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A New Approach Towards Characterizing Microstructural Influence on Material Behavior Under Very High Cycle

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14. ABSTRACT
Very high cycle fatigue (VHCF), in which components undergo fatigue lifetimes well beyond traditional design limits of 107 cycles, is not well understood and is becoming an increasingly prevalent deformation state in aerospace applications. Components are now designed to handle increasingly long lifetimes (>109 cycles), and it is critically important to be able to accurately predict when these components will fail and to intelligently tailor them for improved performance. Toward this end, a new methodology was developed for the small-scale investigation of fatigue crack initiation and growth during VHCF loading, and was used to investigate environmental and microstructural effects on the fatigue lifetimes of the polycrystalline titanium alloy Ti-6242S. Small fatigue crack growth in Ti-6242S, a commonly utilized alloy in aerospace applications, was examined in vacuum and in controlled partial pressures of water vapor, high purity oxygen, and high purity hydrogen.

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Accomplishments/New Findings:

- The creation, in collaboration with Chris Torbet at UCSB, of a new system to perform in-SEM ultrasonic testing. This is, to the best of the investigator's knowledge, the only system of its type in the world, and provides the capability to monitor and track crack growth *in-situ* at the microstructural length scale during Very High Cycle Fatigue (VHCF).
- The successful creation of a new experimental methodology for small-scale characterization that combines ultrasonic fatigue testing at 20 kHz; in-SEM, full-field deformation mapping at the microstructural length scale; and pre- and post-mortem crystallographic characterization via EBSD. The in-SEM deformation mapping at the microstructural length scale necessitated the creation of new chemical techniques for the self-assembly of nanoparticles (used as tracking markers) on Ti6242, and the development of correction algorithms for the complex spatial and temporal distortions that are inherent to SEM micrographs.
- Environment (humidity) played a large role in determining fatigue life in the VHCF regime. The magnitude of this effect was remarkable, as VHCF imparts very low crack opening displacements.
- Interestingly, fatigue lifetimes at 133 Pa high purity H_2 were significantly longer, on the order of a factor of 2x-4x, than lifetimes at the same vapor pressure (133 Pa) of either high purity 0_2 or H_2O.
- In the VHCF regime, there was predominately transgranular crystallographic crack growth with a high propensity of cracking along the basal planes, particularly in primary alpha grains.
- Decelerations in the fatigue crack growth rate were correlated with crack-tip interactions with the local microstructure.
- A change in environmental conditions – such as the introduction of increased levels of oxygen - was found to have the capability to assist a crack in overcoming a microstructural barrier where it had arrested. It is interesting to note the influence of environment on the fatigue crack growth rate even at extremely fast ultrasonic frequencies of ~20,000 cycles per second.
- The relative importance of basal versus prismatic sip in fatigue crack initiation is under on-going debate in the community. It was determined that grains located at the end of FIB notches with low Schmid factor for basal slip and a high Schmid factor for prismatic slip did not initiate visibly detectable cracks (within the resolution of the SEM).

ABSTRACT

Very high cycle fatigue (VHCF), in which components undergo fatigue lifetimes well beyond traditional design limits of 10^7 cycles, is not well understood and is becoming an increasingly prevalent deformation state in aerospace applications. Components are now designed to handle increasingly long lifetimes (>10^9 cycles), and it is critically important to be able to accurately predict when these components will fail and to intelligently tailor them for improved performance. Toward this end, a new methodology was developed for the small-scale investigation of fatigue crack initiation and growth during VHCF loading, and was used to investigate environmental and microstructural effects on the fatigue lifetimes of the polycrystalline titanium alloy Ti-6242S. Small fatigue crack growth in Ti-6242S, a commonly utilized alloy in aerospace applications, was examined in vacuum and in controlled partial pressures of water vapor, high purity oxygen, and high purity hydrogen.

Microstructural heterogeneity dominates the failure process in the VHCF regime. In order to understand what drives damage accumulation and failure in materials of interest to the Air Force under VHCF, there is a need to have small-scale experimental data linking fatigue crack nucleation and growth back to the microstructure. The research funded by this award addressed this gap in experimental data and resulted in in-situ, in-SEM comparisons between small fatigue crack growth behavior and the underlying microstructure under varied environmental conditions. A pronounced increase in the fatigue crack growth rate with increasing partial pressure of H_2O vapor was found, and high purity oxygen was found to be more detrimental than high purity hydrogen. In vacuum, no significant crack propagation was found at the same stress amplitude used in the H_2O vapor tests, in which stable crack growth was observed. Cracks frequently decelerated or arrested at high angle α/α and α/α+β grain boundaries, and demonstrated a pronounced sensitivity to microstructure. Micro-notch tips that were machined by focused ion beam in regions unfavorably oriented for basal slip tended to initiate cracks later, or in some cases not at all.

PROJECT DETAIL

In this research, fatigue crack formation and growth in the near alpha titanium alloy Ti-6242S was examined under very high cycle fatigue (VHCF) loading. To accomplish these investigations, a custom experimental setup was designed and built in order to employ *in situ* ultrasonic fatigue at a cyclic frequency of 20 kHz inside an environmental scanning electron microscope (ESEM)*. The role of environment on small fatigue crack initiation and growth was investigated in vacuum and in variable pressures of saturated water vapor, as well as in laboratory air. The research funded by this award resulted in the first quantitative, full-field, in-SEM comparisons between small fatigue crack growth behavior and the underlying microstructure under varied environmental conditions. A pronounced increase in the fatigue crack growth rate with increasing partial pressure of H_2O vapor was found, and oxygen was shown to be the significantly more detrimental species. In vacuum, no significant crack propagation was found at the same stress amplitude used in the H_2O vapor tests, in which stable crack growth was observed. Cracks frequently decelerated or arrested at high angle α/α and $\alpha/\alpha+\beta$ grain boundaries, and demonstrated a pronounced sensitivity to microstructure. Micro-notch tips that were machined by focused ion beam in regions unfavorably oriented for basal slip tended to initiate cracks later, or in some cases not at all. In addition to the scientific findings described below, this research also successfully demonstrated the usefulness of *in situ* ultrasonic fatigue instrumentation (termed here as "UF-SEM") as a new tool for the characterization of environmental and microstructural influences on VHCF behavior.

There is a growing need to extend the service life of systems and components well beyond traditional fatigue design limits of 10^7 cycles, into what is known as the very high cycle fatigue (VHCF) regime. Researchers have conventionally assumed the existence of a fatigue limit, or threshold stress amplitude below which fatigue life is infinite [1]. This assumption is historically linked to fatigue studies of ferrous metals in the high cycle fatigue (HCF) regime, a fatigue life range of 10^6 to 10^7 cycles [2]. However, recent studies conducted at 30-100 Hz [3, 4] and at ultrasonic frequencies [5, 6] reveal that this assumption may not be a valid design approach for materials operating in the VHCF regime. Even at low applied stresses (well below the conventional fatigue threshold) and at nominally elastic strains characteristic of VHCF, significant damage accumulation at the microstructural length scale can lead to crack initiation and fatigue failure [7-10]. Furthermore, fatigue life in this regime is dominated by crack initiation and the growth of microstructurally small cracks. Thus, a significant portion of the fatigue life involves micro-scale mechanistic responses to cyclic stresses [11]. The sensitivity of cyclic deformation mechanisms to microstructural influences adds complexity and greater uncertainty to lifetime predictions.

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* This system was built in collaboration with C. Torbet at the University of California Santa Barbara.

Ultrasonic fatigue testing has been used since the early 1950s [12, 13] to provide a powerful and time-effective means for interrogating VHCF of a wide range of materials including cast aluminum alloys [14, 15], nickel-base superalloys [7, 16], titanium alloys [10], and high strength steels [17, 18]. Over the past forty years, the technique has been extended to enable VHCF studies under various environmental conditions [7, 19], in crack growth studies [20-22], and in conjunction with other techniques such as synchrotron x-ray imaging [16]. However, the data acquired from ultrasonic fatigue testing largely remains limited to determination of total fatigue life, crack growth rates, and deformation processes that are inferred from fractography and surface microscopy. Observations regarding crack initiation and ultimate failure are linked to microstructure in a before-and-after methodology through grain mapping techniques like electron backscatter diffraction, with limited *in situ* observations. Efforts have been made to track the evolution of deformation and formation of early fatigue damage, such as slip bands, by using methods that probe the damage micro-mechanisms taking place as a function of the number of cycles. For example, replication is a common technique used to obtain surface fatigue damage history as a function of applied cycles [21]. Stanzl-Tschegg et al. [23] used a combination of high resolution SEM and atomic force microscopy (AFM) to investigate fatigue damage in copper polycrystals in the VHCF regime with cycling. This technique was *ex situ* and required a significant amount of time, but added useful information to the current understanding of the progression of fatigue damage.

