Conference Proceedings of the Society for Experimental Mechanics Series

Alan T. Zehnder · Jay Carroll · Kavan Hazeli · Ryan B. Berke
Garrett Pataky · Matthew Cavalli · Alison M. Beese
Shuman Xia *Editors*

Fracture, Fatigue, Failure and Damage Evolution, Volume 8

Proceedings of the 2016 Annual Conference on Experimental and Applied Mechanics

SEM

Springer

Conference Proceedings of the Society for Experimental Mechanics Series

Series Editor

Kristin B. Zimmerman, Ph.D.
Society for Experimental Mechanics, Inc.
Bethel, CT, USA

More information about this series at http://www.springer.com/series/8922

Alan T. Zehnder • Jay Carroll • Kavan Hazeli • Ryan B. Berke
Garrett Pataky • Matthew Cavalli • Alison M. Beese • Shuman Xia
Editors

Fracture, Fatigue, Failure and Damage Evolution, Volume 8

Proceedings of the 2016 Annual Conference on Experimental and Applied Mechanics

Springer

Editors
Alan T. Zehnder
Cornell University
Ithaca, NY, USA

Kavan Hazeli
John Hopkins University
Baltimore, MD, USA

Garrett Pataky
Department of Mechanical Engineering
Clemson University
Clemson, SC, USA

Alison M. Beese
Pennsylvania State University
State College, PA, USA

Jay Carroll
Sandia National Laboratories
Albuquerque, NM, USA

Ryan B. Berke
School of Engineering
University of Liverpool
Liverpool, UK

Matthew Cavalli
Department of Mechanical Engineering
University of North Dakota
Grand Forks, ND, USA

Shuman Xia
Georgia Institute of Technology
Atlanda, GA, USA

ISSN 2191-5644 ISSN 2191-5652 (electronic)
Conference Proceedings of the Society for Experimental Mechanics Series
ISBN 978-3-319-42194-0 ISBN 978-3-319-42195-7 (eBook)
DOI 10.1007/978-3-319-42195-7

Library of Congress Control Number: 2016949027

Printed on acid-free paper

This Springer imprint is published by Springer Nature
The registered company is Springer International Publishing AG Switzerland

Preface

Fracture, Fatigue, Failure and Damage Evolution represents one of ten volumes of technical papers presented at the SEM 2016 Annual Conference & Exposition on Experimental and Applied Mechanics organized by the Society for Experimental Mechanics and held in Orlando, FL, June 6–9, 2016. The complete proceedings also includes volumes on *Dynamic Behavior of Materials*; *Challenges in Mechanics of Time-Dependent Materials*; *Advancement of Optical Methods in Experimental Mechanics*; *Experimental and Applied Mechanics*; *Micro-and Nanomechanics*; *Mechanics of Biological Systems and Materials*; *Mechanics of Composite and Multifunctional Materials*; *Residual Stress, Thermomechanics and Infrared Imaging, Hybrid Techniques and Inverse Problems*; and *Joining Technologies for Composites and Dissimilar Materials.*

Each collection presents early findings from experimental and computational investigations on an important area within experimental mechanics, fracture and fatigue being one of these areas.

Fatigue and fracture are two of the most critical considerations in engineering design. Understanding and characterizing fatigue and fracture has remained as one of the primary focus areas of experimental mechanics for several decades. Advances in experimental techniques, such as digital image correlation, acoustic emissions, and electron microscopy, have allowed for deeper study of phenomena related to fatigue and fracture. This volume contains the results of investigations of several aspects of fatigue and fracture such as microstructural effects, the behavior of interfaces, the behavior of different and/or complex materials such as composites, and environmental and loading effects. The collection of experimental mechanics research included here represents another step toward solving the long-term challenges associated with fatigue and fracture.

Ithaca, NY, USA — Alan T. Zehnder
Albuquerque, NM, USA — Jay Carroll
Baltimore, MD, USA — Kavan Hazeli
Liverpool, UK — Ryan B. Berke
Clemson, SC, USA — Garrett Pataky
Grand Forks, ND, USA — Matthew Cavalli
State College, PA, USA — Allison M. Beese
Atlanta, GA, USA — Shuman Xia

Contents

Chapter 1
Fatigue Damage Precursor Identification Using Nondestructive Evaluation Coupled with Electron Microscopy

B. Wisner and Antonios Kontsos

Abstract Several fatigue failure modes originate at the microstructural level by the activation, interactions and development of what are referred to as "damage precursors" long before the formation of dominant cracks that grow as a function of loading and crystallographic parameters. In this context, this work presents new developments of an in-house developed experimental mechanics approach to evaluate aspects of microstructure evolution and identify validated damage precursors that are active during fatigue loading by combining Nondestructive Evaluation (NDE) methods with ex situ and in situ Scanning Electron Microscopy (SEM). The used NDE methods include real time Acoustic Emission (AE) monitoring from inside the SEM chamber and Digital Image Correlation (DIC) for strain evolution directly at the grain scale. The coupling between quasi in situ microscopy with actual in situ nondestructive evaluation falls into the ICME framework and the idea of quantitative data-driven and multiscale characterization of material behavior. To demonstrate this approach, Aluminum 2024-T3 specimens were tested using a SEM mechanical testing stage under low cycle fatigue to identify and validate the presence of damage precursors, while correlating their presence with specific parameters extracted by the available NDE data. The reported results show how load information could be correlated with both AE activity, DIC strain maps, and microscopic observations of precipitate fracture and microcracks.

