ESRF	**Experiment title:** **Macro and micro stress development in technical materials subjected to mechanical and thermal load**	**Experiment number**: ME - 98
Beamline: ID 15a	**Date of experiment**: from: Sept. 2000 to: Feb. 2002	**Date of report**: 26.02.2002
Shifts: ~ 54	Local contact(s)**:** T. Buslaps, J. Merino, V. Honkimäki, T. d'Almeida, A. Fitch	*Received at ESRF:*

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Report:

Changes in experimental set-up, equipment brought to the ESRF

A main aim of our group has been improving the set-up for stress / strain measurements at ID 15a. This has been done in very close co-operation with Dr. Thomas Buslaps, Mr. Yves Dabin and Mr. Mogens Kretzschmer of the ESRF. Thomas Buslaps has provided us with details of the equipment present and the possibilities for changes within the hutch. Yves Dabin and his group have constructed and implemented a large part of the new set-up in co-operation especially with Dr. Agnès Royer of our group. Mogens Kretzschmer has provided us with adjustments of our samples to translation stages, furnaces etc.

The set-up for stress / strain determination was moved from the triple-axis diffractometer to a separate table at the side of the hutch of ID15B which is at the longest distance to the source. This means, that the basic set-up is available for all stress / strain and other experiments (e.g. experiments on amorphous materials and liquids), which spares several (~3 to 5) shifts that have been necessary for the set-up before. On this table an ω – ciricle and a large Eulerian cradle has been installed. Within this new Eulerian cradle now an x-y-z table can be implemented which as a translation distance in x and y of 50 mm and in z of 50 mm. Thus, it is now possible to determine a strain distribution by moving samples and small industrial components perpendicular and in the beam direction without continuously rearranging and subsequently realigning them. For a rapid and reproducible fixation of the samples on the translation stages several adaptation pieces have been manufactured by M. Kretzschmer. On the ω – circle beneath the Eulerian cradle a profile is fixed, which supports the slits. Thus, now an alignment of the slits directly in the direction of the primary beam can be performed by using a diode for detector. This facilitates the adjustment of the slit translation and rotation significantly, since the slits usually are opened horizontally less than 100µm. Due to this set-up change now the volume element size can be decreased to a width of less than 40µm, which opens up new possibilities for

measurements in layer structures, e.g. coated turbine blades. A further significant improvement of the set-up is the shielding of the primary and the diffracted beam by tubes. The brass tubes covering the diffracted beams have tremendously reduced the background of the spectra. An evacuated steel tube in the primary beam has improved the flux in the medium energy range (~40 keV to 60 keV), which improves measurements on components consisting of a high and a low Z – material such as. measurements on hydroxyapatite coated titanium for medical applications.
Since the profile which supports the slits and the detector is mounted on wheels now it is possible to change the 2θ – value rapidly between measurements and even during measurements. Thus, in case of stress / strain experiments easily a compromise between signal intensity and resolution respectively volume element size can be realised.
Regarding data evaluation a software has been developed which enables a Gauss fit of multiple reflections in a spectrum. This software is based on the IDL language. The software works well for spectra without and with few reflection overlapping. Future work is necessary to improve this software with respect to an on-line data evaluation during the measurements.
Equipment brought to the ESRF consists of a furnace enabling the study of the influence of temperature gradients on the strain / stress distribution e.g. in coated materials and a combined heating - torsion device.

Future requirements and suggestions for stress / strain – experiments at ID 15a
Due to the building of a new hutch with thicker plumbum shielding now the beam size in the experimental hutch can be increased to a size of several mm^2. Thus, the time necessary to acquire significant data now is limited by the photon gain of the detector, especially the detector electronics. Especially with regard to stress / strain experiments at elevated temperature, where dynamic processes occur, an improvement of the detector electronics would be reasonable.
A further improvement could be made by developing a slit system which enables moving the slit size easily during the experiments. This, however is ambitious due to the higy accuracy of the movement of the slit parts required.

User groups which have used the experimental set-up built-up at ID 15a
- Prof. P.J. Withers, Materials Science Center, Manchester University, UK
- Prof. B. Derby, Manchester University, UK
- Dr. V. Stanic, Ancona University, Italy
- Prof. F. Mompean, Madrid University, Spain
- Dr. D. H. Moritz, DLR Köln, Germany

The new set-up was used for experiments regarding stress – strain determination as well as for experiments on amorphous systems and liquids.