Ultrasonic Fatigue SEM (UF-SEM) System

High spatial resolution (≈ 5 nm) imaging of fatigue damage at the microstructural length scale was accomplished using a custom combination of ultrasonic fatigue and scanning electron microscopy, termed UF-SEM and shown in Fig. 1. This system combines ultrasonic fatigue testing instrumentation with a Philips XL30 ESEM. The ultrasonic fatigue instrumentation operates using the principles described in [12] and summarized here. The load line of the system is comprised of Ti-6Al-4V components tuned to a 20 kHz resonant frequency. The components include an ultrasonic converter that imparts a controlled sinusoidal displacement using a piezoelectric material stack, an amplification horn that magnifies the displacement from the ultrasonic converter, a lambda rod, and the fatigue test specimen. The system, which is mounted to a custom built SEM chamber door, is controlled by instrumentation that accurately maintains the resonant frequency within ± 1 Hz in displacement control by monitoring the input displacement to the specimen using a piezoelectric transducer in a closed-loop control system.

Integrating the ultrasonic fatigue instrumentation into an ESEM provides the capability to perform fatigue studies under environmental conditions ranging from vacuum (3.7×10^{-4} Pa) to low partial pressures (133 Pa to 2660 Pa) of selected gases. A gaseous secondary electron (GSE) detector was used for electron imaging in low vacuum and gaseous environments. The fatigue specimen is positioned in the desired imaging orientation using a McAllister Technical Services MB1500 manual manipulator stage with five translational adjustments (including insertion). The numerous degrees of freedom in the manual manipulator stage permitted the observation and tracking of multiple cracks or microstructural features such as large grains, grain clusters or regions of microtexture. Furthermore, rotation of the assembly about the longitudinal axis of the specimen enabled *in situ* EBSD mapping for crystallographic characterization.

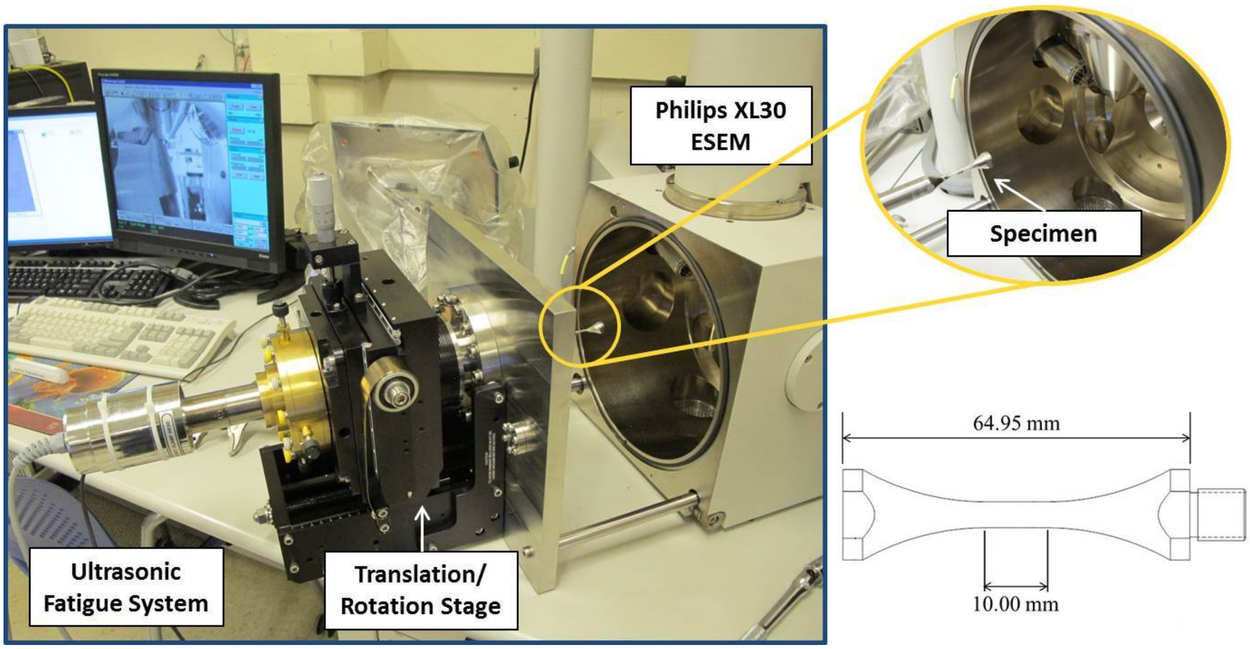


Fig. 1. Ultrasonic fatigue scanning electron microscope (UF-SEM) system combining ultrasonic fatigue at 20 kHz with the high resolution imaging capabilities of a SEM. This system was built in collaboration with C. Torbet, UCSB.

Material and Methods

Material

Ti-6242S, a polycrystalline, near-alpha titanium alloy was provided in the form of a forged disc. The alloy was processed to produce a bimodal microstructure and had a nominal composition of wt.%. 6Al, 2Sn, 4Zr, 2Mo, 0.1Si, and Ti (balance). The microstructure consisted of primary α grains in a transformed β matrix, as shown in Fig. 2. The average primary α grain size measured by the linear intercept method was 12.5 μm ± 5.5 μm. The area fraction of the primary α phase was approximately 30% ± 3%. The measured Young's modulus and yield stress were 121 GPa and 926 MPa, respectively.

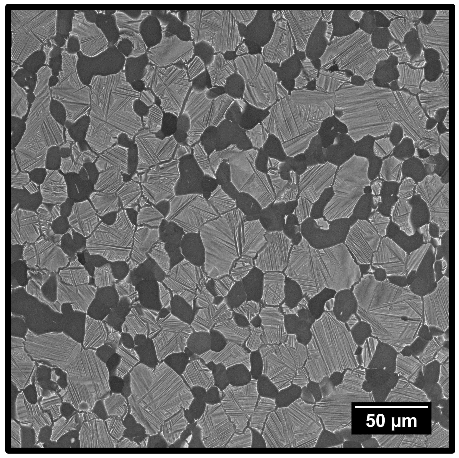


Fig. 2. Back-scattered electron (BSE) micrograph of Ti-6242S microstructure showing primary α grains in a transformed β matrix.

Specimen Preparation

Fatigue test specimens were machined from slices extracted in the circumferential orientation from a forged disc[†]. Cylindrical blanks with a 4 mm diameter and 10 mm long gauge section were cut from the source material. Ti-6Al-4V rod was inertia welded to the specimen ends for shoulder and grip regions. Diametrically opposed surface flats extending from the specimen shoulder regions were machined into the gauge section to facilitate fatigue crack growth observations and microstructural mapping using electron backscatter diffraction (EBSD) techniques. Final machining included low-stress grinding to minimize compressive residual stresses and was completed by Metcut Research Inc. To minimize surface compressive residual stresses, fatigue specimens were electropolished in a solution of 590 ml methanol, 350 ml butyl cellosolve, and 60 ml perchloric acid at -40°C for 90 minutes. Approximately 100 μm was removed from the surface by electropolishing.

To investigate small crack growth behavior, micro-notches were machined in the specimen flats using a FEI Nova Nanolab focused ion beam (FIB) SEM equipped with a gallium ion source operating at 30 kV and a probe current of 3.0 nA. FIB machining processes induce damage by gallium ion implantation that can alter the local mechanical properties. However, previous studies reported the penetration of 30 kV gallium ions to be much less than 1 μm [24]. Furthermore, a reduced probe current of 3.0 nA was used to minimize the depth of gallium ion penetration to less than approximately 200 nm. Three 30 μm long and approximately 15 μm deep FIB micro-notches were machined in the center region of the gauge section of each specimen, with a spacing of 1 mm between notches, as shown schematically in Fig. 3. FIB-

† We gratefully acknowledge our Air Force Research Laboratory collaborators, especially Dr. James M. Larsen, Dr. Sushant Jha, and Dr.Christopher Szczepanski (currently at Special Metals Corp.) for providing material and support.

deposited Pt markers were placed in a 200 μm by 100 μm rectangle centered about each FIB micro-notch to enable alignment of the EBSD and fatigue test field of view (FOV). Local microstructural information was determined in these neighborhoods by EBSD.

