Universal testing machine for mechanical properties of thin materials

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In this work, the design, construction, calibration and compliance measurement of a universal testing machine for tension tests of materials in film geometry are presented. A commercial load cell of 220 N and sensitivity of 1.2345 mV/V is used to measure the applied load. Material strain is measured by movement of the crosshead displacement of the machine with a digital indicator with 0.001 mm resolution and 25 mm maximum displacement, connected to a PC through an interface. Mechanical strain is achieved by an electric high precision stepper motor capable to obtain displacement velocities as low as 0.001 mm/s. The stress-strain data acquired with a GPIB interface are saved as a file with a home-made program developed in LabView 7.0. Measurements of the elastic modulus and yield point of a commercial polymer film (500 HN Kapton) were used to validate the performance of the testing machine. The obtained mechanical properties are in good agreement with the mean values reported by the supplier and with the values obtained from a commercial machine, taking into account the limitations of thin film testing and experimental conditions.

Keywords: Universal testing machine; elastic modulus; thin films; compliance.

En este trabajo se discute el diseño, la construcción, la calibración y la medición de la complianza de una máquina universal para pruebas de tensión de materiales en geometría de pelicula. La carga aplicada es medida con una celda de carga comercial de 220 N y sensitividad de 1.2345 mV/V. La elongación de la muestra es medida a través del desplazamiento del cabezal de la máquina con un indicador de carátula digital con resolución de 0.001 mm y desplazamiento máximo de 25 mm, conectada a una PC a través de una interfase de puerto serial. La deformación mecánica es conseguida con un motor a pasos de alta precisión capaz de conseguir velocidades tan bajas como 0.001 mm/s. Los datos adquiridos a través de una interfase GPIB en tiempo real son guardados en un archivo usando un programa de diseño propio desarrollado en LabView 7.0. El módulo de elasticidad y el punto de fluencia medidos en una pelicula polimérica comercial (Kapton 500 HN) fueron utilizados para evaluar el desempeño de la máquina construida. Los valores de las propiedades mecánicas obtenidas muestran buen acuerdo con los valores reportados por el proveedor y con los resultados obtenidos con una máquina comercial, considerando la geometría de capa delgada.

Descriptores: Máquina universal de pruebas; módulo de elasticidad; capas delgadas; complianza.

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1. Introduction

New methodologies to measure the physical properties of thin films are currently required. Particularly, reported mechanical properties of materials at these dimensions are currently controversial in the scientific literature. Thus, it is necessary to propose techniques for determining mechanical properties of thin films, such as elastic modulus, Poison's ratio and strength. Properties of materials at micro and nanoscale are of considerable interest because of the unique properties associated with small volumes. These unique properties are increasing the importance of thin films and nanostructured materials used in several technological applications. At present, flexible electronics onto polymer substrates [1-2] are being developed for many applications, such as electronic textiles [3] and paper-like displays [4-5]. These electronic devices are normally subjected to high external stresses and large strains due to the flexible material used as substrate [6]. As it is well known, the film properties can be quite different from the bulk properties [7]. Thus, knowledge of the mechanical properties of nanostructured materials is essential to obtain functional and reliable electronic devices. Different methods have been proposed to investigate the mechanical properties of these thin materials. The most common methods are based on X-ray diffraction [8], interferometry [9] and most recently, nanoindentation [10-11]. These methods often require sophisticated instrumentation and do not compel with the conventional definition of mechanical properties, which are defined in terms of conventional tensile testing. In bulk geometry, the mechanical properties are commonly obtained

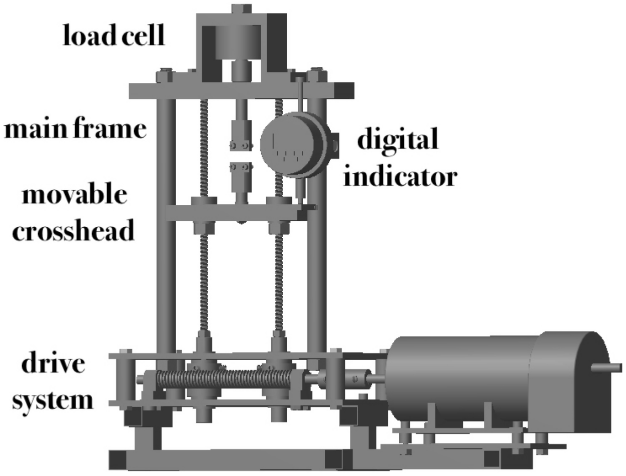

FIGURE 1. 3D view of the universal testing machine.

