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# Novel fabrication technique for NiTi and TiN micro-structures by femtosecond lasers

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**Abstract :** NiTi shape memory alloy (SMA) thin films were deposited onto silicon substrate using pulsed DC magnetron sputtering technique. To obtain crystalline NiTi thin films has to be synthesized at higher temperatures (475 - 525) °C. This high temperature requirement restricts the ease in conventional lithographic procedures. The recent advancements in the laser micromachining lend their applications into the fabrication of miniaturized systems. The femtosecond lasers (FSL) allow non-thermal processing of materials by ablation. This work focuses on the deposition and fabrication of NiTi ( $\approx 1.5 \mu\text{m}$ . thick) and titanium nitride (TiN  $\approx 0.3 \mu\text{m}$ . thick) thin films based miniaturized systems by femtosecond laser bulk micromachining. The NiTi and TiN microstructures were release by bottom silicon etch using reactive ion etching chlorine chemistry (RIE-Cl).

## 1. INTRODUCTION

Among the various micro-actuator domains like; piezoelectric, electrostatic, magnetic and bimorph systems; the NiTi based shape memory alloy materials have got a considerable attention because of their excellent work output and biocompatible nature [1, 2]. In NiTi SMA Systems, the shape memory effect is occurred by the crystal reorientations with either external mechanical or thermal stimuli. The low temperature, low stable phase is FCC known as martensite (B19) and the high temperature, high stable phase is BCC known as austenite (B2) [3, 4]. Despite of its potential applications in bulk form, NiTi SMAs have a wide range of applications in miniaturized system like, micro-actuators [5, 6]. The high power to weight ratio, large achievable strain, low driving voltage and excellent mechanical properties makes NiTi SMAs a promising candidate in micro-actuator domain [7, 8]. Because of it excellent biocompatibility, NiTi based SMAs are used in biomedical devices and surgical tools [9, 10].

When comes to the synthesis and fabrications part, there are several challenges involved in the successful deployment of micro-actuators of NiTi SMAs. The NiTi films have been mainly deposited widely by sputtering technique which provides more flexibility for precise control over the film composition, thickness & crystalline nature of the films [11, 12]. The NiTi thin films needs to be deposited or post deposition annealed at high temperatures for crystallization. The minimum temperature at which the NiTi films undergo crystallization is 475 °C. The phase transformation temperatures are depending on atomic percentage and the crystalline nature of nickel titanium films [13, 14].



The high temperature process requirement of NiTi thin films limits the ease of lithographic procedures and also the conventional lithography takes long procedures [15]. The alternate option barring the conventional lithographic procedures is the use of laser micro-patterning. But the laser micro-patterning has limitation like, accuracy, heat affect zones etc. Among the various laser machining techniques, femtosecond lasers have been outstanding due to their accuracy and minimal damage during the removal of materials [16, 17, 18].

Titanium nitride (TiN) is widely used as coating material because of its significant properties. In thin film form, along with NiTi, it is used as a protective layer for preventing the oxide formation in the Ti rich NiTi thin film surfaces. TiN layer also enhances the mechanical properties and biocompatibility of the NiTi thin films [19, 20]. In recent years, the TiN thin films are being used as heating element in MEMS devices for heating the localized areas [21].

## 2. EXPERIMENTAL

Nickel titanium thin films have been deposited onto P-type (100) silicon substrates by pulsed dc magnetron sputter deposition (PDC) technique. The target used is a 99.99% pure NiTi alloy (Ni45%atTi) disc of 76.2 mm. diameter. The deposition has been carried out at 600 °C using a resistive heater set up for obtaining crystalline NiTi thin films. The process chamber has been evacuated using a pumping combination of rotary vane and cryogenic pumps to a base pressure of  $1.50 \times 10^{-6}$  mbar. The substrate to target distance has been maintained at 90 mm. The process chamber contains another magnetron with titanium target. Since the moisture content in the process chamber degrades the quality of NiTi thin films, Ti has been pre-sputtered in argon ambient to reduce the moisture content in the chamber. The residual gases present in the chamber were monitored using a SRS make closed type RGA. During NiTi deposition, the chamber working pressure was maintained as constant at  $1.00 \times 10^{-2}$  mbar using a VAT manual gate valve, after argon gas with a flow rate of 25 sccm passed into the chamber. The magnetron is powered with 100 W using pulsed DC power supply. A pulsed frequency of 200 kHz and a time reversal of 1.0  $\mu$ s were maintained as the pulsing parameters. Prior to the deposition, the Silicon (100) substrates are cleaned initially by a rinse in deionized water followed by acetone ultrasonic treatment and are then subjected to a HF dip for native oxide removal.

