TEM investigation of ODS tungsten materials

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Tungsten-base materials are candidate materials for structural applications in the high temperature region of plasma facing components for future fusion power reactors (DEMO and beyond) [1, 2]. Of promising interest are oxide dispersion stabilised (ODS) tungstenbased materials, which allow improving the mechanical properties of the base material, in particular the creep strength at high temperatures. It is also expected that such materials exhibit improved resistance to radiation damage, as the numerous interfaces between the particles and the matrix may act as sinks for the irradiation-induced defects. In our study we have used two different additional secondary phases to produce ODS W alloys, of either pure yttria or yttrium where the oxide consists in yttria (Y_2O_3) or addition of TiC. Mechanical alloying (MA) is being used to produce mixing of these materials [3]. The aim is to obtain a homogeneous distribution of oxides or carbides inside the tungsten matrix. The obtained materials are compacted by hot isostatic pressing (HIP) at 1350°C and 200MPa for 3hrs and second HIPping at 1450°C and 200MPa for 3hrs. The density of the bulk materials was above 90 % of the theoretical after the second additional HIP. The best density was observed in the W-2Y about 97%.

In order to assess the microstructure in terms of oxide or carbides dispersion, secondary phase precipitation, grain morphology, and ultimately to improve on the production of these ODS tungsten alloys, SEM and TEM observations are performed on the compacted material. However, the high porosity makes the preparation of transmission electron microscopy (TEM) specimens in a classical way unreliable and further microstructural investigation difficult if not impossible. Focused ion beam (FIB) appears then as a major tool in this study, as it allows milling and observing specimens in any region of the material. We have used for FIB/SEM operation a ZEISS Nvision 40 located at PSI. The SEM picture (Fig. 1) is showing the microstructure of W-2Y2O3 and reveals that the grains are nanometres in size. With the help of the lift-out FIB technique, we were able to prepare TEM lamellas that are 10x5 μm^2 and 150 nm (Fig. 2) thick from the fragile, porous and finegrained microstructure of the tungsten-based materials. For TEM we use a JEOL 2010 with a LaB_6 gun operated at 200 kV. However, TEM sample preparation by FIB introduces fine particle desperation in the surface of the thin lamella that will interfere with the radiation damage we would like to study in the bulk of the material. In order to remove the radiation damage induced by the FIB in the surfaces, we have developed a flash electrochemical polishing technique that is applied after the FIB preparation (Fig. 3).

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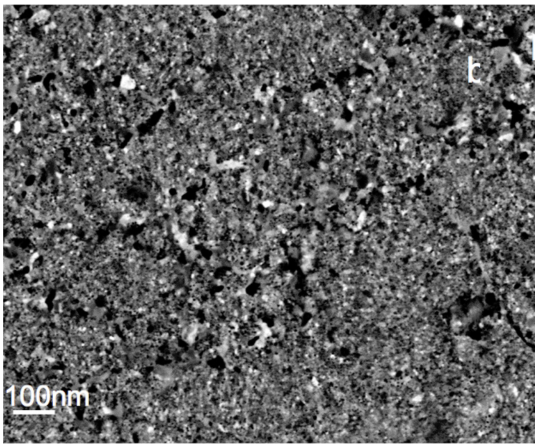

Figure 1. SEM image of nanograin structure of W-2Y2O3 after HIPping under 100 kX (QBSD).

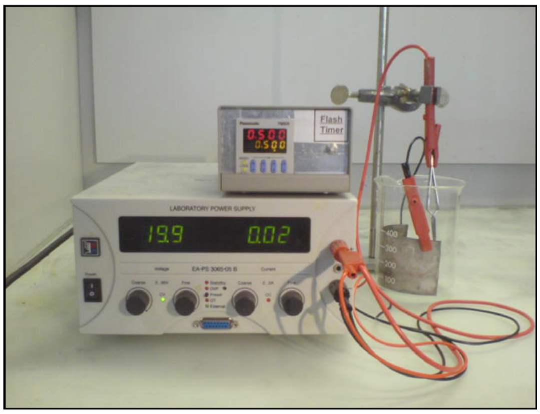

Figure 2. Flash polishing device consisting of flash timer, power supply and NaOH solution in water at room temperature.

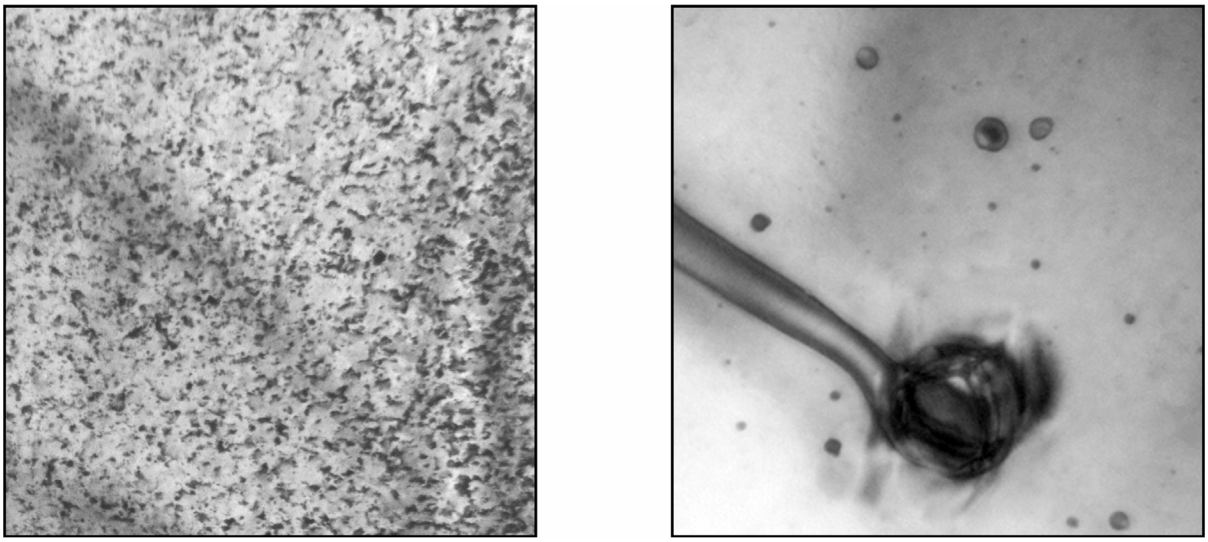

Figure 3. TEM image of (left) W-1Y2O3 thin FIB lamella before flash polishing showing radiation damages from the FIBing and (right) after the flash polishing for 3 seconds.

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