through tensile testing, and there exists different commercial machines for characterization of bulk materials [12-15]. Tensile testing is an effective way to investigate the mechanical properties of materials and it is a well established technique for bulk sample characterization. However, tensile testing is not easily implemented for micro and nano-structured materials due to the small dimensions of the specimen. At present, there exist these testing machines for materials in film geometry [16-18] but they are expensive and its flexibility to make modifications is limited. In this work, the design, construction, calibration and compliance determination of a universal testing machine, specially designed for tensile tests of materials in film geometry is discussed. Our universal testing machine is designed to produce small deformations at low velocities and, consequently, high resolution. The testing machine is sensitive to small loads and permits to obtain the stress-strain curves for materials in film geometry. Elastic modulus, yield point, and maximum stress can be obtained from samples of up to about 10 cm length. An important feature of this device is the simplicity to exchange components according to the user requirements: the low cost, low machine compliance and the high resolution obtained. This machine can also be adapted for compression testing with appropriate samples and grips. However, compression testing will not be addressed in this work.

2. Design and construction

The testing machine was designed to determine the stress-strain curves of thin materials such as polymers and particularly metallic films deposited onto polymeric substrates. Figure 1 shows a 3D illustration of the designed device with 15 cm wide, 55 cm length and 45 cm high as total dimensions. The equipment is capable of analyzing samples up to 10 cm of length. The mechanical design minimizes effects of load introduction in the main frame, drive screws, and the relative movement between the movable crosshead and the drive screws.

As is shown in Fig. 1, the testing machine is composed of five main parts:

i) the main frame,
ii) the drive system,
iii) the movable crosshead,
iv) the load cell, and
v) the digital indicator.

The testing machine is mainly made of stainless steel, excepting some frictional elements like the gears, which were made of bronze.

The main frame includes the rectangular base where the gearbox is placed, the fixed crosshead and the two vertical parallel columns. The drive system includes a stepper motor with variable speed. The gearbox is formed by a worm shaft, and two worm gears, which moves the two drive screws. The movable crosshead is integrated by the bottom grip, two conical fastener tools with internal thread and an adjustable conical ring. The conical fastener tools provide stabilization to the movable crosshead when moving along the drive screws. The load cell used is a LCC-HTC-50 dual stud cell from Load Cell Central Co. [19] which withstands a 220 N maximum load with a sensitivity of 1.2345 mV/V. The load cell can be used for tension or compression testing, it is located on the upper side of the frame and supports the upper grip. The digital indicator measures the crosshead displacement and consists of a digital micrometer from Starret [20] with 0.001 mm of resolution, which is connected with an RS232 interface to a personal computer to acquire data.

To achieve high resolution in the measurements, the device includes a stepper motor used to control both velocity and torque. The drive system achieves displacements as small as 0.001 mm and velocities between 0.001 and 0.1 mm/s. Two stainless-steel grips (one fixed to the load

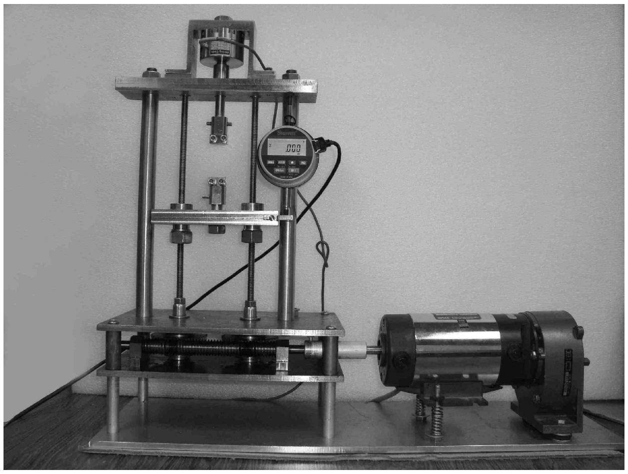

FIGURE 2. Photograph of the universal testing machine. The different components shown in Fig. 1 such as the drive system, load cell, electronic indicator, movable crosshead and stepper motor can be observed.

