ESRF	**Experiment title:** **Macro and micro stress development in technical materials subjected to mechanical and thermal load**	**Experiment number**: ME - 558
Beamline: ID 15a	**Date of experiment**: from: Sept. 2003 to: Sept. 2004	**Date of report**: 08.09.2004
Shifts: ~ 54	Local contact(s)**:** T. Buslaps, M. Di Michiel (ID15a) F. Fauth (ID 31), E. Boller (ID 19)	*Received at ESRF:*

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Report:

Experiments carried out during the Longterm – Project

1. Determination of Internal Stress State in AlSiC MMCp and in-situ thermal loading of the composite Experiment at ID15a (18 shifts), ID31 (9shifts) and ID19 (3 shifts in as industry beam time: IN 420, bought by A. Pyzalla)

In high power electronics thermal conductivity of materials employed and the minimization of thermal mismatch strains between different materials employed in a module become increasingly important. The aim of the PhD thesis of Ulrike Goebel was to study the possibility of using modern particles reinforced metal matrix composites (MMCp) as base-plates for the so-called Insolated Gate Bipolar Transistor Modules (IGBTs). In contrast to common, rather well developed MMCp the material studied here has a volume fraction of SiC in the order of 60 to 70 vol.%, which is about twice of the usual amount of reinforcing ceramic phase. In addition to the inhomogeneity produced by the SiC the composites contain pores and Si particles (fig.1). These Si particles form a second reinforcing structure within the material. Depending on the manufacturing process they can be either distributed homogeneously as isolated particles or they can build three-dimensional structures that incorporate aluminium cells.

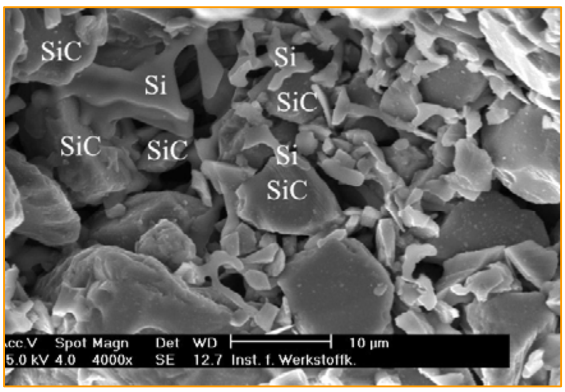

Fig. 1. Si, SiC in AlSiC after deep etching of the Al-matrix /Huber, Degischer 2003/

The aim of the experiments was a) the determination of the macro and micro stresses in the SiC, the Al and especially in the Si particles and b) the determination of the evolution of the micro stresses depending on temperature. The main obstacles to overclimb were the very small volume fraction of Si in the order of 1 up

