



Role of structural parameters of ultra-fine grained Cu for its fatigue and crack growth behaviour

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ABSTRACT

High cycle fatigue (HCF) life time curves and fatigue crack growth rates of bulk ultra-fine grained (UFG) copper deformed by high pressure torsion (HPT) were determined. Cu of two different purities as well as a bimodally structured HPT Cu were investigated. The results show increased HCF properties of the UFG materials compared to coarse grained (CG) Cu. Especially HPT Cu with lower purity shows enhanced fatigue resistance due to higher microstructural stability. Contrary, crack growth rates in HPT Cu are increased. In case of the high purity Cu, cyclic deformation induced coarsening of the UFG microstructure nearby the crack is found at threshold crack growth rates leading to a retardation of the fatigue crack propagation. Within these coarse grains typical fatigue surface slip marks as observed in CG Cu are found.

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1. Introduction

Using high pressure torsion (HPT), disc-shaped specimens are deformed by shear under high hydrostatic pressure [1] and large degrees of deformation can be achieved leading to bulk ultra-fine grained or even nanostructured materials [2,3]. Literature on the fatigue crack growth behaviour of ultra-fine grained materials provided by Equal Channel Angular Pressing (ECAP) [4] and Accumulative Roll Bonding (ARB) [5] reports enhanced crack growth rates when compared to coarse grained (CG) materials due to a straight crack path and less roughness induced crack closure effects [6]. The aim of the work presented here was to investigate the fatigue crack growth behaviour of HPT deformed Cu in the threshold range and microstructural evolution during fatigue loading.

2. Experimental

2.1. Materials

Disc-shaped specimens (diameter 10 mm, height 0.8 mm) of annealed high purity (99.99%) and commercial purity (99.9%) Cu were HPT deformed by two rotations leading to a shear strain of 42 at a radius of 2 mm [7]. The resulting microstructure and grain size depend on the purity, the same HPT deformation leads to a smaller grain size in case of the commercial purity Cu (150–200 nm compared to 300 nm). Furthermore, a bimodally structured high purity HPT Cu was investigated. A slight annealing heat treatment (150 °C for 3 min in an oil bath) leads to

a microstructure consisting of grains with sizes of several microns (~40% of the volume fraction) embedded in a still ultra-fine grained (UFG) matrix.

2.2. Tensile tests

Tensile tests were conducted on miniaturized samples with a parallel gauge length of 2.5 mm and a cross-section of 0.7 mm × 0.7 mm at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ (Fig. 1).

It can be seen that HPT deformation leads to a strong increase in the ultimate tensile strength (UTS) of the material. Furthermore, the resulting strength depends very much on the grain size, meaning that the finer grain size of the commercial purity HPT Cu leads to a higher strength. The tensile tests also show a decreased ductility after HPT deformation indicated by a lower uniform tensile elongation. Some ductility of the high purity HPT Cu can be regained through the heat treatment, although at the cost of UTS. The bimodally structured Cu shows an increase in uniform tensile elongation by 12% when compared to the ultra-fine grained Cu but a decrease of tensile strength by approximately 100 MPa.

The grain sizes and mechanical properties of the investigated materials are summarized in Table 1.

2.3. Ultrasonic resonance fatigue testing system

To investigate the fatigue crack growth behaviour of the small-sized HPT specimens, a set-up previously used for measuring thin foils was adopted [9]. Disc-shaped HPT specimens with a diameter of 10 mm were polished down to a thickness of 200 μm and an elliptical through notch (1.5 μm × 0.5 μm) was introduced in the centre of the specimen. The specimens were electrolytically polished and glued onto a holder over a slot to ensure free standing of the measurement area, see Fig. 2. The holder as part of an ultrasonic resonance fatigue testing system is subjected to longitudinal push-pull vibrations with zero mean strain (i.e. $R = -1$) at a frequency of 20 kHz. The high frequency used makes it possible to investigate fatigue crack growth rates down to 10^{-12} m/cycle . The crack length was measured using a travelling light microscope with a resolution of approximately 2 μm. Additionally, compressed air was applied for cooling. During each measurement the strain of the

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Table 1
Grain sizes and mechanical properties of the investigated materials.

