

NOTCH-RUPTURE BEHAVIOR OF A NICKEL-BASE  
SUPERALLOY AT INTERMEDIATE TEMPERATURES

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ABSTRACT

The phenomenology of notch rupture in a modified IN 100 alloy was investigated. Two conditions were used for the study: an air cooled plus aged treatment (AC) which resulted in about equal smooth section and notch lives and an oil quenched plus aged treatment (OQ) which resulted in equivalent smooth section but much lower notch lives. In contrast to the AC material, the notch lives of the OQ material showed little dependence on stress at high stresses until a threshold level was reached at which point notch life increased dramatically and approached that observed in smooth sections. Notch life increased rapidly as temperature was lowered. More interestingly, notch life was found to be markedly influenced by the temperature/stress path experienced during loading. Load application at lower temperature and/or at higher stress rates tended to eliminate the notch sensitivity. Effects of surface treatment and test environment were also found to be important. Shot peening and/or testing in vacuum improved notch life significantly. Interestingly, it was found that testing for a short time in a vacuum followed by testing in air also enhanced notch life. The notch sensitivity of the OQ material was traced to environmentally induced susceptibility to grain-boundary cracking during stress relaxation under high stresses.

## INTRODUCTION

The majority of engineering structures contain holes, slots, grooves, etc. for attachment of subassemblies and coupling of other components. Such features locally perturb the applied or working loads and these regions of stress concentration are often the life-limiting location in a part. At elevated temperatures, stress-rupture is a potential fracture mode and thus rupture properties in notched details are an important design consideration. This paper explores the notch-rupture phenomenology in a high-strength nickel-base superalloy fabricated by powder metallurgical processing methods. Specifically, factors which influence the behavior of MERL 76, an alloy for use in advanced turbine disks, are evaluated.

## MATERIAL AND SMOOTH-SECTION PROPERTIES

The material investigated is a modified IN 100 alloy designated MERL 76 with analyzed composition (weight percent):

Ni	Cr	Co	Mo	Al	Ti	Nb	Hf	B	Zr	C
54.7	12.3	17.9	3.3	5.0	4.3	1.7	0.74	0.018	0.06	0.025

The material was consolidated from -80 mesh powder by hot isostatic pressing. Two heat treatments which produced extremes in notch behavior were selected for this study; both involved a solution treatment at 1120°C and an aging at 760°C.\* The difference is in the cooling rate from the solution treatment temperature; one sample was quenched in oil and the other was cooled in air, hereafter referred to as OQ material and AC material, respectively. Microstructures, illustrated in Figure 1, showed two significant differences. Firstly, the secondary gamma prime particles are considerably finer in the OQ material. Secondly, except for the blocky primary gamma prime particles which are mainly at grain intersections, the grain boundaries in the OQ material are rather straight and featureless. In contrast, gamma prime particles have precipitated along the grain boundaries in the AC material during cooling from the solution treatment temperature. These microstructural variations caused relatively small differences in smooth-section properties and may be summarized as follows. The OQ material has a higher yield strength (1100 MPa versus 980 MPa) and a lower tensile ductility (18 percent versus 24 percent elongation) than the AC material. Under creep conditions, the

\*Note that neither of these heat treatment sequences are used for MERL 76 components (1).