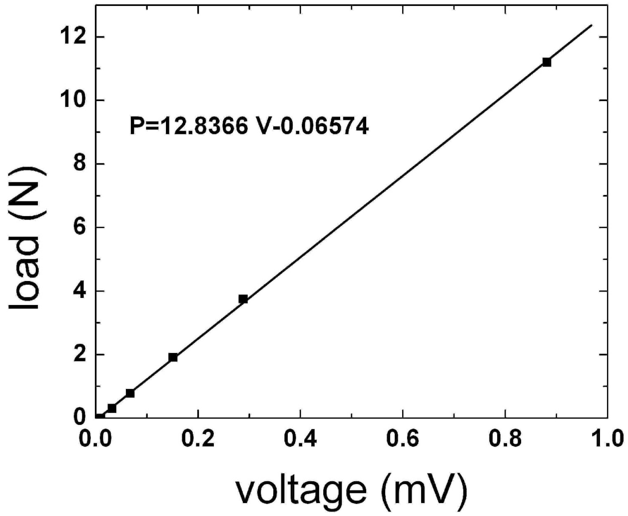

FIGURE 3. Calibration curve showing the relation between applied load and the voltage response of the load cell.

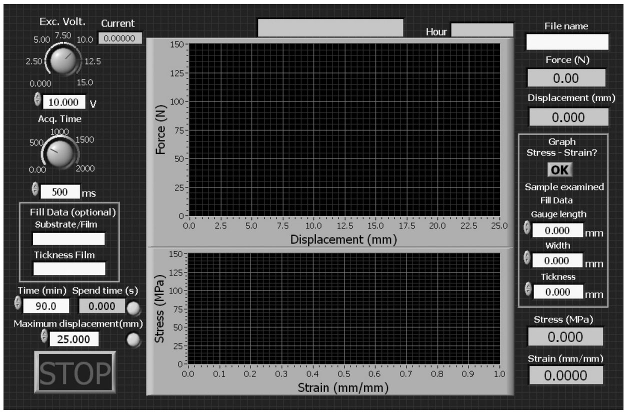
Exc. Volt. 10.000 V
Current 0.00000
Acq. Time 500 ms
Time (min) 90.0
Spend time (s) 0.000
Maximum displacement(mm) 25.000
Force (N) 0.00
Displacement (mm) 0.000
Gauge length 0.000 mm
Width 0.000 mm
Tickness 0.000 mm
Stress (MPa) 0.000
Strain (mm/mm) 0.0000

FIGURE 4. Frontal panel of the program developed in LabView. The program permits to control the excitation voltage and acquire the applied force and the displacement.

cell and the other on the movable crosshead) are used to hold the samples. The grips were designed with smooth surfaces to avoid damage to the soft and thin samples. Each grip is composed by a fixed part and a movable plate joined with two screws to uniformly press the samples. This holding system avoids sliding between sample and grips during tensile tests. The design of this universal testing machine permits the interchange of the different parts such as the load cell, grips and drive screws, in order to extend the user requirements. Figure 2 shows a photograph of the universal testing machine. Stress-strain data are captured and saved in a data file through a GPIB interface controlled with a home-made protocol programmed in LabView 7.0.

3. Performance

In order to determine the performance of the proposed testing machine, following the work done to calibrate the load cell, the methodology used to obtain the compliance of the machine, and the data acquisition are discussed.

3.1. Calibration and data acquisition

The calibration of the load cell was conducted by collecting data of different known applied loads (weights) and measuring its corresponding output voltage.

