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Inducing stable interfacial delamination in a multilayer system by four-point bending of microbridges

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ABSTRACT

The ability to produce stable delamination of thin film multilayer interfaces is a powerful tool for studying the interfacial adhesion within microsystems. In this study, a technique involving the four-point bending of microbridges was applied to initiate stable interfacial delamination within a multilayer system. Microscale pre-notched bridges with clamped-ends were machined into an Al/SiN/GaAs multilayer using focus ion beam milling. A square flat-end indenter was used to induce bending of the bridge by two contact locations. Bridge failure occurred via substrate fracture at the pre-notch, followed by crack deflection, and stable interfacial delamination of the SiN/GaAs interface. Substrate fracture and delamination were identified within the obtained load-displacement curves as a pop-in and region of linear load reduction respectively.

1. Introduction

The interfacial adhesion of thin film multilayers is an important factor affecting the performance and service life of contemporary microelectronic devices as well as micro-electrical mechanical systems (MEMS). Delamination of the thin films can lead to significant reliability issues. For the design of robust microsystems, the ability to assess a particular interface within multilayers, particularly on the microscale, is crucial. Therefore, methods capable of isolating and inducing stable delamination events within multilayer interfaces are of considerable research importance.

One of the most widely accepted methods used for studying interfacial adhesion within thin film multilayers is four-point bending (4 PB) [1,2]. The method has been successfully used to evaluate a variety of 'blanket' multilayers [3–7]. However, in order to apply load to the specimen, the configuration is conventionally applied on the macroscale. Consequently, the method is often unable to isolate the microscale interfaces that exist within 'patterned' multilayers. Top surface [8–10], and cross-sectional nanoindentation [11–13] are methods that have proven effective at initiating microscale delamination in a variety of thin films and multilayers. However, the complex stress state induced in a specimen during an indent is accompanied by unavoidable plastic deformation [9]. As a result, the energy dissipated by plastic deformation is not easily decoupled from the total work. A 'test-specific' practical work of adhesion is therefore typically obtained [14].

Nanoindentation induced deformation of microscale structures fabricated using focused ion beam (FIB) milling has been increasingly utilised for investigating the properties of thin films [15,16]. The deformation of a well-defined structure milled into a microscale feature of interest, allows the feature to be studied under a simple, quantifiable stress-state. For example, microcantilever (MC) bending has been used to investigate the elastic modulus [17], yield strength [18], fracture toughness [19,20], fatigue properties [21], and residual stress [22,23] of thin films. More recently, the fracture toughness of thin films and coatings have been assessed by means of micropillar splitting [24], double-cantilever compression [25], and three point bending of clamped bridges [26,27].

A limited number of studies have applied the indentation of fabricated microscale structures to investigate interfaces [17,28]. Matoy et al. [29], Hirakata et al. [30], and Chan et al. [31], used notched MC configurations to initiate delamination of SiO/W and SiO/Cu, Sn/Si, and Zr/hydride interfaces, respectively. Delamination generally occurred in an unstable manner due to these interfaces being orientated perpendicular to the axis of the MCs; prompting the development of alternative configurations [32].

This present work aimed to develop a microscale method capable of initiating stable interfacial delamination within a thin film multilayer by effectively miniaturising conventional 4 PB. FIB milling was used to fabricate microbridges (MBs) within an Al/SiN/GaAs multilayer; a system considered typical of those found in microelectronic devices. A 4 PB configuration was applied using a flat-end indenter in order to induce a

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linear-elastic stress state. This resulted in the initiation and deflection of a substrate crack, followed by stable delamination of the SiN/GaAs interface. The method is theoretically applicable to both blanket and patterned multilayers.

2. Methodology

2.1. Specimen preparation

An Al/SiN/GaAs multilayer specimen was synthesized for this study. Adhesion of the SiN/GaAs interface was of primary interest as delamination of the passivating SiN film has been a critical issue affecting the reliability of GaAs based microelectronics. Amorphous SiN film/single crystal (001) GaAs substrate specimens were provided by WIN Semiconductors Co. The SiN film was deposited by plasma enhanced chemical vapour deposition (PECVD); the details of which can be found elsewhere [8]. An aluminium film was deposited onto the SiN film by direct-current magnetron sputtering using an Auto 500 Sputter Coater (HHV Technologies, West Sussex, United Kingdom). Deposition was undertaken in an argon atmosphere at a base pressure of 4 mTorr, and a power range from 80 to 250 W.

2.2. Machining of microbridges

MBs were milled into the top surface of the specimens using a Scios DualBeam FIB (FEI, Oregon, USA). The detailed milling procedure can be found elsewhere [33,34]. The MBs were examined using a 7100F scanning electron microscope (SEM) (JEOL Ltd., Tokyo, Japan). The fabricated MBs consisted of an Al layer, SiN layer, and GaAs substrate component with side wall heights of h_{Al}, h_{SiN}, and h_{GaAs} respectively. SEM micrographs of a typical MB are shown in Fig. 1(a); the resultant pentagonal cross-section is illustrated in Fig. 1(b). MB dimensions, including length, L, and width, W, are summarised in Table 1. Wedge-shaped centered pre-notches of height, h_{notch}, with an included angle of 20°, were milled into the substrate component to promote fracture during bending. A milling current of 100 pA resulted in an average notch root radius of 200 nm. Large fillets, with radii, R_{end}, ranging from 3 to 5 μm, were introduced on the clamped ends of each MB to avoid stress concentrations. A centrally located blind hole, further referred to as a 'centring hole', was milled into the top surface of each MB using a low milling current of 50 pA.