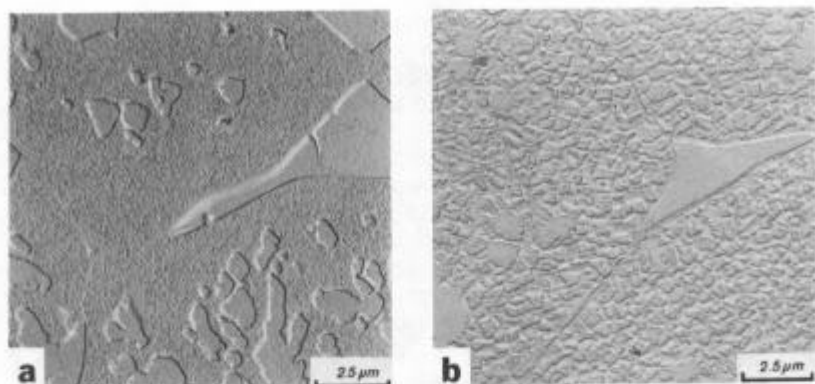


Figure 1. Microstructure of MERL 76 after (a) the OQ (b) AC heat treatments

OQ material has a lower minimum creep rate, slightly longer rupture life and lower rupture ductility than the AC material. However, the stress dependence of the creep-rupture properties of the two materials is similar. Creep deformation in both materials is heterogeneous, involving concentrated shear on few slip planes and orientation formation (2). In spite of these apparent similarities in smooth-section properties in these two materials, vast differences in notch behavior are observed which are described in the following section.

#### NOTCH-RUPTURE BEHAVIOR

The geometry of the bar specimen used in this study is illustrated in Figure 2.

The theoretical elastic stress concentration factor ( $K_t$ ) at the notch root is 3.8. Standard notch tests were conducted at 704°C and at a net-section stress of 690 MPa, which resulted in notch lives of about 2 h for the OQ material and about 100 h in the case of the AC material. For comparison, the rupture lives of smooth specimens are slightly over 100 h for both materials. The notch-brittleness of the OQ material persists to stresses as low as 420 MPa where differences in notch lives between the two materials are over two orders of magnitude.

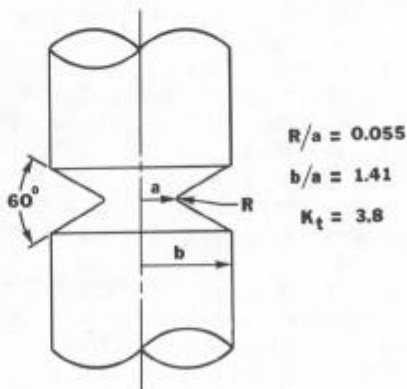


Figure 2. Geometry of the notched specimen

It is customary for metallurgists and material scientists to associate notch-brittleness with "unfavorable" microstructures, environment, and/or deformation mechanisms (3-5). Alternatively, analyses of local stresses and strains in the plastic zone at the notch root, using constitutive equations derived from smooth specimen data, have also been proved successful in understanding some aspects of notch behavior (6-8). A synthesis of both view points is deemed essential for a basic understanding of the notch-rupture phenomenon. The experiments described below are designed to provide the basic framework for establishing such a synthesis. Some of the reasoning behind certain parts of the study is as follows.

Under a net-section stress which is smaller than the macroscopic yield stress, a notched section will deform in a rather complex manner. At sufficiently high stresses during loading and before the maximum load is reached, a small volume of material at the notch root deforms plastically, because of the stress concentration, while the bulk remains elastic. Upon reaching the maximum applied load, the plastic deformation at the notch root is constrained by an elastic core and subsequent plastic flow is, therefore, dominated by a strain-controlled creep relaxation phenomenon. Typically, the stress and the rate of deformation are the highest initially and decrease rapidly with time toward steady state values determined by elastic stresses in the bulk. These considerations indicate that a significant portion of the plastic strain in the notch is accumulated during the initial stages of the test. The nature of this deformation and the accompanying damage depend on the relative contribution of various deformation mechanisms and are determined by factors such as stress, deformation rate, temperature and environment. At high temperatures, a slower rate of deformation results in a larger amount of grain-boundary sliding than a faster rate of deformation (9). Thus, if grain boundary sliding contributes to the notch-brittleness, a fast loading rate should be beneficial. It therefore follows that, in addition to stress and temperature, the stress/temperature path along which a maximum load is applied should be an important notch-rupture test variable.

In general, the variables which govern the notch-rupture behavior of a material fall into four categories: (1) Test parameters such as temperature, stress and, as considered above, stress/temperature path. (2) Specimen parameters such as geometry and surface conditions. (3) Microstructural parameters such as grain size and grain boundary microstructures. (4) Environment. Results presented below illustrate effects of some of the variables in each category.

