Interfacial strength and fracture toughness in integrated semiconductor materials

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Abstract

Two material systems, Si-SiC and GaN-on-diamond, used for compound semiconductor devices were studied in this paper. Firstly, SiC bonded to a Si substrate was characterized by X-ray computed micro-tomography and it was found that a bilayer structure, about 12 μm thick, with distinct density was created at the SiC-Si interface. With prolonged heat treatment at 1000°C for 12 hrs, the density difference between these two layers was reduced by about 9%, perhaps due to diffusion between the materials. In addition, micro-mechanical testing using nano-indentation indicated that the interfacial strength is improved by heat treatment. The second material studied was GaN attached to diamond. A fracture toughness approach based on energy release rate was adopted and the derived mixed mode practical work of adhesion was found to be significantly higher than a common metal-ceramic interface.

INTRODUCTION

High power GaN microwave technology is needed for the next generation of communication and radar technology. However, the traditional way of integrating GaN onto SiC substrates has seen a limit in terms of the capability of heat extraction from the devices. Recently, integrating polycrystalline diamond to the back of GaN based devices has been proven successful, demonstrating in excess of 3x RF power densities [1]. This represents an exciting opportunity to improve the heat dissipation capability of these devices and extend their lifetime due to the high thermal conductivity of diamond.

One critical question is whether the interface between the GaN and the diamond is mechanically robust enough to survive a real application in microwave devices. Thus, we have studied [2,3] previously the interfacial mechanical strength and the potential formation of voids at the GaN - diamond interface when diamond is grown onto the GaN for this integration. GaN-on-diamond materials made by first depositing nano-sized diamond particles as seeds for the diamond growth, followed by a 100 μm thick polycrystalline diamond layer using microwave (MW) plasma chemical vapor deposition (CVD) were considered. However, there are other ways for this integration, such as direct bonding. Initial successful attempts have been reported by BAE Systems [4]. This allows the integration of literally any two materials and it has the huge potential for producing strong and 'atomic-bonded' interfaces. Here in this work, we study several integrated systems including Si/4-H SiC and GaN-on-Diamond. Initial results on the thermal resistance of such devices looked promising [5], however the mechanical stability of these interfaces still needs to be assessed. Due to the small thickness of the individual layers (GaN<1μm), macroscopic mechanical tests on adhesion of the interfaces are impractical.

Conventionally, indentation is a method to measure the hardness of a material such as Vicker's hardness where a sharp tip made of a hard material with known properties (usually single crystal diamond) was pressed into the target sample; by measuring the applied load and the projected area of the ultimate imprint left on the sample surface due to plastic deformation, a single point measurement of the hardness of the tested sample can be derived. Modern instrumented nano-indenters are able to provide instantaneous output of the load and displacement (stress-strain curve), therefore allows the determination of mechanical properties such as the hardness, unloading elastic modulus, fracture toughness, and interfacial adhesion energy.

For a weakly bonded thin film to a rigid substrate, appropriate indentation force applied to the top surface of the thin film would be able to create local delamination, i.e. a blister, where the practical work of decohesion ($J.m^2$) can be derived based on the blister size. For strongly bonded films on a dissimilar substrate, indentation on a cross-section is necessary to create desired local decohesion / crack along the film-substrate interface. The crack trajectory in such bimaterial layered structures is determined by a mutual competition between the direction of 'maximum' mechanical driving force and the 'weakest' microstructural path [6]. In homogeneous, linear-elastic solids, cracks are presumed to follow a path of maximum strain-energy release which for all practical purposes coincides with propagation in the direction where the mode II stress intensity is zero, i.e. the local phase angle is zero. In light of this, the fracture toughness at the bonded interface can be derived based on the indentation force, distance of the indent to the interface, the interfacial crack length and propagation angle. In our current work, we used indentation on the top surface of the thin film to obtain the adhesion energy at the interfaces. In general, for hard film on a rigid substrate, there are two analytical methods available based on Hutchinson&Suo [7] and Marshall&Evans [8]. In this paper, using GaN-on-diamond as an example, the Hutchison&Suo model was used and the results discussed.

