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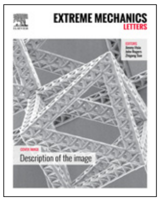

Microstructural evolution and surface strengthening of pulse-laser treated Ti/Ni multilayer thin films

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ABSTRACT

In this study, a picosecond pulse laser was utilized to treat Ti/Ni multilayer thin films to induce desired microstructure change and surface strengthening. It was observed that with the increase of laser pulse-energy, the microstructure of the treated films can be significantly modified. The surface morphology evolves from a homogeneous grain surface, to a cross-hatched pattern surface, and then to a rough melted surface covered with bubbles, voids and cracks. And the cross-section morphology evolves from a multilayered structure to partially intermixed and eventually fully intermixed structure. Due to the precipitation of Ti–Ni intermetallic phase, laser treatment with high pulse-energy led to surface strengthening on Ti/Ni multilayer thin films.

1. Introduction

Laser treatment has attracted more attention recently on micro and nano-scale materials in many ways [1]: fabrication of stable nanoparticles [2,3] and nanocomposite thin films [4] by laser ablation; laser crystallization, laser doping and laser cleaning in semiconductor industry; and laser processing for micro and nanostructured solids, such as laser-induced forward transfer (LIFT) [5] and laser-induced periodic surface structures (LIPSS) [6–9]. LIPSS have been extensively reported and found large potential in micromachining for microelectronics and micro-electro mechanical systems (MEMS) [10,11]. The formation of LIPSS is attributed to interference between the incident beam and scattered beam parallel to the substrate, and the corresponding period and orientation of ripples can be determined by laser beam wavelength and polarization as described by classic theories [12–15]. In addition, laser treatment can modify the phase composition and chemical state of the irradiated surface [16–18], via surface oxidation and intermetallic formation, accompanying the modification of surface properties. Laser treatments have been previously carried out on bulk metallic materials to achieve better mechanical properties such as hardness [19], fatigue [20], wear [21], and corrosion resistance. The mechanical property enhancement is attributed to solid solutions and alloy precipitates induced by laser treatment. The advantage of this technique is the ability to improve surface mechanical properties while confining the modification of both microstructure and chemical composition to a very shallow depth from the surface within a very short interaction time.

For metallic multilayer thin films, few studies have been done on laser treatment. However, laser-treated multilayer films could have broad potential applications. First, laser treatment on multilayer thin films can be a convenient and quick approach to form alloy compared to thermal annealing. Second, laser-induced surface strengthening, protective coating, and texturing could be achieved with suitable material selection and precise laser controlling, increasing the corresponding potential applications of multilayer thin films in aerospace/armor systems, biomedical devices, and photovoltaic/semiconductor industry.

In this work, Ti/Ni multilayer thin films were selected as a model system to perform laser treatment due to their advanced functional properties (e.g. optical and mag-

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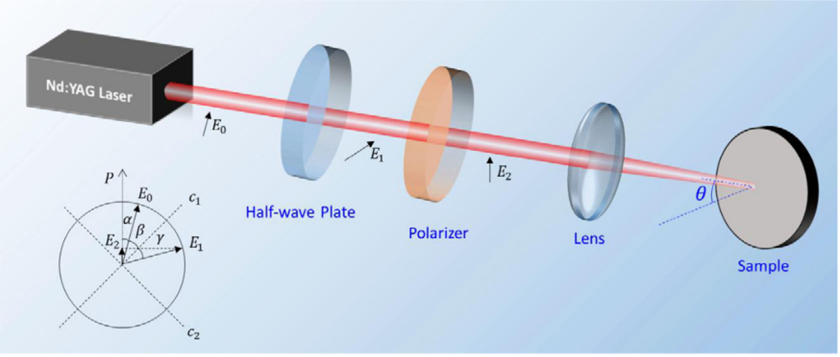

Fig. 1. Schematic of experimental setup for laser treatment on Ti/Ni multilayer thin films.

netic) [22–24] as well as mechanical properties [25]. One of the major applications of Ti/Ni multilayer system is in X-ray optical field for designing highly reflective mirrors, supermirrors, polarizers and other optical elements [26–29]. In addition, Ti/Ni multilayer thin films can be used in a new fabrication process of Ti–Ni shape memory alloys (SMA) by post-processing as-sputtered Ti/Ni multilayer thin films with thermal annealing [30,31] or ion implantation [32]. Ti–Ni SMA have been broadly applied in NEMS/MEMS such as actuators, and bio-medical systems due to their unique mechanical properties and excellent biocompatibility of both Ti and Ni elements [33]. Laser treatment can potentially improve the wear and corrosion resistance and biocompatibility of Ti/Ni multilayer films by forming a Ti–Ni alloy coating on the surface, increasing the probability of applying the Ti/Ni multilayer system for medical applications.

