ESRF	**Experiment title:** Measuring the microscale origins of functional fatigue in materials with martensitic transformations	**Experiment number**: MA-4774
Beamline: ID06	**Date of experiment**: from: 21 June 2022 to: 27 June 2022	**Date of report**: 15 Sep 2022
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Preliminary Report:

Motivation: The aim of this experiment was to investigate the microscale origins of functional fatigue in materials that undergo martensitic phase transformations. Martensitic phase transformations are diffusionless, often reversible solid-to-solid phase transformations between a high-symmetry and a low-symmetry phase. It is the enabling mechanism behind the novel behaviors of a diversity of materials, including shape memory alloys, switchable multiferroics, and transformation induced plasticity (TRIP) steels. However, fundamental knowledge gaps regarding functional fatigue—changes to the material during cyclic loading that diminish its exploitative properties—continue to be a major barrier. Functional fatigue is caused by dislocations generated at the phase interface due to interfacial stress fields. Here, we used dark-field X-ray microscopy (DFXM) to measure these interfacial stress fields during stress-induced martensitic phase transformation for the first time, using a custom miniature mechanical load frame developed and tested by the PI and Co-PIs at the U.S. National Institute of Standards and Technology (NIST) (**Fig. 1**). Using this new capability, copper-aluminum-nickel (CuAlNi) shape memory alloys were mechanically cycled to observe stress-induced martensitic phase transformation, how dislocations developed in connection with interfacial stress fields at the austenite-martensite interfaces, and how these defects influenced the functional fatigue behavior in subsequent cycles.

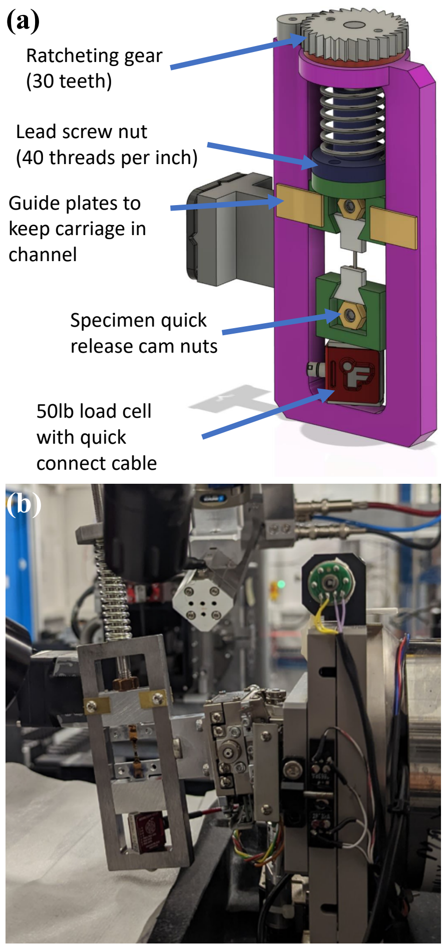

Figure 1. NIST-developed mechanical stage specially designed to be compatible with DFXM on ID06 (a); Stage mounted on the goniometer during beamtime on ID06.

Experiment Overview: Coarse-grained CuAlNi shape memory alloys were loaded in tension using the NIST-developed load frame (**Fig. 1**) while the microstructural evolution was measured inside individual grains using DFXM on beamline ID06. CuAlNi is an ideal material system for this fundamental study, because the martensite domains are known to be relatively large (> 1 μm), making them resolvable using DFXM. Here, we used this model martensitic phase transforming shape memory alloy to understand the microstructural origins of functional fatigue by measuring interfacial stress fields, interface morphology and type, and dislocation generation during stress-induced martensitic phase transformation under tensile loading.

Results: Preliminary results are shown in **Fig. 2** and **Fig. 3**. These examples show the partially phase transformed internal structure of one particular grain, measured in situ during uniaxial tensile loading with DFXM. The resultant microstructures are partially austenite (the parent phase, shown in white) and partially martensite (the transformed or daughter phase, shown in black), i.e., are two-phase mixtures. Thanks to the addition of a new detector on ID06, we have two options for magnification, resulting in both slightly magnified, large field-of-view measurements (e.g., top of **Fig. 2** or bottom of **Fig. 3**), as well as a highly magnified, smaller field-of-view measurements (e.g., bottom left of **Fig. 2**, middle of **Fig. 3**). By slightly tilting, or rocking, the sample about specific axes, we can then relate these micrographs of the sub-surface microstructure morphology to small variations in crystallographic orientation and elastic lattice strain (e.g., bottom middle and bottom right in **Fig. 2**). This allows us to measure, among other things, the chiefly important interfacial stress fields at the austenite-martensite interfaces. We also translated the sample vertically while collecting DFXM measurements, as shown in the bottom of **Fig. 3**, which will be used to reconstruct the 3D morphology of martensite domain structures. The specific phase interface, or habit plane, orientations can be used to identify which martensite variants are present.

