Available online at www.sciencedirect.com

ScienceDirect

Procedia Structural Integrity 7 (2017) 268–274

Structural Integrity Procedia

www.elsevier.com/locate/procedia

3rd International Symposium on Fatigue Design and Material Defects, FDMD 2017, 19-22 September 2017, Lecco, Italy

In-situ investigations of structural changes during cyclic loading by high resolution reciprocal space mapping

Annika M. Diederichs[a*], Felix Thiel[b], Ulrich Lienert[c], Wolfgang Pantleon[a]

[a]Department of Mechanical Engineering, Section of Materials and Surface Engineering, Technical University of Denmark, 2800 Kgs. Lyngby, Denmark
[b]Institute for Metallic Materials, Leibniz Institute for Solid State and Materials Research, 01690 Dresden, Germany
[c]DESY Photon Science, Deutsches Elektronen Synchrotron, 22607 Hamburg, Germany

Abstract

A major failure reason for structural materials is fatigue-related damage due to repeatedly changing mechanical loads. During cyclic loading dislocations self-organize into characteristic ordered structures, which play a decisive role for the materials lifetime. These heterogeneous dislocation structures can be identified using advanced electron microscopy and synchrotron techniques. A detailed characterization of the microstructure during cyclic loading by in-situ monitoring the internal structure within individual grains with high energy x-rays can help to understand and predict the materials behavior during cyclic deformation and to improve the material design. While monitoring macroscopic stress and strain during cyclic loading, reciprocal space maps of diffraction peaks from single grains are obtained with high resolution. High Resolution Reciprocal Space Mapping was applied successfully in-situ during cyclic deformation of macroscopic aluminium samples at the Advanced Photon Source to reveal the structural reorganization within single grains embedded in the bulk material during fatigue.

Keywords: cyclic deformation; fatigue; in-situ x-ray diffraction; reciprocal space mapping; synchroton radiation; aluminium

1. Introduction

The majority of metallic components fail as a consequence of periodically varying stresses causing structural changes in the material, which result in cracks and fracture after a sufficient number of cycles. During mechanical loading of metals, plastic deformation occurs on the microscale by motion of dislocations causing a fraction of dislocations to be stored in the material. Characteristic low-energy dislocation structures develop during cyclic deformation in face-centered cubic metals and consist of dislocation-rich walls and dislocation-free subgrains (Mughrabi et al. 1983). These structures have been extensively studied in copper, while the corresponding microstructural changes in aluminium are less frequently reported. Grosskreutz et al. (1963) and later Madhoun et al. (2003) analysed the reorganization of dislocations in aluminium into 1 to 5 µm large cells during cycling deformation by means of transmission electron microscopy. The details of the progressing self-organization of dislocations into

* Corresponding author. Tel.: +45 45 25 47 38. Fax +45 45 25 19 61.
E-mail address: anmad@mek.dtu.dk

10.1016/j.prostr.2017.11.088

subgrains, however, remain unknown, because it is not possible to study the substructure of grains in the bulk of a relevant sized polycrystal during ongoing deformation by means of electron microscopy.

Such information can be provided by High Resolution Reciprocal Space Mapping (HRRSM). The synchrotron technique (Jakobsen et al. 2006, 2007) enables to follow the microstructure of individual grains embedded within a polycrystalline bulk sample in-situ during deformation by obtaining three-dimensional reciprocal space maps with high resolution ($\Delta q/q = 10^{-4}$), while other techniques such as electron microscopy or conventional x-ray diffraction are either destructive or obtain an average over a number of grains with various orientations. Utilizing a custom-made load frame, the evolution of the subgrains and the associated internal stresses in individual grains of commercially pure, polycrystalline aluminium can be monitored in-situ during cyclic deformation. In this manner, the evolution of substructure can be related in an unprecedented way to the mechanical loading regime experienced by the sample.

2. Experimental investigation

2.1. Material

Tensile test specimens were manufactured from an AA1050 sheet cold-rolled to 90% thickness reduction to a final thickness of 1 mm. Dog bone-shaped specimens with a gauge section of 15 mm in length and 5 mm in width were designed to fit to a custom-made screw-driven load frame. Sample cutting was done by spark cutting and tensile specimens were then annealed at 600 °C for 2 h to ensure complete and homogeneous recrystallization. The microstructure after annealing was investigated metallographically. Using both, light optical microscopy and scanning electron microscopy, grain sizes were estimated to be between 30 µm and 100 µm and homogeneous throughout the entire cross section of the gauge.

2.2. Pre-deformation

Prior to the in-situ investigations by HRRSM, cyclic pre-deformation was carried out in order to introduce a microstructure conform to cyclic deformation in the specimen using an MTS Acumen 3 kN Electrodynamic Test System equipped with Station Manager MTS FlexTest 40 and pneumatic grips. The investigated sample was initially deformed by 1% in tension with a grip speed of 0.015 mm/s and then cycled at a rate of 0.5 Hz under displacement control with a displacement amplitude of 10 µm corresponding to an engineering strain amplitude $\hat{\varepsilon}$ of $6.7\cdot10^{-4}$. 18000 tension-tension cycles were performed with the maximal displacement achieved after 1% tension.

2.3. Experimental set-up at synchrotron facility

For the synchrotron investigations, the sample was equipped with a pre-wired strain gauge Omega KFG-3 350 Ω at the center of the gauge section and aligned with the tension axis to monitor the axial strain in-situ. The sample is mounted in a custom-made screw-driven load frame equipped with a 5 kN load cell as presented in Figure 1a.

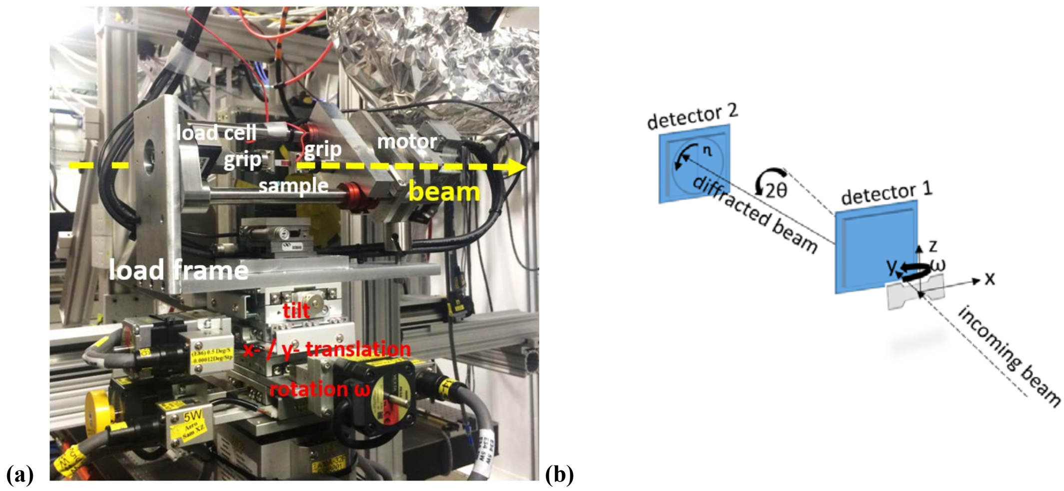

Fig. 1. (a) *Load frame used for High Resolution Reciprocal Space Mapping in-situ during mechanical loading at APS, 1-ID-E. The sample equipped with a strain gauge is positioned in the center of rotation on top of several translation and rotation stages for alignment und acquisition.* (b) *Sketch of the diffraction geometry and the position of the detectors used at APS, 1-ID-E.*