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Technical note

# Quantifying microstructurally small fatigue crack growth in an aluminum alloy using a silicon-rubber replica method

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ABSTRACT

A variation of the replica method is presented for quantifying microstructurally small fatigue crack growth rates in a rolled aluminum alloy. Repliset®, a two-part silicon-rubber compound, was employed to make surface impressions on notched specimens subjected to interrupted cyclic loading. Employing the high resolution capability of the scanning electron microscope, this replication method characterized fatigue crack growth rates of the 7075-T651 aluminum alloy for cracks as small as 10 μm in length.



## 1. Introduction

Through the years, the study of fatigue in metals has resulted in the generalized and quantifiable stages of fatigue damage in metals: incubation; microstructurally small crack growth, physically small crack growth, and long crack growth [1]. The inability of classical linear-elastic fracture mechanics (LEFM) to describe the difference in experimentally observed crack growth rates between the microstructurally small and long crack growth stages has prompted significant research efforts in this area. Attempts to correlate small-crack to large-crack behavior in terms of LEFM have led to the so-called "small fatigue crack" anomaly. Possible explanations for small crack behavior have been attributed to plasticity effects, plasticity-induced crack-closure transients, roughness induced crack closure, microstructural interaction, violation of LEFM, and mixed mode effects [2].

While generating large crack growth data is a relatively straightforward approach, small crack testing can be more cumbersome and difficult to conduct. Methods employed to quantify small crack growth include in-situ scanning electron microscopy (SEM) [3], replicas [4], marker bands [5], electrical potential [6], and ultrasonic surface waves [7]. Of those listed, in-situ SEM methods are deemed the most state-of-the-art due to the high resolution nature of the microscope. Typically, in in-situ SEM small fatigue crack testing, the experimental approach involves using micromechanical stages and interrupted cyclic load profiles that allow for the searching and measuring of fatigue cracks. However, the major drawback of using in-situ SEM to measure microstructurally small fatigue crack growth is that the location of a naturally occurring and dominant fatigue crack is not readily apparent until the crack is hundreds of μm in length. At this crack length, the opportunity to observe nucleation and the early stages of microstructure influenced crack growth is missed. However, this is not the case with the replication method. In general, the replication method involves making replicas of an area of interest using various fast drying/curing or impression materials. The advantage of this method is that a preserved surface is obtained that can be viewed at a later time when the exact location of the dominant crack is known. Typically, the most common replica method employed involves the use of an acetyl cellulose film [8,9]. This method involves pressing the film onto the specimen after swabbing the area with acetone. After the film has dried, it can be removed and viewed under high magnification using SEM. The advantage of using SEM to image the replicas is that cracks as small as 10–20 μm in length can be observed [8]. However, the use of the acetyl cellulose film has several drawbacks that have been previously documented. One issue is that the acetyl cellulose film is known to shrink by about 10% when it is fully dried, meaning that any crack length measured from these replicas are smaller compared to the actual crack length [9]. A potentially more significant issue is an apparent increase in fatigue life compared to non-replica tests due to the acetone being applied to the notch surface during the replica process. The application of acetone is thought to protect the crack tip from the ambi-

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ent atmosphere laboratory environment [9], thus causing the cracks to behave more like fatigue tests under a vacuum environment. Lastly, the application of the acetyl cellulose film is cumbersome and requires 20–30 min for the film to dry, which can mean obtaining a well defined crack growth rate curve is a significantly labor-intensive task.

Despite the aforementioned problems associated with acetyl cellulose film, the basic idea of surface replication is still a valid approach for measuring surface crack growth. As an alternative to the acetyl cellulose film, a two-part silicon mixture (Repliset®) was recently employed with good success. Newman et al. [10] employed Repliset® for crack detection in a NASA related reliability study. They found that Repliset®, through the use of SEM analysis, could detect cracks less than 25 μm with a resolution of 0.1 μm and was highly accurate when compared to destructive analysis. Recently, Repliset® was used to study multi-axial fatigue crack initiation and growth [11]. However, in this work, the capability of the Repliset® replica method to characterize microstructurally small fatigue cracks was not fully realized. As such, the emphasis of this work is to test the hypothesis that of use of the Repliset® replica method for generating small fatigue crack data is a viable method for quantifying the incubation and microstructurally small crack growth regimes. Additionally, the viability of this particular method is presented through the characterization of the incubation and the growth stages of small fatigue cracks in 7075-T651 aluminum alloy.

## 2. Materials and experiments

The 7075-T651 aluminum alloy is a high strength material used heavily in the aerospace industry. This wrought alloy was examined under SEM and optical microscope in its as-received condition. The grains and particles in this material were found to be pancake shaped and were aligned in the rolling direction of the plate. It is also apparent from microstructural analysis that many inclusions were broken during the manufacturing process and distributed along the rolling direction, creating non-uniform particle stringers. A thorough characterization of the microstructure and mechanical properties are presented elsewhere [12–14].

