

## Determination of 3D statistical structural parameters of Ni<sub>4</sub>Ti<sub>3</sub> precipitates in stress-free and compressed Ni-Ti

S. Cao, M. Croitoru, W. Van den Broek and D. Schryvers

EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020, Antwerp, Belgium

nick.schryvers@ua.ac.be

Keywords: Precipitation, FIB/SEM, 3D Reconstruction, Slice-and-View, Shape Memory

A Slice-and-View procedure in a Dual-Beam FIB/SEM instrument is applied for the 3D reconstruction of Ni<sub>4</sub>Ti<sub>3</sub> precipitates in Ni-rich Ni-Ti alloys, in order to investigate their 3D morphology and distribution at the micro-scale and to better understand their significant influence on the B2-(R-B19)-B19' martensitic transformation of the matrix [1].

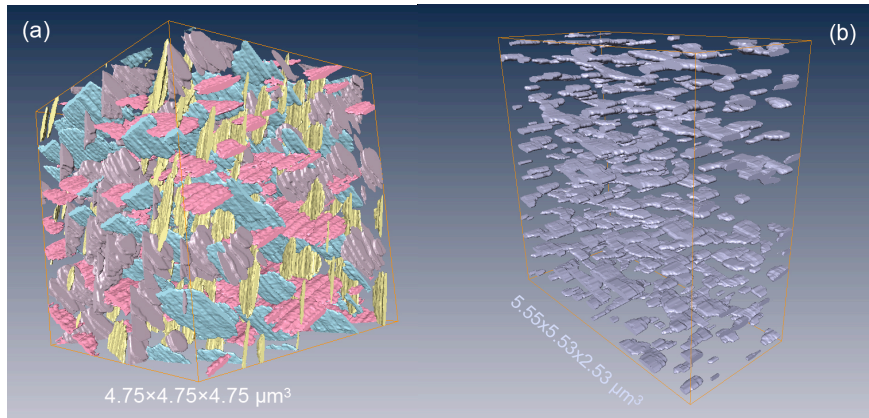
In the present study, a sequence of 2D cross-section images is obtained at the optimized imaging conditions of the Dual-Beam system. After proper image alignment and processing, a sequence of binary images is created in order to achieve the 3D reconstruction, from which both qualitative and quantitative data can be extracted [2].

In the present work, both a stress-free Ni<sub>50.8</sub>Ti<sub>49.2</sub> alloy with all four variants of precipitates (shown in Figure 1a) and a single crystal Ni<sub>51</sub>Ti<sub>49</sub> alloy after <111> compression ageing with precipitates aligned in one family (shown in Figure 1b) are investigated. The geometric mean volume of the precipitates in the stress-free alloy is calculated to be  $8.42 \cdot 10^{-3} \mu\text{m}^3$  with a geometric standard deviation of 6.473. These precipitates reach a volume ratio of 9.6% in the matrix. On the other hand, in the compressed alloy, the precipitates have a geometric mean volume of  $4.95 \cdot 10^{-3} \mu\text{m}^3$  with a geometric standard deviation of 3.760 and reach a volume ratio of 4.3% in the matrix. Figure 2 shows the lognormal volume distributions of the precipitates in both alloys. By the same principle, the surface area, specific surface area, thickness, disc diameter and aspect ratio of the precipitates in both cases are obtained. A morphological simulation with an ellipsoid function is performed on some random selected precipitates in the stress-free alloy and with a disc shape function in the compressed alloy to better understand the real morphology of the single precipitates.

