ESRF	**Experiment title:** **Experimental studies of an x-ray standing wave between long-distance separated crystals**	**Experiment number**: MI-269
Beamline: BM5	**Date of experiment**: from: 26/8/98 to: 1/9/98	**Date of report**: 15/2/99
Shifts: 15	**Local contact(s)**: Dr Alexei Souvorov	*Received at ESRF:*

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Report:

The experiment was performed on the European Synchrotron Radiation Facility (ESRF), BM5 (BL-10) beamline, Grenoble, France. A primary, tunable double-crystal Si(111) monochromator selected synchrotron radiation energy in the vicinity of 9.1 keV (λ = 1.36 Å) from the bending-magnet source. The second, Si(400), monochromator was used, in a non-dispersive set-up, to ensure the highest order of collimation and monochromatization of the beam. The origin of the previously observed intensity fringes in Si(555) 90 degree reflection was established by a measurement of the *transmitted* beam using a PIN diode detector. It was found that, due to the highest degree of symmetry of the <111> crystallographic axis, the multi-beam diffraction fringes occur at similar yet not quite the same angular position of the crystal at different azimuthal orientation of the crystal. This fact has been proven by extensive rotation of the Si(555) crystal around its normal in 0-55 degree range (full symmetry around the <111> axis is 60 degrees). Fringes occurring due to reflection of the fundamental harmonic disappear with the azimuthal rotation, however, fringes occurring due to reflection of higher radiation harmonics remain in the diffraction profile with slightly changed angular locations.

Subsequently, the radiation energy (λ = 1.43 Å) was selected to allow the (800) reflection, at a Bragg angle of 90 degrees, from a InGaAs/GaAs(100) crystal that was mounted at the second axis of the BM5 triple-crystal diffractometer. A novel detector arrangement [1] was used to measure the fluorescence scattering from a nickel-alloy plate placed in the incident beam. In this arrangement a NaI(Tl) scintillator-photomultiplier detector collected fluorescence scattering produced by the back-scattered beams hitting the 1 mm thick nickel-alloy plate. The absorption edge of nickel is below the selected radiation energy used for the data collection. Since the fluorescence yield is much higher than the air scattering contribution, the detected intensity of the back-reflected peaks was very large compared to the air scattered background.

The experimental objective was to measure the intensity profiles of back-reflected beams near the 90 degree Bragg reflection from a thin surface layer while the substrate-reflected beams are "switched off". Since the layer lattice spacing is slightly bigger than the substrate lattice spacing (due to the impurity presence in the layer alloy), it was suggested to scan the incident radiation energy through the regime when the substrate stop to reflect while the layer is still reflecting. A preliminary study at the Monash University laboratory using MoK characteristic line (λ = 0.709 Å) showed that the layer to substrate peak ratio was about 55%. A brief scan around the (400) Bragg reflection at the *same radiation energy*, used for the (800) 90 degree reflection - λ = 1.43 Å, showed this ratio to be about 20%. Experimental diffraction data collected near the 90 degree reflection showed a ratio to be about 1%. This was quite unexpected because the photoelectric attenuation has not changed with the change of the Bragg diffraction order from (400) to (800). Apparently, the existing theory for the *extinction depth* at a 90 degree Bragg reflection (which has been developed for a semi-finite crystal) requires further development for diffraction on thin crystals.

The results obtained show a new possibility for direct measurements of diffraction profiles from a thin crystalline layer. It is impossible to avoid a contribution of the substrate rocking-curve 'tails' into the measured Bragg-diffraction layer intensity profile using a 'standard' (far from 90 degree) Bragg reflection. The present research proves the idea that it is possible to completely "switch off" the substrate-reflected beam while the layer-reflected beam is still measurable.

This pioneering experiment has both, fundamental (development of x-ray diffraction dynamical theory on thin crystals near 90 degree Bragg reflection) and applied (direct precise quantitative characterisation of thin films) implications.

The present studies prove that direct/back-reflected beam interference phenomena can be investigated without expensive, extremely alignment-sensitive, x-ray interferometers. Sensitivity of this novel experimental set-up to the coherent/incoherent interaction of the x-ray wave scattered by the substrate and the layer warrants further research on the single- or bi-crystal diffraction intensity profile studies.