Experimental study of ultra-thin films mechanical integrity by combined nano-indentation and nano-acoustic emission

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Experimental study of ultra-thin films mechanical integrity by combined nano-indentation and nano-acoustic emission

by

Zihou Zhang

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in partial fulfillment of the requirements for the degree of

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ABSTRACT

Advancement of interconnect technology has imposed significant challenge on interface characterization and reliability for blurred interfaces between layers. There is a need for material properties and these miniaturized length scales and assessment of reliability; including the intrinsic film fracture toughness and the interfacial fracture toughness. The nano-meter range of film thicknesses currently employed, impose significant challenges on evaluating these physical quantities and thereby impose significant challenge on the design cycle.

In this study we attempted to use a combined nano-indentation and nano-acoustic emission to qualitatively and quantitatively characterize the failure modes in ultra-thin blanket films on Si substrates or stakes of different characteristics. We have performed and analyzed an exhaustive group of testes that cover many diverge combination of film-substrate combination, provided by both Intel and IBM. When the force-indentation depth curve shows excursion, a direct measure of the total energy release rate is estimated. The collected acoustic emission signal is then used to partition the total energy into two segments, one associated with the cohesive fracture toughness of the film and the other is for the adhesive fracture toughness of the interface. The acoustic emission signal is analyzed in both the time and frequency domain to achieve such energy division. In particular, the signal time domain analysis for signal skewness, time of arrival and total energy content are employed with the proper signal to noise ratio. In the frequency domain, an expansive group of acoustic emission signals are utilized to
construct the details of the power spectral density. A bank of band-pass filters are designed to sort the individual signals to those associated with adhesive interlayer cracking, cohesive channel cracking, or other system induced noise. The attenuation time and the energy content within each spectral frequency were the key elements for this sorting.

In the case wherein no excursion were present on the load-displacement curve, atypical case for adhesive failure only, the final indentation load along with the size of the blister were used to characterize the cohesive fracture energy. In the few cases wherein the film toughness was much higher than the substrate toughness, the unloading segment of the force-indentation depth curve showed a clear demarcation with a much lower slope. Such demarcation arises from the film buckling induced delamination. The delaminated buckled film unloads faster than the indentation process zone within the substrate and thereby become affected by the stored elastic strain energy within the film only. A simple model, utilizing the area under the force-indentation depth curve is derived to calculate the interfacial energy release rate of the film-substrate system. The model assessment is in good agreement with estimates done by four-point-bend testing on the same material system.

The developed experimental protocol may become useful in identifying the prominent failure mechanisms for quick screening of film-substrate, as well as in providing some quantitative measures of the adhesive and cohesive fracture toughness.
CHAPTER 1. INTRODUCTION

1.1. Motivation and Proposed Work

The interconnected layered structure is one of the basic forms of modern micro-electronic devices. The inspiration, that to make interconnected multiple devices on one single chip, of this application was raised even as early as the beginning of modern semiconductor industry, however did not finally come into reality until 1990’s, when fully integrated microelectronicmechanical system (MEMS) was brought by a technological revolution [1]. This technology is based on improved understanding of thin-film deposition and mechanical properties, and the core part is surface micromachining, which is grounded in the use of photolithography meanwhile provides repetitive processing steps, ultimately yields multi-layered interconnected structure [2]. Different layers of the structure can serve as different devices.

Mechanical properties and reliabilities of this structure have been concerned ever since this appearance [3-7]. There are five major failure modes, including ratcheting, voiding, electromigration, cohesive cracking, delamination (adhesive cracking) [4]. In this paper, only the latter two, which are defined as fracture of the structure, are featured. Cohesive cracking is that cracks initiate and propagate in the film only, and it only fractures the film, while does not influence interface and substrate or under layer. Delamination, or adhesive cracking, initiates and fractures interface of two layers. To study such behaviors can help industry to design more reliable devices.
There are a bunch of conventional experimental methods to measure and evaluate fracture of the structures based on fracture mechanics approaches, including superlayer test [9], double cantilever beam test [10], three or four point bending and scratching test [11-12]. However, these conventional techniques are limited by complicated sample preparation, difficulties in grips to match boundary conditions of corresponding models, instrumental resolution and stochastic variation at small length scale. Besides, few of above can simultaneously evaluate both of the fracture modes. Nanoindentation test has the advantages of no prior sample preparation, high localization and high resolution of appliances. However, conventional nanoindentation test still has limitations, such as limited applicable materials system, and inability of study interactions of both modes [13].

Acoustic emission (AE) monitoring is a process-kinetics sensitive technique. It has been successfully utilized in various testing fields [14-16]. By recognizing patterns of real-time AE signals, a possibility is provided that is to monitor occurrence and discrimination of different mechanical process [17]. It has no doubt that AE signals denoting cohesive cracks should have distinguished pattern from those representing adhesive cracks, due to different kinetics of these two process[18-19]. Hence, application of real-time AE monitoring technique in nanoindentation test has the potential of assisting us to evaluate fracture behaviors.

In this paper, we attempt to invent a novel fracture testing technique, featuring nanoindentation combined with acoustic emission (AE) monitoring technique, in order
to synchronously measuring both the fracture modes and their interactions. By testing on samples manufactured by Intel and IBM, relationships between material properties, such as elastic modulus and flow stress, and fracture modes are studied. Real-time acoustic emission signals captured during tests will be recognized and related to corresponding fracture modes. Hence, this technique can not only quantitively evaluate fracture behaviors, but also qualitively detect them underneath.

This thesis is set up by two parts. The first part is the overview of nanoindentation and necessary theories of fracture mechanics for this study, and application of both above to current tests. The second part is a review of acoustic emission monitoring technique, construction of AE signal pattern recognition method, and how it can be employed to current research. Conclusion and bibliography are provided at the end of the paper.

1.2. Introduction of Nanoindentation Technique

1.2.1 Nanoindentation

The idea of indentation was raised from hardness testing, by a simple thought that poking an unknown material with a material of given properties and comparing. After several decades of refinement, concept of hardness finally became the least pressure needed to produce a permanent impression at the center of contact area. By such definition, the most important parameters of indentation test should be the
indentation peak load $P_{\text{max}}$ and the contact area $A_c$, and therefore hardness as its definition is given by:

$$H = \frac{P_{\text{max}}}{A_c} \quad (1.1)$$

As is indicated by its name, different from common indentation, nanoindentation has a smaller length scale in nanometers, comparing to common indentation tests of milli or micrometers. This miniaturized length scale provides on one hand higher resolution for measuring, on the other hand difficulties in measuring squared micron-sized contact area by conventional optical methods. Hence, indentation contact area is replaced by a more easily manageable parameter, indentation depth, for this case. That is why nanoindentation is also called depth-sensing indentation [18]. Along with the development of this technique, nanoindentation test is not only limited in hardness testing, but also applied to testing other materials behaviors such as elasticity, plasticity [17, 19], viscoelasticity [20], stress-induced phase transformation [21] and fracture [22]. For such purposes, links between parameters derived directly from indentation like indentation load and depth, and contact area as well as further corresponding parameters should be established, as will be discussed below.
A typical load-displacement curve derived from indentation test. $P_{\text{max}}$ is the peak indentation load of the test, $h_{\text{max}}$ is the indentation depth at the peak indentation load. $h_f$ is the final displacement. $S$ is the stiffness of the tested material. Adapted from [23].

A typical load-displacement curve is shown in Figure 1.1. It is obvious that there is only elastic response happening during unloading. Hence, stiffness of the tested materials can be found by:

$$S = \frac{dP}{dh} = 2E_r r_c$$

(1.2)

Where, $S$ is the stiffness, $P$ is the indentation load and $h$ the indentation depth, $r_c$ is the radius of contact area, and reduced modulus $E_r$ is defined:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu'^2}{E'}$$

(1.3)

Where, $E$ and $E'$ are elastic moduli of tested material and indenter tip, $\nu$ and $\nu'$ are Poisson’s ratios of tested material and indenter tip, respectively. In our test, diamond is
used as the indenter tip, thus $E'$ is as high as \( \sim 1 \) TPa. For most of measured materials with modulus ranging \( 100 \) Mpa \( \sim 100 \) GPa, reduced modulus value is very close to material’s young’s modulus value.

Due to huge errors from conventional elastic models, in 1992, Oliver and Pharr [23] established a method to estimate contact area based on elastic-plastic response during unloading:

$$
P = B (h - h_f)^m
$$

(1.4)

Where, $B$ and $m$ can be found by power-law fitting unloading curve. At the peak load, combining functions (1.2) and (1.4) yields:

$$
S = \frac{dP}{dh} (P = P_{max}) = mB(h_{max} - h_f)^{m-1}
$$

(1.5)

The contact depth $h_c$, rather than either $h_{max}$ or $h_f$, is then given by:

$$
h_c = \varepsilon \frac{P_{max}}{S}
$$

(1.6)

Where, $\varepsilon$ is a geometry constant depending on the shape of indenter tip. For conical tip, $\varepsilon = \frac{2}{\pi} (\pi - 2) = 0.72$. Since radius of cross sectional area of tip is only dependent on geometry of tip and indentation depth, from the result of (1.6) $r_c$ and $A_c$ can be derived. Apply the value to (1.1) and (1.2), and one can finally find harness and elastic modulus of the tested material.

In general cases, it is impossible that only elastic response exists. More commonly, plasticity comes along with process of indentation. In the definition of
hardness, the term “permanent” usually relates to plasticity. This definition indicates a simple relationship between material’s hardness and yield strength, and under an indentation test, this hardness can be expressed by mean pressure \( P \).

Following experimental research approaches have provided useful and reliable results:

\[
H \approx C \sigma_y
\]  

(1.7)

Where, \( C \) is a constant varying according to \( E/\sigma_y \) ratios. When \( E/\sigma_y \) is high, \( C \approx 3 \); and if \( E/\sigma_y \) is low, \( C \approx 1.5 \). Relationships of \( P_m \), \( H \) and \( \sigma_y \) are listed in Table 1.1.

