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The Fourier-RTOF neutron diffractometer FSS at the 5 MW research reactor FRG-I

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Introduction

At the GKSS research center new experimental facilities have been built around the FRG-I reactor to cover a wide variety of different aspects of materials science, which will help to understand the macroscopic behaviour of materials by studying their microstructure. This covers also the non-destructive measurement of strains within polycrystalline materials and the subsequent calculation of the internal stresses, which are important both from the technical as from the basic research point of view.
The concept and layout of the FSS spectrometer - which stands for *Fourier Strain Spectrometer* - was therefore optimized for this purpose.
From comparison of the positions of Bragg reflections in strained and unstrained specimens the lattice strain is detemined as the relative difference between the two. The fact that the elastic constants of most materials are crystal-orientation dependent and that texture may considerably influence their intensities, led to the decision to use time-of-flight diffractometry to measure the positions of Bragg reflections. Any variations of peak intensities at different angles of observation indicate the presence of texture, the knowledge of which is important in order to calculate stress factors, using the orientation distribution function. Stress factors replace elastic constants when stresses are calculated from strains for textured samples. Since the duty cycle of a high-resolution chopper is too low to allow to perform time-of-flight spectroscopy at a steady state reactor source economically, only a correlation spectrometer can be used. In this case the two competing properties „neutron intensity“ and „resolution“ are decoupled: the resolution in a time-of-flight Fourier spectrometer is determined by the three components: angular resolution (divergent neutron beams), flightpath resolution (sample sizes and aperture outlines) and time resolution (e.g. the maximum frequency of beam modulation and time channel width in this case). If this latter component is further decreased by using a higher modulation frequency, this will not affect the intensity of the neutron beam. There is another advantage of a Fourier spectrometer which becomes important, when the widths of Bragg reflections have to be measured. Peakbroadenings in general can be caused by size effects or microstrain effects. If they need to be analysed, the shape of the resolution function must be accurately known. Using the calculations of V.Kudryashev, we can choose adequate rotor rotational frequency programs to make the resolution function Gaussian, or the first or second derivative of it, so that any deviation from these shapes leads to information about the physical phenomenon which caused the broadening. ([2], [3], [4],).

Technical details of the FSS design

The main features of the spectrometer have been described in [1]. The instrument is operating in a stable and reliable manner over the periods of time necessary at a low power neutron source to arrive at the statistics requested. According to the materials investigated, standard rotor speed programs, delay times and channel widths have emerged from the growing experimental experience, so that a standardized data evaluation can be performed on a PC. The sample positioner control system is connected to the control of the experiment, so that scan measurements (like through-thickness scans or measurements across the full geometrical extension of a sample) can be performed automatically.
Figure 1 is a schematic of the RTOF diffractometer at this time, while there is hope to be able to install an second detector opposite to the existing one, in order to do simultaneous measurements in the directions of two mutually orthogonal scattering vectors. This will reduce the measuring times for tensor determinations by more than a factor of two.

