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Impact of SHI on structural and mechanical behavior of intermetallic NiTi thin films

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A B S T R A C T

In the present investigation, nanocrystalline thin films of NiTi are grown on Si substrate by dc-magnetron co-sputtering using Ni and Ti sputtering targets. These as- grown NiTi thin films are irradiated by 90 MeV Ni ions with fluences 1×10^{12}, 3×10^{12}, 9×10^{12} and 1×10^{13} ions/cm^2, respectively. The elemental composition and depth profile of the pristine film are analyzed by Rutherford backscattering spectrometry. Further, structural, surface morphology and mechanical properties of these films are investigated by X-ray diffraction (XRD), atomic force microscopy (AFM) and nanoindentation techniques, respectively. X-ray diffraction result shows the presence of both austenite and martensite phases in the pristine film with the preferred growth of (110) orientation. The crystallite size is decreased with increase in the ion fluence as compared to pristine film. The AFM images confirm the variation in surface roughness values with the change in the incident ion fluence. The nanoindentation investigation has revealed the enhancement in the mechanical behavior of the NiTi films with ion fluences. The irradiated NiTi film at a fluence of 3×10^{12} ions/cm^2 exhibits higher hardness, elastic modulus and depth recovery ratio and therefore better wear-resistance as compared to other films. This result of nanoindentation indicates the higher ductility of NiTi film in comparison of pristine film and their applicability for Micro-electromechanically system applications (MEMS).

1. Introduction

In the present scenario, the demand for micromachines has increased significantly in various fields such as biomedical, aerospace, biotechnology, industries and various micro-electro-mechanical systems (MEMS) applications [1–3]. Thin films of NiTi have attracted significant research interest due to their excellent biocompatibility and mechanical properties. It is reported that NiTi thin films exhibit superelasticity and shape memory effect that are comparable to the bulk counterpart [4]. The physical origins of these two effects are due to the martensitic transformation between high symmetrical austenite (B_2) and low symmetrical or stress-induced martensite phases (B19′). The appropriate level of biocompatibility and high work output per unit volume of NiTi SMA make it suitable for MEMS-based biomedical and micro-actuators devices fabrication [5–7].

The growth of high purity, quality and stoichiometry NiTi thin film and modification of their properties under swift heavy ion (SHI) irradiation are of immense technological importance for MEMS applications [8,9]. The SHI irradiation technique has attracted the attention of research community for tuning the materials properties, which would be a rapid advancement in the field of materials science [10,11]. At present scenario, ion beam technology is a versatile technology for the development of miniaturized devices which require basic and fundamental understanding about the interaction between ions and matter [12–14]. The deposition of localized energy density into a specified volume is the main advantage of SHI irradiation over thermal equilibrium process [15]. Furthermore, the study of the interaction between ions and matter establishes an interdisciplinary connection between condensed and atomic physics.

As the beam of energetic ions passes through the material, it loses its energy into the material and significantly modifies the material properties. The modifications in material properties depend upon the energy deposited into the material by swift heavy ions; therefore a desired modification in material property could be achieved by choosing the particular beam and fluence, which is not possible by using any other technique [16,17]. The effects of different types of perturbations such as electron, proton, and ion irradiation on microstructure, phase transformation behavior and mechanical properties of bulk NiTi alloy have been reported in literature [18–20], But the investigation of surface characteristic and mechanical behavior of NiTi thin films irradiated with 90 MeV Ni ion irradiation are not adequate and needs to be investigated in detail.

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In the present experiment the effect of SHI irradiation on the structural, topographical and mechanical property of NiTi thin films have been investigated. The SHI irradiation has demonstrated the enhancement of the various properties of nanocrystalline NiTi thin films up to certain fluences and decrements in the properties above that fluence. The variations in structural and mechanical properties at higher fluence are attributed to the grain boundary sliding by imparted of the SHI irradiation on the NiTi thin film, which is explained in detail. This study is also important to use NiTi SMA in a harsh space environment, as in the case of the device designed; the superior tribological and mechanical properties are required [21].

2. Experimental details

In present study, all the thin films of intermetallic NiTi alloy have been deposited by dc-magnetron co-sputtering technique on Si substrate in one deposition. The two separate high purity Ni (99.99%) and Ti (99.99%) targets (Neyco supplier, France) of 3 mm thick and 2-inch diameter have been used for films deposition. A base pressure of 2×10^{-7} torr has been achieved before deposition inside the vacuum chamber by using a rotary and turbo molecular pumps. The deposition has been performed under a pressure of 3×10^{-3} torr using a dynamic throttling valve. Before films deposition, the Si substrate has been cleaned in a mixture of Trichloroethylene and di-water in an ultrasonic bath and then cleaned with the boiled acetone. Further, the films deposition has been performed for 1 h 40 min by two different powers; 100 W for Ti and 50 W for Ni targets, respectively. The deposition of the films has been carried out in argon (~99.9%) atmosphere at the substrate temperature of 550 °C by an AJA Int. make ATC Orion-8 series sputtering system. After deposition, all the deposited thin films have been naturally cooled to room temperature by water cooling arrangement inbuilt to the sputtering system. The distance between the substrate and target holder is kept ~16 cm to get the uniform deposition.

