ESRF	**Experiment title:** High resolution in-situ study of martensitic nucleation in Ni_5Al_3 and Ni_2MnGa single crystals	**Experiment number:** HS 105
Beamline: ID19	**Date of experiment:** from: 11.12.1996 to: *13.12.1996*	**Date of report:** *25.8.1997*
Shifts: 6	**Local contact(s):** Dr. J. Härtwig	*Received at ESRF:* 1 SEP. 1997

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Experimental Report:

The nucleation of martensitic phase transformations is generally still an unsolved problem, despite its importance for a basic understanding of solid state phase transitions and various metallurgical applications. In this experiment the transformational behavior of $Ni_{62.5}Al_{37.5}$ and Ni_2MnGa single crystals with cubic high temperature phases (B2 and $L2_1$, respectively) was investigated, which are model alloys for a potential '*localized soft mode* ' nucleation [l] due to strong phonon softening [2], which plays the role of a "roadmap" for the transformation. This work, being part of a comparative study of displacive phase transitions in the bulk and near surfaces [3], is the first real bulk x-ray study of martensite formation in these alloys. It was realized by a white beam transmission experiment performed in situ with photon energies between 30 and 100 keV and the same crystals as in previous neutron and x-ray scattering experiments [2,3]. ID19 was ideally suited to this experiment due to the availability of a white beam at high energies, mandatory for transmission experiments with bulk samples (-mm), and the unique long distance between source and sample. The corresponding high angular resolution allowed us to detect very sensitively the formation of the first martensite in the "austenite" matrix, showing up as additional sharp Laue spots on the film.

In the following we concentrate on results for $Ni_{62.5}Al_{37.5}$ with a martensitic transformation temperature T_m = 85 K as measured previously by neutron scattering [2]. Fig. 1 shows the

transmission Laue pattern at 300 K. Diffuse streaks in <110> directions appear at approx. 200 K and increase upon further cooling. This is interpreted as increased straining of the austenitic {110} planes, which can be associated with the incipient crystal lattice instability. Fig. 2 shows a Laue diagram at 110 K, where the first sharp spots appear in the region of diffuse streaking, indicating that the first martensite plates have built up in the crystal. This is in remarkable contrast to the conventional value of T,. Taking into account that 0.1 % variation in the alloy concentration changes the transition temperature by ca. 15 K, this points at small concentration fluctuations in the sample with different local transformation temperatures. The formation of a complete ellipse of Laue spots, related to a zone axis of one martensitic plate, can be observed as the sample temperature is further lowered. Three additional ellipses, appearing upon further cooling, indicate the nucleation of different variants of the martensitic phase. However, one of them dominated the Laue pattern in the course of the transformation; in addition, not all variants were built up in subsequent thermal cycles. Fig. 3 shows the two phase regime at 85 K with co-existing austenitic and martensitic Laue spots, whereas fig. 4 refers to the totally transformed sample at 37 K. An important conclusion of this experiment seems to be that pre-transformed domains from local inhomogeneities do not transform the entire crystal by means of an autocatalytic nucleation.
A similar transformational behavior was observed for Ni_2MnGa. However, additional spots appeared ca. 50 K above T_m. To our knowledge, this is the first x-ray experiment showing clear crystallographic evidence for a transition to an intermediate phase that has been proposed due to anomalies in x-ray experiments [4] and confirmed by neutron scattering [5].

Figure 1: $Ni_{62.5}Al_{37.5}$ 300 K
Figure 2: $Ni_{62.5}Al_{37.5}$ 115 K
Figure 3: $Ni_{62.5}Al_{37.5}$ T_m = 85 K
Figure 4: $Ni_{62.5}Al_{37.5}$ 37 K

Acknowledgements: The authors are grateful to S.M.Shapiro and V.V.Kokorin for providing the $Ni_{62.5}Al_{37.5}$ and Ni_2MnGa crystals, respectively. This work was supported by the Deutsche Forschungsgemeinschafi (Grant No. PE 127/3) and Nato Linkage Grant No. 95 1422.

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