Keywords Damage precursors • Inclusion fracture • Particle fracture • Damage detection • In situ • NDE

1.1 Introduction

Aluminum alloys are widely used both because of their high strength to weight ratio as well as the existence of large-quantity and controllable quality manufacturing procedures, developed over the past several decades [1–6]. Given their mass adoption, there is a wide variety of examples in engineering applications in which fatigue and fracture of such alloys becomes a dominant economical parameter, as this relates to life cycle related decisions for existing structures and their continuing use in demanding operation environments encountered for instance in marine and aerospace applications, as well as a crucial design parameter for the next generation of lightweight structures starting from physical metallurgy all the way until final product finish. Given this context, the objective of this article is to investigate the role of state-of-the-art and multiphysics nondestructive evaluation (NDE) based on acoustics, optics and microscopy coupled with direct measurements of microstructural effects in material behavior to detect, identify and analyze the contribution of specific microstructural features on the room temperature fatigue behavior of commercial-grade aluminum alloys used in aerospace.

For this reason this research focuses on the aircraft grade Al alloy, 2024-T3. This alloy is a precipitate-hardened material containing Cu, Fe, Si, Mg, and Mn as the primary alloying elements [1]. The specific alloying and temper process results in Cu and Fe rich hard inclusions/particles with sizes >1 μm that have been reported in literature since 1969 by Grosskreuts and Shaw to be dominant fatigue crack nucleation sites [7]. In addition, Gao et al. identified similar hard inclusions in Al 7075-T6 and 2024-T3 [8]. Related work by Agarwal et al. attempted to connect bulk strain levels with the number of fractured particles based on surface observations [9]. However, to accomplish this task, interrupted tests were performed to allow for surface observations of damaged particles inside a scanning electron microscope (SEM). Therefore for both material synthesis and damage prevention purposes, it becomes crucial to identify and develop a method capable of informing when surface and subsurface particles fracture and what is their effect on local and global mechanical behavior. In this direction, a number of papers have been published further supporting crack nucleation at hard inclusions [3, 10–12].

B. Wisner • A. Kontsos (✉)
Department of Mechanical Engineering and Mechanics, Drexel University, Philadelphia, PA 19104, USA
e-mail: akontsos@coe.drexel.edu

A.T. Zehnder et al. (eds.), *Fracture, Fatigue, Failure and Damage Evolution, Volume 8*,
Conference Proceedings of the Society for Experimental Mechanics Series, DOI 10.1007/978-3-319-42195-7_1

In this framework the innovation recently proposed by the authors [13] is related to what is termed here as microstructure-sensitive Acoustic Emission (AE) monitoring for identifying damage directly at the scale that this occurs in Aluminum alloys. In fact, the term "acoustic emission" is concurrently used to denote both the natural phenomenon [14–17] that occurs in all materials at multiple scales that is related to the energy release that occurs as a result of mostly irreversible processes such as plasticity and fracture [18], as well as the versatile and passive NDE technique that detects travelling elastic waves by the use of a variety of transduces and signal processing methods [19, 20].

Therefore, AE is suitable for application both at the microstructural and structural levels allowing for connections between fundamental science observations and practical applications. Indeed since the work by Hamstad et al. [21] efforts have been made to distinguish the different sources of AE included slip as well as noise, however, such distinction was difficult if not impossible due to limitations in both observational means (e.g. in situ microscopy) as well as lack of advanced digital signal processing capabilities. Despite these deficiencies, Hamstad and others argued that burst AE signals, similar to those emitted during particle fracture are easier to identify, primarily due to their high amplitude and energy as well as their narrowband frequency content, and therefore link them to the activation of actual damage sources. Moreover, McBride et al. [22] attempted to make a quantitative connection between AE amplitude and particle fracture using Al7075 containing particles and comparing it to pure Al with no particles; AE from particle fracture in this investigation was reported to be burst in nature and resulting from large particle (~10 μm), which the authors supported by providing post mortem evaluation of the specimens. Further work by Wells et al. [23] attempted to separate the dislocation motion (slip) related AE sources from particle fracture using the peaks in root-mean-square (rms) value of the AE signals. The hypothesis is such analysis was that particle fracture only happens in tension while slip occurs in both tension and compression. Therefore these authors argued that signals present in both directions of loading are representative of slip and the signals present only in the tension tests are most likely occurring due to particle fracture. Furthermore, AE has been used to identify damage and material degradation of a variety of materials; in a recent example Ai et al. [24] proposed the use of NDE methods, primarily AE to detect micro-defects and cracks in Al A356 by proposing the use of additional AE features such as counts and energy to determine the level of damage in the material.

