

# CHEMICAL AND MICROSTRUCTURAL ASPECTS OF CREEP CRACK GROWTH IN INCONEL 718 ALLOY

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## Abstract

Coordinated surface chemistry and microstructural studies were carried out, in conjunction with crack growth measurements, to better understand environmentally enhanced creep crack growth in Inconel 718 alloy. Creep crack growth response was determined as a function of stress intensity factor ( $K$ ) and temperature in air, pure oxygen, moist and dry argon. Crack growth was found to be thermally activated, with apparent activation energies of about  $287 \pm 46$  kJ/mol in pure oxygen and  $191 \pm 77$  kJ/mol in moist argon. The growth rates were independent of oxygen pressure from 2.67 to 100 kPa at 973 K, and were about four orders of magnitude faster than those in high purity argon. Fractographic examinations showed predominantly intergranular cracking and the presence of copious amounts of niobium carbides. X-ray photoelectron spectroscopic (XPS) examinations showed that the oxides on the fracture surfaces were enriched in niobium oxide near the crack tip, and with iron and molybdenum away from the tip. Parallel XPS studies showed considerable segregation of niobium at the grain boundaries of polycrystals, and significant enrichment of single crystal alloy surfaces with niobium after heating for 1 h at temperatures above 775 K (with surface concentration in excess of 25 at% at 975 K). These results and observations suggest that niobium has a strong surface affinity in IN718 alloy and may be responsible for the environmental enhancement of creep crack growth at high temperatures. The oxidation and decomposition of niobium carbides as a source for segregated niobium and as the rate controlling process for crack growth are discussed. Niobium as a source for embrittlement of other nickel-base superalloys is considered.

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## Introduction

Inconel 718 alloy is widely used in high temperature applications, such as turbine discs in aircraft engines. Its sensitivity to environmentally enhanced crack growth at turbine disk operating temperatures in the range of 700 to 1033 K (*i.e.*, 426 to 760°C) has received considerable attention [1-5]. To improve its performance and to better predict its service life, it is essential to develop a better understanding of the processes of crack growth at high temperatures in this alloy. In this paper, the results of ongoing, coordinated studies of the chemical and microstructural aspects of creep crack growth in an Inconel 718 alloy are summarized to provide new understanding of the mechanism and rate controlling process for crack growth. The importance of niobium, not fully appreciated heretofore in enhancing crack growth in nickel-base alloys, is suggested and discussed.

Extensive studies have been conducted to understand the mechanisms and rate controlling processes for environmentally enhanced creep crack growth in nickel-base alloys since the early 1980s by Floreen *et al.* [1,6], Sadananda *et al.* [3,7], Pelloux and co-workers [8-10], and Pineau and co-workers [5,11]. Much of the work was focused on Inconel 718 because of its technological importance and its environmental susceptibility. Creep crack growth rates (CCGR) in Inconel 718 in air were found to be one to two orders of magnitude faster than those in vacuum or in inert environments [1,3,4]. Similar environmental sensitivity was observed also in other superalloys; for example, Rene-95, Merl-76, IN100 and X750 [3,9,10,12].

Two-stage creep crack growth response was identified, with a stage that is strongly dependent on the crack-tip stress intensity factor  $K_I$  (Stage I) and one with lesser  $K_I$  dependence (Stage II) [12,13]. It is recognized that the reported Stage I may have included transient growth that did not reflect steady-state response [12]. CCGR were also found to depend strongly on temperature, reflecting control by some thermally activated process [3,13]. The apparent activation energy for Stage II crack growth was determined to be 197 kJ/mol in air [13,14], and a much less certain value of 75 kJ/mol in vacuum [7]. Indirect evidence suggested that oxygen pressure may be important in determining crack growth enhancement and fracture mode [4,5]. There has been no direct study, however, of the influence of oxygen pressure on creep crack growth.

Fractographic studies of Inconel 718 showed that crack growth was intergranular in air and in vacuum or helium [3,4,6,7]. Floreen *et al.* [6] reported that the grain boundary facets were covered with small cavities in air and in helium. Sadananda *et al.* [3,7], on the other hand, showed no grain boundary cavities in air or in vacuum, except for those associated with small secondary-phase particles for creep at 811 K. Stucke *et al.* [4] found that cracking in vacuum was mainly transgranular (micro-voids and cleavage) at 811 and 922 K, and intergranular at 977 K. For cracking in air, no transgranular failure was observed except for a mixed mode failure at 811 K. No grain boundary cavities were reported for any of the test conditions [4].

The increased CCGR in air have been attributed to the enhancement by oxygen; principally through enhanced cavity nucleation and growth by high-pressure carbon monoxide/dioxide formed by the reactions of oxygen that diffused into the material with the grain boundary carbides [15,16]. The appropriateness of this mechanism, however, may be questioned by the absence of cavitation on the crack surfaces produced in air. Alternative mechanisms have been proposed; one involving the oxidation of grain boundary particles and release of "embrittling" species onto the boundaries [15,16], and the other, the formation of nickel, iron and chromium oxides (NiO, FeO and Cr<sub>2</sub>O<sub>3</sub>) which may inhibit or promote crack tip damage [5,17-19]. Crack

growth response may be controlled by either short-range or long-range diffusion of oxygen, and oxide formation and fracture mode were shown to depend upon oxygen partial pressure [5]. The precise mechanism, the strong environmental sensitivity of Inconel 718 and the possible influence of other alloying and impurity elements, however, were not addressed.

