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Fatigue crack growth anisotropy in ultrafine-grained iron

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ABSTRACT

Nanocrystalline and ultrafine-grained (UFG) metals produced by severe plastic deformation exhibit often microstructures with elongated grains, which result in orientation dependent mechanical properties. This anisotropy is especially pronounced for the resistance against quasi-static and cyclic crack growth. In order to gain more knowledge about the consequences of anisotropic microstructures in the case of cyclic loading, fatigue crack growth (FCG) experiments were performed on UFG iron processed by high pressure torsion, with a mean grain size of 500 × 400 × 150 nm^3. Samples with four different orientations were prepared and tested with two mean stresses to account for crack closure effects. The FCG rate varies by one order of magnitude between cracks propagating parallel to elongated grains and cracks advancing perpendicular to it. This larger difference is discussed in the light of intrinsic and extrinsic toughening mechanisms. It is concluded that crack closure contributions are reduced in UFG Fe, however, geometric shielding due to more frequently occurring crack branching leads to a significantly higher FCG resistance for cracks perpendicular to the grain elongation. Furthermore, it is observed that grain refinement leads to a transition from transgranular to intergranular fracture. However, it can be shown that this intergranular crack growth of UFG iron under cyclic loading is not the result of grain boundary embrittlement, but occurs due to a blunting and re-sharpening process along the grain boundaries.

1. Introduction

Ultrafine-grained (UFG) and nanocrystalline (NC) metals, i.e. metals with grain sizes below 1 and 100 nm respectively, show various improved mechanical properties compared to microcrystalline (MC) counterparts and have been the topic of a large number of studies in recent years [1–4]. Besides electrodeposition, mechanical alloying and gas-phase condensation, severe plastic deformation (SPD) can be applied to obtain NC and UFG metals. With SPD methods the grain refinement is achieved by the introduction of large plastic strains, which reduces the grain size until an equilibrium between grain fragmentation and restoration processes is reached. Metals processed by SPD methods have in common that in many cases the developing microstructure exhibits elongated grains [5–7] which results in anisotropic mechanical properties [8–12]. This is especially the case for a number of continuous SPD techniques which are able to produce larger volumes of materials and thus are of great interest for industry, as for example accumulative roll bonding (ARB), continuous confined strip shearing (C2S2) and the equal channel angular pressing (ECAP) conform method [13–15]. With other methods, such as high pressure torsion (HPT) and ECAP, the processed material volumes are small, however, comparable features evolve [5–7]. Smaller-sized samples make it possible to conduct a larger number of fundamental studies on these materials by changing process parameters, like the applied shear strain, strain rate and deformation temperature. Especially with HPT a large variety of materials can be processed due to the high hydrostatic pressure, which allows investigations even on high strength and brittle metals. However, elongated grains are also characteristic for NC metals produced by other techniques, like electrodeposition.

Anisotropic microstructures lead to orientation dependent mechanical properties, especially concerning the resistance against static and cyclic crack growth, which is well known from bio materials [16]. In principle, the anisotropy of the mechanical properties is not a drawback and, as in nature, can be used as toughening strategy. For example, fracture toughness was found to be relatively low for cracks introduced parallel to elongated grains [8–10]. However, the existence of this weak crack path leads to a significantly enhanced crack growth resistance for crack

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propagation perpendicular to the elongated grains [8–10]. For example, fracture experiments on HPT deformed UFG ARMCO Fe revealed that the fracture toughness of cracks oriented perpendicular to the shear plane of the HPT process is about 3.5 times higher than the fracture toughness of cracks parallel to it (49.0 MPa $m^{0.5}$ for perpendicular cracks, 14.2 MPa $m^{0.5}$ for parallel cracks) [9]. The same behavior has been found for crack propagation under cyclic loading for UFG Ni produced by HPT, where a significantly higher fatigue crack growth (FCG) resistance was measured for cracks perpendicular to the grain elongation direction, compared to cracks parallel to the elongated grains [12]. In order to improve the understanding of the effect of grain shape on the FCG resistance in UFG and NC metals, FCG tests were performed on HPT deformed UFG iron as a representative of body-centered cubic structures, with various different sample orientations. The experimental results are discussed with focus on intrinsic and extrinsic toughening mechanisms and the role of crack closure and crack tip shielding.

