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# Investigation of particle fracture during fatigue of aluminum 2024

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## ABSTRACT

A significant portion of the mechanical energy that enters into a material subjected to fatigue is expended to nucleate local, early-state damage which is distinct from later observed catastrophic type damage frequently targeted in fatigue investigations. Therefore, identification of early signs of fatigue damage combined with means to monitor its evolution is crucial for understanding the influence of material microstructure on the development of progressive pre-failure sites that can ultimately lead to conditions that favor the development of fatigue damage. In this context, in situ scanning electron microscope (SEM) testing combined with microstructure-sensitive nondestructive evaluation (NDE) is leveraged, in this article, to allow the direct observation of fatigue damage incubation in a precipitate-hardened aluminum alloy, Al 2024-T3. To validate surface observations of such early signs of damage, X-ray Micro-Computed Tomography (μ-CT) scans were made to investigate the relation between particle size and chemical balance with local grain structure and crystallography. In addition, an effort was made to explore the effect of specimen geometry and loading schemes on the occurrence of particle fracture activity as well its evolution throughout the early stages of the specimen life. Furthermore, a machine learning approach was developed in an attempt to post-process the available NDE data and relate it to particle fracture activity.

## 1. Introduction

Fatigue is a complex, stochastic, and multiscale process that varies significantly among different materials. In general, it incubates and nucleates damage at the atomistic scale and gradually evolves spatially leading to observable failure modes, such as cracks. Fatigue damage initiation and subsequent early growth, therefore, requires a significant amount of energy and time to develop and therefore it practically allows its observation at several stages of existence [1,2]. Of particular interest to both material processing and structure-property-behavior relations is consequently the understanding of the earliest signs of fatigue damage and their evolution well before their appearance could be considered detrimental to the structure's useful life. To this effect significant work has been conducted in recent years into development of loading devices capable of performing fatigue at scales where direct observations of relevance to the material can be made. In this context, Biallas and Maier created a survey of in situ devices and discussed various applications including corrosion in water vapor environments, as well as monotonic and cyclic mechanical loading at high and low temperatures [3]. Furthermore, in situ experiments were conducted to study the effects of crack closure [4], incremental crack growth processes [5], crystallography [6] and inclusions [7–9] on crack formation and growth.

Within the scope of understanding and monitoring fatigue in materials, a variety of nondestructive evaluation (NDE) techniques have also enabled both a more detailed understanding of the role various material mechanisms in the evolution of fatigue damage as well as the development of practical methods to monitor it, often at scales that go far above that of laboratory specimen [10–12]. Among other methods, Acoustic Emission (AE) monitoring and Digital Image Correlation (DIC) have proven to be particularly useful across scales for fatigue damage detection and monitoring in materials used in aerospace applications [13–25]. In addition, attempts have been made to link such microstructure effects to AE signals and identify key AE features for detecting dislocation motion [26] and particle fracture validated by post mortem microstructure investigation [17–19,21,23,25,27–33]. Recently, the authors of this article extended the AE monitoring for use inside a Scanning Electron Microscope (SEM) in conjunction with an in situ loading device to observe particle fracture at the grain scale and connect it to real time collected AE waveforms [8,34]. This in situ SEM-AE monitoring has also been shown by the authors to be successful in identifying twin formation in magnesium alloys [35]. Similarly, the DIC method has been used to identify strain localizations resulting from loading and damage mechanisms. DIC has been used to monitor strain evolution in monotonic [24,33,36–38], dynamic [39], and cyclic [40,41] loadings at the meso-scale. Further, DIC has been shown to be

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useful in detecting crack formation at the macroscale on a concrete structure subject to earthquake loads [11,12]. Moreover, recent advances have allowed the extension of DIC to the microscale using images obtained from inside an SEM microscope that make use of microscale patterning methods [41–45] and correct for the distortion introduced by SEM imaging [43,45]. Additionally, AE and DIC have been coupled to provide a deeper understanding of the damage mechanisms investigated and their evolution at the meso-scale to explain crack formation. Recently, the authors used a combined SEM-DIC-AE technique to identify particle fracture directly at the time and scale it occurred for the first time [34].

Specifically, for aluminum alloys, a significant amount of research into the mechanical behavior and characterization has been conducted, across scales, to study damage evolution and crack growth subjected to both monotonic [17,46,47], bending [48], and fatigue loading conditions [1,4,5,7,18,49–56]. Furthermore, particles have been investigated as damage nucleation sites resulting from their fracture [54,57] or debonding [57] with a few of such studies conducted to examine particle fracture as it occurs in Al7075 [7]. Additionally, connections have been made between the size of particles and the likelihood of fracture [58]. However, limited effort has been made to identify the role of particle fracture below the surface within the context of fatigue damage initiation.

