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Effect of Annealing on Hardness and Elastic Modulus of Invar36 Thin Films Deposited by Direct Current Sputtering for Strain Gauge Applications

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Abstract: Invar36 thin film was deposited at room temperature on p-type silicon (100) substrates using DC magnetron sputtering technique. In order to investigate the post-annealing effect on the structural characteristics and mechanical properties of the prepared films, they were vacuum annealed for one hour at different temperatures viz. 200°C, 400°C and 500°C. Composition analysis, phase structure, microstructure and roughness of as-deposited and annealed Invar36 thin films were investigated by energy dispersive X-ray analysis (EDX), X-Ray diffraction (XRD) and Atomic Force Microscopy (AFM). Mechanical properties of Invar36 thin films were studied by nano indentation method. EDX analysis revealed a variation in nickel content with annealing. The XRD measurements indicated the phase transformation of Invar36 thin film with annealing. AFM analysis implied uniform surface morphology of the films, increase of surface roughness and grain size with annealing. The hardness (H) of the film decreased with annealing. Hardness of as-deposited, annealed at 200ºC, 400ºC and 500ºC were found as 8.5±0.96 GPa, 7.64±0.35 GPa, 6.34±0.14GPa and 3.95±1.05 GPa, respectively. The elastic modulus of Invar36 thin films was increased with annealing. Elastic modulus of as-deposited, annealed at 200ºC, 400ºC and 500ºC were found as 157.00±25.49 GPa, 166.0±11.8 GPa, 172.00±9.93 GPa and 176.00 ±10.78 GPa, respectively. These results are explained on the basis of the change of microstructure after annealing and the effect of the same on the mechanical properties of Invar36 thin films for strain gauge applications.

Keywords: Invar36 thin film, microstructure, Elastic modulus, hardness, strain gauge.

1 Introduction

Strain gauge is a fundamental sensing element for pressure sensors, load cells, torque sensors, position sensors and others [1]. Because of number of advantages over foil and semiconductor strain gauges, thin film strain gauges are widely used for many applications [2,3]. Commonly used materials for the development of thin film strain gauges are metals, alloys, cermets, semiconductors and polymers [4].

For certain applications dimensional stability of sensing material over wide temperature range needs to be stable for which the sensing material should possess lower coefficient of thermal expansion (CTE). Invar36 alloydeveloped by Guilleaume in 1898 exhibits low CTE and high dimensional stability over a wide range of temperatures [5]. Hence this alloy is utilized in many industrial as well as in micro-electro-mechanical system applications [6,7,8]. Rajanna et al. [9] studied strain sensitivity and temperature dependence of sputter deposited Invar36 thin film. Kalpana et al[10,11] studied the effect of annealing and thickness on the electrical properties of Invar36 thin films and also measured the gauge factors. In addition to these studies the analysis of structural and mechanical properties is essential to verify whether the sensing element is suitable for strain gauge application or not.

Mechanical properties of thin films often differ from those of bulk materials [12]. The experimental study of mechanical properties of thin films requires the

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development and use of nontraditional mechanical testing techniques. Indentation based methods for mechanical property measurements of thin films are popular and more versatile than any other methods [13]. Nanoindentation is one of the most widely used contact mechanical methods for the determination of mechanical properties of thin films [14,15]. The advantage of nanoindentation is that the films can be used readily without the necessity of peeling them off from the substrate. At the same time, the substrate does not affect the intrinsic film properties measured as long as the indentation size is significantly less than the film thickness [16].

It is well known that post annealing process strongly influences the structural and mechanical properties of thin films. Due to the development of internal stresses during sputtering process Invar36 thin films may not be completely stabilized. As-deposited Invar36 thin films are body centered cubic (BCC) structures and are not completely crystallized in nature. Mechanical properties of Invar36 thin films are very much dependent on the film structure stability; whereas films with face centered cubic (FCC) structure remain stable after heat treatment. Therefore for strain gauge application stabilized Invar36 thin films with FCC structure are preferred. This can be obtained by minimizing the internal stresses by annealing the films in vacuum above their crystallization temperature. Hence in this work the influence of annealing on structural and mechanical properties of Invar36 thin films deposited on p-type silicon (100) substrates by DC magnetron sputtering technique for strain gauge application is systematically investigated.

2 Experimental Details

2.1Materials

The target is an Invar36 foil (procured from ESPI Metals) bonded on copper disc by using silver paste. Although nickel and iron are the major constituents of Invar36 alloy, addition of small amounts of materials such as carbon, manganese etc is known to improve the characteristics of Invar36 alloy. The composition of Invar36 alloy are given in Table elsewhere[10].

