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Fatigue crack growth in SiC particle reinforced Al alloy matrix composites at high and low *R*-ratios by *in situ* X-ray synchrotron tomography

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ABSTRACT

Metal matrix composites (MMCs) offer high strength, high stiffness, low density, and good fatigue resistance, while maintaining cost an acceptable level. Fatigue resistance of MMCs depends on many aspects of composite microstructure. Fatigue crack growth behavior is particularly dependent on the reinforcement characteristics and matrix microstructure. The goal of this work is to obtain a fundamental understanding of fatigue crack growth behavior in SiC particle-reinforced 2080 Al alloy composites. '*In situ* X-ray synchrotron tomography was performed on two samples at low ($R = 0.1$) and at high ($R = 0.6$) *R*-ratios. The resulting reconstructed images were used to obtain three-dimensional (3D) rendering of the particles and fatigue crack. Behaviors of the particles and crack, as well as their interaction, were analyzed and quantified. Four-dimensional (4D) visual representations were constructed to aid in the overall understanding of damage evolution.

1. Introduction

Metal matrix composites (MMCs), composed of a light alloy matrix, such as aluminum alloy, reinforced with ceramic particulates, have a combination of high strength, high stiffness, and low density [1]. Furthermore, MMCs exhibit improved fatigue resistance over monolithic alloys. This fatigue resistance depends on a variety of factors, such as reinforcement particle volume fraction, particle size, matrix and interfacial microstructure, processing-induced inclusions or defects, and testing environment [2–8].

In previous years, an understanding of the precise nature of damage mechanisms in MMCs has been largely limited to examinations of two-dimensional (2D) fracture surfaces or polished cross-sections of the material. These techniques are laborious and restrictive due to the 2D nature of the analysis. Accurately sampling a representative volume of the microstructure by such methods is also difficult. While these surface techniques provide information about microstructure, both the microstructure and state of stress can differ greatly between the surface and the bulk [9,10].

Three dimensional (3D) characterization tools have been recently developed to allow for clear, accurate, and quantitative depictions of the damage behavior of MMCs. A number of techniques have been used for the 3D visualization of MMC microstructures, including serial sectioning techniques using either mechanical polishing coupled with optical microscopy [11,12] or focused ion beam milling and image reconstruction [13–15]. Although serial sectioning is a powerful technique for virtual 3D microstructure generation, it is both time consuming and destructive.

X-ray tomography is an excellent technique that, unlike serial sectioning, is non-destructive and allows for large volumes to be studied with minimal sample preparation, resulting in statistically significant information and relatively large-scale visualization capabilities [16–19]. 3D visualization and quantification of heterogeneous microstructures has been successfully performed in Sn-rich alloys [20], powder metallurgy steels [21], metal matrix composites [22–25] and aluminum and copper alloys [26–28].

Synchrotron radiation has been used for X-ray tomography [22] and holotomography [23,24] of MMCs to visualize their microstructures. SiC particle reinforced aluminum alloy matrix composites have been investigated using X-ray synchrotron tomography to visualize and quantify Fe-rich inclusions and porosity [25] as

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well as the influence of particle size and aspect ratio on tensile fracture [29]. More recently, *in situ*, or 4D experiments (the fourth dimension here is time) have been conducted to understand the deformation behavior in real-time. Tensile damage behavior of SiC particle reinforced aluminum alloy matrix has been studied using *in situ* X-ray synchrotron tomography [30]. *In situ* X-ray synchrotron tomography has been used for 3D observation and quantification of fatigue cracking in Al–Mg–Si alloys [31] and to visualize void volume changes due to thermal fatigue damage in SiC particle reinforced aluminum [32].

Adequate visualization and fracture quantification is critical to the understanding of damage in MMCs. Additionally, a significant amount of statistical characterization and analysis, on features such as particle fracture and crack growth, is required both before and after deformation. *In situ* techniques are particularly well suited for examining the initiation and evolution of damage in MMCs over time, allowing for a sound understanding of the sequence of particle fracture and crack growth during fatigue to be obtained.

In the area of fatigue, a significant amount of work has been done in trying to understand the interaction between the fatigue crack and the SiC particles [33,34]. Although the effect of load ratio (positive *R*-ratio) on fatigue crack growth behavior in MMCs has been investigated [35–37], the precise mechanisms of crack growth as a function of *R*-ratio remain poorly understood. This is partially because most fatigue crack growth studies are limited to predominantly low *R*-ratios. Moreover, the scope of existing studies at high *R*-ratios are limited to optical imaging of cracking at the sample surface and of fracture surfaces [38,39]. At low *R*-ratio crack deflection around SiC particles has been qualitatively shown. At high *R*-ratio, however, the key question is whether the SiC particles crack ahead of the main crack, or whether the fatigue crack grows through the originally intact SiC particles. Post-mortem analysis on the surface of the specimen only shows the final state of fracture, i.e., that the crack has gone through the particle, but it does not account for potential fractures ahead of the crack tip. The precise mechanisms for fracture, not only on the surface but through the thickness, can only be verified by *in situ* X-ray microtomography.