The research discussed in this paper proposes to make use of recent advances in technology to perform experiments inside an SEM microscope for direct observation of micro processes of interest (particle fracture) while recording and monitoring AE events in real time. Furthermore, other NDE techniques such as digital image correlation (DIC) are implemented to assist in the identification of specific time, load and strain values of a priori identified particle fractures to directly correlate recorded AE signals with observable fracture events. Specific AE features are extracted and compared to other AE information obtained from other micro processes in both monotonic and fatigue tests. The overall experimental mechanics approach allows the extraction of parameters that can be leveraged by both crystal and continuum level plasticity models that are concurrently developed by the authors.

1.2 Experimental Procedure

1.2.1 Mechanical and Nondestructive Evaluation Setup

To combine AE signals, microscale DIC images and microstructure evolution while a specimen is undergoing load the experimental setup shown in Fig. 1.1 was used [13]. Tensile loads were applied by a GATAN MTEST2000 load stage capable of supplying both monotonic tension and compression loads as well as performing cyclic tension–tension and compression–compression fatigue tests. For this work, the stage was placed inside a FEI XL30 ESEM under high vacuum and specimens were loaded at a rate of 0.1 mm/min to allow for in situ monitoring and video recording of microstructure evolution without significant blurring. Additionally, high resolution secondary and backscatter electron images were obtained prior to, and after loading to characterize the materials initial and final state and quantify any induced microstructural changes, as well as to perform 2D DIC deformation measurements.

Acoustic Emission signals were monitored and recorded throughout loading using a four-channel data acquisition system consisting of two PCI-2 boards (manufactured by MISTRAS, Inc.). Two piezoelectric sensors (MISTRAS, Pico Sensor) were attached to the back of the specimen and the signals were passed through the SEM chamber by means of specially made BNC connectors mounted on the MTEST stage door. The signals were obtained at a sampling rate of ten million samples per second to avoid the possibility of signal aliasing. Furthermore, the signals were amplified by using 2/4/6 preamplifiers (MISTRAS, Inc.) and were band-pass filtered in the frequency range of 0.2–1 MHz (which is the manufacturer's defined useful range of the PICO sensors used). The peak definition, hit definition, and hit lock out times were set to 100, 400, and

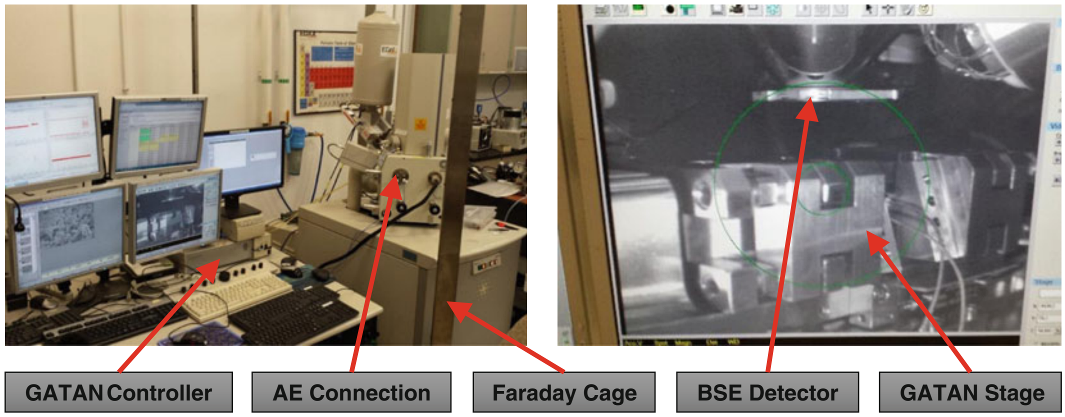

Fig. 1.1 Experimental set up used consisting of a GATAN MTEST 2000N load cell stage, custom door with BNC connectors for up to four AE channels, and Faraday Cage for EMI reduction in AE signals

500 μs respectively based on the signals obtained in previous tests by the authors [13]. Additionally, the entire SEM and AE acquisition system was contained within a Faraday cage designed to reduce the effects of EMI present in the area on the AE signals obtained. In fact, the presence of this cage was capable to reduce the noise floor from 35 to 20 dB allowing for the acquisition of lower amplitude signals (such as those expected from particle fracture) to be captured and analyzed.

