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Applied Surface Science 208–209 (2003) 518–521

applied surface science

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Laser deposition of shape-memory alloy for MEMS applications

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Abstract

We have coated, by laser deposited NiTi films, Si-based cantilevers, of the kind commercially available as atomic force microscopy probes. The samples exhibited peculiar mechanical features ascribed to combination of shape-memory and bimetal effects. Our findings demonstrate the capabilities of laser deposition in fabricating metal alloys, and, at the same time, open the way to a simple process for the fabrication of basic components in microelectromechanical systems.

Keywords: PLD; Shape-memory effect; MEMS; Metal alloy films

1. Introduction

Large interest has recently grown on microelectromechanical systems (MEMS [1]) and on their applications in a huge variety of fields, ranging through microfluidics, biomechanics, biophysics, to optoelectronics and photonics. MEMS realization relies on both the availability of materials with suitable mechanical performances and the development of techniques able to produce small-size samples. In our work, we have combined NiTi, a shape-memory alloy (SMA), with pulsed laser deposition (PLD) in order to fabricate films with shape-memory effect onto Si-based substrates.

SMA [2] belong to a class of materials with the unique ability to recover their shape against strong deformations. In particular, they exhibit an extremely interesting mechanical behavior as a function of temperature: once deformed at low temperature, they will stay deformed until heated above some threshold temperature, when they spontaneously return to the original shape, with a mechanism involving large mechanical strengths. The basic mechanism for a similar behavior is related to the occurrence of a solid-to-solid phase transformation, from the so-called martensite to the austenite phase (in NiTi, a monoclinic $B19'$ and a CsCl-like structures, at low and high temperature, respectively). Thus, SMA devices can be used to replace at one time temperature-sensor and mechanical-actuator functions, as done since several decades in macroscopic systems with bulk SMA components.

The large power-density of SMA, their scalable character, and, in some cases including NiTi, their biological compatibility, make those materials excellent candidates for MEMS realization, which, in turns, demands the development of reliable SMA film deposition techniques. It must be pointed out that the relevant temperatures of the phase transformation turn out to be dramatically dependent on the stoichiometry, and that the mechanical rearrangement involved in the shape-memory effect can be strongly prevented by non-stoichiometric precipitates in a film. Furthermore, austenite phase stabilization can occur

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0169-4332/02/$ – see front matter © 2002 Elsevier Science B.V. All rights reserved.
doi:10.1016/S0169-4332(02)01448-4

in thin layers, limiting the shape-memory function. As a consequence, an efficient deposition technique for SMA must be congruent, clean, and easily scalable to relatively large thickness (typically, above 1 μm).

In the last decade, PLD has been widely employed with ceramic targets, application to metal and metal alloys being much less investigated [3]. On the other hand, some peculiarities of the process, as the congruency, the possibility to operate in ultra high-vacuum regime and to deposit thicker films by simply increasing the number of laser shots (at least, if mechanical stresses suffered by the growing film are kept small enough), can be efficiently exploited also in deposition of NiTi. A few years ago, we have already demonstrated the feasibility of the process [4] and, more recently, we have pointed out the relevance of vapor-phase collisional processes in preserving film composition [5]. In this paper, we report on the use of cantilevered substrates for NiTi films, which demonstrates the potential offered by PLD in the emerging area of MEMS.

2. Experimental setup

In our depositions, we used a conventional apparatus for PLD based on a commercial XeCl excimer laser and a deposition chamber evacuated at a residual pressure below 10^{-7} mbar. Depositions were typically carried out at a relatively large laser fluence (4–8 J/cm^2) and short target/substrate distance (30–33 mm), in order to attain a deposition rate of the order of 0.3–0.4 Å per laser shot. Substrate temperature was set in the range 520–600 °C, with a 20–40 min in situ high-vacuum annealing at the same temperature, followed by free cooling to room temperature.

A NiTi (49:51) pellet was used as target, produced by metallurgical methods aimed at ensuring a small content of impurities. Transition temperatures of a few tens of °C are expected, that allows an easier characterization and eventual exploitation with respect to equiatomic alloys, where the relevant temperatures are below zero. As substrates, we have used both Si/SiO$_2$ (450 nm) wafers and Si$_3$N$_4$ triangular cantilevers, commercially available as AFM probes [6]. This particular choice was motivated by the need of readily available substrates compatible with the mechanical properties of the samples. AFM cantilevers, which exist in a variety of shapes, dimensions, and spring constants, fulfill this requirement. Furthermore, NiTi coated cantilevers can be thought as basic elements of SMA miniaturized devices (e.g. microgrippers, micropumps, all-optical switches, ...), and, being based on silicon, an investigation of their functionality can be relevant in a more general context to assess the performance of Si-based micromachined devices coated by NiTi.