Material	Grain size	Tensile strength [MPa]	Uniform tensile elongation	Fatigue strength (10 ⁹ cycles) [MPa]
1 HPT Cu 99.9%	150–200 nm	580	0.03	120
2 HPT Cu 99.99%	300 nm	410	0.02	65
3 HPT Cu 99.99%–150 °C/3 min	300 nm/1–5 μm	310	0.14	65
4 CG Cu annealed	40 μm	200	0.44	80

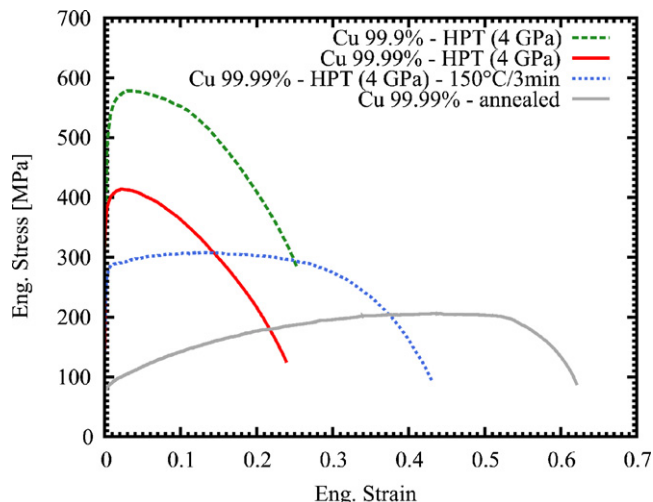


Fig. 1. Engineering stress–strain curves of the investigated materials.

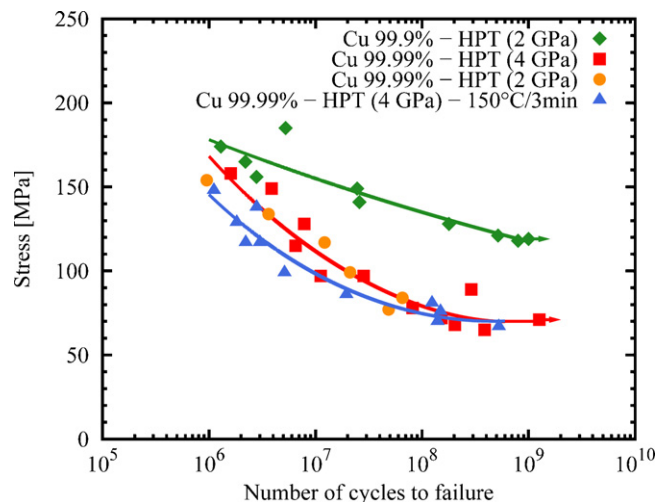


Fig. 3. High cycle fatigue life time curves for HPT Cu.

holder was monitored using a strain gauge. The strain of the sample was determined by calibration with strain gauges on a notchless sample as well as on the holder. This shows an increase of strain by a factor of 1.64 as a consequence of the geometry. The stress amplitude was then calculated from the strain amplitude via Hook's law (Young's modulus for Cu: 120 GPa).

During cyclic loading the crack emerged on both sides of the notch, and crack growth rates were determined in a range of the total crack length $2a$ (including the notch) between 3 and 5 mm. Within the latter limits the microstructure of the HPT specimen can be considered as homogeneous [7]. Fatigue crack growth rates in the threshold regime were determined by the load shedding technique and in addition, constant amplitude tests were conducted. The stress intensity factor ΔK was calculated via Eq. (1) where $\Delta\sigma/2$ is the stress amplitude, a is half of the total crack length and $f(a)$ is a geometrical factor taken from [10].

$$\Delta K = \frac{\Delta\sigma}{2} \sqrt{\pi a} f(a) \quad (1)$$

Fatigue life time curves in the high cycle fatigue (HCF) regime were determined by using a similar set-up and flat dumbbell-shaped specimens as described in [8].