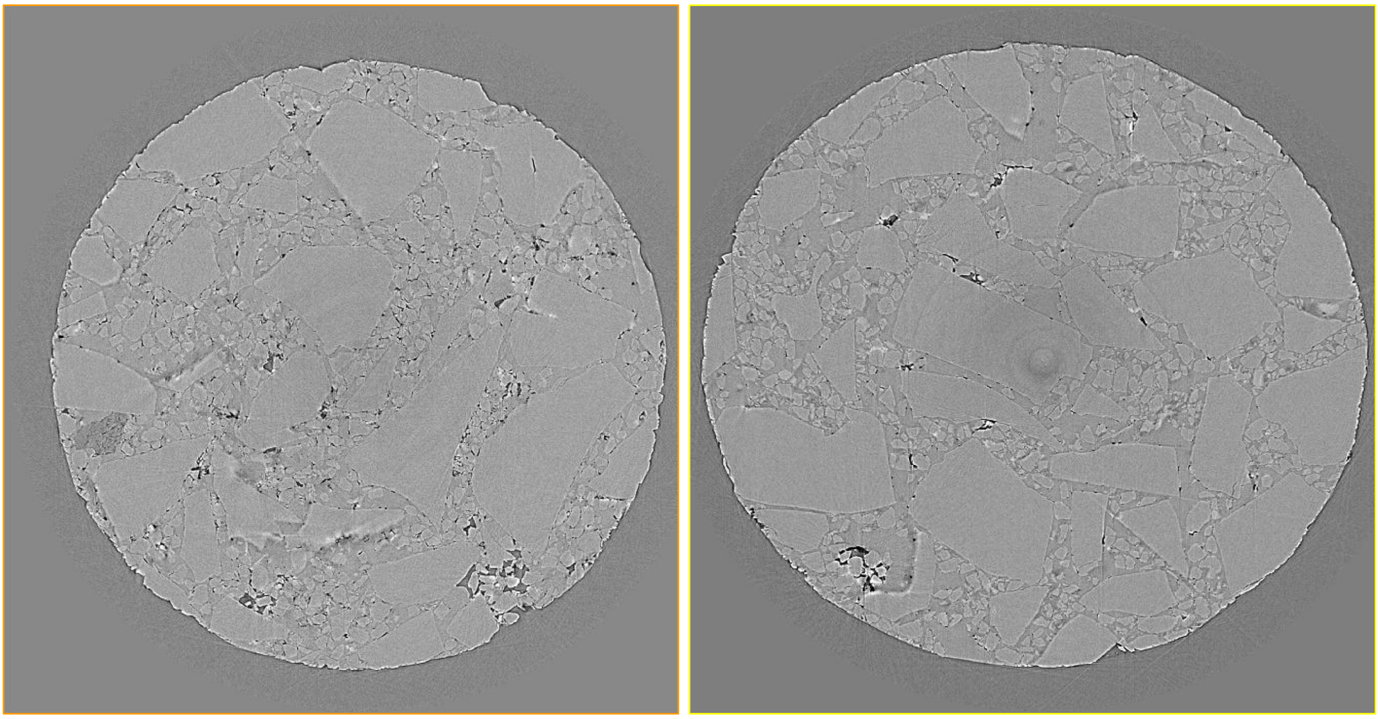

Fig.2: Pores in he center (left) and at the boundary(right) of the AlSiC-baseplates.

to 2 vol.-% and the coarse graininess of the SiC.

The internal macro stresses in the samples were determined on ID31. We overcame the coarse grain problem by strain scanning and afterwards adding the diffractograms obtained at neighbouring points, thus increasing the number of grains contributing to strain analyses. The results of the experiment showed that this appears to be a very reasonable method. The internal stress state obtained revealed significant macro stresses within the plates. In order to determine the cause of these macro stresses we decided to do a tomography experiment (since the PhD thesis of U. Göbel is supposed to be finished soon, we needed to buy additional beam time, 3shifts at ID19). Absorption tomography and also holotomography clearly revealed that the source of the internal macro stresses is the differences in the porosity in the centre and near the boundary of the AlSiC-plates (Fig. 2). These differences in the porosity are due to the infiltration process, where not sufficient liquid aluminium reached the centre of the green body.

