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# Influence of material properties and testing frequency on VHCF and HCF lives of polycrystalline copper

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ABSTRACT

Predicting life times of structures, machines and their parts as well as very small electronic devices for medical purposes is especially difficult for very low stress amplitudes and very high numbers of cycles. The development of the ultrasonic-fatigue method resulted in a reliable testing technique for VHCF loading. Some of its properties are described in this paper. To offer an explanation for the somewhat controversial ultrasonic-fatigue properties reported in the literature, *S-N* tests were performed at ~19 kHz and 20 Hz on polycrystalline copper – a nominally homogenous and ductile material. Moreover, plastic-strain measurements were performed. In addition, micro-structural features and their changes in the high and very-high cycle regimes are reported.

## 1. Introduction

The development of structures, machines and their parts for applications covering several ranges of structural dimensions necessitates a reasonable prediction of their service life and thus the knowledge of their mechanical response to fatigue loading [1–5]. The increase of life times of technical materials becomes more and more important in times of restrictions of natural resources. Basic research on the material response to all kinds of load is needed, as the structure of technical materials is complex and researchers are developing even more complex structures with the aim of improving the material response especially for HCF and VHCF loading (additive manufactured materials [4–6]). These developments cover several ranges of structural dimensions and have led to emphasize the role of a hierarchical treatment of the relevant features [3,7–9]: during cyclic loading, not only the properties of the raw material, but also the subsequent changes of these properties during loading are primarily important, whereby influences from the outside, such as mechanical and thermal loads and environment, have to be considered. The role of the microstructure and the properties of the materials' surfaces on fatigue-crack nucleation has been studied with new imaging techniques by Polák et al. [10] and is summarized by Lukáš [11] and other authors in the ASM Handbook [12]. Earlier research showed that, especially at very low cyclic loads and thus high and very high numbers of cycles, understanding the material response is even more difficult than at higher cyclic loads. Measurements and research in the very-high cycle fatigue (VHCF) range naturally requires extremely long testing times. Hence, from about $10^8$ cycles upwards, the use of a time-saving method is indispensable. Of course, it is necessary to investigate whether the obtained results can be compared with conventional loading conditions and whether or not the results help to assess the realistic cases. As a renowned technique in this field, the ultrasonic-fatigue method has been constantly improved since more than 35 years, so that its reliability and accuracy is at least as high presently as that of fatigue testing equipment working at conventional testing frequencies [15]. Consequently, it has been established as an important technique not only for academic research but also for industrial purposes.

In the following, some features of the ultrasonic technique are described. In the following, some more recent results are reported and compared with measurements at a testing frequency of about 20 Hz. Several different loading conditions such as specimen dimensions and shapes are considered. The results show the capabilities and usefulness of the ultrasonic-fatigue method to characterize the material response in the VHCF-regime, but also the limitations of modeling this response and life-time predictions.

One main aim of this paper is to present the role of microstructural features of a nominally homogenous ductile material

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containing no second-phase particles on fatigue, fatigue crack formation and failure. Polycrystalline copper was used and SEM techniques were applied, which allowed studies from the millimeter-range down to the nanometer-range.

## 2. Material

Three different variants of polycrystalline copper with different purity and production process were studied. A-Cu is an electrolyte 99.98% copper (DIN 1787/17672/1756), B-Cu an electrolytic Cu-ETP (CW004A, DIN E-Cu58/57 (2.0065/60)) and C-Cu is high purity 99.999% copper which was produced with the Bridgeman technique [16,17].

After the heat treatment, the specimens were ground, heat treated and subsequently electrolytically polished again in order to remove eventual residual stresses at the surfaces owing to the machining process. The subsequent heat treatment took place in vacuum. The A-Cu was annealed at 750 °C for 75 min and cooled in air, the B-Cu 680 °C for 25 min and cooled with 300 °C/h in vacuum, the C-Cu 650 °C for 60 min and furnace cooled in vacuum. Aim of the somewhat different heat treatments was to obtain approximately same grain sizes of the three materials. For A-Cu and C-Cu a grain size of ∼60 ± 10 μm resulted and for B-Cu ∼40 ± 10 μm. The mechanical properties and grain sizes are summarized in Table 1.

Different specimen shapes were chosen for the different types of tests (Fig. 1). For the fatigue tests, mostly hourglass shapes with a reduced diameter in their centre were prepared. From A-Cu, rectangular rods with a cross section of $8 \times 8\ \text{mm}^2$ and hour-glass shaped specimens with an inner diameter of 3 mm were machined. B-Cu was machined from 15 mm diameter cold-drawn rods to hourglass shaped specimens with an inner diameter of 3 mm. High-purity copper (C-Cu) specimens were likewise hour-glass shaped, and in addition rods with 7 mm diameter were machined such that two opposite flat areas were obtained (details see [16]). For the thermoelectric measurements, cylindrical rods with a diameter of 5 mm were used [18].