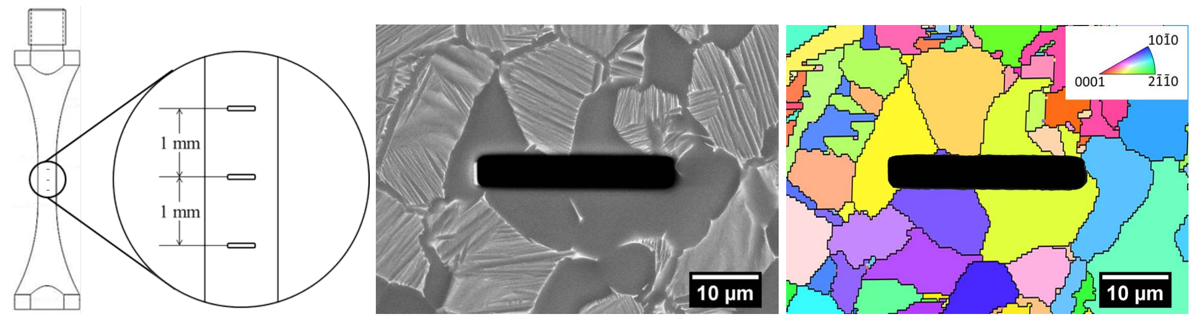


Fig. 3. Schematic of ultrasonic fatigue specimen with surface flats and three FIB micro-notches placed in the center gage section. A BSE image of a FIB micro-notch is shown with the corresponding inverse pole figure (IPF) map of the surrounding microstructure.

Ultrasonic Fatigue Testing

Ultrasonic axial fatigue testing was performed in lab air and in the ESEM at a vacuum level of 3.7 x 10^{-4} Pa. The use of ultrasonic fatigue allowed the attainment of fatigue lifetimes of >10^7 cycles in a matter of hours, rather than days as required by conventional methods. Fatigue testing in laboratory air was accomplished using the ultrasonic fatigue instrument described in [16] and shown in Fig. 4. For testing in laboratory air, observations of fatigue crack growth were made using a Navitar 12X Ultrazoom lens system equipped with a 20X Mitutoyo infinity corrected objective and a 5-megapixel CCD (Point Grey GRAS-50S5C). The custom-built UF-SEM system was used for all in-SEM tests. All tests were carried out at a stress ratio of R = -1 (fully reversed) and a testing frequency of approximately 20 kHz. For all tests, a constant displacement amplitude was maintained to produce a stress amplitude of 400 MPa.

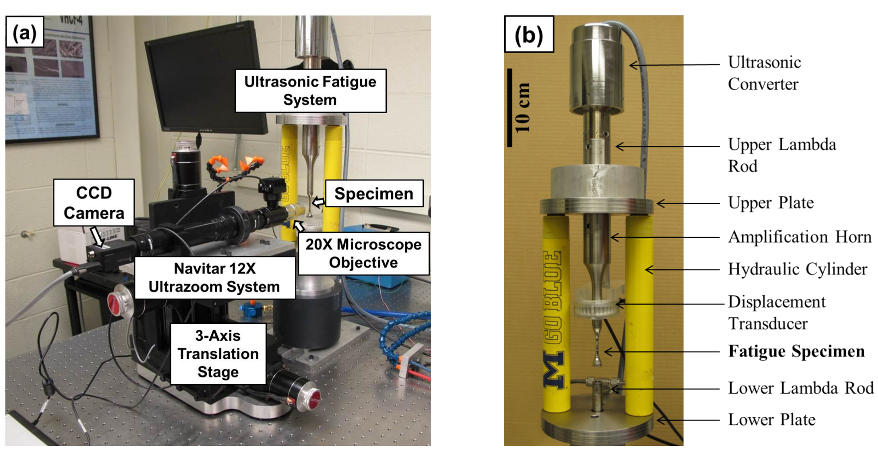


Fig. 4. (a) Laboratory air setup including Navitar 12X Ultrazoom optical system and ultrasonic testing system and (b) a magnified view of the ultrasonic testing system.

Intermittent cycling (200 milliseconds pulse and 3 second pause) was used in both laboratory air and in-SEM tests to minimize the temperature increase associated with high frequency loading. In vacuum, the specimen temperature was maintained to within 10°C of room temperature as determined by thermal imaging (model FLIR SC5000) and K-type thermocouples. In laboratory air tests, specimen temperature was maintained to within 2°C of room temperature as determined by IR imaging. Additionally, forced air cooling was used for tests in laboratory air. No auxiliary cooling methods were used in the in-SEM fatigue experiments.

In-ESEM VHCF fatigue experiments were performed in the following vapor environments to investigate the effect of environment on small crack growth:

- H_2O: 1330 Pa, 665 Pa, 133 Pa, and 65 Pa
- High Purity 0_2: 133 Pa
- High Purity H_2: 133 Pa
- Vacuum
- Lab Air

Cycling was paused every 10,000 to 25,000 cycles, depending on the fatigue crack growth rate (FCGR), to observe damage and capture micrographs for the subsequent determination of crack growth rates. The stress intensity factor range was calculated using the equations of Newmann and Raju [25] for a surface crack in a finite elastic plate, where *c/a* was assumed to be unity. The fatigue crack growth rate, *dc/dN*, was calculated using the secant method. Higher resolution micrographs, described later in this report, were obtained *ex situ* using a Tescan Mira-3 SEM.

Results & Discussion

Environmental Effects on Small Crack Growth Behavior

The effects of the environments listed above are shown in Figure 5. For clarity, fatigue crack growth data from non-fatal cracks was omitted from the plots in Fig. 5. However, the data from non-fatal cracks is in agreement with the reported data for the fatal cracks in each test case. Higher H_2O vapor pressures resulted in significantly increased fatigue crack growth rates and reduced fatigue lifetimes. An order of magnitude increase in fatigue life was observed for tests carried out in significantly lower (133 Pa and 65 Pa) H_2O vapor over those carried out in laboratory air. The crack growth rates for 133 Pa H_2O were moderately higher than 65 Pa H_2O. As seen in Fig. 5, fatigue lifetimes were markedly shorter and crack propagation rates were much higher in laboratory air tests than for in-SEM tests. As the H_2O vapor pressure approached 1330 Pa, the crack length (*c*) vs. cycle number (*N*) curve approached that of lab air, until the two curves overlaid each other.

The impact of high purity hydrogen (H_2) and high purity oxygen (O_2) was examined and it was found that high purity oxygen was significantly more detrimental for VHCF fatigue crack growth. The role of hydrogen versus oxygen has been under debate in the very high cycle fatigue community. The mechanisms underlying the independent roles of these species and their interactions are now under active investigation.