Table 1
Dimensions of fabricated microbridges.

	MB1	MB2	MB3
L (μm)	34.53	34.08	26.56
W (μm)	3.61	3.44	2.92
h_{GaAs} (μm)	1.83	2.05	1.33
h_{SiN} (μm)	0.44	0.44	0.44
h_{Al} (μm)	0.75	0.75	0.68
h_{notch} (μm)	1.28	1.46	0.94
R_{end} (μm)	3.34	3.55	4.64

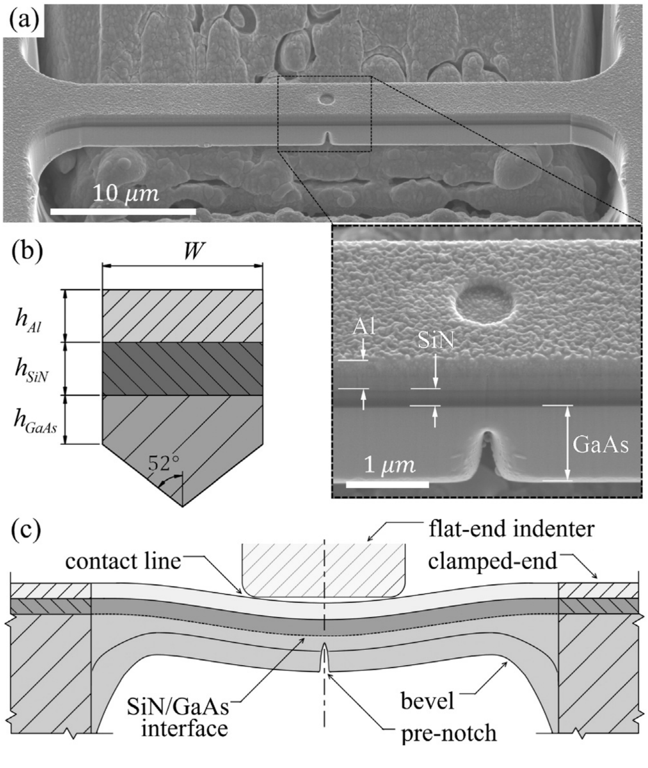

Fig. 1. (a) SEM micrographs of a typical FIB-milled MB (MB1) viewed at 45° tilt. Al, SiN, and GaAs components are labelled. (b) Indicative MB cross-section. (c) Schematic illustration of the 4 PB experimental configuration.

2.3. Nanoindentation

Nanoindentation induced bending of MBs was undertaken using a TI900 Triboindenter (Hysitron Inc., Minneapolis, USA). Bending was applied under a 4-point configuration using a 10 μm square flat-end indenter, whereby the opposite edges of the indenter acted as inner contact lines. A schematic of the experimental configuration is shown in Fig. 1(c). Alignment of the indenter was achieved by evaluating successive indentation impressions applied to an un-milled region of the sample surface. Tilt and rotational alignment of the indenter with each MB was evaluated using AFM and corrected using a tilt-stage. Positioning of the indenter on each MB was achieved by using the 'centring hole' as an optical reference. Indentation on all MBs were undertaken at a loading velocity 10 nm/s.

To allow for SEM examination of the MBs through each step of the fracture sequence, bending was applied incrementally over two separate indentations. During bending, once a particular feature was observed in the P-h data, the indenter was halted and withdrawn. The indenter withdrawal points were categorised into 3 distinct groups: withdrawals after a pop-in, [W1], withdrawals after a pop-in and subsequent decline in load, [W2], and withdrawals after a long term decline in load, [W3].

3. Results

The P-h curves for the 1st 4 PB of MBs are shown in Fig. 2(a). At low displacements all MBs exhibited linear-elastic loading behaviour. At higher displacements all MBs gradually deviated from linearity, followed by the occurrence of a significant initial pop-in. Bending of MB1 and MB2 was then immediately halted. Loading of MB3 continued at a dramatically increased compliance. This was followed by a second significant pop-in, resulting in a load drop to approximately 1000 μN; bending of MB3 was then immediately halted.

SEM examination was used to investigate the deformation behaviour of the MBs after the 1st indentation. MB1 and MB2 observations were indicative of the typical fracture state after the occurrence of an initial pop-in. MB3 observations were indicative of the typical fracture state after a second pop-in. Both fracture states are illustrated in Fig. 3(a) and (b). Substrate fractures were found to have initiated within the radius of the notch for all MBs. Propagation occurred at angles ranging from 0° to 45° from the vertical plane. For example, in MB1, a single substrate fracture was observed, terminating at the SiN/GaAs interface (Fig. 3(a)). In MB3, two substrate fractures were observed; one terminated at the interface, and the other deflected into the interface, resulting in a short asymmetric interfacial delamination (Fig. 3(b)). An impression was observed on the Al surface of each MB due to the contact of the flat-end indenter. Analysis of these impressions indicated

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