

Mechanical Properties of Thin Electrical Films

Silviya Letskovska¹ and Pavlik Rahnev²

Abstract – In this work the base mechanical properties of thin films are described. The attentions is paid of the type of stress state conditions in the system film/substrate, the possible fracture mechanisms. The date for mechanical properties of some commonly used thin films are given.

Key words – mechanical properties, thin film, testing methods.

I. INTRODUCTION

The digital revolution brought about by the development of integrated circuits over the last half century is one of the most astonishing achievements in human history. Most materials used in advanced microelectronic devices are in thin film form. In certain applications, thin films are used as components that carry mechanical loads.

In these applications, the mechanical behaviour behave of the thin-film material is the primary consideration in materials selection. In many other applications, thin films are selected primarily because of their unique electronic, magnetic, optical, or thermal properties. The mechanical characteristics of the materials of choice in such applications are, however, not unimportant because thin films are often subjected to large mechanical stresses during both the manufacturing process and normal operation of the devices.

II. MECHANICAL BEHAVIOR OF THIN FILMS

Generally, mechanical stresses in thin films can be divided into either the so-called intrinsic or growth stresses that develop during the deposition process, or the extrinsic stresses that are induced by external physical effects [1-4].

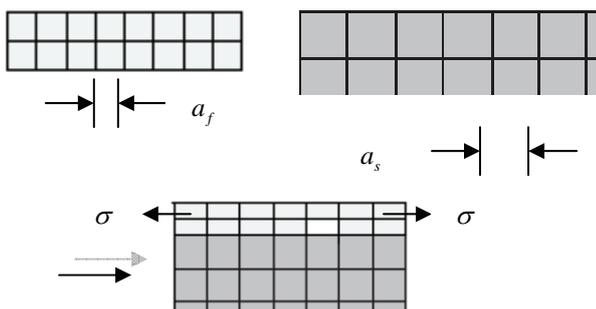


Fig. 1. Film on the substrate.

Thin-film materials are fabricated using very different processing methods than those for bulk materials, such as epitaxial growth and various vapour deposition techniques.

These processing methods often result in development of mechanical stresses in the films. For example - epitaxial stresses arise when films have perfectly coherent interfaces with their substrates (Fig. 1).

There have been many mechanisms proposed for the development of growth stresses, which depend sensitively on the materials systems, deposition technique, and process parameters [5]. These stresses arise because generally films are deposited under *non-equilibrium conditions*. In general, any redistribution of matter will result in film stresses, since the film is constrained by the substrate. Typical behaviour of the average film stress as a function of film thickness is shown in the following Fig. 2.

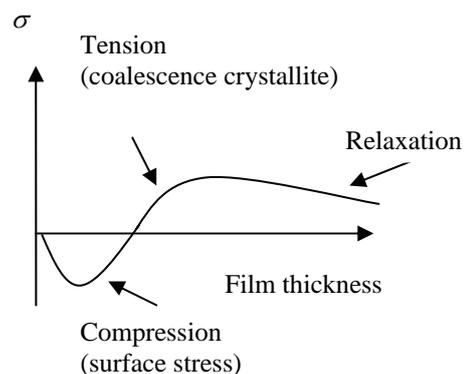


Fig. 2. Film stress as a function of the thickness.

The extrinsic stresses are induced by various physical effects after the film is grown. One of the most commonly encountered examples is the thermal mismatch stress due to the thermal expansion mismatch between a film and its substrate [1]. For an infinitely infinity substrate:

$$\sigma_T = M \Delta T \Delta \bar{\alpha} \quad (1)$$

where: $\Delta \bar{\alpha}$ average, differential thermal expansion; ΔT temperature difference; $M = E / (1 - \nu)$ - the biaxial modulus, for isotropic materials, E - Young's modulus, ν - Poisson's ratio.

The manufacturing process and normal operation of most thin-film based devices often involve large temperature cycles. Therefore, thermal mismatch stresses are inevitable and they can be very high if the thermal mismatch is large and the elastic modulus of the film is large, such as is the case for Cu films used in integrated circuits. Large mechanical stresses are unfavourable in most applications. For example, high

¹Silviya Letskovsta is with of Burgas Free University, San Stefano 62, 8000 Burgas, Bulgaria, silvia@bfu.bg

²Pavlik Rahnev is with of Burgas Free University, San Stefano 62, 8000 Burgas, Bulgaria, rahnev@bfu.bg

levels of residual stress in shape memory alloy thin films may prevent a phase transformation from occurring and may lead to loss of the shape memory effect. For piezoelectric thin films, excessive deformations due to large stresses may also cause the material to behave differently from its ideal function. Even though the presence of large mechanical stresses may sometimes not significantly affect the functional properties of thin films, they may still cause a material to fail by promoting the formation of voids or cracks in the films or by delaminating the films from attached layers.