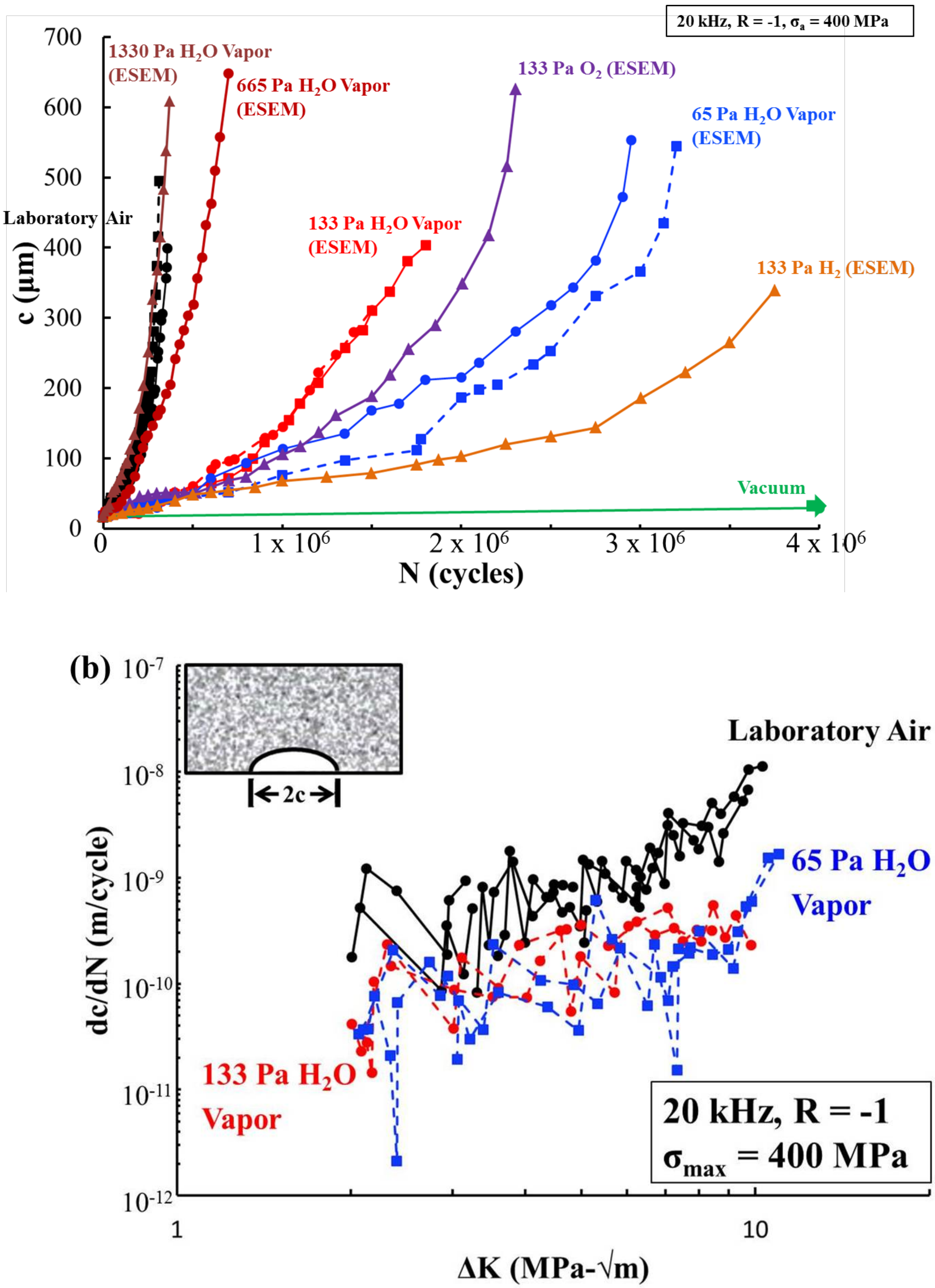


Fig. 5. Fatigue crack growth in laboratory air, high purity hydrogen, high purity oxygen, and water vapor environments. (top) Crack length (*c*) vs. cycle number (*N*) is shown. Fatigue

lifetimes were substantially lower in air than in the vapor H_2O vapor environments, with lifetimes uniformly decreasing as vapor pressure increased. Oxygen was the more deleterious species as compared with hydrogen. (bottom) The fatigue crack growth rate (*dc/dN*) vs. ΔK for three H_2O vapor environments showing a pronounced increase in FCGR for laboratory air versus H_2O vapor environments. Significant dips in the fatigue crack growth rate dc/dN were also found to be correlated to microstructural barriers.

Fatigue crack growth under a 3.7 x 10^{-4} Pa environment (SEM chamber vacuum) was also examined as shown in Figure 5. Small fatigue crack growth rates were lowest in the vacuum environment (3.7 x 10^{-4} Pa). In all vacuum experiments, the crack initiated well after 10^6 cycles and propagated for a small distance before crack arrest. After the crack had arrested for a minimum of 10^7 cycles, the stress amplitude was increased in 10% increments until crack growth resumed. The results of a vacuum experiment shown in Fig. 5(a) are from a test in which an approximately 12 μm long crack was observed at 4 x 10^6 cycles, where σ_{max} = 400 MPa. Further cycling of the specimen to 10^7 cycles resulted in a small increase in crack advance (< 2 μm). Growing fatigue cracks in vacuum using the same stress amplitudes used for environmental tests proved challenging because of their low growth rate. Due to time and cost restraints, in the present work vacuum tests were either step tested to much higher stress amplitudes to cause fatigue failure (up to 700 MPa), or water vapor was introduced to accelerate crack growth.

The work performed under this award significantly contributes to our understanding of the roles of oxygen, hydrogen, and water vapor in fatigue crack propagation and their interactions. This has been under debate in the community – for example, from elevated temperature (550 °C) studies on a Ti-6242 alloy, Sarrazin-Baudoux et al. [26] reasoned that water vapor is the more detrimental species and oxygen plays a secondary role. They came to this conclusion from demonstrations that humidified argon substantially increased fatigue crack propagation rates compared to that observed in humidified argon with added oxygen. They argued that oxygen limits the effects of water vapor on the oxide layer formation that results primarily from water vapor dissociation, and proposed that the formation of such an oxide layer on fresh crack faces is responsible for the observed increase in fatigue crack growth rates. However, Bache et al. [27] investigated the role of internal oxygen content on fatigue crack propagation in a Ti-6Al-4V alloy in low vacuum (13.3 Pa) at room temperature and concluded that oxygen was responsible for the increase in growth rate, partially through enhanced facet formation. Fatigue crack growth rates were also observed to be slightly lower in pure hydrogen gas than laboratory air. They proposed that hydrogen serves to shield the crack tip from harmful species such as water vapor and oxygen and, because the gas was nominally dry (< 3ppm water), oxygen must play a critical role. It is possible that the relative influence of water vapor and oxygen are tied to temperature, which is a topic of interest for future work.

In studies aimed at understanding intrinsic crack growth mechanisms (in the absence of environmental effects) in titanium alloys by vacuum testing, FCGRs in the near threshold regime were lower in vacuum than in air [27-31]. The present study also shoes lower FCGRs in vacuum, even though tests were conducted at 20 kHz instead of the much lower frequencies associated with conventional fatigue testing. The decrease of FCGRs in vacuum could be due to local heating at the crack tip. Sugano et al. [31] reported a sharp increase in fatigue lifetimes (of nominally 500,000 cycles) for a pressure reduction from 10 Pa to 1 Pa in pure Ti up to a testing frequency of 1 kHz. They attributed this increase to gas absorption processes and internal frictional heating of the specimen in vacuum that led to increased plasticity at the crack-tip. A model was postulated for which heating of active slip planes at the crack-tip, on the order of 200 °C, caused a decrease in fatigue crack growth rates through crack-tip blunting and the development of compressive residual stresses around the plastic zone of the crack-tip. In the present study, more work is needed to determine the role of temperature increase on the decrease in FCGR in vacuum and low pressure water vapor at ultrasonic fatigue frequencies, if any. The overall specimen temperature was maintained to within 10 °C of the ambient temperature using a pulse/pause duty cycle, so temperature is not believed to have a significant effect on the crack growth behavior observed in this study.

Crack Initiation

Environment significantly influenced fatigue crack initiation lifetime, N_i, from FIB micro-notches, with the shortest initiation lifetimes in laboratory air and longest in vacuum. Here, crack initiation was defined as the formation of a discontinuity that extended from the micro-notch to a minimum length of 50 nm. As shown in Table 1, N_i ranged from 7 x 10^3 to 3 x 10^4 cycles for laboratory air fatigue tests, while no cracks initiated prior to 10^6 cycles in high vacuum tests at the same stress amplitude (400 MPa). N_i for cracks grown in low vacuum saturated water vapor experiments fell between these values, with a range of 2.6 x 10^4 to 3 x 10^5 cycles to initiate an observable crack.

Table 1. Fatigue crack initiation lifetime[a] ranges from FIB micro-notches for each test environment.

Environment	Cycles to observed crack initiation
Laboratory air	7,000 – 30,000
133 Pa H_2O vapor	26,000 – 300,000
65 Pa H_2O vapor	40,000 – 105,000
3.7 x 10^{-4} (vacuum)	>10^6

[a]Crack initiation was defined as the formation of a crack at least 50 nm long.

Crack initiation behavior was influenced by the microstructural neighborhoods at the micro-notch tips. Although the number of tests was limited, it was apparent that the ease of basal slip in the local microstructure is critical to fatigue crack initiation. Specifically, when the notch tips ended in primary α grains favorably oriented for basal slip, early crack initiation was observed.

Cracks tended to initiate later or decelerate in grains that were not favorably oriented for basal slip.

In several cases and independent of environment, crack initiation did not occur in some micro-notch tip neighborhoods before the specimen failed from a different micro-notch. The common characteristic of the "non-initiating" micro-notch tips was their location in primary α grains with orientations such that basal planes were nearly perpendicular to the nominal crack growth direction, and which therefore exhibited low basal Schmid factors for the prescribed specimen loading direction. Table 2 summarizes the microstructural characteristics of grains located at micro-notch ends in fatigued specimens in laboratory air, 65 Pa H_2O vapor, and 133 Pa H_20 vapor environments, for which a surface fatigue crack did not initiate before fatigue fracture occurred from another micro-notch on the sample at 3.9×10^5 cycles, 2.9×10^6 cycles, and 3.5×10^6 cycles, respectively. The basal Schmid factors ranged from 0.14 to 0.20 and the inclination of the basal plane with respect to the loading axis (φ) was 13° to 17°. These grains also had high prismatic Schmid factors (0.47-0.49).

Table 2. Microstructural characteristics of grains located at micro-notch ends where no cracks initiated.

Environment	Basal Schmid factor	Prismatic Schmid factor	φ (°)
Laboratory air	0.15	0.47	17
133 Pa H_2O vapor	0.14	0.49	13
65 Pa H_2O vapor	0.20	0.48	15

This research demonstrated that environment as well as neighborhood characteristics plays a significant role in small crack initiation behavior in titanium alloys, even at the high frequencies and low loads associated with ultrasonic fatigue. The effect of basal plane orientation on fatigue crack initiation has been studied previously, but those studies mainly focused on the role of microstructure on small crack growth rather than environmental effects. Bache et al. [33] examined short crack growth behavior in a near α titanium alloy and also concluded that the orientation of the basal plane in which a fatigue crack initiates plays a significant role in determining fatigue life scatter. Szczepanski et al. [34] observed a moderate effect of the microstructure adjacent to micro-notches on fatigue crack initiation lifetimes at 20 kHz in a microtextured α + β titanium alloy. Specifically, neighborhoods that were favorably oriented for basal and prismatic slip tended to promote early fatigue crack initiation.

