

BRAGG-EDGE TRANSMISSION AS AN ADDITIONAL TOOL FOR STRAIN MEASUREMENTS

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Solid state information from coherent neutron scattering can be extracted from transmission experiments on polycrystalline materials. The transmission technique has been applied to dynamic structural and phase change investigations and will be developed to cover the fields of strain mapping and structural tomography.

Keywords: Neutron diffraction; Transmission; Transient phase transformations; Dynamic phenomena

INTRODUCTION

A white thermal neutron spectrum, when transmitted through polycrystalline material, is considerably distorted by Bragg edges, which correspond to a scattering angle $2\theta = 180^\circ$. Since all diffracted neutrons are scattered out of the direct beam, transmission edges build up rapidly. High peak-intensity neutron bursts can be detected using the current-mode technique, opening the possibility to do both transient and

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stroboscopic measurements. This new method is relevant to different research areas, like transient phase transformations, melting and solidification, dynamic stress influence at impact or materials under extreme external conditions. An advantage compared to ordinary diffraction is the easier setup of the experiment due to the fact, that the sample can be placed anywhere between the source and the detector. The Bragg-edge method may become a unique tool for the investigation of structural phase-transitions, crystal-structure tomography and strain mapping. Recent developments will also have impact on the determination of the stress-free reference lattice plane distances.

NEUTRON TRANSMISSION DIFFRACTION

Low-energy coherent neutron scattering from polycrystalline materials can be performed with highest resolution in neutron time-of-flight transmission geometry which corresponds to backscattering, with the only difference that the detectors are placed in the forward direction (Fig. 1). Instead of Bragg peaks so-called Bragg edges are observed. Bragg edges represent the sudden discontinuity in the scattering cross section, whenever certain lattice planes are excluded from scattering since the Bragg condition $\lambda_{hkl} = 2 \cdot d_{hkl}$ (for $\theta = 90^\circ$) is no more fulfilled. At a powerful pulsed neutron source like LANSCE in Los Alamos, information about the crystal structural properties can be attained from every neutron pulse. Since each pulse contains the full thermalized neutron spectrum, a large number of Bragg reflections can be observed simultaneously.

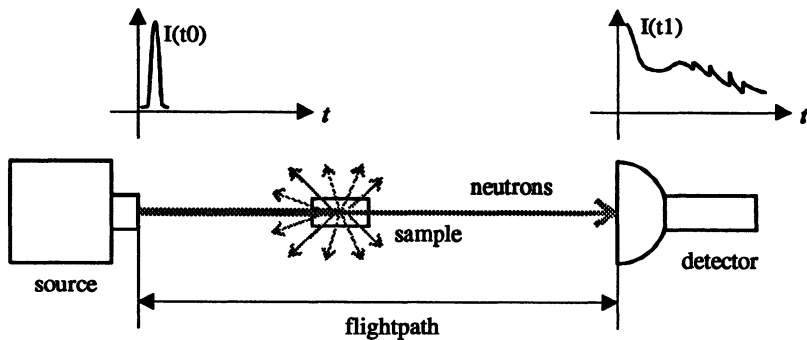


FIGURE 1 Schematic of the transmission setup (from Bless, 1997).

Real-time investigations of changes in the material properties which influence the position, width and height of the Bragg edges (like phase changes, anisotropic strain development or thermal expansion) can be observed with a time resolution between 100 μs and several ms. The measured heights must be normalized with respect either to the initial height (for phases with decreasing volume fractions) or to the final height (for phases with increasing volume fractions). One may also “calibrate” the measurements by measuring phase volume fractions in specimens before and after heat treatment using conventional neutron powder diffraction. First experiences with this new method have already been reported (Priesmeyer *et al.*, 1989; Bowman *et al.*, 1991; Priesmeyer, 1992), but further progress has been made concerning single-shot resolution. A new transient recorder capable of registering successive single-shots over minutes has been designed and used to investigate thermal expansion and phase changes in steel, while at the same time a temperature measurement was made by evaluating the Doppler-broadening of the 4.9 eV neutron resonance in a gold foil attached to the steel specimen.

The spectral intensity ratio between “sample-in-beam” and “open beam” is called transmission and described by

$$T = \exp\{-N \cdot \sigma_{\text{coh.-el.}} - n \cdot x \cdot (\sigma_{\text{inc.}} + \sigma_{\text{abs.}})\} \quad (1)$$

where N is the number of unit cells/ cm^3 and n is the number of atoms/ cm^3 , x the thickness of the specimen, $\sigma_{\text{coh.-el.}}$ the coherent-elastic cross section, $\sigma_{\text{inc.}}$ the incoherent cross section and $\sigma_{\text{abs.}}$ the absorption cross section.

