EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Grain-scale mechanical behavior in an innovative high-entropy shape memory alloy: in-situ martensitic transformation study	**Experiment number**: MA5410
Beamline: ID11	**Date of experiment**: from: 2022/09/22 to: 2022/09/26	**Date of report**:
Shifts: 12	**Local contact(s)**: Eleanore Lawrence BRIGHT, Wolfgang LUDWIG	*Received at ESRF:*

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Report

Introduction

Recently, a novel class of materials has been developed, the high entropy alloys (HEA)1. They are composed of a solid solution containing at least five elements, which leads to several interesting properties such as low stacking fault energies, exceptional strength and ductility in extreme environmental conditions. Besides, shape memory alloys (SMA) are extensively used in industrial applications due to their unique properties, such as superelasticity or shape memory effect; high temperature SMA (HT-SMA) are of particular interest in aerospace sector. For the first time, we have developed a new alloy combining high entropy effect and high temperature superelasticity in 2021: the (TiHfZr)74(NbTa)26 alloy exhibit a reversible martensitic transformation (MT) during mechanical loading. In order to master mechanical properties, a fine understanding of the martensitic transformation occurrence is mandatory. Previous experiments on a “classical” SMA highlighted the role of intergranular and intragranular interactions on martensitic transformation. Therefore the grain-scale deformation response has to be determined; to date, there has been no such measurements on HE-SMA.
So the proposal consisted in an in-situ loading experiment of a polycrystalline (TiHfZr)74(NbTa)26 HE-SMA. The main interest was to track the individual grain behaviour upon loading and to index martensite variants within the grains in order to better understand MT processes that governs macroscopic properties. This has been achieved by coupling diffraction contrast tomography (DCT) and the 3D-XRD technique.

Experimental method:

Two new home developed HE-SMA alloys have been studied: the (TiHfZr)74(NbTa)26 alloy and a Fe-based one. They differ by the crystallographic structure of the austenite phase (CC/FCC).

Specimens for nanox tensile rig were machined from flat specimen obtained by previous rolling and thermal treatments. An in-situ loading test was performed and was stopped at different loading point to acquire DCT patterns. To access strain/stress state, 3DXRD was also performed; 6 measurement points were recorded during loading and 5 during unloading for the polycrystal.

First analysis

- **Fe-based specimen :** despite thermal treatemnts, the final icrostructure was too fine to be analyzed. This was all the more complicated as the material exhibited twinning in its autenite phase.
- **Ti-based alloy** : 4 specimens have been analyzed:
    - The first one had a section of 0.5x0.6 mm^2. The DCT reconstruction made during the experiment revealed far too much grains in the diffracting volume. So the section of following samples has been reduced by polisihng to 0.3x0.3 mm^2.
    - The second sample analsyis failed due to a acquisition software problem.
    - The last 2 samples were studied during loading sequence, with two different vertical beam size : one with a large 0.16 mm, and the other ones with 3 slices of 0.08 mm^2. Microstrcutures have been reconstructed successfully for both samples; only two loading points, correspondind to the higher plastic strain level were not satisfactory. About 1000 grains were indexed at the initial state.

Further works

Besides the continuation of preliminary analysis presented before, the 3DXRD data processng are in progress. Expected results are the orientation, strain and stress fields of individual grains at in the initial state, the elastic domain and as it has started to transform: the final goal is to get these data both in autenite and martensite phase. The experimental results will also be used for / compared to mechanical modeling.