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Validation of mechanical damage monitoring on aluminium freestanding thin films using electrical measurements

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Abstract. This paper describes a new technique allowing the monitoring of damage in metallic freestanding thin films during micro-tensile test by using electrical characterization. After a presentation of the set-up, results obtained on Aluminium thin coatings by using two calculation methods for damage variable are presented and commented.

Introduction

Systems based on Micro- and Nano- Technologies involve thin coatings. The mechanical function is often ensured by the use of moveable part, such as freestanding membranes or cantilever. As a consequence, the mechanical properties of such freestanding part must be known in order to properly design the component or to validate its reliability. The evaluation of elastic properties (elastic modulus, yield stress) may be sufficient when only small and reversible deformations are expected (e.g. accelerometer). The application of a large number of actuations may nevertheless lead to a mechanical damage.

Moreover, large deformations are now considered, when developing components based on flexible electronic for example. The large deformations to be sustained by thin coatings may also lead to some damage in the metallic thin coating. Such damages can lead to an alteration of the properties, both mechanical and electrical.

This paper describes a new technique allowing the monitoring of damage in metallic freestanding thin films during micro-tensile test by using electrical characterization. The mechanical stress is applied with a specific apparatus which was described in the RSI journal [1]. The relevance of using micro-tensile test for the mechanical characterization of freestanding thin layers was demonstrated on 1µm thick aluminium specimen in SPIE [2]. Thus, the whole elastic-plastic behaviour of thin layers can be characterized and provides meaningful data for improving design and reliability of MEMS with freestanding parts. The current paper is focused on a new way to monitor damage, by coupling electrical measurements with the mechanical stress applied on metallic aluminium thin layers.

Relevant dataset of damage for thin films are required to properly design and improve the reliability of new devices. Previous studies have been carried out on bulk and composite materials [3, 4]. Coupling damage monitoring to uniaxial tensile test is well suited to extract the relevant elastic and plastic parameters as well as to establish damage law for thin films.

Description of the testing methodology

The micro-tensile apparatus. The micro-tensile bench was designed by the SIMaP laboratory in order to characterize the stress-strain relationship of thin coatings and to correlate these properties with a microstructural analysis via *in-situ* observations (MEB, AFM...)[1, 5]. Two load cells are available (1.5 N and 20 N) to test different kinds of materials. Several author [6, 7] have pointed out strain measurement as a key issue in the reliability of micro-tensile experiments. This particular point has been here overcome with the use of interferometry technique.

A specific study based on finite element modelling to design optimized specimen geometry lead to the use of double curvature shoulders between the gauge part and the clamping region [1] in order to avoid stress concentration. The length of the gauge has then to be corrected for the calculation of strain in order to take into account the widening near the clamps [8].

The specimens are constituted of thin sputtered aluminium film deposited on a silicon substrate with a freestanding gauge part. Specimens are framed with Silicon in order to allow handling and to calibrate the tensile stage prior to the test.

Fig. 1 shows the typical flow-test: a specimen is fastened into grips and any awkward slipping is prevented. After removing the frame (silicone cutting for example), the grips are then moved away from each other and both load and displacement are monitored and used to calculate the stress-strain curve that gives information on the mechanical properties of the material: modulus of elasticity (E), yield stress (σ_y), ultimate elongation (ε_u), ultimate strength (σ_u) and strain-hardening.

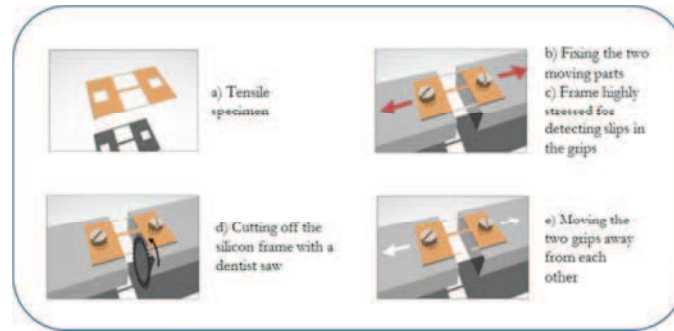


Fig. 1: Flow of an in-plane uniaxial stress test on freestanding coating [1]