<table>
<thead>
<tr>
<th>Region</th>
<th>Response</th>
</tr>
</thead>
<tbody>
<tr>
<td>( P_m &lt; 1.1\sigma_y )</td>
<td>Elastic response</td>
</tr>
<tr>
<td>( 1.1\sigma_y &lt; P_m &lt; H )</td>
<td>Plastic deformation exists but is constrained by surrounding elastic materials. Ratio ( C = H/\sigma_y ) is constant with respect to impressed material and tip geometry</td>
</tr>
<tr>
<td>( P_m \geq H )</td>
<td>Material yields. Plastic region grows, and is independent of increase of ( P_m )</td>
</tr>
</tbody>
</table>

Table 1.1

**1.2.2 Fracture Mechanics of Indentation on Film-Substrate Structure**

It has been proven that simply applying conventional designing principle, which is critical stress-based, is unreliable. Thus we need a substitutive and more convincing principle. The failure of previous theory is due to imperfection of materials. Since this imperfection can be never avoided, the new theory must start with considering defects. If we model the defected materials as following: the body is a linear elastic continuum and there is a crack with matching surfaces and a front tip inside the body, it is obvious that
only if this crack elongates, or propagates, the materials fails, or it is safe and stable. Considering the physical process of crack propagation, it can be simply described as generation of new surfaces and simultaneous reduce of energy stored inside the body. If we focus on the relationship between changes of energy stored and crack surface area, a parameter should be developed:

\[
G = -\frac{\partial U}{\partial A}
\]  

(1.8)

Where \(U\) is the elastic energy stored inside the body and \(A\) is the crack surface area. \(G\) is called “energy release rate”, as is easily understood by its definition. If \(G\) is greater than certain critical value, \(G_{cr}\), or \(\Gamma\), which is called fracture energy, the crack propagates, or the crack will stay stable. It is easily seen that fracture toughness is a materials constant directly related to strength of atomic bonding while independent of boundary conditions, thus what we care is how to calculate energy release rate.

(a)
1) Cohesive Cracking Model

When applying this fracture mechanics theory to indentation on films, according to result given by Jungk et.al [22], the developed model should be considered as two types: mechanical thick and mechanical thin (Figure 1.2). If indentation depth is less than 10% of film thickness, we consider it mechanical thick, otherwise it should be mechanical thin. Energy release rate of cohesive cracks under indentation can be presented as:

\[
G_c^i = \begin{cases} 
\left(\frac{2k}{\eta}\right)^2 \frac{(P/c)^2}{(cH_f)} & \text{mechanical thick} \\
(V^*)^2 \left(\frac{\lambda}{\eta}\right)^2 \left(\frac{1}{E_f H_f^2}\right)^{1/3} \left(\frac{1}{t_f}\right)^2 \left(\frac{p^2}{c}\right) & \text{mechanical thin} 
\end{cases}
\] (1.9)
Where, $\lambda$ is a constant related to Poisson’s ratio and indentation tip geometry, and is approximately 0.013 for Vicker’s indenter and 0.016 for cube-corner indenter. $\xi$ is a geometric parameter derived from experiments, and is equal to 0.038 for cube-corner tip [24]. Tip shape constant $\eta$ is defined by:

$$
\frac{P}{H_f} = A_c = \eta r_c^2
$$

(1.10)

And for both Vicker’s and cube-corner geometry, $\eta$ is approximately 3.53. $E_f$, $H_f$ are the modulus and hardness of the film, respectively, which can be directly derived by (1.1) and (1.2). $t_f$ is film thickness, and $c$ is the total crack length. $V^*$ is a parameter that characterizes plastic affect, which is given by

$$
V^* = \begin{cases} 
1 & \text{if } h_p \leq t_f \\
1 - \frac{(h_f - t_f)^3}{h_f^3} & \text{if } h_p > t_f
\end{cases}
$$

(1.11)

Figure 1.3 Scheme of a film-substrate structure with one cohesive crack only in the structure. $t_f$ is the film thickness, $\sigma_R$ is the residual stress throughout the film. Adapted from [25].
For deposition of film, residual stress cannot be avoided as a byproduct. Apart from indentation-induced stress, residual stress can as well be a resource of driving force for crack propagation. Considering a single layer film bonded by a substrate, a channel crack runs across the thickness of the film, and no additional damage to either substrate or interface. Since crack tip is constrained by substrate that bonds so that the problem is simple plain-strain, change of elastic energy is scaled by film thickness and crack area, namely:

$$\Delta U \sim -\frac{\sigma^2 c t_f^2}{E_f}$$  \hspace{1cm} (1.12)

Hence, the energy release rate is given by:

$$G_c^R = \frac{\partial U}{\partial A} \sim \frac{\sigma^2 t_f}{E_f}$$  \hspace{1cm} (1.13)

And this expression indicates that energy release rate is only related to film thickness geometrically, thus the thinner film is, the more difficult it cracks. Generally, energy release rate under this condition is [26]:

$$G_c^R = Z(\alpha, \beta) \frac{\sigma^2 t_f}{E_f}$$  \hspace{1cm} (1.14)

Where $Z$ is a dimensionless number and depends on Dundur’s parameters:

$$\alpha = \frac{E_f - E_s}{E_f + E_s}$$

$$\beta = \frac{E_f (1 - \nu_f)(1 - 2\nu_s) - E_s (1 - \nu_f)(1 - 2\nu_s)}{2(1 - \nu_f)(1 - \nu_s)(E_f + E_s)}$$  \hspace{1cm} (1.15)
And they represent materials mismatch between film and substrate. When mismatch does not exist, e.g., Dundur’s parameters $\alpha = \beta = 0$, $Z = 2$ or more exactly, 1.976. $Z$ value decreases a little bit if the structure is a soft film on hard substrate, meaning $\alpha < 0$. When substrate is compliant than film, $Z$ value increases rapidly with respect to increase of $\alpha$. For example, when $\alpha > 0.99$, $Z > 30$. Effect of another parameter $\beta$ is secondary and is often ignored [25]. At last, energy release rate of cohesive cracking yields:

$$\sqrt{G_c} = \sqrt{G_c^l} + \sqrt{G_c^R}$$

(1.16)

2) Adhesive Cracking Models

Figure 1.4 Scheme of film-substrate system under indentation when (a) indentation without buckling, (b) indentation with buckling and (c) film buckling after removal of indenter. $V_i$ is the indentation volume, $t_f$ the film thickness and $\alpha$ the length of adhesive cracking. Adapted from [13]
For adhesive cracks, the most commonly used model (Figure 1.4) is developed by Marshall and Evans [27]:

\[
\frac{G_a E_f}{1 - v_f} = \frac{1}{2} t_f \sigma_f^2 (1 + v_f) + (1 - \alpha_1) h \sigma_h^2
\]

\[-(1 - \alpha_1) t_f (\sigma_i - \sigma_B)^2\]  

(1.17)

Where, \(v_f\) is the Poisson’s ratio of the film, parameter \(\alpha\) represents buckling effect, which is given by

\[
\alpha_1 = \begin{cases} 
0 & \sigma_i + \sigma_R \leq \sigma_B \\
1 - \frac{1}{1 + 0.902(1 - v_f)} & \sigma_i + \sigma_R > \sigma_B 
\end{cases}
\]  

(1.18)

The indentation stress \(\sigma_i\) is calculated by

\[
\sigma_i = \frac{V_i E_f}{2 \pi t_f a^2 (1 - v_f)}
\]  

(1.19)

\(a^2\) is the square of adhesive crack radius, \(V_i\) is half of the indentation volume, which is given by integrating tip area function from the machine. The Euler buckling stress \(\sigma_B\) is defined as

\[
\sigma_B = \frac{\mu t_f^2 E_f}{12 a^2 (1 - v_f)}
\]  

(1.20)

Where, \(\mu\) is a constant equal to 14.68. Using these equations, we can determine energy release rate. The problem of this model is, it neglects the influences from substrate. One can tell from the expression from the model, that there is no consideration of materials mismatch in system. It also assumes that what indentation brings in is of elastic
responses. Buckling is an issue but still under the regime of elasticity. Also, lack of consideration of indentation process is unreliability from another point of view. Hence, this model can be only used for brittle film materials with relatively lower interfacial fracture toughness.

![Diagram](image)

**Figure 1.5** A schematic load-displacement curve got from indentation on highly buckled film. There is a sudden change of slope when unloading. $U$ is the work done by film to indenter tip, $P$ and $h$ are defined as previous. Adapted from Wei Hong, personal communication in 2011.

Another approach considers plasticity and indentation load-displacement response. Figure 1.5 plots an indentation test on film with large buckling. There is a clear linear section with discontinuous slope on the unloading. The area under, however, is the work done by the film to the indenter during unloading. Because the unloading is a reversible process, this work should be equal to elastic energy released in the film due to indentation. Neglecting effect of residual stress which is considered small comparing to that caused by indentation, let us consider this work.
At the maximum load and displacement, delamination stops at certain area. The energy release rate of this point should be equal to fracture toughness, or debonding will continue. Assume that the debonding area, with the radius of \( a \), has a circular insertion of eigenstrain of \( \epsilon^* \) in the middle. The insertion is related to plastic zone of axisymmetric indentation, and its radius is \( b \). Analytical solution of this problem is, in term of displacement:

\[
u = \frac{1 + \epsilon^*}{1 - \frac{a^2}{b^2}} \left( r - \frac{a^2}{r} \right) \tag{1.21}
\]

By this expression, the corresponding strains can be represented as:

\[
\begin{align*}
\epsilon_r &= \frac{1 + \epsilon^*}{1 - \frac{a^2}{b^2}} \left( 1 + \frac{a^2}{r^2} \right) \\
\epsilon_\theta &= \frac{1 + \epsilon^*}{1 - \frac{a^2}{b^2}} \left( 1 - \frac{a^2}{r^2} \right) \tag{1.22}
\end{align*}
\]

Where, \( \epsilon_r \) is the radial strain and \( \epsilon_\theta \) is the tangential strain. Then the strain energy density will be derived:

\[
W = \frac{1}{2} \frac{E}{1 - \nu^2} \left( \epsilon_r^2 + \epsilon_\theta^2 + 2\nu \epsilon_r \epsilon_\theta \right) \\
= \frac{E}{1 - \nu^2} \left( 1 + \frac{\epsilon^*}{1 - \frac{a^2}{b^2}} \right)^2 \left( 1 + \nu \right) \left( 1 - \nu \right) \left( \frac{a^4}{r^4} \right) \tag{1.23}
\]
Integrating this density over the annular region, the elastic strain energy is:

\[
U = \frac{\pi Eh}{1 - \nu^2} \left( \frac{1 + \epsilon^*}{\left(1 - \frac{a^2}{b^2}\right)} \right)^2 \left[ (1 + \nu)(a^2 - b^2) + a^2(1 - \nu)\left(\frac{a^2}{b^2} - 1\right) \right]
\]

\[
= \frac{\pi Eh}{1 - \nu^2} \frac{(1 + \epsilon^*)^2}{a^2 - b^2} \frac{[(1 + \nu)b^2 + (1 - \nu)a^2]}{1} \quad (1.24)
\]

\[
= \frac{\pi Eh b^2}{1 - \nu^2} (1 + \epsilon^*)^2 \left( \frac{2b^2}{a^2 - b^2} + 1 - \nu \right)
\]

