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Application for beam time at ESRF – Experimental Method

Quantitative texture analysis of polycrystalline NiTi wires with synchrotron radiation

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Aims of the experiment and scientific background

The analysis of texture (preferred grain orientations) in Ni-rich NiTi shape memory alloys (50.7 at-% Ni) which show two martensitic transformations on cooling from high temperatures (B2→R, R→B19′) is important for two reasons. Firstly, the presence of the texture represents a challenge for advanced crystallographic structure analysis. Secondly, the texture must be expected to be associated with anisotropic functional properties. The relationships between the phase transition behaviour of a solution annealed and aged (400°C, 72 ks) Ni-rich NiTi shape memory alloy in differential scanning calorimetry (DSC) to X-ray powder diffraction (XRD) and neutron powder diffraction (ND) data which considering the presence of the phases B2, B19′, R and Ni_4Ti_3 was described by Sitepu *et al.* (2001a). The results showed that the microstructures in the surface region (provided by XRD) and in the bulk (provided by ND) differ in terms of detected phases and corresponding volume fractions. The agreement between the measured and calculated patterns in all cases improved dramatically when the generalized spherical harmonic (Bunge, 1982; Popa, 1992) description for the preferred orientation (Von Dreele, 1997; Sitepu *et al.*, 2000; Sitepu, 2001) was included in the Rietveld (1969) refinements. The quality of the Rietveld fits was better for the ND than for the XRD patterns. Subsequently, Sitepu *et al.* (2001b) showed that:

1. The Ti-50.7 at-% Ni showed two DSC peaks on cooling from high temperatures and one DSC peak on heating from low temperatures. The volume fractions of B2, B19′ and R-phase change during heating and cooling while the volume fraction of the precipitates stays constant.
2. After the first peak on cooling a mixture of phases was detected. The first distinct DSC peak on cooling is therefore not only due to the formation of R-phase. This was found using both ND and transmission electron microscopy. Moreover the analysis of the energies associated with the first and second DSC peak on cooling suggests that the energies associated with the B2→B19′ and R→B19′ transitions are higher than the energy associated with the B2→R transition.
3. An analysis of the B2 lattice strains associated with the formation of the precipitates and the formation of the R-phase suggests that the precipitates favour the R-phase not only by making the formation of the B19′-phase more difficult but also by forming R-phase halos which decrease coherency stresses.

Wenk (1998) described the advantages and disadvantages of quantitative texture analysis by x-rays, neutron and electron methods. For very special applications such as very small volume in fine grained polycrystalline materials, Wenk (1988) showed that the synchrotron radiation is superior to the other methods. Heidelbach, Riekel and Wenk (1999) experimented with textures of rolled aluminum, aluminum and steel wires, polymer fibers and natural bone materials using the monochromatic X-ray microbeam (≤ 30 μm) at the microfocus beamline of the European Synchrotron Radiation Facility (ESRF). They showed that using both the synchrotron microfocus beam and the quantitative texture analysis method, it was feasible to analyze very small samples that could not be resolved previously.

Experimental method

The anisotropy of spontaneous unit-cell strain at a paraelastic-ferroelastic phase transitions such as in shape memory alloys translates into texture formation in the polycrystalline material, if the transition is stress induced isothermally as well as if it is induced by changing temperature in the presence of stress. The experiments on selected shape memory wires in temperature-stress space will reveal details of the stress-induced crystallite-orientation dependent nucleation at the martensitic phase transition by the observation of texture evolution with an area detector, and it will resolve the effective thermodynamic coupling constants between order parameters (crystallographic stress tensor) and conjugate field (stress tensor) in the polycrystalline material. Of particular interest are the behaviour of the intermediate rhombohedral "R-phase" between the cubic austenite and the monoclinic martensite, the martensite texture in relation to the texture in the original austenite as formed during wire manufacturing. Further, we would like to examine possible spatially inhomogeneous transition behaviour in the wires as observed in macroscopic tensile testing on large cylindrical specimens.

The 3rd generation synchrotron beamlines ID13 and BM16 at ESRF offers both high photon energy as needed for transmission measurements through NiTi as well as a well as practically vanishing divergence needed for Debye-Scherrer-cone registration at a large distance from the specimen. Further, fluctuations of beam position are required to be small to allow collection of consistent

In the present study, the technique described by Heidelbach, Riekel and Wenk (1999) at ESRF to study the quantitative texture analysis of very small volumes in fine grained polycrystalline materials of rolled aluminum, aluminum and steel wires, polymer fibers and natural bone will be applied to various type of NiTi polycrystalline wires with diameters range from 30μm to 152μm purchased from Memory-Metalle GmbH, Rhein in Germany. The wires will be investigated "in situ" at various temperatures (N_2 gas flow cooler/heater) under applied stress, where both axial and torsional forces are of interest. Moreover, the influence of both cold predeformation and high-temperature pre-creeping on the texture of the martensite and austenite phases will be investigated.

Results expected

It is expected to obtain quantitative texture analysis of polycrystalline NiTi wires with synchrotron radiation which cannot be resolved by conventional X-rays and neutron diffraction.

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