P-type silicon (100) wafer (1cm×1cm) with resistivity of 5-10μΩ-cm are used as substrates to deposit Invar36 thin films. Substrates are cleaned by using Radio Corporation of America (RCA) cleaning method [17].

2.2 Deposition of Invar36 Thin Film by DC Magnetron Sputtering

Invar36 thin film was deposited on silicon substrates by DC magnetron sputtering technique at room temperature[18,19]. Planar DC Magnetron Sputtering Unit comprising of diffusion pump backed by a rotary pump was used for

deposition of Invar36 thin film and explained by kalpanaet.al[11].

2.3 Characterization Techniques

In order to study the enhanced properties due to post-deposition annealing of Invar36 thin films they were subsequently annealed in vacuum. Samples were held in horizontal position by a special jig (the substrate holding and heating arrangement). A k type thermocouple was installed with the arrangement for temperature sensing. A PID controller with ±1°C tolerance was employed as temperature controller. The samples were annealed in vacuum (10^{-5} mbar) for one hour at temperatures of 200°C, 400°C and 500°C.The heating rate was 2-3°C/min, and the cooling rate was 1°C/min. Annealing was carried out to increase the crystallanity and stability of the films as well as mechanical properties. The as-deposited and annealed films composition was determined by energy dispersive X-ray spectrometry (EDX) performed using scanning electron microscope (JEOL JSM-5600LV), and the average film composition was found as approximately Ni 36% and Fe 60% atomic weight.

Phase orientation analysis of films was carried out using PANanalytic X-ray diffractometer operated at 40KV, 20mA with CuKα ray tube (λ = 1.542Å) configured in symmetrical θ-2θ mode. The surface roughness of films was measured using Atomic Force Microscope (AFM Xe-70 Park system) in contact mode.

Nanoindentation tests were carried out by employing a TriboscopeNanomechanicalTesting System (Hysitron TI-900, Minneapolis).Three sided pyramidal Berkovich tip of diamond was employed for characterization of as-deposited, annealed Invar36 thin films at 200ºC, 400ºC and 500ºC. Tip diameter of the indenter was 200nm, load resolution was 1nN and depth resolution was 0.04nm. A suitable penetration load was identified by carrying the tests over a range of loads where the depth of indentation was such that it was more than the surface roughness but less than the film thickness to ensure that there is no substrate effect. In all the tests, films were loaded to a maximum load of 300μN and were held constant for 10s followed by unloading. The loading and unloading rate was maintained constant for all nanoindentation tests. The load displacement experiments were repeated at six independent locations on the surface of each film. In order to decrease the possible interaction between adjacent indents, all indents were separated by sufficient distance. Penetration depth of all indents were from 17nm to 36 nm. Measurements from six indents were averaged to determine the mechanical properties of as-deposited and annealed Invar36 thin films.

Hardness and reduced Elastic modulus were determined by means of the Oliver and Pharr's analysis method [20]. This method is the standard method which is frequently used for thin film mechanical property analysis. Elastic modulus was determined using the effective elastic modulus, E_{eff} which is given by the equation

$$E_{eff}=\frac{1}{2}\sqrt{\frac{\pi}{Ac}}\frac{dP}{dh}(1)$$

dP/dh is the slope of the unloading curve and Ac the projected contact area at the maximum load.
The final Elastic modulus, E, is obtained from the expression (2)

$$\frac{1}{E_{eff}}=\frac{1-v^2}{E}+\frac{1-vi^2}{Ei}(2)$$

For a diamond indenter Elastic modulus E_i = 1141 GPa and poisons ratio ν_i=0.07
Poisons ratio of Ni-Fe alloy is ν= 0.27 [21].
The hardness is represented as a ratio of peak load (P_{max}) to the contact area (Ac) as given in equation(3)

$$H=\frac{P_{max}}{Ac}(3)$$

3 Results and Discussion

3.1Effect of post Annealing on Elemental Composition

The properties of deposited Invar36 thin films may get altered due to change in their composition. The composition of films depends on the deposition process parameters such as target power, operating pressure, target to substrate distance and temperature of the substrate. To ensure the invar composition it is necessary to know the proper percentage of iron (Fe) and nickel (Ni) in the deposited thin films**.** The Invar36 thin films of thickness 120 nm deposited on p-type (100)silicon substrates of dimension 5 × 5 mm at ambient temperature were selected for composition analysis in as-deposited and annealed at 200, 400 and 500°C. The composition of as-deposited and annealed films was analyzed at different locations on the film with the help of energy dispersive X-ray analysis (EDX).
The EDX spectra of as-deposited and films annealed at 200, 400 and 500°C are shown in Fig.1 (a-d) respectively. The observed strong peak at 1.8 KeV is attributed to the silicon substrate. The summary of results presented in Table 1 indicates almost same value of Fe and Ni in as-deposited and annealed films with traces of carbon (C) and manganese (Mn). The observed composition of Fe and Ni in as-deposited and annealed films matched with the composition of bulk Invar36 foil. The results indicate annealing has an insignificant effect on the composition of deposited films.