MATERIALS AND MEASUREMENT TECHNIQUE

Two material systems were studied: Si bonded to 4H SiC and GaN-on-diamond. For bonding Si to 4H-SiC, surface-activated bonding (SAB) was used for the manufacture in which the samples are bonded to each other after their surfaces are activated by Ar plasma irradiation to fabricate semiconductor junctions without heating the sample [9,10]. Consequently, junctions of dissimilar materials with different thermal expansion coefficients can be made by SAB. X-ray computed tomography (XCT) on a ZEISS Xradia 510 Versa microscope with voltage of 80 kV (power: 7W) to capture the 3D microstructure at the interface to obtain information for voids and discontinuities (the pixel size used was 0.68 µm, field of view of 1.4x1.4mm, 4x objective len). A Hysitron TI Premier nano-indenter with a Berkvich tip was used to indent into SiC coating surface to probe the interfacial adhesion energy to Si substrate. For the GaN-on-diamond, instrumented indentation was firstly performed on the surface of the GaN films, the blister sizes were measured subsequently using atomic force microscopy. The mechanical properties of the individual layers were obtained by the same system for consistency.

RESULTS AND DISCUSSION

For the Si/4H SiC samples with SiC coating, indentation tests were applied from the top surface in the untreated samples. Subsequently, samples annealed at 400°C and 700°C were tested to obtain the degradation of mechanical properties with annealing time and temperature. Fig. 1a shows a typical delamination introduced by applying an indentation force of about 15 mN and the corresponding load-displacement curve (Fig. 1b). An excursion in the load-depth curve was observed and is correlated to the formation of de-cohesion. For the sample annealed at 700°C, a much higher load is required to create local damage, and is shown in Fig. 1c, where a smaller blister was created. This indicates that the fracture toughness at the Si/4H SiC interface is improved with thermal annealing.

As shown in Fig. 2 and 3, the bilayer structure (a denser material adjacent to a less dense thin layer at the Si side) was created by the bonding prior to annealing in SiC-Si system. With prolonged heat treatment at 1000°C for 12 hrs, there is little change in terms of the width and relative density between these two layers. The thickness remained more or less the same, i.e. it changed from about 12 µm in untreated sample to about 14 µm in 1000°C 12hr condition.

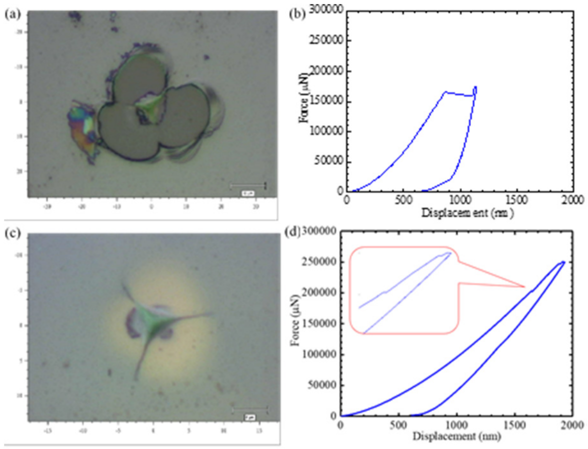

Fig. 1 (a) Optical image of a typical surface blister (local decohesion) created by indentation and (b) the corresponding load-displacement curve in the sample annealed at 400°C; (c) and (d) show a typical blister and indentation curve for 700°C annealed samples.