The mechanical characterization of damaging. Lemaitre and Chaboche [9] have introduced the variable D that characterizes the damage level of the material. The value of the variable D can be calculated using equation (1):

$$D = \frac{\tilde{S}}{S} \quad (1)$$

where S is the nominal section of the specimen and \tilde{S} the effective section. When the material is perfectly non-damaged, \tilde{S} is equal to S and $D = 0$. For a broken material, $\tilde{S} = 0$ and $D = 1$. As the damage of material results in an invisible reduction of the effective area, an effective stress $\tilde{\sigma}$ is calculated as:

$$\tilde{\sigma} = \sigma \frac{\tilde{S}}{S} \quad (2)$$

Combining (1) and (2) gives:

$$\tilde{\sigma} = \frac{\sigma}{1-D} \quad (3)$$

The principle of strain equivalence is based on the assumption that the behaviour law of virgin materials remains valid when replacing the applied stress σ by the equivalent stress $\tilde{\sigma}$. The Hooke's law for a damaged material is then written:

$$\varepsilon = \frac{\tilde{\sigma}}{E} = \frac{\sigma}{(1-D)E} \quad (4)$$

The equation (4) allows the introduction of \tilde{E} , the modulus of elasticity of the damaged material, as a function of the modulus of elasticity of the virgin material E and the variable damage D as given in:

$$\tilde{E} = E(1 - D) \quad (5)$$

Unloading the specimen during the experiments allows measuring the elastic response of the damaged material and so to characterize the evolution of the material damaging with strain rising as shown on Fig. 2. Due to the small film thickness compared to the specimen length, buckling appears even for low compressive load. A particular attention is paid to stay in tensile state when unloading in order to avoid buckling.

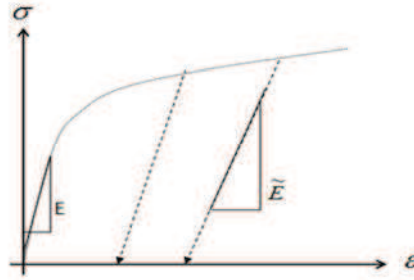


Fig. 2: Schematic view of a uniaxial tensile law, with the Young's modulus and the apparent one when damage is occurring.

The electrical measurement of damaging. The actual current density \tilde{i} , *i.e.* the current flowing through the actual resistive section of the specimen is here introduced by analogy with the actual stress $\tilde{\sigma}$ presented above. According to Lemaitre and Chaboche [9], the actual current density \tilde{i} can be expressed as a function of the nominal current i and the damage variable D as:

$$\tilde{i} = \frac{i}{1-D} \quad (6)$$

The electrical resistance dR of a virgin material depends on the length l and on the section S of the specimen and on the resistivity ρ of the material:

$$dR = \frac{dv}{di} = \rho \frac{dl}{dS} \quad (7)$$

The electrical resistance of a damaged material dR' is:

$$dR' = \frac{dv'}{d\tilde{i}} \quad (8)$$

Combining the equation (6) and (8) give:

$$dR' = (1 - D) \frac{dv'}{di} = \rho' \frac{dl'}{dS'} \frac{1}{1-D} \quad (9)$$

It is assumed that the evolution of the resistivity is only due to geometrical effects induced by the plastic and elastic strains [9]. Thus, in the case of a homogeneous isotropic material, dl' can be written as:

$$dl' = dl(1 + \varepsilon_e + \varepsilon_p) \quad (10)$$

and dS' :

$$dS' = (1 - 2\nu\varepsilon_e - \varepsilon_p) \quad (11)$$

The Bridgman's law says that the resistivity of a structure submitted to a mechanical strain is modified by the geometrical effects weighted by a constant C_1 which depends on the piezoresistivity of the material:

$$\rho' = \rho(1 + C_1\varepsilon_e(1 - 2\nu)) \quad (12)$$

The evolution of the resistance can then be written as:

$$\frac{\Delta dR}{dR} = \frac{dR' - dR}{dR} = \frac{K_e\varepsilon_e + K_p\varepsilon_p + D(1 - 2\nu\varepsilon_e - \varepsilon_p)}{(1 - 2\nu\varepsilon_e - \varepsilon_p)(1 - D)} \quad (13)$$

with:

$$K_e = 1 + 2\nu + C_1(1 - 2\nu)(1 + \varepsilon_e) \quad (14)$$

and:

$$K_p = 2 + C_1\varepsilon_e(1 - 2\nu) \quad (15)$$

Thus, the damage variable D can then be written as a function of the measured electrical resistance as:

$$D = \frac{\frac{\Delta dR}{dR}(1 - 2\nu\varepsilon_e - \varepsilon_p) - K_e\varepsilon_e - K_p\varepsilon_p}{(1 - 2\nu\varepsilon_e - \varepsilon_p)\left(1 + \frac{\Delta dR}{dR}\right)} \quad (16)$$

The electrical measurements of resistance are performed using a four-wires probe (as shown in Fig. 3): a current flow IC is applied and the potential drop VC is independently measured. This method for measuring contact resistance is the same method used in crossed rod contact resistance measurements of Holm [10]: only the contact resistance is measured, independently of the voltage drop in the supply wires and access paths.

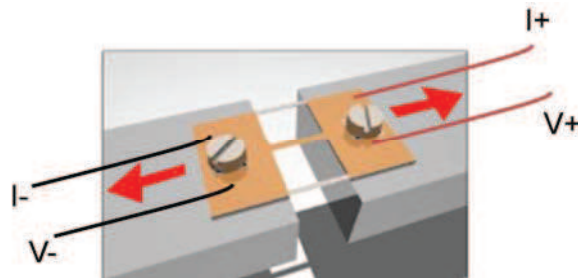


Fig. 3: Example of specimens with position of the four probes for electrical measurements.

Finite element modelling using ANSYS®, were performed in order to ensure that the electrical measurement does not induce strong rising of temperature in the gauge part of the specimen. As it can be seen in Fig. 4 the temperature does not exceed 60°C and then can be neglected for an aluminium specimen.

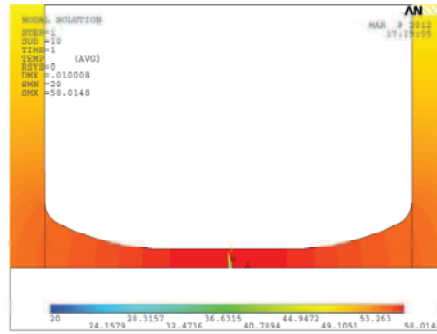


Fig. 4: Temperature in the gauge part of the specimen due to electrical measurement calculated by FEM

Results

Mechanical measurement of damage. Fig. 5 shows a typical stress-strain curve of a multi-cycle loading performed on an aluminium thin film. A total unloading-loading cycle has been performed each 100 μm of elongation until the breakdown of the sample. The damaged modulus of elasticity \tilde{E} has been measured for each unloading and has been linked to the damage variable D using (5). The modulus of elasticity of the first unloading has been taken as a reference. The evolution of D is plotted in the same figure. The damage variable seems to remain close to 0 until the strain reaches 11%. The ultimate variable damage D_u is 0.089 which is quite low for a metallic material and shows that failure is due to a fast final damaging [9]. A damage law cannot be fitted due to the low number of measurements points, despite the already time-consuming experiment.

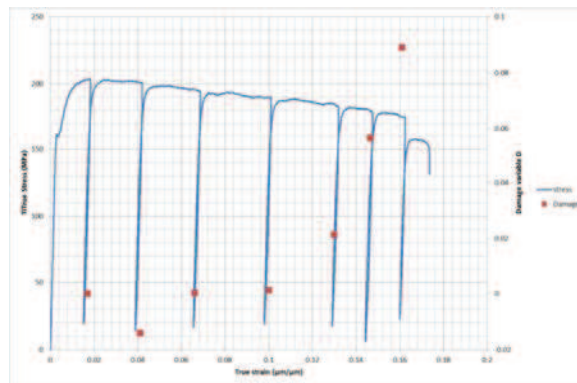


Fig. 5: Stress-strain curve of a multicycle tensile experiment and the evolution of damage as a function of strain on an aluminium specimen.

