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Microfabricated Cantilevers Based on Sputtered Thin-Film $Ni_{50}Ti_{50}$ Shape Memory Alloy (SMA)

by Cory R Knick and Christopher J Morris

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US Army Research Laboratory

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Sensors and Electron Devices Directorate, ARL

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14. ABSTRACT In this work, we discuss the design and fabrication of a nickel-titanium ($Ni_{50}Ti_{50}$) shape memory alloy (SMA) cantilever array that was thermally actuated by harnessing the residual stress difference during the martensite-to-austenite phase transformation during heating. The cantilever devices were fabricated on a silicon (Si) wafer using standard microfabrication techniques and released using a xenon difluoride (XeF_2) dry-etch technique and may therefore be applicable to microelectromechanical system (MEMS) switch or actuator applications. We demonstrated partially released devices capable of tearing a 1–3 µm rib of Si upon thermal actuation.							
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We wish to acknowledge Brian Isaacson for fabrication of devices in the cleanroom.

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1. Introduction

Thin-film shape memory alloys (SMAs) based on nickel-titanium (NiTi) can be engineered into micron-sized structures based on standard lithography and microelectromechanical systems (MEMS) processing, and have already become a primary actuating mechanism in MEMS devices since the work output per volume of thin-film SMAs surpasses that of electrostatic, magnetic, bi-metallic, piezoelectric, and thermo-pneumatic actuators.[1] While the study of thin-film SMA materials is well documented,[2–14] we are focused on microdevice applications for SMAs, which is perhaps a less well-developed area but is nevertheless the subject of several past high-quality studies. Namazu demonstrated a MEMS probe card based on a silicon (Si) cantilever using sputtered NiTi as the actuating material, generating up to 200 µN of force when Joule-heated with 500 mW supplied to the film.[15] Knopf showed an optically driven SMA microactuator using light to heat a SMA beyond its phase transformation temperature, thereby causing actuation.[16] Nakamura demonstrates substrate release of sputtered 10-µm-thick NiTi SMA cantilevers formed into loop actuators, demonstrating repeatable motion up to 20 Hz.[17] Chung uses photolithography to pattern 5-µm-thick NiTi cantilevers on glass substrates that exhibit curling upon exposure to an infrared (IR) light source.[18] In comparing reported material properties of these micromachined or patterned NiTi films, it should be emphasized that different test procedures produce different results. For example, transformation temperatures are affected by externally applied or residual stresses[19] complicating comparisons between free-standing SMA films and films attached to a substrate.

In an earlier report, we discussed our in-house development of a co-sputtering process to produce thin-film $Ni_{50}Ti_{50}$ SMAs with a phase change temperature at or near 60 °C with general repeatability.[20] In this report, we describe a fabrication process for creating dry-etch-released thin-film cantilevers of NiTi.

2. Methods

2.1 Co-Sputtering of $Ni_{50}Ti_{50}$

$Ni_{50}Ti_{50}$ films were co-sputtered using an AJA ATC 2200 co-sputter tool, with independent DC power supplies to 2 separate 4-inch targets. The targets we used were a $Ni_{50}Ti_{50}$ target and a pure Ti target. Substrate rotation was used to obtain optimal uniformity and argon (Ar) pressure was controlled between 2–10 mTorr. These films were characterized using multiple techniques, including electrical resistivity measurements on a 280SI Sheet 4-point Resistivity Measurement System

to quantify film non-uniformity. We verified the equiatomic composition of NiTi by using energy dispersive spectroscopy (EDX) film composition analysis on a Hitachi S-4500 scanning electron microscope (SEM) microelectromechanical system equipped with PRISM[60] Princeton Gamma Tech detector using a beam acceleration voltage of 20 kV. Phase transformation temperature in our NiTi alloy was quantified separately using differential scanning calorimetry (DSC) and temperature-dependent residual stress measurements for 1- and 2-µm films on a Si wafer.

