickel content in the film is substantially reduced at -1150 mV_{SHE}. This is expected based on the potential-pH diagram since this potential is about 200 mV below the Ni/NiO line. Nevertheless, the decrease in Ni(OH)\textsubscript{2}, or the corresponding increase in the Cr\textsubscript{2}O\textsubscript{3} fraction of the film correlates inversely with SCC cracking susceptibility. The most likely explanation for this effect is that it is not the protectiveness of the film which is critical but the generation and absorption of hydrogen which is controlling. It also shows that the major role of Cr in the alloy is in reducing the creep rate rather than in forming a more protective film.

6. Contribution to Our Current Understanding of the Problem

It is important to consider these results in the context of the collective understanding of the problem of IG cracking in high temperature water. These results have added substantially to this body of knowledge in several respects. The mechanism of IG cracking in ultra-high purity Ni-16Cr-9Fe is now well understood to be due to grain boundary void formation, growth and link-up. For the first time, the roles of Cr and C (in solution) on deformation and cracking have been clearly determined. Also, the first definitive set of data on the role of the environment in the deformation behavior of the alloy has been acquired. These results implicate hydrogen as the likely species causing an enhancement in dislocation plasticity in the matrix. The results are also the first to show a substantial increase in steady state creep rate in water over that in an inert environment. The studies on grain boundary misorientation show that CSLBs are more resistant to cracking in high purity water than GHABs. Also, the creep deformation is considerably different for a CSLB-enhanced microstructure over that of a microstructure consisting of a random distribution of grain boundary misorientations. Finally, we have shown how Cr and C affect the formation of the oxide film and that the film composition and thickness does not seem to control the observed IG cracking.

The results of this program have shed considerable light on the mechanism of cracking. They have also prompted important new and better focused questions regarding the cracking mechanism. These include, is hydrogen really the culprit? If so, how does the increased dislocation plasticity translate into an increased propensity for IG cracking? Knowing the role of C in solution, what is the role of C in the form of carbides and how can these be separated? Is the deformation and cracking response of small grain material similar to that for large grain material? What is the mechanism by which carbon in solution or CSLBs change affect grain boundary deformation to make the boundaries
more resistant to IG cracking? A program designed to answer these questions is described in the accompanying renewal proposal.

7. Bibliography


Journal Articles


Conferences


9. Graduate Student Status and Degrees Awarded

9.1 Graduate student status

Two graduate students are being supported on this program and are pursuing advanced degrees. One student (Visit) has achieved candidacy and the other (Jason) has passed the written exam and is preparing for the oral exam.

Visit Thaveeprungsriporn is working on grain boundary misorientation effects on cracking. He has recently completed the construction and benchmarking of the electron backscattering pattern (EBSP) imaging system on the environmental scanning electron microscope (ESEM). He is now engaged in developing thermomechanical treatments to control the CSLB fraction in fine grain samples of Ni-16Cr-9Fe. He will conduct experiments designed to determine the role of grain boundary misorientation in deformation and IG cracking. I expect him to complete his PhD. by August, 1995.

Jason Hertzberg is working on determining the role of carbon in solution and as carbides in deformation and IG cracking of Ni-16Cr-9Fe. He has recently passed his written candidacy exam and is preparing for his oral exam this summer. He should achieve candidacy by December, 1993. I expect that he will finish his thesis by January, 1996.

9.2 Degrees Awarded

During the last three years, three PhDs were awarded to students who were supported on this project.


10. Appendix (copies of 5 recent key papers)


