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Temperature evolution of microstructure of deformed submicrocrystalline cu-zr samples

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Stability of fine-grained microstructure obtained by severe plastic deformation (by ECAP– equal-channel angular pressing) of Cu-Zr samples was investigated by XRD powder diffraction and electron back-scattered diffraction (EBSD). Some of the XRD experiments were performed in-situ in high-temperature chamber and also the time evolution of microstructure was studied.

The addition of zirconium to copper improves the microstructural stability from the region of only slightly above 100 °C to about 400 °C. This also decreases with the number of ECAP passes. With annealing temperature the diffraction profiles became of anomalous shape with a sharp peak and long tails. Simple 2D diffraction patterns indicated the presence of multimodal microstructure leading to wide and narrow components of diffraction profiles. Therefore, bimodal microstructure model was applied for the evaluation and whole measured diffraction patterns were fitted by our own software MSTRUCT [1] as a sum of two Cu phases with different microstructures. This software combines different procedures and algorithms known from the software created by M. Leoni and P. Scardi or by G. Ribárik with some features of MAUD by L. Lutterotti. It appeared that two factors had main influence on the diffraction profiles and, consequently, could be reliably determined from the experiment – microstrain (and/or dislocation density) in the deformed component and the ratio of deformed and recovered fractions.

In temperature dependence, slow decrease of dislocation densities in the whole temperature region was observed while significant drop of deformed fraction appeared above 320 °C. However, this also depends on number of ECAP passes.
Detailed studies of time dependences were performed for Cu-Zr samples at 370 °C. The development of both fractions could be well observed and formally described by the JMAK [2] equation but with extremely low exponent.

Most of measurements were performed with a laboratory vertical diffractometer Panalytical MPD, Cu Kalpha radiation, variable divergence slits and Pixcel 1D detector. In-situ measurements were carried out in the same arrangements with MRI high-temperature chamber with both direct and radiant heating. After partial annealing, selected samples showing still indication of a bimodal microstructure were carefully studied with several laboratory and synchrotron setups. In the latter, powder diffraction patterns were taken with scanning integrating 2D detector in the reflection mode and asymmetric coplanar geometry at mediate energy of 12 keV [3]. High-quality parallel beam synchrotron data lead to the determination of significantly different deformed and recovered fractions with respect to the laboratory data. We assume that this is caused by different grain statistics in the experiments.
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