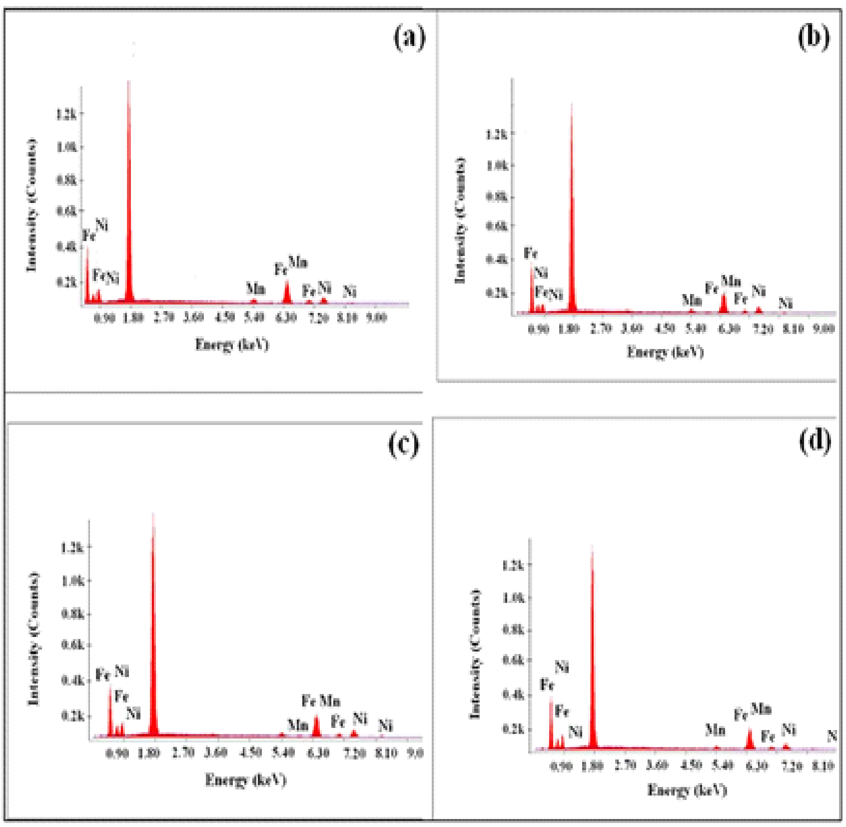

Fig. 1: EDX spectrum of Invar36 thin films in (a) as-deposited (b) annealed at 200°C (c) annealed at 400°C and (d) annealed at 500°C.

Table 1: Composition of as-deposited and annealed Invar36 thin film.

Sl No	Condition of the Film	Fe	Ni
1	As-deposited	59.85	33.06
2	Annealed @ 200 °C	60.72	32.63
3	Annealed @ 400 °C	59.24	35.88
4	Annealed @ 500 °C	59.94	33.13

3.2 Effect of post Annealing on Crystal Structure

X-ray diffraction measurements were performed to analyze the crystal structure of the as-deposited Invar36 thin film and the transformation of the same with post annealing at different temperatures. X-ray diffraction patterns of 120nm thick as deposited as well as annealed Invar36 thin films on p-type (100) silicon substrates are shown in Fig.2(a-d). Mechanical properties of Invar36 thin film are related with preferred orientation. Annealing at different temperatures affected the crystallographic texture of the film. The values of crystallite size, dislocation density, strain, density of grain boundary and lattice constant of as-deposited and annealed Invar36 thin films were calculated from the results obtained by X-ray diffraction studies and are tabulated in Table2.

The as-deposited film exhibits body centered cubic (BCC) structure (α phase) with (220) orientation as evident from diffraction peak observed at 2θ = 65.23° (Fig.2 (a)).

