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THERMAL EXPANSION OF INVAR THIN FILM FOR MEMS APPLICATION

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ABSTRACT

In recent years Invar as a thin film has found application in many electronic industries. Invar as a thin film demands post annealing to get pure gamma phase by alpha to gamma phase transformation reaction. First time, a Rapid Thermal Annealing (RTA) technique is employed for this transformation reaction. The coefficient of thermal expansion (CTE) has been investigated using stress measurement against temperature. A CTE as low as 1.63ppm/K is observed.

Keywords: Invar, Thermal expansion, MEMS.

1. INTRODUCTION

In 1898 Guilleaume discovered that the coefficient of thermal expansion (CTE) of Fe-Ni alloys with f.c.c. phase exhibits a sharp minimum at around 36wt% Ni. This particular alloy, called "Invar" alloy, short for invariable, has almost zero expansion at room temperature[1]. In addition to low CTE, Fe-rich alloys show many other anomalous properties such as (i) large negative pressure effects on the magnetization, dM/dP and on the Curie temperature, dT_c/dP, (ii) a large forced volume magnetostriction, $d\omega/dH$, (iii) an anomalous temperature dependence of elastic constants, (iv) a deviation from the Slater- Pauling curve of the dependence of the spontaneous magnetic moment on the concentration[2]. These anomalous properties are, as a whole, called "Invar anomaly", or "Invar effect". Since its discovery, lots of hypothesis has been put forward to explain the "Invar anomaly". Still, its low thermal expansion coefficient and many exotic behaviors have not yet been fully understood. So far it is widely accepted that compensation of thermal expansion with volume magnetostriction leads to low CTE. It has been reported[3] that for most materials the spontaneous volume magnetostriction is small, but Invar´s large spontaneous volume magnetostriction compensates for normal thermal expansion due to the lattice vibrations, resulting in a dimensionally stable material. A number of theoretical models for describing the Invar effect have been proposed, details of which can be found elsewhere[3].

The Invar alloy was originally developed as a replacement of costly platinum-iridium alloy for standard measure of the metric system. Since then, Invar alloys have been widely used as materials for precision instruments and areas where dimensional stability is of prime importance. In many electronic devices like shadow masks of cathode ray tubes for color televisions, resonant cavities of microwave and laser instruments etc. Invar material is used. In recent years, Invar film has attracted the attention of microelectronic industry as a potential candidate for application in sensors, actuators and microelectromechanical systems (MEMS). Development of Radio Frequency (RF) MEMS switches using metal membranes with capacitive coupling has also been reported[4]. Metal membrane switches show reasonable switching voltages, fast switching speeds and excellent linearity. The cross section of a metal membrane capacitive switch is shown in Figure 1. The detailed structure of such a switch can be found elsewhere[6]. The metallic switch membrane consists of a thin metallic film less than 0.5μm thick.

Application of an electrostatic field between the membrane and the lower electrode causes the formation of positive and negative charge on the conductor surfaces. These charges exhibit an attractive force which, when strong enough, causes the suspended metal membrane to snap down onto the lower electrode and dielectric surface, forming a low-impedance RF path to ground. As switching response is highly sensitive to capacitance and film stress i.e. the distance between suspended membrane and grounded membrane, the suspended film should be insensitive to temperature and CTE of film should matches with substrate to avoid thermal stresses. This requirement makes Invar film an ideal candidate for such an application.

For the two important deposition techniques: sputtering and electroplating, Invar exist either as pure alpha or mixture of alpha and gamma phase directly after deposition. To get Invar property one has to do proper heat treatment to get pure gamma phase by alpha to gamma phase transformation. In present study transformation of sputtered Invar film is investigated. First time, annealing of film by RTA is used to transform the film consisting of alpha and gamma phase into pure gamma phase. Structural and compositional analysis of film is performed to determine the effect on CTE. The CTE was investigated using stress measurement against temperature.

