

**Experiment title:**

Non-equilibrium solidification of undercooled metallic melts

**Experiment****number:**

HS-488

**Beamline:****Date of experiment:**

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12

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**Report:**

The combination of Energy dispersive X-Ray diffraction with electromagnetic levitation technique allows direct investigations of the structure of undercooled melts and their solidification pathways. The binary Co-V alloy was chosen as a model system, because it shows phase competition of three different crystallographic phases, bcc, fcc and an intermetallic phase of tetragonal structure. The solidification pathway in the concentration range of 30 up to 50 at % V was investigated. Multi-recalescence steps of T-t profiles indicate a primary crystallisation of a metastable crystallographic phase. The Co-V samples were processed within an electromagnetic levitation chamber, especially designed for this kind of experiments. They were melted, undercooled and spontaneously solidified in a containerless state. Cooling rates in the order of some K/s were applied. The temperature was controlled by forced convection of cooling gas and measured with a two colour pyrometer in top view. The measurements of the temperature-time profiles are utilised to determine the undercooling (the difference between  $T_1$  and  $T_n$ , where  $T_n$  is the nucleation temperature). As

shown in the previous experiments, with the energy dispersive germanium detector available at ID09 the collection of a diffractogram of metallic melts with good statistics is possible. The integration times of the X-ray diffraction measurements ranged from 0.5 s to several minutes, sufficient for the Co-V system due to the long stability of the observed metastable phases. Figure 1 shows four energy dispersive diffraction spectra measured on a Co50V50 sample. The upper spectrum has been taken on the undercooled liquid in coexistence with the metastable bcc phase. The second spectrum shows the growing of the bcc phase. After further cooling down the sample the intermetallic  $\sigma$ -phase crystallises. Due to the heat of Crystallisation this reaction is accompanied by a remelting of the metastable bcc-phase and therefore only Bragg peaks of the  $\sigma$ -phase are visible in the third spectrum. The last spectrum shows the entirely crystallised sample. The combination of the electromagnetic levitation technique with energy dispersive X-ray diffraction gives access to investigate *in situ* the phase-selection process during solidification of undercooled Co-V melts, and gives direct evidence of primary crystallisation of metastable crystallographic phases in undercooled melts.

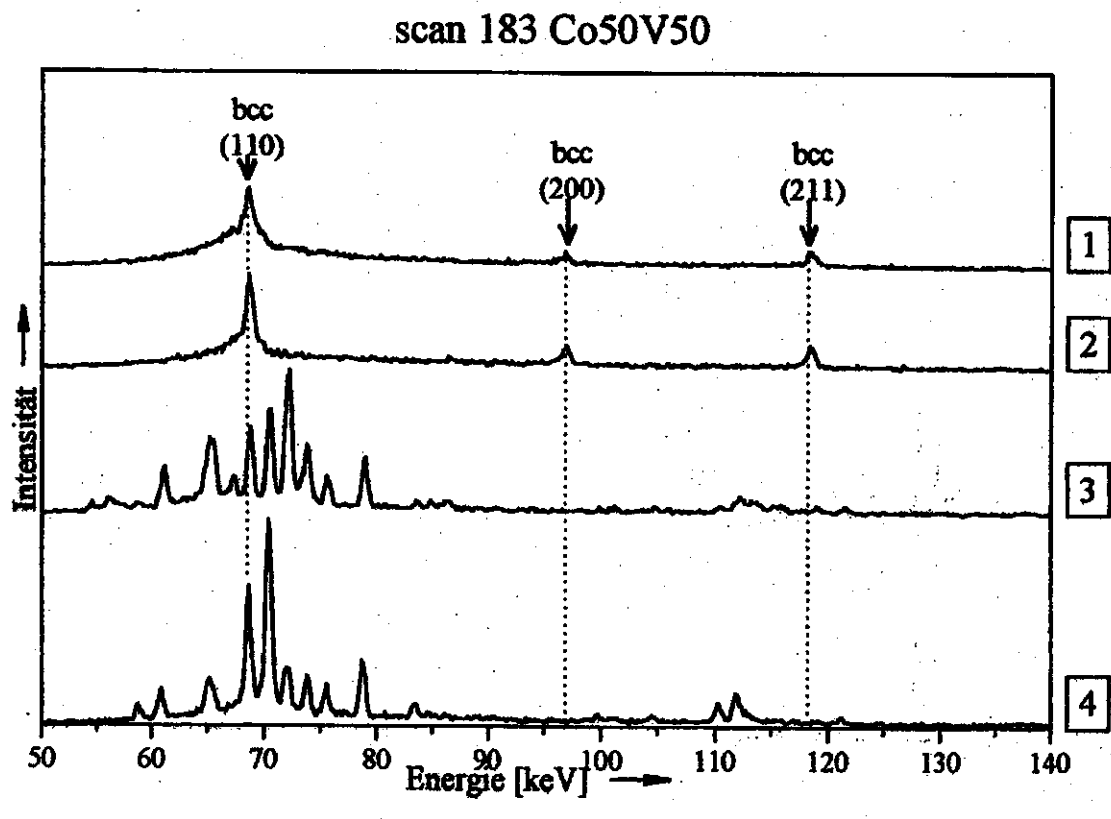


Fig. 1 EDXD spectra of a levitated Co50V50 sample