9. MECHANICAL PROPERTIES

9.1 INTRODUCTION

Interest in the mechanical properties of thin films has grown rapidly over the past three decades hand in hand with general interest in all other properties. The mechanical properties of thin films have been studied from several points of view. The large internal stresses, first found in electroplated films, have been an annoying feature often leading to film fracture and peeling. Early observations of films exhibiting an unexpectedly high structural strength provided another motivation for the study of the mechanical properties of films. The correction of structure and mechanical properties is leading to a better understanding of condensed films, but a quantitative theory of film mechanics seems to evolve only slowly.

The study of the stress in thin films has attracted much attention since the early work of Stoney on electrodeposited films [1]. Most of the recent works have been carried out on evaporated films, due to high internal stresses often found in practical applications of the films. Haffman has summarized the stress behaviour of many materials [2] and it has been extended by Ennos [3]. Intrinsic stress of some evaporated thin film materials have been measured by Koch et al [4] and Abermann et al. [5]. The use of semiconductors in electronic instruments as stress and strain transducers require that a detailed calculation
of the stress and strain in these materials are made [6]. Stress in the semiconducting films may be deduced from changes in the bandgap [7], electrical resistance [8], and superconducting transition temperature [9]. Imai and Uchida [10] and Matukura [11] individually investigated the uniaxial stress effect on germanium p-n junctions and Wortman et al., have theoretically calculated the stress in germanium [12]. Stress measurements of GaAs and Si epitaxial films have also been reported by Chaorong Li et al.[13]. Also from recent reports [14-15] it is clear that stress may substantially affect the electronic and structural properties of thin films.

Although the literature concerning the structural, electrical and optical properties of Ge, Se and Ge-Se films are so extensive, only a few papers on their mechanical properties can be seen. In this chapter stress measurements of vacuum evaporated Ge, Se and Ge\textsubscript{x}Se\textsubscript{1-x} thin films are presented.

9.2 THEORY

Stress in thin film is divided into two parts (i) tensile stress and (ii) compressive stress. The positive value of stress is called tensile stress and the negative value is called compressive stress. If a film is deposited on a thin substrate, the substrate will be bent by a measurable degree. A tensile stress will cause the film surface, to bend it as is concave, and a compressive stress so that is convex. The most common methods
for measuring the stress in a thin film are based on this bending principle. In general, the total stress $S$ is equal to the sum of the external, thermal and intrinsic stresses.

$$S_{\text{total}} = S_{\text{external}} + S_{\text{thermal}} + S_{\text{intrinsic}} \quad \text{(9.1)}$$

The combination of thermal and intrinsic stress is called internal stress. In technological applications, the total stress must be kept small. The intrinsic stress is the predominant component in many thin film systems and has been the subject of most of the investigations.

The intrinsic stress in the film can be related to the end deflection of the composite cantilever, or the edge or center deflection of the circular plate substrate, by applying the standard theory of elasticity. This relation is obtained by assuming the following factors:

(i) The film strains the substrate which bends until equilibrium is reached.

(ii) The film-substrate bond is strong enough to suppress slippage.

(iii) The substrate is linearly elastic, homogeneous and uniformly thick.

(iv) The bending displacement is small compared with the thickness of the substrate.

(v) The width of the substrate is less than half the length.

(vi) The stress is uniform throughout the film thickness.
(vii) No stress relief or changes in elastic constants take place as the film is built up.

Some of the assumptions are hard to justify. For instance, stress in a film is not expected to be uniform because of the presence of a free surface. The frequent curling away of the films on peeling is, of course, the result of a nonuniform stress. Stress relief is known to occur in many systems. The measured displacements often are not much smaller than the substrate thickness (≈ 50µm). Timoshenko [16] solved the problem for large deflections when a bending moment is applied only to two opposite edges of the plate, a condition which is similar to the film-substrate case. With the assumptions outlined above, by the theory of bending plate [17], the expression for the mean intrinsic stress \( S \) (stress per unit cross-section of the film) can be written in the form originally derived by Stoney [3] as,

\[
S = \frac{E D^2 \delta}{6 l^2 M d} \text{ N/m}^2
\]

where

\( d = \) film thickness
\( E = \) Young's modulus of the substrate, \( (\text{mica in the present study}) \)
\( D = \) substrate thickness
\( \delta = \) final tension
\( M = \) distance between scale to mirror
\( l = \) length of the film
In the above expression, the factor $1/1-v$ given in the original equation, is neglected due to the cantilever geometry of the present study [18].

