Adhesion energy in nanogap InP/InGaAs microcantilevers

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The adhesion energy is measured between InGaAs quantum wells that have collapsed across a 125 nm air gap in an InP/InGaAs heterostructure. The method relies on measuring the unadhered length and shape of collapsed microcantilevers with optical interferometry. The adhesion energy is found to be 72±16 mJ m −2. Since the air gap is much smaller than has been measured previously, the influence of van der Waals forces across the gap was included in theoretical modeling. It was found that the forces should not cause significant deviation from the standard adhesion models unless the adhesion energy drops below 25 mJ m−2.

One of the most problematic issues in micromachining is “stiction,” where attractive forces between surfaces can cause distinct mechanical structures to permanently adhere to one another. It is a primary cause of micromachined device failure and, as such, has been studied by many authors in great detail. The adhesion energy between surfaces—perhaps the most critical parameter in quantitative studies of stiction—is defined as the energy per unit area required to separate two adhering surfaces. For most micromachined structures to date, the adhesion energy together with spring constants and certain geometrical and environmental information have been able to determine whether or not two structures will separate after an etch release or actuation. However, with current nanofabrication, feature sizes and mechanical gaps are decreasing well below a micron. In this letter the adhesion energy between cantilevers and substrates with InGaAs/InP quantum well surfaces is studied at small gap spacings on the order of 100 nm. A model which includes the influence of van der Waals forces across the air gap is used to describe the unadhered length of a collapsed cantilever. The average adhesion energy measured for two InGaAs surfaces in contact is found to be 72±16 mJ m−2 and the deformation is in close agreement with the theory.

The adhesion energy is measured with the method developed by Mastrangelo and Hsu and de Boer and Michalske using a microcantilever array. Their measurements examined the adhesion energies of polysilicon beams with a variety of surface treatments. Previous measurements of InP adhesion energy have also been performed using microcantilevers, but apparently no previous work has been done on InGaAs surfaces. This material system will likely have interesting quantum properties in nanomechanically tuned optoelectronic devices. In this work, optical interferometry is used to obtain additional information on cantilever shape.

The specific measurement method is as follows. A cantilever collapses after its length surpasses a critical length. Close to the critical length, the cantilever bends in an arc like fashion such that only its tip touches the substrate. A cantilever significantly longer than the critical length adheres over a long portion of its length thereby attaining an s-shape. To better assess the full range between complete separation and s-shape collapse, two types of test structures were fabricated. The first one consists of an array of 5 μm wide beams attached to a common anchor. The length of each beam increases from 5 to 50 μm in increments of 2.5 μm. An array of 200 μm long cantilevers forms the other type; the width of all the beams in this array was either 10, 20, or 40 μm.

The cantilever is modeled as an Euler-Bernoulli beam with the van der Waals forces acting as a load:

\[
\frac{d^2}{dx^2} \left( \frac{EI}{dx^2} u(x) \right) = A \frac{1}{6\pi y(x)^3},
\]

where \(E\) is the elastic modulus of the nitride, \(I\) the second moment of inertia of the cantilever, \(u\) the deformation, and \(y\) the distance between the cantilever and the substrate. The Hamaker constant \(A\) quantifies the strength of the van der Waals forces. An iterative finite element algorithm solves Eq. (1) and also calculates the unadhered length \(s\) which is the distance between the hinge points and the point where the cantilever touches the surface. Figure 1 shows the difference between the deflections of the cantilevers with and without taking into account the van der Waals forces across the air gap. van der Waals forces cause the shape to deviate from the s-shape. The deviations are only evident at small adhesion energies (<25 mJ m−2). At high energies, the difference between the shapes with and without van der Waals forces is negligible. Neglecting van der Waals forces—\(A=0\)

FIG. 1. (Color online) Each curve shows the difference between the shapes of the collapsed cantilever with and without taking van der Waals forces across the air gap into account. As the adhesion energy increases, the unadhered length of the beam decreases.

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in Eq. (1)—the relation between unadhered length and adhesion energy becomes

$$\Gamma = k \frac{E r^3 h^2}{s^4}. \quad (2)$$

The factor $k$ takes the different boundary conditions into account. For an arc-shaped cantilever it is 3/8; for $s$-shaped cantilevers it is 3/2.

