As expected from the extreme deformation involved in ball milling and confirmed by X-ray diffraction (XRD) line profile analysis, powder particles prepared by this process are considered as formed by nanocrystallites. Indeed, in these systems, structural knowledge relies on global information given by XRD because the high strain contrast prevents from good TEM imaging condition. However the recent TEM ACOM method (1) should be able to overcome this difficulty. TEM-ACOM is based on the scanning by a narrow parallel beam combined to the acquisition of the local diffraction patterns. Indexation of the diffraction pattern data provides a map containing phase information and crystallographic orientation on each point of the scanned area. This method gives similar maps as SEM-EBSD with the specific advantage to be reliable even in presence of a high strain level. Therefore information like grain microstructure and local distortion can be reached.

In the present work, TEM-ACOM has been applied to ODS steel powders prepared by ball milling. According to XRD analysis, these materials developed for fuel cladding application have a fine scale microstructure (crystallite size ~ 30 nm). The TEM ACOM experiment was carried out with a parallel beam (probe size 1nm) scanned with a 2.5 nm step on FIB sections taken in ODS steel particles. The data indexation was refined in order to have an angular resolution of ~ 0.2°.

Figure 1 displays the orientation map obtained on an as milled ODS steel particle. The color coding of local orientation directly reveals that the microstructure is formed of long grains (width ~ 50-100 nm length up to several microns). At first sight, the microstructure does not seem made of 30 nm crystallites as indicated by XRD. But misorientation profiles taken across a grain (Fig. 2 and 3) reveal that within the grains, there are domains of about 30 nm separated by thick dislocation walls. These low misorientation domains correspond to the coherent domains measured by XRD line width. Indeed there are no contradictions between the XRD nanoscale domains and the TEM ACOM long grains; only XRD is blind to the local texture between domains.

TEM ACOM appears then as a very appropriate method to analyze complex micro/nanostructures and to provide relevant information. For instance in ODS steel powders, information on local texture as given by TEM ACOM are of significant importance for understanding the evolution under further processing like consolidation and extrusion.

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Fig. 1: TEM-ACOM orientation map of a ball milled ODS particle FIB section. Note that numerous grains exhibit a very high anisotropy (shape factor > 30). The gradual color change within the grains indicates local distortion. The orientation map is overlapped in each point with the indexation reliability value.

Fig. 2: High magnification view of TEM-ACOM map of the as-milled powder. Using ACOM analysis tool, a profile of misorientation showing the local and cumulated distortion is taken in a elongated grain along the line marked in white. The cumulated misorientation corresponds to the global deformation.

Fig. 3: Misorientation profile revealing domains without misorientation (i.e. coherent domains) or with high and uneven misorientation corresponding to dislocation walls or entangled dislocations.