Note that debonding area \( A = \pi a^2 \) and indentation plastic zone \( A_p = \pi b^2 \), such expression can be rewritten as:

\[
U = \frac{EhA_p}{1 - \nu^2} (1 + \epsilon^*)^2 \left( \frac{2A_p}{A - A_p} + 1 - \nu \right) \quad (1.25)
\]

And the energy release rate is:

\[
G_a = -\frac{\partial U}{\partial A} = \frac{EhA_p}{1 - \nu^2} (1 + \epsilon^*)^2 \frac{2A_p}{(A - A_p)^2}
\]

\[
= U \frac{2A_p}{(A - A_p)[(1 - \nu)A + (1 + \nu)A_p]} \quad (1.26)
\]
1.2.3 Equivalent Tip and Plastic Zone

![Schematic equivalence of pyramidal indenter tip (left) and conical tip (right). Results [28] show that the tips are equivalent when conical one inscribes the pyramidal tip. $\Theta$ and $\Psi$ are indenter angles, respectively. Adapted from [28].](image)

Both the adhesive fracture models share the assumption that the indenter tip is asymmetric. However in our test, a cube-corner tip is used. Hence there is a need to find an equivalent asymmetric, or conical tip, for our test to apply models and find plastic zone. According to Qin et al [28], base area of a pyramidal indenter (Berkovich and cube-corner etc.) should be equivalent to that of an axisymmetric indenter whose indenter angle equals to angle of this pyramidal indenter (Figure 1.6). Namely, cube-corner tip is equivalent to a 35.3 angled axisymmetric tip. That means, indentation area using cube-corner indenter should be approximately 1.654 times that using equivalent axisymmetric tip.

According to Kramer et al [29], radius of plastic zone area and radius of axisymmetric indenter area have a relationship as following:
\[ \zeta = \frac{b}{r_i} = \begin{cases} 
3 & \text{for } \frac{r_i}{t_f} \leq \frac{1}{3} \\
1 + C_1 \left( \frac{r_i}{t_f} \right)^n & \text{for } \frac{r_i}{t_f} > \frac{1}{3} 
\end{cases} \] (1.27)

Where, \( r_i \) is the radius of the indentation area; \( C_1 \) and \( n \) are constants which equal to 1.27 and -0.5 respectively. Thus, if we have the indenter tip’s depth-area relationship function \( A(h) \), the plastic zone area will be:

\[ A_p = \frac{\zeta^2 A(h)}{2.736} \] (1.28)

1.2.4 Role of Excursions

During indentation, when there is a sudden large increase of compliance of the system, a pop-in will occur on the load-displacement curve, which is also referred as an “excursion” (Figure 1.9(a)). There are a lot of resources that can result in the increase of compliance, including plasticity [17, 30] fracture [22, 31] and other sources. As the compliance increases, as a response under the same load, deformation is larger, and the indenter tip sinks in the system deeper. Therefore, the area under an excursion should be the work done by indenter tip to the system. In our experiments, we hardly observe evidence of phase transformation. This work done is finally transformed to elastic as well as plastic deformation energy, cohesive cracking energy and adhesive cracking energy, namely:

\[ \delta U_{sys} = W_{ex} = \delta U_p + \delta U_e - \delta U_c - \delta U_a \] (1.29)
Where, \( \delta U_{sys} \) is the change in energy of the system, \( W_{ex} \) is area under excursion, which equals to work done by indenter tip, \( \delta U_c \) is the energy change by cohesive cracking and \( \delta U_a \) is the energy change by adhesive cracking. If we do a little transformation for (1.29):

\[
W_{ex} \left( 1 - \frac{\delta U_p + \delta U_c}{W_{ex}} \right) = \gamma W_{ex} = -(\delta U_c + \delta U_a)
\]

(1.30)

Where, \( \gamma \), which denotes non-fracture portion of work done, is approximately 0.15 for some cases. However, this ratio is not a universal constant. It varies according to mechanical propertied of the system. For example, for ductile samples, this ratio should be lower than that for brittle and high-stiffness samples.

According to the definition of energy release rate and fracture energy, energy release rate and fracture energy are equal when crack propagation stops under load. Note that in our case, fracture is relatively small which is in micro-scale, which yields:

\[
\Gamma = G_{cr} = -\frac{\partial U}{\partial A} \approx -\frac{\delta U}{A}
\]

(1.31)

Combining (1.30) and (1.31), the relationship between fracture and excursions is expressed:

\[
\gamma W_{ex} = -(\delta U_c + \delta U_a) = \Gamma_c A_c + \Gamma_a A_a
\]

(1.32)

Where, \( \Gamma_c \) and \( \Gamma_a \) are cohesive and adhesive fracture energy, respectively, \( A_c \) and \( A_a \) are areas of cohesive and adhesive crack, respectively, which can be measured directly from tests.
1.3 Acoustic Emission Technique on Film Fracture Analysis

When a physical process involving sudden release of elastic energy appears in a system, elastic waves generated and emitting can be detected and measured by acoustic emission (AE) monitoring. The resource of the process can be fracture, stress-induced phase transformations as well as plastic instabilities such as acceleration of existing dislocation and initiation of new slip system. Both theoretical and experimental approaches prove that energy of AE signals has a linear relationship with energy generated by corresponding physical process. Namely,

\[ \sum AE_{process} = \alpha_{AE} E_{process} \]  

(1.33)

Where, \( \sum AE_{process} \) is the sum of measured relative acoustic energy, \( E_{process} \) the energy of corresponding process and \( \alpha_{AE} \) a scaling coefficient that varies with respect to systems and physical processes [32].

AE monitoring is also sensitive to process kinetics, which allows discriminating different types of processes. Hence this technique can be used for in-situ evaluation of specific mechanical processes. The signals captured by AE monitoring can provide following information about the process: 1) time when the process occurs; 2) energy level of the process and 3) type of the process.

Conventional AE monitoring involves surface detection provided by large sensor attached to the surface, which was used for to evaluate certain processes during indentation and scratching tests on a variety of materials. However, this technique has
some defects that limit its applications. First of all, the energy level capable to detect is relatively high. Previous studies have proved that the technique is applicable to evaluate transition from elastic to elastic-plastic deformation by a measured linear relationship between AE energy and work of plasticity induced excursions [16]. Except for signals at the beginning of such excursions, no other signal is detected, which indicates process involved with lower energy changing, namely dislocation nucleation, are incapable to capture. Other processes with similar energy level, such as oxide film fracture, is neither capable to detect. In fact, the technique is impotent for process occurring under 1mN. Additionally, effect of AE detection is highly influenced by multiple wave reflections, and it leads to the dependence on size. Finally, types of signals will be affected by shape of indenter tip [16, 33]. Due to these defects, this approach is not applicable to micro- and nano-scaled testing, especially tests on small-scale systems such as thin films and interface. Therefore in this paper, novel nano-AE monitoring is involved. Nano-AE monitoring relies on the sensor attached directly to local indentation contact by coupling with indenter tip, which yields higher sensitivity to localization and synchronicity. Due to such, it is possible to utilize the technique in measuring physical processes that happens under 1mN.

An AE event is detected and recorded as a signal when amplitude of its waveform generated by the sensor is above noise floor (NF) and triggers the recorder (Figure 1.7). The system noise floor is usually measured when there is no desired test being operated with the appliance, and it denotes the components of surroundings only
without response of sensor. NF is scaled by the unit of sound intensity, dB. In our system, NF is calculated by:

\[
NF = 60\, dB + 20 \log \left( \frac{x_{\mu V}}{1.0\mu V} \right)
\]  

(1.34)

Where, the term 60dB corresponds input that is inevitable. Gain voltage \( x \) is detected by the sensor, and varies under different surroundings.

![Figure 1.7 A typical AE event detected at trigger level of 69 dB. Both the axis is in arbitrary unit (AU).](image)

Each AE signal is recorded in the time length of 1\( \mu \)s, at sampling rate of 10MHz. Frequency response is from 100kHz to 2000kHz. Delay time, meaning sum of data points before the trigger one is 50 as default. The noise floor is 69dB. We assumed that during the experiment happens only fracture process, including both cohesive and adhesive fracture. Energy integrated from AE signals relating to delamination should have linear relationship with work of adhesion, and similar calculation is valid in cohesive cracking case. Due to the same system that all signals are detected in one test, coefficient \( \alpha_{AE} \) is a constant for each test. Namely, a relationship is achieved:
Where, $\delta U_c$ and $\delta U_a$ are of the same definitions in (1.18). Therefore, if all corresponding signals are detected and recognized, fracture energy is possible to be evaluated by combining (1.23) and (1.19):

$$
\Gamma_c = \frac{\gamma W_{ex}}{A_c} \times \frac{\sum AE_c}{\sum AE_c + \sum AE_c}
$$

$$
\Gamma_a = \frac{\gamma W_{ex}}{A_a} \times \frac{\sum AE_a}{\sum AE_a + \sum AE_a}
$$

Signals resulting from delamination might have the sound intensity up to 3 or 4 orders lower than that resulting from cohesive cracking, depending on structure components. In our experiment, cohesive cracking signals can be detected up to 2-3 orders higher than the noise floor (Figure 1.8). If we lowered the trigger and keep it above noise floor, no additional signal appears. This leads to a situation that adhesive cracking signals might be detected around the noise floor, and could be submerged by systematic background noises, which result from air flow, transient electronic jump or friction between tip and specimen [34]. Hence it is difficult if one wants to tell the difference of a debonding signal from background noise signals simply by sound intensity characterization. Unlike cohesive signals (Figure 1.7), adhesive signals cannot either differentiate themselves from background noises by the shape of waveforms. Such that pattern recognition work need to be done.
Figure 1.8 AE signals characterization at trigger levels of (a) 69dB (b) 70dB and (c) 71dB. Equivalent cohesive signals (green dots) in square appear in similar positions of load-displacement curves (red) from same experiment set. Unlike these cohesive AE signals, other signals including adhesive AE signals and noise do not survive when increasing the trigger level.
CHAPTER 2. METHODOLOGY

2.1 Experimental Setup

a. Equipment

The nanoindenter used for the experiments is Hysitron© TriboIndenter T950 and the controlling interface is TriboScan© v9.8. The nanoindenter provides several functions as follow: 1) Indentation and load-displacement curve plotting; 2) In-situ surface scan; 3) Acoustic Emission (AE) monitoring; 4) Automatic calculation of modulus and hardness. These functions are all utilized throughout whole experimental procedure. Surface scanning images are read and analyzed with TriboView©.