It appears worthwhile, therefore, to re-examine the mechanisms and rate controlling processes for environmentally assisted creep crack growth in nickel-base alloys. Because of the presence of moisture in air, the possible influence of hydrogen needs to be considered as well. Crack growth experiments and microstructural examinations were carried out on a commercial Inconel 718 alloy plate. Complementary surface chemistry studies were conducted on an Inconel 718 alloy single crystal to determine the kinetics and mechanism of reactions with oxygen, and to investigate the segregation of niobium. The results are summarized in the following sections, and their implications on environmentally assisted creep crack growth in nickel-base alloys are then discussed.

### Material and Microstructure

An Inconel 718 alloy single crystal and a 3 mm thick commercial Inconel 718 (polycrystalline) alloy plate (nominal composition in weight percent: Ni 53.7, Cr 18.1, Fe 18.0, Nb 4.85, Mo 2.9, Ti 1.0, Al 0.42) were used [20,21]. Standard heat treatment was used for the commercial alloy: 1255 K, 1 h, AC; 1005 K, 8 h, FC; 894 K, 8 h, AC. Compact-tension specimens (width: 50.8 mm; thickness: 3.2 mm) were used for all creep crack growth experiments. Single crystal samples were polished to expose the (100) surface, and were heat treated as follows: homogenize, 1375 K, 36 h + 1435 K, 2.5 h, FC; solution anneal, 1200 K, 10 h, AC; age, 1000 K, 48 h, AC.

Representative microstructures for the single crystal and polycrystalline material are shown in Fig. 1. The single crystal (Fig. 1a) showed relatively large planar  $\text{Ni}_3\text{Nb}$  precipitates along {100} planes [20]. In addition, large (irregularly shaped) niobium carbide (NbC) particles con-

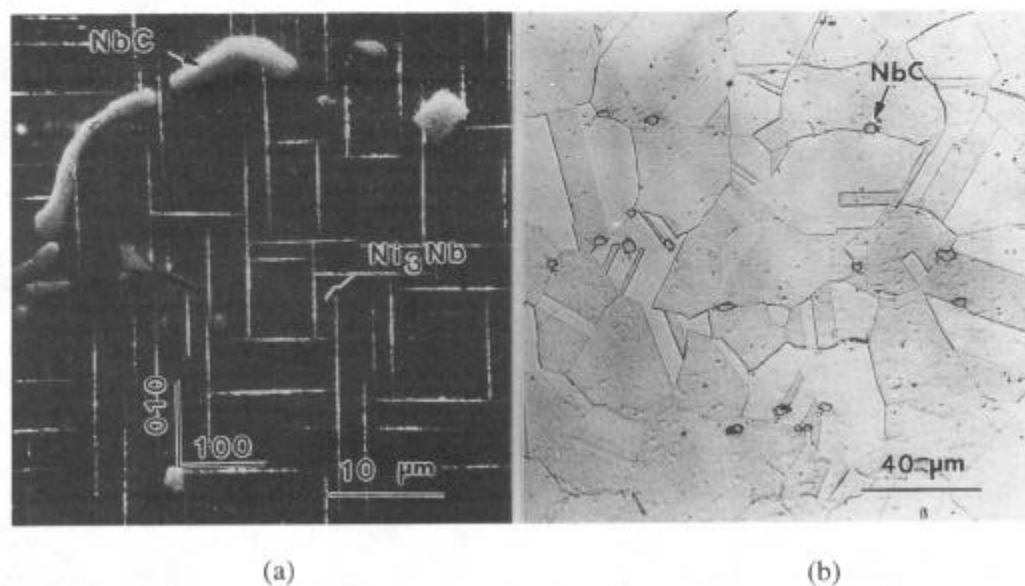


FIG. 1. Representative microstructure for (a) Inconel 718 alloy (100) single crystal, and (b) commercial Inconel 718 (polycrystalline) alloy plate.

taining about 10 at% (atomic percent) titanium, were observed. The microstructure for the commercial alloy was typical of Inconel 718, and showed the presence of NbC particles (Fig. 1b).

### Creep Crack Growth Response

Creep crack growth rate (CCGR) data from tests in oxygen and moist argon at three temperatures are shown in Figs 2a and 2b, respectively [21]. The tests included an examination of oxygen (partial) pressure (2.67, 20 and 100 kPa) at 973 K, Fig. 2a. Crack growth rates in oxygen at 973 K were consistent with those of Floreen in air [13] and were approximately twice that in moist argon. The rate was nearly four orders of magnitude higher than that in pure argon near  $K = 60 \text{ MPa}\sqrt{\text{m}}$  [21]. The rates are essentially independent of oxygen pressure between 2.67 and 100 kPa. The very slow growth rate in pure argon (versus oxygen) suggested that a pressure dependence can be expected at lower oxygen pressures (below 2.67 kPa). The mechanistic implication of this pressure independence at the higher pressures needed to be considered. It was noted that only those data in the linear region (above  $25 \text{ MPa}\sqrt{\text{m}}$ ) were to be identified with steady state growth [21]; those in the non-linear region below  $25 \text{ MPa}\sqrt{\text{m}}$  reflected transient (or non-steady state) response [12,21,22] and were excluded from further analyses.