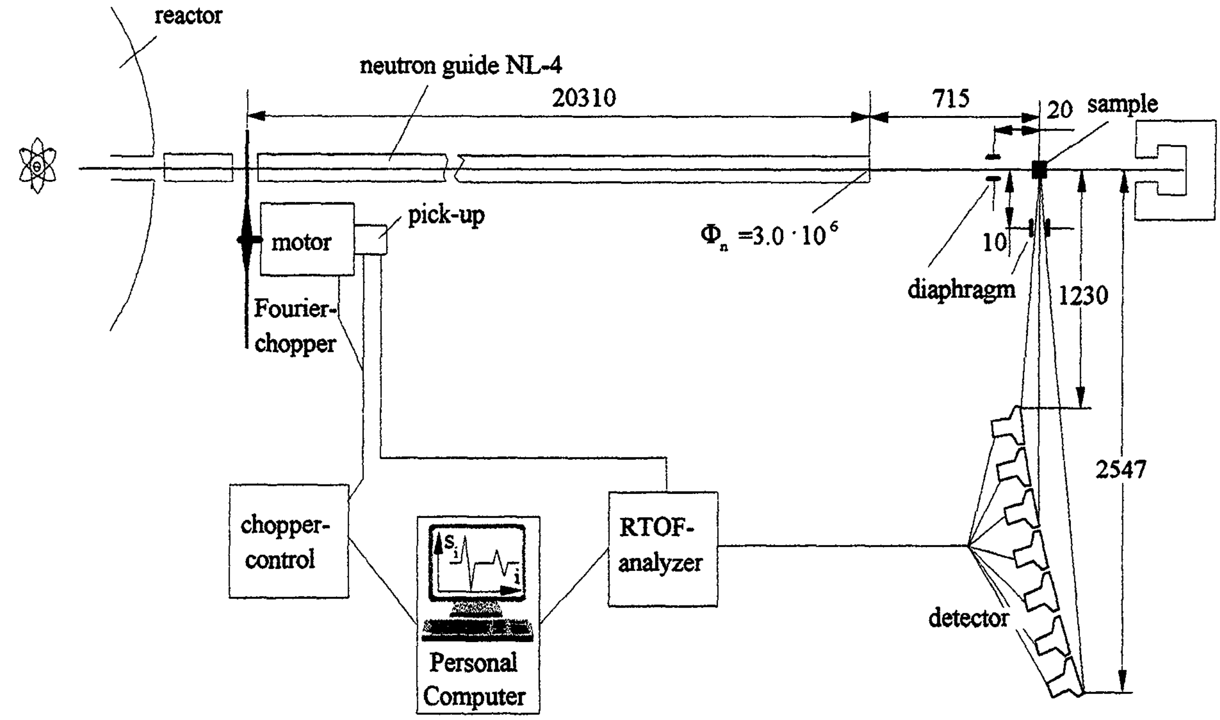

Fig. 1: Schematic outline of the FSS setup (dimensions given in mm)

The detector bank is equipped with 16 ^{6}Li-glass sintillator/photomultiplier units (5" diameter), aligned according to the optimization calculations described in [2]. Figure 2 shows, how the detectors were lined up with a certain tilt from the ideal time-focussing curve to achieve a larger area coverage of the detectors. So a typical run in our case for a 4x4x25mm^3 scattering volume can be done in about 3 hours for steel.

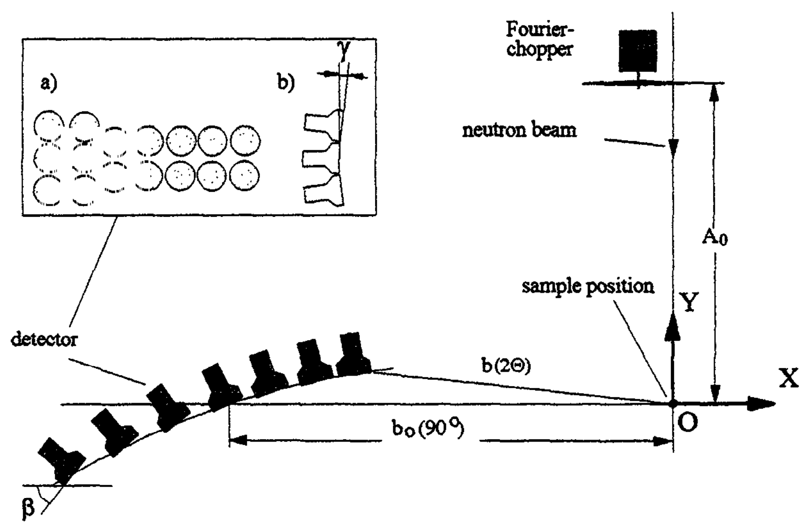

Fig.2: The detector positions are optimized for maximum neutron capture probability at minimum deviation from the time-focussing curve

The nominal spectrometer resolution is determined by three components: the angular and the flight path uncertainties - combined as the geometric component and the time component. It has been calculated and experimentally veryfied, as can be seen in Figure 3.

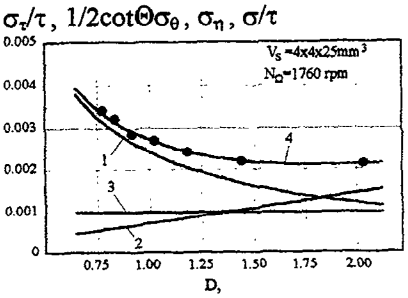

Fig. 3: Components and the resulting relative resolution σ/τ of FSS for a G_1 - shape reflection
1- time component σ_τ/τ,
2- term $1/2\cot\Theta\sigma_\theta$ of the geometric component
3- term σ_η of the geometric component
4- resulting relative resolution σ/τ and experimental values.

