

Bimodal grain size distributions in UFG materials produced by SPD: Their evolution and effect on mechanical properties

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Abstract. The mechanical properties of bulk ultrafine-grained materials produced by severe plastic deformation can be modified (sometimes enhanced) by a mild annealing treatment which leads, in some cases, to a bimodal grain size distribution, characterized by a good combination of strength and ductility. Bimodal grain size distributions can also evolve during cyclic deformation at rather low homologous temperature. Here, the conditions under which bimodal grain size distributions evolve and how they affect the mechanical properties, as studied by the authors and as reported so far in the literature, will be reviewed and discussed.

1. Introduction

Bulk ultrafine-grained (UFG) metals produced by equal channel angular pressing (ECAP), the most widespread severe plastic deformation (SPD) technique, have attracted much attention during recent years because of their extraordinary strength properties [1]. As will be discussed in detail below, *bimodal grain size distributions* in UFG metals, consisting of regions with coarsened grains amidst the original UFG grain structure, have been found repeatedly in bulk ECAP-processed UFG material of copper, nickel and α -brass under different, frequently not well-defined circumstances, for example

- after cyclic deformation at room temperature,
- after long-time anneals at room temperature or short-time anneals at higher temperatures, and
- shortly after ECAP-processing.

The effect of bimodal grain size distributions on the monotonic and the fatigue strength properties has been beneficial in some cases but not in others. The aim of the present work is to present some new results, to clarify some seemingly contradictory findings, to identify in more detail the conditions under which bimodal grain structures evolve and to discuss their effects on the mechanical properties.

2. Occurrence of bimodal grain size distributions, effects on monotonic and fatigue strength

2.1. Evolution of bimodal grain size distributions during strain- and stress-controlled fatigue

In an early fatigue study on UFG copper (99.98%), an interesting observation was made, namely that, during fatigue in strain control at room temperature (RT), a strong cyclic softening occurred. At the same time, it was noted that the originally strongly ECAP-hardened and more or less homogeneous UFG grain structure had undergone a marked coarsening in locally confined regions, whereas the original UFG microstructure was retained in the remaining regions [2]. Similar results were obtained

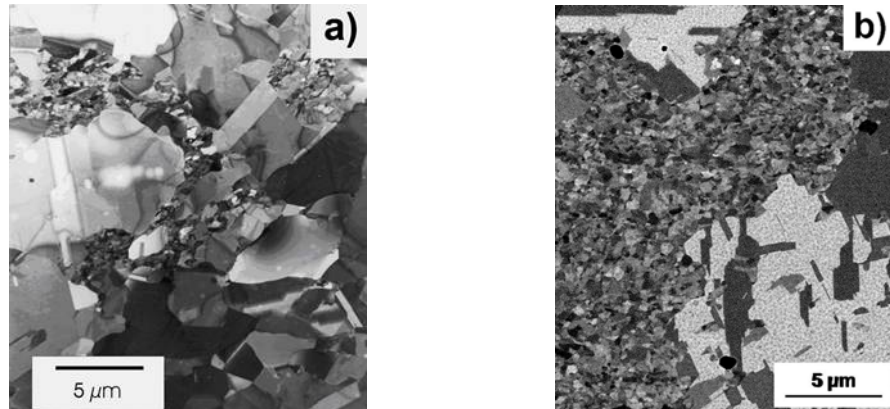


Figure 1. Examples of bimodally coarsened UFG copper. a) TEM micrograph of “optimal” bimodal grain size structure in annealed high-purity (99.99%) UFG copper [3,4]. b) SEM micrograph of bimodal grain size structure in commercial purity (>99.95%) UFG copper, < 3 weeks after ECAP.

on fatigued high-purity (99.99%) UFG copper [3,4] in which clearly *bimodal grain size distributions* were found and, less clearly, on fatigued high purity (99.99%) nickel [5]. It follows that, in the as-ECAP-processed UFG copper, the grain structure is microstructurally unstable at RT, i.e. at a rather low homologous temperature of ca. 0.2. The process of *local* grain coarsening during deformation has been identified as thermally activated temperature- and strain-rate-dependent *dynamic recrystallization* [3,4]. It is remarkable that, in stress-controlled fatigue of high-purity UFG copper, almost no cyclic softening occurred and local grain coarsening was almost negligible [3,4].

Normally, in material of lower *purity*, grain coarsening and cyclic softening would be expected to be impeded. This seemed to be confirmed in work on commercial purity UFG copper [6], however after fatigue in stress control. Only very recently, the same authors found softening and bimodal grain coarsening in strain control. Thus, final conclusions concerning the effect of purity remain vague.

