MECHANISTIC CONSIDERATIONS OF OXYGEN ENHANCED CRACK GROWTH IN INCONEL 718

Robert P. Wei¹, Zhi-Fang Huang¹, Christopher F. Miller² and Gary W. Simmons²

¹Department of Mechanical Engineering and Mechanics
²Department of Chemistry
Lehigh University
Sinclair Laboratory, 7 Asa Drive
Bethlehem, PA 18015 USA

Abstract

A coordinated, multidisciplinary study was conducted to elucidate the mechanism for oxygen enhanced crack growth in Inconel 718 at high temperatures. Sustained-load tests were conducted in high-purity argon and oxygen, at 873 to 973 K, to characterize the kinetics of crack growth. The reactivity of the alloy, Nb, NbC and Ni₃Nb with oxygen was determined by x-ray photoelectron spectroscopy (XPS). Crack growth experiments, in oxygen at 973 K, were interrupted and the specimens were charged with hydrogen and fractured in UHV to determine the state of oxidation of various elements, particularly ahead of the crack tip. The existence of a damage zone ahead of the crack tip had been demonstrated previously by reloading interrupted specimens of comparable alloys in argon at lower temperatures and stress intensity levels. A mechanism for oxygen enhanced crack growth in Inconel 718, based on the formation of a brittle niobium oxide film ahead of the crack tip from the oxidation of grain boundary Ni₃Nb and Nb-rich carbides, is proposed and discussed.

Introduction

Inconel 718 is widely used in high temperature applications, such as turbine discs in aircraft turbine engines. Its sensitivity to oxygen enhanced crack growth (OECG) at operating temperatures in the range of 700 to 1000 K (or about 430 to 750°C) has long been recognized. The presence of oxygen can increase the rate of crack growth by more than 4 orders of magnitude (*i.e.*, >10⁴X) over that in inert environments (for example, ultrahigh purity argon). To improve its performance and to formulate mechanistically based probability methods for reliability analysis and life-cycle management, it is essential to develop improved understanding of the processes of crack growth (*i.e.*, damage evolution) in this alloy at high temperatures. In this paper, the results of a coordinated, multidisciplinary study, at Lehigh University, of oxygen enhanced crack growth in an Inconel 718 alloy are summarized. These results, building upon the work reported at the 1995 Symposium [1], provide new understanding of the mechanisms for the enhancement of crack growth by oxygen, and further insight into the role of the various alloying elements.

Efforts to understand the mechanisms for this enhancement have been made since the 1970s, for example [2-11]. Floreen and Raj [2] have categorized the various mechanisms into two groups. The first group involves environmentally enhanced formation and growth of cavities, or microcracks, at grain boundaries ahead of the crack tip. One such mechanism attributed the enhanced crack growth to the reaction of oxygen with grain boundary carbides to produce high pressure CO/CO₂, which enhanced cavity growth and grain boundary separation [4,5]. The second type is associated with preferential formation of a grain boundary oxide layer at the crack tip, either through the formation of oxides of nickel and iron (NiO and FeO) [6-8], or through a two step process of forming first NiO and FeO and subsequently Cr₂O₃ [9].

More recent studies at Lehigh University and the University of Maine at Orono [1,12-19] on an Inconel 718 alloy and a Ni-18Cr-18Fe ternary alloy suggested that niobium could play a significant role, and raised concerns regarding the viability of these proposed mechanisms for crack growth enhancement by oxygen. The results showed that sustained load crack growth rates in oxygen at 973 K were increased, for example, by more than 10⁴X over those in highpurity argon for Inconel 718 [1,12]. It was suggested that the enhancement in crack growth was caused by the formation and rupture of a non-protective and brittle Nb₂O₅-type oxide film at the grain boundaries; the films being formed through the oxidation and decomposition of niobiumrich carbides and, perhaps, oxidation of γ'' (Ni₃Nb) precipitates at the grain boundaries at a much higher concentration (~10:1, Ni₃Nb:NbC). Crack growth rates in the ternary alloy, on the hand, were found to be essentially unaffected by oxygen [19]. A comparison of these results with those on a range of nickel-based superalloys in the literature showed the strong dependence of the environmental sensitivity factor (i.e., the ratio of crack growth rates in the deleterious and inert environments) on Nb concentration [13]. The sensitivity factor increased by more than 10⁴X for Nb from 0 to 5 wt pct, albeit the sensitivity varied widely among the alloys. These findings suggested that the role of Nb on enhancing crack growth in oxygen. heretofore not recognized, needs to be carefully examined. The "insensitivity" of the Ni-18Cr-18Fe ternary alloy, with copious amounts of M₂₃C₆ carbides at the grain boundaries, calls into question the viability of both groups of proposed mechanisms.

