Experimental investigations on the nanomechanical properties of sputter deposited Ni-Mn-Ga ferromagnetic shape memory thin films

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***Abstract*— NiMnGa thin films were dc magnetron sputter deposited onto well cleaned substrates of Si(100) and glass in high pure argon environment of vacuum 0.01 mbar at low sputtering power of 36W and their mechanical properties were investigated by nanoindentation technique using calibrated Berkovich indenter. Results reveal that the films posses elasto-plastic nature. The elastic modulus of the films was found to be thickness as well as substrate dependent, whereas, the indentation hardness of the films was found to be thickness dependent but substrate independent. The highest elastic modulus of the films obtained was 151.02 GPa and highest indentation hardness of the films was found to be 5513.3 MPa.**

Key words: Ni-Mn-Ga thin films, ferromagnetic shape memory materials, nanoindentation, elastic modulus, hardness and mechanical properties.

I. Introduction

The development of intelligent miniaturized systems call for the utilization of variety of new materials and their combinations with varying mechanical, magnetic and thermal characteristics. Therefore, the investigation of basic properties of such materials and their coupling responses are inevitable to acquire in-depth knowledge of the materials for the development of such modern systems. NiMnGa alloy is one of such advanced functional material possessing strong magneto-mechanical coupling. As they are mainly attracted for actuator applications, knowledge of their mechanical properties are imperative to understand their elastic responses. NiMnGa alloy is capable of producing large reversible magnetic field induced strain of up to 10% by the re-orientation of structural variants in self accommodation process associated with ferromagnetic shape memory effect [1-8]. NiMnGa thin films find potential applications primarily in the field of miniaturized actuators and sensors. Literature survey reveals that the mechanical properties of d.c. magnetron sputter deposited NiMnGa thin films have not been much investigated and reported and hence the present work.

Indentation based methods for mechanical characterization of materials have become popular recent days and been widely recognized for their potential in obtaining fundamental insights of several aspects of mechanical behavior of materials [9,10]. Mechanical properties of materials change dramatically when their dimensions are reduced very small. Thus the knowledge of mechanical properties of materials on the micro and naometer scales is required for the development of miniaturized devices [11]. In recent days, there has been a growing interest on the tribological and mechanical analysis of surfaces and films of new materials as they have found resourceful applications in various hybrid systems like micro and nano electro mechanical systems (MEMS and NEMS) which requires actuation and sensing mechanisms with high efficiency. Where such high efficacy actuation mechanisms are concerned, one is immensely interested in understanding of the physical mechanism of the thin film elements and hence the knowledge of their mechanical responses becomes inevitable for designing actuators which work on micro and nano scale dimensions [12]. There is considerable interest in the use of nanoindentation as a measure of characterizing mechanical properties of such small-scale specimens, notably, the Young's modulus and hardness [13]. Hence, in the progress of micro and nano systems technology, nanoindentation has gained importance for the estimation of nanomechanical properties.

In indentation based methods, the material to be investigated is indented with a conical or pyramidal or spherical indenter and the force-depth relationships during the loading and unloading cycles are recorded. In all indentation tests, the indenter is loaded at a specific rate and the load is then held constant for a period of time while the displacement is measured, subsequently unloaded. From the load-displacement curves, the mechanical properties of the materials are extracted. By nanoindentation method, it is possible to determine the two important properties of the materials viz. Young's modulus (E) and the hardness (H) in a single test [9,12,14,15]. The another vital term connected to mechanical properties is 'elastic recovery ratio', which is defined as the ratio of residual indentation depth to the maximum indentation depth (h_r/h_{max}). This is an important parameter to explain the mechanical behavior of the material. For a complete elastic deformation, $h_r/h_{max} = 0$ and for a complete plastic deformation, $h_r/h_{max} = 1$. The values of elastic recovery ratio in-between 0 and 1 of indicates the degree of elastio-plastic nature of the material. Since Ni-Mn-Ga is a strong magneto-mechanical responsive material; the present study was primarily intended to know the mechanical properties such as elastic modulus (Young's modulus) and indentation hardness of the d.c. magnetron sputter deposited NiMnGa thin films using nanoindentaion technique.

II. EXPERIMENTAL DETAILS

Ni-Mn-Ga thin films were sputter deposited onto well cleaned substrates of Si(100) and glass using D.C. magnetron sputtering unit under high pure argon atmosphere of 0.01 mbar at a low sputtering power of 36W. The target used was a 2" diameter thin disk which was wire cut from the prepared bulk alloy in our laboratory using vacuum induction-melting technique. The composition of the sputtering target is $Ni_{50}Mn_{30}Ga_{20}$. The composition of the deposited thin films was identified using electron probe micro analysis (EPMA) technique. The thickness of the thin films was measured using an atomic force microscope (AFM). The elastic modulus and hardness of the thin films were determined by nanoindentation studies using calibrated Berkovich indenter. The Nanoindentation tests were performed on the thin films at 3 different places for each sample. The loading and unloading rates were 30 sec with a pause of 10 sec for a maximum load of 500 μN.

III. RESULTS AND DISCUSSION

The composition of the films was found to be: $Ni_{55}Mn_{24.5}Ga_{20.5}$. The typical nanoindentation load-displacement curves obtained for three different NiMnGa samples are shown in Fig. The mechanical properties extracted from the loan-displacement curves of those samples are given in the Table.