2. Experimental details

In this study, the Ti/Ni multilayer thin films were prepared using the same procedure as in previous work [25]. Briefly, they were deposited on single crystal Si (100) wafers by a dual DC magnetron sputtering system (Orion-5-UHV from AJA International, Inc, MA) with pure Ti (99.995%) and Ni (99.999%) targets. The Si wafers were cleaned by standard RCA cleaning and dipped in deionized water before deposition. The base pressure in the main sputtering chamber was around 1×10^{-7} mbar, and the Ar partial pressure during deposition was around 5×10^{-3} mbar with Ar gas flow rate at 10 sccm. During deposition, the DC powers of both Ti and Ni were fixed as 100 W and alternating Ti and Ni layers were grown on the Si substrate layer by layer starting with Ti and ending with Ni. The sputtering time of each layer was programmed in order to achieve the same thickness for both Ti and Ni layers. In this work, 500 nm thick Ti/Ni multilayer film was deposited with individual layer thickness of 20 nm.

The aforedeposited Ti/Ni multilayer thin films were subsequently treated by a Nd:YAG pulse laser (Leopard SS, Continuum, Santa Clara CA) with pulse duration of 120 picosecond (ps) and wavelength of 1064 nm. Fig. 1 shows the schematic of the experimental setup for the laser treatment. The Nd:YAG laser has a fixed pulse energy of 260 mJ. The combination of a half-wave plate and a polarizer was used as an optical attenuator to adjust the pulse-energy according to the following relationship:

$$\begin{cases} E_1 = E_0 \\ \gamma = \beta \end{cases} \tag{1}$$

$$E_2 = E_1 \cos(\beta + \gamma + \alpha) = E_0 \cos(2\beta + \alpha) \tag{2}$$

where E_0, E_1, E_2 are the light vector magnitudes from the laser, and after passing through the half-wave plate and polarizer, respectively, α is the angle between the polarization direction of the laser beam and the polarizer (which is assumed to be vertical), β and γ are the entering and exiting angles of the laser beam relative to the fast axis of the half-wave plate. During experiment, β was adjusted by rotating the half-wave plate to vary the deposited pulse energy which was measured by a power-meter (Ophir, 10A-V2, MA) placed just before the sample surface. In this work, pulse-energy dependency was studied with a wide energy range from 25 to 150 mJ. A plano-convex lens was used to focus the original laser beam of 10 mm diameter down to a spot with 3 mm diameter. The incident laser beam was directed on the Ti/Ni multilayer surface at an incident angle of 67.5° due to the high reflectivity of sample surface. Each treatment consists of 50 successive pulses with a typical pulse repetition rate of 5 Hz. A three-axis translational stage was used to precisely position the laser spot at a desired location on the sample.

For microstructure characterization, scanning electron microscopy (SEM) was used to examine the evolution of both cross-section and surface morphologies, and X-ray diffraction (XRD) with Cu Kα radiation source was used to study the crystallinity of the non-treated and laser-treated films. Fine scanning was performed with a step of 0.06°/s in order to capture detailed modifications of phase composition and chemical state after irradiation. Atomic force microscopy (AFM) was used to further examine the surface features for laser-treated samples. For mechanical testing, nanoindentation was carried out by an Ubi1 nanomechanical test instrument (Hysitron, Inc., MN) with a Berkovich indenter tip for both non-treated and laser-treated Ti/Ni multilayer films. During indentation, the sample underwent a trapezoidal loading described by a 10 s loading, 5 s hold at maximum load, and 10 s unloading. A minimum of 16 indents were performed on each sample