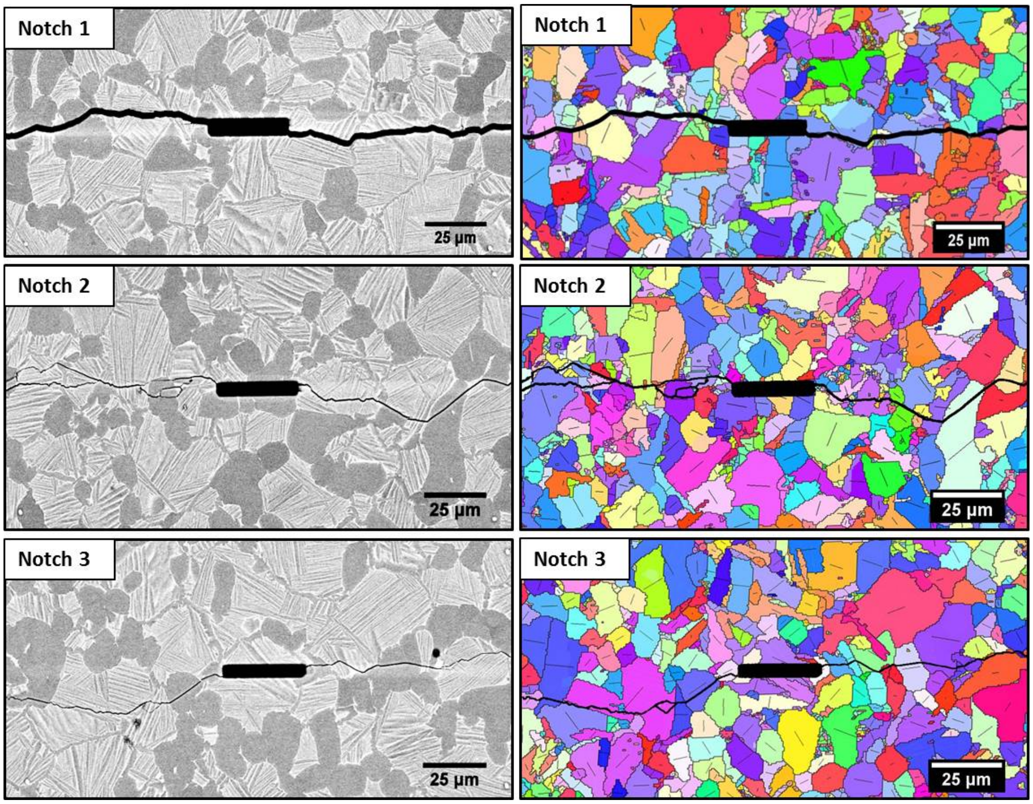


Fig. 6. Small fatigue cracks propagated in a 133 Pa saturated H_2O vapor environment from three FIB micro-notches machined into the same test specimen. The specimen failed at a fatal crack that was initiated and grown from Notch 1. Transgranular, crystallographic crack growth was observed in each case.

Microstructural Effects on Fatigue Crack Growth

Cracks propagated transgranularly along specific crystallographic directions in the small crack region and up to crack lengths of approximately 1 mm. In primary α grains, cracks tended to propagate along basal planes in the early stages of growth. An example of this is shown in Fig. 6 for an in-ESEM test in a 133 Pa saturated water vapor environment. Here, an overlay of the grain orientation maps from EBSD with crack paths is provided to demonstrate that cracks propagated along basal planes in primary α grains. Another example of early propagation along basal planes is shown in Fig. 7 for a test in laboratory air, where the crack propagated along basal planes in two primary α grains before arresting at a high angle α/α grain boundary.

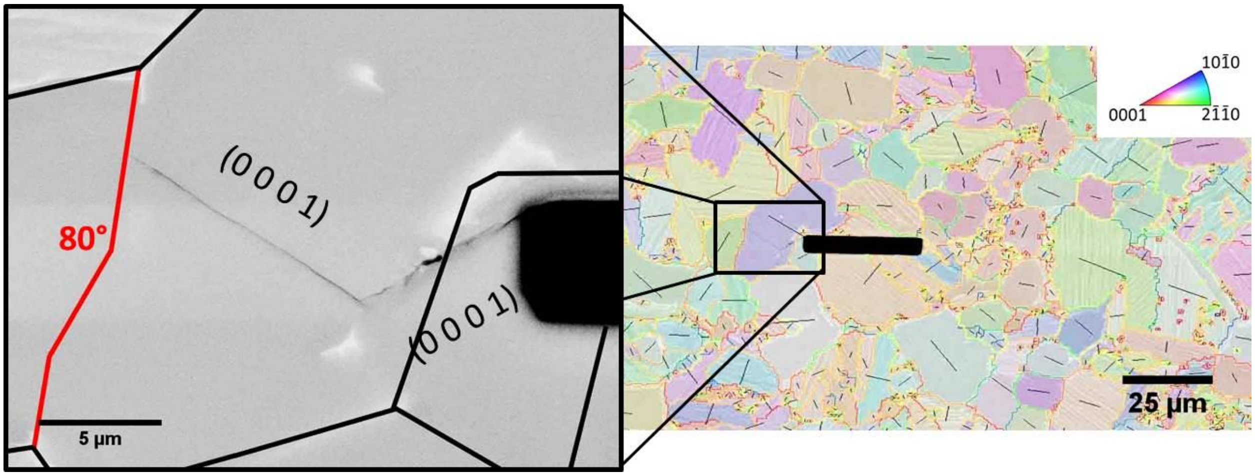


Fig. 7. A small fatigue crack was initiated and grown in laboratory air. The right image shows the local microstructure surrounding the notch with the IPF map overlaid, where black lines denote basal plane traces. The SEM micrograph on the left shows the fatigue crack propagated along basal planes and arrested at a high angle α/α grain boundary after 3.0 x 10^4 cycles.

Significant local variability in the small fatigue crack growth rate was observed in each environment, and was frequently correlated with microstructural features such as high misorientation angle grain boundaries and phase boundaries. Fig. 7 shows a fatigue crack that propagated in laboratory air along basal planes in two neighboring primary α grains for 3.0 x 10^4 cycles before arresting for the duration of the test (3.1 x 10^5 cycles) at an α/α grain boundary with a misorientation of approximately 80°. Fig. 8 shows a fatigue crack grown in 133 Pa H_2O vapor that arrested for approximately 10^5 cycles at an α/α+β phase boundary. The misorientation angle between these two grains was low and likely not responsible for the reduction in crack growth rate observed at this boundary. Rather, this reduction could be an effect of the crack crossing from a primary α grain to a transformed β phase region, as lamellar regions have a higher fatigue crack growth resistance [35]. Crack arrest at the grain boundary may also have been affected by the low basal Schmid factor of the transformed β region, as grains that are unfavorably oriented for basal slip have been observed to impede crack growth in titanium alloys [34].