The following figure shows the three modes, in which the spectrometer can be used.
Row *a* shows the time vs. frequency functions (u is normalized to the maximum frequency Ω_{max}) , which lead to the reflexion peak shapesshown in row *b* and *c*. The measured peaks in row *c* were fitted and the residues (multiplied by a factor of ten) are also shown.

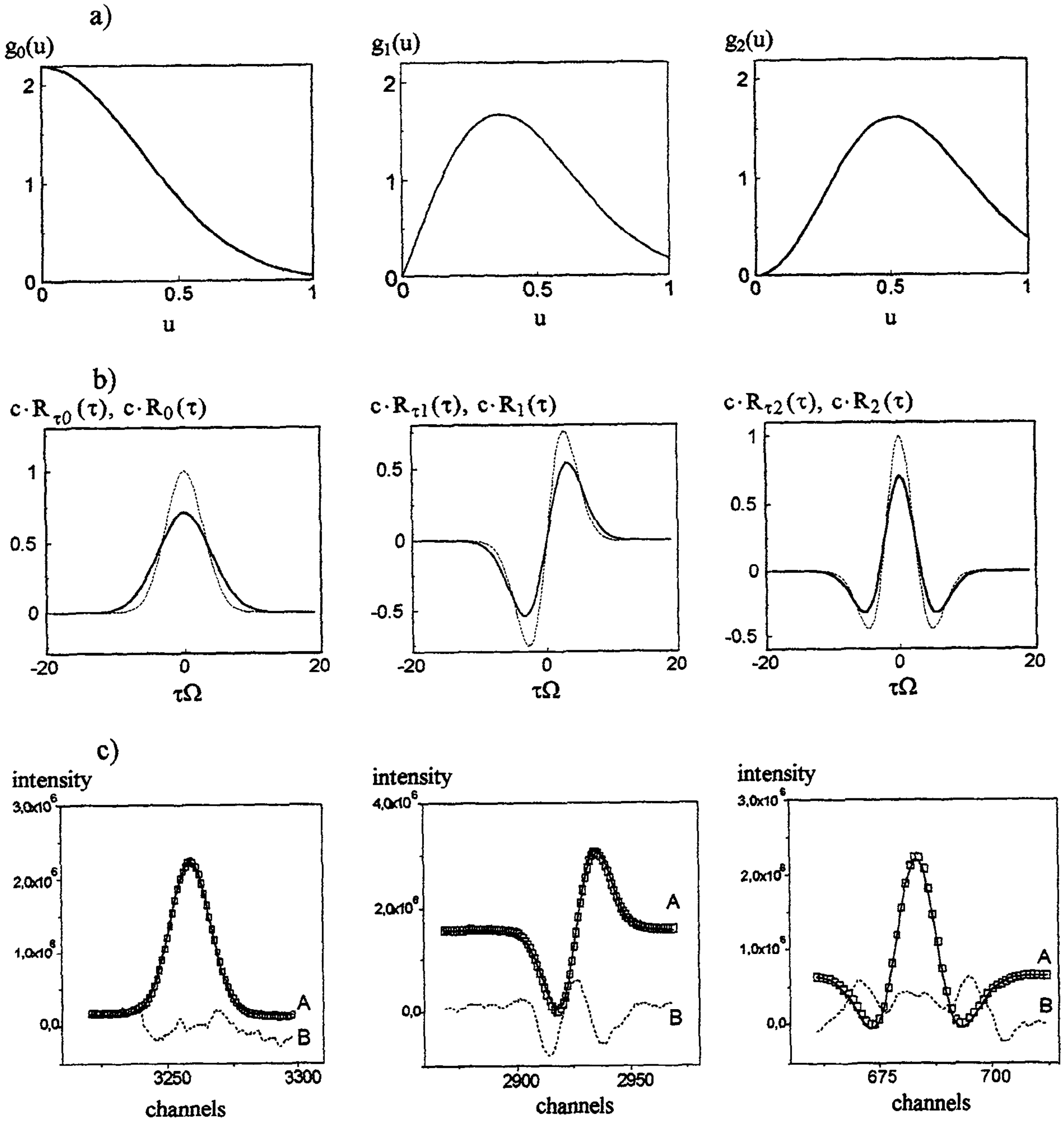

Fig. 4: Main rotor frequency programs and resulting peakshapes (b.) = calculated, c.) = measured and fitted) used at FSS

The development of Rietveld refinement software is underway; it has to be adopted to the $g_1(u)$ rotor frequency program
Ancillary equipment that has been developed and used is a stress rig to apply tensile forces in-situ to the samples measured, which includes a calibrated strain gauge setup to check the applied forces and an x,y,z,ω_1,ω_2 positioner, which is PC-controlled and can be adapted to different experimental tasks. A furnace, which will allow to measure specimens under uniaxial stress while heated, is under construction.