The specimens employed in this study were single-edge-notch tension specimens. The specimens contained a semi-circular notch radius, $r = 2.41$ mm and were orientated in the longitudinal direction. This radius was selected based on the size of a common fastener-hole size employed in an aircraft component of interest. In order to remove residual machining marks and burs, and to reduce compressive residual stresses, the notch of the specimen was prepared by submerging the specimens in a chemical solution of 94% phosphoric acid and 6% nitric acid at a temperature of 85 °C for 5 min. Prior to testing, the notch of each specimen was rinsed in ethanol and then dusted with particle-free compressed air.

Stress-life fatigue tests were conducted on a servo-hydraulic load frame for a range of applied stress levels and stress ratios. The tests were conducted in load control with frequencies ranging from 5 to 20 Hz, depending on the expected number of cycles to failure, for three stress ratios ($R = P_{min}/P_{max}$): −1, 0, and 0.5. Anti-buckling plates were employed for all $R = -1$ tests. All tests were conducted in ambient laboratory air and humidity and were terminated when full separation of the specimen was achieved. For the application of the replicas, the cyclic tests were stopped at the mean cyclic load. For the $R = 0$ and 0.5 tests, this was a tension load, and for $R = -1$, the mean load was zero. Before the application of each replica, the notch was dusted with particle-free compressed air. A static tip mixing nozzle and dispensing gun, as shown in Fig. 1a, were used to dispense the Repliset® material onto the notch. In order to support the silicon mixture as it dried, tape was applied over the notch of the specimen with one side left open to provide an opening to squeeze the Repliset® through (Fig. 1b and c). Once the notch was completely filled with the Repliset® material, the tape was closed over the opening to prevent the Repliset® from seeping out. After approximately 5–10 min, the tape was carefully pulled away of the specimen, starting from one edge and pulling toward the opposite edge. Due to the adhesive nature of the tape, the Repliset® replica was securely attached to the tape when it was pulled away. The replica was then mounted on a SEM stub and any extra tape was trimmed away. It is important to note that since the notch was dusted with the compressed air, no evidence of debris was observed on the replica surfaces. The above procedure for making replicas was repeated at intervals of approximately 1000 cycles, such that approximately 25–30 replicas were obtained during the life of each specimen. The replicas were then sputter-coated in gold–palladium to maximize surface conductivity for SEM imaging. Starting from the final replica and proceeding backwards to the earliest replica, the lengths of the cracks along the bore of the notch were measured under SEM. It is worth noting that SEM imaging of the replicas were conducted as quickly as possible since, at high magnifications, the replica surface can be damage by the electron beam due to its limited conductivity even with the gold–palladium coating. Fig. 1d shows a set of Repliset® replicas that are ready for imaging.

## 3. Results and discussion

In order to determine if the Repliset® replica process affected the fatigue crack growth rate, the results of the stress-life fatigue tests with replicas were compared to stress-life fatigue tests without replicas. Specimens were subjected to the same stress amplitudes and the surface preparation as described previously. Fig. 2 compares the number of cycles to failure for the replica and non-replica tests. It is clearly seen that the replica tests showed more variability compared to the non-replicas tests for the $R = 0$ and −1. While the maximum and minimum fatigue life for some of the replica tests were outside the bounds of non-replica tests, overall the mean life of the replica tests compared very closely to the non-replica tests and the differences could be attributed to statistical variance commonly observed in fatigue testing. However, more tests are likely needed to accurately assess the statistical effect of the Repliset® replica process on crack growth.

A representative SEM image of a surface fatigue crack taken from a replica is shown in Fig. 3a. In this image, the fatigue crack initiated and propagated from an iron-rich intermetallic particle. It is noted that the surface crack grew symmetrically from the particle, and only the right side of the crack is shown. As shown in Fig. 3a, the crack path was observed to be somewhat tortuous relative to the microstructure. Fig. 3b shows the crack length versus number of cycles for a surface crack located in the notch bore, where the crack length is measured horizontally. The crack length is seen to grow exponentially once the crack length reaches approximately 50 μm in length (point B). From point B to point D, the crack length accelerates rapidly. To illustrate the inherent effect of the microstructure on crack growth, Fig. 3c shows the crack growth rate as a function of surface crack length. It was observed that this particular surface crack experienced crack growth acceleration and retardation from points A through D. The large observed acceleration and retardation in crack growth occurred around point C as a result of the crack propagating through a particle stringer. The large increase in crack growth rates beginning around point C in Fig. 3c corresponds to the rapid increase in crack length observed in Fig. 3b. Some of the variability in crack growth rates could possibly be attributed to the measurement of crack length through the use of SEM imaging. Also, some of the variability could