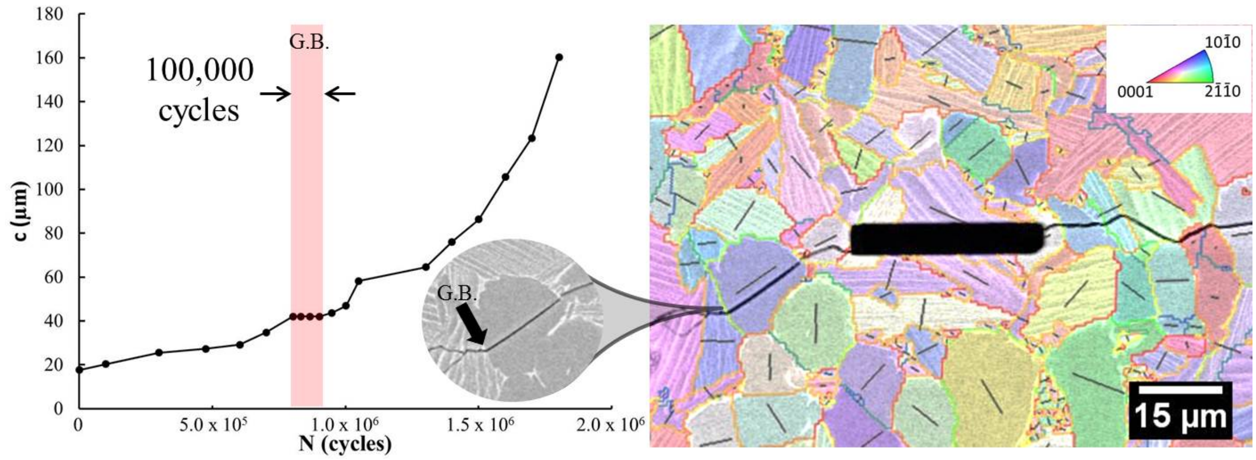
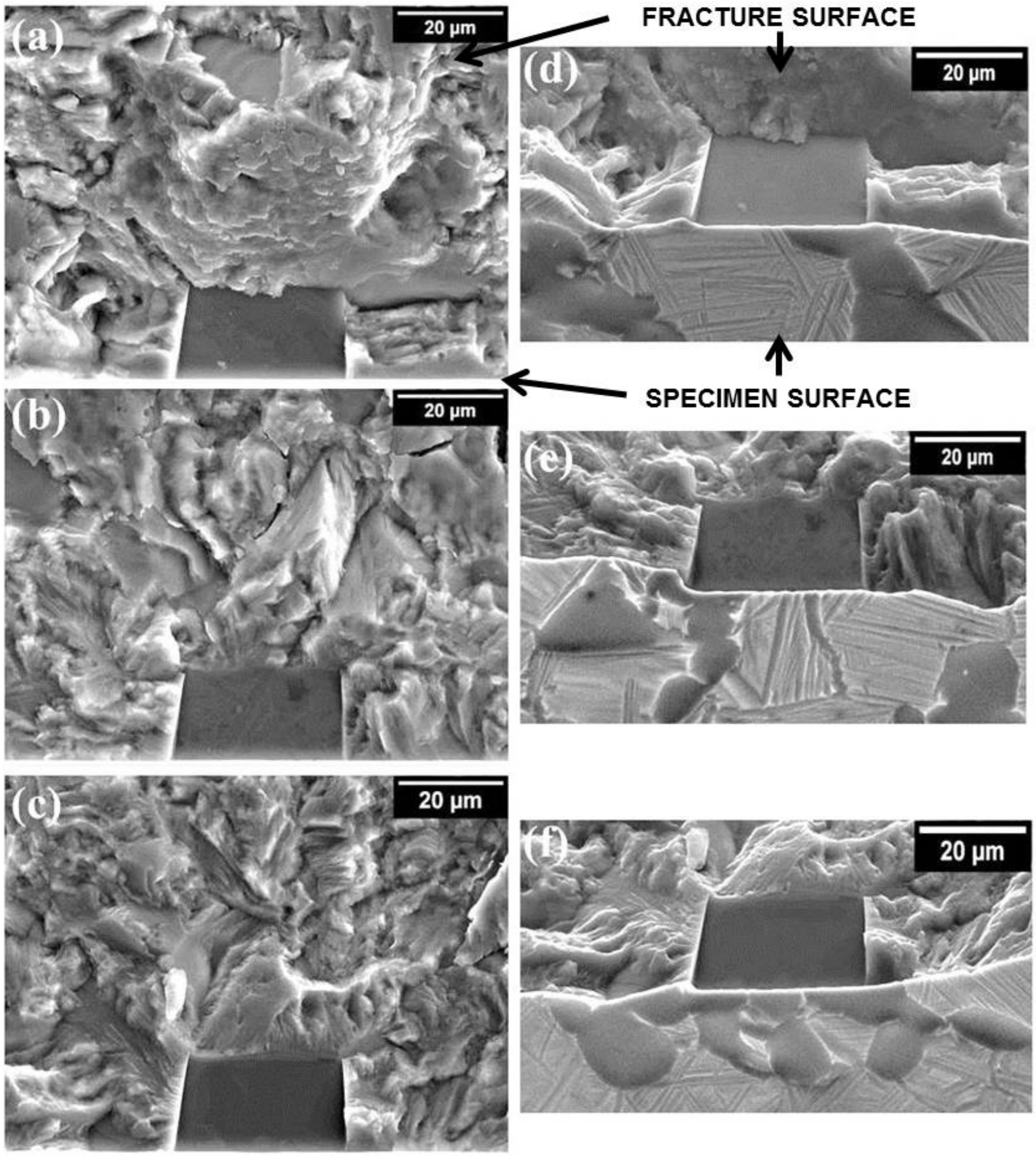


Fig. 8. A small fatigue crack was initiated and grown in 133 Pa H_2O vapor. A micrograph of the local microstructure surrounding the notch and the propagated crack with the IPF map overlaid is shown. The black lines denote basal plane traces. The left side fatigue crack propagated along basal planes in α grains and arrested at the α/α+β phase boundary indicated by the arrow for approximately 10^5 cycles.

Fractography

In both the 133 Pa H_2O vapor and 65 Pa H_2O vapor environments, fatigue cracks propagated transgranularly and were microstructurally sensitive in the low ΔK regime. Observed fracture surface features for the two low vacuum water vapor environments were similar, consistent with the moderate difference in fatigue crack growth rate. Example fractographs obtained in the early crack growth region for each environment are shown in Fig. 9. Macroscopically smooth faceted crack growth across primary α grains was observed for ΔK < 7.0 MPa√m, as shown in Fig. 10. Correlation of primary α grain facets with the adjacent surface EBSD maps showed that faceting predominantly occurred along basal planes. Higher magnification imaging of faceted surfaces revealed crack growth markings, or bands, indicative of a slowly advancing crack, rather than a cleavage-like fracture mechanism. The frequency of primary α facets decreased with increasing crack length. This is attributed to an increase in the crack-tip stress intensity as the crack gets longer and thus, an increase in plastic deformation at the crack-tip.

9.  Fig.

Fracture surfaces of fatal cracks for (a) laboratory air, (b) 133 Pa H_2O vapor, and (c) 65 Pa H_2O vapor. The images on the left show the specimen fracture surface as viewed along the loading direction. The images on the right show the corresponding fracture surfaces on the left with a BSE image of the adjacent surface microstructure as viewed by a 45° tilt with respect to the loading direction.. Facetted fracturing is observed in each of the test environments in the early stage crack growth region.

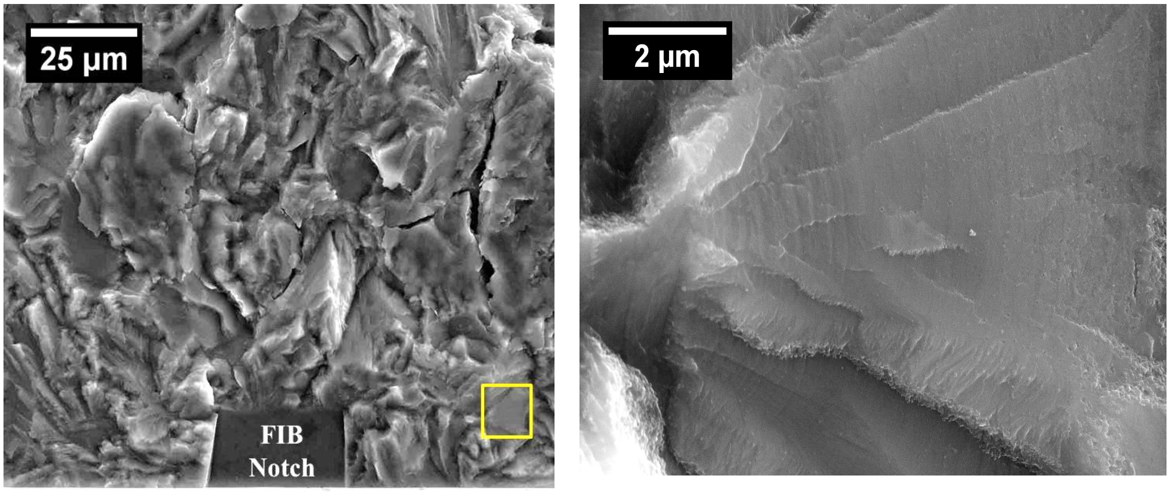


Fig. 10. SEM micrograph of the fracture surface of a fatal crack propagated in the 133 Pa saturated water vapor environment. The right image is a high magnification of the area in the yellow box in the left image, and shows a macroscopically smooth primary α facet with distinct crack growth features indicating that the facet was created by a slowly advancing crack ($dc/dN \approx 1.8 \times 10^{-10}$ m/cycle) rather than a cleavage mechanism. The crack propagation direction is from bottom to top.

Crystallographic, microstructurally sensitive fatigue crack growth was also observed in laboratory air. The fracture surfaces of fatal cracks propagated in laboratory air showed primary α grain faceting during early crack growth. EBSD maps of fractured primary α grains that intersected the specimen surface indicated that low ΔK crack advance took place mainly along basal planes, but likely took fewer numbers of cycles for facet formation. Higher magnification micrographs of macroscopically smooth faceted surfaces showed little to no striation-like markings (Fig. 11), in contrast with the features shown on a faceted primary α grain in Fig. 10. This was in agreement with the observed increase in fatigue crack growth rate in laboratory air versus vacuum environments, where a faster fracture in laboratory air produced fewer fatigue markings on faceted planes. Some facets with coarse band-like features were also observed in the laboratory air experiments, but not in the saturated water vapor and vacuum environments at equivalent crack lengths.