2.2 Dry-Etch Release Fabrication Process for a $Ni_{50}Ti_{50}$ Cantilever

Our wafer frontside dry-etch release process is depicted in Fig. 1. Starting with a 4-inch Si wafer with a 170-nm platinum (Pt) backside layer and a 600-nm silicon nitride (Si_3N_4) frontside layer, we etched lithographically patterned regions of Si_3N_4 down to the Si substrate. Following this, we liftoff patterned a thermally evaporated 200-nm chromium (Cr)/Pt layer to create a differential stress distribution across each cantilever when combined with the SMA. Pt was selected to withstand the 600 °C anneal required to crystallize NiTi, after we demonstrated that gold (Au) could not survive the necessary anneal. Following this, a 1- or 2-µm film of $Ni_{50}Ti_{50}$ was blanket sputtered and annealed at 600 °C for 1 h without breaking vacuum following the sputter step. This layer was then wet-etch patterned with a 2:2:20 solution of hydrogen fluoride (HF), nitric acid, and deionized water (DI) for 60 s. We then capped the NiTi with a 120-nm sputtered layer of Au. A positive tone 5214 resist layer was patterned and developed to protect the Si_3N_4 during the xenon difluoride (XeF_2) final dry-etch release.

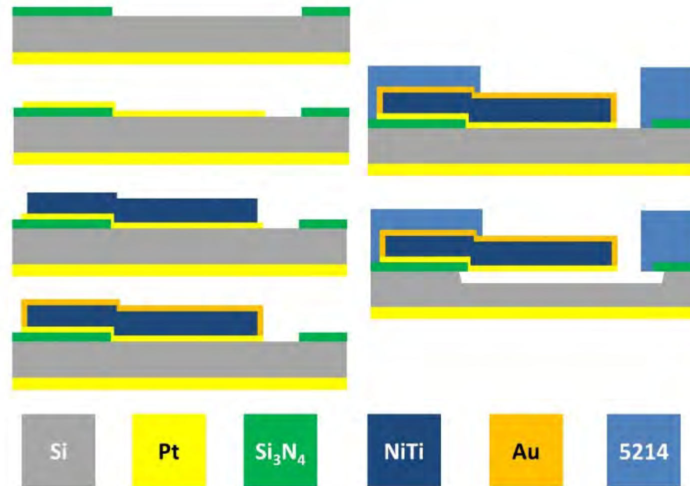

Fig. 1 Frontside Si dry-etch release process with Pt contacts and an Au sputter cap

3. Results and Discussion

Details about NiTi sputter process development are documented previously.[20] Film uniformity as measured by resistivity measurements was less than 7% across a 4-inch wafer. The onset of SMA phase change for an equiatomic $Ni_{50}Ti_{50}$ recipe was 58 °C as found by the DSC method, and 60 °C as found by temperature-dependent residual stress measurements. Up to 62 MPa was available for actuation based on these measurements.

3.1 Wet-Etch Patterning NiTi

Figure 2 shows SEMs of the wet-etch patterned NiTi devices after a 6-min wet etch using a 1:1:20 solution of HF/nitric acid/DI. Figure 2a shows how only a portion of the NiTi was cleared during the wet etch. We performed an additional 3-min etch to clear the remaining NiTi. Figure 2b shows the etch progress after the initial 6 min, demonstrating no lateral undercut of NiTi under the 5214 resist mold. Figure 2c shows a NiTi device not quite completely cleared of NiTi. Figure 2d confirms a 1-µm NiTi thickness, and the relatively smooth surface indicates that the NiTi etch progresses from the sides, rather than from the surface downwards. This indicates a surface coating developed during the NiTi deposition or anneal that is relatively resistant to the wet etch.