Here, key results from the earlier studies [12-19] are briefly summarized, and more recent results from coordinated surface chemistry and fracture mechanics studies [20-22], to elucidate the role of niobium on crack growth in Inconel 718 in oxygen at high temperatures, are presented. Based on the findings and information in the literature, the mechanisms for crack growth and the role of niobium and of other elements are considered. A 3.2-mm thick plate of commercial Inconel 718 alloy was used for the crack growth and companion surface chemistry studies [12-20]. The nominal composition of the alloy, in wt pct, is as follows: Ni 53.7, Cr 18.1, Fe 18.0, Nb 4.85, Mo 2.9, Ti 1.0, Al 0.42, and the corresponding atomic percentages are Ni 52.9, Cr 20.2, Fe 18.7, Nb 3.0, Mo 1.8, Ti 1.2, and Al 0.9. The alloy was heat treated as follows: solution annealing in argon at 1225 K for 1 h, and aged in argon at 1005 K for 8 h,

followed by 894 K for 8 h. The average grain size was about 45 μ m. Pure Nb and Ni₃Nb, and specially prepared films of NbC were used for the supporting surface oxidation studies.

Suggestion of the Embrittling Role of Niobium [1,12-19]

Crack Growth Response

Crack growth rate data from tests in high-purity oxygen and moist argon at three temperatures are summarized in Figs 1a and 1b, respectively [1,12]. The tests included an examination of oxygen (partial) pressure (2.67, 20 and 100 kPa) at 973 K, Fig. 1a. The growth rates were essentially independent of oxygen pressure between 2.67 and 100 kPa, and the rate in oxygen near $K = 60 \text{ MPa-m}^{1/2}$ was more than four orders of magnitude (*i.e.*, >10⁴X) faster than that in high-purity argon (with an estimated partial pressure of residual oxygen below 10^{-17} Pa). Crack growth was found to be thermally activated, with an apparent activation energy of $287 \pm 46 \text{ kJ/mol}$ in pure oxygen [12].

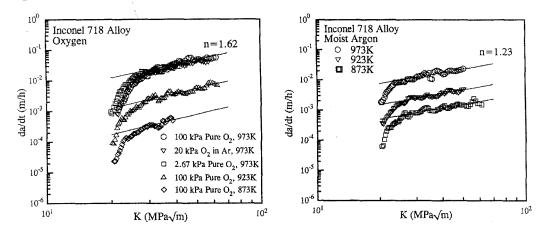


Figure 1: Influence of temperature on the kinetics of environmentally enhanced crack growth in (a) pure oxygen and an oxygen-argon mixture, and (b) in water vapor (moist argon) [1,12].

Fracture Surface Morphology and Microstructural Examinations

Fractographic analysis showed that growth was intergranular in oxygen, and moist and highpurity argon from 873 to 973 K. A representative microfractograph for crack growth in oxygen at 973 K, typical of those at other temperatures, is shown in Fig. 2a [12]. For comparison, the fracture surface exposed by impact fracture at LN₂ temperature after hydrogen charging is

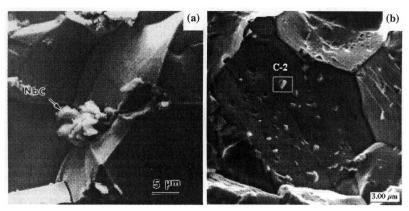


Figure 2: SEM microfractograph of surfaces produced by (a) sustained-load crack growth at 973 K in 100 kPa oxygen, and (b) by *in situ* fracture of an electrolytically charged specimen at LN₂ temperature [12,14].

shown in Fig. 2b [14]. The morphology of fracture surfaces produced in oxygen is characterized by smooth separation along grain boundaries and clear facets. Many niobium-rich carbides were observed; the feathery appearance of those at the grain boundary triple points indicated reactions with oxygen. The morphology of surfaces produced in argon (not shown) was quite different [12]. The grain boundary surfaces were covered by densely populated, cavity-like features, approximately 0.5 μ m in diameter. The difference in fracture surface morphologies suggested that crack growth occurred by different micromechanisms, and is consistent with the large differences in crack growth rates. The presence of smaller niobium-rich carbides along the grain boundaries has been identified by energy dispersive spectroscopic (EDS) analysis and analytical electron microscopy [14], and is readily seen from Fig. 2b for the hydrogen charged sample. The morphology and distribution of γ'' precipitates along the grain boundaries are shown in Fig. 3 [15], and those of the smaller niobium-rich carbides are given in [14].