Calibration measurements were conducted by steps over a range of 0 to 12 N with an elapsed-time of 1 min between each calibrated load in order to avoid hysteretic effects; *i.e*, the voltage returns to cero value after removing the load. A series of calibrated loads were applied in increasing order. The output voltage of the load cell was captured through a high-resolution programmable voltmeter HP 3458A. Figure 3 shows the obtained linear behavior between the applied load (P) and the output voltage (V) as obtained from the load cell. The equation describing this relationship can be expressed by:

$$P = 12.8366V - 0.06574 \tag{1}$$

where the applied load P is given in Newtons and the output voltage V in milivolts.

This linear behavior confirms the information provided by the manufacturer and permits to obtain a relationship to be used into the program as a transduction signal.

According to the supplier, the load cell can support maximum loads of 220 N, but it is not desirable to reach this limit given that supplier guarantees a linear deviation of 0.15% at full load.

The data acquisition system uses a GPIB interface of National instruments to control the applied excitation voltage of the power supply and to collect the corresponding output voltage of the voltmeter. The GPIB interface is controlled with a home-made program developed in Labview 7.0. Figure 4 shows the frontal panel of the implemented program and the different parameters used to obtain the stress-strain curve. The designed program permits to select the acquisition time, step-time, and the excitation voltage for the load cell, and requires the length, width, and thickness of samples as entries. The force-displacement and stress-strain curves are plotted in real time during testing. Using this program, parameters as data acquisition time, displacement, and applied force can be captured and saved in real time for subsequent analysis.

3.2. Machine compliance determination

In mechanical testing of materials, when a strain gage or an in-situ element cannot be used to measure the real material strain, it is customary to use the machine crosshead displacement to measure the applied strain. Measurements conducted by crosshead displacement need to be calibrated by taking into account the machine compliance C_m. In order to calibrate the machine compliance ($C_m = 1/k_m = \delta/P$, where k_m is the stiffness constant, δ the crosshead displacement, and P the applied load), a specific experiment was conducted by tensile testing by using a stiff metallic sample (as compared

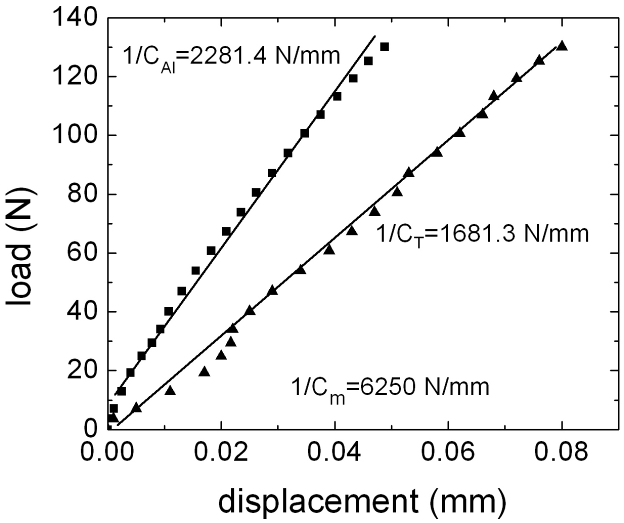

FIGURE 5. Rigidities obtained from the universal testing machine to calculate the total and material compliances.

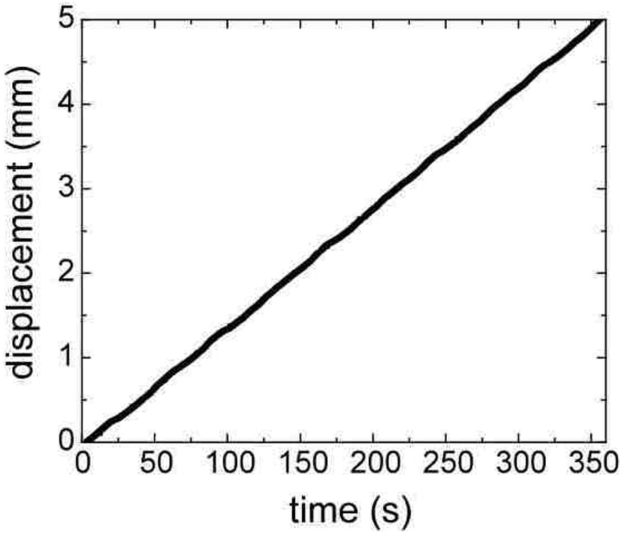

FIGURE 6. Displacement of the movable crosshead vs. time during a tensile test.