2. EXPERIMENTAL PROCEDURE

In present case, Invar films were prepared by magnetron sputtering. Invar target of 3” diameter and 1.5 mm thickness has been obtained from *GOODFELLOW*, whose composition is determined by EDX to be 63.94%Fe, 35.66% Ni and 0.4% of Mn. n-type (1 0 0) Si with 100nm of Si_3N_4 diffusion barrier is used as substrate. The sputtering chamber is pumped down to a vacuum base pressure of $2\text{-}7\cdot10^{-7}$ mbar before deposition. Then argon is fed into the chamber up to a pressure between 5-10 μbar. The films were produced in constant power mode. Generally, before depositing on actual sample, pre-sputtering is done for 1-3 minutes. This ensures removal of any impurity present on target surface and stability of power which usually fluctuates during first few minutes of sputtering. The sample holder swayed across the target with certain frequency. In present case, the sample holder disc was given oscillation of ¼ rotation. This gives films with uniform deposition rate. Every time sample holder sways across the plasma, some thickness of film gets deposited over the substrate. To see the effect of pressure and power on deposition rate, a series of samples were sputtered for short duration. Then thicknesses of such films were measured using X-ray reflectometry. After appropriate calibration, thickness per oscillation for Invar is calculated. Then number of oscillation is set to get desired thickness of film. X-ray diffraction measurements were performed to analysed the phase present in deposited films using Cu $K_{□}$ radiation in a Philips X`pert. Composition was analysed using a *Leo 1550* SEM equipped with EDX. Element standards of Ni, Fe and Si have been used and the spectrum obtained is then analysed by Stratagem software which enables precise analysis of thin film composition by varying the primary electron beam energy.

The film is annealed using AST SHS10 system with N_2 flowing into quartz chamber during thermal processing to avoid the oxidation of Invar film. Samples are heated to 600°C with maximum possible ramp and held for 5, 30 and 60s. After that, annealing chamber is set to cool down to room temperature within 250s.
Stress in the film can be measured by Laser-Scanning method which measures substrate curvature. The stress in the thin film is then calculated from the radius of curvature of the substrate using the Stoney formula[7]

$$\sigma = \frac{Eh^2}{(1-\nu)6RT} \qquad (1)$$

Where E /1-ν is the biaxial elastic modulus of the substrate *h* is the substrate thickness, *T* is the film thickness, *R* is the substrate radius of curvature, σ is the average film stress. To measure the CTE, change in stress with temperature is measured, which is given by the following equation

$$\frac{d\sigma}{dT} = \frac{E_{\text{Film}}}{1-\nu_{\text{Film}}}\left(\alpha_{\text{Substrate}} - \alpha_{\text{Film}}\right) \qquad (2)$$

where α_{Film} and $\alpha_{substrate}$ are CTE of film and substrate respectively. Thus, in curves representing stress vs temperature the slope gives the dσ/d*T*, E_{film} has been measured, ν_{Film} and $\alpha_{substrate}$ are standard value so that, α_{Film} can be calculated. Stress is measured using *TENCOR FLX-2908*. Prior to actual stress measurement of film, curvature of the substrate is measured before deposition to subtract initial bending present in substrate. Samples were subjected, in nitrogen atmosphere, to temperature cycle between room temperature and 200°C at heating rate of 0.8°C/min and cooling rate of 0.5°C/min.

3. RESULTS AND DISCUSSION

The sputtered film is analysed for its composition and structure. Composition of 63.56wt% Fe and 36.44wt% Ni is found which is almost same composition as of the target. Figure 2 shows the XRD pattern of as prepared sputtered film showing the mixture of alpha and gamma phase. The reported temperature at which complete transformation of alpha phase occurs varies widely in literature for conventional annealing techniques. Transformation of Invar thin film using RTA is performed. Since no literature has been found describing RTA of Invar film, several experiments at different time and temperature have been performed. XRD pattern of RTA treated film annealed at 600°C for different time is shown in Figure 3. From XRD, it can be inferred that there is complete transformation of alpha phase to gamma phase even at 5s of RTA treatment. Though transformation is completed after 30s and 60s of RTA, there is a formation of some oxide which is evident from onset of oxide peak. The sharpness of peak i.e. small FWHM indicates that there is some grain growth. By using Scherrer formula for gamma peak, grain size of as prepared and RTA treated samples were found to be 10nm and 150nm respectively.Generally grain growth is accompanied by structural rearrangement and relationship between texture evolution and grain growth can be found elsewhere[8].