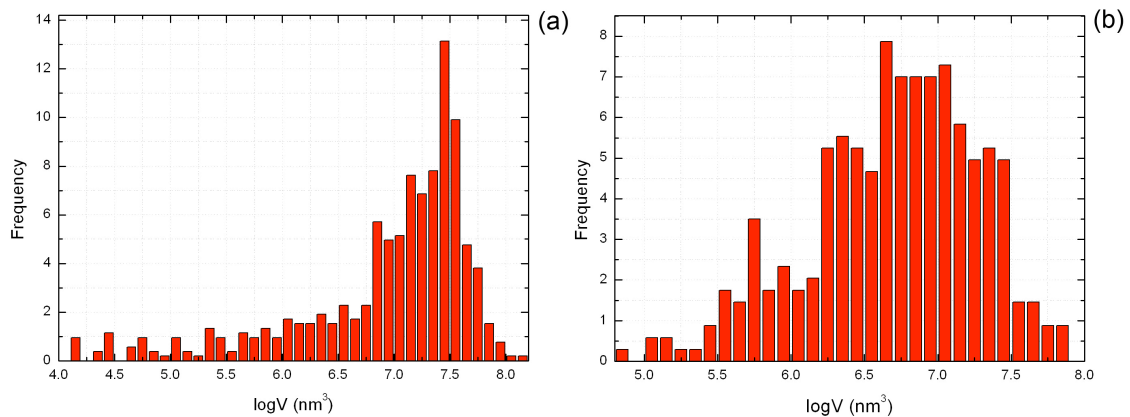
The Pair Distribution Function (PDF) of the precipitates is obtained via the coordinates of their mass centers, in order to study the different distributions of the precipitates in the compressed and stress-free alloy. As shown in Figure 3, in the compressed alloy, the nearest neighbor distance between the precipitates is found to be 0.56  $\mu\text{m}$ , while in the stain free sample, it is 0.44  $\mu\text{m}$ , with an extra peak at 0.26  $\mu\text{m}$  due to intersecting precipitates.

These parameters indeed reveal the different characters of the 3D configuration of precipitates in the alloys treated under different conditions so that the present work clearly indicates that Slice-and-View is a valuable tool for the investigation of the 3D distribution, morphology and related parameters of technologically important precipitates in alloys such as Ni-Ti.

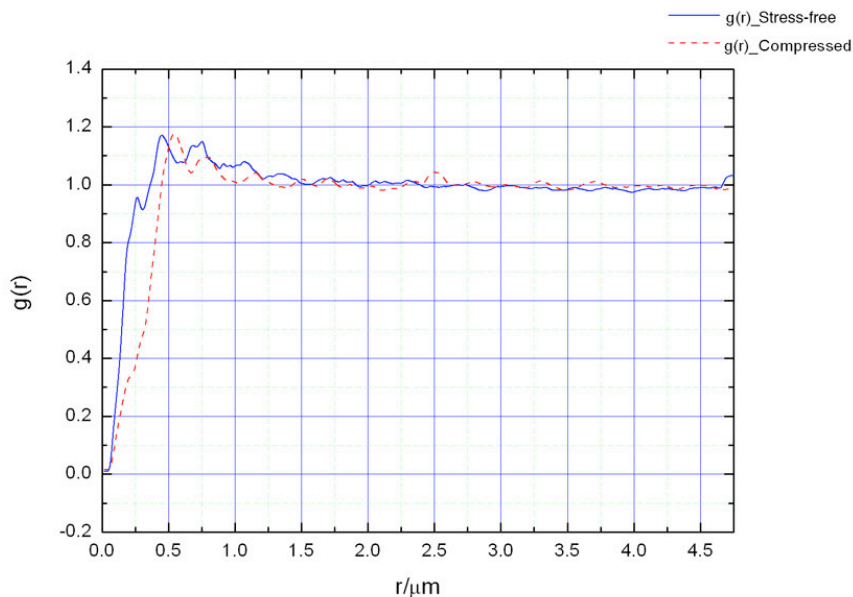
1. K. Otsuka, X. Ren, *Progress in Materials Science* **50** (2005), p. 511
2. S. Cao, W. Tirry, W. Van den Broek, D. Schryvers, *Journal of Microscopy* **233** (2009) 61-68
3. This research was supported by the Flemish FWO and EC MC MULTIMAT projects.
4. Samples were kindly provided by M. Nishida from Kyushu University, Jp (stress-free) and C. Somsen from Ruhr-Universität Bochum, Ge (compressed)



**Figure 1.** 3D reconstructions of the (a) stress-free and (b)  $\langle 111 \rangle$  compressed Ni-Ti alloy clearly showing the single family in the latter case.



**Figure 2.** Lognormal volume distribution of the precipitates in the (a) stress-free and (b) compressed Ni-Ti alloy.



**Figure 3.** Pair distribution function (PDF) of the precipitate mass centers in the stress-free (full blue) and compressed (dashed red) Ni-Ti alloy.