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Cyclic deformation response of ultrafine pure Al

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Abstract

The cyclic deformation response of ultrafine pure Al produced by cryo-rolling was investigated using low-cycle fatigue and electron microscopy. The ultrafine pure Al was cyclically deformed under a fully reversed total strain control ($R = -1$) corresponding to a plastic strain amplitude range of 8×10^{-4} and 1.4×10^{-2}. It was found that cryo-rolled pure Al is very susceptible to cyclic softening, resulting in a much lower fatigue life compared with the coarse-grained counterpart. Shear banding and associated grain coarsening were the most prominent microstructural features of the cyclically deformed cryo-rolled pure Al. Furthermore, annealing led to a slight decrease in the cyclic softening rate, giving a slight increase in the fatigue life of cryo-rolled Al.

Keywords: Ultrafine grained microstructure; Cryo-rolling; Fatigue; Low-cycle fatigue; Shear bands

1. Introduction

Ultrafine-grained (UFG)/nanocrystalline (NC) materials are a new generation of potential structural materials. Among these materials, bulk UFG metals and alloys are increasingly attracting attention due to their extraordinary properties such as exceptionally high strength and hardness compared with their coarse-grained (CG) counterparts. However, most of the work on UFG materials has focused on their monotonic deformation behaviour, while their cyclic deformation has received much less attention. This represents a major gap in our ability to develop applications for these materials as knowledge of the fatigue properties of UFG/NC materials is essential to assess their usefulness in structural components.

In general, the limited studies of the effect of grain refinement on the fatigue life of UFG/NC materials have shown an increased fatigue life (in comparison with their CG counterparts) in the high-cycle fatigue (HCF) regime, where the applied strains are in the elastic range, whereas in the low-cycle fatigue (LCF) regime, where the applied strain are in the plastic range, refinement of the microstructure does not appear to improve the fatigue life [1–5]. This effect of grain size is somewhat expected when considering the effect of the higher strength of the UFG/NC materials in the elastic region and their lower ductility in the plastic region [3,6].

The increased endurance limit and higher fatigue life in the HCF regimes indicates a higher resistance of UFG materials to crack initiation. In this regard, the study of the notch-sensitivity of UFG Cu produced by equal-channel angular pressing (ECAP) showed a higher notch-sensitivity compared with its CG counterpart [7]. In addition, it was shown that the microstructure remains stable except for the plastic zone in the vicinity of the crack tip in which clearly elongated grains were detected. In regard to fatigue crack growth, the results of different studies on different UFG materials produced by ECAP revealed a reduced fatigue crack threshold and an enhanced fatigue crack growth rate [8–12]. This behaviour was mainly attributed to the lower tortuosity of the crack path and reduced crack closure due to grain refinement [8,12].

In the low-cycle fatigue region, the deterioration of fatigue life has been reported to be due to microstructural instabilities. These microstructural instabilities are mostly

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observed in the form of shear banding and grain coarsening [2,3,13–19]. To what extent these two phenomena (shear banding and grain coarsening) are contributing to cyclic softening is still not clear, but shear banding has been reported in both LCF and HCF regardless of cyclic softening or cyclic stability [1,2,18,20,21], while grain coarsening has been only observed in LCF [2,3,13,22]. On the other hand, in contrast to the detailed grain coarsening observations, the microstructural evidence of shear banding has not been systematically documented in the literature with indirect indications of shear band formation, such as surface banding, and being relied upon. The first microstructural evidence of shear banding was reported in studies on fatigued high-purity UFG Cu with a grain size of 300 nm [19], and commercial purity UFG Al with a grain size of 800 nm [18]. The width of shear bands reported in the above-mentioned studies was relatively coarse and the bands contained several grains, slightly rotated compared with the adjacent area. In contrast, Wong and co-workers observed micro-shear bands composed of tangled dislocations with a thickness much lower than the grain size. According to the model proposed by Höppel [19], grain coarsening and shear banding are correlated phenomena which occur concurrently. However, there is as yet insufficient microstructural evidence to verify their proposed model.