Result of stress measurements of as prepared sample is shown in Figure 4. Such a thermal cycle consist of a heating and cooling cycle. The room temperature stress after deposition is as high as 850MPa and of tensile character. During heating, some irreversible reactions such as dislocation motion and annealing out of vacancies take place leading to plastic deformation reactions. During cooling cycle, the stress increases monotonically till room temperature. From the curve it can be inferred that the irreversible reaction completes around 160°C as after 160°C, heating and cooling cycle superimpose on each other inferring elastic deformation. The slope of the curve can be found by linear fitting of the cooling curve and CTE can be calculated from the equation 2. A high negative slope is exhibited which means that CTE of the film is higher than substrate material. Elastic modulus measurement by nanoindentation has been used to measure elastic modulus of sputtered film. It is evident from Figure 5 that there is very little scatter among the data points and average value has been calculated to 95+/-11.5 for sputtered sample. From this value, the biaxial modulus is found to be 140Gpa. CTE of 2.8×10^{-6} /K for silicon[9] substrate is used. As a result very high CTE of 13.5 ppm/K is obtained in this case which is much higher than bulk Invar. This is due the fact that the deposited film has mixture of alpha and gamma phase. Stress cycle of film after RTA is shown in Figure 6, which is quite different from the curve shown in Figure 5. The positive slope implies a CTE of film which is smaller than that of the substrate. The calculated CTE values after RTA range from 1.63 to 1.69ppm/K for cooling and heating cycle respectively which is quite close to CTE of bulk alloy[10] with same chemical composition. It is evident from the figure that after RTA, the stress-temperature curves coincide for heating and cooling, hence they are fully reversible and there is no plastic deformation. The magnitude of the tensile stress at room temperature for RTA treated sample is lower than as deposited film due to some stress relaxation mechanism. However it is still quite high which is not desirable for certain applications. Stress also affects the CTE which is known to decrease with increasing stress for Invar in elastic limit[10]. Stress(σ) dependency of CTE(α) is given by[11]

$$d\alpha_{total} / d\sigma = -1/E^2 (dE/dT) \quad (3)$$

Since elastic modulus of Invar alloys increases with temperature (upto Curie temperature) owing to its "Invar anomaly", $d\alpha_{total} / d\sigma$ would be negative in the range of elastic limit and Curie temperature. Though dE/dT is well established for bulk Invar[11], similar study for nanocrystalline Invar thin films are not reported so far. So quantitative analysis of influence of stress on CTE of sputtered film is beyond the scope of this paper.

4. CONCLUSION

The objective of this work is to investigate the feasibility of RTA treatment of sputtered Fe-Ni films for high throughput in industry. It is demonstrated that Invar property can be seen in film treated in RTA for time duration as low as 5 second at 600°C. Typical stress-temperature curves obtained for Invar material on Si substrates show line with positive slope. The measured CTE value of about 1.63ppm/K is similar to those reported for bulk Invar alloys.