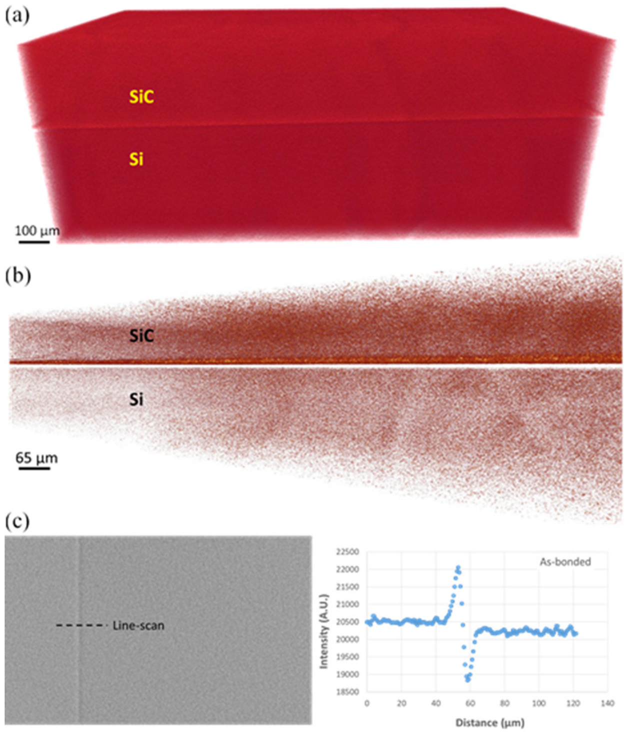

Fig. 2 (a) 3D reconstruction of Si/4H-SiC bonded structure, prior to annealing, showing a denser layer with an adjacent lower density layer at the interface; (b) visualization of this interface structure by contrast thresholding; (c) a line-scan across the interface in a typical slice of reconstructed X-ray tomography stack showing the change of grey scale over the distance.

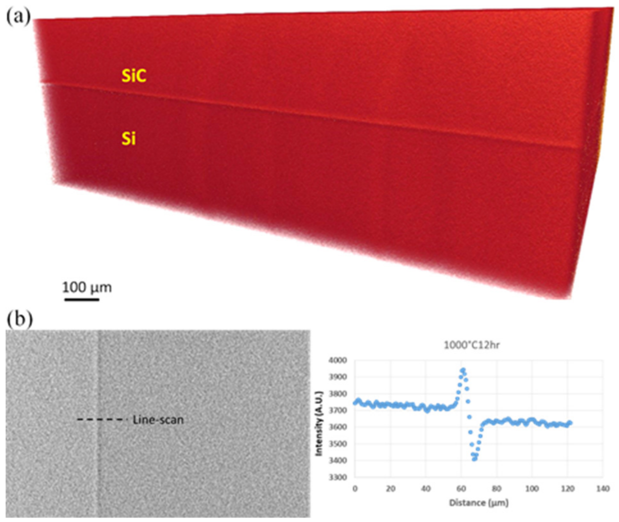

Fig. 3 (a) 3D reconstruction of Si/4H-SiC bonded structure after heat treatment at 1000℃ for 12 hrs showing the bilayer at the interface prior to annealing; (b) a line-scan shows the change of intensity (grey-scale) ie material density across this interface.

The density, however, reduced by about 9% after the annealing, i.e. there could be a diffusion process during the heat treatment. This is consistent with the nano-indentation measurement that the interfacial adhesion was improved upon heat treatment.

For the GaN-on-diamond material, it comprises of about 1 μm thick GaN on a 100 μm diamond substrate. Ex-situ micro-mechanical testing using nano-indentation was undertaken on the surface of the GaN to initiate damage at the interface hence the evaluation of its decohesion energy. In the present work, a fracture mechanics approach was adopted and it assumed elastic deformation as plastic effect; this is as due to the thin GaN film buckling only becomes pronounced for films with high residual stresses on relatively soft substrates [7,12]. The strain energy release rate (*G*), or the crack driving force was derived as a measure of the practical work of adhesion. The condition for crack propagation is when the strain energy release rate exceeds the resistance to crack growth ($\Gamma_{(\Psi)}$) which is usually a mixed mode term comprising the tensile stress perpendicular to the crack, σ, and shear stress along the interface, τ, at a particular phase angle Ψ.

$$\Psi = tan^{-1}\left(\frac{\tau}{\sigma}\right) \tag{1}$$

Mixed mode fracture is considered to be pertinent to interfacial crack formation due to the mismatch in elastic properties between the two dissimilar materials.