## Test Parameters

The effect of stress on the notch lives of the OQ and the AC material at 704°C is illustrated in Figure 3. For comparison, the rupture lives of smooth specimens, which are rather similar for these two materials, are also included in the figure. The notch lives of the OQ material show two distinctly different stress dependencies. At stresses over about 420 MPa, the OQ material has short notch lives which are relatively insensitive to stress. At stresses slightly lower than 420 MPa, the notch lives increase abruptly by more than two orders of magnitude and approach the rupture lives of smooth specimens. In contrast, the notch lives of the AC material are considerably longer, equal to or slightly lower than the rupture lives of smooth specimens in the stress range studied, and are more stress dependent.

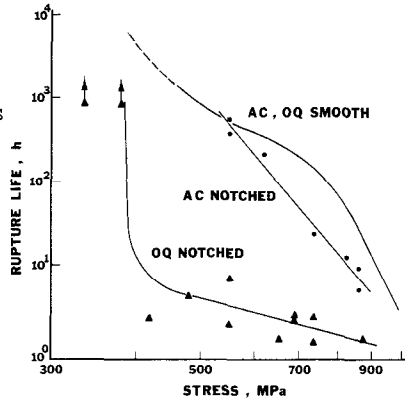


Figure 3. Effect of stress on 704°C rupture lives of smooth ( $K_t=1$ ) and notched specimens with the OQ and the AC heat treatments

The effect of temperature on the notch life of MERL 76 at 690 MPa is shown in Figure 4. The temperature dependence of notch lives for both the OQ and the AC materials appears to be similar. There is a rapid increase in notch lives below 704°C and notch-brittleness is absent at about 675°C.

The conventional procedure for creep-rupture or notch-rupture testing is to equilibrate the specimen at the test temperature before applying the load by placing standard weights on a load pan. This procedure precludes controlled variation of loading rates. Therefore, the effect of loading rate on notch life of the OQ material was studied using

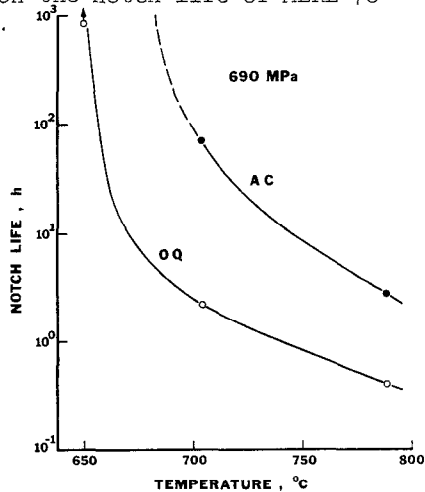


Figure 4. Effect of temperature on rupture lives of notched specimens with the OQ and the AC heat treatments at a net-section stress of 690 MPa

a servo-hydraulic testing machine. Notch specimens were equilibrated at 704°C before loading at various rates to a net-section stress of either 550 MPa or 690 MPa. Results are illustrated in Figure 5. At a net-section stress of 550 MPa, a very significant improvement of rupture lives was observed at loading rates 4135 MPa/minute or higher. However, no effect of loading rate was observed if the net-section stress was increased to 690 MPa.

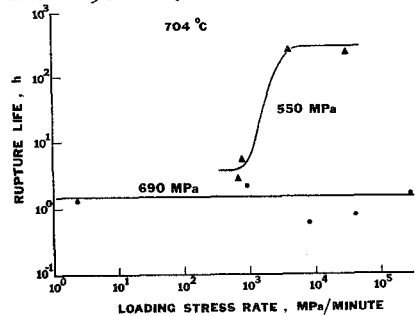


Figure 5. Effect of loading rate on 704°C rupture lives of notched specimens with the OQ heat treatment at net-section stresses of 550 MPa and 690 MPa.

To study the effect of loading temperature on notch-rupture life, specimens were first equilibrated at a selected temperature. The load was applied in a conventional manner until a net-section stress of 690 MPa was reached, maintained at 690 MPa for 0.5 h and during subsequent temperature increase to 704°C. Results, illustrated in Figure 6, show that the notch-brittleness associated with isothermal testing at 704°C was eliminated when the loading temperatures were lower than about 480°C.