Analysis of the mechanical behavior (displacement versus temperature) of the deposited samples was carried out by using an ad hoc setup, employing a temperature controlled Peltier stage and a tilt/twist measurement system based on an optical lever method. Namely, a low intensity and tightly focused diode laser beam was sent to the cantilever vertex, and deflection was measured by a four quadrant photodiode acting as a position sensitive detector. When needed due to large deflection angles, a CCD detector with an appropriate optics has been used. Moreover, the setup allows for optothermal operation by an infrared laser beam used to locally warm up the sample in measurements which will be presented elsewhere.

3. Results and discussion

The main goal of our investigation is the assessment of the shape-memory effect in NiTi films deposited by PLD. Direct or indirect diagnostics tools can be exploited to this aim. Among indirect methods, the most frequently used is based on the analysis of XRD patterns acquired at different temperatures (above and below transition temperatures). Fig. 1 shows an example for a film deposited with 4×10^4 laser shots onto Si/SiO$_2$, which exhibits remarkable structural differences as a function of temperature. The peak at $2\theta \sim 43.7^\circ$, ascribed to (0 0 2)-B19′ reflection, disappears at high temperature, whereas the signal around $2\theta = 42.5^\circ$, attributed to the (1 1 0) reflection of the CsCl-like structure, follows an opposite behavior. This confirms the occurrence of a phase transformation, even if it must be noted that the transformation into martensite is not fully developed, as confirmed by the non-negligible signal around 43° in the low temperature pattern. This circumstance, along with shift and broadening of the corresponding peaks, suggests that the phase transformation is partially inhibited due to

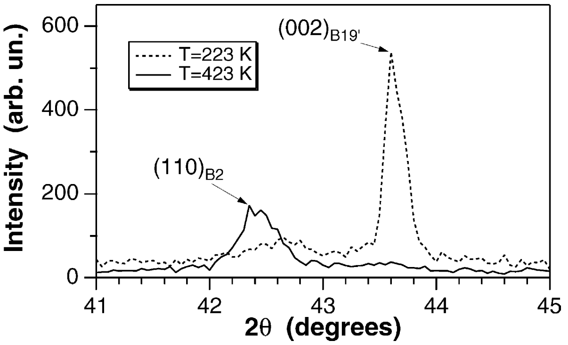

Fig. 1. XRD pattern acquired at two different temperatures on a film deposited onto Si/SiO_2 with 4×10^4 laser shots. Peak identification is shown on the plot.

mechanical interactions with the substrate, as expected in relatively thin films, or due to precipitates formed at the interface with the SiO_2 layer.

Temperature-resolved XRD patterns, being carried out at (generally two) fixed temperatures, do not allow in any case a full reconstruction of the transformation behavior. To this aim, the electrical resistance versus temperature analysis, which relies on the dependence of the electrical transport properties on the actual structure of the SMA film, is much more useful. An example is reported in Fig. 2(a) referring to a film deposited with 5×10^4 laser shots, which shows the occurrence of an hysteretic behavior typically associated with the dissipative effects of the solid-to-solid phase transformation. Similar measurements, carried out as detailed in [5], allowed us to estimate the relevant temperatures of the transformation in films of different thickness. The data summarized in Fig. 2(b) demonstrate the important role of thickness and the occurrence of phase stabilization phenomena in thinner films, where the interval between relevant temperatures is larger and the hysteresis loop is less defined (i.e. its area is smaller). At the same time, the derived temperatures turn out to be rather similar to those of the NiTi pellet used as the target, especially for thicker films. This confirm the congruent character of the deposition process.

Even if indirect measurements provided us with a viable tool to assess and characterize shape-memory effect in our samples, functional (i.e. mechanical) investigations are needed to evaluate their applicative potential. Different approaches can be adopted for the displacement involved in shape-memory to take place. For instance, free-standing films [4] or suitable micromachining of the coated substrate [7] can be exploited. Alternatively, micromachined cantilevers can be used as flexible substrates for the deposition of the SMA film. Contrary to [8], where specific Si-based beams were used in RF-sputtering deposition of NiTi, we chose commercial Si_3N_4 cantilevers, which offer the advantage to be readily available and fully characterized in terms of mechanical properties.