Based on the geometry and properties of the blister created by nano-indentation in the GaN film, such as the width, $2b$, the height, δ, the thickness, h, the elastic modulus, δ, and the Poisson's ratio, ν, the buckling stress, σ_b, which is the stress required to create such a buckle or blister, and the residual stress in the film, σ_r, can be expressed as

$$\sigma_b = \frac{h^2\mu^2 E}{12b^2(1-\nu^2)} \tag{2}$$

$$\sigma_r = \sigma_b(1 + \frac{c_1\delta^2}{h^2}) \tag{3}$$

where $c_1 = 0.2473(1+\nu) + 0.2231(1+\nu^2)$, $\mu^2 = 14.68$ for unpinned blisters and 42.67 for pinned blisters [7]. The mixed mode practical work of adhesion, $\Gamma_{(\Psi)}$, can then be represented by

$$\Gamma_{(\Psi)} = c_2[1 - (\frac{\sigma_b}{\sigma_r})^2]\frac{(1-\nu)h{\sigma_d}^2}{E} \tag{4}$$

where $c_2 = \frac{1}{1+0.9021(1-\nu)}$, and for a circular blister as in the current result, the mixed mode phase angle which represents the relation between normal and shear forces ahead of the crack tip at the interface, can be expressed by

$$\Psi = tan^{-1}\left(\frac{\cos(\omega)+0.2486(1+\nu_1)\delta/hsin(\omega)}{-\sin(\omega)+0.2486(1+\nu_1)\delta/hcos(\omega)}\right) \tag{5}$$

where $\omega = 52.1$ in the case of a rigid substrate like diamond assuming no elastic mismatch.

Therefore, the mode I adhesion energy caused by the force normal to the interface and opens the crack at zero phase angle, Γ_I, can be derived as

$$\Gamma_{\mathrm{I}} = \Gamma_{(\Psi)}\{1 + tan^2[\Psi(1-\lambda)]\} \tag{6}$$

where $\lambda = 0.3$ for a brittle interface as is appropriate for the current material structure.

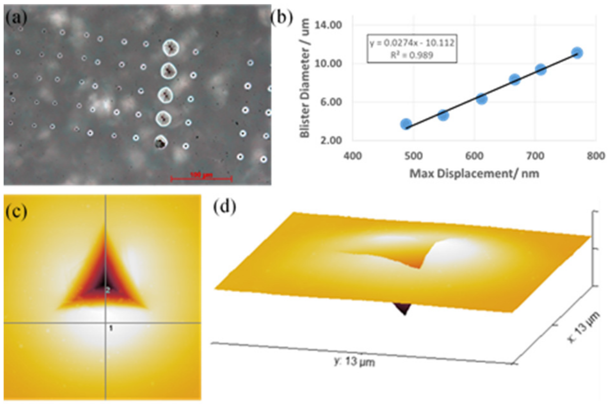

Fig. 4 (a) Optical image showing a typical group of indents made on the top surface of GaN-on-diamond with different indentation loads; (b) the blister size (diameter) was measured and plotted as a function of the maximum load applied; (c) atomic force microscope (AFM) was used to measure the size and depth of the indents as shown in (d).

Based on the experimental data, Fig. 4, and applying equations (1-6), the mode I energy was found to be less than 10 J·m^{-2}. This value is higher than reported in literature such as less than 0.5-2 J·m^{-2} for metals on ceramic [11], suggesting high mechanical stability of this interface. Further refinement of the data derived from the current work will be presented in a future paper. Errors associated with the parameters used for the analytical solutions including residual stresses, film thickness, modulus and Poisson's ration are being analyzed. To further confirm the derived results, other methods proposed by Marshall&Evans [8], and micro-mechanical testing using a micro-cantilever bending geometry are being developed.

CONCLUSIONS

Two types of heterogeneous interfaces were studied in the present work. SiC attached to Si was studied using XCT and nano-indentation method. It was found that thermal treatment increased the interfacial strength. Furthermore, the interfacial toughness in terms of the practical work of adhesion was evaluated in GaN attached to diamond substrate. A method proposed by Hutchinson&Suo was used and the mode I energy release rate appear higher than metal on ceramic systems. Further work on refining the results are ongoing and will be presented in a future paper.

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