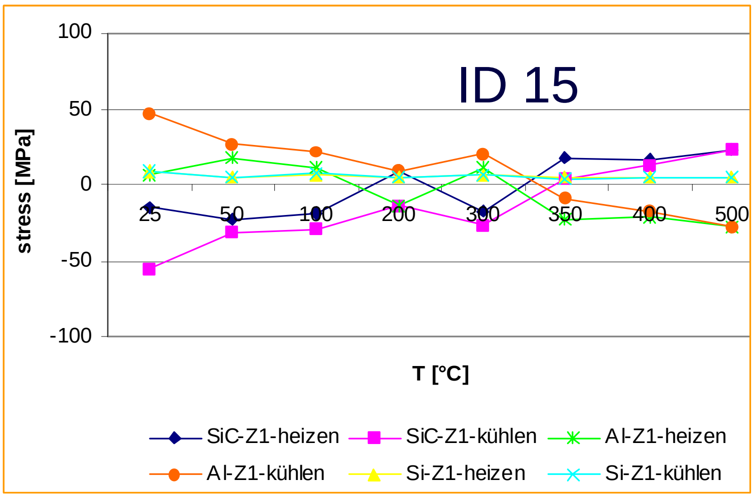

Fig.3: Phase specific internal stresses during heating and cooling in AlSiC

Despite the small volume fraction of Si – particles we managed to obtain sufficiently well resolved diffractograms for all phase from small samples. By in-situ heating experiments doing thermal cycles up to 400°C we could determine the turning point with full relaxation of the internal stresses in the samples and see also the onset of strains and stresses during cooling in the different phases (Fig.3). The results obtained show also the interesting detail that the Si structure in the aluminium matrix appears to more or less stress-free, but shows very broad reflections compared to a Si powder standard. This implies that the Si structure contains areas with high tensile as well as high compressive strains/stresses, depending on whether the Si particle is close to an SiC particle or completely surrounded by the aluminium matrix. The internal stress

development also indicated maximum temperatures that are not be reached e.g. by a brazing process if the AlSiC – plates are supposed not to distort during manufacturing. Publications about the results of the experiments are in preparation.

2. First in-situ creep-experiment: Determination of pore evolution in MMCs during creep (9 shifts on ID19 in spring 2004)

The aim of our first beam time at ID19 was an in-situ creep experiment and the determination of pore evolution during creep of MMCp. Pore evolution in MMCp using conventional microscopy strongly suffers from artefacts such as closing pores by material smearing into them during grinding and polishing or particle fracture occurring due to heavy loads imoosed in the grinding or polishing process. Further on only a three-dimension al picture of the pore evolution permits us to determine the change in pore volume fraction and other geometrical parameters during the creep process.

