

## About the measurement of dynamic mechanical properties of bi-layer systems

S.N. Goyanes<sup>a,b,\*</sup>, A. Roncaglia<sup>a</sup>, F. Saavedra<sup>a</sup>, G.H. Rubiolo<sup>a,b,c</sup>

<sup>a</sup> *Dpto Física, Facultad de Ciencias Exactas y Naturales, Universidad de Buenos Aires, Ciudad Universitaria, Pabellón I, (1428) Buenos Aires, Argentina*

<sup>b</sup> *Consejo Nacional de Investigaciones Científicas y Técnicas (CONICET), Buenos Aires, Argentina*

<sup>c</sup> *Dep. de Materiales, Comisión Nacional de Energía Atómica (CNEA), Av. Del Libertador 8250, (1429) Buenos Aires, Argentina*

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### Abstract

The bi-layer system has been well developed for many engineering structural components in the aircraft, automotive, microelectronics and modern space industries in the last few decades. Consequently, a good determination of its dynamic mechanical properties is important for an appropriate design of its industrial application. These studies are usually performed by a three-point bending test using a dynamic mechanical analyzer (DMA). In this work, we show that the three points bending test on a bi-layer system carried out with the test tool provided in a commercial DMA, for example the dynamic mechanical thermal analyzer (DMTA) Rheometric IV, can lead to erroneous results. Measurements on polymer-hard substrate bi-layer systems around the glass transition temperature of the polymer,  $T_g$ , give larger changes in elastic modulus and loss tangent than they are. This problem is not observed at temperatures lower than  $T_g$ . The design of new test tool and specimen geometry for the three points bending test is shown together with its application to the measurement of the dynamic elastic properties of a polyvinyl chloride (PVC)–aluminum bi-layer system.

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### 1. Introduction

In the last years, materials like symmetric sandwich metal–polymer–metal have been developed to reduce noise and vibrations in car and ship structures. Thanks to the relaxation phenomena associated to the polymer's glass transition, compounds with high loss factor and a significant stiffness are expected. Moreover asymmetric laminated metal–polymer compounds have been considered for regulating elastic modulus in modern aerospace industries. Therefore, the determination of their dynamic mechanical properties is important for a proper design in their industrial application.

In former times, the asymmetric compounds made up by a beam of relatively high stiffness glued to a polymeric beam have been used to obtain dynamic mechanical properties of materials too soft to support their own weight [1–6]. This

technique was also suggested to study the effects induced by modifications of the polymer morphology in the interfacial region [4–6]. These types of studies are usually performed by means of three points bending or torsion tests with a dynamic mechanical thermal analyzer (DMTA).

The aim of this work is to show that the three points bending dynamic test on polymer-hard substrate bi-layer beams carried out with inadequate tools can lead to erroneous results around the glass transition temperature of the polymer,  $T_g$ . We suggest new design for the tools and the sample that can help to solve these problems. The experimental results obtained using the commercial tool are compared against those obtained with our tool design. Finally, both results are compared against the ones predicted from the classical theory of bi-layer beams assuming perfect interfaces.

### 2. Experimental

Two different bi-layer beams were considered in this work. One of them is prepared with materials of similar

\* Corresponding author. Tel.: +54-11-4576-3353;

fax: +54-11-4576-3357.

E-mail address: goyanes@df.uba.ar (S.N. Goyanes).