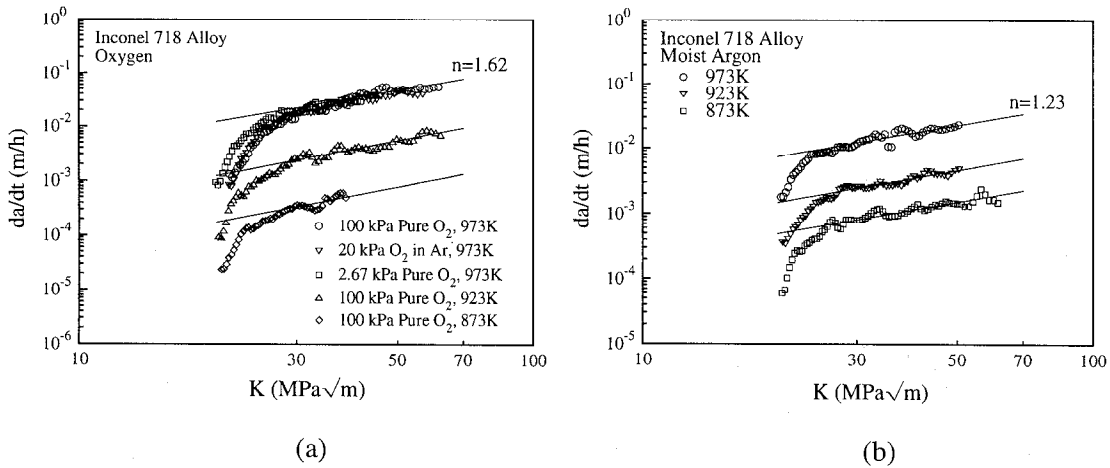


FIG. 2. Influence of temperature on the kinetics of creep crack growth (a) in pure oxygen, including the effect of oxygen partial pressure, and (b) in water vapor (moist argon) [21].

The temperature and K dependence of steady state CCGR was represented by an Arrhenius relationship of the following form [21]:

$$\frac{da}{dt} = AK^n \exp\left[-\frac{Q}{RT}\right]$$

where da/dt is the steady state CCGR; A is a temperature independent coefficient; K is stress intensity factor; the exponent, n, represents the slope on the log-log plot; Q is the activation energy; R is the universal gas constant; and T is absolute temperature. By pooling the data at all three temperatures, the exponents n for oxygen and moist argon were determined to be 1.62

and 1.23, respectively (see Fig. 2). Arrhenius plots of the data at  $K = 40 \text{ MPa}\sqrt{\text{m}}$  are shown in Fig. 3 [21] along with data from the literature [13,14].

Activation energies for oxygen and water vapor were determined to be  $287 \pm 46 \text{ kJ/mol}$  and  $191 \pm 77 \text{ kJ/mol}$ , respectively, at the 95% confidence level [21]. The value in water vapor compares well with the value of  $197 \text{ kJ/mol}$  reported for air by Floreen [13] and Sadananda and Shahinian [14]. It was suggested that the difference in activation energies between oxygen and moist argon reflects a difference in the rate controlling process and, perhaps, a change in the deleterious species (oxygen versus hydrogen, or oxygen plus hydrogen) for crack growth enhancement [21]. The possible controlling processes included reactions with the newly formed crack surfaces, diffusion through the matrix or grain boundaries and reactions with specific elements of the microstructure, and will be considered later.

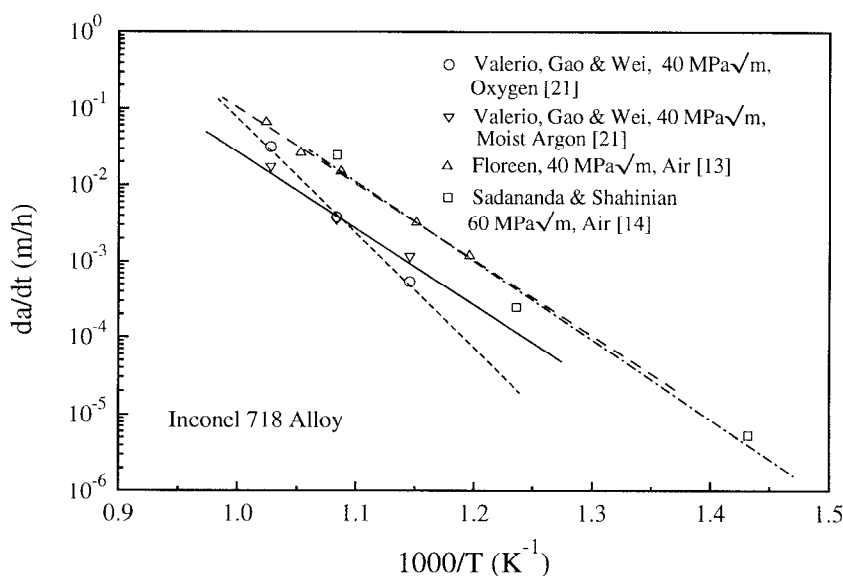


FIG. 3. Arrhenius plots showing temperature dependence of creep crack growth rates in oxygen and water vapor [21] in comparison with the data of Floreen [13] and Sadananda and Shahinian [14] in air.