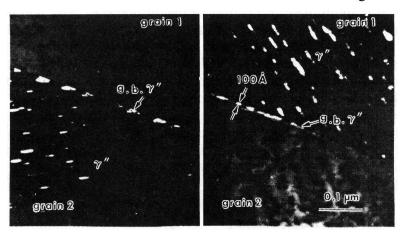


Figure 3: Dark field image showing the distribution of γ'' precipitates along the grain boundary in Inconel 718 alloy [15].

Reactivity of Inconel 718 Surfaces and Grain Boundary Enrichment of Niobium

Studies of polished surfaces of Inconel 718 single crystals and polycrystals, and of grain boundary surfaces exposed by *in situ* fracture of hydrogen charged samples were carried out by x-ray photoelectron spectroscopy (XPS) [1,17,18]. Strong enrichment of Nb was observed on the alloy surfaces after heating to 973 K for 1 h in ultrahigh vacuum (UHV). Enrichment was evidenced by the increased Nb signal in the XPS spectra (Fig. 5). Its extent was determined from analyses of the spectra, and represented an increase from 2.1 at. pct for a freshly sputtered surface to 20 to 35 at. pct (*i.e.*, more than 10 fold increase in Nb). The final concentration

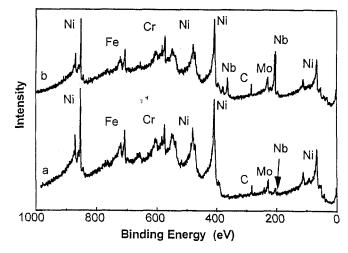


Figure 4: X-ray photoelectron spectra of Inconel 718 alloy single crystal: (a) as ion sputtered, and (b) after heating to 975 K [18].

depended somewhat on the sample and the sputtering conditions. It was suggested that the results had been affected by residual oxygen that was left on the edges of the specimens after ion sputtering. The evolution of CO during heating, and confirmed by temperature programmed reaction experiments, suggested that surface enrichment of Nb was associated with the oxidation of NbC at the surface and that the surface mobility of Nb was high. XPS analyses of grain boundary surfaces also indicated enrichment of Nb by about 4-fold relative to the bulk concentration. This enrichment (*versus* the thermally induced enrichment) has been attributed to the preferential formation of γ'' (Ni₃Nb) precipitates at the grain boundaries (occupying about 25 pct of the boundary surfaces) during aging (see Fig. 3).

Confirmation of the Principal Embrittling Role of Niobium [20-22]

The foregoing findings suggested that the enhancement of crack growth had resulted from the oxidation and decomposition of niobium rich carbides at the grain boundaries ahead of the crack tip during crack growth, increasing the niobium surface concentration by about 10 fold [1,13,16]. This niobium was believed to be in the form of a brittle, niobium oxide (Nb₂O₅), which promoted grain boundary cracking in the alloy [1]. The γ'' (Ni₃Nb) precipitates along the grain boundaries were expected to participate in crack growth enhancement, but no direct evidence for their oxidation was provided. A series of studies, therefore, was conducted to further test the hypothesis that Nb played an essential role in the enhancement of crack growth in oxygen at high temperatures. The goals of the studies were as follows: (a) to provide direct evidence for the oxidation of NbC and Ni₃Nb, (b) to assess the reactivity of the alloying elements in Inconel 718 with oxygen, and (c) to determine the extent of oxygen penetration ahead of the crack tip and the oxidation state of the various alloying element [20-22]. The principal findings are summarized in the following subsections.