It is clear that the mechanical properties of thin films are very important. It has long been recognized that the mechanical properties of thin films can be very different from those of their bulk counterparts [1, 4, 6]. This phenomenon is generally referred to as a size effect. Some properties can be simply explained through extrapolation of bulk behaviour, but other properties depend sensitively on mechanisms that remain illusive [6]. The strength of thin films is a prime example - a substantial amount of experimental evidence has shown that thin metal films can support much higher stresses than the same material in bulk form. This strengthening has generally been attributed to dimensional and microstructural constraints on dislocation activity in thin films [7]. Dimensional constraints are imposed by the interfaces and the small dimensions typically encountered in thin films, while microstructural constraints arise from the very fine grains often found in thin films. In bulk materials, microstructural constraints dominate the plastic behaviour of the material. However, when material dimensions are comparable to microstructural length scales - as is typically the case for thin films - free surfaces and interfaces play an important role as well. For example, dislocations can exit the material through free surfaces, while strong interfaces can prevent them from doing so. Consequently, strong interfaces lead to a higher cumulative dislocation density in the film and result in a higher flow stress and a greater strain-hardening rate. This behaviour cannot be captured by classical plasticity theories and motivates a strong interest in developing new models to describe it. In addition to dimensional constraints, the microstructure of thin films also affects their mechanical properties. In summary, understanding the deformation mechanisms for thin films is not only important to take full advantage of the materials and improve device reliability, but also important expand to our knowledge of the processing-structure-property relationship, one of the basic tasks in materials science.

The traditional mechanical testing methods used for bulk materials cannot be applied directly to the study of thin films because of the thickness of these materials.

The techniques to characterize the mechanical behaviour of thin films can generally be divided into two main categories:

- Direct testing of thin films deposited on substrates, which involves minimum sample preparation. The film properties are, however, implicitly embedded in the experiment data, and significant post-processing effort is usually required in order to extract the intrinsic film properties;

- Mechanical characterization of freestanding films, which requires careful specimen processing and handling.

These techniques can yield explicit and accurate elastic-plastic properties of the films.

III. TECHNIQUES FOR STUDY

Several of the most widely used techniques in both categories.

3.1. Techniques for films on substrate: the substrate curvature and nanoindentation.

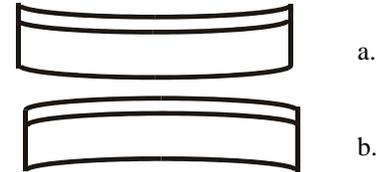


Fig. 3, a, b. Deformation of substrate and film.

Intrinsic stress results from the microstructure created in the film as atoms are deposited on the substrate. Tensile stress results from microvoids in the thin film, because of the attractive interaction of atoms across the voids. Tensile stress - film wants to be "smaller" than the substrate because it was "stretched" to fit. (Fig. 3a). Compressive stress, the film wants to be "larger" than the substrate, because it was "compressed" to fit (Fig. 3b).

In the substrate curvature measurement, strains are imposed by varying the temperature of the film/substrate system if the coefficients of thermal expansion (CTE) of the film and the substrate are different. The stress in the film causes the film/substrate system to bend, the curvature of which can be measured using optical methods. Since the curvature of the substrate may not be zero, it is necessary to measure the substrate curvature prior to film deposition. The change of the substrate curvature, Δk , can be related to the film stress, σ_f , through the Stoney equation [1]:

$$\Delta k = \frac{6\sigma_f h_f}{M_s h_s^2} \quad (2)$$

where: $M_s = E_s / (1 - \nu_s)$ is the biaxial modulus of the substrate, h_f , h_s - the film and substrate thicknesses, respectively. Given M_s , h_f and h_s the film stress can be readily determined as a function of temperature by measuring the curvature of the film/substrate system as a function of temperature. It should be noted that the imposed strain is equibiaxial if both the film and the substrate are thermally isotropic ($\sigma_{xx} = \sigma_{yy}$, $\sigma_{zz} = 0$). The technique is often used to study the thermal mechanical behaviour of metal films on ceramic substrates.