The coherent-elastic scattering cross section is given by

$$\sigma_{\tau}(\lambda) = \frac{\lambda^2 \cdot z_{\tau}}{2 \cdot V_{\text{cell}}} \cdot |F(\tau)|^2 \cdot e^{-2W} \cdot d_{\tau} \quad \text{and} \quad \sigma_{\text{coh.-el.}}(\lambda) = \sum_{\tau \text{ for } 2d_{\tau} > \lambda} \sigma_{\tau}(\lambda) \quad (2)$$

where the unit-cell structure factor

$$F(\tau) = \sum_i \bar{b}_i \cdot \exp(2\pi i \tau \cdot d) \cdot \exp(-2W_i) \quad (3)$$

comprises the nuclear properties of the scatterer, crystal structure selection rules and multiplicities as well as thermal effects, described by the Debye–Waller factor.

THE PRECISION LIMITS OF BRAGG-EDGE PARAMETERS

In the field of residual stress assessment using neutron diffraction, lattice spacings must be determined with a error of less than 10^{-4} . Residual stress states exist in components even if they are not subjected to external load. They develop in a very complicated, highly coupled thermal/metallurgical/mechanical process during welding, casting or forging or as a result of other manufacturing processes. Recent research (Priesmeyer *et al.*, 1994) on the development of residual stresses includes their calculation, for which certain material constants are important to know. These include the time development of the buildup of incompatible phases as well as the interrelation between phase transformation kinetics and internal stress, so-called transformation strains.

The relative reflex intensities yield information on phase volume fractions (Meggers *et al.*, 1994a,b). In transmission experiments the high precision attained in ordinary backscattering geometry concerning profile parameters of Bragg edges is preserved. Two-dimensional macro- and microstrain can be accurately measured. Figure 2 shows a section of a typical current-mode single-shot transmission spectrum for α -iron (bcc) of 2.5 cm thickness, collected at 10 m detector position within 7 ms. In order to improve the statistical accuracy, multiple single-shots can be accumulated.

Edge height, slope and position are extracted from the transmission spectra. The shape of Bragg edges is generally a convolution of the contributions of the natural and mosaic spread of lattice spacings, the resolution function and strain gradient influences. An evaluation procedure similar to conventional Rietveld refinement is being developed. As a first attempt to use existing software, transmission spectra were differentiated. The resulting peaks were fitted as single peaks, while differentiation tends to increase noise and distort the continuous background (Fig. 3).

The parameters describing the resolution function are also fitted. Accuracies achieved for peak positions were about $4 \cdot 10^{-4}$ for a measuring time of 250 s, but can further be increased.

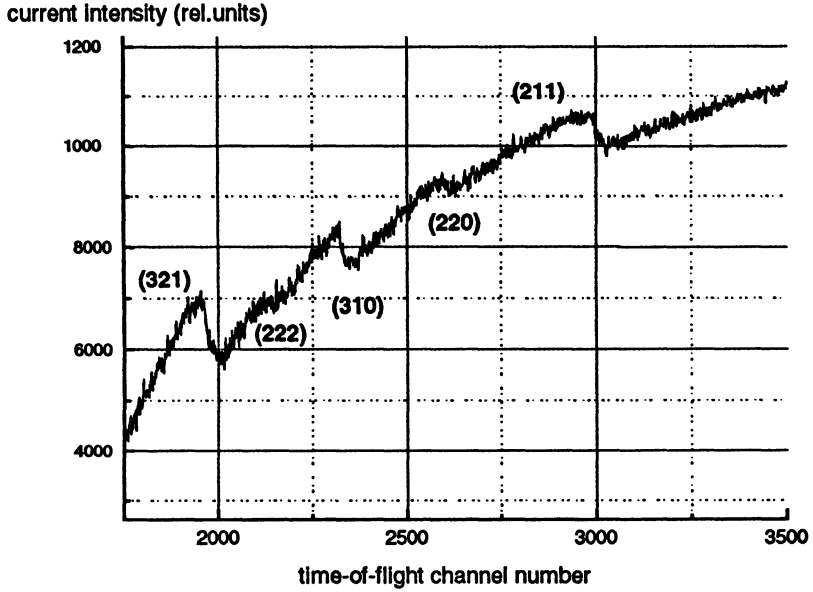


FIGURE 2 Partial single-shot spectrum at 10 m flight-path.