Titanium nitride thin films have been deposited onto Si/NiTi stacks, without breaking the vacuum after the NiTi deposition, by reactive pulsed DC magnetron sputter deposition. The ratio between the sputter gas (Argon) and reactive gas (Nitrogen) has been kept constant. The target used is a 99.99% pure Titanium disc of 76.2 mm diameter. The depositions have been carried out at a substrate temperature of 300 °C using a resistive heater set up. The process chamber working pressure is maintained as at  $5.00 \times 10^{-3}$  mbar using a manual gate valve, after argon and nitrogen gases were passed into the chamber. The magnetron is powered with 100 W using pulsed DC power supply along with a pulsed frequency of 200 kHz and a time reversal of 1.0  $\mu$ s.

The microstructure studies of NiTi thin films have been carried out using an X-ray diffractometer (Rigaku Smart Lab X-ray diffractometer). To study the phase transformation temperatures with XRD, a heating mechanism was attached to the sample loading stage. The surface topography of the NiTi thin films have been carried out by scanning electron microscopy (SEM - Ultra high resolution scanning electron with monochromatic imaging spectroscopy) and Bruker atomic force microscopy (AFM). The thickness of the NiTi film was measured by cross-sectional scanning electron microscopy. The micro-wrapper and micro-heater structures were bulk micro-machined by using femtosecond laser (Clark MXR). The micromachined NiTi structures were release by bottom silicon etch using RIE-Cl. The heat generated by the TiN microheaters was measured using Fluke IR thermal camera.

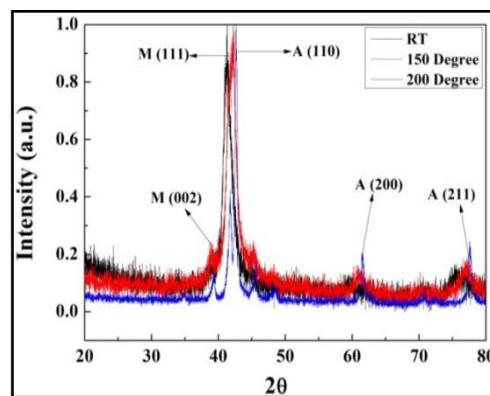
### 3. RESULTS

#### 3.1. Microstructural studies (Identification of phase transformation temperature)

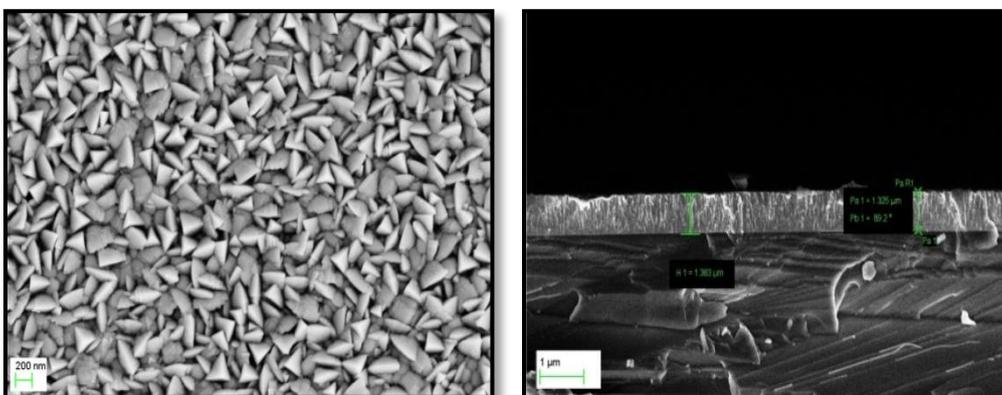
The NiTi film deposited at 600 °C subjected to X-Ray diffraction (figure 1) studies both at room temperature and at elevated temperatures. The room temperature XRD spectra reveal that, the NiTi film is in martensitic phase with more orientated towards (111) plane at 41.7°. Upon increasing the sample stage temperature (150 – 200 °C), the film is found to be transforming to austenitic phase. The peak corresponding to the (111) orientation was shifting to the (110) orientation at 42.6°. In addition, another peak corresponding to the (200) orientation of the austenite phase was also found to be appearing along with the temperature raise. This confirms the phase transformation behavior of the NiTi thin films [3, 4].