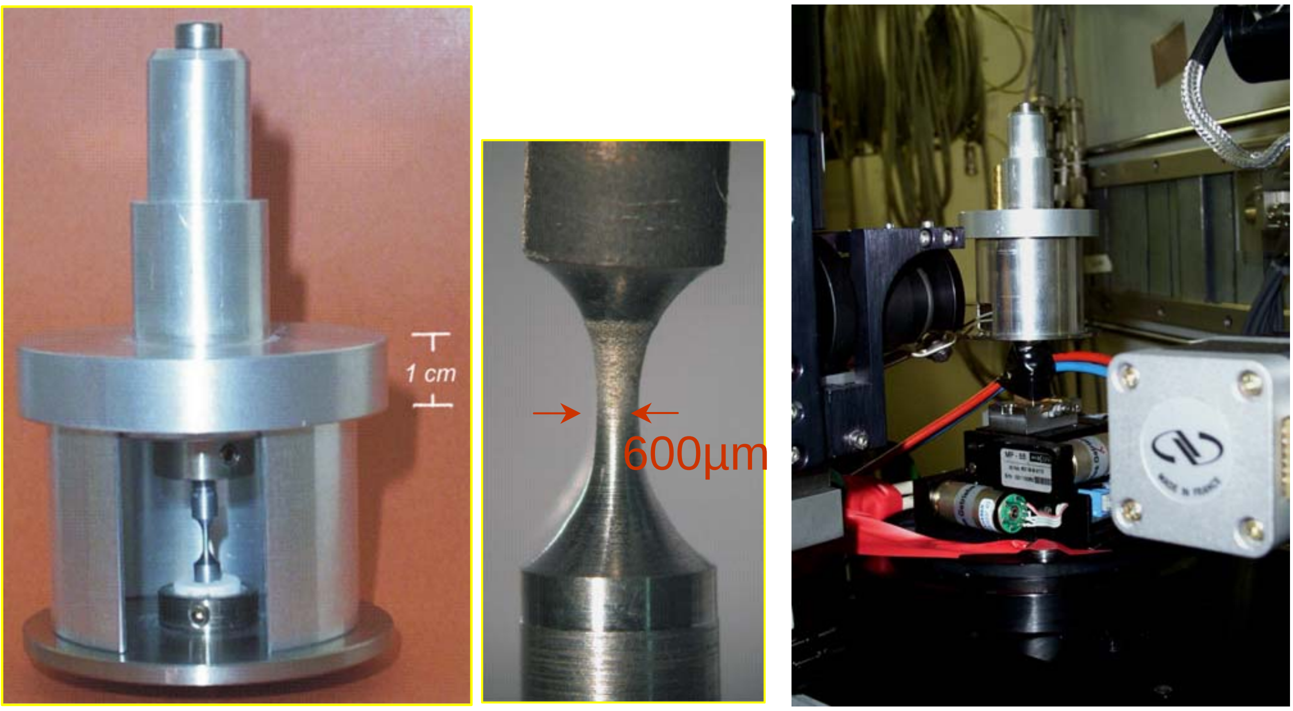

Fig. 4: Left: creep device, center: creep sample, right: creep device mounted at ID19

The main problem to be solved in order to perform the first in-situ creep experiment was the construction of an appropriate creep device. Since the stability of the device with respect to the centre of rotation and the minimization of vibrations is crucial we constructed it in a way, that allows us to load the sample without needing a motor (Fig. 4). This is accomplished via a spring. By taking springs with different stiffness, the load on the sample can be varied. The construction principle of the creep device further allowed us to rotate it around even 360° without changing the absorption conditions. The sample is induction heated, which allows us to reach the creep temperature rapidly.
For the experiment we chose to Al-base MMCs where we expected strong differences in their creep behaviour: AlSi25Cu4Mg1 and AA6061+SiC. In order to allow for fast creep experiments during the beamtime available we chose small loads and high temperatures (350°C and 400°C). One difficulty which became apparent during the experiment was the strong increase of creep speed in the later stage II of the creep process which produced artefacts due to the lengthening of the samples.