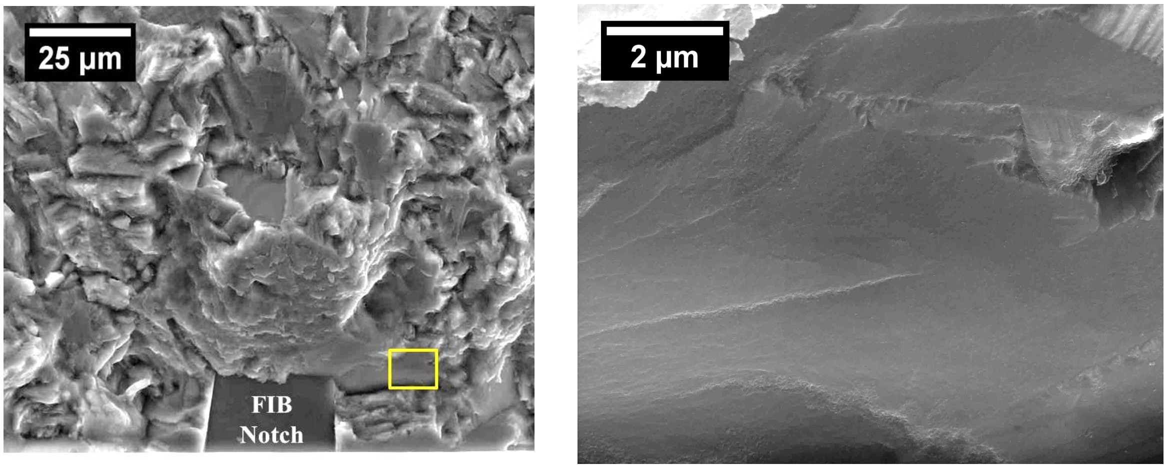


Fig. 11. SEM micrographs of the fracture surface of a fatal crack propagated in laboratory air. At right is a high magnification micrograph of the area in the yellow box in the left image, and shows a macroscopically smooth primary α facet with no striation-like features. The crack propagation direction is from bottom to top ($dc/dN \approx 6.2 \times 10^{-10}$ m/cycle).

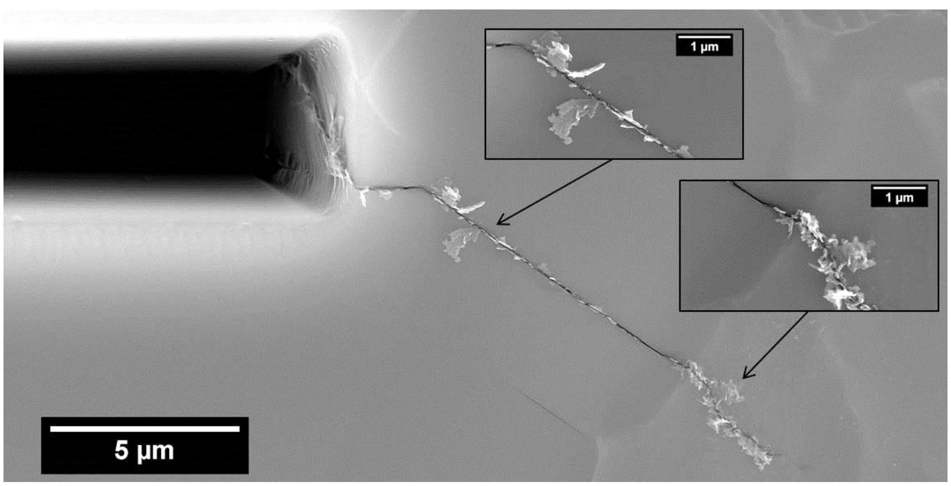


Fig. 12. SEM micrograph of extruded material from a surface crack of a specimen fatigued in vacuum (3.7×10^{-4} Pa). The extruded material transitions from a thin feather-like structure in the primary alpha grain to a globular extrusion upon entering the adjacent lamellar region.

Interestingly, a thin material appears to have been ejected from the crack faces and is still adhered to the specimen surface, as shown in Fig. 12. This phenomenon was observed in numerous tests and depended on microstructure and environment. The test shown in Fig. 12 was conducted in vacuum, and exhibits a transition from a feather-like extrusion in a primary α grain to a more globular morphology in the adjacent transformed β region. The frequency and intensity of the observed extruded material was greater in fatigue tests conducted in vacuum and partial pressures of water vapor than laboratory air. However, note that the specimens tested in laboratory air were subjected to high velocity air jets, which may have removed extruded material. Sugano et al. [31] observed a similar feature on the specimen surface of a titanium alloy in vacuum fatigue tests at a pressure of 6.7 x 10^{-3} Pa, $R = -1$, and 10^8 cycles. The detected ribbon-like extrusions occurred at slip bands on the specimen surface. They also observed a decrease in intensity and fraction of these extrusions in specimens fatigued in laboratory air versus vacuum. The mechanism for the creation of these features is likely related to crack closure effects and oxide layer formation. More analysis is underway to determine the composition of these formations and the mechanism by which they are created.

Summary

An *in situ* combined ultrasonic fatigue scanning electron microscope system (UF-SEM) for high resolution observations of fatigue damage accumulation and subsequent crack initiation and growth behavior was designed and built. The system was successfully used to examine the microstructural and environmental dependence of crack initiation and propagation in the near alpha titanium alloy Ti-6242S. In-SEM small fatigue crack growth behavior was compared to fatigue tests in laboratory air using a different ultrasonic fatigue instrument that operates from the same principles as the UF-SEM system. Our work resulted in the following findings:

- Fatigue crack growth rates determined by in-SEM tests increased with increasing partial pressures of H_2O vapor. A pronounced increase in fatigue crack growth rate was observed in laboratory air compared to 133 Pa and 65 Pa H_2O vapor environments.
- In vacuum, no significant crack propagation was observed at the same stress amplitude used in laboratory air and H_2O vapor tests, in which stable crack growth was observed.
- The impact of high purity hydrogen (H_2) and high purity oxygen (O_2) was examined, and it was found that high purity oxygen was significantly more detrimental for VHCF fatigue crack growth.
- Fatigue crack initiation lifetime (N_i) was shortest in laboratory air and longest in vacuum.
- Cracks frequently decelerated or arrested at high angle α/α and $\alpha/\alpha+\beta$ grain boundaries and demonstrated sensitivity to microstructure that has typically been observed in small crack growth behavior.
- The local microstructural neighborhood near the micro-notch tips influenced crack initiation life. Micro-notch tips in regions unfavorably oriented for basal slip tended to initiate cracks later or in some cases not at all.
- Primary α grain faceted fracture along basal planes was observed in each environment. Facets from specimens fatigued in vacuum and H_2O vapor environments frequently showed fatigue markings indicative of a low ΔK crack advance mechanism, while no such markings were observed from laboratory air tests.

The results of the present study provide a basis for future studies to probe the mechanisms that underlie small crack initiation and growth behavior in the VHCF regime.

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Graduate Students:	Mr. Jason Geathers (PhD expected June 2016)

Publications and Other Products:

Geathers J, Torbet CJ, Jones JW, Daly S. Examination of Small Fatigue Crack-tip Microstructural Interactions at Very Long Lives Using Ultrasonic Fatigue and Scanning Electron Microscopy. *In progress*.

Geathers J, Torbet CJ, Jones JW, Daly S. Investigating Environmental Effects on Small Fatigue Crack Growth in Ti-6242S Using Combined Ultrasonic Fatigue and Scanning Electron Microscopy. International Journal of Fatigue, Vol. 70: 154-162, 2015. DOI: http://dx.doi.org/10.1016/j.ijfatigue.2014.09.007 **(Top 25 Hottest Articles in IJF for Fall 2014:** http://top25.sciencedirect.com/subject/engineering/12/journal/international-journal-of-fatigue/01421123/archive/57/)

Geathers J, Jones JW, Daly S. Characterization of Microstructural Effects on Small Fatigue Crack Growth Mechanisms in Ti-6242S. Microscale and Microstructural Effects on Mechanical Behavior, 51st Annual Technical Meeting of the Society of Engineering, West Lafayette, IN 2014.

Geathers J, Jones JW, Daly S. Investigating the Role of Microstructure on the VHCF Behavior of Ti-6242S. Micromechanics of Fracture and Fatigue Symposium Proceedings, Society of Experimental Mechanics Annual Conference & Exposition, 2013.