Reactions of Nb Compounds with Oxygen

To provide direct evidence for the reactions of Nb compounds with oxygen, specimens of Nb, Ni₃Nb and a specially grown film of NbC were given the same exposure to oxygen at 873, 923 and 973 K and analyzed by XPS [20]. Representative spectra for Ni₃Nb and the NbC film at 973 K are shown in Fig. 5 along with the component spectra for Ni₃Nb and NbC and the

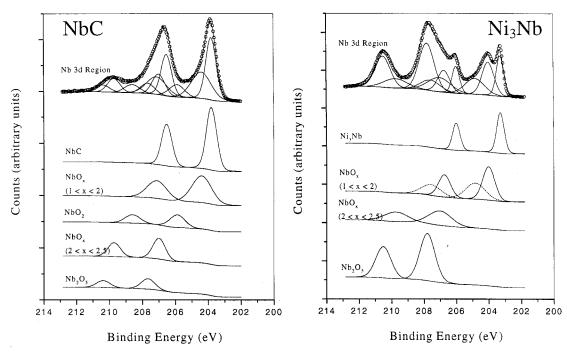


Figure 5: XPS spectra (Nb region: $3d_{3/2,5/2}$) of NbC and Ni₃Nb after oxidation at 973 K in 5 x 10^{-4} Pa of O₂ for 45 min. [19]

various oxides that were formed. The results confirm the fact that both Ni₃Nb and NbC react with oxygen at these temperatures to form niobium oxides of ranging stoichiometry, with a greater propensity for Ni₃Nb to oxidize to Nb₂O₅ than NbC. As such, oxidation of both NbC (*i.e.*, Nb-rich carbides) and Ni₃Nb could contribute to the enhancement of crack growth at high temperatures.

Reactivity of Inconel 718 at High Temperature

To determine its reactivity to oxygen, polished and ion-sputtered surfaces of Inconel 718 were analyzed by XPS under two sets of conditions [21]. Ion sputtering at a beam voltage of 5.0 kV for 25 minutes was used to establish contaminant-free starting surfaces for study. Each of the surfaces was heated in ultrahigh vacuum (UHV) for 15 min. to bring it to 973 K, and was held at temperature under one of the following two conditions. The first involved holding the sputtered surface in UHV (at a background pressure of 8 x 10⁻⁷ Pa, corresponding to partial pressures of O₂, H₂O, CO, CO₂ and hydrocarbons) for 60 min. to determine the short-term thermal stability of the alloy and to provide a reference for the oxidation reactions. The second involved an exposure to 5 x 10⁻⁴ Pa of oxygen at 973 K for 45 min. to determine the reactivity of the various alloying elements. The oxygen pressure of 5 x 10⁻⁴ Pa was chosen principally for experimental convenience and to reflect the anticipated low concentration (or "equivalent" pressure) of oxygen ahead of the crack tip.

Comparisons of the normalized, relative-peak-area percentages for the sputtered and reacted surfaces are shown in Fig. 6 [21]. The relative-peak-area percentages were computed from the XPS peak areas for each elemental region that had been normalized using their corresponding sensitivity factors. Figure 6a shows that the surface heated in UHV at 973 K was enriched in Cr. Al and Nb along with a modest increase in Ti. Less than 20% of the surface Cr was oxidized (Cr₂O₃), while over 75% of Ti (TiO), Al (Al₂O₃) and Nb (Nb₂O₅) were oxidized. Oxidation of these elements was caused, most likely, by reactions with the dissolved atomic oxygen in the specimen and the residual O₂ and H₂O in the preparation chamber. Exposure to oxygen (5 x 10⁻⁴ Pa) at 973 K, on the other hand, resulted in the preferential oxidization of Cr, Fe and Nb, while Ni decreased considerably and remained unoxidized (Fig. 6b). The preferential oxidation of Cr and Fe at low oxygen pressures is well-established [9,10]. The observed Nb enrichment in the form of Nb₂O₅ demonstrates the high reactivity of Nb in Inconel 718 over a range of low oxygen pressures (from UHV to 5 x 10⁻⁴ Pa O₂). Excluding the negligible increase in Ti, the enrichment and oxidation of Cr, Al and Nb demonstrates that these phases are highly reactive at low exposures and are, hence, possible causes for the enhancement of crack growth.

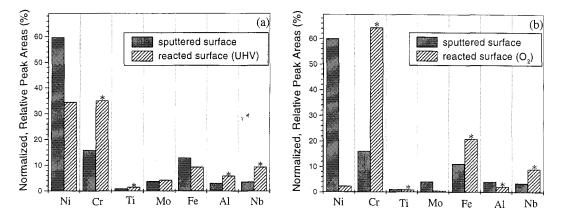


Figure 6: XPS results showing the normalized, relative-peak-area percentages for surfaces of Inconel 718: sputtered surface *versus* exposure to (a) UHV for 60 min. at 973 K, (b) 5 x 10^{-4} Pa O₂ for 45 min. at 973 K; The "*" indicates oxidation.