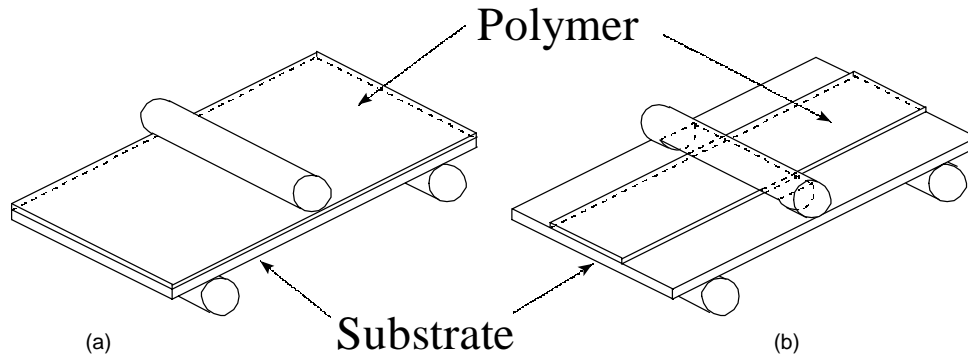


Fig. 1. Experimental arrangement for the three point bending test: (a) the classical design of the bi-layer beam sample and the drive shaft clamp; (b) our design.

storage modulus (epoxy resin–polyvinyl chloride (PVC)) and the other with very dissimilar ones (aluminum–PVC). Processing of the bi-layer beam is achieved by gluing together the simple beams with an anaerobic adhesive.

The PVC used was a commercial product from Heling Argentina. The aluminum is a 1060-T0 alloy (min. 99.6% Al), and the epoxy resin is a diglycidyl ether of bisphenol A (DGEBA, 100 pbw (parts by weight)) with methyl tetra hydrophthalic anhydride (MTHPA, 90 pbw) as hardened and diglycidyl ether and heterocyclic amine (*N*-metil imidazol, 0.7 pbw) as accelerant, all of them from CIBA (Argentina). The resin was cured 4 h at 80 °C and post cured 8 h at 140 °C.

The tool redesigned by us is the drive shaft clamp that deforms the center of the sample in the three point bending test. Fig. 1 shows the schematic diagrams of the sample geometry and its arrangement for the test in the classical proposal and in our proposal. We adopt the following notation for the sample geometry:  $h_{\text{PVC}}$  is the thickness of the PVC beam and  $h_s$  refers to the thickness of the aluminum or the epoxy beams indistinctly, the same follows for the width  $w$ . The values of these parameters for each type of bi-layer beams are given in Table 1.

In order to evaluate the contribution of the anaerobic adhesive, a bi-layer sample with the geometry shown in Fig. 1a was made by gluing together two simple beams of PVC.

The dynamic mechanical tests were performed with a dynamic mechanical thermal analyzer, Rheometric DMTA IV, in the three point bending mode. The test were performed with a deformation  $\varepsilon = 1.5 \times 10^{-4}$ , at a frequency of 1 Hz, and with a heating rate of 2 K/min in the range of temperatures between room temperature and 120 °C.

Table 1  
Geometry of the bi-layer beams

Type of bi-layer	$h_s$ (mm)	$h_{\text{PVC}}$ (mm)	$w_s$ (mm)	$w_{\text{PVC}}$ (mm)
Epoxy–PVC	1.135	0.79	7.45	5.00
Aluminum–PVC	0.985	0.55	8.35	5.00

### 3. Results and discussion

The Fig. 2 shows the dynamic mechanical characterization of the aluminum beam. The loss tangent,  $\tan \delta$ , increases exponentially with the temperature following the typical behavior of the high temperature background of polycrystalline metals and the storage modulus  $E'$  decreases as expected. The measured values agree with those reported in the literature [7–9].

The dynamic mechanical spectra of the bi-layer beam PVC–adhesive–PVC and of the simple beams of PVC and epoxy are shown in Fig. 3. The PVC exhibits a main mechanical relaxation at 74 °C. The epoxy resin has its glass transition temperature at,  $T_g \approx 130$  °C and therefore only the left hillside of its main mechanical relaxation is displayed in the loss tangent. Any observable effect of the adhesive layer appears on the dynamic mechanical spectrum of the compound beam PVC–adhesive–PVC. All values of the measured dynamic mechanical properties agree with published data [10–13].

