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Revealing Transformation and Deformation Mechanisms in NiTiHf and NiTiAu High Temperature Shape Memory Alloys Through Microstructural Investigations

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Shape memory alloys (SMAs) are ‘smart’ materials which are able to change their shape in response to changes in temperature. This unusual behavior arises from a solid-state phase transformation, which can be utilized to generate force. These extraordinary properties have made them of great interest to the automotive and aerospace industries for potential light-weight solid-state actuator applications. An actuator is any mechanism which converts energy, such as heat or electricity, into motion. SMAs outshine traditional actuating systems such as pneumatics, hydraulics, and DC motors due to their remarkably high power-to-weight ratios [1]. By replacing heavy conventional actuating systems, they offer the possibility of higher reliability, lighter weight and increased capability while lowering space and power consumption. This will lead to improved efficiency and reduced emissions, particularly in aircraft. There is currently a drive toward developing SMAs which can be used in high temperature environments, for applications such as fuel control valves within jet engines [2].

Two systems are at the core of this effort: NiTiHf and NiTiAu. The addition of hafnium (Hf) and gold (Au) dramatically increases the viable operating temperature window in these alloys. NiTiHf can be tailored to achieve a highly favorable balance of properties, including high strength, stability, and work output at temperatures approaching 300°C [3]. This behavior is strongly influenced by the formation of nano-scale precipitates, known as *H-phase*. These precipitates raise transformation temperatures and enhance shape memory behavior, while improving stability by suppressing plasticity during transformation. Less is known about the NiTiAu system. Recent constant-force thermal cycling (CFTC) experiments have demonstrated work output at temperatures above 400°C, as well as notable compositional insensitivity. Microstructural investigations have shown the presence of two types of secondary phase in these alloys, which may be responsible for the remarkable properties observed.

Advanced scanning transmission electron microscopy (STEM) based characterization techniques are being used to explore the mechanisms responsible for the unusual behavior seen in both alloy systems. In order to advance the reliability of these alloys to where commercialization is viable, a comprehensive understanding of the important microstructure-property relationships will need to be developed. Two compositions of NiTiHf ($Ni_{51}Ti_{29}Hf_{20}$ and $Ni_{50.3}Ti_{29.7}Hf_{20}$) and four compositions of NiTiAu ($Ni_{11}Ti_{49}Au_{40}$, $Ni_{10.3}Ti_{49.7}Au_{40}$, $Ni_{10}Ti_{50}Au_{40}$, $Ni_{9}Ti_{51}Au_{40}$) were prepared by vacuum induction melting at NASA Glenn Research Center. Small cylindrical bars were cut for mechanical testing and heat treatments. Nano-scale needles of aged material were trenched and thinned to electron transparency using a focused ion beam (FIB) on a FEI Nova 600. High angle annular dark field (HAADF) STEM was performed using a probe-corrected FEI Titan 80-300 at 300kV.

Investigations have highlighted the powerful effects of temperature, composition, and aging on critical alloy properties. Structural analysis of the H-phase precipitate was completed in previous publications

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[3], but recent efforts have been aimed at understanding how the precipitates interact with the surrounding matrix in such a way that the martensitic transformation is near-perfectly accommodated.

An accurate volume fraction of H-phase precipitates is necessary for developing models which accurately capture the mechanisms occurring during operation. This is challenging due to their small size, and as a result, estimates in literature vary across almost two orders of magnitude. Figure 1 shows the result of recent STEM-tomography work performed using FIB-fabricated nano-scale needles of NiTiHf. The precipitates are rich in the heavier element hafnium, and therefore show up brighter in HAADF STEM. The needle is placed in a Fischione 2050 On-axis tomography holder, which allows for 360^o rotation within the microscope. 180 HAADF STEM images, such as that in Figure 1a, were obtained in 1^o increments, to compensate for any particle shadowing and martensite diffraction contrast ambiguities. Post-processing is performed using TomoJ™, MIPAR™, and Avizo® software. For the needle shown in Figure 1, the precipitate volume fraction is calculated to be 14.3%. Similar efforts are planned for different aging conditions in this system. This technique allows for a larger, more representative sample volume compared to other techniques such as atom probe tomography (APT), and can be tailored based on the precipitate size and density within the sample. [4]

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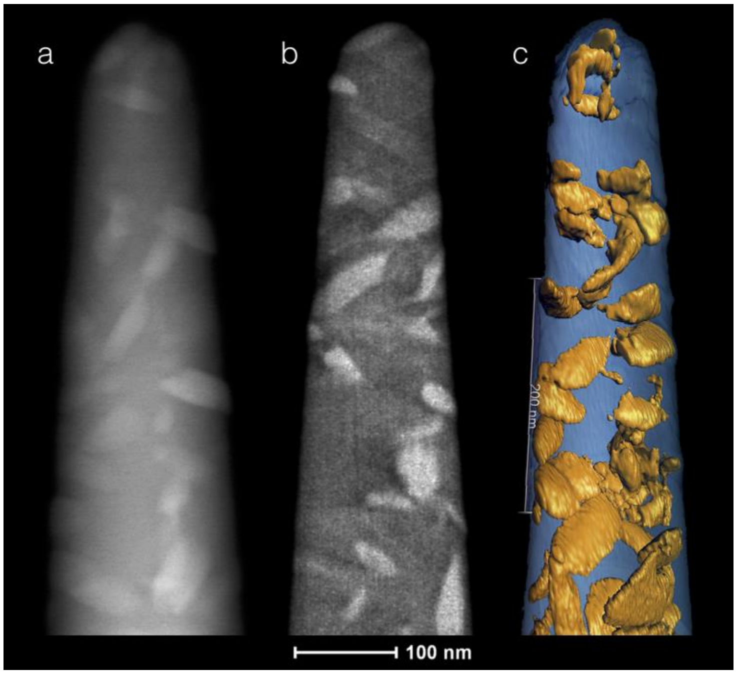

Figure 1: A needle fabricated from a $Ni_{50.3}Ti_{29.7}Hf_{20}$ alloy, aged 315 hours at 550^{o}C, used for H-phase volume fraction analysis. (a) An example of 1 of 180 HAADF STEM images used in creating the back-projection, a frame of which is shown in (b), which formed the building blocks for the 3D rendering shown in (c). The calculated volume fraction of precipitates in this sample is 14.3%.

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