#### 3.2. Surface topography

The FE-SEM images of NiTi film deposited at 600 °C is shown in figure 2a. From the images, it can be concluded that the films are in crystalline nature with grain sizes of about  $\approx 250$  nm., this corroborates the X-Ray diffraction findings. The thickness of the NiTi film was measured from SEM imaging of fractured cross-sectional samples (figure 2b) is about  $\approx 1.5$   $\mu\text{m}$ . and using this, the rate of deposition of the films can be calculated to be  $\sim 25$  nm/min.



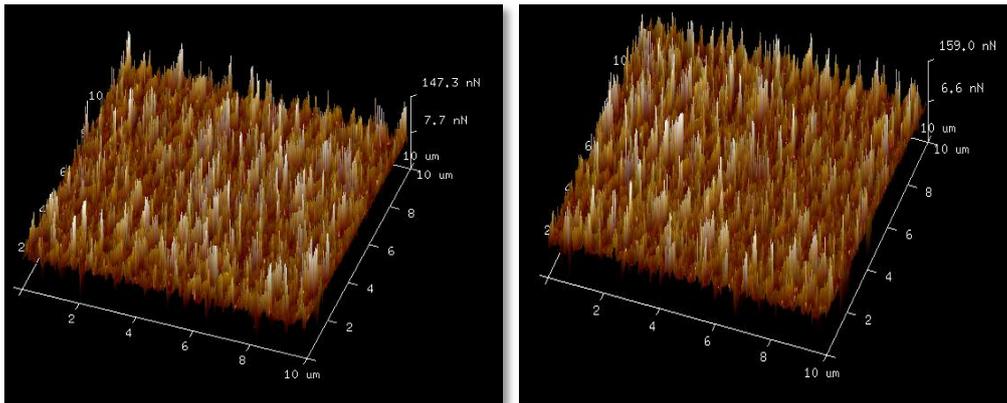
**Figure 1:** High temperature X-Ray diffraction spectra of pulsed DC sputter deposited NiTi films



**Figure 2:** (a) Scanning electron microscopic image of the surface topography of pulsed DC sputter deposited NiTi thin films, (b) Fractured cross-sectional image of NiTi films

The 3D topography of the NiTi film at room temperature is shown in figure 3a. The film surface has an RMS roughness of about 20.6 nm. Figure 3b. Shows 3D topography of the NiTi film at elevated temperatures (150°C). Both the room temperature and high temperature scans were carried out at the same spot on the sample surface. There was no much change observed on the sample surface with

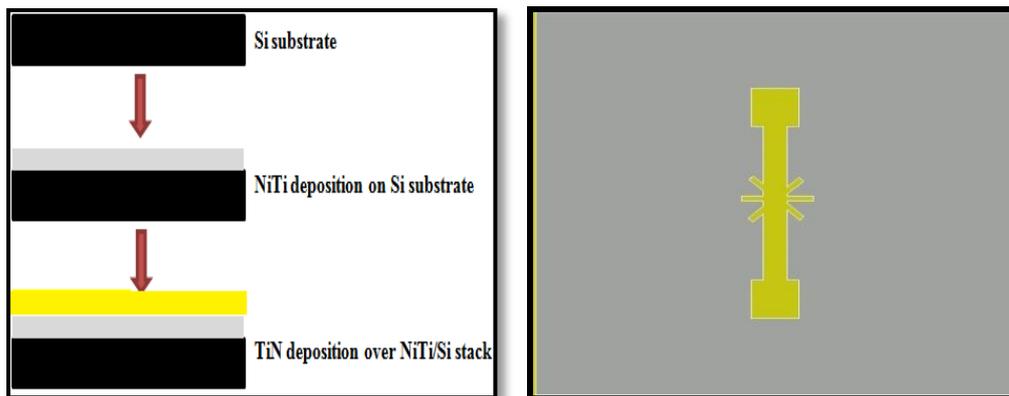
temperature. In general, the surface roughness of the thin films used to be more with crystalline nature. Here, both the XRD and SEM findings corroborate with the AFM results.