The dynamic mechanical characterization of the bi-layer beams epoxy–PVC and aluminum–PVC are shown in Figs. 4 and 5. The data include measurements over samples with the geometry and the arrangement for the test in the classical

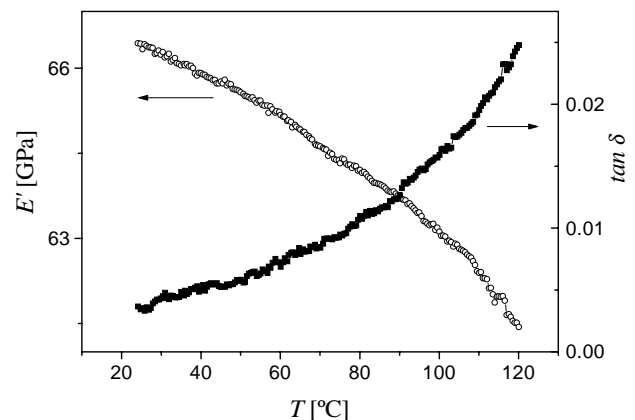


Fig. 2. Dynamic mechanical characterization of the aluminum beam.

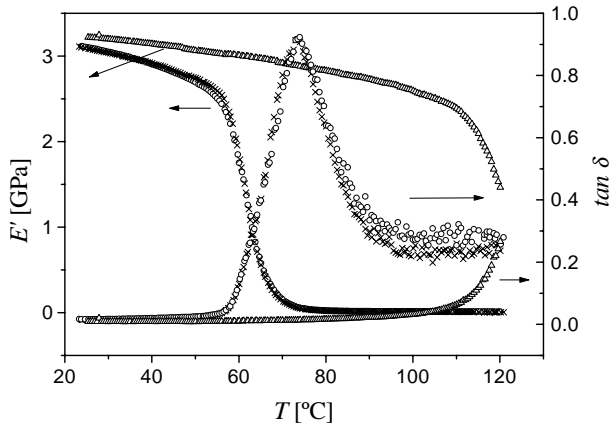
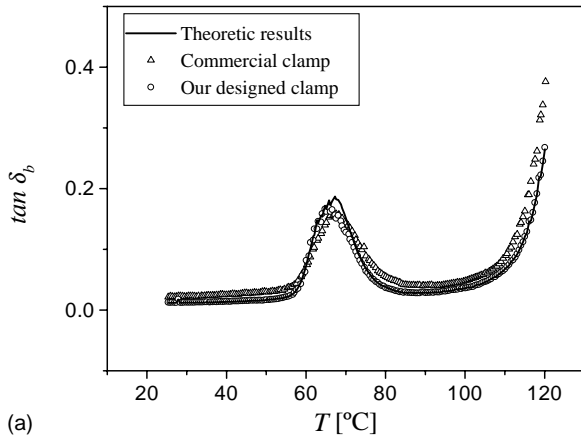
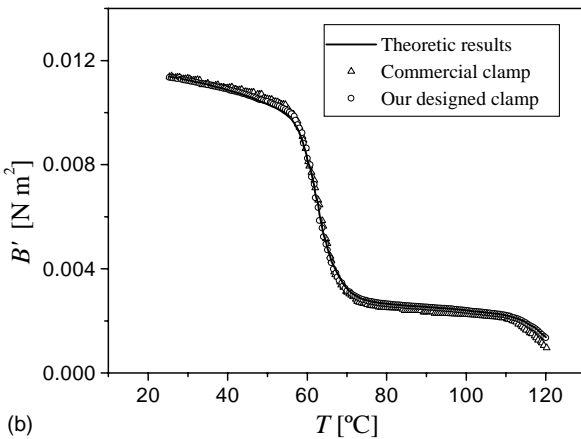


Fig. 3. Dynamic mechanical characterization of the simple epoxy and PVC beams and the compound beam PVC–adhesive–PVC. ( $\Delta$ ) Epoxy, ( $\circ$ ) PVC; ( $\times$ ) PVC–adhesive–PVC.