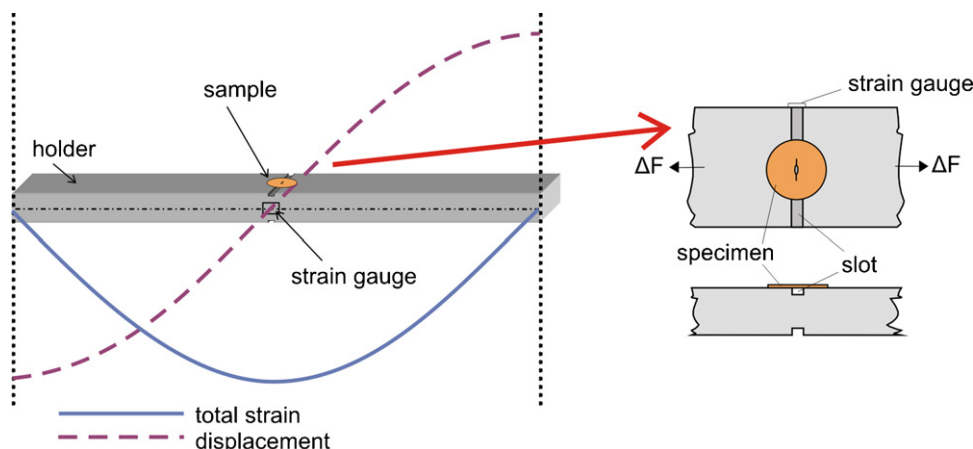


Fig. 2. Experimental set-up for fatigue crack growth measurement of HPT specimens (schematic).

3. Results and discussion

3.1. Fatigue life time curves

When comparing the HCF curves (Fig. 3), commercial purity HPT Cu shows the best fatigue performance which is not only due to the smaller grain size and the higher tensile strength but also due to a better microstructural stability. Grain coarsening was found to decrease the fatigue life time of high purity HPT Cu at high numbers of loading cycles [8]. The high purity bimodally structured HPT Cu shows decreased HCF properties in the range of 10⁶–10⁸ cycles to failure when compared to its not thermally treated counterpart which can be explained by the initially lower tensile strength of the material. At numbers of cycles to failure higher than 10⁸, deformation induced grain coarsening determines the life time and leads to an overlapping of the fatigue life time curves.

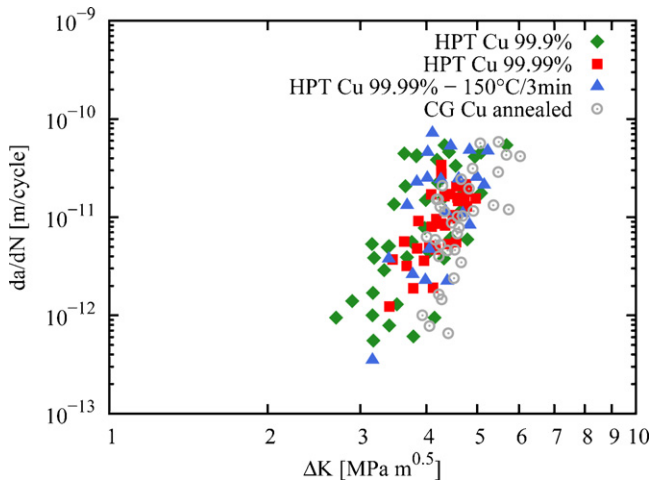


Fig. 4. Fatigue crack growth rate versus stress intensity factor ΔK .