The film annealed at 200ºC (Fig. 2(b)) exhibits a mixture of α and γ phases. The observed diffraction peak (220) at 2θ = 65.12° corresponds to α phase which represents BCC structure. The peak observed at 2θ = 43.12° corresponds to crystal orientation of (111) which represents FCC structure of the film. Film annealed at 200ºC indicates onset of crystallization compared to as-deposited film

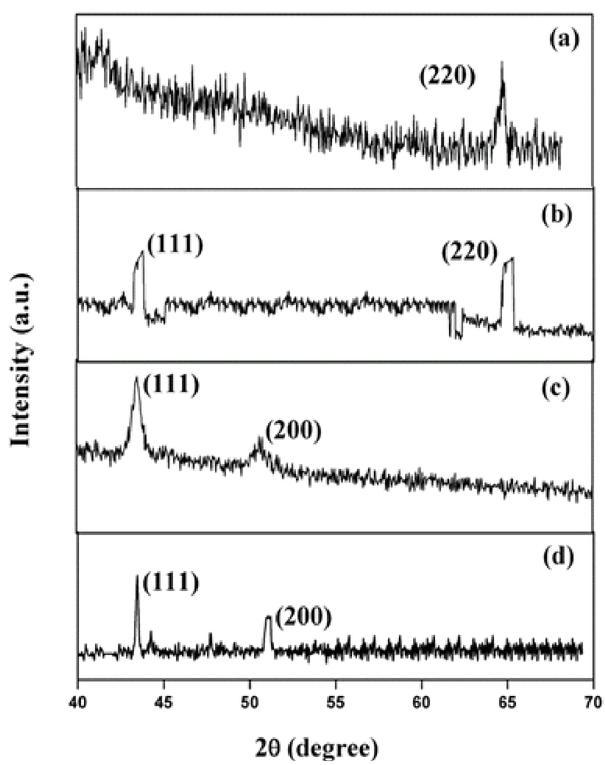

Fig.2:XRD patterns of Invar36 thin films in (a) as-deposited (b) annealed at 200°C (c) annealed at 400°C and (d) annealed at 500°C

[22,23].Thefilm annealed at 400°C (Fig. 2(c)) exhibits pure γ phases. The observed two diffraction peaks, one at 2θ = 43.47° with a crystal orientation of (111) and other at 2θ = 50.82° with a crystal orientation of (200) corresponds to FCC structure. This indicates phase transformation of the deposited films due to annealing [24, 25].

The film annealed at 500°C (Fig. 2(d)) exhibits γ phases with increased crystallite size. The observed two diffraction peaks at 2θ = 43.71° and 2θ = 50.81° correspond to γ phase with (111) and (200) orientations respectively. This indicates a complete transformation of the deposited film from BCC to FCC phase [26,27].Phase transformation of invar thin films after annealing is supported by previous findings. G.Dumpich et al[24]studied the structure of evaporated Fe68Ni32 thin films by X-ray diffraction. They observed the occurrence of mixed BCC and FCC phases for as-prepared films and the disappearance of the BCC phase after annealing. Anand Kumar Dokania et al. [8] prepared invar thin films by magnetron sputtering and analyzed the phase using XRD studies. They observed combination of BCC and FCC phases in as-depositedfilms and FCC phase in annealed films using rapid thermal annealing (RTA) at 600°C.

The average crystallite size of as-deposited film with an orientation of (220) exhibited a crystallite size of 13.07 nm. The films annealed at 200, 400 and 500ºC showed a crystallite size of 14.46, 28.9 and 36.1 nm respectively with an orientation of (111)[Table 2]. The observed difference in crystallite size of as-deposited film with annealed films is attributed to the presence of large amount of structural defects due to deposition process as well as beginning of accumulation and condensation of adatoms [28]. An increase in crystallite size in the film annealed at 200ºC is attributed to onset of crystallization and decrease in the number of structural defects induced in film during the deposition process. The observed significant increase in crystallite size in the film annealed at 400ºC is attributed to the reorientations of the grains that are taken place as a result of changes in grain boundary and dislocation densities. Further increase in crystallite size in the films annealed at 500°C is attributed to the densification of film with accompanied grain growth, micro pore vanishing and decrease of stress. Results are in agreement with previous works. Senoy Thomas et al. [29] obtained vapour deposited thin films of Fe-Ni and carried out structural and micro structural characterization. The XRD pattern of as-deposited thin film was found to be amorphous in nature without any sharp diffraction peaks. As films were annealed at 100,200 and 300 ºC there was an increase in the grain growth represented by sharp FCC peak. .R. Abdel-Karimet.al[30] obtained crystallite sizes of Fe-Ni alloys between 20 and 30 nm from the XRD peaks at (111).

The variation of dislocation density of as-deposited and annealed at 200, 400 and 500ºC Invar36 thin film at respective diffraction peaks is tabulated in Table 4. The observed high value of dislocation density of as-deposited film is attributed to the presence of defects, grain boundaries and surface irregularities. As the films are annealed a gradual decrease in its value is observed because of vanishing of dislocations due to annealing.