It is interesting to note that most studies to date have used UFG materials produced by ECAP, whereas there is a broad range of severe plastic deformation (SPD) processes, including accumulative roll bonding, high-pressure torsion and cryo-rolling, which are expected to have different microstructural characteristics which may then lead to other cyclic deformation processes, especially softening. Therefore, in the current work an ultrafine pure Al produced by cryo-rolling was utilized to study the cyclic deformation response of a single-phase ultrafine material. The microstructural characteristics of UFG materials produced by cryo-rolling is different from the ECAPed materials in terms of grain boundary energy, misorientation and stability. Therefore, the aim of the present work is to use an ultrafine, commercially pure Al as a model material to understand fatigue response and damage mechanisms of ultrafine microstructures produced by cryo-rolling, with and without subsequent annealing.

2. Experimental procedure

A commercial-purity Al ingot with the chemical composition given in Table 1 was cut into 10 mm × 42 mm × 50 mm blocks. The blocks were homogenized at 530 °C for 1 h before cryo-rolling.

Table 1
Chemical composition of commercial-purity Al (in wt.%).

Al	Fe	Si	Mg	Zn	Other
99.7	0.11	0.064	0.024	0.01	0.09

Following homogenization, the solutionized blocks were dipped in liquid nitrogen for 15 min and then rolled from 10 mm to ~2 mm thickness. To prevent cracking, the total thickness reduction (~80%) was achieved in about 20 passes with 0.4 mm reduction in each pass. After each pass, the plates were immersed in liquid nitrogen for over 2 min before further reduction. To study the effect of annealing on the fatigue properties of our cryo-rolled (CR) samples, a group of rolled sheets were subjected to annealing heat treatment at 275 °C for 15 min [23]. The aim of heat treatment was to recover the heavily deformed microstructure to obtain sharper grain boundaries and lower dislocation density inside the cells/subgrains.

The microstructure was characterized before and after deformation using electron microscopy. Transmission electron microscopy (TEM) observations were carried out using a JEOL JEM-2100 microscope operated at 200 kV. The TEM samples were sectioned parallel to the rolling direction and the foils were cut from both the deformed area (close to the fracture region and far enough from the shoulder) and undeformed shoulder region. Then the samples were carefully mechanically polished to a thickness of about 70 μm followed by low-energy ion-beam milling using a Gatan 691 PIPs machine. Fractography and surface relief observations were performed by scanning electron microscopy (SEM) using Leica S440 and Leo 1530 microscopes.

The geometry of the fatigue samples used in this study is shown in Fig. 1. The axes of the flat dog-bone-shaped samples were parallel to the rolling direction. The sample surfaces were polished to a 1 μm finish before electropolishing. Electropolishing was performed in a solution of 20% nitric acid and 80% methanol at −30 °C and a voltage of 13 V.

The LCF tests were conducted on a MTS 25 kN servo-hydraulic load frame in accordance with ASTM E606-92 [24]. Fatigue tests were conducted under a fully reversed constant total strain amplitude ($R = -1$), using a frequency of 0.5 Hz and a triangular waveform. The plastic strain amplitude was calculated from the width of the hysteresis loop closest to the midlife [25]. The total strain range considered in this study was chosen such that the plastic strain amplitude range ($\Delta\varepsilon_p$) applied to the samples was between 8×10^{-4} and 1.4×10^{-2}. Failure was determined by a drop in the tensile load of 10% from the maximum tensile stress. Anti-buckling guides were used to prevent buckling under

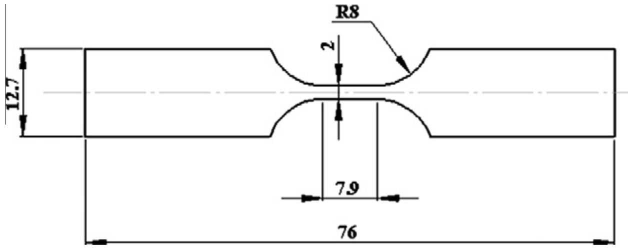

Fig. 1. Schematic of the fatigue samples used in this study (dimensions in mm).