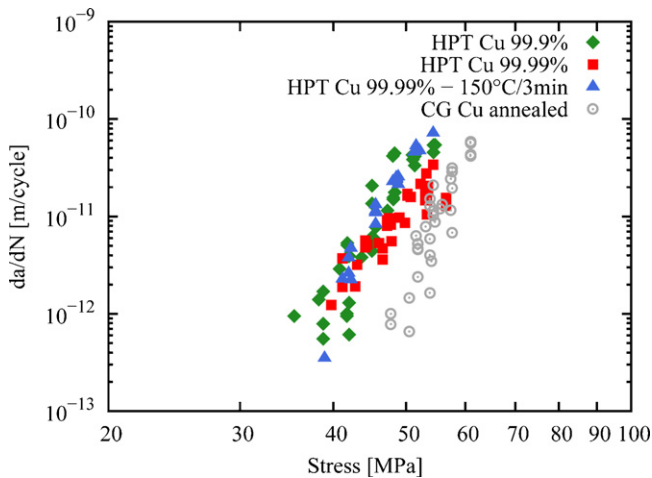


Fig. 5. Fatigue crack growth rate versus stress amplitude.

3.2. Fatigue crack growth threshold

The results of the fatigue crack growth rate measurements are plotted in Figs. 4 and 5, as a function of stress intensity factor and stress amplitude, respectively.

It can be seen from both figures that all different types of HPT Cu show a lower fatigue crack growth threshold than CG Cu. The threshold stress intensity factor for HPT Cu is around 3 MPa \sqrt{m} , which is close to the effective threshold value $\Delta K_{th,eff}$ measured for bulk CG Cu [9]. CG Cu shows a zig-zag crack path and slip marks that cover the surface in a range of 100 μm on both sides of the crack. In contrast, crack paths in HPT Cu are rather straight, see

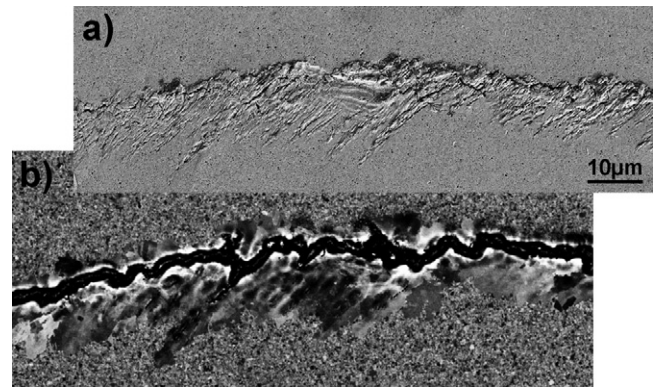


Fig. 7. (a) Surface slip marks in high purity HPT Cu at a stress amplitude of 42 MPa and a crack growth rate of 5×10^{-12} m/cycle and (b) ECC image of the same sample area.

Fig. 6, leading to higher crack growth rates.

This can be related to the reduced effect of roughness induced crack closure in fine grained materials [11]. There can also be seen a tendency for the commercial purity HPT Cu to have the lowest threshold in combination with the straightest crack path (Fig. 6a). Furthermore, the decreased slope of the crack growth curve of high purity HPT Cu in Fig. 5 is an indication of lower crack growth rates in this material. Nevertheless the differences between all tested materials are rather small. Investigations by other researchers (e.g. [4]) on high purity UFG materials show clearly increased crack growth rates in the threshold regime in comparison to CG materials. Contrary to the present study their works were performed under positive load ratios ($R > 0$) where crack closure has more influence than at $R = -1$ [12].

When comparing the fatigue life with the crack growth behaviour of the tested HPT materials, the improved fatigue resistance of commercial purity HPT Cu can be related to a reduced sensitivity of the ultra-fine grained and high strength material to crack initiation, while the same ultra-fine grained structure leads to a decreased resistance to crack growth.

The representation of crack growth rate against stress amplitude in Fig. 5 has been chosen because the large scatter in Fig. 4 raises the question whether the concept of ΔK derived from linear elastic fracture mechanics can be applied to these miniaturized specimens with non-standard geometry.