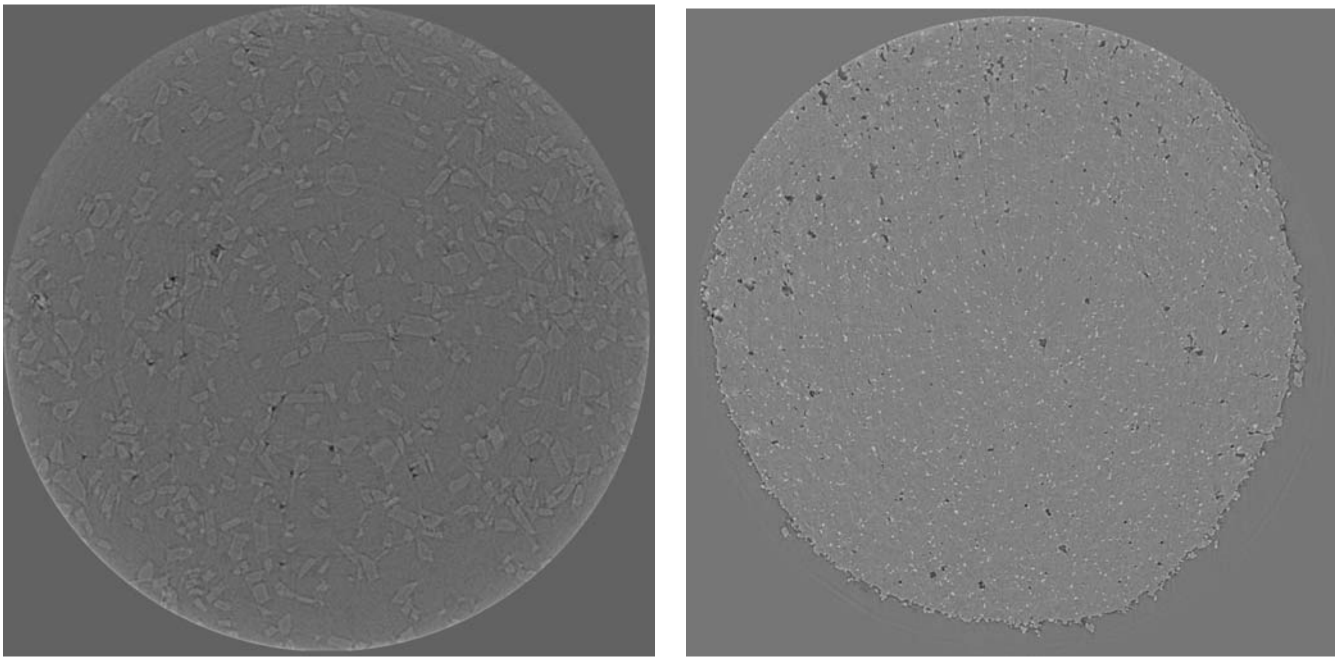

Fig. 5: Slices of the tomogram a) AA6061+Al_2O_3 at 360°C, b) AlSi25Cu4Mg1 at 400°C, Artefacts are due to the movement (lengthening) of the samples during the creep process

Though a few samples broke due to their small diameter necessitated by the necessary spatial resolution to be obtained (0.7µm), for both materials successful creep experiments were performed. They revealed that in case of AlSi25Cu4Mg1 large areas of connected pores become visible during the creep process. The evaluation of the tomograms done so far indicate that the pore volume and their shape change during the creep process and are different for different temperatures. In case of AA6061 in contrast we see fewer pores and the pore volume appears to be less different for different creep temperatures (Fig. 5). The pore volume also appears to increase not as fast and as strongly as in AlSi25Cu4Mg1. Further data evaluation is in progress. Additional creep tests with macroscopic samples have been done and the samples investigated at ID19 are subjected to complementary microscopy.

3. First experiment combining diffraction and tomography (ID15, 18 shifts, June 2004)

In order to show that combined experiments (where two experimental methods are used simultaneously) to characterize the microstructure development of a sample are possible we did a world-wide first experiment combining diffraction and tomography with high energy synchrotron radiation. We used both the monochromtic beam as well as the white beam transmitted through the monochromator. The white beam was shielded by a beam tube in order to decrease the scattering background.

The experimental set-up consisted of a tomography camera, sample stage with rotation, szintillator counter and energy dispersive detector (Fig. 6). During tomography and white beam measurement the sample remained at the sample place. For the diffraction with the monochromatic beam it was translated into the monochromatic beam. In both energy dispersive and angle dispersive diffraction set-ups the gauge volume in the sample was defined by slits.

The sample was situated in the creep device used already at ID19, which however had been further developed. We chose a CuZn – alloys, German standard CuZn40Pb2 and CuZn37 for the combined creep and diffraction experiment. In

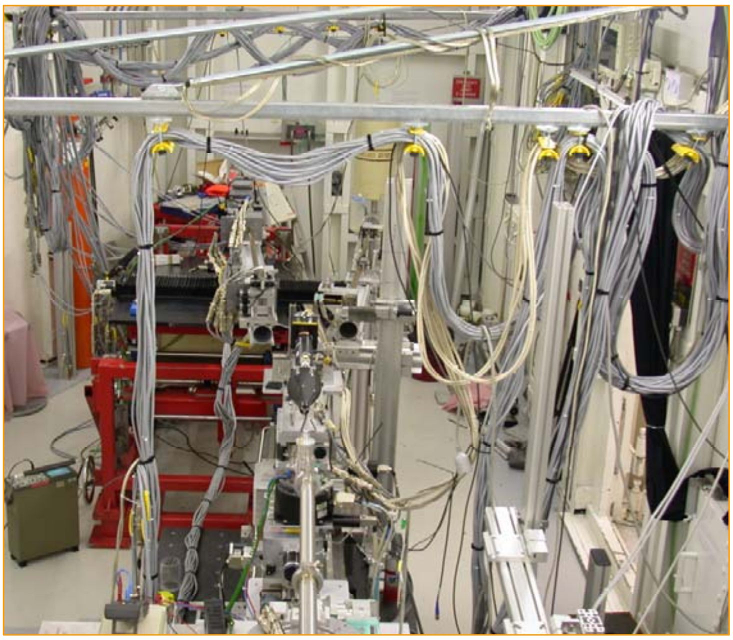

Fig. 6: Set-up for the combined tomography-diffraction experiment at ID15a

order not to suffer from coarse grain effects and to see creep occurring within a reasonable time frame we used material that was 4.47% deformed prior to the experiment.