Experiences and experimental results

The predominant frequency program used for the strain measurements is the $g_1(u)$-window, which produces the first derivative of a Gaussian as peak shapes. The reason for trying other than Gaussian shapes was the basic idea that the contribution of low frequencies beginning from zero is the „DC component" and long-period „AC components", which have no or at least negligible influence on the peak positions and can therefore be cancelled to the advantage of more measuring time for the spectrum. The calculations [3] have then shown that for a given period of measurement time the „first derivative Gaussian" peak shape mode gives the highest precision for the peak position and is therefore the most suitable form for the type of measurements the FSS spectrometer was designed for.
An example is given in the next figure, which shows the measured shift in peak position in a tensile stress experiment on TMCP steel, a material used in offshore applications.

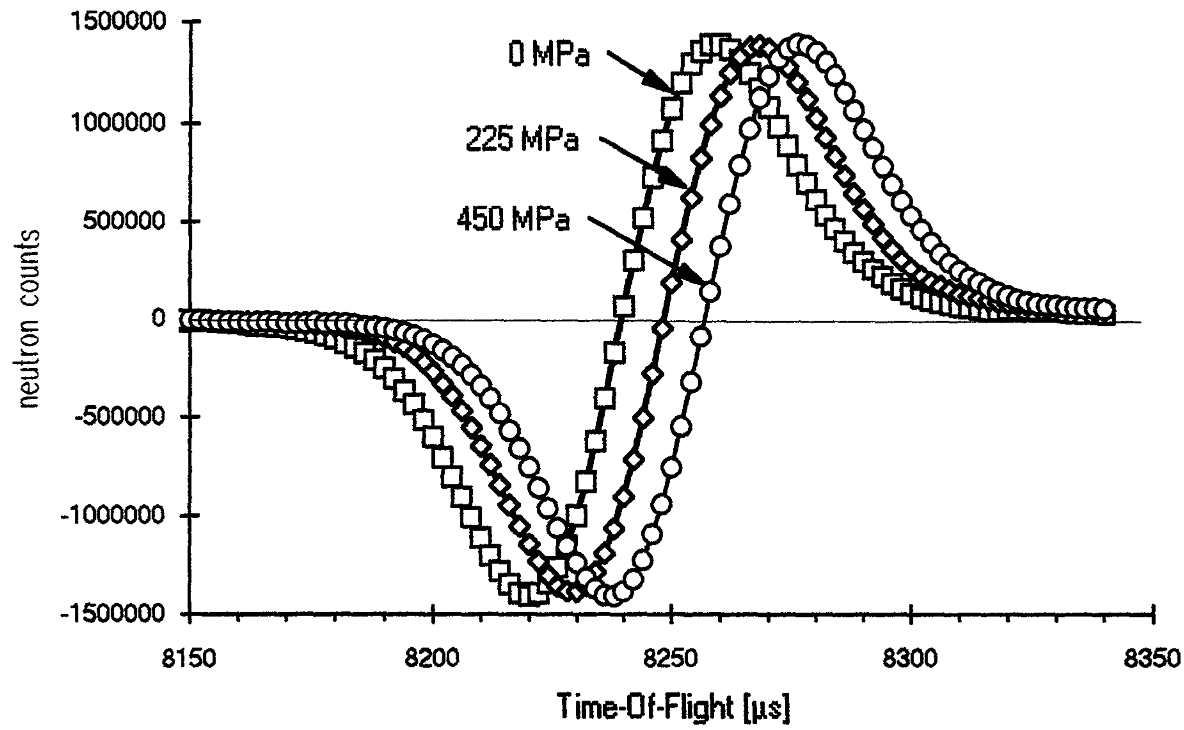

Fig. 5: Measured peakshift in a uniaxial-tension experiment (first derivative of Gaussian)

A fixed phase relationship between the opening of the neutron chopper and the optical pickup must be guaranteed. The experience with FSS has shown that the peak parameters „position" and „width" depend on the stability of this phase. Deviations from the ideal phase angle result in an asymmetry of the peak shape. Measurements done with very high statistical accuracy show, that a phase angle of zero degrees strictly can only be achieved for a certain part on the neutron diffraction spectrum, since the flighttime of the neutrons between the rotor and the stator already in principal causes phase differences: for the fast neutrons, the full opening is not yet reached, while the slow neutrons arrive at the stator only after the maximum opening. In practise the asymmetry due to this effect may be tolerated, if the distance between rotor and stator is less than 5 mm. In order to appreciate the influence of phase errors, calculations have been made, which resulted in a fitting function, which takes these errors into account, so that they can be eliminated in the peak position determination [5].

