



## PHiTEM

# Platform for High Temperature Materials

### Scope of project

With the establishing of the platform of high temperature materials and multi-scale characterisation of micro-structure and mechanical behaviour of these materials, with a focus on irradiated materials, a unique facility in Switzerland is generated in the field of development and characterisation of advanced materials for advanced future energy power plants.

### Goals and activities

The four experimental devices purchased within the CCEM project, have been installed, tested and became fully operational in July 2007.

The focused ion beam (FIB) has been installed in a controlled area at the PSI. The nano-indenter has been installed in the Hot Laboratory at the PSI. These two devices are now available for characterizing radioactive as well as inactive specimens (see pictures).

At the Empa in Thun a nano-indentation device for a scanning electron microscope (SEM) as well as a dual-beam FIB prototype have been installed (see pictures). Both devices have been tested and are now fully operational.

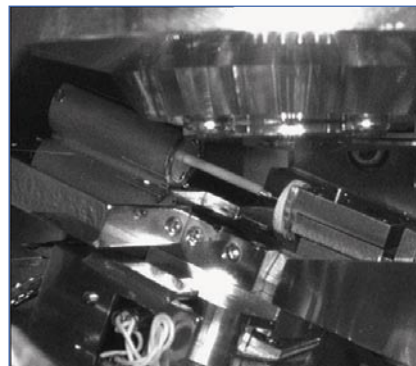
### Main scientific results

Some preliminary results have been obtained on the mechanical behaviour of single crystalline tungsten micropillars, which were fabricated by using a FIB device. The micropillar diameter ranged from 500 nm to 2  $\mu\text{m}$ , while the aspect ratio (height to diameter ratio) was kept constant, equal to 3. All the micropillars had their main axis along the  $\langle 100 \rangle$  direction. The mechanical response of the fabricated micropillars was investigated by means of compression tests, which were carried out using a load-controlled flat punch indenter.

Micropillars with a diameter of 1  $\mu\text{m}$  or 2  $\mu\text{m}$  were investigated in this preliminary study. The load was applied onto the micropillars at a constant rate of 50  $\mu\text{N/s}$ . The maximum load was held constant for a time equal to twice the load-



Above: The FIB device (left) and the nano-indenter (right) at the PSI.



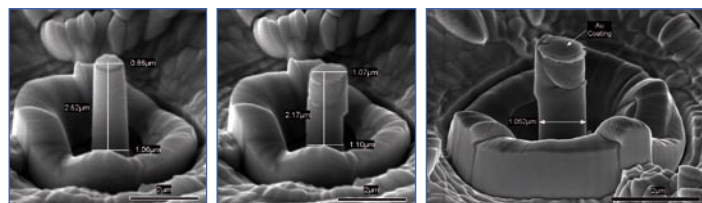
Below: The indenter stage from Hysitron, which was adapted for a SEM (left) and The Orsay Physics ion gun integrated into a Tescan SEM (right) at the Empa Thun.

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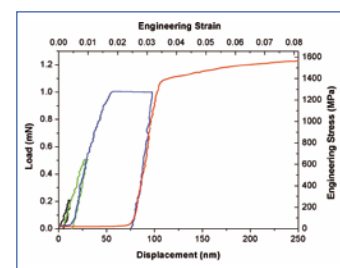
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ing time. The unloading was carried out at a constant rate of 50  $\mu\text{N/s}$ . The micropillars were loaded and unloaded several times, with increasing loads, until the yield point is reached. The applied load ranged from 200 to 4000  $\mu\text{N}$ . SEM images of a 1  $\mu\text{m}$  diameter micropillar before and after compression up to a maximum load of 3 mN are shown (pictures). The load-displacement curves obtained for a 1  $\mu\text{m}$  diameter micropillar are reported (see figure), together with the engineering stress-strain curves calculated using the initial diameter of the micropillar equal to 1  $\mu\text{m}$  and the initial height of the micropillar equal to 3070 nm ( $= 2520 \text{ nm} / \sin(55^\circ)$ ), as shown in the pictures. Comparison between the engineering stress-strain curves obtained for the 1  $\mu\text{m}$  and the 2  $\mu\text{m}$  diameter micropillars showed that the yield stress of tungsten micropillars (about 1200 MPa) does not depend significantly on the diameter of the micropillar. The elastic modulus of the single crystalline tungsten micropillars was determined from the unloading curves to be approximately 490 GPa. Specimens for transmission electron microscopy (TEM) are now being cut out from the tungsten micropillars using the FIB device located at the PSI, which is not an easy task due to the inherent brittleness and high hardness of tungsten.

Apart activities on tungsten, a number of TEM specimens from various materials have been prepared using the FIB device located at the PSI. In addition, the hardness of the PM2000 ODS ferritic steel has been measured by using the nano-indenter before and after severe plastic deformation (SPD) by Equal Channel Angular Pressing (ECAP) and before and after irradiation with helium ions to a dose of 0.7 dpa.



From left to right: SEM images of a 1  $\mu\text{m}$  diameter tungsten micropillar before and after compression up to a maximum load of 3 mN. A high resolution SEM image of the deformed micropillar.



Load-displacement curves and engineering stress-strain curves.