proposal and in our proposal. The Figs. 4a and 5a show the loss tangent of the bi-layer beam,  $\tan \delta_b$ , while Figs. 4b and 5b show the real part of the complex bending stiffness,  $B'$ , defined as [14]:

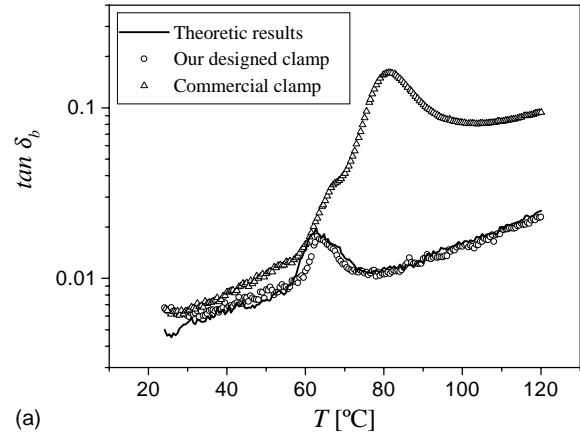


(a)

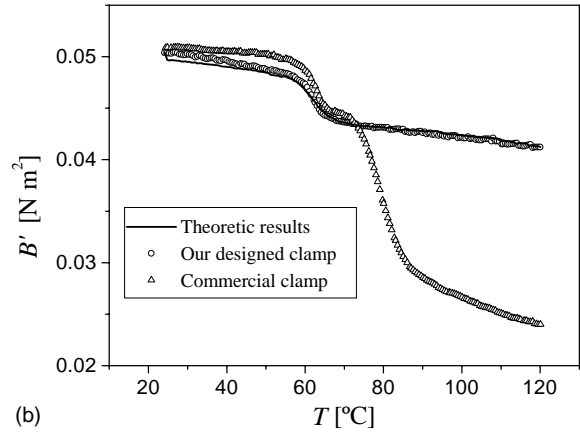


(b)

Fig. 4. Dynamic mechanical characterization of the bi-layer beam epoxy–PVC. The solid line shows the theoretic results using Schwarzl’s model [16].



(a)



(b)

Fig. 5. Dynamic mechanical characterization of the bi-layer beam aluminum–PVC. The solid line shows the theoretic results using Schwarzl’s model [16].

$$B' = \frac{l^3 F}{48D} \cos \delta_b \quad (1)$$

where  $l = 28$  mm is the length of the sample,  $F$  the applied force,  $D$  the displacement and  $\delta_b$  is the phase angle between  $F$  and  $D$ .

Both sample geometries and experimental arrangements give similar results on the bi-layer beam of epoxy–PVC. This fact does not occur with the bi-layer beam of aluminum–PVC. The loss tangent obtained with the geometry and the arrangement for the test in the classical proposal shows a main peak with a shoulder over its left hillside. This effect is also observable in the real part of complex bending stiffness,  $B'$ , as a second relaxation. The main relaxation is a little displaced to high temperatures in respect to the measured glass transition temperature of the PVC. Instead of this, the sample geometry and experimental arrangement proposed by us gives a result with only one mechanical relaxation whose maximum coincides with the position of the shoulder in the result mentioned above.

In order to clarify the discrepancy observed between the experimental results, we calculated the expected behavior of a bi-layer system considering an ideal interphase for the

coupling between phases by using the model proposed by Schwarzl [15,16]. Following this model, the PVC layer and the substrate layer (aluminum or epoxy, where it corresponds) have complex tensile moduli

$$E^* = E'(1 + i \tan \delta)$$

and

$$E_s^* = E_s'(1 + i \tan \delta_s)$$

respectively, then the complex bending stiffness of the bi-layer beam  $B^*$ , defined as the ratio of bending moment to curvature, is written as

$$B^* = B'(1 + i \tan \delta_b) = E^* I_a + E_s^* I_{as} + \frac{E^* A E_s^* A_s (h + h_s)^2}{4(E^* A + E_s^* A_s)} \quad (2)$$

where  $I_a$ ,  $A$ , and  $h$  are the second moment of area of the cross section, the cross sectional area and the thickness of the PVC and  $I_{as}$ ,  $A_s$ , and  $h_s$  the corresponding quantities for the substrate layer.