Elastic modulus and Indentation Hardness:

Fig. reveals that the initial and final portions of the loading and unloading curves for the films with labels 4.1 and 4.6 merge each other in these regions below 0.05 mN, even though they take different paths beyond which. It can also be observed that the maximum indentation depth and residual indentation depths for the film with label 2.4 is greater than that of other two films. This may be due to the film thickness and the effect of the substrates for indentation process.

The average value of the residual indentation depth (h_r) for our film of thickness 600 nm is found to be 47.81 nm and the average maximum indentation depth (h_{max}) for the same sample is 70.67 nm. The average value of residual indentation depths and the average maximum indentation depths for the films of thickness 450 nm are found to be almost equal which seem to be substrate independent. But, the results reveal that the elastic modulus and hardness of the thin films are substrate dependent.

For, Ni-Mn-Ga system, the stresses viz. thermal, mechanical and magnetic are not totally mutually exclusive, and hence the mutual dependence of these three parameters may collectively influence the material characteristics. This collective response is quiet complex to interpret for the Ni-Mn-Ga intermetallic alloy due to its multifunctional nature and the evidences of the formation of various commensurate pre-martensitic microstructures (which might show different properties). Hence, due to the mutual dependencies of thermal, mechanical and magnetic forces in these special materials, unless we dynamically test and systematically investigate the occurrence of micro structural changes in these materials during the indentation process through some in situ arrangements, it will be quiet difficult to explain the entire mechanical behavior of such special alloy systems under deformation processes.

From our experimental observation we found that, the Young's modulus of the film labeled 4.1 was found to be 100.33 GPa , and for the same thickness the Young's modulus of Ni-Mn-Ga film labeled 4.6 was found to be 151.02, which are of the order of bulk alloy reported [16] and the thin films prepared by r.f. magnetron sputtering method [17] . These observations reveal that the value of elastic modulus is substrate dependent. The elastic modulus of the thin films also found to be thickness dependent (by comparing these values for the films labeled 4.1 and 2.4) as reported in literature that the thinner the film the larger the elastic modulus [18].

One could expect that softening of the elastic constant occurs at the transition points, particularly, at structural transitions due to stress-induced martensitic transformation (superelasticity) [19,20]. But such inference could be resolved through suitable electron microscopy analysis of the indented area that too dynamically during loading and unloading process of nanoindentation, which is challenging.

IV. CONCLUSSIONS

Ni-Mn-Ga thin films were prepared onto well cleaned substrates of si(100) and glass and their mechanical properties namely, elastic modulus and hardness were investigated by nanoindentation technique. The load-displacement curves reveal that the films show elasto-plastic nature. The elastic modulus of the NiMnGa thin films was found to be thickness as well as substrate dependent, whereas, the indentation hardness of the films was found to be thickness dependent and substrate independent. The highest indentation hardness obtained was 5513.3 MPa and the corresponding elastic modulus was found to be 151.02 GPa.

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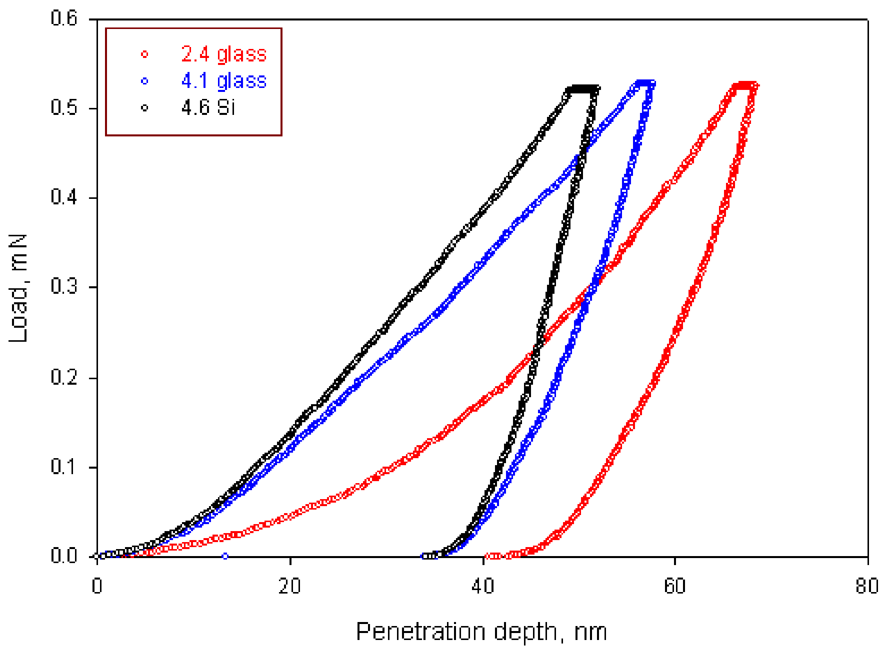

Fig. Typical Load-displacement (P-h) curves of NiMnGa thin films

TABLE

Nanomechanical Properties of NiMnGa thin films

Substrate	Film Thickness (A°)	Load (mN)	H_{IT} (MPa)	E_{IT} (GPa)	HV (Equivalent)	h_{max} (nm)	h_r (nm)
Glass (4.1)	4500	0.5	5353.8	100.33	495.82	55.36	32.47
Si(100) (4.6)	4500		5513.3	151.02	510.49	51.33	34.44
Glass (2.4)	6000		3251.8	76.58	301.15	70.67	47.81

H_{IT} : Nanoindentation Hardness
E_{IT} : Elastic Modulus
h_{max} : Maximum indentation depth
h_r : Residual indentation depth