On the Removal of the amorphous Layer in NiTi Shape Memory Alloy during TEM Sample Preparation

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NiTi shape memory alloys are multifunctional materials that undergo a martensitic transformation that gives rise to their unique shape memory effect and superelasticity properties. Recent advancements in transmission electron microscopy (TEM) techniques, coupled with sophisticated in-situ holders, have significantly enhanced our ability to study these properties at a nanoscale. The focused ion beam (FIB) method is widely regarded as state-of-the-art for fabricating samples for both ex-situ and in-situ TEM analysis. In this study, we conducted in-situ thermal analysis of NiTi alloys using TEM and observed an unexpected change in transformation temperatures attributed to the presence of an amorphous layer that formed during the FIB fabrication process. To address this challenge, we developed a flash polishing procedure that successfully removed the amorphous layer. However, upon transferring the flash-polished sample to a micro-electro-mechanical systems (MEMS) device, we discovered the reappearance of the amorphous layer. Subsequent investigation revealed that sputtering of atoms during transfer was the source of amorphization. To further understand the evolution of the amorphous layer, we subjected the flash-polished sample transferred onto the MEMS device to multiple cycles of heating and cooling. Our findings shed light on the dynamic behavior of the amorphous layer and its impact on the transformation properties of NiTi alloys, offering insights for the development of more effective fabrication and processing techniques.