The molecular beam epitaxy (MBE) grown InP/InGaAs heterostructure consists of the three lattice matched layers: a 5 nm InGaAs layer on top of the InP substrate, a 100 nm InP sacrificial layer, and a 5 nm InGaAs layer at the top. Then 3.2 $\mu$m of structural silicon nitride were deposited with plasma-enhanced chemical-vapor deposition at a temperature of 250 °C and at a pressure of 900 mTorr. A high deposition power of 200 W minimized the internal stress gradient in the nitride. A CF$_4$ and SF$_6$ based plasma etch transferred the cantilever pattern onto the nitride. Then the top InGaAs layer was etched for 120 s in a H$_2$SO$_4$ : H$_2$O$_2$ : H$_2$O 1:8:100 wet etch. After a thorough rinse, the cantilevers were released in a stirred HCl : H$_2$O 1:2 etch which was heated to 45 °C. Four samples with the following etch times were prepared: 2.5, 3, 4, and 4 h. The etch preferentially undercut along the (100) directions. The photoresist strip, each sample was baked at 170 °C for 3 min on a hot plate to reduce its adsorbed water content. Finally, an oxygen plasma cleaned the surface of organic residue.

As seen in Fig. 2 optical microscopy revealed light and dark regions within each nitride structure after the release. The light regions correspond to an air gap which scanning electron microscopy (SEM) images later confirmed. Hence, the longest pinned beam could simply be determined by optical microscopy of the array of 5 $\mu$m wide cantilevers.

Interferometric measurements with a ZYGO white light interferometer revealed the shape of the collapsed cantilevers. Figure 3 shows the profile along the top of the beam. The two reference points which mark the beginning and the end of the transition were set manually. The horizontal distance between them yields the unadhered length while the vertical distance gives the initial step height. The smooth line indicates the $s$-shape expected of a collapsed cantilever in the absence of forces across the air gap. The measurements of the initial step height deviated from the expected 100 nm by 25 nm. Atomic force microscopy measurements of a step etched into the sacrificial InP layer confirmed this deviation. After the nitride was peeled of using carbon tape, Auger electron spectroscopy confirmed the presence of InGaAs on the underside of the cantilevers.

In order to increase accuracy, a large number of measurements were taken. The horizontal resolution of the interferometer limited the number of measurements. The 5 $\mu$m wide beams were below the resolution of the system. Table I shows the compiled results. The nitride had a thickness of 3.22±0.05 $\mu$m. Nanoindentation measured an elastic modulus of 243508-2 Makowski, Gawarikar, and Talghader Appl. Phys. Lett. 89, 243508 (2006)

![Fig. 2](image1.png)

**FIG. 2.** (Color online) Top: Air gap between the nitride and the substrate appears as a light region. This micrograph reveals that the transition from released to pinned cantilevers occurs in this array of 5 $\mu$m wide cantilevers. Bottom: In the SEM micrographs of the third and fourth cantilevers, the air gap appears as a dark region under the tip of the right cantilever.

![Fig. 3](image2.png)

**FIG. 3.** (Color online) Interferometric measurements reveal the shape of a collapsed 20 $\mu$m wide cantilever. The smooth line indicates the expected shape. The profile corresponds to the cantilever indicated in the top picture.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Width ($\mu$m)</th>
<th>Energy (mJ m$^{-2}$)</th>
<th>$n$</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>80±20</td>
<td>67</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td>54±21</td>
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<td>3</td>
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<td>2</td>
</tr>
<tr>
<td>4</td>
<td>40</td>
<td>70±36</td>
<td>27</td>
</tr>
<tr>
<td>All</td>
<td>72±16</td>
<td>187</td>
<td></td>
</tr>
</tbody>
</table>

**TABLE I.** Adhesion energy derived from the unadhered length of the cantilevers. The error bars resulted from a statistical analysis and error propagation. The confidence interval of student’s t distribution is 95%.
lus of 164±10 GPa. The unadhered length varied within each structure and across the sample. All measurements of the adhesion energy of the s-shaped beams match within their error bars. The energy of adhesion does not exhibit a dependence on the width of the structure. The measurements obtained from the arc-shaped beams give significantly higher values. Because of the high uncertainty inherent in this method, this result is disregarded. Converging all measurements gives an adhesion energy of 72±16 mJ m$^{-2}$ for two InGaAs surfaces in contact. The shape of the collapsed cantilevers did not differ significantly from the s-shape. In accordance with the model which predicts a noticeable difference only below an adhesion energy of 25 mJ m$^{-2}$ the influence of the van der Waals forces on the shape could not be noticed at 72 mJ m$^{-2}$.

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