Proper interpretation of the experimental results is not easy because the mechanical response is complicated by the temperature change. Moreover, the strain level that can be imposed is limited by the difference of CTEs between the film and the substrate and the maximum temperature change. The

microstructure of the films may also change during the thermal cycles.

Nanoindentation [8] is a technique that can quickly probe the mechanical properties of various thin films deposited on substrates (fig. 4).

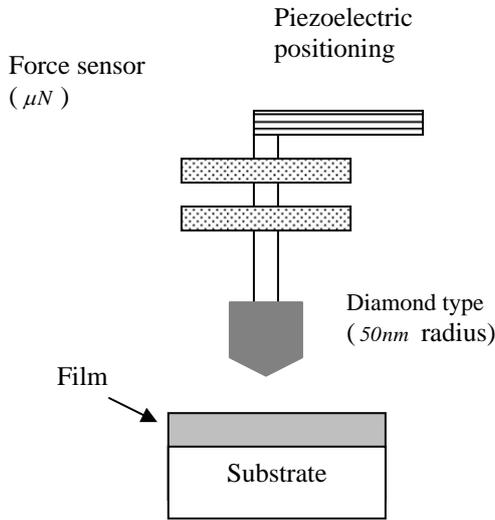


Fig. 4. Nanoindentation test method.

The hardness H is defined as the ratio between indentation load P and projected contact area A ($H = P/A$). The indentation modulus M can be extracted using the following equations:

$$S = (\gamma 2M \sqrt{A}) / \sqrt{\pi} \quad (3)$$

The contact stiffness S is obtained from the slope of the initial portion of the elastic unloading curve; γ is a correction factor for a specific indenter tip shape, e.g., 1.08γ for a three-sided pyramidal indenter. The hardness, H , is proportional to the material yield stress. Nanoindentation on thin films has uncertainties due to well-known experimental limitations that make it difficult to interpret the experimental data accurately. Most notable among these are the effects due to presence of the substrate, densification of the film as a result of large hydrostatic stresses, issues with tip calibration, surface roughness, and size effects as a result of the non-homogenous strain field. Considerable effort has been devoted to understanding these issues and to relating nanoindentation results to intrinsic material properties [9]. The information that can be acquired from the nanoindentation is also limited. For example, nanoindentation is not suitable for measuring the work-hardening behaviour or the residual stress in the film.

In addition to the substrate curvature technique and nanoindentation, a number of dynamic techniques are available for determining the elastic properties of thin films on substrates. These techniques include surface acoustic wave spectroscopy (SAWS) and surface Brillouin scattering (SBS). These techniques typically require knowledge of the density of the film and only provide information on the elastic behaviour of the films.

3.2. Techniques for freestanding films: the microtensile test [10] and the bulge test [11].

These techniques require some sample preparation, but they can be readily applied to measure intrinsic film properties without any substrate effects, and to obtain thin film constitutive behaviour with relatively large applied strains.

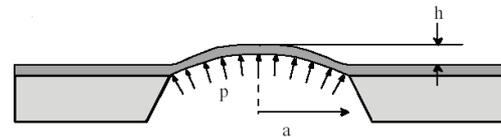


Fig. 5. Bulge test schematic.

The microtensile test is the analogy of its bulk counterpart. Due to difficulties associated with sample handling at the micron or submicron scale, microtensile testing often suffers from alignment and gripping problem, which often leads to inaccuracy in the strain measurement. Spaepen and colleagues [12] have developed a diffraction-based technique for measuring the local strains of a freestanding film by patterning a square array of photoresist islands on the film surface. There are still some uncertainties in the strain measurement due to transverse wrinkling of the freestanding film. Since the film needs to be removed from the substrate before mounting it on the testing stage, residual stresses in the film cannot be measured. The sample handling also limits the thickness of the film that can be tested. Alternatively, a thinner film can be tested by depositing the film on a compliant polymeric substrate and by stretching the film/substrate composite structure. The local stresses in individual grains in the film can also be measured by means of x-ray diffraction.

There is a variation of the microtensile test, the so-called membrane deflection experiment (MDE) that was developed by Espinosa and colleagues [13]. In this technique, a dogbane-shaped freestanding film stripe is micro fabricated with an enlarged contact area at its centre. A nanoindenter tip is used to apply line loading on this contact area and the load is continuously recorded. A nanoindenter tip is used to apply line loading on this contact area and the load is continuously recorded. The gauge section of the freestanding film undergoes a pure stretch and the displacement is measured by means of a full-field interferometric method. This technique involves less specimen handling and offers accurate strain measurement.