#### Specimen Parameters

Notch specimens with only one geometry, given in Figure 1, were used in this study; the only specimen parameter that was studied in some detail was surface treatment. Five conditions were examined: as-machined, electropolished, electropolished plus nitrogen ion implanted, as-machined plus prestrained, and as-machined plus glass bead peened. Nitrogen ions were implanted at 100 Kev and at a dose of  $10^{17}$  ions/cm<sup>2</sup>. To prestrain a specimen it was first equilibrated at a temperature at which the OQ material is notch insensitive, loaded to the predetermined stress level and then the load was released. Prestraining and peening were anticipated to have similar effects on notch-rupture behavior through the worked surface layer and residual stress pattern which were introduced. These surface treatments were applied to the OQ material, and specimens were tested conventionally at a standard test condition of 704°C/690 MPa. Results, given in Table 1, show that shot peening significantly increases notch lives while other surface treatments have no beneficial effect. Beneficial effects associated with shot peening are usually considered to be the deformation

structure and the accompanying compressive residual surface stresses. Conversely, the poor response of the prestrained specimens may be attributed to a relatively smaller and less homogeneous deformation which could have resulted in a deformation structure and a residual stress pattern quite different from those in the peened specimens. Except for the alloying effect, ion implantation may be viewed simply as shot peening in atomic scale. The low notch lives of the nitrogen implanted specimens may be due to a high mobility of the nitrogen atoms at the test temperature used. If this is the case, implanting different atomic species may well produce different results.

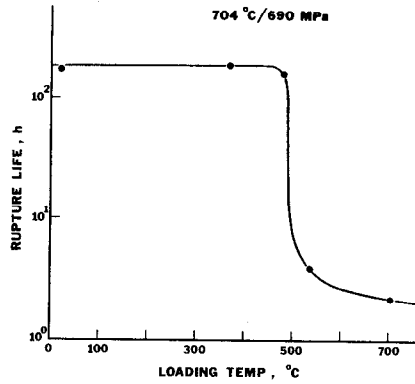


Figure 6. Effect of loading temperature on 704°C rupture lives of notched specimens with the OQ heat treatment at a net-section stress of 690 MPa

#### Microstructural Parameters

Since details of crack nucleation in an air-tested specimen were obscured by oxidation, crack initiation behavior at the notch root was studied by testing electropolished specimens in a vacuum. The tests were interrupted periodically for crack inspection using a scanning electron microscope. Microcracks were detected on the notch surfaces of either the OQ or the AC materials after about 50 h of testing at 704°C/690 MPa. In the OQ material microcracks preferentially nucleated at and propagated along grain boundaries as shown in Figure 7a. The crack nucleation sites were somewhat different for the AC material in which microcracks formed at several locations (Figure 7b): at gamma prime:gamma interface (arrow 1), within large gamma prime particles (arrow 2), at machining cracks normal to the stress axis (arrow 3) and at grain boundaries. The first three types of microcracks propagate transgranularly in a direction normal to the stress axis. Grain boundary microcracks were seldom observed in the AC material at test

Table 1 Effect of Surface Treatment on Notch-Rupture Life

Surface Treatment	Life, h
As-machined	2.3
As-machined	2.1
Electropolished	0.5
Electropolished + N <sub>2</sub> <sup>+</sup> Implanted	0.6
Electropolished + N <sub>2</sub> Implanted	1.5
As-machined + Prestrained	
827 MPa at 20°C	0.5
690 MPa at 370°C	1.7
As-machined + Peened (6.5N)	113.0*
As-machined + Peened (11.5N)	95.0*