### Fractographic Observations

Fractographic analysis showed that creep cracking was intergranular in all environments and temperatures used in this study [21]. Figures 4a to 4c show typical microfractographs for fracture surfaces produced in oxygen, moist argon and pure argon at 973 K. The morphology of fracture surfaces produced in oxygen is characterized by smooth grain boundary separation and clear facets, Fig. 4a. Many niobium carbides were observed; their feathery appearance indicated reactions with the environment. The water vapor fracture surface was also smooth, but the grain boundary surfaces were covered with many small particle-like features, Fig. 4b. The appearance of the argon fracture surface was very different from the others. The grain boundaries were covered by densely populated cavity-like features which are approximately  $0.5 \mu\text{m}$  in diameter, Fig. 4c. The difference in fracture surface morphology for the argon specimen suggests crack growth proceeded by a different fracture mechanism, which is consistent with the significantly lower CCGR. To determine grain boundary composition, samples were electrolytically charged with hydrogen and broken in impact. The predominantly intergranular fracture shown by these samples is indicated in Fig. 4d.

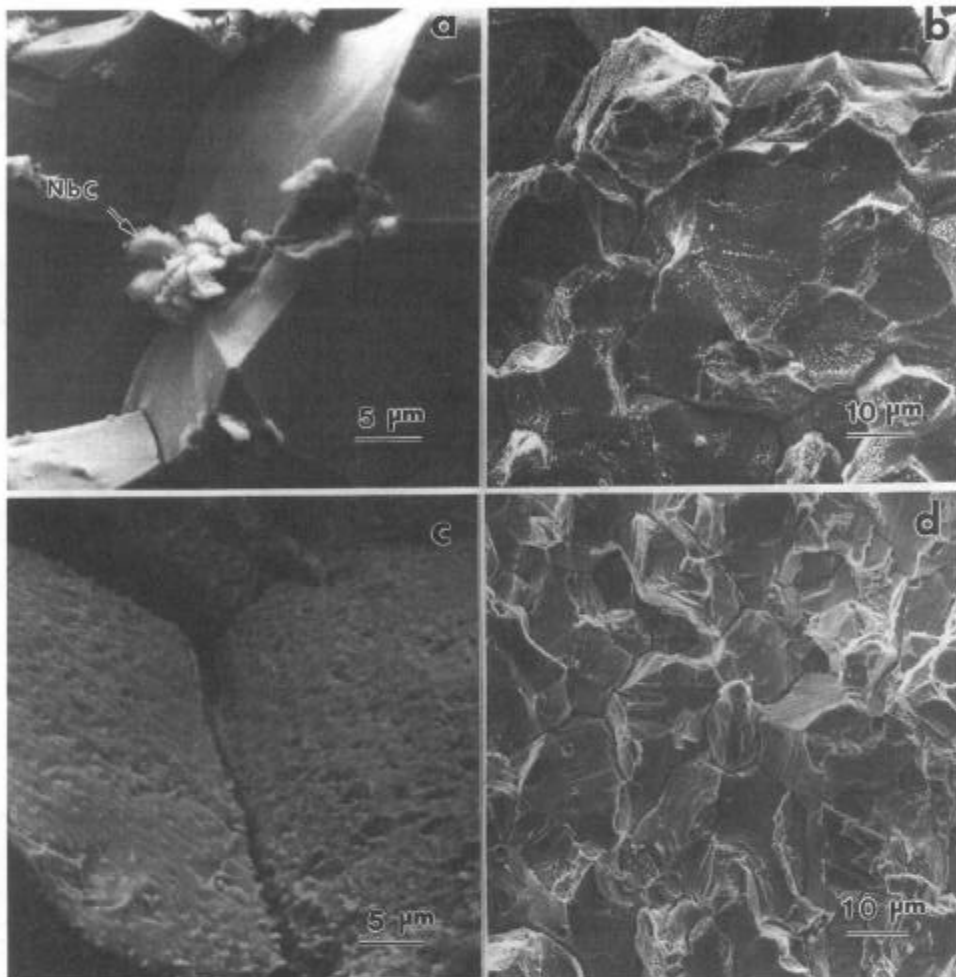


FIG. 4. SEM micrographs of fracture surfaces produced by creep crack growth at 973 K (a) in 100 kPa oxygen, (b) in water vapor (moist argon), (c) in pure argon, and (d) by *in situ* fracture of an electrolytically hydrogen charged specimen at room temperature inside the XPS chamber [20,21].