Experiments carried out during the Longterm – Project
The focus of the experiments carried out during the Longterm – Project has been on the determination of macro and micro stresses in technical materials subjected to either mechanical or thermal loading or to a combination of both mechanical and thermal loading.
Initial experiments performed focused on an optimisation of the set-up and on determining the development of texture and intergranular (micro) strains in different bcc and fcc alloys deformed by tensile and compressive deformation. The results of the experiments verified calculations of the texure and microstrain development simulated by numerical models.
A larger part of the experiments were dedicated to the determination of the strain and stress distribution in hot extruded aluminum metal matrix composites and in friction stir welds. The manufacturing processes in both cases exert strong plastic deformations at high temperature on the material. The analyses revealed that in case of hot extrusion the process parameters billet temperature and extrusion speed had a strong influence on the microstructure, the texture and the strength whereas their influence of the residual stress state was low. Instead the residual stress state in the hot extruded composites was merely determined by the differences in the properties of the metal matrix and the particles. Thus, in extrudates made of particles reinforced aluminium alloys the micro residual stresses are of particular importance. In case of the friction stir welds a strong influence of the process parameters welding speed, tool speed and tool diameter on the residual stress

distribution has been found. Due to the high local resolution obtainable and the high penetration depth here the distribution of the three prinicpal residual strain / stress components could be determined. For all samples investigated critical high tensile residual stresses were present in the heat affected zone where also a strength minimum is present and the microstruncture is in an overaged state. These are the reasons for failure occuring exactly at this position during tensile tests and during fatigue tests.

The largest part of the measurement time was dedicated to in-situ tensile tests of composites at elevated temperature. These measurements have been possible for the first time using high energy synchrotron radiation since due to the high photon flux the data acquisition time is in the order of several ten seconds which means that only a very limited change of the strain level due to relaxation processes occurs during the data acquisition for a spectrum. High energy synchrotron radiation enables measurements in a closed furnace so that the deformation process at elevated temperature can be studied in-situ. Aluminium metal matrix composites reinforced with silicon particles and different amounts of alumina have been investigated. The experiments comprised tensile tests at different temperatures and up to different strain levels. Further on, relaxation tests were performed at different temperatures and from different strain levels. Also some complementary creep tests were performed at different temperatures and stress levels. The results of the tensile tests at elevated temperatures revealed information about the load sharing between the aluminum metal matrix and the particles. Due to the availability of the whole spectrum texture development was monitored at the same time as the strain / stress development. The data revealed no significant texture changes for both the matrix and the particles in the AlSi – alloy whereas a change of the particles orientation was observed for the alumina particles in AA 6061 + Al_2O_3. The relatively high volume amout of particles of 15 vol.% up to 25 vol.% for different samples inhibited texture formation as well as interngranular strain formation in the metal matrix. In AA6061 + Al_2O_3 at high load stresses severe boadening of the reflections of the metal matrix and also some reflection broadening for the particles was observed which can be attributed to the formation of micro strains. The relaxation tests also reveal information about the load sharing behaviour of the matrix and the particles. The time necessary for the acquisition of a spectrum could be reduced to 30s. It can be expected that, due to the larger beam size possible after the rebuiliding of the hutch, the data acqusition time can be further shortened so that even more information about the first stage of the relaxation process will be available now. The data show the influence of the temperature and the total strain on the relaxation behaviour of the elastic strains in the composites. Besides the time dependent strain change has been determined.

During the experiments performed in February 2002 the possibilities for very small gauge volumes were explored. Gauge volumes of a very limited width are necessary for an exploration of the phase composition, the strain and the texture change in hidden layers. These hidden layers are of particular importance in systems where a bond coat is situated between a substrate and a thick outer layer protecting the substrate e.g. from corrosion or high temperature oxidation. Choosing a slit width of 40 µm it was possible to study the phase composition, the texture and the strain in coated turbine blades and hydroyapatite coatings on titanium substrstes. In case of the turbine blades especially the influence of the oxidation of the bond coat, which has a thickness of only 150µm was investigated. In case of the hydroxyapatite coating the main interest was on the influence of a mixed hydroxyapatite – titanium intermediate layer on the strains in both substrate and the outer hydrxyapatite layer. The data obtained on the layer structures are still under evaluation.

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

Habilitation

A. Pyzalla, Analyse stark plastisch verformter Werkstoffe mit Beugungsverfahren („Analyses of strongly plastically deformed materials using diffraction methods"), Faculty for Mechanical Engineering, Ruhr-Universität Bochum, Germany, July 2001

PHD – Theses

- S. Aris: Simulationen der Textur und der texturbedingten Kristallitmikrodehnungen (Simulation of texture and texture induced crystallite microstrains), Faculty for Process Sciences, TU Berlin, Autumn 2000
- J. Wegener: Mikrostruktur, Textur und Eigenspannungen von stranggepressten und reibrühr-geschweißten Aluminiumlegierungen (Microstructure, Texture and Residual Stresses in hot extruded or friction stir welded aluminum alloys), Faculty for Process Sciences, TU Berlin, Summer 2001

Graduate Theses

- E. Lima: Mikrostruktur, Textur und Eigenspannungen von Reibrührschweißnähten aus Al 6013 („Microstructure, Texture and Residual Stresses in AA 6013 Friction Stir Welds"), TU Berlin, Germany, Winter 2002
- B. Reetz: Untersuchungen zum Verformungsverhalten von partikelverstärkten Metallmatrixverbund-werkstoffen am Beispiel von AA 6061 + Al_2O_3 (Experimental determination of the deformation mechanisms of the MMC of AA 6061 + Al_2O_3), TU Berlin, Germany, Summer 2001

Erhard – Höpfner – Prize

The graduate thesis of Björn Reetz was voted best graduate thesis in materials science and structure research in Berlin in 2001 and awarded the Erhard – Höpfner – Prize.