By analyzing tilting of the coated cantilever upon sample heating and cooling, we observed a peculiar

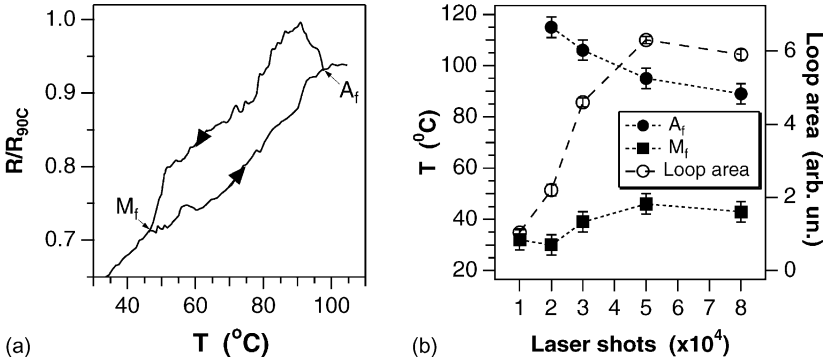

Fig. 2. (a) Electrical resistance vs. temperature curve (data normalized to the resistance at 90 °C) in a film deposited onto Si/SiO_2 with 5×10^4 laser shots; temperatures relevant for the transformation as deduced from the measurement are marked in the plot (A_f and M_f being the finish point of transformation into austenite and martensite phase, respectively). (b) Behavior of the relevant temperatures for the transformation as deduced by analyzing films grown with different number of laser shots, i.e. different thickness (the average growth rate being 0.3–0.4 Å per laser shot); the behavior of the area inscribed in the hysteresis loop is also shown (open circles), measured in arbitrary units.

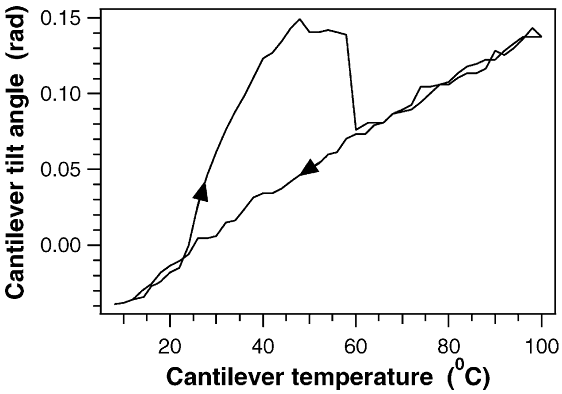

Fig. 3. Tilting angle as a function of the temperature for a NiTi coated microcantilever (triangular shape, length 320 μm, spring constant 0.01 N/m). Twist of the cantilever during the measurement was negligible, as well as the displacement of uncoated cantilevers observed in similar conditions.

behavior, shown, for instance, in Fig. 3. Most striking features are the relatively large absolute value of the tilting angle and the occurrence of a hysteresis loop. We note that, for the 320 μm long cantilever used for the measurement shown, the maximum tilting observed roughly corresponds to a linear displacement of the cantilever tip in the tens of μm range. Furthermore, the critical temperatures which can be identified in the loop are once again compatible with those of the phase transition for the material used as the target. Uncoated samples were also analyzed with the same apparatus, showing negligible displacements and absence of any hysteresis.

A complete interpretation of the observed behavior is out of the scope of the present paper. Several phenomena should be considered to this aim, as, for instance, the bimetal effect and the occurrence of a pre-load in the deposited films due to mechanical stresses accumulated during the growth. However, we remark that important applicative perspectives are offered by the simple system we have realized. For instance, a temperature controlled actuator can be built, with the possibility to operate in a proportional regime (corresponding to the linear portion of the hysteresis loop) or in a strongly non-linear regime (corresponding to temperatures close to the transition), depending on the thermal history. Furthermore, due to the micrometer size of the sample, temperature change can be easily (and quickly) accomplished by a variety of methods, for instance by sending a small current through the metal alloy film, or even by low-power laser irradiation of the cantilever. The optothermal control of the coated cantilevers, which can be of interest in the realization of all-optical switches for photonics applications, has been already verified and will be presented in a forthcoming paper.

4. Conclusions

NiTi films have been grown by PLD onto Si-based substrates. Results demonstrate the attainment of shape-memory effect, so confirming PLD as a viable technique for deposition of SMA films. In particular, deposition onto microcantilevers and subsequent investigation of the mechanical behavior as a function of the sample temperature suggest that basic components of MEMS prototypes with a wide range of possible applications can be fabricated by PLD in a simple, clean and congruent process.

Acknowledgements

Work supported by CNR through Progetto Finalizzato MSTA II, “Leghe a memoria di forma”.

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