The Young's modulus of the mica substrates ($E$) has been evaluated by making use of the formula,

$$E = \frac{12 m g l_1^2 D_1}{\delta b d_1^3} \text{ N/m}^2$$

where

- $m$ = load applied at the free end of the mica sheet.
- $g$ = acceleration due to gravity
- $b$ = breadth of the mica substrate
- $l_1$ = length of the mica substrate
- $D_1$ = distance between the scale and the mirror
- $\delta$ = shift in the scale reading
- $d_1$ = thickness of the mica sheet

9.3 EXPERIMENTAL

To measure the mechanical stress, an aluminium plate is bent, and cut accordingly (Fig 9.1) and has provisions for fixing the mica strip and a glass substrate for stress and thickness measurements respectively. This set up is used to fix the arrangements inside the coating unit. Using a 0.3m conventional vacuum coating unit (12A4, Hind Hivac, India) germanium or Selenium or $\text{Ge}_x\text{Se}_{1-x}$ is evaporated from a tungsten conical basket and deposited onto the well-cleaned glass substrate and mica.
Fig. 9.1 Schematic diagram of the front view of the stress measuring aluminium plate setup.
strip. The rate of evaporation is properly controlled and has been maintained throughout the evaporation. All evaporation processes have been carried out under a vacuum of \(2.66 \times 10^{-3}\) Pa. Using multiple beam interferometer the thicknesses of the films have been measured.

In order to observe the deflection with the aid of a scale and telescope from the free end of a mica strip cantilever, a mirror has been fixed at the free end of the mica strip. As the weight of the mica strip is small, the weight of the mirror has to be comparable with that of the mica strip. Hence a suitable mirror is fabricated using microscopic cover glass slides (Bluestar) and fixed at the free end of the mica strip.

Due to the practical difficulties in studying the mechanical properties of hot wall deposited films, only vacuum evaporated semiconducting films have been used in the present study.

9.4 RESULTS AND DISCUSSION

Knowing the distance from the mirror to the telescope, deflection, length, breadth and thickness of the mica strip, the Young's modulus of the mica strip (substrate) has been evaluated as \(4.88 \times 10^{10}\) N/m\(^2\) using the formula 9.2. The stress values in the films are calculated using the relation 9.2.

The variation of stress \(\sigma\) with the thickness of vacuum evaporated Ge, Se, and \(\text{Ge}_x\text{Se}_{1-x}\) films are given in figs. 9.2, 9.3.
Fig. 9.2 Variation of stress with thickness of vacuum evaporated Ge films.
Fig. 9.3 Variation of stress with thickness of selenium films.
Fig. 9.4 Dependence on stress on vacuum evaporated $\text{Ge}_x\text{Se}_{1-x}$ films.
and 9.4. In all the figures three distinct regions are observed in the lower, middle and higher thickness ranges. In germanium films, the stress increases steeply with thickness, attains maximum at 60 nm and decreases thereafter before attaining constant values. The maximum stress observed is $5.66 \times 10^9$ N/m$^2$.

In Selenium films a gradual rise in stress with thickness is observed in the first region. After an almost flat second region, stress decreases gradually with thickness. No thickness independent region is observed in the higher thickness range. The maximum stress is $1.2 \times 10^9$ N/m$^2$.

The stress of Ge$_x$Se$_{1-x}$ film shows a gradual rise in the lower thickness region and attains maximum ($6.95 \times 10^9$ N/m$^2$) at the thickness of 76 nm, after which a gradual decrease is observed.

Similar behaviour of stress with the thickness of the films has been observed by many workers on different materials, Viz., silver and gold [19], copper [20], antimony [21], LiF [22] and SnO [23].

In the present study the stress in the semiconducting films deposited on mica substrate is tensile in nature. The stress in the low thickness films as indicated by the first region is mainly intrinsic. No intrinsic stress is added to the films after they reach a specific thickness (60 nm for Ge, 144nm for Se and 76 nm for Ge$_x$Se$_{1-x}$ films).
It can be said that the last holes in the island stage of the films are filling up when the stress reaches a maximum values at thicknesses mentioned above.

The behaviour of the stress of the films may be explained by the model proposed by Willock et al.[19]. This model is based on the evidence that the island in island structure film consists of many microcrystallites. As the film grows, the crystallites anneal out, leading to a net volume of the islands. This results in the occurrence of tensile stress.

Since not much information is available in the literature regarding the mechanical properties of Ge, Se and Ge\textsubscript{x}Se\textsubscript{1-x} films, the results obtained in the present study could not be directly compared. However the order of stress values determined is in agreement with that of Silicon[24], hydrogenated Silicon [25] evaporated tellurium films [26] and GaAs films [27].
REFERENCES