#### **Possible Role of Niobium in Enhancing Crack Growth**

The fractographic results suggest that environmentally enhanced cavitation cannot be the dominant mechanism for enhanced creep crack growth, and an alternate mechanism needs to be considered. The smooth intergranular separation suggests some form of an "interfacial embrittlement" mechanism. Evidence that suggests the role for niobium is summarized in the following subsections.

#### **Segregation of Nb and Other Elements at (100) Surface and Interactions with Oxygen.**

Thermally induced segregation of alloying and impurity elements was found on Inconel 718 alloy (100) single crystals by x-ray photoelectron spectroscopy (XPS) [20]. The unexpectedly

strong surface enrichment of niobium (Nb) after heating to 975 K for 1 h in ultrahigh vacuum (UHV) may be seen from Table 1, which compares the bulk and surface concentrations of the principal alloying elements, as determined by atomic adsorption spectroscopy and XPS, respectively [20]. The change in surface composition at this and other temperatures (from 300 K) is shown in Fig. 5. The (100) surface appeared to be relatively stable up to about 575 K, above which it began to be enriched in chromium, principally at the expense of nickel. At temperatures above 775 K, Nb and Ti began to segregate strongly to the surface, accompanied by some increase in the surface concentration of Mo. Segregation of these elements was coupled with a continued decrease in Ni and a sharp decrease in the amounts of Cr and Fe. At 975 K, the Nb surface concentration had increased from 2.1 at% on a freshly sputtered surface to 23.4 at% (Table 1); the final concentration was somewhat dependent on sample or sputtering conditions, and ranged from 20 to as high as 35 at%. These changes in the surface composition took place even though the sample had been thermally aged for more than 48 h at 1000 K. No sulfur segregation was observed [20].

Table 1: Composition (atomic %) of Inconel 718 Alloy Single Crystals [20]  
(Oxygen and Carbon Free Basis)

Composition	Ni	Cr	Fe	Nb	Mo	Ti	Al	B
Bulk	53.6	20.2	19.0	3.1	1.7	1.1	0.9	0.02
Ion Sputtered at 300 K	54.9	17.7	21.1	2.1	1.9	--	--	--
After 1 h at 975 K	37.9	18.5	12.1	23.4	2.9	4.1	--	--

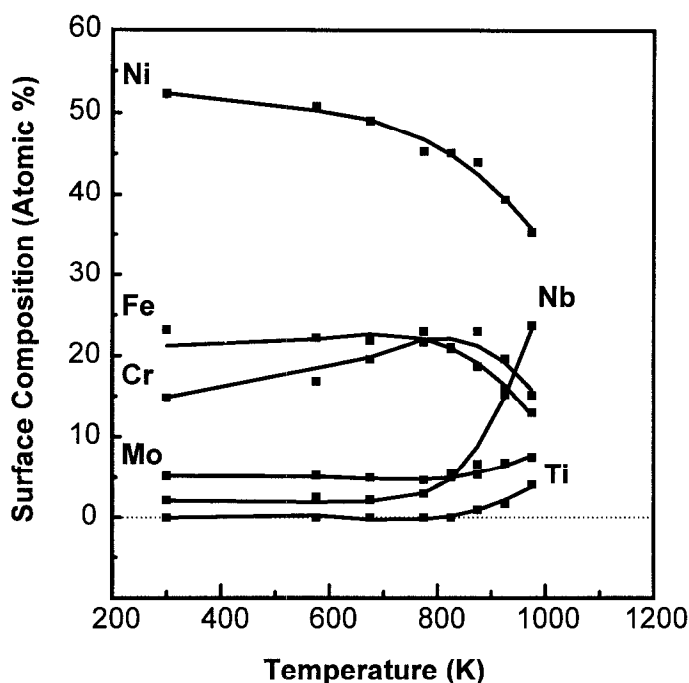


FIG. 5. Surface composition of the Inconel 718 (100) single crystal surface after heating for 1 h in vacuum at the indicated temperature [20].

More detailed studies of the kinetics and extent of niobium segregation at 1000 K were made. The results (Fig. 6) showed a strong influence of pre-adsorbed oxygen on Nb segregation. The lesser amounts of segregated Nb in vacuum suggested that the earlier results, shown in Table 1 and Fig. 5, had been affected by residual oxygen on the edges of the specimen. The formation of CO, detected during heating and confirmed by temperature programmed reaction (TPR) experiments (Fig. 7), suggested that surface enrichment of Nb was associated with the oxidation of NbC at the surface. In the TPR experiments, a surface dosed with 100 L of oxygen at 575 K was heated at  $10 \text{ K s}^{-1}$  while the rate of CO evolution from the surface was monitored with a mass spectrometer. The activation energy for NbC oxidation was estimated to be about 266 kJ/mol by analysis of the leading edge of the TPR trace (rate independent of

