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# OBSERVATION OF MARTENSITE PHASE TRANSFORMATION IN SHAPE MEMORY ALLOYS USING A COMBINED XRD-DIC TECHNIQUE

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#### **Abstract**

Shape memory alloys (SMA), Ni-Ti alloy for example, have widely industrial applications because of their superelasticity or shape memory properties. However, the potential coexistence of three principal phases (Austenite, Martensite, and R phase) depending on the various thermomechanical loading conditions thus exhibiting different transformation deformation, makes further industrial applications a difficult task. For any tentative of multi-axial modeling validation, it appears relevant to get an estimation of strain field correlated with the local volume fraction of phases during a mechanical test. Combined DIC-XRD observation has been carried out for that purpose.

**KEYWORDS:** Digital Images Correlations, X-ray diffraction, Shape-Memory Alloys

#### Introduction

When a quasi static loading is applied to a equiatomic Ni-Ti alloy strip, phase transformation occurs (Austenite towards Martensite) in form of localization bands after bypassing the stress threshold. At increasing loading, either localization bands enlarge or new sets of localizations bands initialize elsewhere. NiTi alloy is on the other hand well known to develop a possible R-phase, intermediate between Austenite an Martensite associated with a different free transformation strain [1]. Both Digital Images Correlations (DIC) and X-ray diffraction (XRD) are consequently relevant techniques for an in-situ experimental observation of phase kinetic during a tensile test. On one hand, DIC grants us a global and continuous observation of strain field, which helps us to capture the evolution of localizations bands, and on the other hand, XRD allows a local evaluation of phase, that is the inner source of localizations.

## **Experimental setups**

As we can see in Figure 1, 1D strip of the quasi equiatomic Ni-Ti alloy (with a rectangle form in the center zone length  $L=10\,\mathrm{mm}$ , width  $l=3\,\mathrm{mm}$  and thickness  $h=0.3\,\mathrm{mm}$ ) is installed in a loading machine placed in the XRD chamber. The surface of specimen is covered by a white speckle pattern to provide enough contrast for Digital Images Correlation analysis. To prevent any perturbation between the setups of two techniques, a Prism is needed to send the image of specimen's surface toward a camera.

Displacement-controlled 1D tensile test is carried out at T=300K with a loading speed  $\dot{u}=0001mm.s^{-1}$  (corresponding to a longitudinal strain rate  $\dot{\varepsilon}=10^{-4}s^{-1}$ ). The loading have been held at three displacement steps during the loading (U=0.3mm, 0.4mm, 0.6mm) and one during unloading (U=0.3mm) to perform the XRD measurement. The corresponding stress / strain<sup>1</sup> curve is reported in Figure 2).

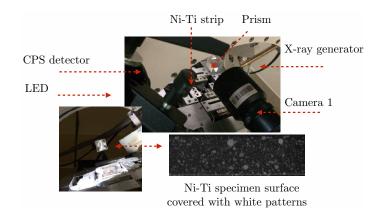


Figure 1: Experimental setups

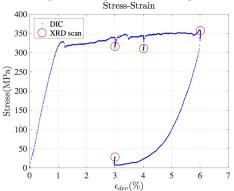


Figure 2: Quasi-static 1D tensile loading - Stress-Equivalent strain curve

### **Experimental observations**

Equivalent strain  $\varepsilon_{dev}$  and corresponding strains rate  $\dot{\varepsilon}_{dev}$  fields obtained just before the 1st XRD-scan are plotted in Figure 3 illustrating the band localization. The evolution of

 $<sup>^1</sup> average$  Levy-Mises equivalent strain calculated using DIC over the observable area  $10\times 3 mm^2$ 

strain rate field indicates the direction of propagation for phase transformation.

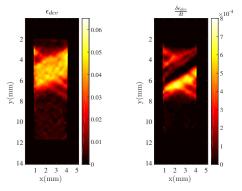


Figure 3: Equivalent strain  $\varepsilon_{dev}$  (left) and strain rate  $\dot{\varepsilon}_{dev}$  (right) fields at the 1st XRD scan

When motors are held, X-ray scans have been carried out along the center line of Ni-Ti strip. The diffraction spectra reported in Figure 4 show significant differences between bands and matrix. Indeed, three different diffraction patterns that belong to Austenite, Martensite, R phase are identified, proving the co-existence of the three phases during 1D quasi-static loading (Figure 5).

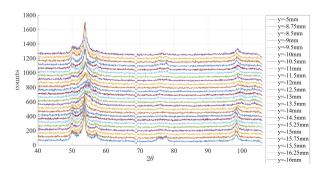


Figure 4: XRD scans along the strip.

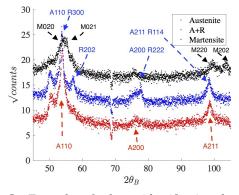


Figure 5: Example of phase identification by powder diffraction theory.

By adopting proper optimization procedures (detailed elsewhere), we calculate the volume fraction distribution of each phase. Strain field and volume fraction distribution

are compared. From Figure 6, we observe that inside localizations bands, Martensite is nearly saturated  $f_M = 100\%$ . Austenite phase is dominant in the matrix out of the band and R-phase is concentrated at the interface between matrix and bands.

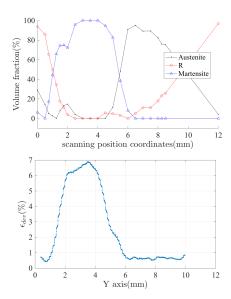


Figure 6: Comparison between Volume fraction distributions (Up) and strain field (Down)

#### **Conclusion**

DIC provides continuous global observations for displacement, strain and strain gradient fields during tensile test. XRD permits to have a local evaluation of different phases at the same time. The combination of these two different experimental techniques states that during the tensile strengthening of equiatomic Ni-Ti alloy the three possible phases coexist. More importantly, R phase is concentrated in the region at the interface between localization bands and matrix, thus potentially indicates that R phase is always produced upstream of the Martensite phase. These observations give insightful informations for the validation of numerical codes of super-elastic materials.

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