\*Test terminated with no failure

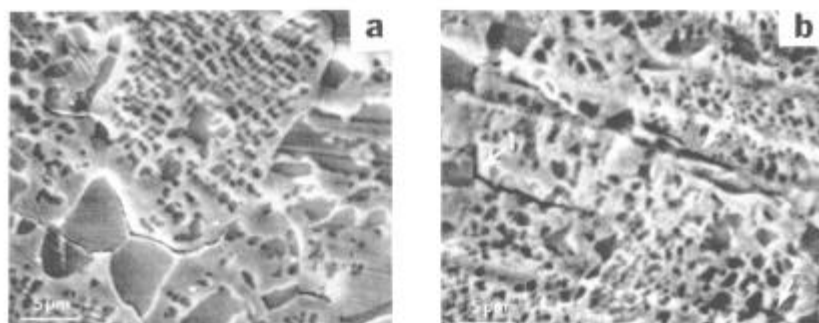


Figure 7. Microcracks observed in the notch root during tests at 704°C/690 MPa in a vacuum (a) the OQ specimen after 65.5 h (b) the AC specimen after 111 h.

times less than 120 h. However, with increased test times, the development of grain boundary microcracks became increasingly evident and would eventually lead to intergranular fracture. These differences in crack nucleation behavior between the OQ and the AC materials may be attributed to a synergistic effect of two factors. Firstly, the grain boundaries in the OQ material are subjected to higher stresses because of the more creep-resistant grain matrix. Secondly, the grain boundaries in the OQ material are less resistant to shear deformation because of a lack of grain-boundary pinners (Figure 1).

Because of the intergranular fracture, grain size is expected to have an effect on notch-rupture life. Data published elsewhere show that the notch-rupture of MERL 76 increases with increasing grain size (10).

#### Environmental Parameters

The effect of testing in a vacuum was studied using the OQ material which was tested conventionally at a standard condition of 704°C/690 MPa. Results are given in Table 2 which show that environment plays an important role. However, the magnitude of the rupture life improvement is surprising. More importantly, it has also been observed that testing 6 h in a vacuum followed by testing in air also dramatically increases the notch-rupture life. This observation is consistent with the expectation that the crucial part of a

Table 2 Effect of Test Environment on Notch-Rupture Life

Environment	Life, h
Air	2.3
Air	2.1
Vacuum, $10^{-5}$ Torr	178.5*
Vacuum, $10^{-5}$ Torr	559.0*
Tested 6 h in vacuum followed by air test	104.5*

\*Test terminated with no failure



notch test is in the initial stages where most of the deformation takes place. Further, it also suggests that it is the interaction of air (or one of its constituents) during the plastic deformation, rather than oxidation per se, which causes the premature notch failure. It is also clear from results cited in this and the previous section that both the crack nucleation and crack growth characteristics are modified by the environment.

#### ANCILLARY TESTS

One of the puzzling aspects of the present study was the extreme contrast in smooth and notch behavior. The notch properties showed great differences whereas the smooth section creep tests showed rather similar characteristics (Figure 3). However, it should be realized that the notch deformation is not under constant stress. As described previously, the creep deformation at the notch, after a full load has been applied, is dominated by a strain-controlled relaxation phenomenon. To gain insight into the notch-rupture behavior, therefore, stress relaxation in the OQ and the AC materials at 704°C was studied using smooth creep specimens. Relaxation data were obtained by pulling a specimen to a predetermined tensile strain or stress at which the elongation of the specimen was kept constant and the decrease in stress with time was recorded. An interesting result of this study is that the OQ specimens fractured in less than 1 h during stress relaxation when the initial stresses exceeded 1240 MPa which corresponds to a tensile strain of only about 0.02. No fracture was observed in the case of the AC specimens tested to stresses up to the tensile strength. As notches also experience high stresses initially, there is an obvious correlation between the observed relaxation behavior and the notch-rupture behavior. Thus, the simple stress relaxation experiments capture some of the basic features of the notch-rupture phenomenon and provide data for analyses without consideration of the complicated multiaxial stress state at the notch for which no analytical solution exists. Because of space limitation, an analysis of the stress relaxation data and a full discussion of the notch-rupture results will be deferred to a future paper.

#### ACKNOWLEDGEMENTS

The work was supported by the Air Force Office of Scientific Research Contract F49620-77-C-0083, under the direction of Dr. A. H. Rosenstein.

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