2.2 Introduction of bimodal grain size distributions by annealing to enhance the fatigue properties

Fatigued UFG copper has always been found to have larger fatigue lives, compared to those of conventional grain size (CG) copper in a Wöhler (S-N) plot but not in the Coffin-Manson plot that is typically used for LCF (Low Cycle Fatigue) representations [2,3,4]. Since it is known that good LCF performance requires a sufficiently high ductility [7] and in view of the lowered ductility of the strongly ECAP-hardened UFG material, the rather unsatisfactory LCF fatigue performance is understandable. In an attempt to remedy this deficiency, Höppel et al. [3,4], compare also [8], subjected their UFG copper of high purity (99.99%) to a mild annealing treatment in order to enhance the ductility at the expense of a moderate loss of strength. In a systematic search for the most suitable annealing treatment with respect to enhancing fatigue life, a 2 h anneal at 170°C was found to be *optimal*. In the annealed material, a *bimodal grain size distribution* was found, with coarse grains of some μm grain size embedded in the original UFG grain structure. With increasing annealing time, the volume fraction of coarse grains increased. Figure 1a shows, as an example, a TEM micrograph of the annealed UFG copper with the optimal bimodal grain size distribution. The annealed UFG copper with the bimodal grain size distribution exhibited a remarkable enhancement of fatigue lives by a factor of 7 in the Coffin-Manson plot and an overall excellent fatigue performance, implying that the optimal bimodal grain size distribution provides a good compromise between fatigue strength and ductility.

2.3 Evolution of bimodal grain size distributions during ageing at room or higher temperatures

It is not always possible to obtain bimodal grain structures by annealing. So far, in the case of bulk SPD-processed UFG metals, bimodal grain size distributions have only been encountered or produced in the case of copper, nickel and α-brass, while considerable efforts to introduce a bimodal grain

structure by annealing in commercial purity UFG aluminium failed [8,10], since grain coarsening always occurred in a more or less homogeneous manner. The crucial factor seems to be the competition between recovery and recrystallization [9]. After strong and rapid initial recovery, favoured by high stacking fault energy, not enough driving force is left for subsequent recrystallization. This is probably the case encountered in aluminium.

For both UFG high-purity copper and UFG commercial purity aluminium, grain coarsening was noted after resting at room temperature for some months (up to a year) after ECAP-processing. However, whereas the typical bimodal grain size structure developed in the case of UFG copper, grain coarsening in UFG aluminium occurred more or less homogeneously as noted previously above.

2.4 Evolution of bimodal grain size distributions (shortly) after ECAP

Commercial purity copper, subjected to 8 ECAP passes, route B_c, at rather high ECAP deformation rate (5 mm/s and 10 mm/s) with back pressure [11], and inspected after a rest of some weeks at RT after ECAP-processing, revealed regions with bimodal grain structures. This was most marked in the centre of the billet (cross section 20 mm × 20 mm) and progressed with further RT-resting. An example of the locally coarsened grain structure is shown in Fig. 1b. The coarsening observed probably resulted from an *in situ* anneal due to the deformation heating during (rapid) ECAP-processing, being stronger in the centre than at the periphery because of less heat loss. A simple estimate predicts a temperature rise that is proportional to the ECAP deformation rate (unpublished).

3. Exploitation of bimodal grain size distributions to enhance monotonic and fatigue strength

As already discussed, beneficial effects of bimodal grain size distributions have been noted in some cases. Shortly after the work of Höppel et al. [3,4], Ma and his group succeeded in producing copper with an excellent combination of good monotonic strength and ductility by cold rolling at liquid nitrogen temperature, followed by an annealing treatment which again led to a bimodal grain size distribution [9]. Based on this success, Ma and others have since advocated the advantages of using material with a bimodal grain size distribution for obtaining good strength and ductility.

However, this expectation is not always borne out in practice. For example, in the case of UFG α-brass, a bimodal grain size distribution was introduced successfully by annealing, and although the parameters of the annealing treatment were varied systematically, only a marginal improvement of the fatigue performance was achieved [8,10]. It appears that the success of such annealing treatments depends not only on finding the optimal annealing conditions but also on the material.

