Evaluating the interfacial toughness of GaN-on-diamond using blistering method with nano-indentation

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Abstract

An improved analysis of the interfacial toughness using nanoindentation induced blistering of thin films on stiff substrates is demonstrated on GaN-on-diamond. The Hutchinson-Suo analysis requires accurate measurement of blister dimensions, conventionally measured using 2-D line-scans from 3-D topographical maps. The new meteorology overcomes shortcomings of this technique by fitting the 3-D analytical solution of a clamped Kicrchoff plate to the topological map of the blister. This allowed for quantification of interfacial toughness of smaller blisters in GaN-on-diamond, previously assumed invalid for analysis due to inadequacies of the line-scan analysis. Three samples were investigated and found to have interfacial toughness ranging from 0.6–1 J m^{-2}. Additionally, the relationship between residual stress in the GaN and interfacial toughness was investigated using photoluminescence spectroscopy. In all cases, the GaN was found to be under increased compression at the diamond interface by up to -0.81 GPa, although no correlation with interfacial toughness was observed.

1. Introduction

Nanoindentation induced blistering is an approach for measuring interfacial toughness of films on thick substrates developed in the 1970-80s. The thin film is indented, introducing compressive stress which, if large enough, will cause film debonding and subsequent stress relaxation through buckling. The most commonly used analytical solution for extracting interfacial toughness is that demonstrated by Hutchinson and Suo. This method calculates GIc from the blister radius, a, and the buckling height, δ. These dimensions can be measured from milled 2-D cross-sections (e.g., focussed ion beam milling) or 3D surface maps (e.g., Atomic Force Microscope, AFM, profiles or Digital Holographic Microscopy, DHM, profiles). For the former, it was found that FIB milling results in relaxation of stresses in the film, causing crack closure or opening and inaccurate measurements of the buckle radius and height. For the latter, the highest point of the blister above the surface is usually taken as the buckling height. This approximation is valid for blisters large relative to the indented area. However, it is less appropriate when the indent takes up a significant portion of the buckled area, a scenario which will become more common with increasing integration of strongly bonded brittle films. For such systems, accurate and reliable quantification of the interfacial toughness is vital for improved reliability.

2. Results

In this work, we used gallium nitride (GaN)-on-diamond as a model system where polycrystalline diamond is grown onto a GaN backside. An interlayer, such as SiNx, is required to protect the GaN from the harsh diamond growth conditions (>800 °C, H2 plasma) and ensure good adhesion between the diamond and GaN. The large coefficient of thermal expansion (CTE) mismatch between diamond and GaN poses a significant challenge in creating a robust material. Mismatch in CTE induces stresses during manufacture which can be high enough to cause delamination and failure of the device. A mechanically stable interface is vital for wide uptake of GaN-on-diamond in high power RF amplifier applications. Three GaN-on-diamond samples have been used to introduce an improved blister analysis that can be applied to a range of brittle films on stiff substrates. The novel method proposed for the extraction of buckle height and blister radius was to fit a clamped Kirchoff plate undergoing buckling to the entire blister 3-D surface topology, rather than using manually drawn 2-D line-scans. Whilst the line-scan analysis has been validated for GaN-on-diamond against alternative analyses, it is believed that there is a systemic underestimation of δ as well as an overestimation of the error in the measured dimensions. Following this, the interfacial toughness values are used to glean insights into the relationship between interfacial toughness and residual stresses in the GaN layer.

Fig. 1(a) presents examples of line-scans and a cross-section of the 3-D fitted buckle for a blister, showing good agreement between fit and experimental data. This figure demonstrates a fundamental limitation in the line-scan analysis as the maximum deflection is expected at the centre of the indented area, impossible to measure using this meteorology. Whilst in this example the measured δ from the line-scans is not too dissimilar from the fitted δ, this is a result of cracking of the protective SiNx layer rather than accurate measurement. Additionally, the vagaries in the line-scan analysis are underlined; the measured δ is dependant on where the line-scans were taken from.

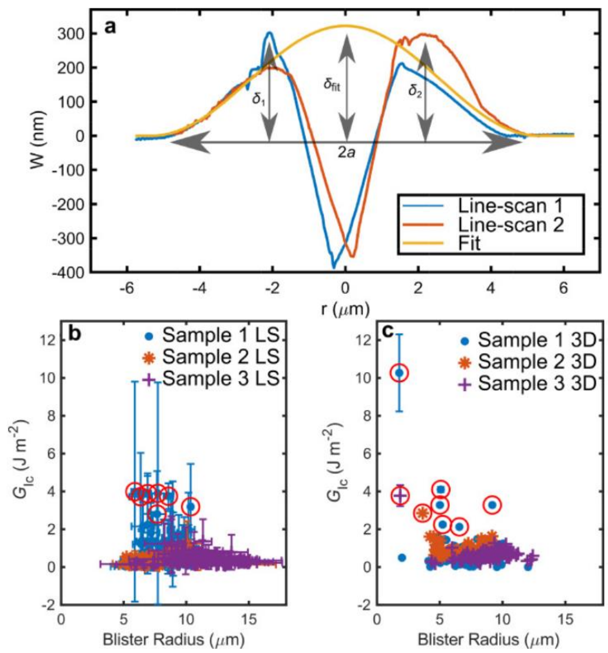

Fig.1. (a): Line-scans of a blister, and the fitted Kirchoff plate undergoing buckling. Marked are the blister diameter, 2a, and the maximum buckle height extracted using line-scans (δ1,2) and the fitted Kirchoff plate (δfit). (b) and (c) show interfacial toughness against measured blister diameter for 3-D fitting and line-scan analysis respectively. Error bars are ± 1 standard deviation and outliers, calculated using a 95% Grubbs test, are circled in red.

3. Conclusions

A novel analysis method has been developed in this work for improved calculation of interfacial toughness using nanoindentation induced blistering. This involves fitting the analytical solution for the shape of the blister to the experimental data, removing the influence of the indented area in measurement buckle height. This has been demonstrated for a GaN-on-diamond material system to improve the reliability of calculation of interfacial toughness compared to conventional 2D line-scan analysis. In addition, it has been shown that smaller blisters, previously assumed to violate assumptions in the Hutchinson Suo model, are in fact consistent with larger blisters and can be used in calculating using our new meteorology.