**Figure 3:** AFM 3D image of pulsed DC sputter deposited NiTi film (a) room temperature, (b) elevated temperature

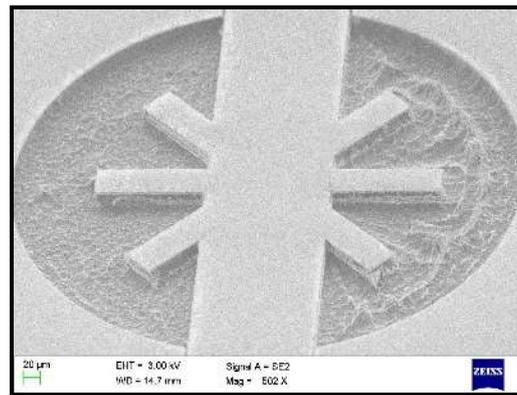
### 3.3. Microstructure fabrication

An AutoCAD design of the planned micro-wrapper and thin film heater was made before proceeding with the fabrication steps, which shown in figure 4. The process flow of the thin film deposition is shown in the figure 4a. The NiTi thin film was directly deposited onto Si (100) substrate. TiN thin film was deposited on Si/NiTi stack to form the complete Si/NiTi/TiN stack. The NiTi wrapper and TiN thin film heater structures design have shown in figure 4b. Conventional lithography procedures cannot be used here for fabricating the micro devices because the TiN and NiTi films were deposited at higher temperature, which hardens the photoresists used for patterning the structures, making it difficult for etching [15].



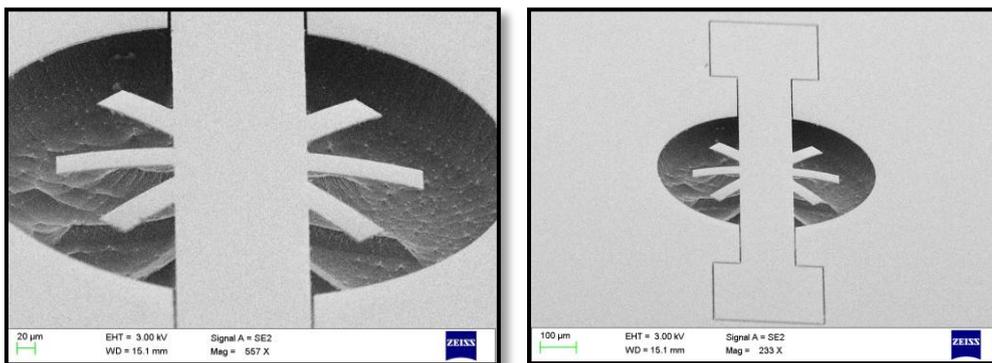
**Figure 4:** (a) Process flow of Si/NiTi/TiN stack (b) The CAD design of NiTi/TiN micro structures

An ultrafast femtosecond laser (Clark MXR) was used to do the bulk micro-machining of NiTi and TiN thin films to make wrapper and heater structures respectively. Focused ion beam (FIB) milling is not used for machining purpose because; the Gallium ions get implanted in NiTi film making it NiTiGa [22]. Also, the time taken to machine the materials is larger for FIB compared to FSL [23]. The image of the laser micromachined cantilever beam array prior to bottom Si etch is shown in figure 5.



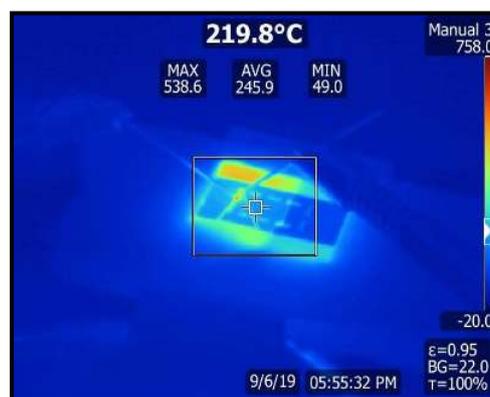
**Figure 5:** Femtosecond laser micro-machined NiTi micro-wrapper and TiN micro-heater structures

The bottom Silicon of NiTi micro-cantilever beams were etched using reactive ion etching Chlorine chemistry (RIE-Cl) resulting the release of the NiTi wrapper structures as shown in figure 6 [24].



**Figure 6:** SEM images of the NiTi micro-wrapper and TiN micro-heater structures after bottom Silicon etch

The TiN thin film micro-heater was powered using Kiethely source through electrical probes and tested their heating capabilities at various input powers. The Fluke IR thermal camera was used for measuring the temperatures generated by the micro-heaters. The highest temperature obtained was 538.6 °C and is shown in figure 7.



**Figure 7:** IR image of the temperature generated by TiN micro-heater

#### 4. CONCLUSION

NiTi and TiN thin films were deposited by pulsed DC magnetron sputter deposition technique. The phase transformation effect in NiTi SMA thin films were studied using HT-XRD analysis. An easy and short process for fabricating the NiTi SMA micro-actuators and the localized heating element was realized. Femtosecond laser micromachining technique makes the NiTi and TiN microstructure fabrication processes more compatible without using conventional lithography procedures. The bottom silicon was etched by RIE-Cl to release the freestanding NiTi microstructures. The heat generated by the TiN micro-heater can be used to actuate the NiTi microstructures.

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