The variation of strain and density of grain boundary of as-deposited and annealed at 200, 400 and 500ºC Invar36 thin film at respective diffraction peaks are tabulated in Table 2. The result indicates that as-deposited films exhibit high values of strain and density of grain boundary. The decrease in their values with annealing is attributed to release of stress and lattice/grain boundary relaxation and recrystallization due to diffusion.

It is evident from X-ray diffraction studies that annealing of Invar36 thin films resulted in increase in their lattice constant [Table 2]. The as-deposited Invar36 thin films exhibited a lattice constant of 1.76 Å for the BCC phase with (220) orientation whereas it is increased to 2.09 Å for annealed at 200ºC with same orientation and phase. The films annealed at 200ºC also showed a lattice constant of 2.72 Å for the FCC phase with (111) orientation. The films annealed at 400 and 500ºC exhibited a lattice constant of 3.58 Å and 3.60 Å respectively with FCC phase and (111) orientation. The increase in lattice constant is attributed to the expansion of unit cell due to annealing [31]. Kyung Hunn Han et al. [28] obtained lattice constant for 60nm thick Fe-Ni film with 40%Ni as 3.59Å. G.Dumpich et al[24] calculated lattice constant from the XRD peaks. For as deposited the value obtained for BCC phase was 2.86 Å and for FCC phase 3.5 Å. For annealed films they obtained lattice constant as 3.595 Å. M. Kadziolka-gawel et.al[32] obtained lattice constant of Fe-Ni alloy as 3.599 Å. From these comparisons it is observed that the lattice constant values obtained in the present work are in line with previous findings.

3.3 Effect of Post Annealing on Microstructure and Surface Roughness

Atomic force microscopy (AFM) was employed to analyze the influence of post annealing on microstructure and surface roughness of Invar36 thin films prior to nanoindentation tests to fix up the depth of indentation. If the surface roughness is too high it leads to the reduction of penetration depth which in turn causes the measurement of hardness and Elastic modulus values dispersive and uncertain. AFM images of 120 nm thick Invar36 thin films of as deposited, annealed for one hour at 200°C,400°C and 500ºC deposited on Si(100) substrates are shown in Fig. 3. All films were scanned through 5µm × 5µm area in contact mode to determine surface roughness.

The AFM image of as-deposited Invar36 thin film shows a very smooth surface with uniform accumulation of small grains and a root mean square (RMS, Rq) roughness of 1.12 nm (Fig.3(a)). The observed insignificant grain growth in as-deposited films is attributed to the presence of defects and stresses developed during the deposition process.

The AFM image of Invar36 thin film annealed at 200°C shown in Fig.3 (b) shows a smooth surface with a roughness of 1.55 nm. It indicates the onset of grain growth

Table 2: XRD analysis data of as-deposited and annealed Invar36 thin films.

Condition of The film	**Phase of The film**	**Orientation**	**Crystallite size (nm)**	**Dislocationd ensity ($10^{16}/m^2$)**	**Strain(10^{-3})**	**Density ofgrainbou ndary ΔV_F**	**Latticeco nstant(Å)**
As-deposited	BCC	(220)	13.09	58.3	2.64	7.78	1.76
Annealed@ 200 °C	BCC	(220)	4.9	416.4	7.05	21.44	2.09
	FCC	**(111)**	**14.46**	**47.8**	**2.3**	**7.03**	**2.72**
Annealed@ 400 °C	**FCC**	**(111)**	**28.9**	**11.9**	**1.19**	**3.4**	**3.58**
	FCC	(200)	13.9	5.17	2.4	7.3	3.11
Annealed@ 500 °C	**FCC**	**(111)**	**36.1**	**7.6**	**0.9**	**2.78**	**3.60**
	FCC	(200)	17.8	31.5	1.93	5.69	3.10