The prepared NiTi thin films have been irradiated at Inter-University Accelerator Centre (IUAC), New Delhi at the room temperature with 90 MeV Ni ions at different fluences of 1×10^{12}, 3×10^{12}, 9×10^{12} and 1×10^{13} ions/cm^2 by 15 UD Tandem Accelerator. During irradiation, a high vacuum (6×10^{-7} torr) is maintained inside the irradiation chamber. The ion beam is focussed on the spot size 1×1 cm^2 over the film area using a magnetic scanner (raster scanning 1×1 cm^2). During the irradiation experiment, the beam current is kept ~2 pnA (particle nanoampere) and the charge state of the Ni ion is found 7+. The final energy of the ions coming out of the accelerator after the switching magnet can be calculated by following equation;

$$E = [E_{deck} + (1 + q)V_T]$$

where q is the charge state of ions after stripping, V_T is terminal potential in MV, and E_{deck} is the deck potential of MC-SNICS source. The electronic energy (S_e) and nuclear energy loss (S_n) have been calculated by the SRIM-2008 code and found to be 1.7×10^3 eV/Å, 0.003×10^3 eV/Å, correspondingly [22]. Furthermore, the range of 90 MeV Ni ions beam has also been calculated by SRIM 2008 computer code in NiTi matrix (density = 7.013 gm/cm^3) and determined as 8.23 µm. The calculated value of S_e is too high than that of S_n; therefore, modifications are mainly attributed due to S_e effect. The values of the fluences have been decided by measuring the charge arising on the film surface under the secondary electron suppressed geometry.

The composition of elements and depth profile of the pristine film have been estimated by Rutherford backscattering spectrometry (RBS). The room temperature crystal structure or phase of pristine and films irradiated at different fluences have been carried out by the Bruker D8 Advance X-ray diffractometer (XRD). The XRD characterization has been performed with a Cu K_α radiation of wavelength (λ = 1.54 Å) with a scan rate of 0.6°/minute at UGC-DAE-CSR Indore. Furthermore, surface features of the pristine and irradiated thin films have been characterized by atomic force microscopy (AFM) (Bruker Nanoscope V system) in tapping mode with a Si_3N_4 cantilever. The mechanical properties of the pristine and the irradiated NiTi thin films have been analyzed by nanoindentation tester equipped (CSM Instrument) with a diamond Berkovich Type indenter tip. Nanoindentation test has been performed on three different positions of the films surface to calculate the average hardness and elastic modulus values. Every nanoindentation test consisted of 8-sec linear loading segment to a peak load and 10-sec holding and again 8-sec linear unloading segments. Mostly the holding period has used to diminish the time-dependent effects (creep effects) caused by the specimen. All these characterizations have been done at room temperature before and after irradiation.

3. Results and discussion

3.1. Rutherford backscattering spectrometry

RBS is a non-destructive technique and it gives the precise estimation of the composition and thickness of the deposited film. The NiTi alloy thin film is very sensitive to the elemental composition and the composition uniformity over the surface. The phase transformation behavior and mechanical strength of the NiTi alloy also depend upon the elemental composition. A small variation in composition (0.1 at.%) could cause a shift in transformation temperature by around 10 °C; consequence shape memory effect occurred below or above room temperature [23]. The superelasticity behavior is also affected by the elemental composition of films. Therefore, the fabrication of the desired composition in the thin film and also the exact determination of the elemental composition of the films are very essential. Fig. 1(a)-(b) represents the simulated RBS spectrum and depth profile for the pristine film deposited on Si substrate at 550 °C. To obtain the film thickness and elemental composition present in the pristine film, the RBS data has been simulated by the SIMNRA software and the fitting of data is shown in Fig. 1(a) [24]. The simulated concentration of the Ti is found to ~43.3 at.% and the concentration of the Ni is found to be ~56.7 at.%. The estimated film thickness is found to be ~270 nm, respectively. Furthermore, Fig. 1(b) shows the graph of the atomic concentration of Ni and Ti versus depth of the film. This Fig. shows the uniform distribution of elements (Ni and Ti) content through the films thickness. A significant diffusion of NiTi into Si substrate (~39 nm) is also observed after the deposition. This diffusion possibly may be due to higher temperature deposition of Ni and Ti.

3.2. X-ray diffraction

Fig. 2 represents the room temperature X-ray diffraction pattern plotted for pristine and the irradiated NiTi thin films. The pristine and irradiated NiTi films represent the several diffraction peaks which indicate the polycrystalline nature of the films. The XRD pattern exhibits two major peaks corresponding to NiTi, in pristine and also in the films irradiated at different fluences. The XRD pattern of the pristine film deposited at 550 °C shows two sharp peaks corresponding to austenite and martensite phase. The XRD patterned of pristine film exhibits the austenite peak located at 2θ = 42.5° (JCPDS file no. 65-5537) corresponding to cubic (110) plane, and martensite peak at 43.9° (JCPDS file no. 77-2170) corresponding to monoclinic (002) plane, respectively. The presence of martensite phase at room temperature in the pristine film could be due to higher bi-axial stress. This type of behavior of martensite phase consistent with the data reported by Martins et al. [25]. The growth of austenite phase corresponding (110) plane in the pristine film could be due to the migration of atoms toward plane with lesser surface energy: here body-centered cubic crystal structure (bcc) which have minimum surface energy along the (110) plane. Due to the minimum surface energy, (110) plane should be favourable in bcc structures [26].

The NiTi thin film irradiated at fluences of 1×10^{12} ions/cm^2 shows