b. Setup and Testing Procedure

Cube-corner shaped indentation tip combining an AE monitoring transducer is chosen to perform the whole experiment (Figure 2.1). Samples, manufactured respectively by IBM and Intel, are mounted firmly in the machine, and indented 5-16 times under different final loads on the surface. We used open-loop indentation under load-control mode. Loading and unloading rate were fixed (~ 500 µN/s), between which there is a 20-second holding of load, to allow delayed response such as creep and plasticity. Each sample is indented up to a depth equals to specific times of (top) film thickness. According to our previous research (Figure 2.2 [36]), usually the maximum indentation depth for a sample is 1 or 3 times thickness, to avoid excessive yielding zone area from covering delamination area. However for the sample that is thinner than 30 nm,
the depth can be 3-4 times, due to precision of the machine. Along with the indentation, AE signals were synchronously captured at the noise floor (trigger level) of 69dB. After each indentation, a load-displacement curve along with AE signals is automatically generated (Figure 2.3 (a)). In the meantime, a specific local area (according to size of an indentation) of the sample surface would be scanned both in topology and in gradient, in order to determine cracking behavior under indentation (Figure 2.3 (b)).
Figure 2.1 (a) SEM Micro-graph of a cube-corner indentation tip. Ideally tip bluntness is less than 100nm. Adapted from [35]. (b) Setup of experiments

Figure 2.2 FEM result of area of large yielding (grey area) compared to delamination crack (white bar) with respect to indentation depth. It is cleared that if indentation reaches over 3 times of film thickness, one cannot observe delamination from surface. Adapted from [36]
Figure 2.3 (a) Nanoindentation load-displacement curve. Each figure features AE signals (green dots) and its real-time position, as well as excursions, as shown inside squares (b) Surface scanning images: topology (left) and gradient (right)
Figure 2.4 Schematic determinations of crack lengths. Cohesive crack length is the mean value of each length of the crack, rooting from corner of impression to the crack tip, namely \( c = \frac{c_1 + c_2 + c_3}{3} \). Adhesive crack length is given by the average value of the measured crack lengths along three directions perpendicular to impression edge. Along each direction, the measured horizontal value \( a \) should be from horizontal position of the lowest point, to that of the first point whose altitude is below 1 nm due to resolution of scanning and surface roughness.
2.2 Measurement of Hardness

Only when elastic modulus measured is close to the standard value, hardness that is simultaneously measured is trustable. However, using Oliver-Pharr model, mechanical properties measured of film are influenced greatly by those of its substrate, because main assumption of this model is that indentation depth is ignorable comparing to the whole thickness of sample. Previous researches [19] show that only when \( h_{\text{max}} / t_f \leq 0.1 \) precise results can be acquired. For most of our samples whose thickness is equal or less than 100nm, direct applying Oliver-Pharr model for modulus and hardness measurement is impossible due to noise floor of the machine (~30nm-50nm). Therefore there are only two ways to get precise measurement. The first one is to find the portion of unloading curve that represents unloading in film (some of fittings). The second, due to default fitting criterion of the machine that is based on percentage of final loads, measurement should be based on a limit of \( h_{\text{max}} / t_f \leq 2 \), especially if \( E_f \) is much smaller than \( E_s \) (Figure 2.5).
Figure 2.5 Example of measurement of elastic modulus and hardness by fitting part of the curve. The fitted part should have the depth smaller than film thickness, and the final indentation depth should be smaller than two times the film thickness. The fitted sample is #007, whose Young’s modulus is 15.6 GPa, equivalent to reduced modulus of 14.78 GPa. The measured reduced modulus is 15.02 GPa.
2.3 AE Signal Pattern Recognition

Cohesive AE signals are always of high sound intensity, and adhesive AE signals have sound intensity as high as that of background noises. Therefore in the real test, it cannot be avoided that to capture AE signals denoting interfacial delamination, a lot of background noises must be as well recorded. AE signals related to cohesive cracks are so easy to find. Either through previous research [16, 33], simultaneous appearance with excursions or huge acoustic energy involved can identify this type of signals at the first stage. This situation leads us a challenge that to separate gold from sand, a characterization method must be developed. Through this method, all signals will be divided into three types: cohesive AE signals, adhesive AE signals and background noises.

2.3.1 Time-Domain Signal Analysis

The AE signal is analyzed first in the time domain to identify the signals related to adhesive, cohesive and background noise. We utilized the skewness of each AE signal to sort the cracks and non-crack signals. According to Scala et al [35], an AE event corresponding to a fracture events is reflected with a skewness; \( \alpha_3 < 0 \). The signal skewness is defined as:

\[
\alpha_3 = \frac{m_3}{m_2^{3/2}}
\]  

(2.1)

Where,
reflects skewness of a signal voltage \( v \) according to time \( t \). A typical data set is shown in Figure 2.6 (a) for ULK 2.4 of 500 nm film. All the data below the Zero datum are possible candidate to correlate to fracture events.

The second step in the analysis is the time of travel of different types of waves, wherein the shear waver emanating from a delamination is the slowest and the longitudinal wave emanating from a cohesive channel crack is the fastest (Figure 2.6 (b)). We utilized the characteristic time, \( \tilde{t} \), wherein most of the signal energy is received by the AE transducer. \( \tilde{t} \) is defined as the first moment of the energy within 3-30 s window, covering the initial signal rise time with respect to time,

\[
\tilde{t} = \frac{\sum_{i=30}^{300} t(i) v^2(i)}{\sum_{i=30}^{300} v^2(i)}
\]

Distribution of \( \tilde{t} \) for a surviving set of the AE events is shown in Figure 2.6 (c). It is clear that there is a cluster of lower values that should correspond to cohesive crack event, as well as another cluster of higher values that correspond to adhesive crack event. The remaining intermediate time of travel may correspond to the background noise. To identify the cut off level as marked on Figure 2.6 (c) bottom, we utilized the probability
density distribution (PDD) to identify these cut-off, as shown in Figure 2.6 (c) top. The cutoff is defined as 65% of the characteristic time level that correspond to the peak of the PDD as the left boundary, and 5% above the peak of the PDD as the right. With this second step of the filter we can identify the adhesive, cohesive and the background signals.

The third step of the analysis will further identify those signals with high energy level above the mean, and thereby clearing the ambiguity of the low signal to noise ratio. The filter comprised of the total level of energy, $E_{\text{signal}}$ received by the transducer within the rise-time, 2.5-7.5 s window. It is defined by

$$E_{\text{signal}}(t) = \sum_{i=1}^{n} (t(i) - t(i - 1)) \times v^2(i)$$ (2.4)

The distribution of the energy levels of the surviving events from the second filter step is shown in Figure 2.6 (d) bottom. Constructing the PDD will separate the background noise from the adhesive and cohesive signals, as shown in Figure 2.6 (d) top. We identified the first cut-off level at 10% above the energy level that correspond to the peak of the PDD. The second cutoff energy level commence with the first $PDD(0)$ after the peak and identify the demarcation between adhesive and cohesive signals. The two demarcation lines in Figure 2.6 (d) correspond to these two limits.
Figure 2.6 (a) Schematic cutoff by skewness. (b) Attenuation characterization differences of cohesive (top) and adhesive (bottom) AE signals. Adhesive AE signals have significant longer attenuation time. (b) Time-domain signal analysis by characteristic time $PDD$ (top) and result (bottom). Threshold of adhesive signals from cluster is confirmed by 1.1 times the time value of peak probability density. (c) Further classification by signal energy. Similar to (b), $PDD$ (top) and result (bottom) are schemed.
As the figure indicates, noises and adhesive signals cluster around certain level of energy as well as characteristic time (all signals in (c) are the results from (b)). This situation calls for a further understanding to differentiate one type to another.

2.3.2 Frequency-Domain Analysis

In this section, power spectral density (PSD) will be used to find the nature of frequency structures of the AE signals. Based on PSDs, band pass filters (BPFs) will be constructed to describe signal patterns by making the signals go to and forth each filter.

(1) Nature of Noises

To understand the main noise resource, intrinsic vibration of the machine, indentation tests on air are done and 272 AE signals generated are collected for frequency-domain analysis. PSD is plotted by calculating averaged squared magnitudes of FFT results of signals (Peter Sherman, personal communication, 2012).

\[
\text{PSD} = 10 \times \log \tilde{A} \quad (1:512) \quad (2.5)
\]

Where,

\[
\begin{cases}
Aemat^i = \frac{[FFT(v^i)]^2}{1024} \\
\tilde{A} = \frac{\sum_{i=1}^{cn} Aemat^i}{cn}
\end{cases} \quad (2.6)
\]

\(i\) refers to signal count number, and is scaled by \(10^{-7}\) s, and \(cn\) the total number of the signals. Number 1024 is used because of the sampling rate (10MHz) and time length
(0.1024 ms) of a signal. The PSD is in dB, and is symmetric through the whole normalized frequency range. Hence only half of the PSD is picked, and frequency is normalized by original frequency over 10MHz, as plotted in Figure 2.7 (b).
PDF of Relative Pwr @ Ctr. Fs:.015/.03/.05/.07/.09/.11/.13/.15/.17/.19

Relative amount of power in the given bw
Figure 2.7 (a) examples of AE noise signals captured during air indentation. (b) PSD (vertical, in dB) of AE noise signals with respect to normalized frequency (original frequency over 10 MHz). Calculation based on 272 air indentation AE noise signals. (c) Power distributions with respect to frequency ranges. (d) Signal simulations by frequency range [0, 1.1 MHz] (red) and origin signals (blue). For each signal waveform, horizontal axis is scaled in $10^{-7}$ s and vertical in mV.

Several peaks are observed throughout the whole frequency range (Figure 2.7 (b)). Frequency ranges higher than ~ 1.1 MHz filter only ignorable portions of wave energies (Figure 2.7 (c)). Simulation of waveforms by band pass filter [0, 1.1 MHz]
covers most of details of a signal (Figure 3.4 (d)). Therefore, peaks in 0-1.1MHz should be under scrutiny, especially 6 peaks at the frequencies of 390 kHz, 520 kHz, 650 kHz, 780 kHz, 910 kHz and 1040 kHz. It is noticed that both absolute amplitude (~ 65dB) and local amplitude (~ 13dB) of the peak at 390 kHz are the highest. In the meantime, frequency band [300 kHz, 500kHz] filters highest energy portion of the whole wave (Figure 2.7 (c)). Considering that other resources, such as air flow, contribute limited to noise nature comparing to mechanical intrinsic vibration, it is clear that frequency of 390 kHz is the intrinsic frequency of the machine. Therefore, the rest 5 peaks might characterize useful signals, e.g., cohesive and adhesive fracture AE signals.

