Environmental and In Situ SEM/TEM

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Tensile / Compression Module in the SEM LEO 982

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Tensile / Compression Modules in combination with a microscope are the standard measurement tool in material testing to determine structure-property-relationships. Through the combination the influence of localized changes of material properties (e. g. cracks, pores, grain size or grain orientation) on the strain-stress curves are analysed. If a visible light microscope is used the resolution is limited to about 1 µm. For a higher resolution or to use the extended detection possibilities of the scanning electron microscopic (SEM) it is necessary to apply the Tensile / Compression Module in the vacuum chamber of the SEM. In this poster technical prerequisites of the adaptation of the module and first results are presented.

We used the SEM LEO 982 with Gemini optic from the company Carl-Zeiss and a Tensile / Compression Module from Kammrath & Weiss in the „Narrow version“ allowing Electron Backscatter Diffraction (EBSD) -applications. The module is especially adapted to the available space in the LEO 982. The maximum strength of the module is 5 kN being both applicable in tension and in compression mode. Here the compression mode is demonstrated on a metal foam. The tensile mode is introduced on a Teflon stripe.

The solid construction of the Tensile / Compression Module guarantees the stable and reproducible use even at high loads. Therefore the loading capacity of the sample stage has to fit the weight of the module, i.e. the stage should not produce mechanical vibrations and instabilities like creeping at this load even for high resolution images. Furthermore suitable flanges for the feedthrough of signals for the control of the module are required. If it is planed to perform SEM analysis at tilted samples (as it is necessary for EBSD-measurements in combination with in situ stresses) there has to be enough space close to the pole piece to tilt the stage without touching any detectors while the working distance is kept reasonable small. In our case the maximum tilt of the module is 50° (see figure 1), requiring the use of pretilted samples to fulfil a 70° incident angle of the electron beam with respect to the surface of the sample. On one hand it is not possible to tilt the module more than 50°. On the other hand it is not desirable to tilt the module less than 50° since the modules support will cover the EBSD-detector for this case.

On mounting the module it should be considered additionally that the module does not cover the SEM-detectors (SE, BSE, EDX, etc.) for all operation conditions. The standard requirements for specimens in the SEM are the vacuum suitability and the conductivity. For tensile / compression tests more requirements need to be considered. The size and shape of the sample have to fit to the fixation mechanisms of the module.

The procedure for in situ experiments is determined by the material properties of the specimen and the question to be examined. The important material characteristics to notice are the geometric (homogeneous or heterogeneous structure) and deformation properties (fast, slow or no relaxation after load), e. g. for samples without relaxation it is possible to interrupt the load and to image the area of interest at a static specimen and not during movement. Therefore a higher image quality and series of images at different sample position and different magnifications but with identical conditions are possible. As an example figure 2 shows two image series with two different magnifications during the stepwise compression of a aluminium metal foam. Furthermore an insignificant relaxation is a necessary prerequisite also for long time measurements, such as EDX-Mappings.

Future work will focus on the EBSD analysis of grain size and grain orientation measurements with in situ tensile and compression. For this purpose the SEM is equipped with an EBSD system by EDAX. This system uses a high speed CCD camera (Hikari) to keep the measurement time within reasonable values and to avoid misinterpretation through creeping and relaxation of the sample during the measurements. The reduced spatial resolution of the fast Hikari camera is expected to be no limitation for the EBSD-recognition since it is aimed to analyse orientation but not a detailed phase analysis.

