



Experiment title:

Time development of martensitic phase transition in Ni₃Sb studied by time resolved single crystal diffraction

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Report:

The martensitic transition between the high-temperature D0₃ phase (cubic with four atoms in primitive cell) and the low-temperature slightly distorted orthorhombic phase of Ni₃Sb has been followed in real time by time-resolved monochromatic single crystal diffraction. By quenching the sample it was possible to follow the transition by registering the progressive splitting, respectively the disappearance of the splitting, of individual cubic Bragg reflexes.

Small spherical single crystals with a diameter of about 200 μm of an alloy with the nominal compositions Ni₇₂Sb₂₈ were prepared by diamond grinding. In order to follow the progress of the phase transition, the samples were rapidly heated or cooled with a temperature step passing through the phase transition temperature. The temperature was calibrated with the phase transition temperature of Ni₇₂Sb₂₈, which for decreasing temperature is at about 560°C [1]. The time evolution of individual Bragg reflexes was followed using monochromatic X-rays with an energy of 56 keV and an X-ray Image Intensifier/CCD detector. Only a small part of the CCD (corresponding to an individual Bragg peak) was read-out, allowing a picture frequency of one image in two seconds.

Fig. 1 shows as a typical result of a measuring procedure, the decay of the (422) reflex of the cubic $D0_3$ high-temperature phase into reflexes of the orthorhombic low-temperature phase after quenching from 575°C to 545°C. This leads to a many-fold splitting of the reflex. The time in seconds after quenching is marked in the Figure:

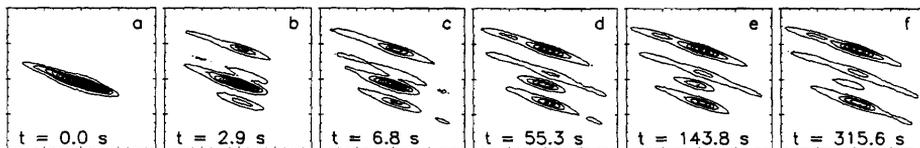


Figure 1.

Fig.2 (a) shows the time dependence of the transformation from the cubic high-temperature phase to the orthorhombic low-temperature phase in a 20 degree cooling step from 575°C down to 555°C. A slow begin is followed by an increase in transformation velocity with a consecutive tendency for saturation. The kinetics of the early stage of transformation is as expected if nucleation with increasing time is promoted by autocatalysis, i.e. an increase in the number of nucleation sites.

Already after the next cooling following an in-between heating to 620°C (1 minute) the transformation is much faster (the initial retardation does no longer occur, Fig. 2(b)), presumably because the nuclei of the low temperature phase have already been created in the preceding cycle and not been destroyed during the short-time heating.

Fig. 2 (c) show the time dependence of the transformation when the temperature is lowered to 545°C, Fig. 2 (d and e) those for a quenching from 560°C to 540°C and 535°C, respectively. The strong dependence of the transformation rate on temperature is evident.

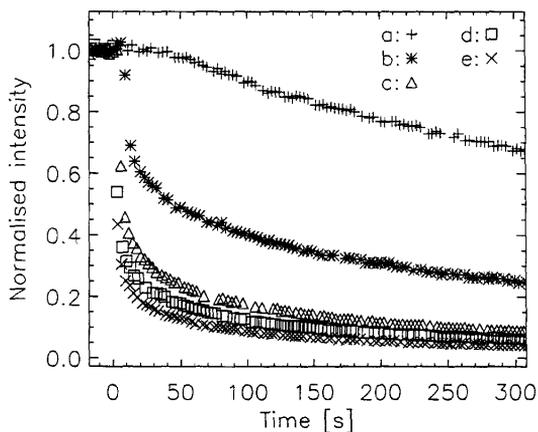


Figure 2, Intensity of (422) cubic reflex.