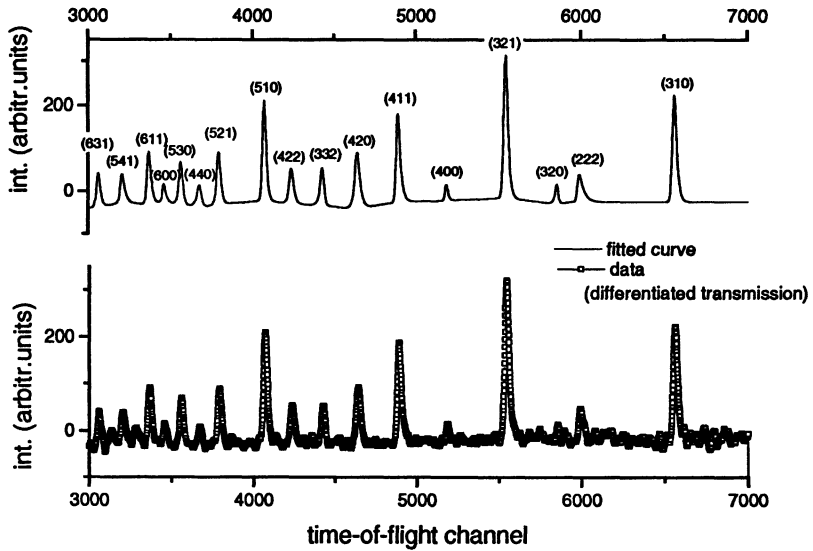


FIGURE 3 Differentiated time-of-flight transmission spectrum (bottom) and fit (top) of bcc-iron (from Vogel, 1996).

TEMPERATURE MEASUREMENT BY DOPPLER-BROADENING OF NUCLEAR RESONANCES

Many of the possible applications of single pulse transmission (like solid state phase changes, development of transformation stresses, thermal stresses during cooling after welding) require the knowledge of the temperature of the specimen. It is known that Doppler-broadening of low-lying neutron resonances can be exploited to determine temperatures. A series of experiments has been made using ferritic steel together with a 17 μm thick gold foil. Figure 4 shows a current-mode detection spectrum containing both edges and a resonance.

Both resonance Doppler-broadening and Bragg-edge shift are functions of the temperature of a specimen. While the position of the Bragg edges is influenced by the combined effects of thermal lattice expansion and strain a resonance is only subjected to thermal Doppler-broadening. In technological applications it may not be possible to mix dopant and alloy without changing the metallurgical properties of the latter. Details of spectral differences due to temperature are shown in Figs. 5 and 6.

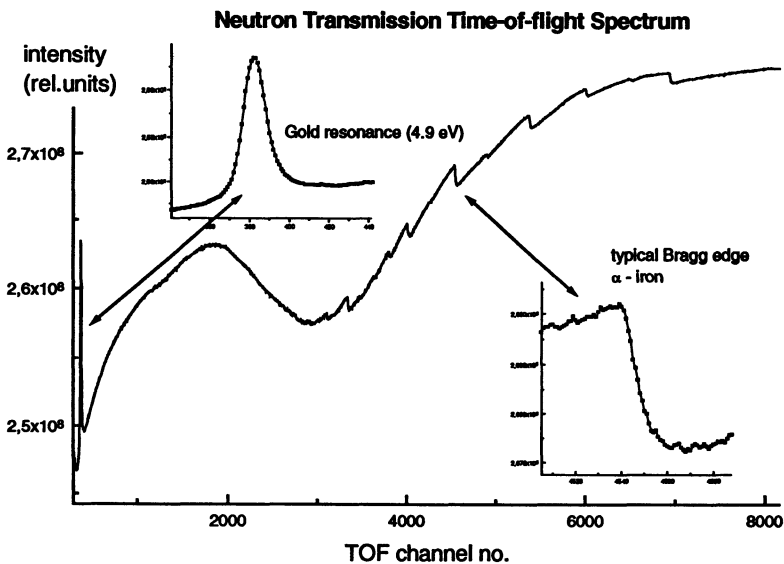


FIGURE 4 Simultaneous current-mode recording of the 4.9 eV gold resonance and several edges of bcc-iron.