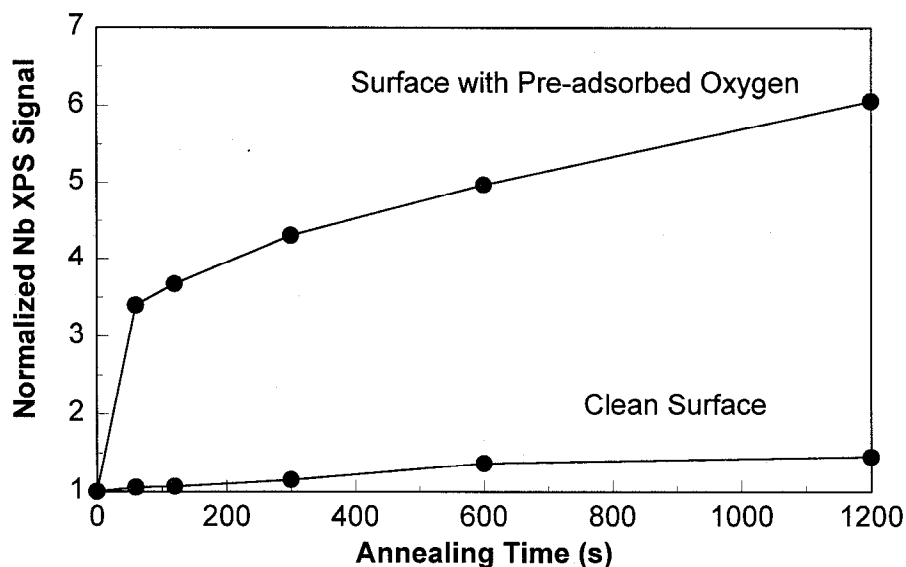


FIG. 6. Influence of pre-adsorbed oxygen on Nb segregation at 1000 K on (100) single crystal surface of Inconel 718 alloy.

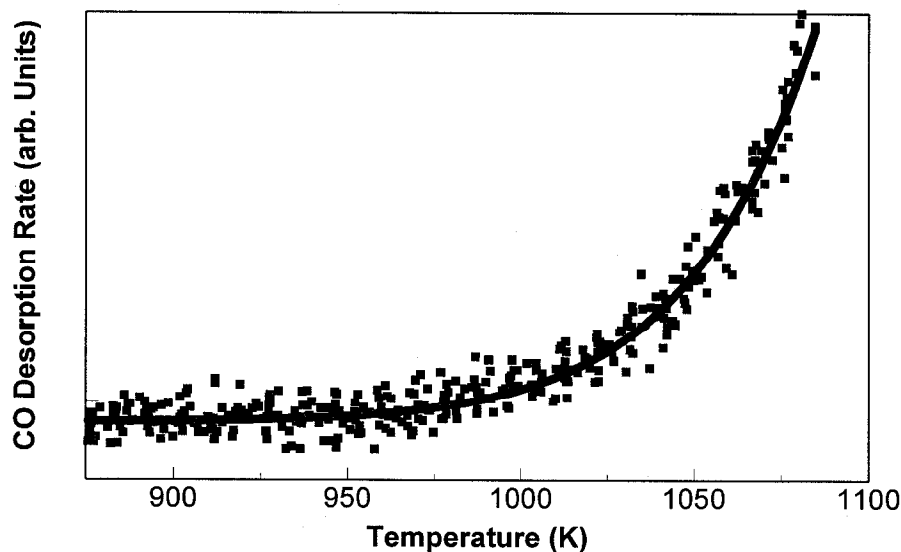


FIG. 7. Rate of CO evolution from (100) single crystal surface of Inconel 718 alloy in TPR experiments at a heating rate of  $10 \text{ K s}^{-1}$ .



surface coverage), and is consistent with the value of 287 kJ/mol reported for crack growth in oxygen [21]. Niobium oxides formed on the surface have been identified by XPS to be principally  $\text{Nb}_2\text{O}_5$ , which is known to be non-protective and brittle [23].

#### Grain Boundary Segregation of Niobium.

The thermally induced enrichment of (100) surface suggested that Nb may segregate at grain boundaries of polycrystals during heat treatment, and may participate in the subsequent environmental enhancement of crack growth. A hydrogen charged sample of the polycrystalline Inconel 718 alloy was, therefore, fractured *in situ* inside the UHV chamber of a XPS system and analyzed [20]. The average surface composition of the predominately intergranular fracture surface, based on measurements of only Ni, Cr, Fe, Nb and Mo, is given in Table 2, and clearly showed the considerable grain boundary enrichment by Nb [20]. Due to surface roughness and the small amount of transgranular fracture, the concentration of Nb on the grain boundaries might have been higher than that shown in Table 2. In contradistinction to the thermally induced segregation of Nb at the free surface of a single crystal, the observed segregation here may reflect preferential formation of  $\text{Ni}_3\text{Nb}$  precipitates at the boundaries during aging (see Fig. 8). Because of the presence of NbC particles at the boundaries (see Fig. 4a), further segregation of Nb from the decomposition of these particles during testing at high

Table 2: Grain Boundary Composition (atomic %) of Inconel 718 Alloy [20]

Composition	Ni	Cr	Fe	Nb	Mo
Bulk	52.8	21.1	18.6	3.0	1.7
Grain Boundary	49.1±5.3	18.9±2.9	15.0±2.9	12.3±3.4	4.7±1.1