The results of the experiments revealed that it is possible to perform experiments where tompography and diffraction are performed simultaneously during one experiment. The tomography data obtained for the two-phase alloys CuZn40Pb2 showed the development of the pores during the creep process. Significant differences are apparent for creep at 300°C and 400°C (fig.8). Strong differences are visible in the pore shapes in the single phase brass CuZn37 and the α/β-brass CuZn39Pb2. The diffraction data shows that reflection width decrease in the early stage of the creep process and remain constant after a certain pore volume fraction is reached. Further on, the diffractograms reveal that as soon as the pore volume fraction increases strongly a fibre texture forms in the α/β-brass CuZn39Pb2 (fig.7).

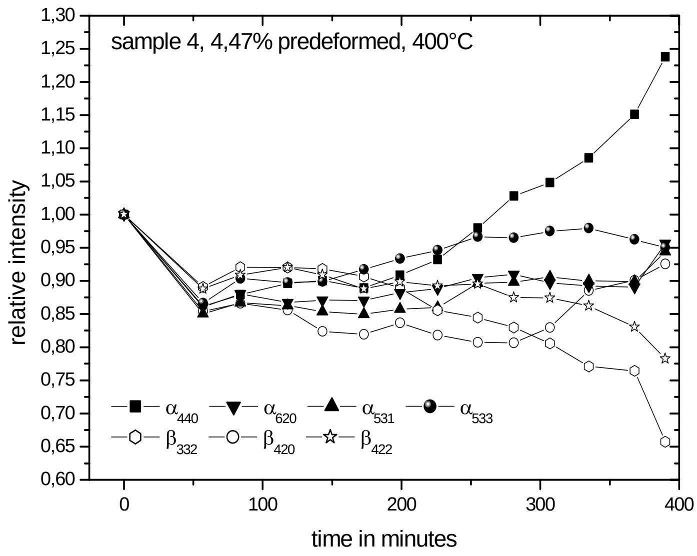

Fig. 7: Intensity changes during creep (energy dispersive data)

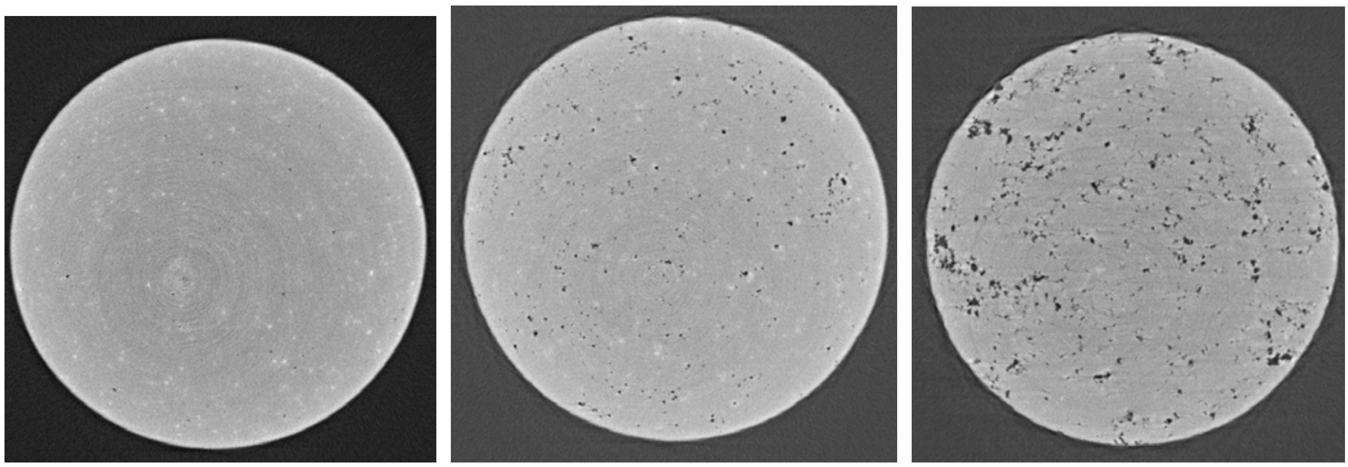

Fig. 8: CuZn39, left: start of the creep process, centre: intermediate state, right: before failure

The quantitative evaluation of the diffraction data has been done, but in case of the tomography data is still in progress.