The following figure is included here as an example for the simultaneous determination of reflections from different phases, a task for which time-of-flight experiments are most suitable. The specimen was a composite steel containing both fcc and bcc structures.

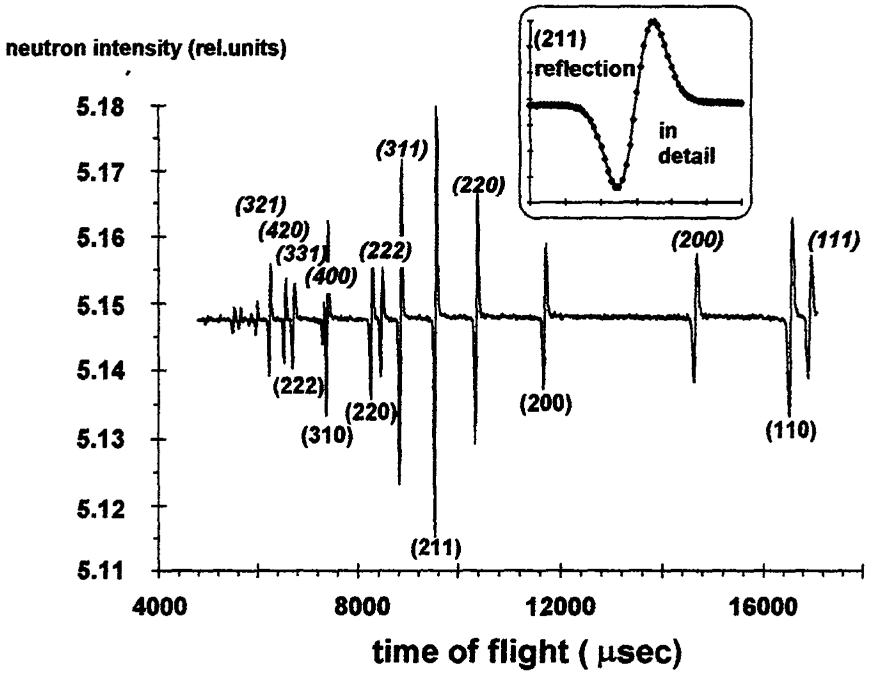

Fig. 6: Simultaneous measurement of α- and γ- phase in composite steel

Our research program concentrates on the validation of finite-element calculations of residual stresses by nondestructive neutron diffraction, in order to arrive at reliable theoretical predictions which can be verified experimentally at a few representative points. The following figure shows the results of measurements on a quenched steel cylinder of 100 mm length and 30 mm diameter, which was a non-transforming austenitic material. Residual stresses in this case are pure thermal stresses. The specimen and the FE calculations were supplied by Prof.Dr.Hougardy of the Max-Planck-Institut für Eisenforschung in Düsseldorf. Calculations and measurements are in good agreement.

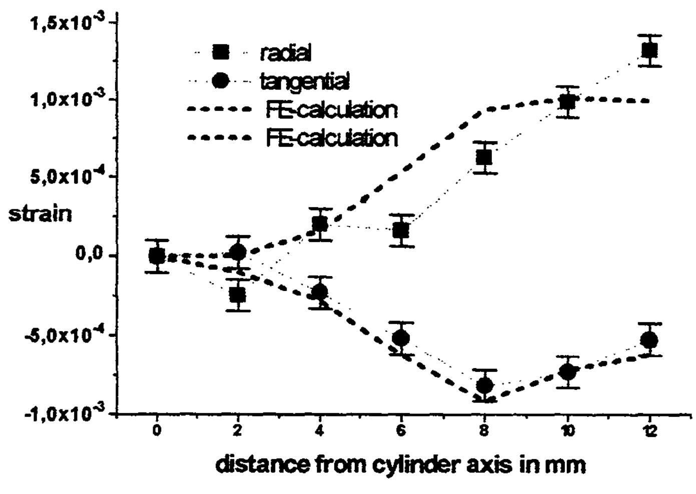

Fig. 7: Radial and tangential strains in a quenched steel cylinder, compared to FE calculations

An experiment on quenched cylinders which contain phase transformation stresses due to the formation of martensite is ongoing.