In this work, the fatigue behavior of particle reinforced aluminum alloys at a high *R*-ratio (0.6) is compared to that at low *R*-ratio (0.1) by *in situ* X-ray synchrotron tomography. In particular, the goal of this work is to obtain a fundamental understanding of how the fatigue crack interacts with SiC particles, and the role of particle fracture, both ahead of and at the crack tip, in controlling fatigue crack propagation. This behavior for high *R*-ratios versus low *R*-ratios will be discussed after quantitative analysis through 3D visualization of segmented particles and crack coupled with the examination of numerous 2D slices.

2. Material and experimental procedure

The material used in this study was a 2080 aluminum alloy (3.6% Cu, 1.9% Mg, 0.25% Zr) reinforced with 20 vol.% SiC particles (average particle size of 25 μm). The materials were processed by blending SiC and Al powders, compaction of the powder mixture, hot pressing, and hot extrusion (Alcoa Inc., Alcoa, PA). Details of the powder metallurgy process for fabrication of these composite materials can be found elsewhere [5].

Fatigue crack growth experiments were carried out on single edge notched (SEN) specimens. The specimens were machined by electrical discharge machining (EDM) parallel to the extrusion axis with the crack growing normal to the extrusion axis, i.e., in the short transverse axis (L–S orientation). The specimens were approximately 1 mm thick, 2.7 mm wide, and 15 mm long, as shown in Fig. 1(a). A microforce testing system (MTS Tytron 250) was used to perform pre-cracking *ex situ* in tension–tension fatigue at the same load ratios as those used during the *in situ* experiment for each sample (frequency of 4 Hz, $\Delta K \approx 5$ MPa $\sqrt{m}$). The resulting pre-crack length for the sample tested under $R = 0.1$ was 0.95 mm, while that for $R = 0.6$ was 0.55 mm.

The fatigue experiments were performed at the Advanced Photon Source (APS) at Argonne National Laboratory under load ratios of 0.1 and 0.6, at a frequency of 1 Hz, using a sinusoidal waveform. X-ray synchrotron tomography was performed at the 2 BM beamline of the APS. Details of the APS beamline 2-BM have been described elsewhere [29,40–42]. Using the standard detector, a 2016 × 2016 pixel PCO Dimax CMOS camera coupled with a LuAG:Ce scintillator screen (used to convert the X-rays to visible light), typical exposure times of 250 ms per projection were obtained. In this configuration, a projection was collected every 1/8° for 180°. The voxel size for $R = 0.6$ experiments was 1.47 μm and for $R = 0.1$ was 1.8 μm. The tomography at one time step can be completed in about 10–15 min. The two-dimensional (2D) projections were reconstructed in 3D using a filtered back-projection algorithm.

A specially designed loading stage, depicted in Fig. 1(b), was used for the *in situ* tomography. The load was transmitted from top to bottom of the stage using a polymer PMMA (Polymethyl methacrylate) sleeve. PMMA was used because it is essentially X-ray transparent. The specimen was inserted from the top of the stage and was clamped between the actuator and the load cell. The stepper motor had a captive linear actuator capable of 8 μm per step and a total stroke of 25 mm. The load cell had a capacity of 500 N. The load was applied to the specimen by automatic control of the actuator using feedback from the load cell. The fatigue tests were run at constant load amplitude. After a specified number of cycles were completed, the load was held constant at a value slightly lower than the maximum load and the sample was scanned. A slight overload was obtained in the higher *R*-ratio sample due to the difficulty in controlling the load. The location and magnitude of the overload was recorded and the appropriate change in damage was quantified.

From the obtained stack of reconstructed images, the SiC particles were segmented from the Al alloy matrix. Due to the similarity in gray scale between the center of a particle and the Al alloy matrix, a novel and semi-automatic segmentation algorithm known as Livewire® (Mimics, Materialise, Ann Arbor, MI) was used, and is described in detail elsewhere [29]. This algorithm takes advantage of the large gradient in grayscale at the interface between the particle and the matrix [43,44]. The crack was also segmented at various stages of growth using a grayscale thresholding tool and subsequent close visual comparison to the original tomography images for removal of anything falsely segmented as part of the crack, such as the dark silicon-rich inclusions. The segmented data was then exported to Avizo® Fire (Visualization Sciences Group, Burlington, MA) for 3D rendering and microstructural quantification. Selected volumes for each *R*-ratio were considered for 3D quantification. These volumes were selected such that the width was equal, the height encompassed the full crack throughout that width, and the length captured the full propagation of the crack. Videos were made for the high *R*-ratio sample to show crack growth, particle fracture, and crack-particle interaction throughout the *in situ* fatigue experiment in 4D.

3. Results and discussion

The differences in fatigue crack growth damage at the tip of the crack, and the subsequent interactions between the crack and the SiC particles, at low and high *R*-ratio, are quite striking. A 2D comparison of the evolution of damage under a load ratio of 0.1 (135 μm