This article targets first the identification of particle fracture activity and then to understand the processes that cause it. To achieve this goal, testing and NDE monitoring inside the SEM are performed with surface strain and acoustic emission activity recorded simultaneously. In addition, X-ray μ-CT data were obtained to link surface observations with the evolution during cyclic loading of key microstructural features such as particles and voids. Subsequently, the evolution from particle fracture to crack growth was observed and discussed in terms of particle size, particle chemical balance, local grain structure, and crystal orientation. Additionally, this information was leveraged with a machine learning approach to cluster the AE data and link the results to in situ damage observations.

## 2. Experimental procedure

### 2.1. Material

Aircraft grade precipitate-hardened aluminum 2024-T3 rolled sheets with a 2.5 mm nominal thickness were used in this investigation. The aluminum was used in the as-received condition which contains up to 5% Cu, 1.8% Mg, 1% Mn, 0.5% Fe, 0.5% Si and trace amounts of Cr and Zn. A section of the sheet was mechanically polished to 0.05 μm alumina suspension and lightly etched with Keller's Reagent for initial characterization and pertinent results are shown in Fig. 1. The grains are found to be relatively equiaxed with only a few of them elongated in the rolling direction. Additionally, a preference for grains aligned with [0 0 1] and [1 0 1] directions was observed as shown in Fig. 1a. Furthermore, the grains were distributed in size as found in Fig. 1b showing a range between ~15–120 μm with the majority being between 30 and 60 μm. Fig. 1c reveals a texture necessitating the need for consistent loading parallel to the loading direction to mitigate texture effects in damage evolution. Additionally, the particles present in the aluminum were examined by energy dispersive spectroscopy; the chemical content was identified as primary particle types including $Al_7Cu_2Fe$, $Al_2CuMg$, and $Al_2Cu$ which agrees with related information in the literature [59–61].

### 2.2. Specimen preparation

In order to localize damage and assist with observations, specimens were initially designed to have sharp notches similar to previous work by the authors [8,34]. In addition, nanoindentations were applied with a grid spacing of 100 μm to further localize the damage while also creating a set of fiducial markers for in situ monitoring. Fig. 2a and b show the sharp notch geometry with the indentation

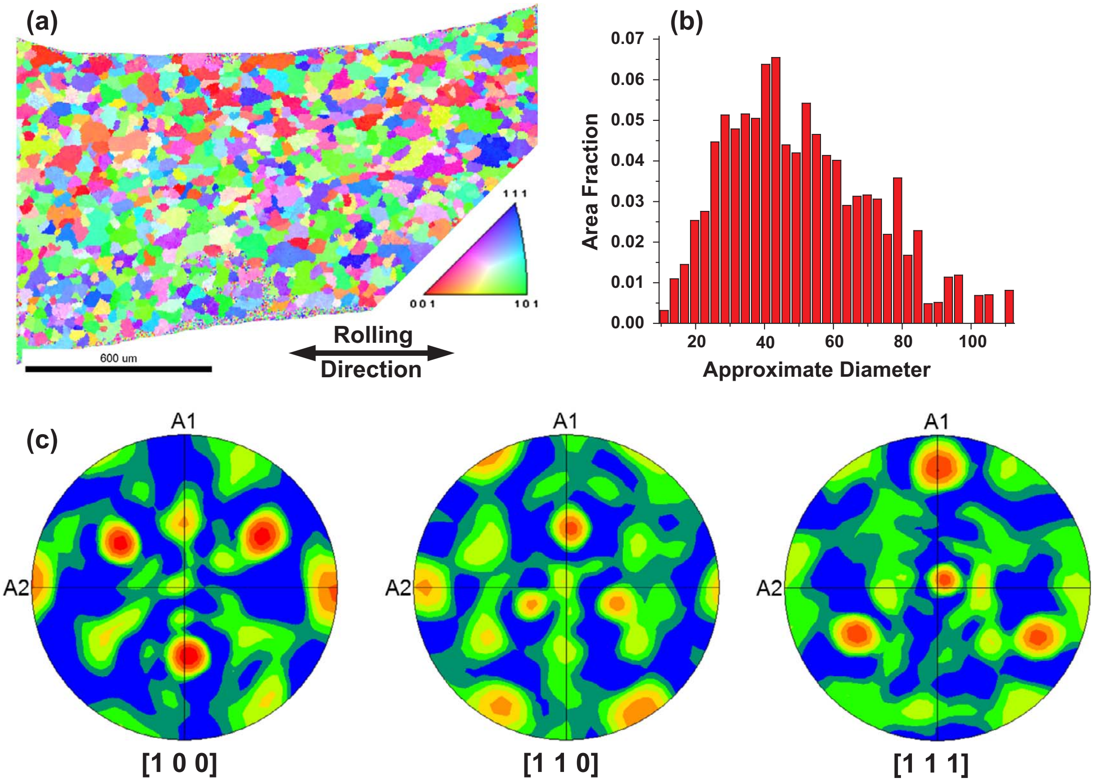

**Fig. 1.** Surface characterization including (a) inverse pole figure showing a variety of grain sizes. (b) Grain size histogram. (c) Pole figures showing a slight rolling texture where A1 is the transverse direction and A2 is the rolling direction indicated in (a).