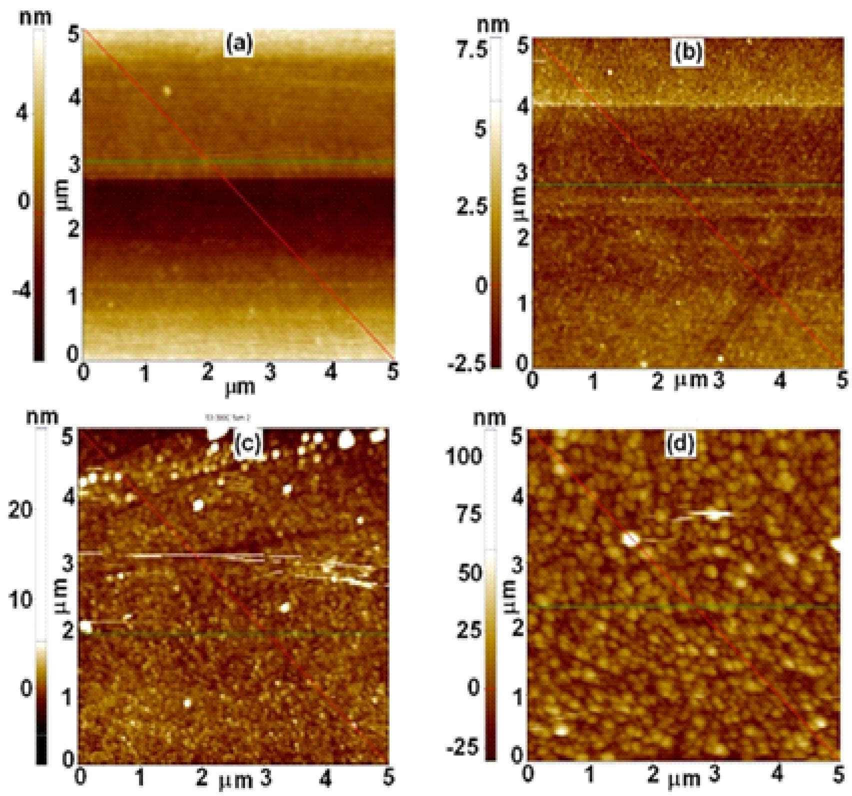

Fig. 3: AFM surface images of Invar36 thin films in (a) as-deposited (b) annealed at 200ºC (c) annealed at 400ºC and (d)annealed at500ºC.

The AFM image of Invar36 thin film annealed at 500°C shown in Fig .3(d) shows a rougher surface with RMS roughness of 12.25 nm. It is observed that annealing of the deposited films at higher temperature results in increase of grain size which is attributed to crystallization due to release of internal stresses [29]. The AFM (Fig.4) results are consistent with XRD (Fig.2) results.

The variation of RMS roughness with annealing temperature of Invar36 thin film is shown in Fig. 4. It is observed that annealing of the films results in increase in RMS roughness of the film as compared to as-deposited film. A two-fold increase in the value of RMS roughness is observed in the film annealed at 400ºC as compared to film annealed at 200ºC. A drastic increase in its value is observed in the film annealed at 500ºC.

3.4 Effect of Post Annealing on Mechanical Properties

Mechanical properties such as Elastic modulus and hardness of 120nm thick Invar36 thin films deposited on p-type silicon (100) substrates were evaluated using nanoindentation technique. Hardness and Elastic modulus of films depend on various factors such as yield stress, elastic modulus of substrate, film/substrate adhesion, film thickness, and indenter geometry. Typical load-displacement curves of as-deposited and annealed films are shown in Fig.5. They contain information about the elastic behavior and plastic deformation of thin film. To minimize the substrate, effect the load applied is limited to 300μN. At the same load, the annealed films exhibit a larger displacement as shown in Fig.5.

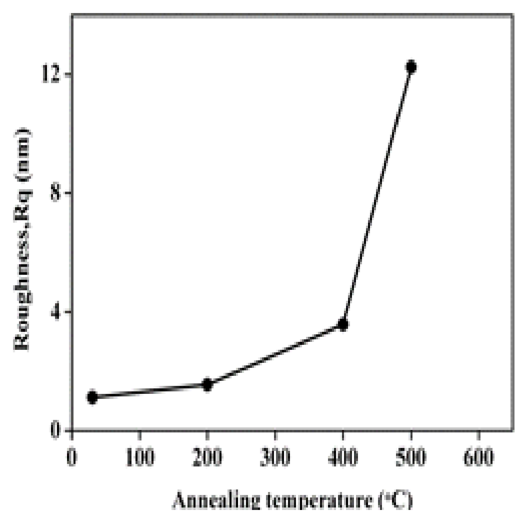

Fig.4: Surface roughness of Invar36 thin film at different annealing temperatures.

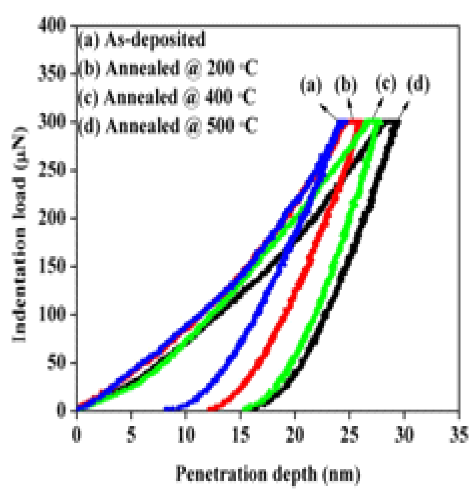

Fig. 5: Load-displacement curves of Invar36 thin films in (a) as-deposited (b) annealed at 200ºC (c) annealed at 400ºC and (d)annealed at500ºC.