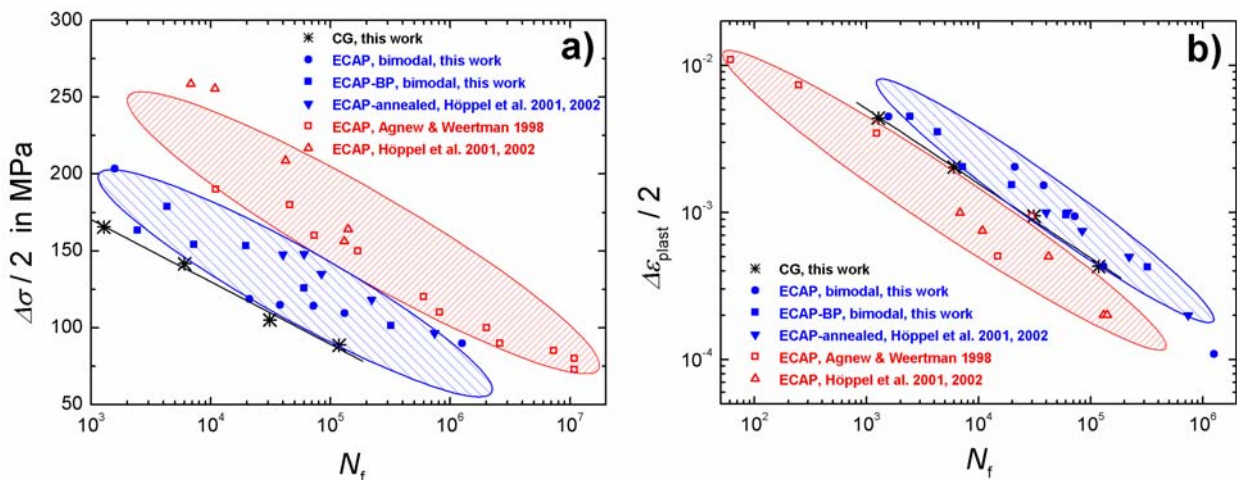


Figure 2. Fatigue life data of commercial purity UFG copper, processed by ECAP without and with back pressure (ECAP-BP). a) Wöhler (S-N) plot, stress amplitude $\Delta\sigma/2$ vs. number of cycles to failure, N_f , showing roughly: $N_{f, \text{CG}} \leq N_{f, \text{bimodal}} \leq N_{f, \text{UFG}}$. b) Coffin-Manson plot, plastic strain amplitude $\Delta\varepsilon_{\text{pl}}/2$ vs. N_f , showing roughly: $N_{f, \text{UFG}} \leq N_{f, \text{CG}} \leq N_{f, \text{bimodal}}$.

In order to assess the effects of ECAP-processing with back pressure [11], commercial purity copper which had been ECAP-processed (12 passes, route B_c) with and without back pressure (BP) was fatigued in strain control. Aroused by an unusually large scatter of the fatigue lives, the authors discovered more or less large regions of a bimodally coarsened grain distribution in the initial UFG grain structure, inherited either from ECAP (see Section 2.4) or from resting at RT after ECAP (Section 2.3). The fatigue life data obtained in this study are displayed in Figures 2a and 2b in Wöhler (S-N) and Coffin-Manson plots, respectively, with some other published data. It should be noted that, while there is a broad scatter band, most ECAP-processed specimens exhibited larger fatigue lives than the CG material, not only in the Wöhler (S-N) but also in the Coffin-Manson plot. This quite remarkable enhancement of fatigue performance is similar to the improvement achieved in the case of high-purity UFG copper by annealing, discussed in Section 2.2. In the present case, the initial bimodally coarsened grain structure of admittedly ill-defined origin obviously had a beneficial effect.

4. Summary and Conclusions

Summarizing and confining ourselves to the well investigated cases, mainly on UFG copper, the following conclusions are drawn with respect to the factors governing the occurrence of coarsening, the evolution of bimodal grain size distributions and their effects on the mechanical properties:

- Some UFG metals (e.g. copper or nickel), exhibit a strong tendency to undergo *local* grain coarsening by *dynamic recrystallization* and to develop *bimodal grain size structures* during mild annealing or mild cyclic deformation at rather low temperature (RT). Other UFG materials, e.g. aluminium, also exhibit grain growth at RT but in a rather uniform manner.
- Bimodal grain size structures can very probably evolve by *in situ* annealing during ECAP, due to the heat production and temperature rise, especially at rather high ECAP deformation rates.
- In strain-controlled fatigue and, to a much smaller extent, in stress-controlled fatigue, bimodal grain size distributions can evolve, accompanied by cyclic softening.
- Ageing for short times at elevated temperatures or at RT for longer periods (months, years) can lead to *local* grain coarsening and formation of bimodal grain size distributions.
- Local grain coarsening is expected to be impeded in less pure UFG material. However, bimodal grain size distributions have been found in commercial and high purity materials. Hence, convincing evidence of such an effect of (im)purity is currently lacking.
- Introduction of bimodal grain size distributions by an annealing treatment can sometimes (not always) enhance the monotonic and fatigue strength properties of UFG materials to different degrees, depending on the annealing conditions, degree of bimodality and the material.
- Tailored enhancement of mechanical properties by annealing in order to introduce bimodal grain size distributions is not always possible and requires “optimal” annealing conditions.

References

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