(2) Wave Structure of Cohesive Fracture Signals

a. Construction of Band Pass Filter

Using time-domain analysis, cohesive signals can be easily identified. Note that the natures of waves caused by cohesive fracture and adhesive fracture are different. Waves generated by cohesive fracture and dispersing are longitudinal waves which have higher characteristic frequency and wave speed, and waves generated by adhesive fracture are shear waves with lower characteristic frequency and wave speed. Theoretically speaking, for a homogeneous, isotropic and condensed material, characteristic frequency ratio between shear wave over longitudinal wave is the square root of the ratio between shear modulus over elastic modulus of the material ($\frac{G}{E}$). Considering the relationship between the two moduli, the value is actually $\frac{1}{\sqrt{2(1 + \nu)}}$, where $\nu$ is the Poisson’s ratio. For a silicon compound, this value is around 0.65. Namely,
if the characteristic frequency of longitudinal wave is 1 MHz, that of shear wave should around 650 kHz. Therefore, to find characteristic frequency of cohesive fracture signals will be a major clue for finding that of adhesive fracture signals.
Figure 2.8 (a) PSD of cohesive fracture signals from tests on sample ULK2.4 (black) and that of noises (red, same as Figure 3.4 (b)). (b) Power distribution with respect to frequency ranges of these cohesive fracture signals.
The PSD of cohesive fracture signals captured from tests on the sample ULK2.4 is plotted (Figure 2.8 (a)). Though the PSD amplitude of cohesive fracture signals is relatively high comparing to that of noise throughout the whole frequency range, it is still obvious that in the frequency range [900 kHz, 1.2 MHz], both PSD amplitude as well as wave structure are extremely unique. Power distribution (Figure 2.8 (b)) also shows apart from machine’s intrinsic frequency (390 kHz), energy filtered also has a large portion in the ranges [900 kHz, 1.1 MHz] and [1.1 MHz, 1.3 MHz]. These evidences indicate the characteristic frequency should lie in the range [900 kHz, 1200 MHz].

(a)
Figure 2.9 Red lines are filtered waveforms and blue lines are original waveforms. Horizontal axis is time, scaled with $10^{-7}$ s and vertical axis is voltage in mV. (a) simulation of waveform of a cohesive fracture signal by frequency range $[0, 1.1 \text{MHz}]$. (b) A noise (top) and a cohesive fracture signal (bottom) passing through the band pass filter $[800 \text{ kHz}, 1 \text{ MHz}]$. (c) A cohesive fracture signal passing through band filters (from top to bottom): $[0, 200 \text{ kHz}]$, $[200 \text{ kHz}, 400 \text{ kHz}]$, $[400 \text{ kHz}, 600 \text{ kHz}]$, $[600 \text{ kHz}, 800 \text{ kHz}]$ and $[800 \text{ kHz}, 1 \text{ MHz}]$. For each signal waveform, horizontal axis is scaled in $10^{-7}$ s and vertical in mV.

Simulation of cohesive fracture signals given by frequency range $[0, 1.1 \text{ MHz}]$ covers as well most of the details of cohesive fracture signals (Figure 2.9 (a)). This range is divided evenly into 5 different band pass filters, and cohesive signals are made to pass through these filters (Figure 2.9 (c)). It is observed that when passing through $[800 \text{ kHz}, 1 \text{ MHz}]$, filtered waveform has a huge envelope at the beginning of the signal, comparing to the result by other filters. On the other hand, this huge envelope is unique to this type of signal – no other signal has it (Figure 2.9 (b)). Meanwhile, the filtered waveform shows a quick and hugh attenuation (ratio of integrated energy over first 22.5 µs and over 22.5-45 µs). Hence, this filter alone can characterize cohesive signals.
b. Pattern Recognition Based on Band Pass Filter

As plotted in Figure 2.9 (b) and (c), when passing through the filter [800 kHz, 1 MHz], a cohesive fracture signal has a big waveform envelop within the window (0, 30 µs), therefore the first filter is constructed based on total level of the energy of filtered wave, $E_{\text{filtered}}$ within the window:

$$E_{\text{filtered}}(t) = \sum_{i=1}^{n} v_{i,\text{filtered}}^2 (t) \times [t(i) - t(i - 1)]$$  \hspace{1cm} (2.7)

Where, $v_{\text{filtered}}(t)$ is the filtered waveform function with respect to time. We define the threshold as the average energy value plus 3 times the standard deviation of the signals in each single test (Figure 2.10).

The second filter is constructed based on the fact that the filtered energy attenuation from the window 0-22.5 µs to the following window 22.5-45 µs of the cohesive fracture signals is as well far from the average level. We scale this attenuation by a ratio, $R_{CO}$, between energies integrated in such two windows:

$$R_{CO} = \frac{E_{\text{filtered}}(22.5 \, \mu s)}{E_{\text{filtered}}(45 \, \mu s) - E_{\text{filtered}}(22.5 \, \mu s)}$$  \hspace{1cm} (2.8)

The cut-off boundary is defined as average value plus 1.5 times the standard deviation of $R_{CO}$ values of all signals in one single test. Schematic process of these two filters is plotted in Figure 2.10.
Figure 2.10 Schematic sorting cohesive fracture signals process for (a) ULK2.4 which has both cohesive and adhesive fracture modes and (b) #005 which has only adhesive fracture.
(3) Wave Structure of Adhesive Fracture Signals.

a. Construction of Band Pass Filter

Similar to what claimed above in the time-domain analysis part, differences between noise and adhesive fracture AE signals are small. Yet as is addressed above, if characteristic frequency of cohesive fracture signals (which represent longitudinal waves) lies in the range [900 kHz, 1.2 MHz], and the characteristic frequency ratio between longitudinal over shear waves is ~0.65, characteristic frequency of adhesive fracture signals (which represent shear waves) should be approximately in the range [600 kHz, 700 kHz]. There is a frequency peak at ~650 kHz in PSD figure, but it is believed that amplitude of this peak does not reflect much information. For some of the samples, for example, Intel sample #317, which only contains noise and adhesive fracture signals, amplitude of this peak is lower than that of air indentation noise, however for other samples, for example, IBM sample 96SJG4 (cohesive fracture signals removed), amplitude of this peak is higher (Figure 2.11 (a)). It is also noticed that at certain range (approximately [600 kHz, 640 kHz] as shown in squared part of Figure 2.11 (a) bottom), PSD amplitude of signals captured from tests is higher than that of air indentation noise. Though difference is still slight, considering expected adhesive fracture signals only take ~5% of overall signals in quantity, this difference between pure adhesive fracture signals and noises could be huge. The other evidence is from power distribution (Figure 2.101(b)). Energy portion in the range [500 kHz, 700 kHz] for #317 is slightly higher than that of noise.
PDF of Relative Pwr @ Ctr. Fs: .015/.03/.05/.07/.09/.11/.13/.15/.17/.19

Relative amount of power in the given bw
Figure 2.11 (a) PSDs (top) and amplified at squared range of PSDs (bottom) of air indentation noises, Intel sample #317 and IBM sample 96SJG4. (b) Power distributions of Intel sample #317, which only contains noise and adhesive fracture signals. Comparing to Figure 2.8 (c), circled value is slightly higher. (c) A cohesive fracture signal (from sample ULK2.4, 1099th) passing through the filter [800 kHz, 1 MHz] (top) and partial amplified waveform (bottom). Wave length and attenuation rate is measured. (d) A proposed adhesive fracture signal (from sample #005, 1190th) filtered by [600 kHz, 700 kHz], containing large pristine envelope, one significant reflection and another reflection submerged by noise (squared). (e) A filtered noise fitting description of pattern.

For each signal waveform, horizontal axis is scaled in $10^{-7}$ s and vertical in mV.

As is confirmed above, characteristic frequency of adhesive fracture signals lies in the frequency range [600 kHz, 700 kHz]. For cohesive fracture signals, wave length is ~12 µs. Therefore, characteristic wave length of adhesive fracture signals should be longer. It can also be observed that in filtered cohesive fracture signals, there is a large pristine envelope at the beginning of signal, followed by two envelopes that are smaller in amplitude but same in wave length. Between each two envelopes, amplitude drop is ~1/3 (Figure 3.7 (c)). The nature of this pattern is that, pristine envelope represents the appearance of the mechanical process, and the following two are nothing but the
reflections (echo, resonance) of the wave generated by the process, and for each time it reflects, attenuation rate is $r = \frac{1}{1 - \frac{1}{2} \cdot 2.25}$ (could be higher). Therefore, filtered adhesive fracture signals should also have similar wave pattern, where, large pristine envelope has a longer wave length (~20-24 µs) and 1 or 2 reflection envelope(s) follows (Figure 2.11 (d)). The reason for uncertain amount of reflections is that energy of pristine envelope is relatively small, and after 2 times of attenuation, the last reflection is easily to be submerged by noises. It is assumed that the noise patterns are totally random; hence it is not avoidable to have a filtered noise pattern fitting this description (Figure 2.11 (e)). After scrutiny, only 5 out of 274 signals fit the description of this pattern, thus the error floor is ~1.8%.

b. Pattern Recognition Based on Band Pass Filter

To recognize designated pattern shown in Figure 2.11 (d), similarly to recognition of cohesive fracture signals, when passing through band pass filter [600 kHz, 700 kHz], integrated energy defined in (2.7) within the window 0-20 µs should be significantly larger than the level of air indentation noise. The average energy in this window for noises is 0.1266 (AU). Hence the first threshold for this energy is set to be 0.14 (AU).

To avoid recognizing a waveform pattern shown in Figure 2.12 (a), the second cut-off is set as 0.07 (AU) for integrated energy within the window 0-10 µs. In the meantime, to avoid a flat envelope pattern, shown in Figure 2.12 (b), the maximum
filtered waveform function value with in the window 0-12 µs should be sufficiently large, and 150 (mV) is selected to be the cut off.