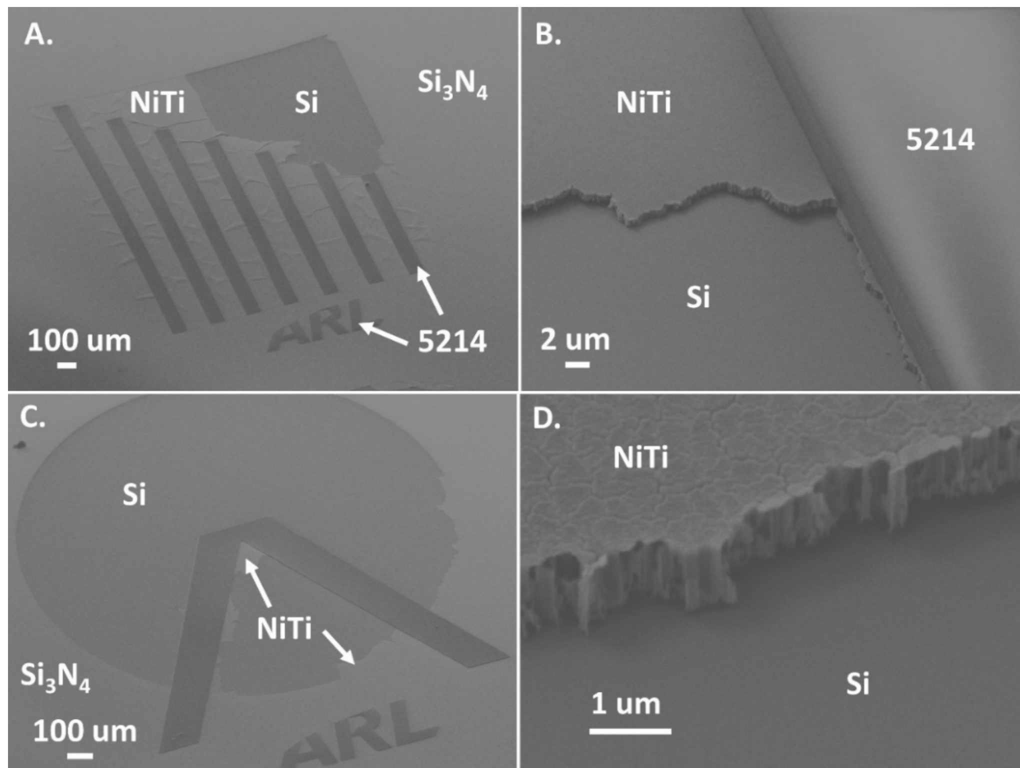

Fig. 2 SEMs after the NiTi wet-etch patterning with HF

In a previous process, we attempted to fabricate SMA actuators to be used as electrical switches with Au contacts. The Au contacts in this process became discolored and melted after the 600 °C vacuum anneal; therefore, we used Pt electrodes instead of Au, since Pt is known to withstand high temperature anneals better than Au. The Pt contacts were robust against the 600 °C anneal, as evident in Fig. 3, which shows SEMs of NiTi devices after the 600 °C anneal, wet-etch patterning of the NiTi. A 120-nm Au capping layer was also sputtered. Figure 3a shows a 200-nm Pt contact /1-µm NiTi /120-nm Au cantilever array device. Figure 3b shows that Si is etched during the NiTi wet-etch patterning step using a 1:1:10 solution of HF /nitric acid /DI. Figure 3c shows a 2–4 µm wet-etch undercut of NiTi from the patterning step and Fig. 3d shows a cantilever edge with a Pt base layer and Au-capped NiTi.

