| ESRF | **Experiment title:** Non-equilibrium solidification of undercooled metallic melts | **Experiment number:** HS-178 |
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**Report:**

Undercooling is a necessary precondition for the non-equilibrium solidification of a metallic melt into solid metastable phases whose crystallographic structure is different from the stable modification. Containerless processing, like the electromagnetic levitation technique, is an effective way to achieve substantial undercoolings.

In the concentration range of 42 up to 60 at % V the system Ni-V shows a two- or three-step crystallization event upon undercooling. Multi-recalescence steps of T-t profiles indicate a primary crystallization of a metastable crystallographic phase, but direct evidence has still been missing.

In May 1997 we were successful in the direct observation of the crystallization process, combining our levitation facility with the energy dispersive x-ray diffraction (EDXD) device at beamline IDO9. : Located in an ultra high-vacuum chamber, the spherical samples (5 mm diameter) were processed within a high frequency levitation coil. The temperature was measured by a two-colour pyrometer and controlled by a variable flow of cooling gas. High

energy radiation with high intensity was applied to perform time resolved investigations on our levitated samples and to work at low scattering angles, laid down to the geometry of the levitation chamber. With the available energy dispersive germanium detector the collection of one spectrum within one second has been realized.

Figure 1 shows three energy dispersive diffraction spectra measured on a Ni42V58 sample at a fixed scattering angle $2\Theta$ of 5°. The upper spectrum has been taken on the undercooled liquid at a temperature of 1200°C, 60°C under the liquidus temperature. The middle one shows the diffraction pattern of the undercooled sample at 1174°C, partly solidified in the metastable bcc-phase. When the temperature of the sample is kept constant after the primary solidification, the stable $\sigma$ -phase crystallizes in a second reaction step after a time, ranging between some seconds and one minute. The second reaction is accompanied by a remelting of the metastable bee-phase and only the stable a-modification could be observed upon cooling to ambient temperatures.

The present experiments give direct evidence of primary crystallization of metastable crystallographic phases in undercooled melts proofed by synchrotron radiation measurements.

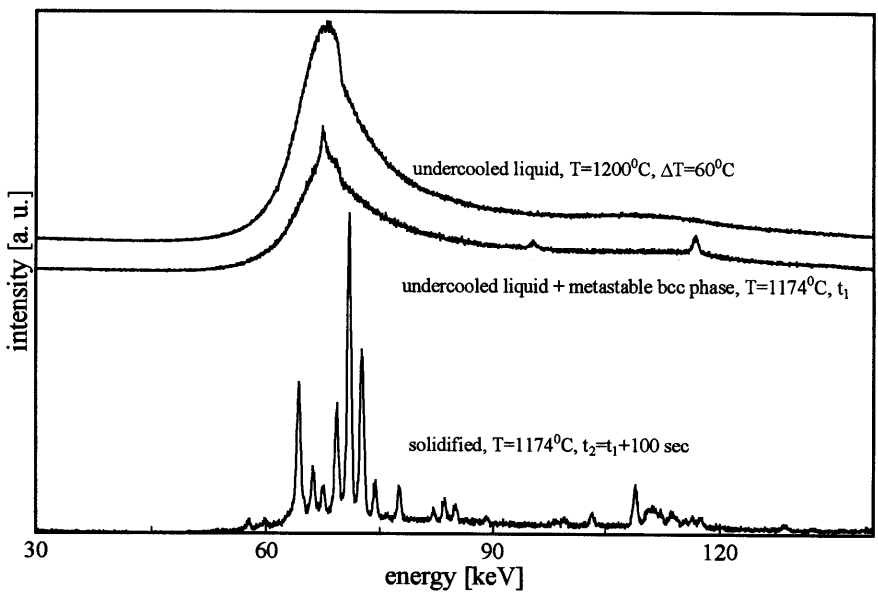


Fig. 1 EDXD spectra of a levitated Ni42V58 sample, measured at different times upon undercooling