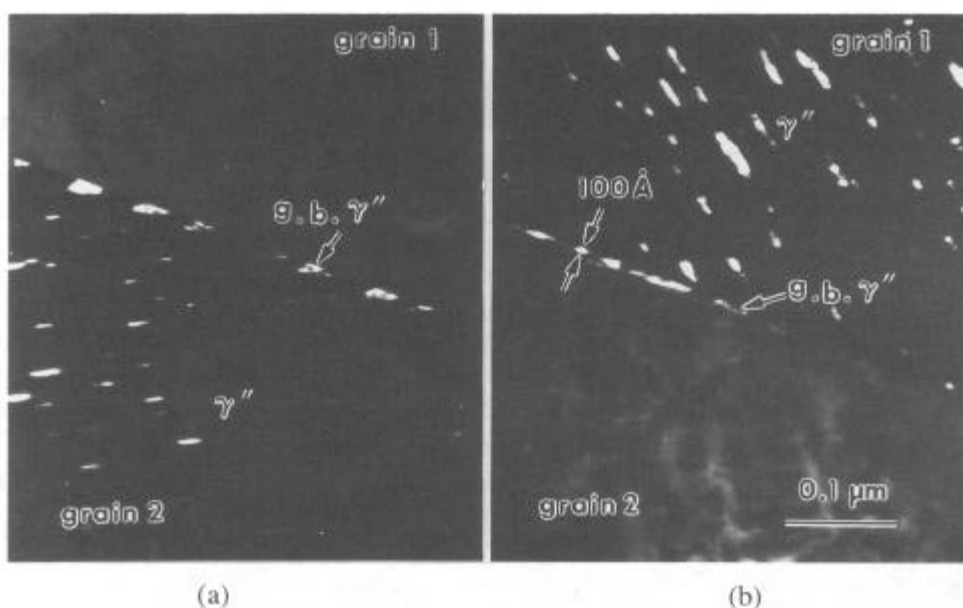


FIG. 8. Dark field images obtained using (a)  $(\frac{1}{2} 1 0)\gamma''$  reflection of grain 1, and (b)  $(1 \frac{1}{2} 0)\gamma''$  reflection of grain 2, showing the high concentration of  $\gamma''$  ( $\text{Ni}_3\text{Nb}$ ) at the grain boundary in the polycrystalline Inconel 718 alloy.

temperatures cannot be precluded. As in the case of the single crystals, no segregation of sulfur at the grain boundaries was detected in the XPS spectra [20].

#### Preferential Oxidation of Nb at the Crack-tip.

Figure 9 shows the distribution of principal alloying elements in the surface oxides formed on the fracture surface of a specimen following creep crack growth in pure oxygen at 923 K. Exposure time to oxygen was estimated to be 3 to 21 h depending on distance from the crack tip (0 to 18 mm). Preferential oxidation of niobium was noted near the crack-tip (~10 at% versus ~3 at% in the bulk) principally at the expense of nickel. Away from the crack-tip (*i.e.*, at longer exposure times), there was enrichment of Fe and Mo in the surface oxides.

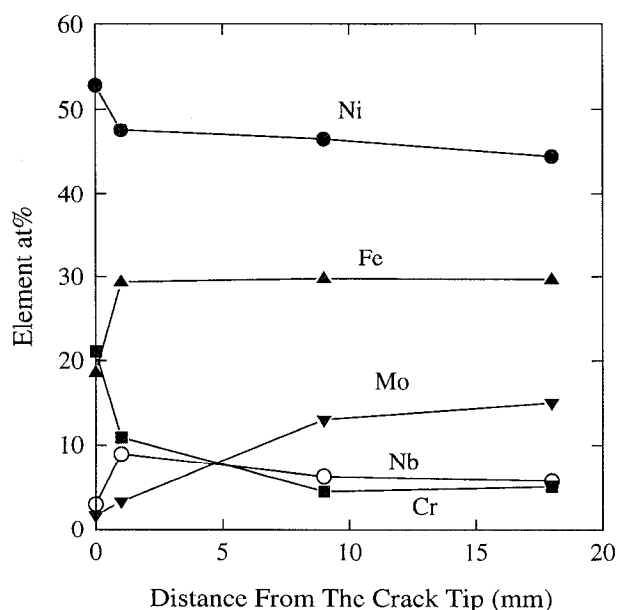


FIG. 9. Distribution of principal alloying elements in the surface oxides formed on a creep crack growth specimen tested in pure oxygen at 923 K. Data at zero distance are the bulk composition of the alloy.