## 3. Experimental procedure

### 3.1. Principles of ultrasonic fatigue

The ultrasonic-fatigue method is a resonance technique based on cyclic movement of inertia-masses: the specimens are attached – usually with screws – to the ultrasonic transducer of an ultrasonic generator. A loading frequency of typically 19 kHz turned out to be the most convenient, as it allows the use of specimen dimensions which are similar to conventional fatigue specimen shapes and, in addition, allow sufficient reduction of the damping heat. Different specimen shapes, either with a constant cross-section or with an hourglass shape are used, depending on the required measurements. For our *S-N* measurements, hourglass shapes were used (Fig. 1a), where the mostly stressed (97%) volume extends about 1.25 mm from the center which has a diameter of 3 mm. The accuracy of the strain amplitude measurement was approximately 2%. Hence, the statistical variation of the stress amplitudes lay between 0.5 and 1 MPa. Sufficient cooling was provided by external fans and the utilization of intermittent loading in the ultrasonic tests: the pulses, usually containing $1.9 \times 10^4 - 3.8 \times 10^4$ cycles, were interrupted periodically by pauses of variable length to ensure that the temperature increase in the specimen center was limited to only a few °C. During the actual *S-N* experiment, on-line frequency measurements allow – after prior calibration – the registration of the temperature in the mostly stressed specimen volume.

Resonance in the ultrasonic tests is obtained by appropriate dimensions and shapes of all the vibrating parts of the load train. When this is the case, a standing wave with maxima of the vibration amplitude at the transducer and specimen ends is generated, and the maxima of strain and stress, which are shifted by 90°, are located at the center of the specimen and the transducer. If the specimen is attached to the transducer with just one end, a mean stress of zero ($R = -1$) is generated. If different mean loads are required, the second end of the specimen is connected to a part of a mechanical or hydraulic machine's frame. In order to attain reliable and accurate data, it is essential to have feedback control. To this end, induction or capacity sensors measure the vibration amplitudes after appropriate calibration, and micro-strain gages serve additionally for control purposes. A vibration gauge serves as feed-back, controlling the vibration with an accuracy of ±1%. The total strain amplitudes are calibrated using a strain gauge in the area of maximum strains. The specimen volume with almost constant stress/strain amplitude is determined by the approximately 3 mm long center part (see Fig. 1a: 97% maximum strain/stress amplitude) of the specimen with a diameter of 3 mm, namely $\sim 21\ \text{mm}^3$. Further details regarding the ultrasonic testing technique can be found in [14,15].

As ultrasound tests are displacement controlled, i.e. total strain controlled, stresses (or loads) cannot be measured directly, but are determined from the measured total strain amplitude $\Delta\varepsilon_{tot}/2 = \Delta\varepsilon_{el}/2 + \Delta\varepsilon_{pl}/2$. If the plastic strain amplitudes are negligible, $\Delta\varepsilon_{tot}/2$ can be used directly to calculate the axial stress amplitude $\Delta\sigma/2$ with Hooke's law: $\Delta\sigma/2 = E \cdot \Delta\varepsilon_{tot}/2$. If this is not the case, $\Delta\varepsilon_{pl}/2$ must be subtracted from $\Delta\varepsilon_{tot}/2$. In order to minimize the damping heat, pulse-pause sequences are followed and, in addition, forced air is used for cooling as described above [18]. Experiments can be performed at different temperatures and in different environments by attaching climate chambers, furnaces, vacuum chambers or environmental chambers containing corrosive or inert liquids or gases. With such equipment, *S-N* curves as well as ($\Delta a$ vs. $\Delta K$)-measurements can be determined.

### 3.2. Measuring procedure at ∼19 kHz ultrasonic-fatigue and at 20 Hz testing frequency

In this paper, measurements of (*S-N*) at ultrasonic frequencies of ∼19 kHz and servo-hydraulic frequencies (∼20 Hz) are reported, applying the principles described in Sections 2 and 3.2.

The *S-N* experiments were started with a stress amplitude of 10 MPa in the fatigue tests. At conventional frequencies using servo-hydraulic equipment frequency measurements were performed under load control. In order to enable a comparison with the ultrasonic measurements, a similar training, i.e. ramp-loading, pre-treatment was chosen (Fig. 2). The specimens were

**Table 1**
Mechanical properties of the three kinds of copper of different purity.

| Material | Purity (wt.%) | *E*-Modul. (GPa) | $R_m$ (MPa) | $R_{p,0.2}$ (MPa) | A | MH (HV) | Grain size (μm) |
|---|---|---|---|---|---|---|---|
| A-Cu | 99.98 | 130 | ∼200 | ∼130 | 30 | 35–70 | 60 |
| B-Cu | 99.990 | 127.7 | ∼271 | ∼203 | ∼12 | ∼87 | 39 |
| C-Cu | 99.999 | 120 | 250 | 140 | 30 | 50 | 60 |