DIC is performed using images obtained from the SEM during loading. The speckle pattern is achieved using Cu particles with average diameter of 300 nm applied to the surface of the specimen through a filter [25]. This method allows for the microstructural changes to be observed though the pattern. Images are analyzed using VIC-2D from correlated solutions to calculate strain localizations. A subset size and step size of 400 μm and 15 μm respectively for the low magnification DIC. High magnification (1000×) resulted in a subset size of 10 μm and a step size of 0.7 μm.

1.2.2 Specimen Preparation

A commercially available 2.5 mm-think rolled Al 2024-T3 sheet was obtained; 28 mm long double notch specimens were cut by EDM from the sheet as shown in Fig. 1.2a. The sharp notch region was designed to localize plasticity for observations in the SEM. Prior loading, the specimens were ground flat and polished to 0.05 alumina suspension for Electron Backscatter Diffraction (EBSD). A representative map is presented in Fig. 1.2b showing grains of ~100 μm mixed with smaller grains on the order of 30 μm. Given the EBSD results in Fig. 1.2b it was concluded that there is no observable texture in the as-received material.

Al 2024 primary alloying elements include Cu, Fe, Mg, Mn, and Si. Consequently, metallic inclusions with sizes greater than 1 μm are abundant. These inclusions/particles have been shown to lead to the formation of fatigue cracks caused both by elastic mismatches between the surrounding matrix and the particles, as well as by geometric discontinuities due to the particles shape. Such effects cause either debonding of the particle from the surrounding matrix [3, 26] and/or particle fracture [3, 10–12]. To determine the composition of these particles and identify possible candidates for fracture, Energy-Dispersive X-ray Spectroscopy (EDS) maps were produced (Fig. 1.2c) since hard inclusions typically have high concentrations of Fe and Cu.

1.3 Results and Discussion

Two sets of experiments were conducted to investigate the detection of fatigue damage precursors. Specifically, specimens were initially loaded monotonically up to a point that the first signs of damage prior to crack nucleation appeared. Then, monotonic tests were conducted until several surface particles were seen to fracture to determine the associated AE signals. Finally, this information was investigated by performing cyclic tension-tension testing.