To ascertain the existence of an "oxygen embrittled" zone ahead of the tip of a growing crack, interrupted experiments were carried out on other nickel-based alloys [22]. In these experiments, sustained-load crack growth in oxygen was stopped, and the specimen was partially unloaded. The test chamber was evacuated and back-filled with ultrahigh purity argon (with $<10^{-17}$ Pa O₂), and the specimen was then reloaded to the same test load. Representative crack growth response for a specimen of a Nb containing alloy tested at K = 33 MPa-m^{1/2} and 873 K is shown in Fig. 7. The results showed the presence of an embrittled zone of about 80 μ m over which the crack growth rate decreased from the pre-interruption rate in oxygen to that for argon. The profile is consistent with that of the concentration profile associated with oxygen diffusion.

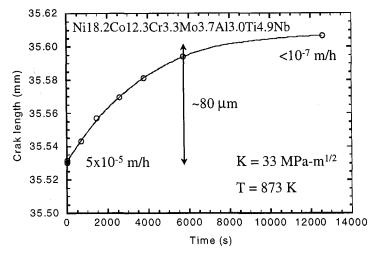
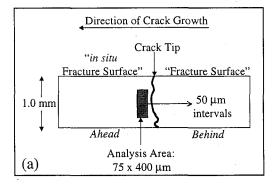


Figure 7: Representative crack growth response for a P/M alloy in ultrahigh purity argon following prior growth in oxygen [22].

To determine which of the elements might be oxidized ahead of the crack tip in the Inconel 718 alloy, interrupted tests were conducted in 135 kPa of oxygen at K = 60 MPa-m^{1/2} and 973 K. Small samples containing the crack tip region were cut from interrupted test specimens. They were electrolytically charged with hydrogen, and broken under ultrahigh vacuum inside the preparation chamber of the XPS spectrometer and analyzed by XPS [21]. The instrument was operated in its high spatial resolution mode to provide an approximate analysis area of 75 x 400 µm. The regions of the fracture surface used for analysis are illustrated schematically in Figure 8a. The "in situ fracture surface" is the area ahead of the crack tip and is produced by in situ fracture in UHV, and the "fracture surface" is the area behind the crack tip produced during the crack growth experiment. XPS analyses were performed at 50 µm intervals along the crack



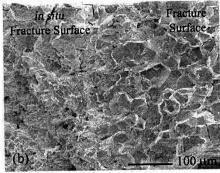


Figure 8: Crack-tip region of an interrupted crack growth specimen of Inconel 718 that was analyzed by XPS: (a) schematic representation and (b) a representative SEM microfractograph of the region [20].

growth direction, paralleling the 75 μm dimension of the analysis window. Detailed procedures are given in [21].

A representative SEM microfractograph of the crack tip region of the interrupted crack growth specimen is shown in Fig. 8b. The *in situ* fracture region on the left was exposed by impact fracture following hydrogen charging, under vacuum, inside the XPS spectrometer. It consisted of a transgranular region (approximately the left one-third of the microfractograph) that was unaffected by oxygen (and subsequent hydrogen charging) and an intergranular region that represented the oxygen affected region (OAR). The OAR is the area that is affected by the penetration of oxygen ahead of the crack tip during crack growth, and is to be identified by chemical analysis. The remaining region to the right, consisting principally of intergranular fracture, was produced by crack growth in 135 kPa of oxygen at 973 K.

The profile of the various elements, in terms of normalized, relative-peak-area percentages, are given as a function of distance along the fracture surface in Fig. 9a. The 0 μ m position represents the starting location for the XPS analyses; the 900 μ m region encompassed the areas ahead and behind the tip of the interrupted crack. The elemental profiles show a substantial increase in Cr and Nb beginning at 200 μ m, while Ni and Fe decrease considerably, but no surface Al. The various elements were found to be in their metallic state over the first 150 to 200 μ m, and to be oxidized to different stoichiometry along the surface.