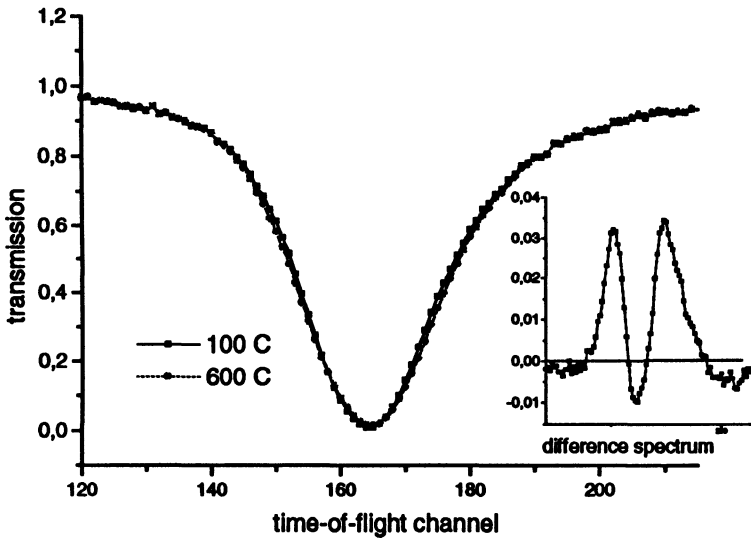


FIGURE 5 Resonance broadening between 100°C and 600°C.

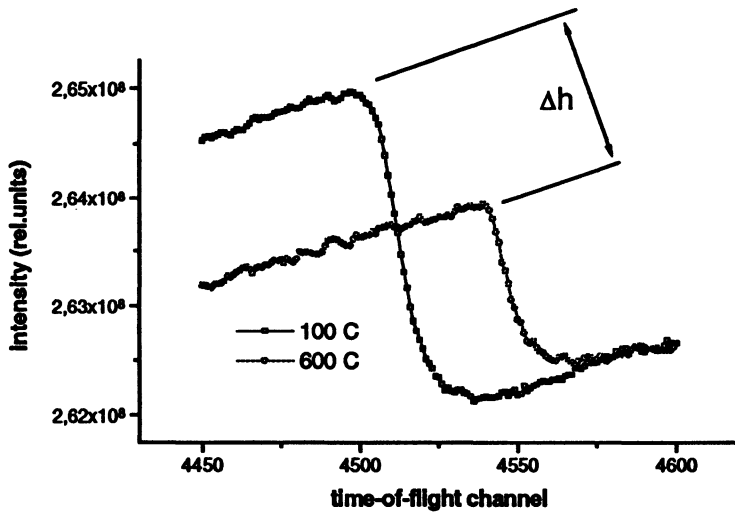


FIGURE 6 (321)-reflection in bcc-iron, shifted in time-of-flight by thermal expansion and reduced in height by Δh according to the Debye–Waller factor.

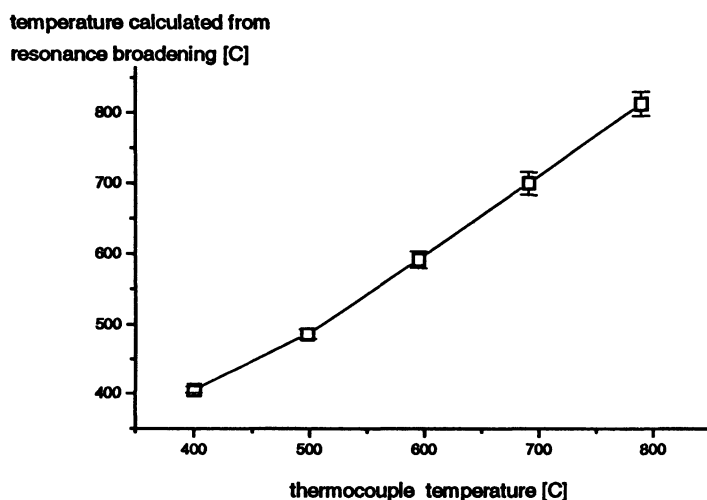


FIGURE 7 Comparison between temperatures derived from thermocouple and resonance broadening measurements (from Stalder, 1996).

Bragg edges may vary in height for two reasons: the Debye–Waller factor will decrease the diffracted intensity with increasing temperature and diffusion-controlled phase changes may reduce the volume fraction of a certain phase. In steel up to 600°C intensity reduction can be fully attributed to the Debye–Waller factor. The principle feasibility of the method has been demonstrated by a series of measurements from room temperature to 1000°C. A better knowledge of the spectrometer resolution function and of the open beam intensity will be needed to arrive at shorter times. In order to derive temperature values from resonance broadening, the method described by Mayers *et al.* (1989) was used. Resonance shape fitting will be used later.