As shown in Figure 2.11 (d), within the windows where contain envelopes, 0-20 µs, 20-40 µs and 40-60 µs, the waveform should be wavy and no flat envelope as plotted in Figure 2.12 (c). Dispersion coefficients of absolute value of waveform function within these three windows, $S_1$, $S_2$ and $S_3$ are defined in (2.9). To ensure enough waviness, cut-off boundary of each coefficient is set to be 52%.

\[
\begin{align*}
S_1 &= \frac{\text{Stdev.} \left( |v_{filtered}(0: 20\mu s)| \right)}{\text{Avg.} \left( |v_{filtered}(0: 20\mu s)| \right)} \\
S_2 &= \frac{\text{Stdev.} \left( |v_{filtered}(20: 40\mu s)| \right)}{\text{Avg.} \left( |v_{filtered}(20: 40\mu s)| \right)} \\
S_3 &= \frac{\text{Stdev.} \left( |v_{filtered}(40: 60\mu s)| \right)}{\text{Avg.} \left( |v_{filtered}(60: 80\mu s)| \right)}
\end{align*}
\]  

(2.9)

The following filter is constructed based on energy attenuation ratios between integrated energies within neighboring windows, $R_{1,2}$ and $R_{2,3}$, which are defined in (2.10):

\[
\begin{align*}
R_{1,2} &= \frac{E_{filtered}(20 \mu s)}{E_{filtered}(40 \mu s) - E_{filtered}(20 \mu s)} \\
R_{2,3} &= \frac{E_{filtered}(40 \mu s) - E_{filtered}(20 \mu s)}{E_{filtered}(60 \mu s) - E_{filtered}(40 \mu s)}
\end{align*}
\]  

(2.10)

Prospective waveform should have the attenuation ratio values in the range [2, 4.5] as discussed above. However, considering two conditions:
(i) If attenuation from the first window to the second window is large, e.g., \( 4.5 \geq R_{1,2} \geq 2 \), the third energy envelope in the window 40-60 \( \mu s \) could not be identified from white noise floor if the second-time attenuation ratio, \( R_{2,3} \), is large. Therefore we choose \([1.5, 4.5]\) as the confidence limit of \( R_{2,3} \).

(ii) If attenuation from the first window to the second window is small, e.g., \( 2 > R_{1,2} \geq 1.5 \), for a prospective waveform, the second-time attenuation ratio should be greater than the value of \( R_{1,2} \). Hence confidence limit of \( R_{2,3} \) in this case is set to be \([R_{1,2}, 4.5]\).
Figure 2.12 Some eliminated signal examples. (a) Energy in first window is large overall but small within the first 10 µs. (b) A flat envelope that contains large energy in the first window. (c) Flat envelopes throughout the windows but attenuate from one window to another. For each signal waveform, horizontal axis is scaled in $10^{-7}$ s and vertical in mV.
After passing these filters, examples of resulting signals are shown in Figure 2.13(a). All the limits and thresholds are estimated by statistical analysis on both tests on samples and air indentation tests.
Figure 2.13 (a) Example of sorted adhesive fracture signals from (from top to bottom) #005, ULK2.4. (b) Frequency structures of adhesive fracture signals and air indentation. Comparing to Figure 2.11 (a), squared zone shows more significant difference. For each signal waveform, horizontal axis is scaled in $10^{-7}$ s and vertical in mV.
2.3.3 Energy of a Signal

When successfully getting cohesive and adhesive AE signals, delamination acoustic energy $AE_a$ and cohesive cracking acoustic energy $AE_c$ are then expressed in (2.11):

$$\begin{align*}
E_{i \text{coh}} &= E_{\text{signal}}(7.5 \mu s) - E_{\text{signal}}(2.5 \mu s) \quad \text{for cohesive signals} \\
E_{i \text{adh}} &= E_{\text{signal}}(10 \mu s) - E_{\text{signal}}(2.5 \mu s) \quad \text{for adhesive signals} \\
AE_a &= \sum E_{i \text{adh}} \\
AE_c &= \sum E_{i \text{coh}}
\end{align*}$$

Figure 2.14 Scheme of calculating acoustic energy of a signal. Vertical axis is in arbitrary unit and horizontal axis is in 0.1 µs.

The reason for different ranges chosen for cohesive (2.5 µs ~ 7.5 µs) and for adhesive (2.5 µs ~ 10 µs) fracture signals is due to different attenuation behaviors of the two types. Figure 2.14 shows a typical cohesive fracture signal that attenuates at ~ 7.5 µs. Time segment window length is around 5 µs. Considering ratio of 0.65, the window
length for adhesive fracture signal should be \(~7.5\mu s\), and the total attenuation time should be \(~1\mu s\).

**2.3.4 Role of Trigger Level**

The analysis is constructed based on one simple fact, that the adhesive signals are not definitely discriminated from background noises at the time range that triggers the acoustic emission sensor. Hence it must be applied when a lower trigger level is set (for our case, use 69 dB as trigger level instead of 70 dB that is automatically chosen by the machine) to enclose more signals, most of which are noises but the useful signals are also included. To demonstrate this fact, we have done sets of experiments. In each set, the only difference from one experiment to another is the trigger level selected. On the IBM sample ULK 2.4, we tested using final load of 4 mN, and the trigger levels were set 69 dB, 70 dB and 71 dB respectively (Figure 1.8). The analysis result shows that only in test at 69 dB we can find useful adhesive signals. No signal of this type is found when trigger level increases (Table 2.1). On IBM sample 96SJG4, we did 3 tests under the final load of 3 mN, two of which were at 70 dB trigger level and the rest was at 69 dB (Figure 2.15). The analysis result is consistent to what we have on ULK 2.4 sample, that only from the test at lower trigger level we can find useful signals (Table 2.2).

<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
</tr>
</thead>
<tbody>
<tr>
<td>ULK 2.4 @69 dB</td>
<td>116</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>ULK 2.4 @ 70 dB</td>
<td>5</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>ULK2.4 @71 dB</td>
<td>2</td>
<td>0</td>
<td>2</td>
</tr>
</tbody>
</table>

**Table 2.1** Result of AE analysis of tests on ULK 2.4 at different trigger levels
Figure 2.15 Tests on IBM sample 96SJG4 with final load of 3 mN. From top to bottom, trigger levels are set: 71dB, 70dB and 69 dB. There are 3, 8 and 150 events detected in tests.

<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
</tr>
</thead>
<tbody>
<tr>
<td>96SJG4 @70 dB-1</td>
<td>3</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>96SJG4 @70 dB-2</td>
<td>8</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>96SJG4 @69 dB</td>
<td>150</td>
<td>6</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 2.2 Results of AE analysis of tests on 96SJG4 at trigger levels of 70dB and 69 dB.
CHAPTER 3. RESULT AND DISCUSSION BY GROUPS

The samples are sorted by elastic mismatch between top layer and under layer \((E_I/E_S)\). For each sample, mechanical properties, fracture energies (based on analytical models, if possible) are found. Acoustic Emission signals detected are captured and sorted. Based on signal sorting results, fracture modes are identified, fracture energies are also estimated.

3.1 Hard Substrate: \(\sigma_{fr}^f/\sigma_{fr}^s < 1\) for All \(E_I/E_S\) Ratios

There are 4 samples (Intel samples #005, #302, #317 and #772 tested in this group. Overall information is listed in Table 3.1.

3.1.1 Intel Sample #005

Intel sample #005 has a single SiC:H (50%C 50%Si) layer of 100 nm thickness on the substrate of silicon. Young’s modulus of the film is 11.5 GPa with residual stress of 60 MPa. There are 9 tests on Intel sample #005. All tests show evidence that only adhesive fracture appears. There is no excursion observed on the load-displacement curves. 1221 AE events are captured in these 9 tests.
<table>
<thead>
<tr>
<th>Sample #</th>
<th>Film / Substrate</th>
<th>( \tau_f ) (nm)</th>
<th>( \sigma_f ) (GPa)</th>
<th>( E_f ) (GPa)</th>
<th>( E_s ) (GPa)</th>
<th>( \frac{\sigma_f}{\sigma_s} )</th>
<th>( \frac{E_f}{E_s} )</th>
<th>( \Gamma_a (\sqrt{f/m^2}) )</th>
<th>( \Gamma_c (\sqrt{f/m^2}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>#005</td>
<td>SiC:H/ Si</td>
<td>100</td>
<td>1.385±0.026</td>
<td>3.6</td>
<td>11.5</td>
<td>157</td>
<td>0.385</td>
<td>0.073</td>
<td>-</td>
</tr>
<tr>
<td>#302</td>
<td>SiC:H /Si</td>
<td>30</td>
<td>0.484±0.107</td>
<td>3.6</td>
<td>5</td>
<td>157</td>
<td>0.134</td>
<td>0.032</td>
<td>-</td>
</tr>
<tr>
<td>#317</td>
<td>Porous SiOC:H / Si</td>
<td>100</td>
<td>0.484±0.107</td>
<td>3.6</td>
<td>5</td>
<td>157</td>
<td>0.134</td>
<td>0.032</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.37±0.13**</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.15±0.06**</td>
</tr>
<tr>
<td>#772</td>
<td>Porous SiCx:H / Si</td>
<td>50</td>
<td>1.653±0.21</td>
<td>3.6</td>
<td>5</td>
<td>157</td>
<td>0.459</td>
<td>0.032</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.15±0.06**</td>
</tr>
</tbody>
</table>

**Table 3.1** Profiles and test results of samples in group 1. The “Exp” columns in fracture energies category are based on acoustic emission measurements of tests. The “model” columns in fracture energies category are based on analytical models. The DCB results are provided by Intel Corporation. Error bars are one time standard deviation from the average. *: Fracture energy value is estimated by (1.17) Marshall-Evan’s model. **: Fracture energy value is estimated by (1.26) insertion model.
Figure 3.1 (a) A typical load-displacement curve of test on Intel sample #005 and (b) corresponding AFM scanning topological (left) and gradient (right) images. AFM images show only delamination happens during indentation.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>AE_a</th>
<th>AE_c</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>84</td>
<td>3</td>
<td>0</td>
<td>2.278222</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>131</td>
<td>2</td>
<td>0</td>
<td>1.535826</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>136</td>
<td>2</td>
<td>0</td>
<td>1.660309</td>
<td>0</td>
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<tr>
<td>4</td>
<td>120</td>
<td>3</td>
<td>0</td>
<td>2.0936</td>
<td>0</td>
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<tr>
<td>5</td>
<td>150</td>
<td>5</td>
<td>0</td>
<td>3.69412</td>
<td>0</td>
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<tr>
<td>6</td>
<td>150</td>
<td>6</td>
<td>0</td>
<td>4.771472</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>150</td>
<td>7</td>
<td>0</td>
<td>4.858548</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>150</td>
<td>3</td>
<td>0</td>
<td>1.870343</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>150</td>
<td>3</td>
<td>0</td>
<td>2.130288</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 3.2 Results of signal sorting based on tests on #005. No cohesive fracture signal detected. Only adhesive fracture signals detected.