Separating and equating the real and imaginary components in Eq. (2), we obtain  $B'$  and  $\tan \delta_b$  for the bi-layer beam as a function of the storage moduli and the loss tangents of the PVC and the substrate layer. The calculated results are illustrated in Figs. 4 and 5 with solid lines. As it can be seen there is an excellent agreement between the model and the experimental results obtained using our sample geometry and experimental arrangement.

In our opinion, the failure occurring when the measurement is done with the sample geometry and the arrangement for the test in the classical proposal comes from a compressive deformation mode over the polymeric layer (evidenced by the great intensity of relaxation). This happens only when the stiffness of the substrate is very high and the stiffness of the polymer is very low as near its glass transition temperature. The sample geometry and experimental arrangement proposed by us suppress that spurious mode of deformation because the tool pressures over the substrate.

#### 4. Conclusions

In this work we propose a new design for the sample geometry and the drive shaft clamp that deforms the cen-

ter of the sample. The new design must be used in the dynamic three point bending test of bi-layer beams when the compound is prepared with materials of very dissimilar storage modulus. It helps to save the failure in the experimental results obtained on the same type of compound by using the sample geometry and tools provided with the commercial equipments. The theoretic results predicted by the Schwarzl's model assuming perfect interfaces between the bi-layer's constituents fall in good agreement with the experimental data obtained with the sample geometry and experimental arrangement proposed by us.

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#### References

- [1] T.J. Dudek, *J. Composite Materials* 4 (1970) 72.
- [2] A.A. Roche, J. Dumas, M. Romand, in: *Proceedings of the European Adhesion Congress EURADH'92*, Karlsruhe, Germany, DEHEMA, 1992, p. 238.
- [3] T. Nicholas, *Shock Vibr. Bull.* 38 (1968) 13.
- [4] D.J. Macon, *Polymer* 42 (2001) 5285.
- [5] J. Bouchet, A.A. Roche, E. Jacquelyn, *J. Adhesion Sci. Technol.* 15 (2001) 345.
- [6] J. Bouchet, A.A. Roche, E. Jacquelyn, *J. Adhesion Sci. Technol.* 15 (2001) 321.
- [7] Q.P. Kong, B. Cai, G. Gottstein, *J. Mater. Sci.* 36 (2001) 5429.
- [8] T.S. Kê, *Phys. Rev.* 71 (1947) 533.
- [9] G.W.C. Kaye, T.H. Laby, *Tables of Physical and Chemical Constants*, Longman, New York, 1995, p. 44.
- [10] E.A. Collins, C.A. Daniels, C.E. Wilkes, in: J. Brandrup, E.H. Immergut (Eds.), *Polymer Handbook*, Wiley, New York, 1975, p. V-41.
- [11] F. Povolò, S.N. Goyanes, *J. Appl. Polym. Sci.* 61 (1996) 359.
- [12] S.N. Goyanes, J.D. Marconi, P.G. König, M.D. Martin, I. Mondragon, *J. Alloys Compd.* 310 (2000) 374.
- [13] N.D. Alberola, G. Merle, K. Benzarti, *Polymer* 40 (1999) 315.
- [14] T.V. Duggan, *Stress Analysis and Vibrations of Elastic Bodies*, Temple Press, London, 1964, p. 89.
- [15] F. Schwarzl, *Acustica* 8 (1958) 164.
- [16] B.E. Read, G.D. Dean, *The Determination of Dynamic Properties of Polymers and Composites*, Adam Hilger Ltd., Bristol, England, 1978, p. 122.