The last figure shows the result of a recent experiment on a steel plate, which was plastically deformed by 10% and released afterwards. The residual stresses induced by this plastic deformation cause compression in the direction of the tensile force applied and tension in the two orthogonal directions. By plotting the strains as a function of the orientation factor Γ, the crystalline elastic anisotropy is accounted for. The Reuss model predicts a linear relation between collinear stress and strain for the plain strain case:

$$\varepsilon_{\|} = \sigma_{\|} \cdot (s_{11} - 2\,S \cdot \Gamma)$$

where $$S = s_{11} - s_{12} - \frac{s_{44}}{2}$$

and $$\Gamma(hkl) = \frac{\left(h^2k^2 + h^2l^2 + k^2l^2\right)}{\left(h^2 + k^2 + l^2\right)^2}$$

A uniaxial tensile experiment therefore results in a linear relation, when the strains derived for different peaks (with Miller indices hkl) are plotted against $\Gamma(hkl)$. On the other hand, from the slope of this linear curve, the existance of tensile or compressive residual stresses may be concluded. As shows the formula, a positive slope indicates tension, while a negative slope indicates compression. The errors on the strain values in the following figure are below 1×10^{-4}.

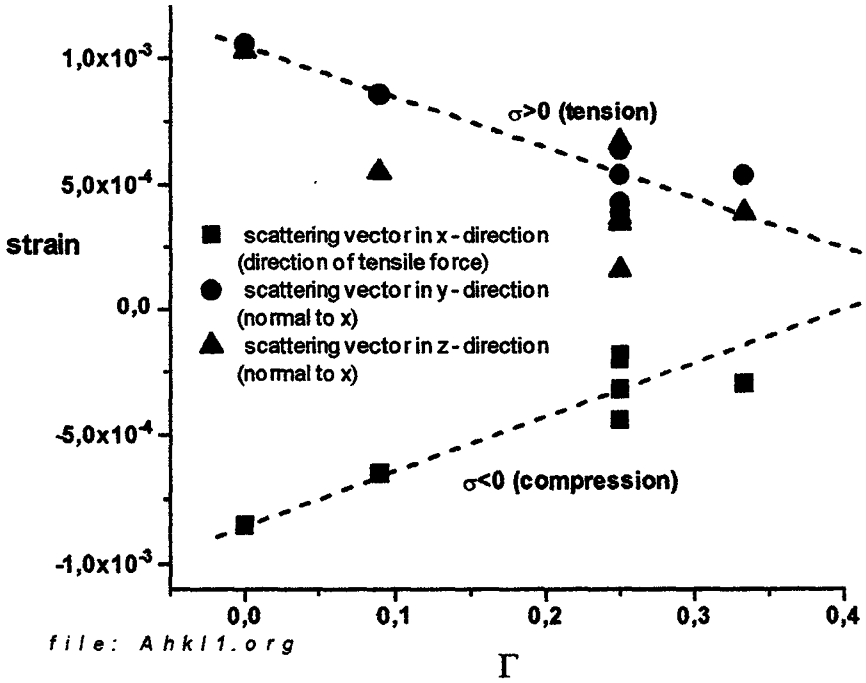

Fig. 8: Plain residual stresses in a plastically deformed steel plate

The specimen was investigated under seven different aspect angles, in order to arrive at seven independent linear relations to be able to calculate the full strain tensor. The analysis of this experiment is not yet completed.

Summary

The FSS RTOF- diffractometer, which was designed and optimized for residual stress measurements, has proven to be a reliable precision instrument. Examples of recent experiments are given.
When residual stress states in polycrystalline materials are to be measured, neutron time-of-flight spectrometry offers a number of unique advantages:
Since a large number of reflexions can be observed simultaneously, which originate from the same scattering volume, this method is especially useful in the investigation of multiphase materials like modern MMC's (metal matrix composites). By choosing a scattering angle of 90°, the optimal scattering volume is defined. A fixed geometry simplifies the setup of different sample environments considerably: heavy loads, furnaces or cryostats can be installed within short times.
With increasing operating experience it is likely, that progress in even better neutron untilization can be expected.

Acknowledgements

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