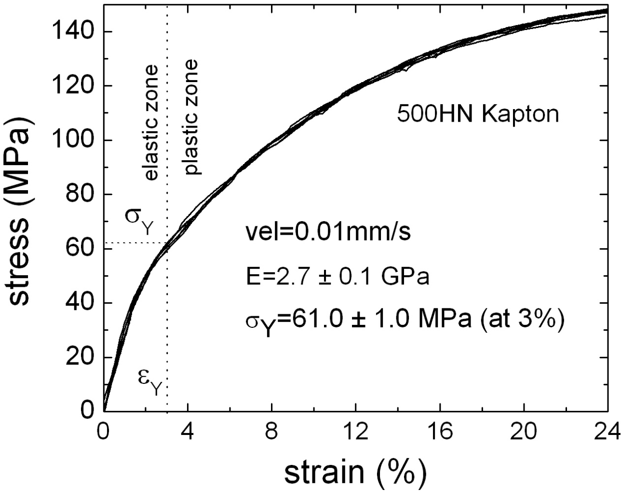

FIGURE 7. Six stress-strain curves obtained from the Kapton foil through our home-made machine, showing the elastic and plastic zone of the polymer.

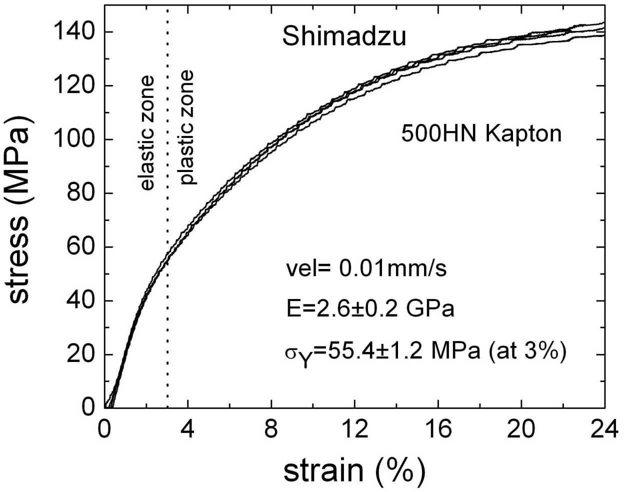

FIGURE 8. Stress-strain curves for Kapton samples obtained with a Shimadzu testing machine under similar conditions than Fig. 7.

with soft materials). Compliance was obtained through tensile testing by using aluminum samples with 35 mm gage lenght, 4.2 mm wide and 0.46 mm thick. The tensile test was done by applying loads from 0 to 140 N, limited by the capacity of the load cell. Strain was measured simultaneously by the machine crosshead displacement and by a commercial strain gage (Vishay ED-DY-062AK-350) bonded to the mid-part of the sample. The strain gage was connected to a Vishay model P3 strain indicator to record the gage signal. Half bridge configuration was used in order to minimize temperature effects. The total compliance measured by the crosshead displacement (C$_T$) is a sum of the compliance of the analyzed material (C$_{Al}$) and the compliance of the machine (C$_m$), simulating a series spring system. Since C$_T$ and C$_{Al}$ are measured during the experiment, the next relation can determine the machine compliance:

$$C_T = C_m + C_{Al} \tag{2}$$

Figure 5 shows load-displacement curves for the analyzed sample measured by the machine-crosshead and strain gage (simultaneously). The values of the compliances (inverse of the load-displacement slopes) measured by both methods are C$_{Al}$=0.43 μm/N and C$_T$=0.59 μm/N. According to Eq. (2), the measured values yield a machine compliance of C$_m$=0.16 μm/N. As it will be further discussed in connection with experiments on Kapton, the compliance of our machine is significantly low, confirming that our universal testing machine is appropriated to obtain mechanical properties of materials with low modulus, thin films, and polymers.