Geathers J, Daly S, Jones JW. A Method for In Situ Capture of Cyclic Strain Accumulation during Ultrasonic Fatigue of Structural Alloys. AIP Conference Proceedings, American Institute of Physics, Ste. 1 No 1 Melville NY 11747, 2012.

Product: A system for in-SEM studies of very high cycle fatigue mechanisms at 20 kHz, designed and built in collaboration with Mr. Chris Torbet at UCSB.

Technical Presentations (J. Geathers):

"Studying Small Fatigue Crack Growth Behavior in Ti-6242S Using Ultrasonic Fatigue and Scanning Electron Microscopy", The Minerals, Metals, and Materials Society Conference, Orlando, FL, 2015 **(INVITED)**

"Investigating Microstructural and Environmental Effects on VHCF Crack Formation in Ti-6242S" Plenary presentation, 6th International Conference on Very High Cycle Fatigue, Chengdu, China , 2014 **(PLENARY, INVITED)**

"Investigating Small Fatigue Crack Growth in Ti-6242S Using In-situ Ultrasonic Fatigue in an ESEM", Air Force Office of Scientific Research Project Review, Kirtland Air Force Base, Albuquerque, NM, 2014

"Examining the Role of Microstructure and Environment on Small Fatigue Crack Growth Behavior in Ti-6242S" , Wright Patterson Air Force Base, Dayton, OH, 2014**(INVITED)**

"Investigating Environmental and Microstructural Effects on Small Fatigue Crack Growth Mechanisms in Ti-6242S", Microscale and Microstructural Effects Symposium, 51st Annual Technical Meeting of the Society of West Lafayette, IN, 2014

"Investigating Microstructural and Environmental Effects on VHCF Crack Formation in Ti-6242S" International Student Paper and Presentation Competition, Society of Experimental Mechanics Conference, Greenville, SC, 2014 **(1st PLACE)**

"Investigating Small Fatigue Crack Growth in Ti-6242S Using In-situ UF-ESEM," A Lifetime of Experience with Titanium Alloys: An SMD Symposium in Honor of Jim Williams, Mike Loretto and Rod Boyer, The Minerals, Metals, and Materials Society Conference, San Diego, CA, 2014 **(INVITED)**

"Experimental Investigations of Full-Field Deformation at the Microstructural Length Scale", Deformation and Transitions at Grain Boundaries III, Materials Science and Technology Conference, Montreal, Quebec, 2013 **(INVITED)**

"Investigating Short Fatigue Crack Growth in Ti-6242S Using In-situ UF-ESEM", Titanium and Titanium Alloys Symposium, Materials Science and Technology Conference, Montreal, Quebec, 2013

"Investigating the Role of Microstructure on the VHCF Behavior of Structural Materials" Micromechanics of Fracture and Fatigue Symposium, Society of Experimental Mechanics Conference, Lombard, IL, 2013

"Combining DIC and Ultrasonic Fatigue to Investigate the VHCF Behavior of Structural Materials" Fatigue in Materials Symposium, The Minerals, Metals, and Materials Society Conference, San Antonio, TX, 2013

"A Method for In Situ Capture of Cyclic Strain Accumulation during Ultrasonic Fatigue" Ultrasonic Fatigue of Advanced Materials and Systems Symposium, The Minerals, Metals, and Materials, Society Conference, Orlando, FL, 2012

Honors and Awards (2012-2015)

- J. Geathers, Overall Best of Show, TMS Student Poster Competition (2015)
- J. Geathers, 1st Place, Structural Materials Division, TMS Student Poster Competition (2015)
- J. Geathers, 1st Place, Society of Engineering Science Student Competition (2014)
- J. Geathers, 1st Place, Society of Experimental Mechanics International Student Competition (2014)
- J. Geathers, 2nd Place, Engineering Graduate Symposium Poster Award, University of Michigan (2013)
- J. Geathers, Scholar Power PhD Candidate Achievement Award (2013)
- S. Daly, James W. Dally Award for Contributions to Education and Research Excellence in Experimental Mechanics, Society of Experimental Mechanics, 2016.

- S. Daly, National Academy of Engineering U.S. Frontiers of Engineering Symposium Attendee, 2015.
- S. Daly, Invited Speaker, Gordon Conference on Physical Metallurgy, 2015.
- S. Daly, Cover, Journal of Materials Science, July 2015.
- S. Daly, Student-Nominated for the Golden Apple Teaching Award, University of Michigan, 2015.
- S. Daly, College of Engineering 1938E Award, University of Michigan, 2014.
- S. Daly, Presentation to University of Michigan Board of Regents (as one of three university-wide example promotions), 2014.
- S. Daly, Lindseth Lecturer, Cornell University, 2014.
- S. Daly, Journal of Strain Analysis Young Investigator Lecturer, 2014.
- S. Daly, Student-Nominated for the Golden Apple Teaching Award, University of Michigan, 2014.
- S. Daly, Mechanical Engineering Department Achievement Award, University of Michigan, 2014.
- S. Daly, Best Paper of the Year, International Journal of Solids and Structures, 2014 (for Best Paper published in IJSS in 2013).
- S. Daly, NSF CAREER Award, 2013.
- S. Daly, Robert Caddell Memorial Materials & Manufacturing Award (with graduate student Adam Kammers), The University of Michigan, 2013.
- S. Daly, Hetényi Award, Society of Experimental Mechanics, 2013.

1.

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A New Approach Towards Characterizing Microstructural Influence on Material Behavior Under Very High Cycle Fatigue

Grant/Contract Number
AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".

FA9550-12-1-0394

Principal Investigator Name
The full name of the principal investigator on the grant or contract.

Samantha Hayes Daly

Program Manager
The AFOSR Program Manager currently assigned to the award

Dr. David Stargel

Reporting Period Start Date

09/01/2012

Reporting Period End Date

08/31/2015

Abstract

Very high cycle fatigue (VHCF), in which components undergo fatigue lifetimes well beyond traditional design limits of 107 cycles, is not well understood and is becoming an increasingly prevalent deformation state in aerospace applications. Components are now designed to handle increasingly long lifetimes (>109 cycles), and it is critically important to be able to accurately predict when these components will fail and to intelligently tailor them for improved performance. Toward this end, a new methodology was developed for the small-scale investigation of fatigue crack initiation and growth during VHCF loading, and was used to investigate environmental and microstructural effects on the fatigue lifetimes of the polycrystalline titanium alloy Ti-6242S. Small fatigue crack growth in Ti-6242S, a commonly utilized alloy in aerospace applications, was examined in vacuum and in controlled partial pressures of water vapor, high purity oxygen, and high purity hydrogen.

Microstructural heterogeneity dominates the failure process in the VHCF regime. In order to understand what drives damage accumulation and failure in materials of interest to the Air Force under VHCF, there is a need to have small-scale experimental data linking fatigue crack nucleation and growth back to the microstructure. The research funded by this award addressed this gap in experimental data and resulted in

in-situ, in-SEM comparisons between small fatigue crack growth behavior and the underlying microstructure under varied environmental conditions. A pronounced increase in the fatigue crack growth rate with increasing partial pressure of H_2O vapor was found, and high purity oxygen was found to be more detrimental than high purity hydrogen. In vacuum, no significant crack propagation was found at the same stress amplitude used in the H_2O vapor tests, in which stable crack growth was observed. Cracks frequently decelerated or arrested at high angle α/α and $\alpha/\alpha+\beta$ grain boundaries, and demonstrated a pronounced sensitivity to microstructure. Micro-notch tips that were machined by focused ion beam in regions unfavorably oriented for basal slip tended to initiate cracks later, or in some cases not at all.

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Geathers J, Torbet CJ, Jones JW, Daly S. Investigating Environmental Effects on Small Fatigue Crack Growth in Ti-6242S Using Combined Ultrasonic Fatigue and Scanning Electron Microscopy. International Journal of Fatigue, Vol. 70: 154-162, 2015. DOI: http://dx.doi.org/10.1016/j.ijfatigue.2014.09.007 (Top 25 Hottest Articles in IJF for Fall 2014: http://top25.sciencedirect.com/subject/engineering/12/journal/international-journal-of-fatigue/01421123/archive/57/)

Product: A system for in-SEM studies of very high cycle fatigue mechanisms at 20 kHz, designed and built in collaboration with Mr. Chris Torbet at UCSB.

Changes in research objectives (if any):

None.

Change in AFOSR Program Manager, if any:

None.

Extensions granted or milestones slipped, if any:

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AFOSR LRIR Number

LRIR Title

Reporting Period

Laboratory Task Manager

Program Officer

Research Objectives

Technical Summary

Funding Summary by Cost Category (by FY, $K)

	Starting FY	FY+1	FY+2
Salary			
Equipment/Facilities			
Supplies			
Total			

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Appendix Documents

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