ESRF	**Experiment title:** Feasiblity and Optimisation of High Energy Synchrotron Residual Stress Analyses on the Example of a Multiphase Round Robin Sample issued by the VAMAS – TWA 20 group	**Experiment number:** MI – 338
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Report:

In previous experiments (e.g. HS241, HS549) it was proven, that residual stress analyses by high energy synchrotron radiation offer numerous advantages compared to conventional X-ray diffraction as well as to neutron diffraction. Among these advantages is the extreme intensity of the high energy synchrotron beam, which permits measurements in the bulk of samples at short measurement times. Due to the high intensity and parallelism of the high energy synchrotron beam, gauge volumes in the order of only several 100 μm^3 can be achieved. Futheron, as a consequence of using a white beam a multitude of reflections is available simultaneously. Therefore, the new technique for residual stress analysis using high energy synchrotron radiation offers a range of new applications.

In order to further qualify this method we are interested in a comparison between residual stress analysis by high energy synchrotron radiation and residual stress analysis by neutron diffraction. In case of neutron diffraction people working at different neutron sources have established a task group for the definition of 'best practice' techniques that works within the framework of the Versailles Project on Advanced Materials and Standards (VAMAS, TWA 20). The objectives of the VAMAS TWA 20 group are e. g. to establish accurate and reliable procedures for non-destructive residual stress analyses in crystalline materials by neutron diffraction and to conduct inter-laboratory comparisons to determine the extent to which reproducible results can be obtained from different diffractometers. A number of 3 samples: an SiC/Al_2O_3-composite, an SiC powder and a sample consisting of sintered Al_2O_3 was subjected to an investigation by high energy synchrotron diffraction.

Due to the residual micro stresses, the reflections from the SiC and the Al_2O_3-particles arise at energy levels which differ from those which are evaluated on the stress-free reference materials (SiC powder and sinterd Al_2O_3). The samples proved to be rather coarse grained with respect to the gauge volume, which was here 1200µm x 80µm x 100µm .

The gauge volume size was determined by scanning an Ag-foil of 15µm thickness. Due to radiation safety reasons the height of the gauge volume could not be extended. Also the length and the width of the volume could not be chosen larger since the gauge volume had to be inside the samples of app. 2mm thickness. Therfore, the sample was oscillated in by +-5° in the angle Ω so that a high number of grains diffracted. The spectrum reveals a severe overlapping of reflections. This is due to the high number of polytypes existing for the SiC. This high number of polytypes as well as the composition of the composite of two non-cubic phases results in comparatively weak reflections. We chose the 2θ -angle 7° therfore as a compromise between intensity and energy resolution. Although we would have liked to change this angle this proved to be impossible with the current set-up since the slits would have had to be completely new aligned.

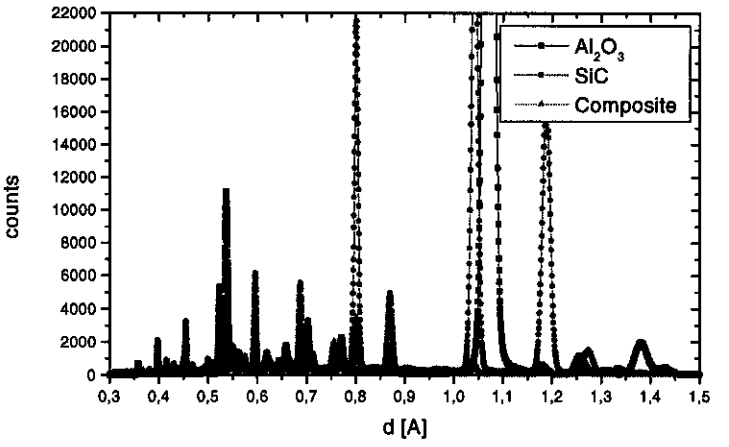

Fig. 1: Spectrum of the powders and the composite

The spectra indicate that strong peak overlapping is present. The position of the reflections in spectra recorded at different times proved to be higly reproducible (difference between 5 E-5 nm typically). The strain obtained in in-plane and out-of-plane direction in good agreement with neutron diffraction revealed compressive residual strains in the SiC and tensile residual strains in the Al_2O_3 (fig.2).

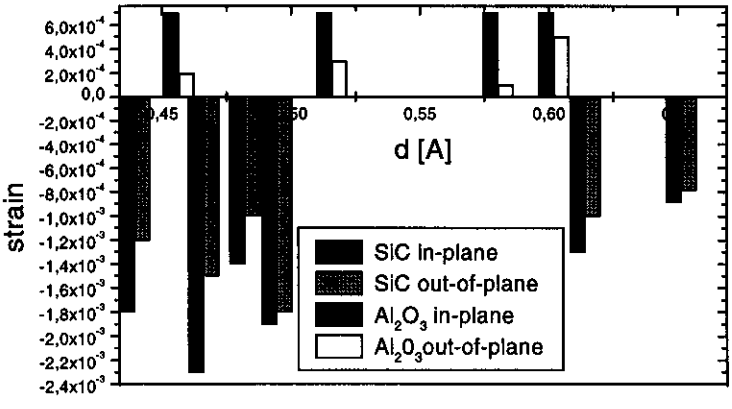

Fig. 2: Strains of different lattice planes in in-plane and out-of-plane direction of the SiC/Al_2O_3 - composite

The value of theses strains due to elastic ansiotropy is slightly different for different lattice planes.
Further residual stress analyses were performed at different locations in the sample checking for material inhomogneities. Apart from that we performed measurements using the d-sin$^2\psi$-technique. Also through-surface scanning was done on the reference sample and the composite, since this technique due to the smaller divergence of the synchrotron beam compared to neutrons seems to be favourable for near surface residual stress analysis. The analysis of the data and the development of the formalism for the energy dispersive measurements is still in progress.