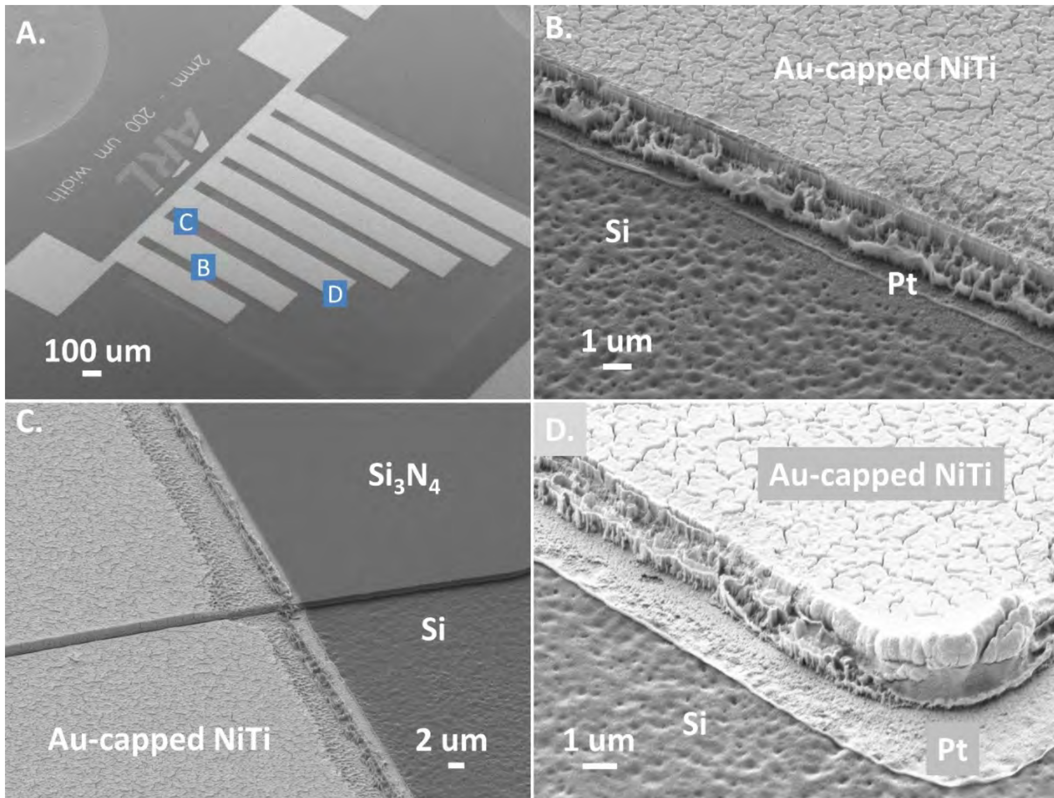

Fig. 3 SEMs after NiTi wet-etch patterning with HF and a 120-nm Au cap layer

Since the 6-min etch at 1:1:20 concentration etched the exposed Si region, we performed an addition wet-etch experiment doubling the concentration of HF and nitric acid, so that a solution of 2:2:20 was used. With this higher HF and nitric acid concentration, the NiTi was completely etched after just 60 s and the exposed Si did not significantly etch since exposure time to the etchant was minimized. The 2:2:20 concentration was used for all subsequent process iterations with very acceptable results.

3.2 Dry-Etch Release of NiTi Devices

A series of XeF_2 dry etches allowed us to discover that the Si_3N_4 layer was removed in the etch before the devices were completely undercut. Therefore, in all successive dry-etch experiments, we patterned 5214 resist so that only the exposed Si was exposed to gas phase XeF_2, effectively solving the Si_3N_4 etch problem. Figure 4 shows the partial release of NiTi cantilevers after 100 and 700 XeF_2 dry-etch cycles, also demonstrating that 5214 protected Si_3N_4 does not etch. After 700 cycles, the cantilevers were partially released, with a small Si rib traversing the length of the cantilevers apparent in the SEMs. In Fig. 5, we show the etch undercut versus the number of XeF_2 cycles on a wafer quarter (insert), which allows us to have control over the undercut depth achieved. All devices were inspected in the SEM after dry etching to confirm the extent of the release.