3.3. Microstructural evolution

High purity HPT Cu showed grain coarsening during fatigue loading in the HCF regime. Therefore the microstructure in the vicinity of the crack was investigated at different constant stress amplitudes. Fig. 7a shows fatigue slip marks near the crack on the surface of a high purity HPT Cu sample. Their lengths reach up to 20 μm which exceeds the original grain size of 300 nm by about

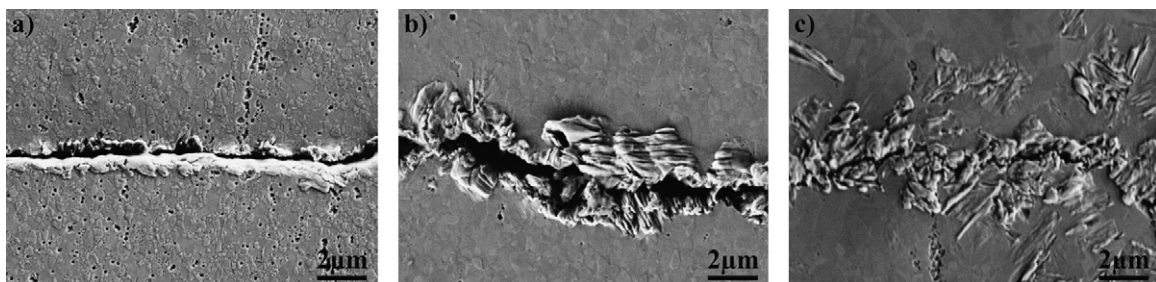


Fig. 6. Fatigue crack paths in HPT deformed Cu at crack growth rates 2×10^{-11} m/cycle: (a) commercial purity, (b) high purity, and (c) high purity bimodally structured Cu.

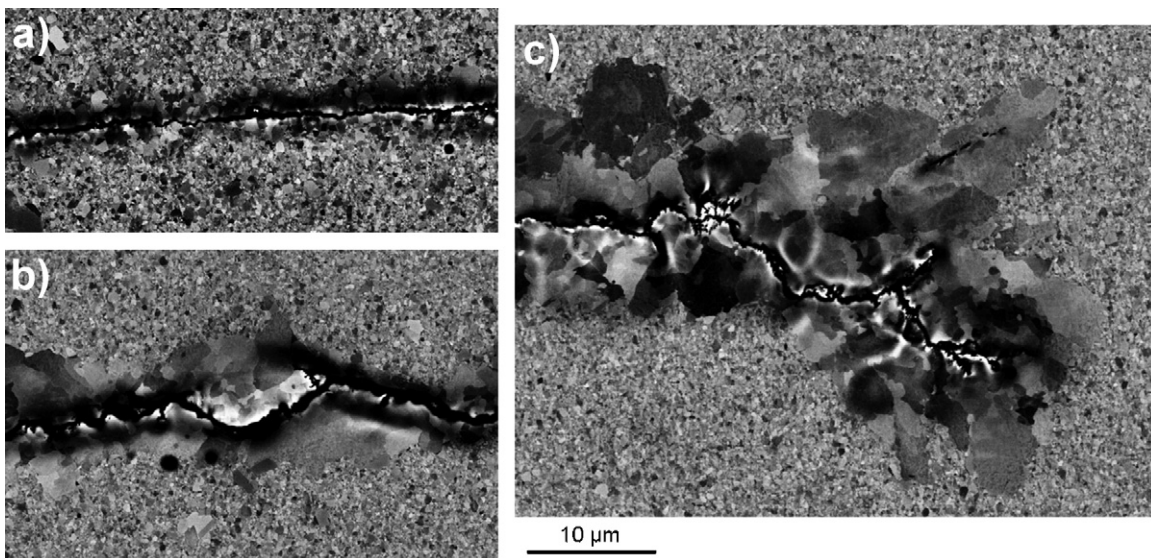


Fig. 8. Microstructure near the crack in high purity HPT Cu at (a) 55 MPa and 4×10^{-11} m/cycle, (b) 49 MPa and 2×10^{-11} m/cycle, and (c) 42 MPa and 7×10^{-12} m/cycle.

two orders of magnitude. Fig. 7b is an electron channelling contrast (ECC) image at the same position after repolishing of the surface. The pictures show that there indeed occurs grain coarsening nearby the crack and that surface slip marks form within these coarsened areas. In the remaining UFG area no surface slip marks can be found.

