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## Peeling Experiments of Double Side Adhesive Tapes Suggests the Feasibility of Graphene Nanocomposites with Gigantic Toughness

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Using a MTS micro-tensile testing machine equipped with a video camera, we studied the peeling dynamics of double sided adhesive tapes under imposed displacement. A standard *T*-peel test was considered for the tape system in order to assess its toughness and strength. The strength necessary to separate the folds and the total energy required to detach them were thus measured. The effects of the fold length and width both on the strength and energy adsorption were also evaluated, suggesting new strategies for the design of super-tough bio-inspired materials, mainly the need of a nanoscopic thickness thus suggesting graphene as the ideal candidate to produce super-tough nanomaterials.

## **Keywords:**

Since the inception of structural bonding, peel tests have been an integral part of the adhesive performance specifications, and have played an important role in the development of adhesives.

The phenomenon of adhesion has attracted attention due to scientific challenges it poses as well as for their industrial importance. Science of adhesion is truly interdisciplinary, involving a great variety of different interrelated physical phenomena like friction, fracture, mechanics of contact, visco-plastic deformation and interfacial properties, such as debonding and rupture of adhesive bonds.<sup>1–3</sup> Detailed mechanisms of such a complicated mixture of phenomena are not yet well understood.

Tests of adhesion are essentially fracture tests designed to study adherence of solids and generally involve normal pulling off<sup>4</sup> and peeling. Peeling also provides a rich insight into fracture mechanics as the dynamics is highly nonlinear and shows a variety of instabilities and complex phenomena. Furthermore, peeling experiments are comparatively easy to setup in laboratory and the recorded response helps to extract useful information on the features of the system.

Thus, adhesion is a common phenomenon in our daily life, concerning two materials sticking to each other. It is,

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sive behaviors quantitatively, since one must take into account not only the microscopic adhesive properties<sup>5,6</sup> of the surface but also the macroscopic deformation of the adhesive tape.<sup>7</sup> The first detailed experimental study on peeling of an adhesive tape was due to Maugis and Barquins.8 These experiments carried out at constant pull speed condition show that peeling is jerky within a window of pull speeds accompanied by acoustic emission.9,10 Constant load experiments have also been carried out recently.<sup>11, 12</sup> The dynamical properties of an adhesive tape have been studied by observing the morphology of the peeling front.<sup>13</sup> Thus peel-testing is a well established methodology in a lot of applications, involving adhesives and also nanomaterials,14 and has been used to a limited extent even in the biomaterials field.<sup>15-17</sup> The single peeling theory of Kendall has been extended to multiple peeling in Ref. [18], including large deformations and pre-stretching.

however, considered to be difficult to understand adhe-

In this letter, in order to quantify the mechanical properties of an adhesive tape system, load-extension curves are measured by peeling thanks to a MTS micro-tensile testing machine equipped with a video camera. In our case, we have double sided adhesive tape with a number of folds, thus the delamination will happen between different folds. For this case the *T*-peel test<sup>19, 20</sup> is often the more suitable procedure.

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**Fig. 1.** Schematic figure of: (a) *T*-peeling classical sample; inset (b) our (here two) folded samples.

The T-peel test was applied for mechanical investigation of the peel behaviour according to ASTM D 1876.19 The word "T-peel" indicates the existence of two peel arms, which are bent by 90° each. A schematic representation of the T-peel test, including sample geometry, direction of loading, respective to sample orientation and our peculiar folded geometry is shown in Figure 1. The initial distance between the clamps was 20 mm, and the peel rate was 5 mm/min. The recorded data of the peel test, force as a function of displacement, were used to determine the peel force  $F_{peel}$ . The recorded peel force and displacement of the end of the tape were plotted separately for each individual peel test. Table I lists the dimensions of each sample. MTS Series software was used to control the test machine and to collect the test data. Tensile tests were carried out under laboratory conditions. It is important to ensure that the bonded portion of the specimen remains perpendicular to the applied load.

In Figures 2–4 typical load versus crosshead displacement curves are shown for samples A and similar results where obtained for samples B and C. Also characteristic peeling configurations, obtained from video record, are reported. Experiments with four different folded samples, namely 1, 2, 3 and 4 folds were considered. Each point in the load-displacement diagrams labelled as a, b, c,... corresponds to a critical point of the peeling failure. Figure 2(a) considers one fold and shows the pseudoplastic plateau region (a)–(b) until 18 mm of displacement. The final stiffening (b)–(c) represents the tape traction.



**Fig. 2.** Peel test on samples A of double sided adhesive tape with one (a, A1) or two (b, A2) folds.

The curve in Figure 2(b), showing peeling test of two fold sample, has a longer plateau region composed by shorter sub-plateau. The decreasing in length of sub-plateau is a consequence of a partial delamination in each fold during the peeling process, as we directly observed. Figures 3 and 4 report the results for three and four folds of samples.

The peeling force increases with the transversal length *L*, since for a rigid tape we expect  $F_{\text{peeling}} = 2\gamma L/(1 - \cos \theta)$ ,<sup>7</sup> where  $\gamma$  is the surface energy and  $\theta$  is the peeling angle (here  $\pi/2$ ). For *N*-fold of double sided adhesive tapes the dissipated energy is  $w = 2(2N - 1)WL\gamma$ . Thus the expected dissipated energy density  $\psi = (w/v) = ((2N - 1)WL\gamma)/((2N - 1)WLt) = \gamma/t$ , where *t* is the thickness of the fold. Nano thickness is thus expected to be

Table I. Sample dimensions and adhesive properties ( $\gamma = 0.135$  N/mm); 1, 2, 3 or 4 folds were considered for each sample.

Sample	<i>L</i> [mm]	W [mm]	Number of folds	Dissipated energy (Exp.) [N mm]	Dissipated energy (Theo.) [N mm]	Peeling force (Exp.) [N]	Peeling force (Theo.) [N]
$\overline{A_1}$	10.0	10.0	1	32.1	27.0	2.0	2.7
$A_2$	10.0	10.0	2	73.6	81.0	2.2	2.7
$A_3$	10.0	10.0	3	140.5	135.0	2.6	2.7
$A_4$	10.0	10.0	4	175.4	189.0	3.3	2.7
$B_1$	10.0	20.0	1	63.1	54.0	2.0	2.7
$B_2$	10.0	20.0	2	162.7	162.0	2.3	2.7
$\overline{C_1}$	20.0	10.0	1	70.8	54.0	5.3	5.4
$C_2$	20.0	10.0	2	180.0	162.0	6.4	5.4

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Fig. 3. Peeling test on sample A of double sided adhesive tape with three folds (A3).



Fig. 4. Peel test on sample A of double sided adhesive tape with four folds (A4).

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Fig. 5. Comparison between dissipated energy experimentally measured and analytically calculated.



Fig. 6. Comparison between peeling force experimentally measured and analytically calculated.

fundamental to mimick biological super-tough materials, suggesting graphene as the best candidate.

The decohesion energy values presented in Figure 5 correspond to the area under the load-displacement curves, up to the tape pure stretching, and are compared with the calculations  $w_{\text{theo}}$  assuming a plausible value of  $\gamma = 0.135$  N/mm. Figure 6 shows the comparison between experimental and analytical calculated peeling forces (same value of  $\gamma$  used in the comparison of Fig. 5).

Our experimental results suggest the validity of our concept, that is the feasibility of materials with gigantic toughness thanks to graphene based nanocomposites.

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