Combined neutron and synchrotron diffraction data structural refinement of R-phase shape memory alloy

Husin Sitepu
Crystallography Laboratory, Virginia Tech, Blacksburg, VA 24061, USA < sitepu@vt.edu>
Jonathan P. Wright
European Synchrotron Research Facility (ESRF), BP 220, 38043 Grenoble Cedex, France

Abstract

The R-phase in *nickel-titanium* (NiTi) shape memory alloy 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. In order to understand the mechanism of the transformation, it is necessary to know the crystal structures of the R-phase. The crystallographic preferred orientation (*i.e.* texture) and the non-transformed austenite can cause serious systematic errors in the structural study of the engineering NiTi shape memory alloy [1,2]. The crystal structure refinement of R-phase phase in $Ti_{50.75}$·$Ni_{47.75}$·$Fe_{1.50}$ alloy synchrotron high resolution powder diffraction data using Rietveld refinement with generalized spherical harmonic description for preferred orientation correction showed that the sample consists of minor cubic phase and the space group was $P\bar{3}$ [2].

In this study, combined refinements of the R-phase in $Ti_{50.75}$·$Ni_{47.75}$·$Fe_{1.50}$ ternary alloy using both the neutron and diffraction data sets together were carried out using these space groups, giving a final series of models for comparison ($P\bar{3}$, $P3$ and $P31m$). The results showed that the reasonable space group for R-phase is $P\bar{3}$ as the six refined atomic positions were converging and the crystallographic R_{WP} and $R(F^2)$ figures-of-merits are 7.59 and 7.32% for synchrotron and 10.87 and 9.65% for neutron. Also, the comparison of the Rietveld fit shows that the peak intensities are quite different in the two data sets. This is due to the negative scattering length for Ti, so that by exploiting combined neutron and X-ray refinements there is excellent discrepancy between the two atomic sites. Trial refinements initially using only the synchrotron data sometimes found a false minimum with somewhat worse *goodness-of-fit* indices, however, when both data sets were used the same minimum was always recovered [3]. Therefore, it can be concluded that the combined synchrotron and neutron structural refinement provides accurate crystal structure parameters for R-phase in the $P\bar{3}$ model when applying correction to intensities using the generalized spherical harmonic description.

References

[1] Sitepu, H., Schmahl, W.W., Khalil Allafi, J., Eggeler, G., Dlouhy, A., Tobbens D.M. and Tovar, M. (2002). *Scripta Materialia,* **46,** 543-548.
[2] Sitepu, H. (2003). *Textures and Microstuctures.* **35**, 185-195.
[3] Sitepu, H., Wright, J.P., Hansen, T., Chateigner, D., Brokmeier, H.-G., Ritter, C. and Ohba, T. (2005). *Materials Science Forum,* **4955-497,** 255-260.