2. Material and experimental methods

In this study ARMCO-iron with the composition given in Table 1 was used as a model material for bcc metals. Discs with 30 mm diameter and 7.5 mm height were subjected to HPT at room temperature at a nominal pressure of 2.8 GPa for 10 rotations, which yields a von Mises strain of ε_{vM} ~ 70 at a radius of 15 mm. The grain size of the as-received and HPT deformed microstructure was determined by electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM), see Fig. 1. The as-received microcrystalline Fe exhibits equiaxed grains with a mean grain size of ~15 µm (Fig. 1(a)). As can be seen in Fig. 1(b), HPT deformation results in a microstructure with grains elongated in the shear direction of the process, with the shortest grain length parallel to the axial direction. The elongated grains are furthermore slightly tilted to the shear plane by about 20°. The grain dimensions are approximately 500 × 400 × 150 nm^3, or about 310 nm in diameter for globular grains with an equivalent volume, i.e. microstructure consists of pancake-shaped grains. EBSD images of UFG Fe with different viewing directions are projected on a cube in Fig. 1(c) to provide a better visualization of the microstructure. The substantial grain-refinement was accompanied by a hardness increase from 95 HV in the as-received state to 410 HV in the HPT-deformed state (see Table 1), which was measured between a radius of 2 and 15 mm of the HPT disc. Yield strengths were estimated with the Tabor rule ($\sigma_y \approx 3 * H * 1\ MPa/HV$) [17], which are in good agreement with the measured strength of a similarly HPT deformed pure Fe [9].

Compact-tension specimens were produced from the HPT deformed material, with W = 5.4 mm, B = 1.3 mm and a = 1.0–1.3 mm. The notches were introduced by a diamond wire saw and further sharpened to a radius of 5–30 µm by razor blade grinding. Cyclic compression loading (R = 10) with a resonance testing machine (Rumul Russenberger + Müller, Switzerland) was used to create short and open fatigue pre-cracks, which allow to start the experiments with crack-closure free conditions [18]. For the analysis of the FCG anisotropy, samples were taken with three different orientations in respect to the HPT process. The nomenclature of the specimens was chosen according to the standard E399 [19], with the first letter indicating the normal direction of the crack plane and the second letter indicating the direction of the crack growth. The directions axial (A), radial (R) and tangential (T) refer to the geometry of the HPT deformed disc and are depicted in Fig. 1(d). As shown in Fig. 1(d), four different sample orientations (T-A, R-A, T-R and A-T) were prepared and tested.

An electrodynamic testing instrument, Instron ElectroPuls™, with a 250 N load cell was used for the cyclic loading of the samples. The specimens were tested with a sinusoidal force and a frequency of 45 Hz at load ratios of R = 0.1 and R = 0.7. The potential drop technique (PDT) was used to measure the crack length in-situ during the FCG test. More details about the PDT can be found elsewhere [12]. The effective threshold stress intensity factor range $\Delta K_{th,eff}$ was determined with the load rising method [18], where the experiments were started at a low ΔK (1.5 MPa $m^{0.5}$) and step-wise increased (0.2 MPa $m^{0.5}$ increment size) until the first crack growth is detected. $\Delta K_{th,eff}$ lies between this and the previous ΔK level. If the crack growth decelerates and stops, which means that the material shows cyclic R-curve behavior and crack closure or other shielding mechanisms have built up, the load is increased for a further increment of 0.2 MPa $m^{0.5}$. When ΔK is equal or larger than the threshold stress intensity factor range of a long crack ΔK_{th}, the crack propagates without stopping until failure.[1] Following the standard E647 [20], small partitions of the crack length and cycle number data were repeatedly fitted by a polynomial function of second order and the derivative calculated to get the FCG rate da/dN. The tests were stopped before the samples failed by overload fracture. The final crack length was measured optically and the value compared to the crack length determined from the PDT. Finally, the samples were cyclically loaded until failure.

After the FCG tests images of the samples were examined with a field emission gun SEM "LEO Gemini 1525". The crack path was inspected on the side faces of the specimens at different crack lengths and hence at different ΔK values and quantitative roughness parameters were calculated from SEM images. The true and the projected length was measured from these profiles and the linear roughness parameter determined by $R_L = (true\ length)/(projected\ length)$. Furthermore the maximum roughness amplitude of the profile was measured and the arithmetic average of the absolute values R_a calculated. The crack profiles were subdivided into 100 nm long segments and the deflection angles from the straight crack path measured.

2.1. Validity of FCG data

Due to the limited size of HPT discs the CT samples used in this study are smaller than the minimum recommendations of ASTM E647 [20], with W = 5.4 mm. Therefore additional care has to be taken that linear elastic fracture mechanics (LEFM) is applicable and that plane strain conditions prevail to allow a comparison of FCG data. Hence, data can only be taken as valid when the maximum size of the plastic zone is small compared to the uncracked ligament and the thickness of the samples. The size of the monotonic plastic zone was calculated for plane strain conditions with [21]:

Table 1
Material properties of MC and UFG Fe, including the grain size, d_m, hardness H and yield strength σ_y.

	MC Fe	UFG Fe
Production route	as-received	High pressure torsion
dm	15 µm	500 × 400 × 150 nm^3
H	95 HV	410 HV
σ_y*	285 MPa	1230 MPa
Composition	0.009 wt% C, 0.060 wt% Mn, 0.009 wt% P, 0.007 wt% S, balance Fe	

[1] The quantities can also be named as the short-crack threshold $\Delta K_{th,eff}$ and the long-crack threshold ΔK_{th}. With the load shedding procedure only the long-crack threshold can be determined.