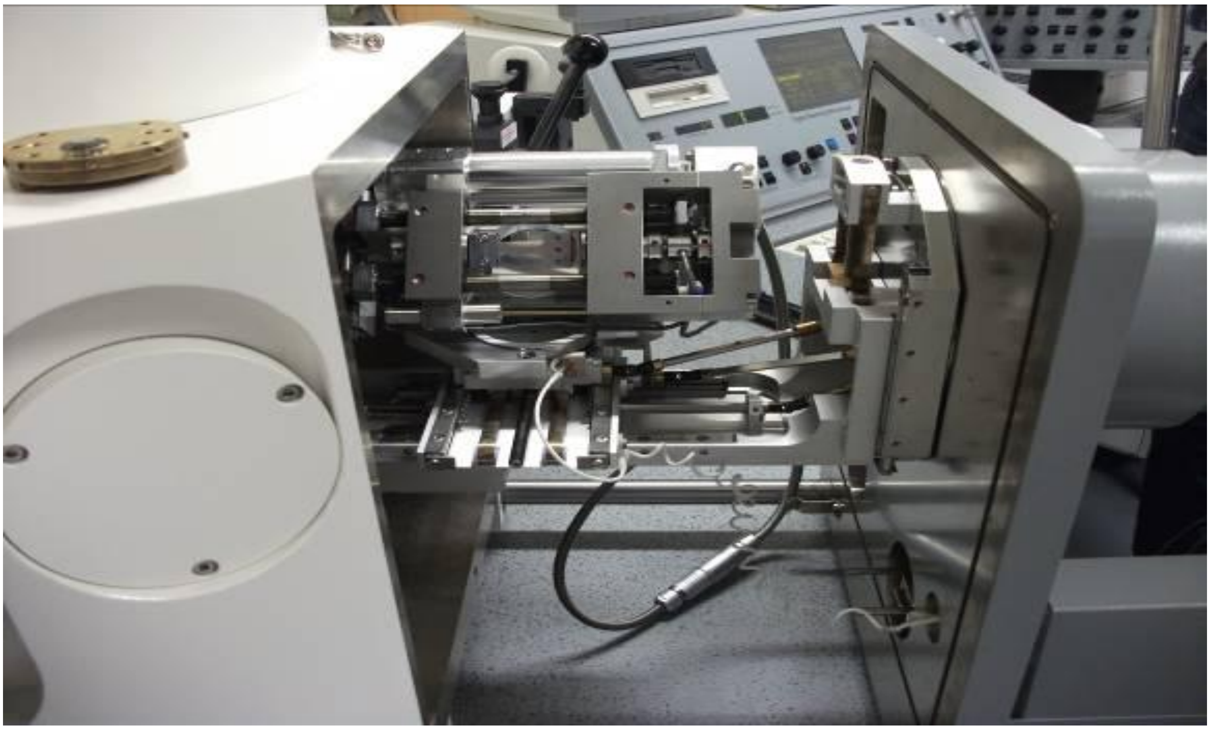

Figure 1. Tilted Tensile / Compression Module for EBSD tests

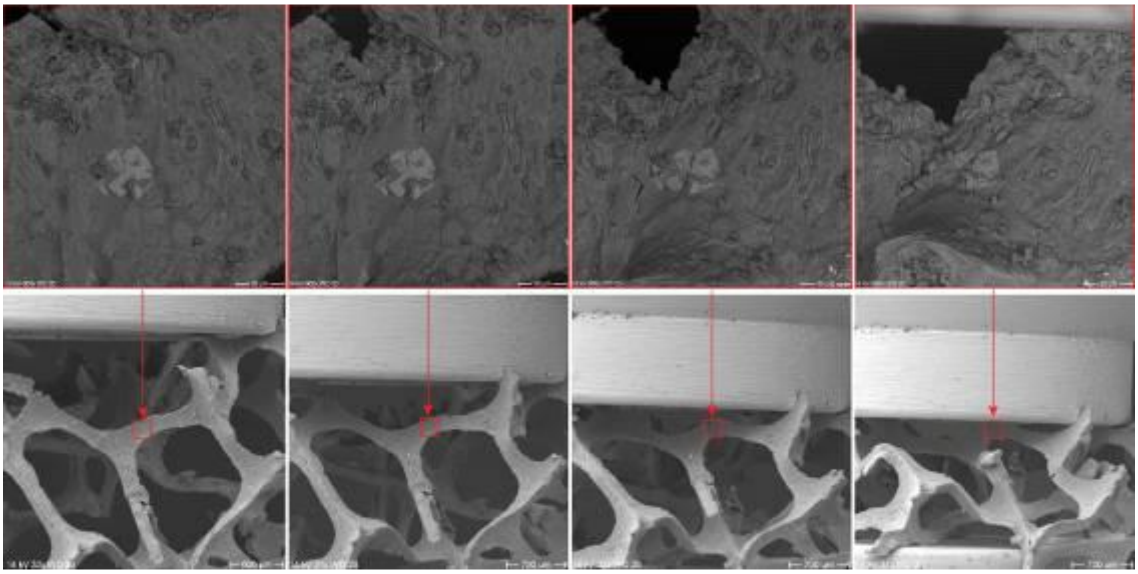

Figure 2. Compression of a metal foam. The first line shows the growing of a rip. The second line shows an