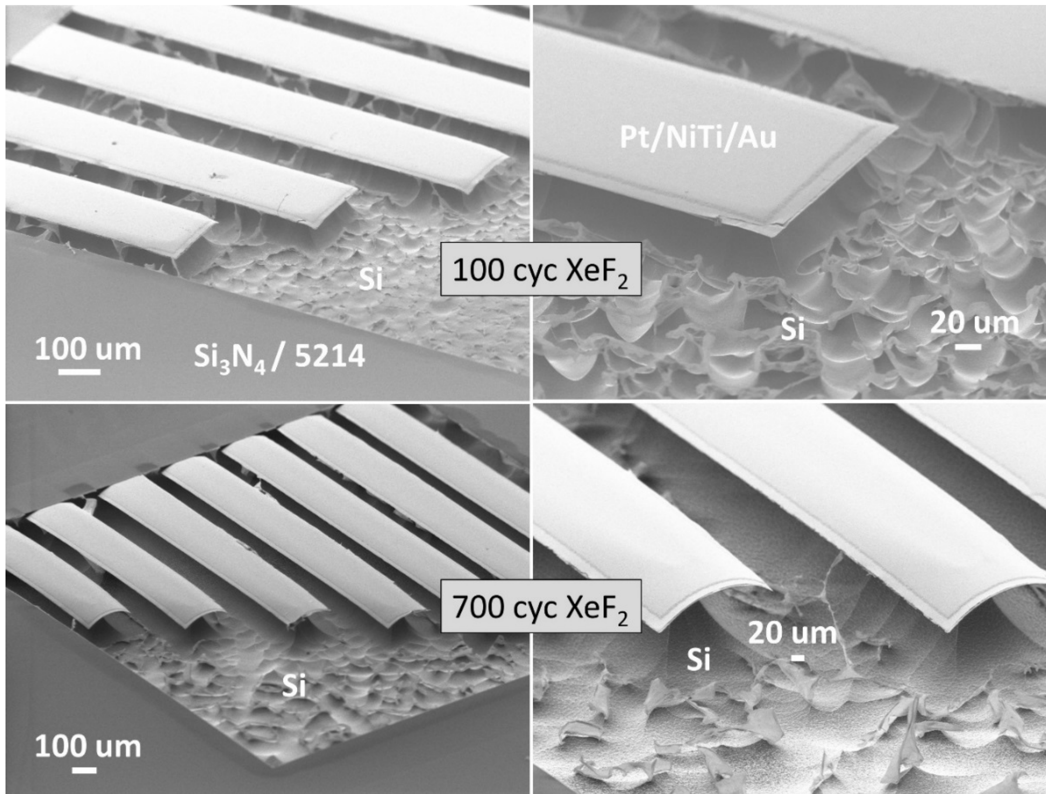

Fig. 4 Partially dry-etch-released NiTi cantilever arrays after 100 and 700 cycles of a XeF_2 dry etch

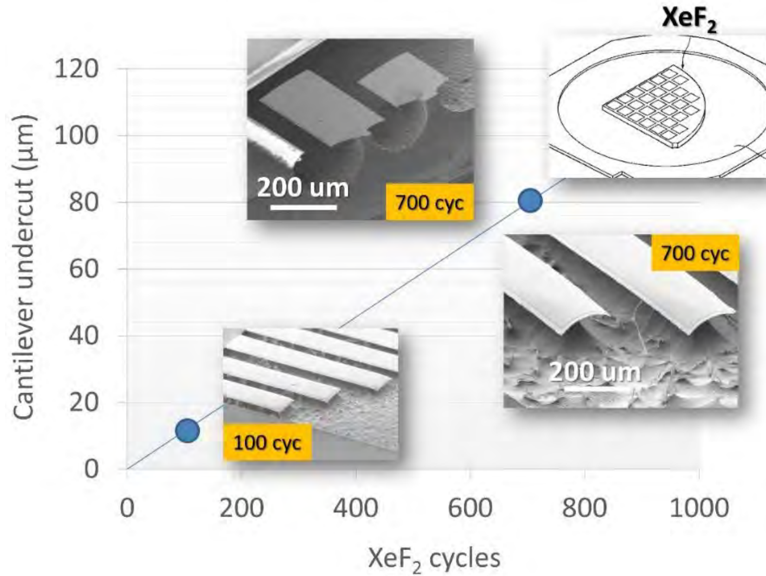

Fig. 5 Cantilever dry-etch undercut vs. number of XeF_2 dry-etch cycles

3.3 Thermal Actuation of NiTi Cantilevers

We took the partially released devices after 700 cycles of the XeF_2 etch and heated them to 80 °C on a hotplate while recording video. The initially flat devices, still anchored to a small rib of Si folded upward after heating, effectively ripping the rib of Si, as shown in Fig. 6. This demonstration shows that NiTi films of 1 µm thickness can provide enough force upon thermal actuation to mechanically disrupt the Si network underneath. Figure 7 shows video frame snapshots taken at 35 and 80 °C while directly heating a wafer piece part on a hotplate under a microscope, where thermally induced upward curling is evident. The cantilever width and length are 200 and 750 µm, respectively, here. We were also successful in actuating 100- and 50-µm-wide devices.