#### Discussion

These initial results strongly suggested that the segregation and enrichment of niobium at grain boundary surfaces played an important role in the environmental enhancement of creep crack growth in Inconel 718 alloy. The precise mechanism, however, still needs to be established. Available evidence suggests that the mechanism involves the formation and fracture of a brittle niobium oxide ( $\text{Nb}_2\text{O}_5$ ) film on the boundary surfaces which reduces the alloy's crack growth resistance. Oxidation and decomposition of NbC particles at the boundaries appear to be the principal source of niobium. The segregated niobium at the grain boundaries is expected to also contribute to the embrittlement. As the source of oxygen would be that of an external (pseudo-equilibrium) surface oxide, the mechanism would be consistent with the observed independence on oxygen pressure from 2.67 to 100 kPa. The rate controlling process would be identified with the reactions of oxygen with niobium carbides (266 *versus* 287 kJ/mol) or with the segregated niobium. The lower activation energy for crack growth in moist argon and air (about 190 kJ/mol) still needs to be understood. It may reflect the additional embrittlement

contribution from hydrogen, and a transfer of the controlling process. Further studies are underway to critically examine this and other hypotheses in developing a more complete understanding.

It has been long recognized that not all nickel-base superalloys are highly sensitive to environmentally enhanced crack growth [3,7,10,24]. Bain and Pelloux [10] showed that creep crack growth rates in air at 923 K can differ by more than three orders of magnitude between different superalloys with very similar mechanical properties. They attributed the difference in sensitivity to the influence of grain boundary structure and composition on oxidation response. It was suggested that elements which segregate to grain boundaries (*e.g.*, C, B, Zr, S and P), grain size, and grain boundary precipitate size and morphology might be important [10]. None of the existing mechanisms, however, can explain these differences in creep crack growth response. The role of niobium now offers a possible explanation. Environmental sensitivity for the various alloys is shown in terms of the ratio of growth rates in air ( $\dot{a}_e$ ) and those in vacuum or argon ( $\dot{a}_r$ ) in Fig. 10. The trend strongly suggests the influence of niobium, and indicates the need for further mechanistic understanding.

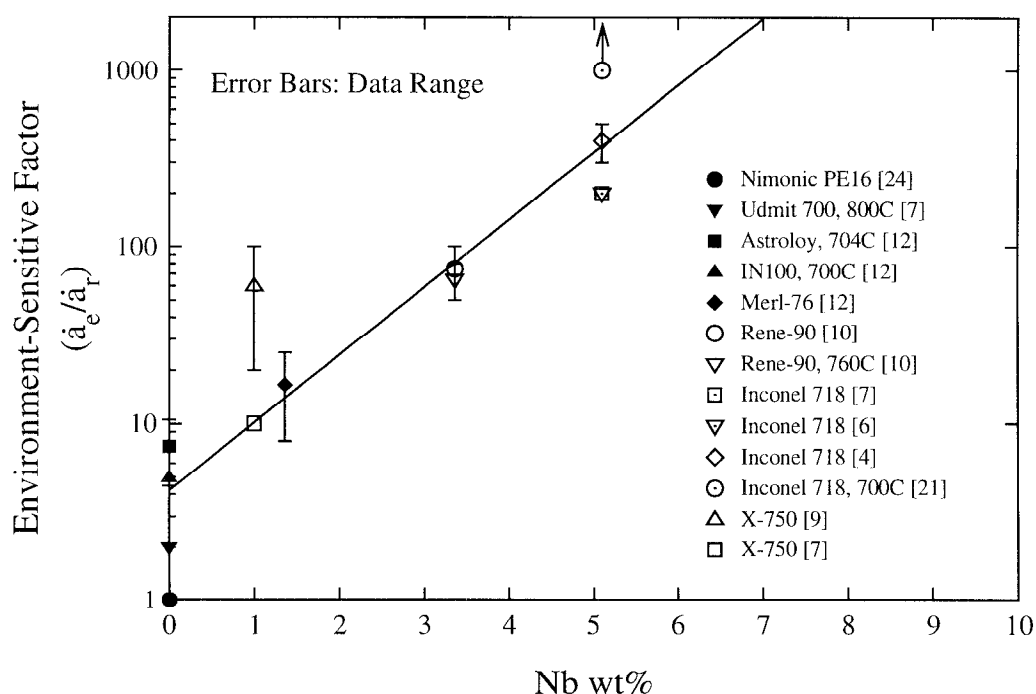


FIG. 10. Environmental crack growth sensitivity of nickel-base superalloys as a function of niobium concentration [4,6,7,9,10,12,21,24]. (Tests at 650°C, except as indicated in the figure.)

### Conclusion

Coordinated fracture mechanics, surface chemistry and microstructural studies were carried out to better understand environmentally enhanced creep crack growth in Inconel 718 alloy. Steady state crack growth was found to be thermally activated, with apparent activation energy of  $287 \pm 46$  kJ/mol in pure oxygen and  $191 \pm 77$  kJ/mol in water vapor, at the 95% confidence level. No oxygen pressure dependence was observed for pressures between 2.67 and 100 kPa. Oxygen and possibly hydrogen are identified as the embrittling species for environmentally enhanced creep crack growth. The results suggest the segregation of niobium to grain boundaries and

its reaction with oxygen and hydrogen to be responsible for the enhancement of creep crack growth in Inconel 718. The segregation resulted in part from the decomposition of NbC particles at the boundary and the rate controlling prior crack growth appears to be that of the oxidation of NbC. Niobium as a source for embrittlement of other nickel-base alloys is suggested. Further studies are recommended to better understand environmental enhancement of crack growth in the nickel-base superalloys.

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