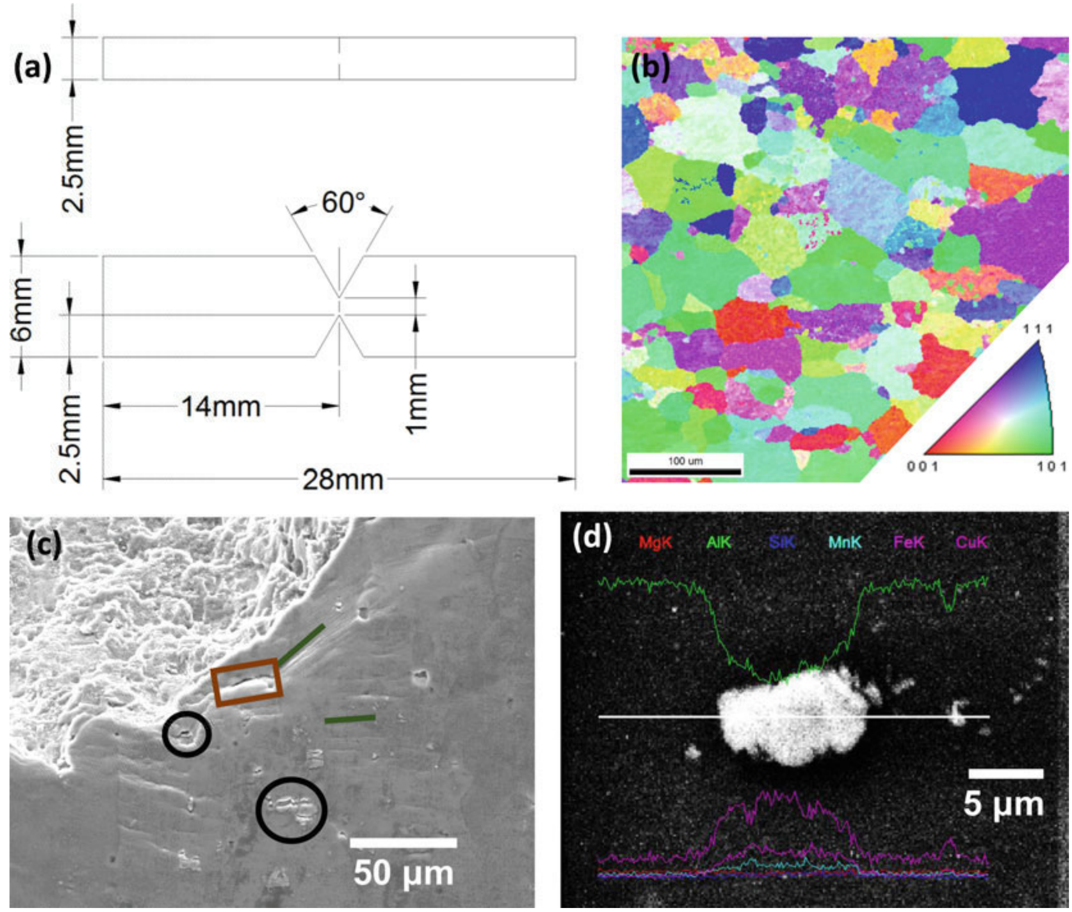

Fig. 1.2 (**a**) Specimen Geometry with notches to localize the plastic zone. (**b**) EBSD map of the region showing grains on ranging from 20 to over 100 μm. (**c**) Notch region after loading indicating slip (*lines*), void nucleation (*boxes*), and particle fracture (*circles*) as initial damage prior to failure. (**d**) Single particle of interest depicting high Fe and Cu content (hard inclusion)

1.3.1 Monotonic In Situ SEM Testing

Based on the related literature, the relatively larger and hard inclusions tend to be the particles that fracture first [7, 9, 22]. Therefore, an area near the notch root with a number of inclusions with sizes greater than 1 μm was selected for monitoring in the SEM. Secondary Electron imaging was used with frame averaging of four images to reduce the effects of noise without causing blurriness due to the motion. Low frequency continuous signals were recorded continuously as shown in Fig. 1.3a. These AE signals obtained during the stages of first application and subsequent initial stages of loading generally served the purpose of identifying the characteristics of noise (due to both hardware and setup issues) and perhaps some early plasticity which were definitely though distinct with respect to later observed and possibly particle fracture related AE content. Specifically, burst-type signals started to appear later in the load cycle, usually slightly before or at loads near yielding loads (Fig. 1.3a). Live SEM monitoring of particles at the magnification and resolution required to see fracture events resulted in a FOV of approximately 300 μm^2. Consequently, only a select number of particles could be monitored in real time, while any other fractured ones could be inferred from comparison of images before loading and after failure.

The orange line in Fig. 1.3a indicates the point after which particle fracture was observed, given the procedure described herein. In addition, Figs. 1.3b, c show the monitored region before and after this loading point. More than one particle fracture events was observed in these microscopy images, as well as other mechanisms such as void formation and growth. Figure 1.3d highlights some of these mechanisms.

Additional information can be extracted for the AE data shown in Fig. 1.3a that can be potentially used to identify occurring damage. Specifically, in Fig. 1.4a there are two locations of absolute energy jump, the first being at the onset of "high frequency" (in this case higher than 300 kHz) in Fig. 1.4b, while the second occurs later at the same time as observed