The location and size of the OAR ahead of the crack tip was determined using the XPS data for Ni, Cr and Nb. The extent of Ni, Cr and Nb oxidation was found by (a) curve fitting the XPS spectra, (b) normalizing the oxide signal for each element and (c) fitting the data for each with a sigmoidal curve (Fig. 9b). Because Ni in Inconel 718 is known to oxidize in 1.0 Pa O₂ at 973 K [9,10], it was assumed that Ni oxidized at least to the crack tip during sustained-load crack growth. As such, the crack tip position was estimated to be at 300 μm, corresponding to the first indication of Ni oxidation. The limit of the OAR is identified with the beginning of oxidation of

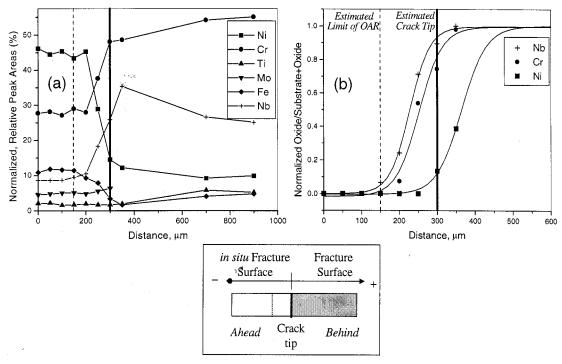


Figure 9: XPS results for Inconel 718 surfaces produced by crack growth in oxygen at 973 K and $K = 60 \text{ MPa-m}^{1/2}$ showing: (a) variation of normalized, relative peak area percentages of the alloying elements, and (b) normalized oxidation profile for Nb, Cr, and Ni, in relation to the estimated boundaries of the oxygen affected region (OAR) [21].

Nb (the most reactive of the three elements), and is estimated at 150 μ m. The estimated size of the OAR (or distance of oxygen penetration ahead of the crack tip) is, therefore, approximately 150 μ m (at K = 60 MPa-m^{1/2}), which is consistent with the direct measurement of 80 μ m in another alloy at about one-half the K level [22].

The XPS data provided direct evidence for the oxygen penetration ahead of the crack tip in concert with crack growth. It caused the enrichment and oxidation of Nb and Cr to form oxides of ranging stoichiometry in the OAR. The extent of oxidation is consistent with the expected oxygen concentration profile associated with diffusion. Oxidation of Nb within the OAR provides support for crack growth mechanisms that involve Nb oxidation ahead of the crack tip. Within the OAR, Ni and Fe remained essentially in their metallic state (i.e., not oxidized). These findings bring the role of Ni and Fe in enhancing crack growth into question. It is to be recognized that the chemical changes ahead of the crack tip, and the extent of Ni and Fe oxidation at low oxygen exposures near the crack tip were not considered in the previous studies that proposed mechanisms involving Ni and Fe [4,5,9,10]. Figure 10b shows that Nb oxidized further ahead of the crack tip than Cr. This preferential oxidation of Nb at low levels of oxygen is consistent with the high-temperature oxidation results on Inconel 718 (Fig. 6), which showed Nb to have oxidized to a greater extent in UHV than Cr, while Ni and Fe remained unoxidized. Although aluminum oxidation and enrichment were also observed during heating in UHV, the absence of aluminum on the fracture surface suggests that it did not contribute to the oxygen enhanced crack growth in Inconel 718.

Summary

These results, taken *in toto*, confirm the postulated role of niobium in enhancing the rate of crack growth in oxygen in Inconel 718 at high temperatures. The enhancement mechanism is the oxidation and decomposition of niobium rich carbides at the grain boundaries to form a brittle film of Nb₂O₅-type oxides on the boundary surfaces, and implied a high degree of mobility for Nb that is "freed" through the oxidation and decomposition of the niobium-rich carbides. The XPS results on the reactions of oxygen with Ni₃Nb showed that Nb was preferentially oxidized over Ni, and that the reactions were more rapid than those with NbC. The Ni₃Nb precipitates, therefore, are also expected to play a significant role in the enhancement of crack growth in Inconel 718. The fact that the results indicate that Ni and Fe did not oxidize ahead of the crack tip suggests that the mechanisms involving the oxidation of these elements may not be viable. Although Cr was oxidized, the absence of environmental sensitivity in the Ni-Cr-Fe ternary alloy suggests that it also may not be a player. These results also demonstrated the efficacy of a coordinated multidisciplinary approach, and it is hoped that the approach can be applied to develop further mechanistic understanding of environmentally enhanced crack growth in other nickel-based superalloys.

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