**In situ Synchrotron X-Ray Diffraction Studies of Ni-Rich Ni\textsubscript{50.3}Ti\textsubscript{29.7}Hf\textsubscript{20} High Temperature Shape Memory Alloy**

O. Benafan\textsuperscript{1}, R.D. Noebe\textsuperscript{1}, A. Garg\textsuperscript{2,1}, G.S. Bigelow\textsuperscript{1}, S.A. Padula II\textsuperscript{1}, D.J. Gaydosh\textsuperscript{3,1}, N. Schell\textsuperscript{4}, R. Vaidyanathan\textsuperscript{5}

\textsuperscript{1}NASA Glenn Research Center, Structures and Materials Division, Cleveland, OH 44135, USA  
\textsuperscript{2}University of Toledo, Toledo, OH 43606, USA  
\textsuperscript{3}Ohio Aerospace Institute, Cleveland, OH 44142, USA  
\textsuperscript{4}Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, D-21502 Geesthacht, Germany  
\textsuperscript{5}Advanced Materials Processing and Analysis Center, Materials Science and Engineering Department, University of Central Florida, Orlando, FL 32816, USA

Shape memory alloys (SMAs) are a unique group of materials that have the ability to change their shape in response to thermal and/or mechanical stimuli. This distinctive ability is a result of a diffusionless, solid-to-solid martensitic phase transformation between a high temperature, higher symmetry austenite phase and a lower temperature, lower symmetry martensite phase. Through this martensitic transformation, SMAs can generate recoverable shape changes of several percent strain even when opposed by large stresses resulting in high work output that is competitive with, or superior to, conventional hydraulic or pneumatic actuators. In addition, SMAs can provide both the sensing and actuation functions in a single mechanism, significantly reducing design complexities. Hence, SMAs are a promising alternative to conventional actuators in aerospace and automotive applications.

Currently available SMAs are primarily limited to binary NiTi alloys that generally exhibit a phase transformation at temperatures from slightly below room temperature to around 100 °C. This has severely restricting the environment in which they can be used, particularly for high temperature actuator applications. To address this limitation, the addition of Pt, Pd, Zr or Hf to binary NiTi has been shown to increase transformation temperatures. More recently, a Ni-rich Ni\textsubscript{50.3}Ti\textsubscript{29.7}Hf\textsubscript{20} (at.\%) HTSMA, developed by NASA Glenn Research Center, has been shown to possess unparalleled bulk mechanical and functional stability at the macroscopic level, with relatively high transformation temperatures (Figure 1) and promising properties at lower raw material cost when compared to typical NiTi-X (X = Pt, Pd, Au) HTSMA. The excellent dimensional stability and high work output for this alloy were attributed to a coherent, nanometer size precipitate phase observed using transmission electron microscopy. Preliminary results indicate that this precipitation strengthening alloy leads to outstanding load-biased shape memory behavior, with little or no irrecoverable strain, and excellent superelastic qualities [1-5].

**Figure 1**: Normalized synchrotron x-ray diffraction patterns of Ni\textsubscript{50.3}Ti\textsubscript{29.7}Hf\textsubscript{20} alloy during heating under (a) 0 MPa and b) 200 MPa stress showing the transition from B19' martensite to cubic B2 austenite.
In order to understand the mechanisms of how these precipitates actually improve shape memory behavior, it is necessary to determine the effect of stress and temperature on the mechanical response of the alloy while simultaneously following changes in the alloys’ internal state and microstructure. This was accomplished using in situ synchrotron x-ray diffraction at the High Energy Materials Science Beamline (HEMS), DESY. The experiments consist of loading the sample to 100/200 MPa, followed by thermally cycling under stress between room temperature and 300 °C with x-ray data collected continuously. Normalized diffraction spectra during the heating portion of the cycle are shown in Figure 1. These plots illustrate the transition from the monoclinic martensite (B19') to the cubic austenite (B2) while heating at different stress levels. The data is used to quantify the role of the internal stresses, phase fractions and texture evolution on the thermomechanical behaviour of this material. The lattice strains shown in Figure 2 indicate that the alloy reverts completely back to its original state once the load is removed without any evidence of residual strain or texture change. The shape memory behavior is unhindered by plastic deformation so that only transformation behaviors are evident.

![Figure 2: Lattice strain evolution of (a) (021)_M martensite and (b) {211}_A austenite peaks during thermomechanical cycling.](image)

Overall, the results emphasize the importance of advanced characterization techniques such as in situ synchrotron diffraction in truly understanding the complex mechanical and transformation behaviors of shape memory alloys. Funding from the NASA Fundamental Aeronautics Program, Aeronautical Sciences Project is gratefully acknowledged. The authors thank A. Stark for technical support and helpful discussions. This work has benefited from the use of the HEMS beamline at the German high-brilliance synchrotron radiation storage ring PETRA III on DESY. A detailed manuscript on this work is in preparation.

References