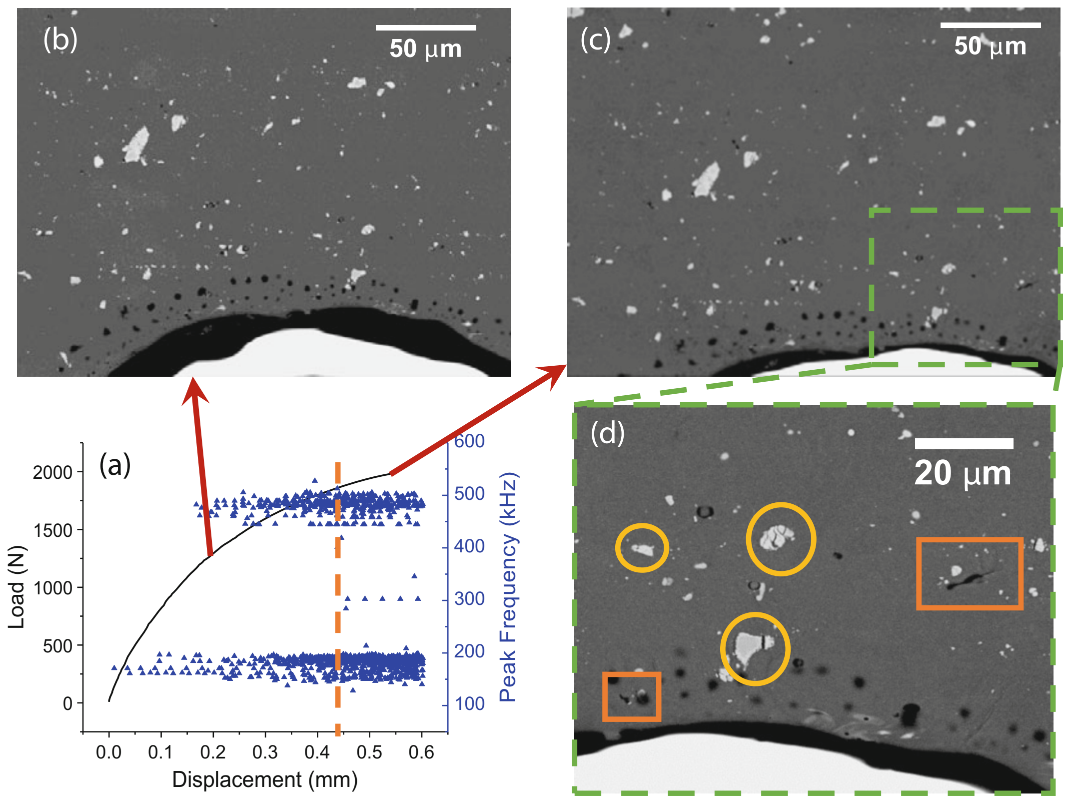

Fig. 1.3 (**a**) Load profile with AE Peak frequency overlaid. The vertical line indicates the location of observed particle fracture. (**b**) SE micrograph of the monitored region prior to particle fracture. (**c**) SE micrograph of the monitored region after loading stopped because of observed particle fracture. (**d**) Higher magnification image showing particles fractured (*circles*) and voids growing (*boxes*)

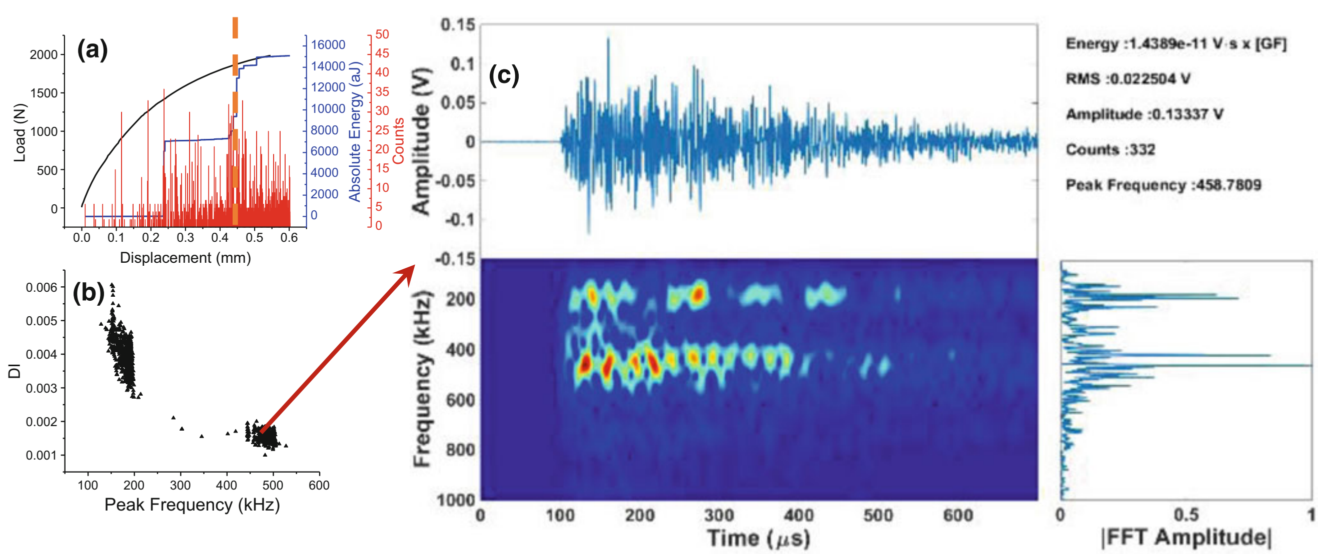

Fig. 1.4 (**a**) Load profile with cumulative AE Absolute Energy overlaid showing a jump in energy at the time of observed particle fracture. Again, the vertical line indicates the location of observed particle fracture. (**b**) AE Damage Indicator plotted against Peak Frequency showing a cluster of signals with High Frequency and low DI. (**c**) The AE signal that occurred at the time of observed particle fracture plotted with SFFT and FFT to show frequency evolution

particle fracture suggesting damage signals between 450 and 550 kHz signals. These jumps occurred because of the monitoring of sudden increase in AE activity, captured by the spikes in the counts bar plot in Fig. 1.4a. Taking advantage of the real time recorded video, the time and displacement of the observed particle fracture can be estimated. Knowing that particle fracture is itself a sudden event possibly resulting in burst-type acoustic emissions, efforts were made to identify the

