Phase composition and quantitative texture analyses of R-phase in *nickel-titanium* shape memory alloy by Rietveld refinement with WIMV method

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Abstract

The R-phase in *nickel-titanium* (NiTi) shape memory alloy (SMA) has been widely used in both industrial and fundamental research because this alloy has shape memory effect or superelasticity and also small thermal hysteresis which are very useful for actuators applications. The crystal structure of R-phase in Ti-Ni-Fe ternary alloy X-ray powder diffraction data showed that the space group is $P3$. While preferred crystallographic orientation (*i.e.* texture), can cause serious systematic errors not only in crystal structure determination but also phase compositional analysis, the fiber texture NiTi rod had not been corrected. Subsequently, the third generation synchrotron X-ray sources at the ESRF in Grenoble, which provides X-ray beams of higher energy and intensity than laboratory X-ray sources, for determining the crystal structure and phase fraction of the R-phase in Ti-Ni-Fe ternary alloy (3 mm in diameter and 8 mm in height) was used. The high resolution synchrotron powder diffraction data of the R-phase were subsequently analyzed using the GSAS Rietveld refinement with generalized spherical harmonic (GSH) description for preferred orientation. The results showed that (i) the GSH description is an excellent descriptor for phase analysis and structure refinement; (ii) the sample consists of minor cubic phase and (iii) no significant improvement in fit is found when the inversion center is removed from the $P\bar{3}$ model, suggesting that the space group is indeed $P\bar{3}$ and not P3.

In the present study, neutron diffraction phase analysis during thermal cycling of R-phase in Ti-Ni-Fe ternary alloy have been carried out using the D1A high resolution neutron powder diffractometer at ILL Grenoble. The neutron diffraction experiments were conducted because neutrons provide much higher intensity at high 2θ-Bragg angle and better atomic position and/or thermal parameters than X-rays. The results derived from Rietveld refinement with GSH description for neutron diffraction data agree well with the synchrotron data. Finally, the quantitative texture analysis of R-phase problem, which provides a wealth of information about the Ti-Ni-Fe ternary alloy properties but had not been conducted by the authors will be undertaken with great care in the present study. To the best of our knowledge there has been no analysis providing information on the quantitative texture analysis of R-phase in Ti-Ni-Fe ternary alloy SMA. In the present study, the ILL D20 high intensity powder diffractometer was used for measuring 1368 whole neutron powder diffraction data (χ=0°-90° and ϕ=0°-360°, with step size of 5°) of the R-phase. The Rietveld software *MAUD* [5], which is the most versatile texture treatment because the orientation distribution function (ODF) can be entered explicitly, was used to analysis the data. This procedure provides the quantitative description of texture simultaneously with the crystal structure of R-phase in Ti-Ni-Fe tenary alloy SMA.