Electrical measurement of damage using theoretical method. Fig. 6 shows the stress-strain curve and the evolution of the electrical resistance R for a ductile aluminium thin film during a multicycle experiment. It can be observed that the electrical resistance rises continuously along the experiment until necking appears and then rises abruptly.

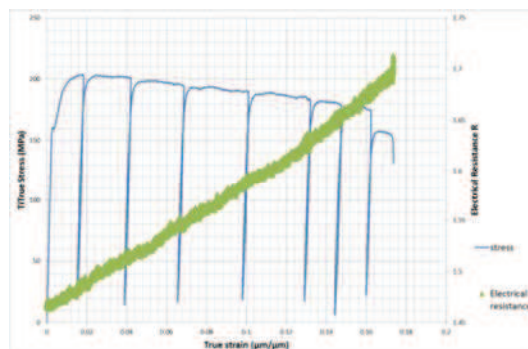


Fig. 6: Stress-strain curve and evolution of resistance as a function of strain for aluminium specimen during a multicycle experiment

The evolution of the damage variable D using equation (16) in order to take into account the evolution of the piezoresistivity and the evolution of the section due to the conservation of the volume in plastic deformation is shown in green on Fig. 7. It appears that the variable damage D decreases continuously during the test towards negative values what is physically impossible. Thus, the correction of the influence of the geometrical modifications are overestimated in equation (16). The explanations of this phenomenon will be discuss later in the paper.

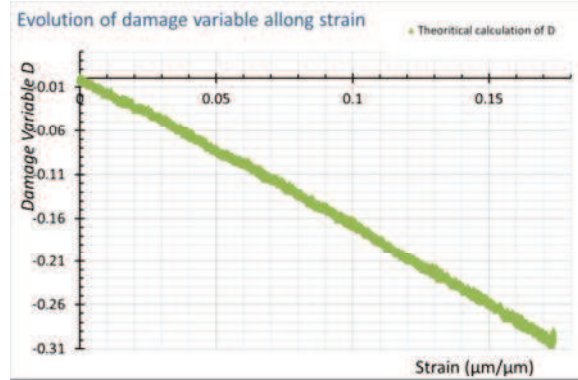


Fig. 7: Evolution of the damage variable D calculated with both theoretical formula and using multicycle experiment.

Electrical measurement of damage using multicycle experiments results. Fig. 6 shows that the electrical resistance R rises continuously during the experiment. However, the mechanical characterization of damage shows that there is no damage until strain reaches 11%. Thus, it can be assumed that the evolution of the electrical resistance R from 0 to 11% of strain, in this experiment, is only linked to the geometrical effect. A linear regression on $D = f(\varepsilon)$ with ε in the range [0% ; 11%] shows that the evolution of R is strongly linear with a slope of 1.227 for a correlation coefficient equal to 0.997. The electrical resistance corrected from the geometrical effects R_{corr} is then expressed as:

$$R_{corr} = R - 1.227\varepsilon \quad (17)$$

The evolution of the corrected electrical resistance R_{corr} is then only linked to the damage of the material and the damage variable D can be calculated by:

$$D = \frac{R_{corr} - R}{R} \quad (18)$$

The evolution of variable damage D using this method is shown in blue on Fig. 8. The value of D remains close to 0 until strain reaches 11.8% and then increases linearly until strain reaches 17.3% which corresponds to the necking of the specimen. The ultimate value of D is 0.017.