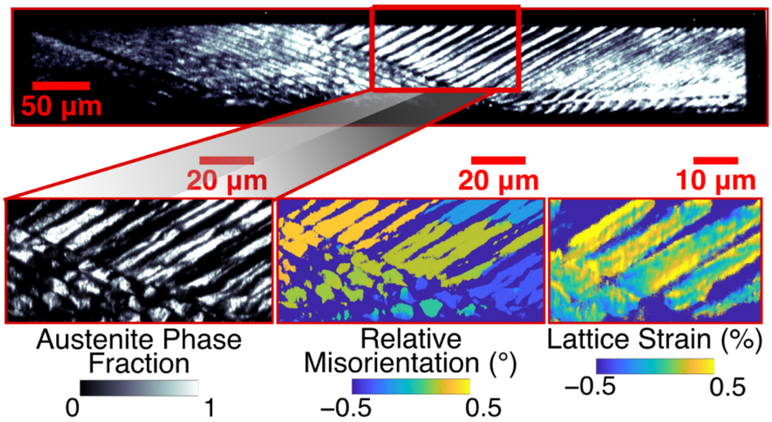

Figure 2. Example measurements of a partially phase transformed, austenite-martensite two-phase mixture. (The austenite is white, and the martensite is black.) These measurements are multiscale (thanks to the addition of a new detector on ID06) and can be used to measure the important interfacial stress fields at austenite-martensite interfaces, as well as small changes in orientation—as all show here.

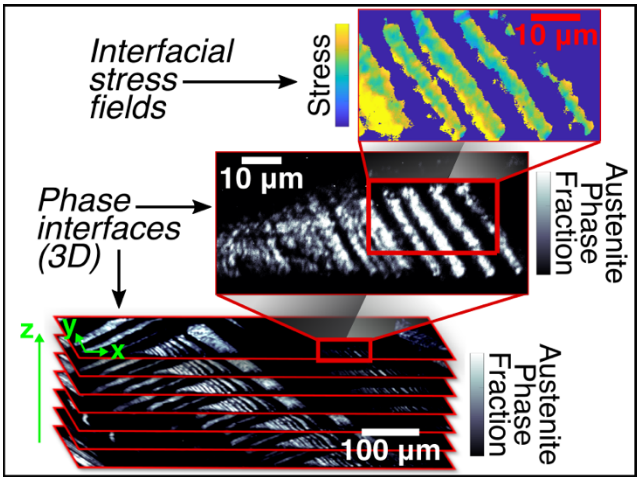

Figure 3. Example measurements of a partially phase transformed, austenite-martensite two-phase mixture. (The austenite is white, and the martensite is black.) These measurements are multiscale and can be used to measure the important interfacial stress fields at austenite-martensite interfaces.

<u>Current and Future Plans:</u> The results shown in **Fig. 2** and **Fig. 3** represent just a small fraction of our measurements. We were able to identify a sample with three neighboring grains all with high-intensity Bragg reflections suitably positioned for the DFXM microscope, and we collected 3D DFXM measurements in each of these three grains at multiple loading and unloading steps. It should also be noted that the reconstructions and images shown in **Fig. 2** and **Fig. 3** have not yet been subjected to any optimized analysis. In current and future work, we will fine tune the parameters involved in these reconstructions to improve image quality, angular resolution, and spatial resolution, and use the results to shed new light on the microstructural origins of functional fatigue. The expected results include the first-ever measurement of sub-surface interfacial stress fields during stress-induced martensitic phase transformation that are quantitatively related to the specific martensite variant, the generation of dislocations, and the effects of these features on subsequent cycles. The results will be used to develop a continuum-scale model for predicting the magnitudes of interfacial stress fields. This experiment will support advancements to understanding functional fatigue in martensitic phase transforming materials, used to accelerate the commercialization of energy saving solid-state technologies.