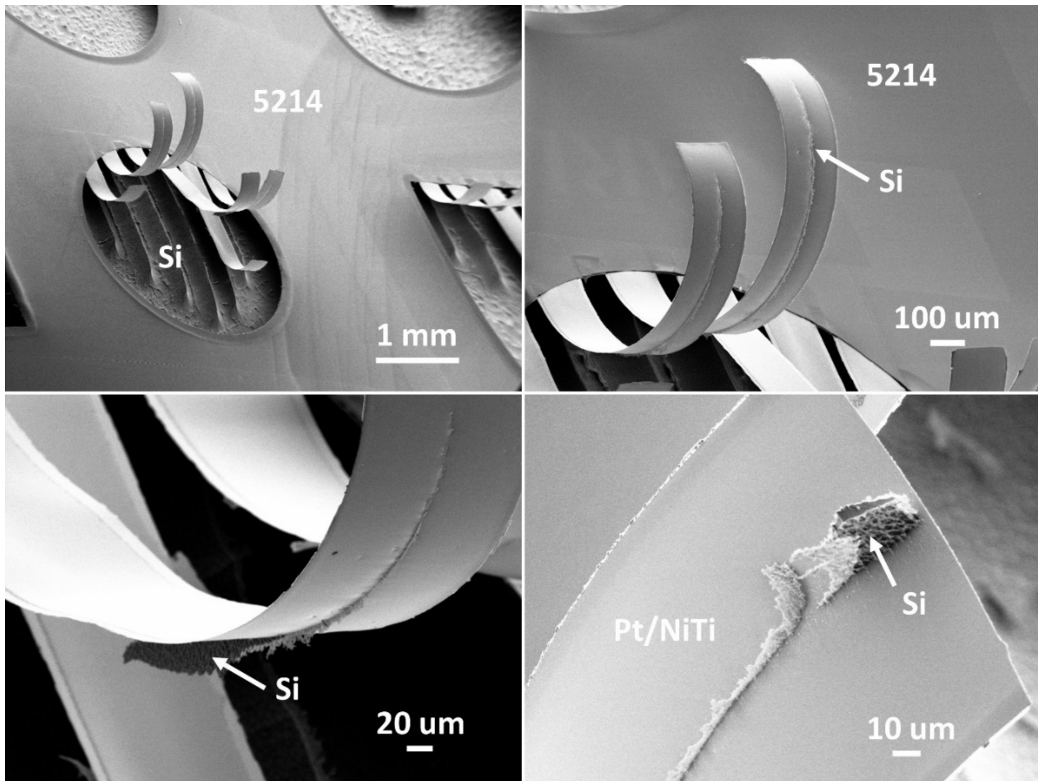

Fig. 6 NiTi devices thermally actuated after heating to 80 °C

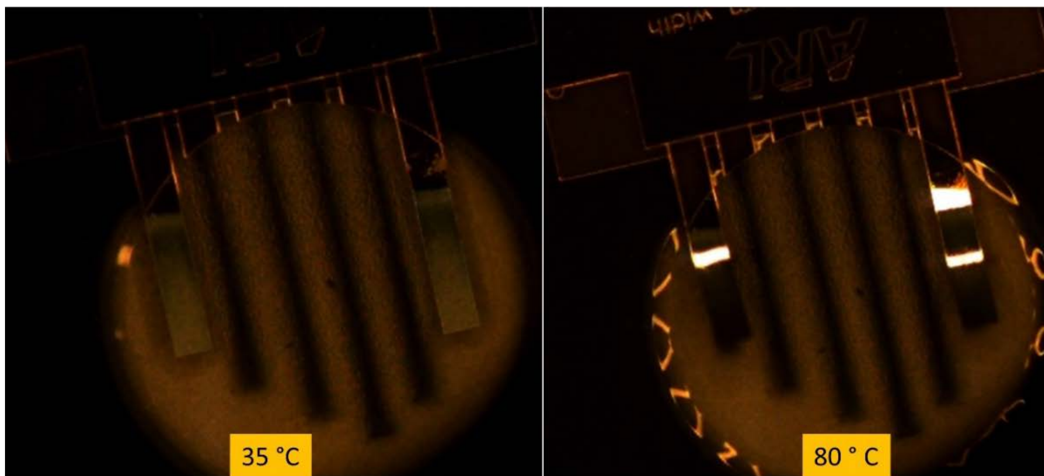

Fig. 7 Video frame snapshots taken at 35 and 80 °C while directly heating a wafer piece part on a hotplate under microscope, where thermally induced upward curling is evident. Cantilever width and length are 200 and 750 µm, respectively.

4. Conclusions

We developed a process flow that allowed us to fabricate prototype arrayed cantilever devices consisting of co-sputtered $Ni_{50}Ti_{50}$ cantilevers, which were successfully released with a wafer frontside dry-etch process. Thermal actuation of NiTi cantilevers was demonstrated by directly heating wafer piece parts on a hotplate and provided enough actuation force to rip through a 1–3 µm rib of Si, which ran along the underside of partially released cantilevers.

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List of Symbols, Abbreviations, and Acronyms

Ar	argon
Au	gold
Cr	chromium
Cu	copper
DI	deionized water
DSC	differential scanning calorimetry
EDX	energy dispersive spectroscopy
HF	hydrogen fluoride
IR	infrared
MEMS	microelectromechanical systems
Ni	nickel
Pt	platinum
SEM	scanning electron microscope
Si	silicon
Si_3N_4	silicon nitride
SMAs	shape memory alloys
Ti	titanium
XeF_2	xenon difluoride

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