The machine compliance value is constant and needs to be considered to determine the real value of the elastic modulus of a material under test, if the crosshead displacement is used to measure strain. To determine the real elastic modulus (E) of a material under axial tension it is necessary to take into account the machine compliance. This can be done using a spring-in-series system. The elastic modulus as determined with the machine crosshead displacement (E_T) needs to be corrected to obtain the real modulus E, by the relation [21,22]:

$$E = \frac{E_T}{1 - \frac{C_m E_T A}{L}} \qquad (3)$$

where Cm is the measured machine compliance, A the sectional area, and L the gage length.

4. Results and validation

A commercial polymer, 500 HN Kapton, was initially used as a benchmarking specimen by its well-known properties reported by the DuPont Co. supplier [23]. Tensile tests were conducted using rectangular geometries of Kapton films of 40 mm length, 5 mm wide and 0.125 mm thick. The gage length of the samples was always 20 mm. The displacement velocity of the movable crosshead during tensile experiments was maintained at 0.01 mm/s in all cases.

Figure 6 shows a plot of the displacement of the movable crosshead vs. time, where high stability can be observed when it moves along the drive screw with the sample gripped. From Fig. 6, a constant behavior of the velocity and very low mechanical noise during the crosshead displacement is evident. Therefore, the movement of the movable crosshead does not have additional effects, such as vibrations or speed changes that could affect the tensile tests.

Figure 7 shows a group of six superimposed stress-strain curves as obtained from different samples of the Kapton foil. The six curves are difficult to visualize given their high reproducibility. From this figure, it can be observed that Kapton has a linear elastic behavior below 1.8% strain. The total strain applied to the samples was always 24%, which is far from the ultimate strain reported by the supplier (72%). This high strain can not be achieved by our machine giving the mechanical limits of the device.

In Fig. 7, the initial slope of the curve (elastic zone) corresponds to the elastic modulus. The value of the real elastic modulus (E) calculated from the different stress-strain curves and corrected through Eq. 3 were estimated in 2.7 ± 0.1 GPa as mean value. This value is 1.1% larger than the E_T value measured from the stress-strain curves directly obtained from the machine. The low dispersion of data affirms the high reproducibility of the measurements inducted on Kapton foils. The yield point (σ_Y) was determined as the stress obtained at 3% of the deformation (ε_Y) as shown in Fig. 7. The mean value of this parameter estimated in 61.0±1.0 MPa. The stress obtained at 24 % strain was ranged from 146.1 to 149.3 MPa, with an average of 147.6±1.0 MPa. For comparison, the mean values of the elastic modulus and the yield point for Kapton as provided by the supplier are 2.5 GPa and 69 MPa at 3% of strain [23], respectively. The small differences measured by our testing machine (9 and 11%) can be due to the difference in the geometry of the samples used by the manufacturer (dog-bone), according to the ASTM D-882-91 standard, and the different test velocity (10 mm/s) used. In order to compare our results, Fig. 8 shows results of four similar samples of Kapton tested in a commercial Shimadzu machine model AGI-100, with a load cell of 500 N, using a crosshead speed of 0.01 mm/s. From results, the elastic modulus and the yield point were estimated in 2.6 ± 0.2 GPa and 55.4 ± 1.2 MPa, respectively. Thus, although the values are somewhat similar, it is very difficult to obtain the same values than those reported by the supplier, if different test conditions are used.

It is known that thickness and specimen length can affect the stress-strain curves of materials, especially those in film geometry [24]. Different test conditions can cause variations on the measurements of mechanical properties of the polymers. For example, notorious effects in the plastic zone due to the testing velocity have been reported in ref [25]. In several reports [26,27] the analyzed samples deviate from the size and geometries required by the ASTM standards, and sometimes the different values measured of certain mechanical properties can not be comparable. Thus, testing of micro and nano-materials demands to establish new standards and fix the test conditions, size and, geometry of the samples, to obtain reproducible mechanical properties.