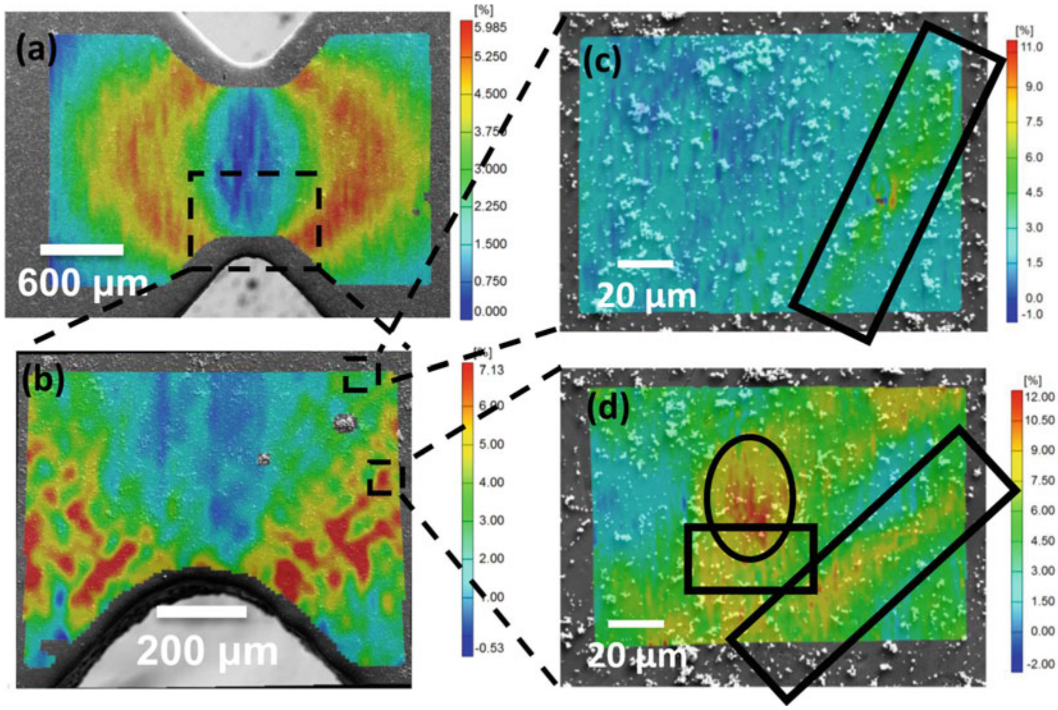

Fig. 1.5 (**a**) Full Field X direction Strain profile (Specimen was loaded in the X direction) showing geometrically induced strains concentrations as semi-circles. (**b**) Higher magnification DIC was performed revealing the "continuous" strain field (**a**) contains further localizations. Further magnification reveals Strain localizations around *Slip lines* (**c**) as well as around pre-existing voids (**d**). Locations of slip are represented by *boxes* and the void location is identified by the *circle*

specific AE signals resulting from that source (Fig. 1.4c). To this aim, Short time Fast Fourier Transfer (SFFT) results in Fig. 1.4d reveal the frequency content of the signal distributed over time and shows multiple bursts of ~500 kHz throughout the duration of the AE signal in Fig. 1.4c. This observation may suggest that there may be multiple AE signals from multiple sources activated at the same time window of a few 100 ms which then were combined into one AE signal due to the particular hardware and software settings. On-going work by the authors (not reported here) utilizing continuous and not hit-driven waveform streaming has the potential to determine if AE signals such as the one reported in Fig. 1.4c represent one or possible multiple fractures. Additionally, the AE activity can be used to identify key features for determination of a damage indicator, such as the one depicted in Fig. 1.4b based on previous work of the authors [13, 19] which could then be used for possible live detection of particle fracture in both monotonic and fatigue loading conditions.

The concurrent use of other NDE methods such as DIC could add further information and assist in the identification of conditions, e.g. strain localizations that lead to particle fracture and increase, therefore, the reliability of damage source identification using the AE method. In this context, Fig. 1.5 shows related DIC measurements made for this type of specimen. It can be seen using multiscale deformation measurements that "macroscopic" plasticity (Fig. 1.5a) near the notched regions results from "microplasticity" effects (Fig. 1.5b) manifested by the pattern strain localizations radiated in distinct directions from the notch that either induce particle fracture or result from it. The strain measurements in Fig. 1.5b resemble dislocation-related cell patterns observed in aluminum alloys. In addition, localizations around voids (Figs. 1.5c, d) can be seen to combine themselves and result in the localizations seen in Fig. 1.5a.

1.3.2 Fatigue In Situ SEM Testing

The same methodology was used for cyclic testing for a maximum of 45 cycles at 85 % of the measured ultimate strength of the material with R = 0.1. In the tests performed, no change was observed on the surface of the specific FOV used as shown in Fig. 1.6, however similar AE activity to that obtained from particle fracture in monotonic loading was observed from the first cycle possibly suggesting that particles are fracturing somewhere in the material. Further fatigue tests are required with either higher loads or more cycles to identify the source of this AE activity.