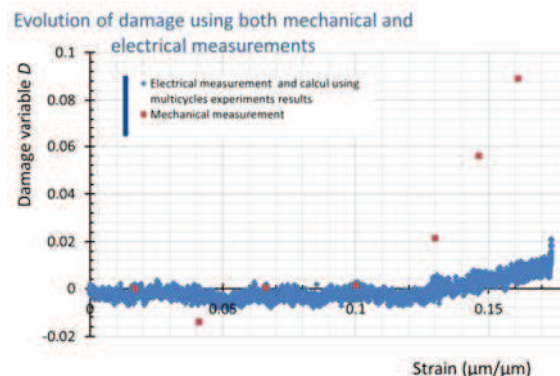


Fig. 8 : Evolution of the damage variable D using both mechanical and electrical measurements.

Discussion

Interest in damage characterization of thin films. This experiment highlights the fact that monotonic experiments are not enough in order to characterize the elasto-plasticity of material: for this material not to have information about the damage leads to estimate that material begins to damage at the maximum stress σ_{max} which is about 1.5% of strain whereas the first apparition of damage occurs at 11%. Thus, the decrease of stress for strain between 1.5% and 11% are linked to the softening of the aluminium. This phenomenon has been observed on other specimens of the same batch during monotonic experiment without electrical measurement. It cannot then be induced by the multicycle or the electrical measurement.

Comparison between mechanical damage and evolution of electrical resistance during multi-cycle test. Fig. 8 shows the evolutions of damage calculated using mechanical and electrical methods. For both the methods, there is no damage measured until the strain reaches 11%. Then the damage variable value increases until the failure of the specimen. Nevertheless, the electrically measured damage variable remains lower than the mechanically one. The phenomenon that leads to the damaging of the thin film could explain this difference. The damaging of ductile metallic materials is led by initiation, growth and coalescence of cavities [11, 12]. Lemaitre and Chaboche [9] highlight that the electrical measure of damage underestimates the value of the damage variable D in such a case.

Discussion on the theoretical calculation of damage with electrical measurement. The theoretical method used to calculate the evolution of damage from the electrical measurements leads to negative values of the damage variable D which is physically impossible. Three hypotheses are still under investigation in order to explain this phenomenon.

The first one is the folding-unfolding phenomenon of the specimen during the loading. The residual stress introduced during the fabrication of the specimen leads to an initial transversal folding of the specimen. During the earliest stage of the loading, the specimen gradually unfolds until it reaches a relative planarity. Then the specimen folds again and remains in this state until failure appears. The hypothesis is that the energy that is usually dissipated in the transversal contraction of the specimen during an experiment on a macroscopic sample is here used to successively fold and unfold the specimen.

The surface effects could also take part in the overestimation of the geometrical effect in the characterization of damage. Indeed, the volume to surface ratio is much more important for a thin film specimen than for a macroscopic one, and then the surface effects are intensified in comparison with the volume effects for a thin film specimen.

Finally, the microstructure of the film could have an influence on the electrical and mechanical properties of the film. The theoretical calculations are based on the hypotheses of an isotropic and homogeneous material. The specimen comes from a batch with a grain size of approximately 20nm [8]. Thus, there are only 4 and 5 grains in the thickness of the specimen and the hypotheses of homogeneity and isotropy must then be reappraised.

Conclusion

The characterization of mechanical damage of 1 μ m thick freestanding aluminium films during micro-tensile experiments has been performed using both mechanical and electrical methods. It has been highlighted that monotonic stress-strain curves give not enough information to fully characterize the elastic-plastic response of a material. Indeed, the decrease of stress when strain reaches 1.5% is linked to softening of aluminium whereas damage occurs only for strain above 11.8%.

The multi-cycle experiments - without electrical measurements - are used to correlate the estimation of the damage variable D using electrical measurements with a mechanical approach. Several unloading-loading cycles have been performed every 50 μ m, until the specimen failure. The damaged modulus of elasticity \tilde{E} has been measured for each unloading phase and has been linked to the damage variable D .

A theoretical calculus of the damage variable D from electrical measurement has been presented but lead to unreliable results. Three hypotheses that could explain this phenomenon still under investigation are discussed.

Another method based on both electrical measurement and multicycles results is developed. This method gives value of damage variable D lower than the mechanically calculated one. The explanation of such results lays in the phenomena that lead the damage of the specimen materials. The shape of the evolution of damage variable D is similar for both the methods.

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