5. Conclusions

The design, construction, calibration and compliance measurement of a universal testing machine for tensile tests on thin and soft materials were discussed. The design has the capability to obtain displacement as small as 0.001 mm and maximum loads of 220 N. The estimated compliance machine value was 0.16 μm/N as measured with a stiff material. The mechanical properties of a 500HN Kapton polymer film were measured and compared with the mean values reported by the supplier as well as independent testing using a commercial testing machine. The corrected average elastic modulus and the yield point of Kapton film determined with our home-made testing machine were 2.7 GPa, and 61.0 MPa. This elastic modulus value was corrected in about 1.1% for the case of Kapton, and represents a slight difference as compared with the mean values reported by the supplier; and is also in reasonable agreement with the values obtained with the Shimadzu machine. The performance and low compliance value of our testing machine indicate that it is appropriate to obtain reliable mechanical properties of compliant materials in thin and soft materials. Our testing machine permits to interchange different elements according to the user requirements. An additional advantage of our testing machine is the lower cost and smaller size compared to other commercial machines. Future efforts will address the use of this equipment to obtain the mechanical properties of thin metallic films (pure and alloys) with different nano-thickness deposited on polymeric substrates.

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1. C.T. Huang. C.L. Shen, C.F. Tang, and S.H. Chang, *Sensors & Actuators A* **141** (2008) 396.
2. S.A. Wilson, R.P.J. Jourdain, and Q. Zhang, *Mat. Sci. Eng. R* **56** (2007) 1.
3. E. Bonderove and S. Wagner, *IEEE Electr. Dev. Lett*. **25** (2004) 295.
4. J. Rogers, *Science* **291** (2001) 1502.
5. Y. Chen *et al.*, *Nature* **423** (2003) 136.
6. F. Macionczyk and W. Brückner, *J. Appl. Phys.* **86** (1999) 4922.
7. A.I. Oliva Arias, F. López Garduza, and V. Sosa, *Ingeniería* **10** (2006) 57.
8. P. Goudeau *et al.*, *Thin Solid Films* **49** (2001) 398.
9. W.N. Sharpe Jr., B. Yuan, and R.L. Edwards, *J. Microelectromech Syst.* **6** (1997) 193.
10. CS. Oh, H.J. Lee, S.G. Ko, S.W. Kim, and H.G. Ahn. *Sensors & Actuators A* **117** (2005) 151
11. R. Saha and W.D. Nix, *Acta Mater.* **56** (2008) 1399.
12. P.C. Lehning Jr, *US Patent* **3203232** (1965).
13. W.T. Gloor, *US Patent* **3323357** (1967).
14. R.J. Simonelli, *US Patent* **5913246** (1999).
15. S. Bergs and F. Sweden, *US Patent* **6148676** (2000).
16. A.V. Sergueeva, J. Zhou, B.E. Meacham and D.J. Branagan, *Mater. Sci. Eng. A* **526** (2008) 79.
17. K. Yang *et al.*, *Acta Mater*. **58** (2010) 967.
18. O. Kulyasova, R.K. Islamgaliev, B. Mingler, and M. Zehetbauer. *Mat. Sci. Eng. A* **503** (2009) 176.
19. Load Cells, *Dual Stud Load Cell Tension/Compression Model: LCC-HTC,* http://www.800loadcel.com.
20. Starret, Athol, MA, and US, *Catalog No. 31B* (2007) 192.
21. U. Ramamurty and A. Paul. *Acta Mater.* **52** (2004) 869.
22. N. Huber and Ch. Tsakmakis, *J. Mater. Res.* **13** (1998) 1650.
23. DuPont, Circleville, OH, and US, *Technical Data Sheet* (1999) 1.
24. Y.H. Zhao *et al.*, *Scripta Mater.* **59** (2008) 627.
25. *Handbook of Polymer Testing*, Edited by R. Brown (Rapra Technology, Shrewsbury, Shropshire SY4 4NR, UK. 2002). p. 97
26. K.M. Youssef, R.O. Scattergood, K.L. Murty, J.A. Horton, and C.C. Koch. *Appl. Phys. Lett.* **87** (2005) 091904.
27. Y.H. Zhao *et al.*, *Adv. Mater.* **18** (2006) 2494.