3.1.2 Intel Sample #302

Intel sample #302 has a single Porous SiOC:H (29% Si, 18% C, 53% O) layer of 30 nm thickness on the substrate of silicon. Young’s modulus of the film is 5 GPa with residual stress of 60 MPa. There are 6 tests on Intel sample #302. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial fracture is detected. There is no excursion observed on the load-displacement curves. Overall 330 AE events are detected during tests.
Figure 3.2 (a) A typical load-displacement curve of test on Intel sample #302 and (b) corresponding in-situ AFM scanning topological (right) and gradient (left) images. The images show evidence of plasticity, and inability to measure any kind of fracture.
Table 3.3 Results of signal sorting based on tests on #302. Even though adhesive fracture cannot be observed directly from the figure, portions that adhesive signals take are higher than noise error (1.8%, test #3 and #6). This can be considered the evidence of delamination.

3.1.3 Intel Sample #317

Intel sample #317 has a single Porous SiOC:H (29% Si, 18% C, 53% O) layer of 100 nm thickness on the substrate of silicon. Young’s modulus of the film is 5 GPa with residual stress of 60 MPa. There are 13 tests on Intel sample #317. All tests show evidence that only adhesive fracture appears. There is no excursion observed on the load-displacement curves. In fact, materials of #317 and #302 are identical, the difference is in thickness. Similar to #017, significant slope change makes this sample available for application of (1.26). Overall 1727 AE events are capture during tests.
Figure 3.3 (a) A typical load-displacement curve of test on Intel sample #317 and (b) corresponding AFM scanning topological (left) and gradient (right) images. AFM images show only delamination happens during indentation.
Table 3.4 Results of signal sorting based on tests on #317. No cohesive fracture signal detected. Only adhesive fracture signal detected.

3.1.4 Intel Sample #772

Intel sample #772 has a single Porous SiC$_x$:H (80%C 20%Si, cured) layer of 50 nm thickness on the substrate of silicon. Young’s modulus of the film is 5 GPa with residual stress of 50 MPa. There are 18 tests on Intel sample #772. All tests show evidence that only adhesive fracture appears. There is no excursion observed on the load-displacement curves. Similar to #017, significant slope change makes this sample available for application of (2.26). The in-situ AFM images also indicate evidence of plasticity. 1658 Events are captured during tests.
Figure 3.4 (a) A typical load-displacement curve of test on Intel sample #772 and (b) corresponding AFM scanning topological (left) and gradient (right) images. AFM images show only delamination happens during indentation.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>AE_3</th>
<th>AE_C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>68</td>
<td>1</td>
<td>0</td>
<td>0.844958</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>46</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>75</td>
<td>1</td>
<td>0</td>
<td>0.76071</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>74</td>
<td>2</td>
<td>0</td>
<td>1.401863</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>97</td>
<td>1</td>
<td>0</td>
<td>0.709211</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>90</td>
<td>5</td>
<td>0</td>
<td>3.79596</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>133</td>
<td>5</td>
<td>0</td>
<td>3.316918</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>129</td>
<td>1</td>
<td>0</td>
<td>0.596476</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>150</td>
<td>2</td>
<td>0</td>
<td>1.294621</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>50</td>
<td>1</td>
<td>0</td>
<td>0.694578</td>
<td>0</td>
</tr>
<tr>
<td>11</td>
<td>51</td>
<td>2</td>
<td>0</td>
<td>1.278544</td>
<td>0</td>
</tr>
<tr>
<td>12</td>
<td>65</td>
<td>2</td>
<td>0</td>
<td>1.374423</td>
<td>0</td>
</tr>
<tr>
<td>13</td>
<td>81</td>
<td>6</td>
<td>0</td>
<td>4.185427</td>
<td>0</td>
</tr>
<tr>
<td>14</td>
<td>92</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>15</td>
<td>81</td>
<td>3</td>
<td>0</td>
<td>2.055813</td>
<td>0</td>
</tr>
<tr>
<td>16</td>
<td>112</td>
<td>6</td>
<td>0</td>
<td>4.283566</td>
<td>0</td>
</tr>
<tr>
<td>17</td>
<td>132</td>
<td>5</td>
<td>0</td>
<td>4.007663</td>
<td>0</td>
</tr>
<tr>
<td>18</td>
<td>132</td>
<td>8</td>
<td>0</td>
<td>5.587492</td>
<td>0</td>
</tr>
</tbody>
</table>

**Table 3.5** Results of signal sorting based on tests on #772. No cohesive fracture signal detected. Only adhesive fracture signal detected.
Table 3.6 Profiles and test results of samples in group 2. The “Exp” columns in fracture energies category are based on acoustic emission measurements of tests. The “model” columns in fracture energies category are based on analytical models. The DCB results are provided by Intel Corporation. Error bars are one time standard deviation from the average. *: Fracture energy value is estimated by (1.17) Marshall-Evan’s model. **: Fracture energy value is estimated by (1.26) insertion model.
3.2 Soft or Comparable Substrate: \( \frac{\sigma_f}{\sigma_Y} \geq 1 \) for All \( \frac{E_f}{E_s} \) Ratios

There are 9 samples (Intel samples #008, #049, #096, #227, #811, #829 and IBM samples ULK2.4, 96SJG4) tested in this group. Overall information is listed in Table 3.6.

3.2.1 Intel Sample #008

Intel sample #008 has a SiCN layer of 25 nm thickness on SiCO:H under layer of 500 nm thickness. Young’s moduli of the film is 120 GPa and of the under layer is 9 GPa with high residual stress (unknown number) in the film. There are 9 tests on Intel sample #008. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial fracture is detected. Excursions sometimes can be observed on the curves, however further evidence (AE analysis) indicates they are related to plasticity rather than fracture. Overall 1106 events are captured during tests.
Figure 3.5 (a) A typical load-displacement curve of test on Intel sample #008 and (b) corresponding in-situ AFM scanning topological (right) and gradient (left) images. The images show evidence of plasticity, and inability to measure any kind of fracture.
Figure 3.6 (a) Another typical load-displacement curve of test on Intel sample #008 and (b) corresponding in-situ AFM scanning topological (right) and gradient (left) images. The images potential possibility of delamination and it is believed to appear on the interface between under layer and substrate. Excursion related to plasticity can be observed.
### Table 3.7 Results of signal sorting based on tests on #008. Even though adhesive fracture cannot be observed directly from the figures, portions that adhesive signals take are higher than noise error (1.8%, all except the last two tests). This can be considered the evidence of delamination. No cohesive fracture signal is observed.

<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>$AE_a$</th>
<th>$AE_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>74</td>
<td>3</td>
<td>0</td>
<td>2.165309</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>80</td>
<td>4</td>
<td>0</td>
<td>2.654203</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>103</td>
<td>2</td>
<td>0</td>
<td>1.696208</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>120</td>
<td>3</td>
<td>0</td>
<td>1.888071</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>129</td>
<td>3</td>
<td>0</td>
<td>2.034421</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>150</td>
<td>5</td>
<td>0</td>
<td>3.5006</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>150</td>
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<td>0</td>
<td>1.412863</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>150</td>
<td>1</td>
<td>0</td>
<td>0.596654</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>150</td>
<td>4</td>
<td>0</td>
<td>3.029076</td>
<td>0</td>
</tr>
</tbody>
</table>

**3.2.2 Intel Sample #017**

Intel sample #017 has a SiC:H (50%C 50%Si) layer of 100 nm thickness substrate of silicon. Young’s modulus of the film is 99 GPa with residual stress of 64MPa (compressive) in the film. There are 9 tests on Intel sample #017. All tests show evidence that adhesive fracture appears. When indenting on the sample it breaks. However, delamination is still able to be estimated by measuring bared substrate. Significant slope change makes this sample available for application of (1.26). Note that if 1/3 of local part of film breaks responsive force from the film is 2/3 of the force applied on it by the tip during loading, while indentation depth hardly changes. Therefore the work in Figure 1.5 for #017 is 2/3 of the expected value. Overall 1008 AE events are captured during tests.
Figure 3.7 (a) A typical load-displacement curve of test on Intel sample #017 and (b) corresponding in-situ AFM scanning topological (left) and gradient (right) images. (c) Schematic determination of delamination area.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>$\text{AE}_a$</th>
<th>$\text{AE}_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>63</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
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<td>85</td>
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<td>1</td>
<td>0.787109</td>
<td>49.35762</td>
</tr>
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<td>19.09337</td>
</tr>
<tr>
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<td>1</td>
<td>1.793655</td>
<td>56.89905</td>
</tr>
<tr>
<td>7</td>
<td>150</td>
<td>1</td>
<td>2</td>
<td>0.689527</td>
<td>53.74315</td>
</tr>
<tr>
<td>8</td>
<td>150</td>
<td>3</td>
<td>3</td>
<td>2.164703</td>
<td>101.4171</td>
</tr>
<tr>
<td>9</td>
<td>150</td>
<td>4</td>
<td>2</td>
<td>3.0151</td>
<td>93.6922</td>
</tr>
</tbody>
</table>

Table 3.8 Results of signal sorting based on tests on #017. The cohesive signals represent breaking of the sample, and due to inability of measure surface cracking, these results cannot be used to estimate fracture energies.

### 3.2.3 Intel Sample #049

Intel sample #049 has a SiC:H (50%C 50%Si) layer of 100 nm thickness on substrate of silicon. Young’s modulus of the film is 175 GPa with residual stress (unknown number) of 1031 MPa (compressive) in the film. There are 9 tests on Intel sample #049. All tests show evidence that only adhesive fracture appears. There is no excursion observed on the load-displacement curves. Overall 1082 events are captured during the tests.
Figure 3.8 (a) A typical load-displacement curve of test on Intel sample #008 and (b) corresponding in-situ AFM scanning topological (left) and gradient (right) images. Only adhesive fracture is observed. There is no excursion on the load-displacement curve.
Table 3.9 Results of signal sorting based on tests on #049. No cohesive fracture signal detected. Only adhesive fracture signal detected.

<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>$AE_a$</th>
<th>$AE_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>3</td>
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<td>1.974915</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>92</td>
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<td>0</td>
<td>0.747198</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>96</td>
<td>3</td>
<td>0</td>
<td>2.060732</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>104</td>
<td>6</td>
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<td>4.359272</td>
<td>0</td>
</tr>
<tr>
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<td>0.80367</td>
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</tr>
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</tr>
<tr>
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<td>4</td>
<td>0</td>
<td>2.458389</td>
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</tr>
</tbody>
</table>

3.2.4 Intel Sample #096

Intel sample #096 has a SiC:H (90% C, 10% Si) layer of 100 nm thickness on SiC:H (50% C, 50% Si) under layer of 1000 nm thickness. Young’s moduli of the film is 200 GPa and of the under layer is 350 GPa with residual stress of 1000 MPa (compress) in the film. There are 3 tests on Intel sample #096. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial fracture is detected. Excursions related to plasticity can be observed sometimes. Overall 450 events are captured during tests.
Figure 3.9 (a) A typical load-displacement curve of test on Intel sample #096 and (b) corresponding in-situ AFM scanning topological (right) and gradient (left) images. The images show evidence of plasticity, and inability to measure any kind of fracture.
Table 3.10 Results of signal sorting based on tests on #008. Even though adhesive fracture cannot be observed directly from the figures, portions that adhesive signals take are higher than noise error (1.8%). This can be considered the evidence of delamination. No cohesive fracture signal is observed.