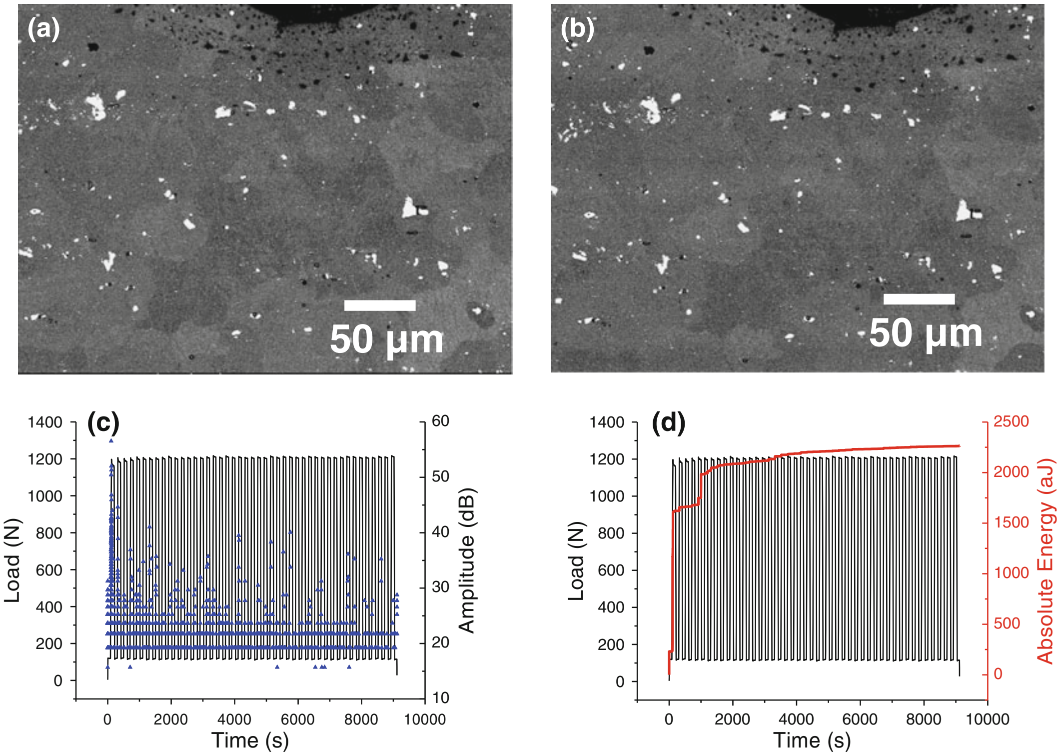

Fig. 1.6 (**a**) SEM image of the region prior to cyclic loading. (**b**) SEM image of the region after cyclic loading showing no change. (**c**) Load vs time curve with AE amplitude overlaid showing a large number of signals during the first cycle with the number of hits decreasing after. (**d**) Absolute energy curve overlaid over the load curve showing two spikes of energy at cycles one and five suggesting the accumulation of damage in these cycles

1.4 Conclusion

Al 2024-T3 fails in fatigue from cracks nucleated from particles fracturing or debonding. NDE methods coupled with in situ SEM observations allowed for the identification of AE sources at the length scale they occur. Specifically, AE data was coupled with information obtained from DIC and changes in the microstructure to identify damage precursors in Al 2024. Particle fracture was connected to representative waveforms, and this information could then be used to quantify a material's state based on the number of signals obtained. The information obtained by AE and DIC could be also fed into models to improve estimates of remaining useful life based on information of the materials physical state.

Acknowledgements The authors would like to acknowledge the financial support received by the Office of Naval Research under the Young Investigator Program, Award #N00014-14-1-0571. The authors further acknowledge the technical support received under the National Aeronautics and Space Administration Space Act Agreement, No. SAA1-19439 with Langley Research Center. Finally, the authors acknowledge the technical support received from Sandia National Labs (SAND2016-1174O)

References

1. Davis, J.R., Davis, J.R.: Aluminum and Aluminum Alloys. ASM International, Materials Park, OH (1993)
2. Esawi, A.M., Aboulkhair, N.T.: Bi-modally structured pure aluminum for enhanced strength and ductility. Mater. Des. **83**, 493–498 (2015)
3. Hahn, G., Rosenfield, A.: Metallurgical factors affecting fracture toughness of aluminum alloys. Metall. Trans. A **6**(4), 653–668 (1975)
4. Hirsch, J., Al-Samman, T.: Superior light metals by texture engineering: optimized aluminum and magnesium alloys for automotive applications. Acta Mater. **61**(3), 818–843 (2013)
5. Saito, Y., et al.: Ultra-fine grained bulk aluminum produced by accumulative roll-bonding (ARB) process. Scr. Mater. **39**(9), 1221–1227 (1998)
6. Vasudevan, A.K., Doherty, R.D.: Aluminum Alloys—Contemporary Research and Applications: Contemporary Research and Applications. Elsevier, Amsterdam (2012)