PIEZOELECTRIC STRAIN GAUGE FOR STATIC AND DYNAMIC LOAD

A stress rig has been developed suitable for transmission experiments. In order to adopt it to a pulsed neutron source, a special device was designed on the basis of an annular piezoelectric actuator (Bless, 1997, patent pend.).

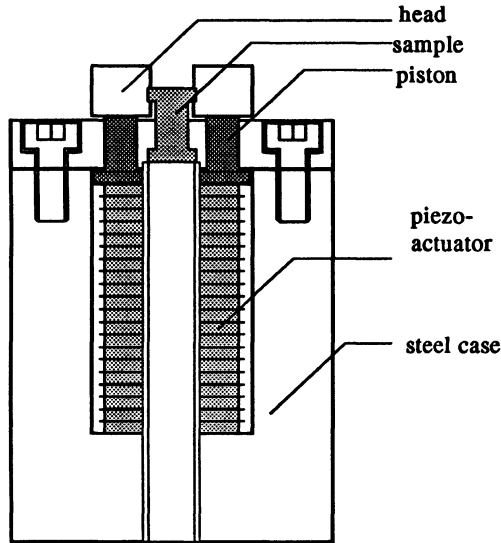


FIGURE 8 Schematic of the piezoelectric uniaxial stress rig (from Bless, 1997).

The outlines are shown in Fig. 8.

The rig can be triggered by the neutron source and the load be applied at certain time intervals; different strain rates can be used to investigate strain-rate dependent effects. The dynamic features were tested using the setup shown in Fig. 9.

For a mechanically prestrained steel specimen, the neutron diffraction result vs. strain gauge comparison is given in Fig. 10.

BRAGG-EDGE TRANSMISSION SPECTROSCOPY TO DETERMINE THE STRESS-FREE REFERENCE LATTICE SPACING d_0

Under the assumption of a two-dimensional stress state (e.g. thin specimens), Bragg-edge transmission measurements may offer an opportunity for *in situ* measurements of d_0 . Strain in the direction determined by the angles ϕ and ψ can be described by the following well known

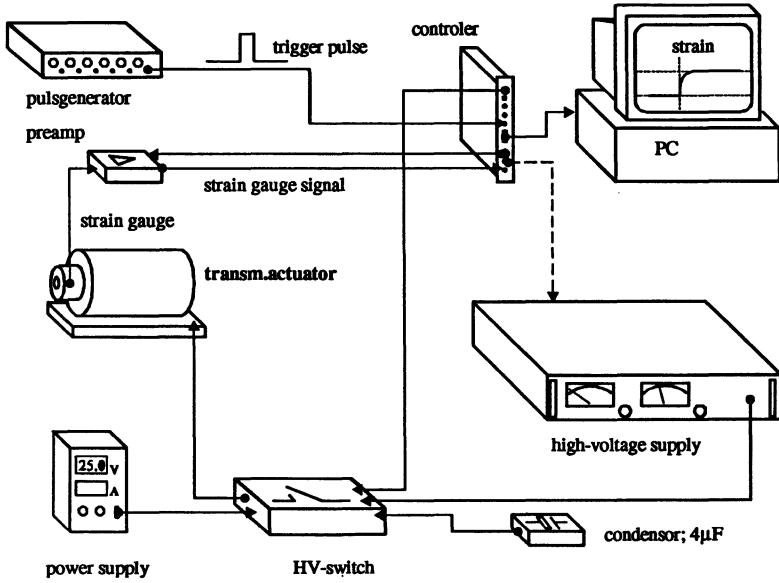


FIGURE 9 Setup to characterize the dynamic features of the transmission stress rig (from Bless, 1997).

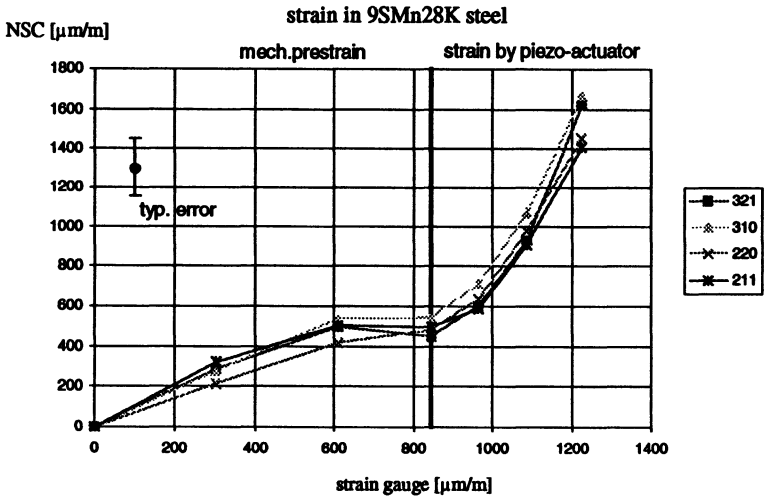


FIGURE 10 Strain in steel specimen: mechanical prestrain (left side), piezoelectrically applied strain (right side) (Bless, 1997).