The area of coarsened grains increases with decreasing stress amplitude and accordingly lower crack growth rate. Fig. 8a–c compares the microstructure nearby the crack at applied stress amplitudes of 55, 49 and 42 MPa, respectively. At the highest stress amplitude the grains in the vicinity of the crack roughly maintain their original size (Fig. 8a) while at the lowest stress amplitude they expand up to a size of several micrometers within a radius of approximately $10 \mu\text{m}$ around the crack tip (Fig. 8c). The coarsening leads to a behaviour contrary to the behaviour of CG materials where the cyclic plastic zone size decreases with decreasing stress amplitude [11]. It should be emphasized that this grain coarsening near the crack occurs only at crack growth rates beneath 10^{-10} m/cycle.

It is known that the strain energy dissipated per unit surface created during cyclic loading is directly proportional to the stress amplitude and inversely proportional to the crack growth rate [13]. In the threshold regime, the crack growth rate decreases faster than the stress amplitude, thus it can be assumed that the required acti-

vation energy for dynamic recrystallization and grain coarsening in the high purity UFG material is provided by an increased dissipated energy during cyclic loading at low stress amplitudes and near threshold crack growth rates.

Applying constant amplitude loading at various stress amplitudes on high purity HPT Cu with a bimodal microstructure, grain coarsening was not observed (Fig. 9). Fatigue slip marks can only be found in the initially larger grains, as can be seen in Fig. 6c. The higher stability and therefore originally fine grain size in this material leads to higher crack growth rates when compared to UFG HPT Cu. This is a hint for a retardation of the fatigue crack propagation through grain coarsening.

4. Conclusions

In the present work the high cycle fatigue behaviour and the near threshold crack growth rates of HPT deformed samples were determined. The materials under investigation were commercial and high purity Cu as well as a bimodally structured high purity HPT Cu. The HPT materials exhibit increased HCF life but also increased crack growth rates indicating that the UFG microstructure shows a better resistance against fatigue crack initiation but a lower resistance against crack propagation compared to CG material. In addition to these general findings, the stability of the UFG structure is of utmost importance. It is increased in the case of commercial purity HPT Cu leading to superior HCF life time. The high purity HPT Cu coarsens during fatigue loading and even during crack propagation at very low crack growth rates. This leads in the first case to an earlier crack initiation and therefore decreased HCF life time, and in the second case to a retardation of the crack growth rate. Although the bimodally structured HPT Cu has a higher microstructural stability than the high purity UFG HPT Cu, an improvement of the fatigue performance did not occur.

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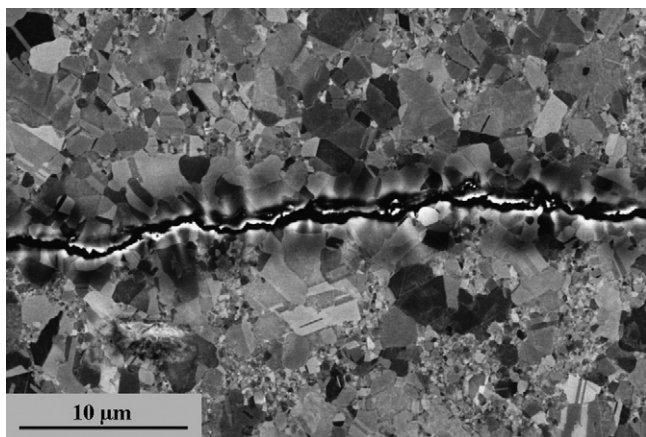


Fig. 9. Microstructure near the crack in case of the bimodally structured high purity HPT Cu (43 MPa and 3×10^{-11} m/cycle).

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