5. REFERENCES

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FIGURES

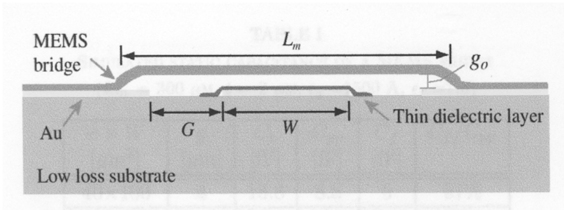

Figure 1 Cross section of a shunt MEMS capacitive switch[5]

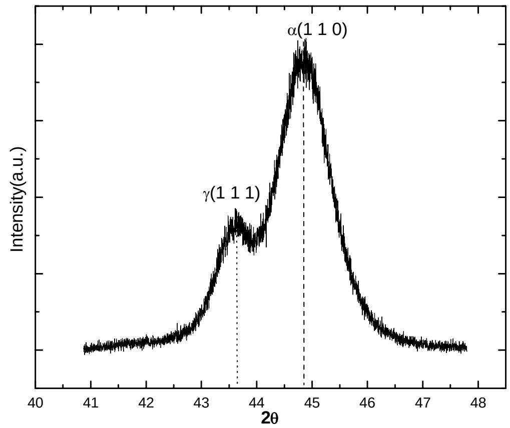

Figure 2 XRD pattern of as prepared film.

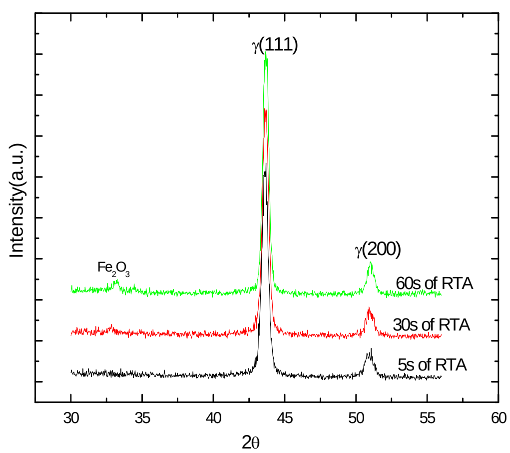

Figure 3 XRD pattern of film after RTA treatment for different time.

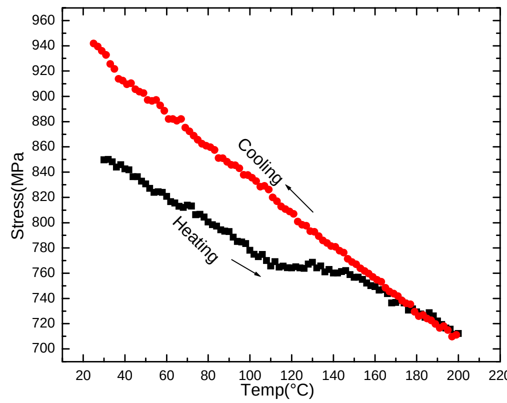

Figure 4 Stress cycle for as prepared sputtered film

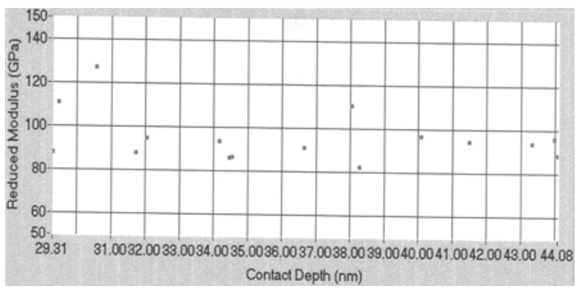

Figure 5 Results of nanoindentataion for Elastic modulus of sputtered film.

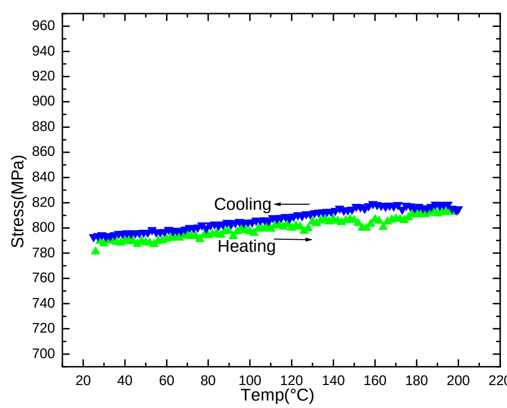

Figure 6 Stress cycle of film after 5s of RTA