ESRF	Experiment title: First KOSSEL investigations by means of hard X-rays at ESRF	Experiment number: MI-I 85
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Names and affiliations of applicants (*indicates experimentalists):

Hans-Jurgen Ullrich (a), Jurgen Bauch (a), Jens Brechbuhl (a), Thomas Buslaps (b) and Andreas Freund(b)

(a) Institut fur Werkstoffwissenschaft der TU Dresden, Mommsenstr. 13, D-01 062 Dresden (Germany)

(b) ESRF Grenoble (France)

Report:

1. Aim of the experiment

In 1992 the first synchrotron excited KOSSEL-patterns were produced at DESY HASYLAB in Hamburg, applying X-ray films and white beam spectrum. The exposure time was in order of minutes. Using image plate , we succeeded to reduce the exposure time by one order.

So far, the investigated materials are composed of elements in the region of Cu to Mo, in the periodic table. For this region, the intensity spectrum of the Beam line L at DORIS HASYLAB, where we have been performing the experiments up to now, is well suited. The aim of our experiment at ESRF was to produce KOSSEL-patterns with shorter X-ray wavelength, e.g. Ku-lines of substances with an atomic number > 50. To perform such experiments, we were recommended to use the monochromatic Beam line ID15B with the KOSSEL camera, which we developed in 1994. The excitation of hard fluorescence X-rays in the specimen , by means of the monochromatic spectrum of ID15B was so weak, so that a useful KOSSEL pattern could not be obtained with in the short exposure time and suitable image plates were not available. To achieve exposure times in order of a minute, we used the opportunity to measure at D5 with a white beam spectrum.

2. Experiments

With the provisional experimental set-up, KOSSEL patterns with low contrast for InSb (λ-In-Kα =0,0512 nm, λ-Sb-Kα =0,047 nm) and W (λ-W-Kα=0,0209 nm) could be produced. The back-reflection KOSSEL patterns of InSb and W had sharp lines and LAUE spots. Because-of the short wavelength of the- K-radiation, the KOSSEL

cones have wide opening angles and the KOSSEL lines are almost straight curves, analogous to the KIKUCHI lines of electron diffraction.

3. Data evaluations

To evaluate the crystallographic information, a computer program has been developed. Figure 1 illustrates the simulation of a InSb KOSSEL pattern (figure 2). The [001] axis of the crystal lattice goes through the centre of the film , i.e. the (001) net plan is parallel to the film. In InSb and W more than 1000 KOSSEL-lines could appear over the specimen surface in back reflection. Even though only some lines could be recorded on the film , the information was enough to determine the crystallographic orientation with high accuracy in reference to the specimen co-ordinate system $\Delta\alpha \pm 0{,}05°$ and to measure the lattice parameter with an accuracy of $\Delta a/a=10^{-3}$ (because high disperse reflexes could not be observed).

4. Comparison to the proposal

Despite the provisional experimental set-up it was possible to obtain KOSSEL-lines applying high energy X-rays in back-reflection geometry. The first experiment shows that **Kα-lines** of materials with high atomic number can be used to characterise a micro-volume of a given specimen.

5. Conclusion

In order to improve the quality of these preliminary results the experiment should be performed with a dedicated equipment (for example our KOSSEL camera specially developed for synchrotron beam excitation) using a white beam of high energies and higher flux. To the dedicated KOSSEL camera belongs a tv-microscope, which enables searching a spot on the specimen to investigate a local volume of compact sample.

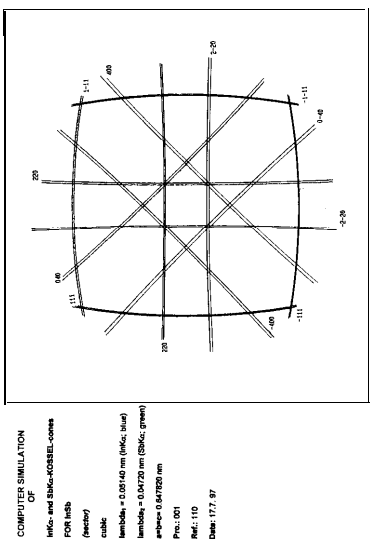

fig. 1

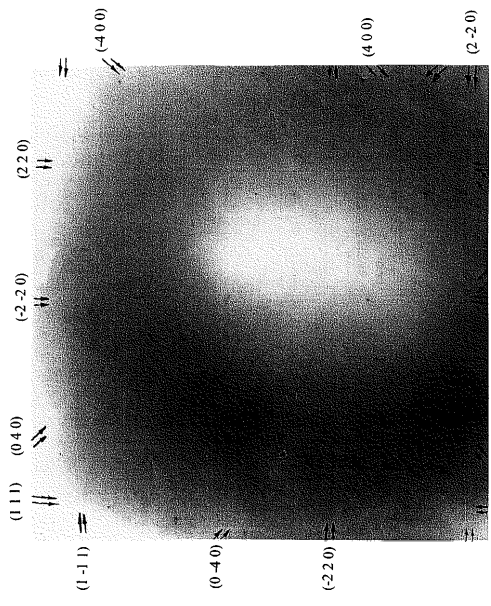

fig. 2