The bulge test (Fig. 5) is another powerful technique for measuring the mechanical behaviour of freestanding thin films. In this technique, freestanding thin films are obtained by opening a window in the substrate using micromachining techniques. The film is deflected by applying a uniform pressure to one side of the freestanding membrane. The mechanical properties of the film are determined from its pressure – deflection behaviour. Compared with microtensile testing, the bulge test technique has the unique advantage of precise sample fabrication and minimal sample handling. There are virtually no issues related to specimen alignment and film wrinkling due to Poisson’s effect since the film is supported by Si substrate at all edges. Moreover, the residual stress in the film can be measured. With some care,

freestanding films as thin as 50 nm can be prepared and tested. The results, derive after testing of mechanical properties some films are given in Table 1.

Table 1.

System	Yield σ_{ys} (MPa)	Modulus E (GPa)	Thick h (nm)	T ($^{\circ}C$)	G_c (J/m^2)
$Ta_2N / Al / Al_2O_3$	190	70	178	20	7
$Ta / Al - Cu / Al_2O_3$	298	70	500	20	5.6
$Ta_2N / Au / Al_2O_3$	517	80.8	200	20	1.4
Nb / Al_2O_3	2000	103	105	-	0.95
Ta_2N / Al_2O_3	-	-	100-6000	20	5.5-9.0
$Si_xN_y / SiO_2 / Si$	-	171	1000	20	1.5

Note: G_c - Adhesive Fracture energy.

CONCLUSION

Thin films are an essential component of all advanced electronic devices. Understanding of failure modes in these devices, especially interface delamination, requires a knowledge of the mechanical behavior of the films. Techniques for measuring the mechanical behavior of thin films are being developed and applied. Because the films are formed by condensation from the vapor phase, their microstructures, and hence their mechanical properties, are quite different from those of bulk materials of the same chemical composition. While the general principles of conventional mechanical testing are applicable to thin films, conventional test equipment and techniques are not. It is clear that the functional properties of thin films (single and multi layers) could be determinate using modern methods and devices to analyze the properties of the surface.

These methods allow to receive data for the Young's modulus, hardness, when very small (nanodimensional) volumes of material is used for investigation. The determination of these properties is of primary importance for creating new material structures and also for prediction of possible fracture mechanism.

REFERENCES

- [1] W. D. Nix, Mechanical properties of thin films, Metall. Trans, A 20, 2217 (1989).
- [2] J. Proost, F. Spaepen, Evolution of the growth stress, stiffness, and microstructure of alumina thin films during vapour deposition, J. Appl. Phys. 91, 204. (2002).
- [3] F. Spaepen, Interfaces and stresses in thin films, Acta Mater. 48, 31 (2000).
- [4] R. P. Vinci, J. J. Vlassak, Mechanical behaviour of thin films, Annu. Rev. Mater.Sci. 26, 431 (1996).
- [5] W. D. Nix, B. M. Clemens, Crystallite coalescence: A mechanism for intrinsic tensile stresses in thin films, J. Mater. Res. 14, 3467 (1999).
- [6] S. M. Spearing, Materials issues in micro electro mechanical systems (MEMS), Acta Mater., 48, 179 (2000).
- [7] E. Arzt, Size effects in materials due to microstructural and dimensional constraints, A comparative review, Acta Mater. 46,, 5611 (1998).
- [8] W. C. Oliver, G. M. Pharr, Measurement of hardness and elastic modulus by instrumented indentation, Advances in understanding and refinements to methodology, J. Mater. Res. 19, 3 (2004).
- [9] X. Chen, Y. Xiang, J. J. Vlassak,: A Novel Technique of Measuring the Mechanical Properties of Porous Materials by Nanoindentation with Application to Low-k Dielectric Thin Films, submitted (2005).
- [10] D. T. Read, Y.W. Cheng, R. R. Keller, J. D. Mc Colskey, Tensile properties of free - standing aluminium thin films, Scripta Mater. 45,, 583 (2001).
- [11] J. J. Vlassak, W. D. Nix, A new bulge test technique for the determination of Young's modulus and Poisson's ratio of thin films, J. Mater. Res, 7, 3242 (1992).
- [12] H. B. Huang, F. Spaepen, Tensile testing of free-standing Cu, Ag and Al thin films and Ag/Cu multilayers, Acta Mater. 48,, 3261 (2000).
- [13] H. D. Espinosa, B. C. Prorok, B. Peng, Plasticity size effects in free-standing submicron polycrystalline FCC films subjected to pure tension, J. Mech. Phys. Solids 52, 667 (2004).