The load-displacement curves indicate overlapping of as-deposited (Fig.5(a)) and annealed at 200°C (Fig.5(b)) curves during loading cycle and they follow different path during unloading. The maximum indentation depth for film annealed at 200°C is observed to be more than that of the as-deposited film. The load-displacement curves of films annealed at 400°C (Fig.5(c)) and at 500°C (Fig.5(d)) overlap each other during loading cycles up to a load of 100 μN and follow different paths with further increase in load as well as during unloading cycle. The observed shift during the loading and unloading cycle is attributed to softness of the film as a result of annealing.

The hardness of as-deposited and vacuum annealed at 200ºC,400ºC and 500ºC Invar36 thin films are evaluated from the load-displacement (p-h) curves shown in fig.6.It is known that hardness of alloy thin film depends on film stress and microstructure. In particular grain size of the film affects the hardness. The stress state of a film can also affects its hardness. The as–deposited film exhibits higher hardness and as the films are annealed, there observed a decrease of hardness. This is attributed to the relaxation of stress with annealing. From the microstructure study of Invar36 thin film shown in Fig.2 and Fig.3 it is evident that as the annealing temperature is increased from room temperature to 500°C there observed an increase of grain size. According to Hall-Petch behavior of thin films as the grain size is increased the hardness of the film decreases [34,35]**.** Plots of hardness as a function of indenter penetration depth, for as-deposited and annealed Invar36 thin films are shown in Fig.6. It is evident that there is very little scatter among the data points and the error bars represent the standard deviations of the measurements among six indents.

The variation of Invar36 thin film hardness with annealing temperature is shown in Fig.7. Each point is an average of six indentations with standard deviations. Hardness of as-deposited film is found as 8.5±0.96GPa. The hardness decreases to7.64±0.35 GPa after annealing at 200ºC, 6.34±0.14GPa after annealing at 400ºC and 3.95±1.05GPa after annealing at 500ºC.

The reduced Elastic modulus is determined from the slope of the initial unloading part of the load-displacement curves shown in Fig.5, using equation (1). Elastic modulus of as-deposited and annealed Invar36 samples at six indentations are calculated using equation (2) and plotted against penetration depth for the same load as shown in Fig.8. It is evident that there is very little scatter among the data points and the error bars represent the standard deviations of the measurements among six indents.

The variation of Invar36 thin film Elastic modulus with annealing temperature is shown in Fig.9. Each point is an average of six indentations with standard deviations. Elastic modulus of as-deposited film is found as 157.00±25.49 GPa. The Elastic modulus is increased after annealing at 200ºC to 166.00±11.8 GPa, after annealing at 400ºC it is increased to 172.00±9.93 GPa and 176.00 ±10.78 GPa after annealing at 500ºC.The increase of Elastic modulus is attributed to grain growth of the Invar36 thin film [35,36]. Yu-Jung Hsu et.al [28] found the elastic modulus of d.c magnetron sputtered invar films of 67%Fe-33%Ni, approximately 184 to 189 GPa and the result obtained in the present investigation are also on the same line. Xiamin Liu et.al [34] studied the effect of annealing on hardness and elastic modulus of electroplated Fe-Co-Ni thin films. The hardness of as-deposited films was obtained around 4.8 GPa, which decreased to 3.6 GPa after annealing at 400ºC.

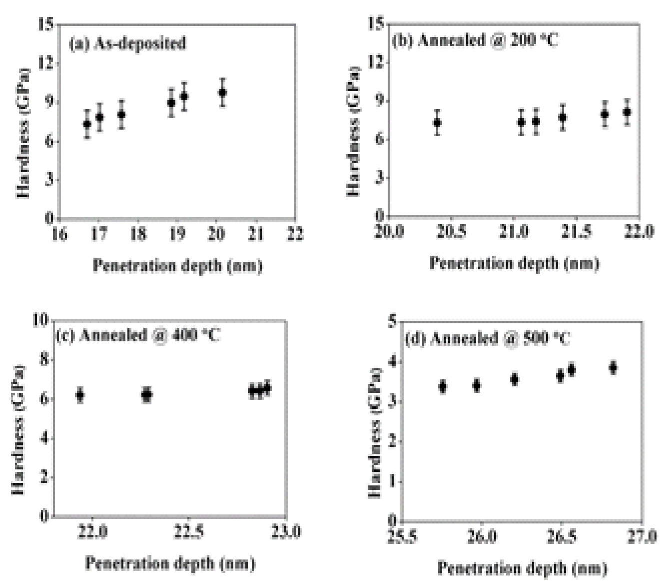

Fig.6: Hardness of Invar36 thin films at different penetrations depth.