expression:

$$\begin{aligned} \frac{d_{\phi,\psi} - d_0}{d_0} = & \frac{1 + \nu}{E} \cdot \{ \sigma_{xx} \cdot \cos^2(\phi) + \sigma_{xy} \cdot \sin(2\phi) + \sigma_{yy} \cdot \sin^2(\phi) - \sigma_{zz} \} \\ & \cdot \sin^2(\psi) + \frac{1 + \nu}{E} \cdot \sigma_{zz} - \frac{\nu}{E} \cdot \{ \sigma_{xx} + \sigma_{yy} + \sigma_{zz} \} \\ & + \frac{1 + \nu}{E} \cdot \{ \sigma_{xz} \cdot \cos(\phi) + \sigma_{yz} \cdot \sin(\phi) \} \cdot \sin(2\psi) \end{aligned} \quad (4)$$

Assuming biaxial stress ($\sigma_{zz} = 0$) and taking $\phi = 0$, the equation further simplifies to:

$$\frac{d_{\phi,\psi} - d_0}{d_0} = \frac{1 + \nu}{E} \cdot \sigma_{xx} \cdot \sin^2(\psi) - \frac{\nu}{E} \cdot \{ \sigma_{xx} + \sigma_{yy} \} \quad (5)$$

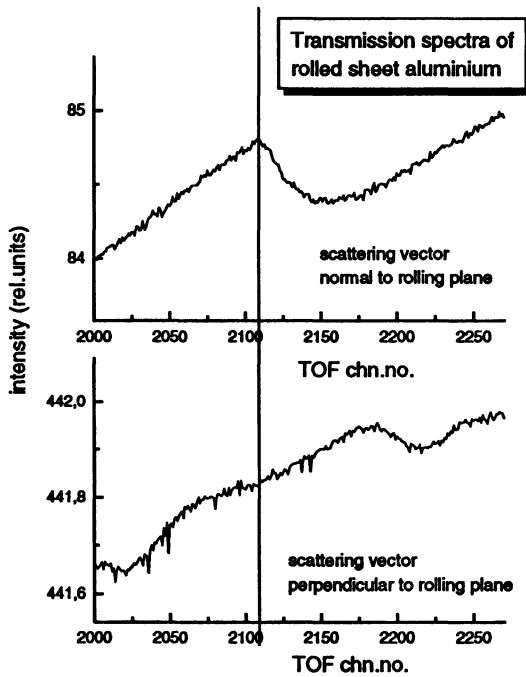


FIGURE 11 Neutron transmission near the (311)-edge in aluminium for two orthogonal positions of the specimen.

The angle $\psi = \psi^*$ for which $d_{\psi^*} = d_0$ may be calculated from the following relation:

$$\sin^2(\psi^*) = \frac{\nu/E}{(1 + \nu)/E} \cdot \left\{ 1 + \frac{\sigma_{yy}}{\sigma_{xx}} \right\} = \frac{\nu/E}{(1 + \nu)/E} \cdot \left(1 + \frac{m_y}{m_x} \right) \quad (6)$$

where m_x and m_y are the slopes of the $d(\sin^2(\psi))$ curves, measured for two orthogonal directions $\phi = 0^\circ$ and $\phi = 90^\circ$. A plot of measured d -spacings vs. $\sin^2(\psi)$ for any two ϕ_A and $\phi_A + 90^\circ$ will lead to the relevant slopes and the angle $\psi = \psi^*$.

BRAGG-EDGE TRANSMISSION AND TEXTURE

A recent measurement of the transmission of rolled sheet aluminium at LANSCE has shown how texture can considerably change the shape of a neutron transmission spectrum (Fig. 11). Evaluation of these findings is still in progress. In general, it should be possible to derive information about the textured state. Since for in-depth strain profiling the neutrons penetrate a certain material layer, consequences for the interpretation of strain data in textured materials may also be anticipated.

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