Restricted moments method to evaluate the microstructure evolution from X-ray peak profile analysis in copper single crystals deformed *in-situ* under very high cycle fatigue

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Many mechanical systems are submitted to stresses repeated for a very large number of cycles during their lifespan and can break under stress amplitudes lower than their ultimate tensile stress (fatigue of materials). Materials fatigue design is thus crucial in engineering and requires the accurate characterization of mechanical behavior of materials under cyclic stress to ensure the safety and reliability of the structures throughout their life service. Mechanical properties in crystalline materials depends on their microstructure, e.g. density of dislocations present. Several techniques, including transmission electron microscopy (TEM), synchrotron x-ray diffraction and neutron diffraction techniques, and electron backscattered diffraction (EBSD), were used to evaluate the dislocation density in plastically deformed materials. However, all these techniques are affected from several limitations in distinguishing the influences of domain size and dislocation density. During the last two decades, Groma et al. developed a new method, the so-called variance method, for the evaluation of the dislocation density and its fluctuation in plastically deformed specimens with the only limitation of the coherent domain size that should be larger than 1 μm. This method is based on the asymptotic behavior of the second- and fourth-order restricted moment in the tail portion of the x-ray diffraction peak.

The present research aims to use the variance method for estimating dislocation density when a very large number ($\sim 10^9$) cyclic loadings is applied on the single crystal with an applied stress significantly lower than the yield stress so that deformation is almost entirely elastic. The monocrystalline copper specimen was loaded using a 20 kHz ultrasonic fatigue machine mounted on the six-circle diffractometer available at the DiffAbs beamline on the SOLEIL synchrotron facility (France). Since we are interested to investigate the very high cycle fatigue domain, the amplitudes of the cyclic stresses range from 7 to 91 MPa. The diffraction patterns were acquired with a 2D hybrid pixel X-ray detector (XPAD S140) which integration time has been synchronized with the fatigue rig. The diffraction data are evaluated by the restricted moments method of the tail portions of individual Bragg peaks, for estimating the microstructure evolution in terms of dislocation density and spatial distribution of the dislocations. The results of these experimental data will be presented and discussed.

Keywords: diffraction, synchrotron, fatigue, microstructure, dislocations