Publications relating to work done at the ESRF

- E.B.F. Lima, J. Wegener, C. Dalle Donne, G. Goerigk, T. Wroblewski, T. Buslaps, A.R. Pyzalla, Dependence of the Microstructure, Residual Stresses and Texture of AA 6013 Friction Stir Welds on the Welding Process , Z. Metallkunde 94 (2003) 908 – 915
- A. Pyzalla, W. Reimers, Study of Stress Gradients using Synchrotron X-ray Diffraction, Chapter 13 in: „Analysis of Residual Stress by Diffraction using Neutron and Synchrotron Radiation“, (A. Lodini, M.E. Fitzpatrick eds.), Taylor and Francis, New York, USA (2003)
- S. Dieter, A. Pyzalla, A. Bauer, N. Schell, J. McCord, K. Seemann, N. Wanderka, W. Reimers, Correlations between Magnetic Properties of Single and Multi-layer CoFe/SiO2 Thin Films and their Microstructure, Texture and Internal Stress State, Z. Metallkunde 95 (2004) 163 - 175
- A.R. Pyzalla, B. Reetz, A. Jacques, J.P. Feiereisen, O. Ferry, T. Buslaps, W. Reimers, In-situ Investigation of Stress Relaxation in Al/Si - MMCs using High Energy Synchrotron Radiation, Z. Metallkunde 95 (2004) 624 – 630

- A.R. Pyzalla, B. Reetz, A. Jacques, J.P. Feiereisen, O. Ferry, T. Buslaps, W. Reimers, Relaxation of Phase Specific Elastic Strains/Stresses During Deformation of Al/Si – MMCs, at Elevated Temperatures, MECASENS-Proceedings and J. Neutron Research, in print
- H.C. Pinto, A.R. Pyzalla, W. Reimers, T. Buslaps, V. Honkimäki, L. Zhang, Y. Dabin, Micro-structural Damage in Thermal Absorbers induced by High Energy Synchrotron Light Irradiation, MECASENS, J. Neutron Research, in print
- H. C. Pinto, A. R.Pyzalla, W. Reimers, T. Buslaps, V. Honkimäki, L. Zhang, Y. Dabin, Investigation of Synchrotron Radiation induced Damage in Thermal Aluminium Absorbers, J. Materials Science and Technology, accepted
- R.V. Martins, U. Lienert, L. Margulies and A. Pyzalla, Determination of the radial crystallite micro-strain distribution within an AlMg3 torsion sample using monochromatic high energy synchrotron radiation, Mat. Sci. Eng. submitted

Habilitation, PhD theses' and graduate theses' with significant contributions by the experiments using synchrotron radiation at the ESRF

PhD – Theses (in preparation)
- Ulrike Göbel, Distortion and internal stress in Al-SiC baseplates for power electronic modules, TU Wien, autumn 2004
- Karolina Zimnik, Determination of internal stress and texture in small components using high energy synchrotron radiation, TU Wien
- Rodrigo Santiago Coelho: Microstructure, internal stress state, texture and performance of novel dissimilar metallic joints, TU Wien
- Marcin Moscicki: Microstructure, internal stress state and crash behavior of aluminum alloy welds, TU Wien
- Björn Reetz: Microstructure changes and mechanical behavior of multiphase CuZn-alloys (brass) during loading at elevated temperature, TU Berlin

Diploma Theses (in preparation)
- Nastin Jank: Microstructure and internal stress of novel dissimilar metallic joints, TU Wien
- Andrea Pernack: Microstructure and internal stresses in CuZn-alloys