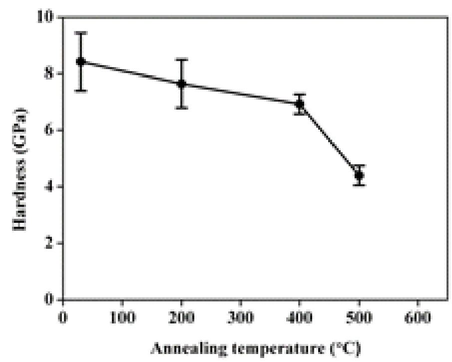

Fig. 7: Hardness of Invar36 thin film at different annealing temperatures.

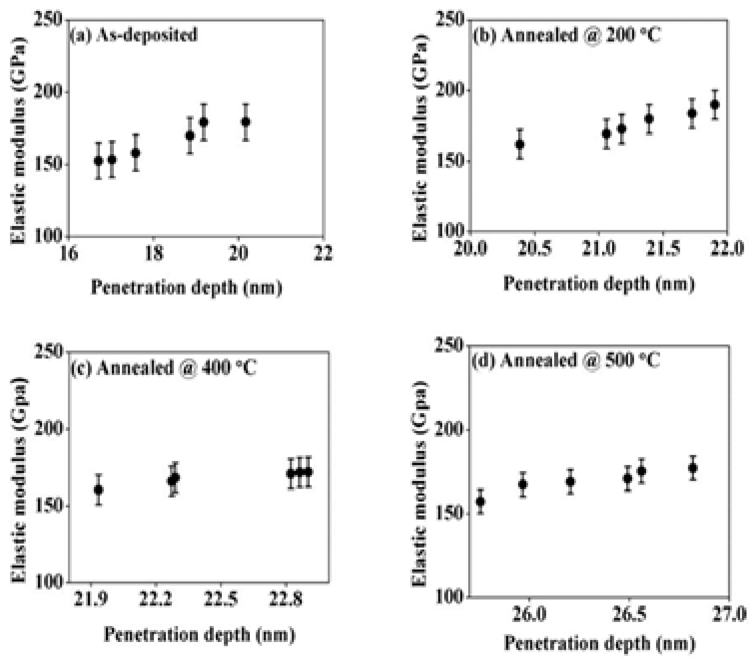

Fig. 8: Elastic modulus of Invar36 thin films at different penetrations depth.

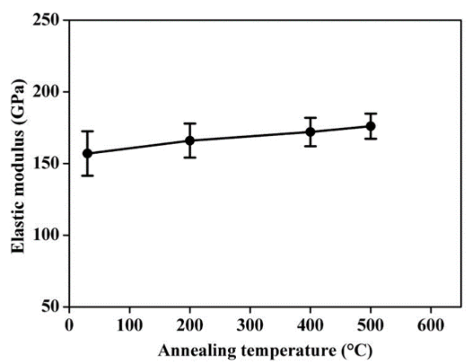

Fig. 9: Elastic modulus of Invar36 thin film at different annealing temperatures.

4 Conclusions

Invar36 thin films are deposited on p-type silicon (100) substrates at room temperature by DC magnetron sputtering. The as-deposited films are annealed in vacuum(10^{-5} mbar) for one hour in the temperatures of 200°C, 400°C and 500°C. The effect of annealing on composition, structural and mechanical properties of Invar36 thin film are studied. EDX analysis of Invar36 thin film shows the concentration of Ni in the invar region for all conditions. XRD analysis of Invar36 thin film reveals the increase of crystallite size from 16nm to 36 nm and phase transformation from BCC to FCC with post annealing. The surface roughness of as-deposited film is found lesser than annealed films and the roughness of annealed films are found lesser than 13nm.

Mechanical properties like hardness and Elastic modulus of Invar36 thin film are investigated by nano indentation technique. The hardness of as-deposited film is found as 8.5±0.96 GPa, which is decreased to 7.64±0.35 GPa after annealing at 200ºC, 6.34±0.14GPa after annealing at 400ºC and 3.95±1.05 GPa after annealing at 500ºC. The value of Elastic modulus of as-deposited film is found as 157.00±25.49 GPa, it is increased to 166.00±11.8 GPa, 172.00±9.93 GPa and 176.00 ±10.78 GPa after annealing at200ºC, 400ºC and 500ºC, respectively. Post annealing effect brings favorable changes in structural and mechanical properties of Invar36 thin films which are suitable for strain gauge applications.

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