3.2.5 Intel Sample #227

Intel sample #227 has a SiCN:H ES layer of 100 nm thickness on low-K SiCx:H under layer of 2000 nm thickness. Young’s moduli of the film is 130 GPa and of the under layer is 6.4 GPa with residual stress of 350 MPa (compress) in the film. There are 10 tests on Intel sample #227. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial fracture is detected. Excursions related to plasticity can be observed on load-displacement curves. Overall 1500 AE events are captured during tests.
Figure 3.10 (a) A typical load-displacement curve of test on Intel sample #227 and (b) corresponding in-situ AFM scanning topological (left) and gradient (right) images. The images show evidence of plasticity, and inability to measure any kind of fracture. Excursions related to plastic deformation of under layer can be observed on curves.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>$AE_a$</th>
<th>$AE_c$</th>
</tr>
</thead>
<tbody>
<tr>
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<td>5.49557</td>
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<td>0</td>
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<td>0.538859</td>
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</table>

Table 3.11 Results of signal sorting based on tests on #227. Even though adhesive fracture cannot be observed directly from the figures, portions that adhesive signals take are higher than noise error (1.8%, all except tests #3, #4 and #6). This can be considered the evidence of delamination. No cohesive fracture signal is observed.

3.2.6 Intel Sample #811

Intel sample #811 has a SiCN:H ES layer of 25 nm thickness on low-K SiCx:H under layer of 2000 nm thickness. Young’s moduli of the film is 130 GPa and of the under layer is 6.4 GPa with residual stress of 350 MPa (compressive) in the film. There are 16 tests on Intel sample #811. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial fracture is detected. Excursions related to plasticity can be observed on load-displacement curves. Overall 1941 AE events are captured during tests.
Figure 3.11 (a) A typical load-displacement curve of test on Intel sample #811 and (b) corresponding in-situ AFM scanning topological (left) and gradient (right) images. The images show evidence of plasticity, and inability to measure any kind of fracture.
Table 3.12 Results of signal sorting based on tests on #811. Even though adhesive fracture cannot be observed directly from the figures, portions that adhesive signals take are higher than noise error (1.8%, all except the first test). This can be considered the evidence of delamination. No cohesive fracture signal is observed.

3.2.7 Intel Sample #829

Intel sample #829 has a SiCN:H ES layer of 10 nm thickness on low-K SiCx:H under layer of 2000 nm thickness. Young’s moduli of the film is 130 GPa and of the under layer is 6.4 GPa with residual stress of 350 MPa (compressive) in the film. There are 6 tests on Intel sample #829. From in-situ AFM scanning images, neither of the fracture modes is observed. Black round hole whose shape is different from that of tip indicates plasticity happens during indentation. However, by AE analysis, interfacial
fracture is detected. Excursions related to plasticity can be observed on load-displacement curves. Overall 401 AE events are captured during tests.

![Figure 3.12](image)

Figure 3.12 (a) A typical load-displacement curve of test on Intel sample #829 and (b) corresponding in-situ AFM scanning topological (left) and gradient (right) images. The images show evidence of plasticity, and inability to measure any kind of fracture.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>AEₐ</th>
<th>AEₑ</th>
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</table>

Table 3.13: Results of signal sorting based on tests on #829. Even though adhesive fracture cannot be observed directly from the figures, portions that adhesive signals take are higher than noise error (1.8%). This can be considered the evidence of delamination. No cohesive fracture signal is observed.

3.2.8 IBM Sample ULK 2.4

IBM sample ULK 2.4 has four 500 nm thick layers of Low-K materials (unknown) on the substrate of silicon. Young’s modulus of the film is 6 GPa with unknown residual stress. There are 9 tests on IBM sample ULK 2.4. In the initial two tests only adhesive fracture observed, while the 7 tests show evidence that both cohesive and adhesive fracture happening when indenting on this sample. Excursions are observed on the curve, coincident with appearance of cohesive fracture. First excursion comes at around indentation depth of 500 nm. Overall 1227 signals are captured during tests.
Figure 3.13 (a) A typical load-displacement curve of test on IBM sample ULK 2.4 and (b) corresponding AFM scanning topological (left) and gradient (right) images. There are several excursions appearing on the curve, beginning at about indentation depth of 500 nm. Coincidently, cohesive fracture appears as well as adhesive fracture.
Figure 3.14 Measured $\Gamma_a = \gamma \frac{W_{ex}}{A_a} \times \frac{\Delta E_a}{\Delta E_a + \Delta E_c}$ vs. analytical model values.
<table>
<thead>
<tr>
<th>Test</th>
<th>Number of all events</th>
<th>Number of adhesive signals</th>
<th>Number of cohesive signals</th>
<th>$AE_a$</th>
<th>$AE_c$</th>
<th>$W_{ex}$</th>
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<th>$\Gamma_c$</th>
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</tr>
</tbody>
</table>

**Table 3.14** Results of signal sorting based on tests on ULK2.4. Both cohesive and adhesive signals are captured for all tests but the 8th test.
3.2.9 IBM Wafer 96SJG4

IBM sample 96SJG4 has a SiCxNy layer of 45 nm thickness on the substrate of silicon. Young’s modulus of the film is 85.3 GPa with residual stress of 430 MPa. There are tests on IBM sample 96SJG4. All tests show evidence that both cohesive and adhesive fracture happening when indenting on this sample. Excursions are observed on the curves, coincident with appearance of cohesive fracture. First excursion comes at around indentation depth of 150-200 nm. Overall 531 AE events are captured during tests.
Figure 3.15 (a) A typical load-displacement curve of test on IBM sample 96SJG4 and (b) corresponding AFM scanning topological (left) and gradient (right) images. There are several excursions appearing on the curve, beginning at about indentation depth of 160 nm. Coincidently, cohesive fracture appears as well as adhesive fracture.

![AFM images](image)

Figure 3.16 Measured $\Gamma_a = \frac{W_{ex}}{A_a} \times \frac{AE_a}{AE_a + AE_c}$ vs. analytical model values.
Table 3.15 Results of signal sorting based on tests on 96SJG4. Both cohesive and adhesive signals are captured for all tests.
CHAPTER 4. SUMMARY AND CONCLUSIONS

An experimental approach is developed to measure fracture behavior of ultra-thin film structure. Nanoindentation can not only measure and estimate mechanical behavior such as elastic modulus, hardness and fracture energy, but also provide simultaneous measurements of both cohesive and adhesive fracture energy by Acoustic Emission monitoring technique. By time-domain and frequency domain analysis, signals generated from AE monitoring are recognized and sorted into groups that denote cohesive cracking, debonding and background noises respectively. Pattern recognition criterions are constructed. It has been proved that different types of AE signals can be used to divide system energy released into corresponding varieties for further analysis. The main advantages of the technique are: samples are easier to fabricate; different modes of fracture can be evaluated at the same time; devastation to structure is relatively ignorable; residual stress effect is reduced. However, technical limit of this technique is also obvious, the major one of which is inability to measure thinner film and hard film on softer substrate system. Finally, fracture mode map is developed (Figure 3.19) which needs future work to extend.

According to test results, when film is over 10 times stiffer than the under layer, we will not observe any fracture. This is due to plasticity of under layer, which is independent of film thickness. Rapid yielding of under layer greatly releases concentration of stress in film, and threshold of crack initiation hence will be never reached. Large vertical deformation caused by plasticity of under layer also concaves the
surface in-situ, therefore delamination measurement based on vertical protrusion fails. On the other hand, due to combination plasticity and ultra-small film thickness of top layer, fracture is neither observed, though from the trend, there should be delamination happening underneath. However, delamination can be detected by acoustic emission monitoring.

Observed adhesive cracking happens in structure featuring harder film with elastic modulus ratio ranging from 0.71 to 5.71 as well as softer and low stiffness film. Adhesive crack length increases with respect to increment of indentation depth (Figure 3.17 (a)). It should be noticed that protrusion on surface caused by indentation may not be delamination, sometimes it could only be plastic zone. To validate delamination measurements, plastic zone size is estimated by (1.27) and (1.28). Instead of maximum indentation depth, contact depth $h_c$ is used to find equivalent conical tip. The results are shown by ratio between adhesive crack lengths over plastic zone radius. It is clear that measured protrusion size is significantly larger than plastic zone for these samples (Figure 3.18). Thereby it is proved the measured areas are delamination area for these samples. Intel sample #017 is a special example of this mode. The sample has extreme high hardness of over 20 GPa (to compare with its substrate, silicon, whose hardness is 10.5 GPa under same test set), and this means this film is highly brittle and unable to sustain large strain. When indenting on the sample, delamination initiates and propagates, and then due to high interfacial strength that strongly constraint film’s horizontal deformation, film buckles until at some level it breaks. However, delamination is still able to be estimated by measuring bared substrate. On delamination-only samples, by
our sorting techniques only adhesive fracture signals can be recognized without any cohesive fracture signal detected.

Cohesive crack appears if top and under layer have comparable mechanical properties. Conventional theory considers cohesive crack initiate at first, then from its root delamination initiates. However according to our results, delamination appears at very initial stage, then cohesive crack initiates from the root, and insipiently propagate as it is free-standing. When cohesive crack tip finally reached the edge of delamination area, as conventional theory predicts, crack root fractures interface and continues delamination. Therefore cohesive crack length is always smaller than radius of delamination area (Figure 3.17 (b)). A good result of fracture energy estimation is got from acoustic emission monitoring.
Figure 3.17 (a) Normalized adhesive crack length (vertical) with respect to normalized indentation depth (horizontal) of #005 and #017. (b) Normalized adhesive and cohesive crack length (vertical) with respect to normalized indentation depth (horizontal) of ULK 2.4
Figure 3.18 Elastic mismatches versus ratio of radii of delamination over estimated plastic zone.
Figure 3.19 Fracture mode map. This map shows a tendency of relationship between fracture mode and material mismatch. Horizontal axis represents the elastic mismatch and vertical axis represent plastic mismatch, between film and its substrate. *: Delamination of these samples is only estimated based on AE monitoring.


