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Hydrogen Ion-Induced AlN Thin Layer Transfer: An Elastomechanical Study

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The change in the micro-structural properties of epitaxially grown AlN as a function of different H-fluences and thermal evolution was elucidated by nanoindentation and by atomic force microscopy. In this study, we investigated 2 µm-thick AlN layers grown epitaxially on sapphire. The elasto-mechanical properties were measured using a Nanoindenter with continuous stiffness measurement attachment. The AlN samples were implanted with hydrogen ions at 50 keV with various fluences ranging from $0.5 \times 10^{17} cm^{-2}$ to $3 \times 10^{17} cm^{-2}$. The elastic modulus and hardness were carefully determined for each sample. The samples were then annealed in air at temperatures ranging from 300°C to 600°C for 5 min to study the influence of pre-layer splitting treatments on the mechanical properties.

Introduction

In recent years, the use of III-nitride compound semiconductors such as GaN, AlN, InN has seen a phenomenal growth in the optoelectronics industry. The exploitation of the full potential of these emerging technologies faces major challenges, namely the lack of high crystalline quality material and the high cost associated with it. In fact, bulk III-nitride wafers are expensive (only 2 inch bulk GaN wafers are commercially available) or still under development. In general, sapphire is used to epitaxially grow III-nitride layers. Nevertheless, because of the high lattice mismatch between these semiconductors and sapphire, the grown layers are generally defective. Higher quality substrates can be obtained by growing thicker layers on sapphire and their subsequent release from the seed substrate (1). The cost of these bulk materials is still too high to compete with alternative technologies (e.g., SiC). One of the possibilities to reduce the material cost would be the application of the ion-cut process to cleave several thin layers from the same wafer and transfer them onto foreign and cheap substrates (2,3). In principle, this ion-cut, a generic process based on light ion (H and/or He) implantation and wafer bonding, can be used to transfer bulk quality fine layers onto foreign substrates thereby achieving heterostructures frequently unattainable by other methods such as epitaxy. The transfer of several layers from the same wafer is believed to reduce the material cost.

In this study, we investigate the evolution of the mechanical properties during the splitting process of AlN. The mechanical properties were investigated by nanoindentation testing. During nanoindentation testing, the elastic modulus and hardness are the most frequently measured properties. The elastic modulus and hardness of a material can be extracted from the load-depth curve. The load-depth curve is composed of a loading step followed by an unloading step. The loading step may consist of elastic deformation followed by a plastic yield. The unloading step consists of plastic deformation after yield has occurred. Hardness is defined as the resistance to penetration or resistance to permanent deformation due to a load or force from a sharp object. On the other hand the elastic modulus or Young's modulus is defined as the tendency of a material to deform elastically along the axis the forces are acting on. In nanoindentation testing, the hardness is defined as the ratio of the maximum load (from the load-depth curve) to the projected area. The elastic modulus is defined as the ratio of the slope of the unloading curve measured at the tangent to the data point at the maximum load and the projected area.

In this work, the change in the micro-structural properties of epitaxially grown AlN has been investigated as a function of different H-fluences and thermal evolution by nanoindentation, and by atomic force microscope (AFM).

Experimental Set-up

A 2μm-thick AlN layer was epitaxially grown on sapphire. The AlN samples were implanted with hydrogen ions at 50 keV with various fluences ranging from $0.5 \times 10^{17}cm^{-2}$ to 3×10^{17} cm^{-2}. The elastic modulus and hardness were carefully determined for each sample. A virgin AlN sample was also used as benchmarking. The samples were then annealed in air at temperatures ranging from 300 ^{o}C to 600 ^{o}C for 5 min to study the evolution of the mechanical properties throughout ion-induced AlN thin layer splitting. The sample with the critical H-fluence for layer splitting $1 \times 10^{17}cm^{-2}$ received repeatedly higher annealing temperature treatments increasing in 50 ^{o}C steps. The elasto-mechanical properties were measured using the Nanoindenter XP by Agilent with continuous stiffness measurement (CSM) attachment. The CSM attachment allows the continuous measurement of the contact stiffness throughout the loading cycle not just at specific depth. With a continuous measurement of the stiffness, one can carefully calculate the elastic modulus and hardness of materials as a continuous function of depth. A triangular pyramidal diamond Berkovich tip was used as the indenter. The indenter approaches the sample surface at a rate of 10 nm/sec. Once the maximum load is reached, the load on the sample is maintained constant for 10 seconds. Then, the indenter is unloaded by 90% of the maximum applied load followed by a hold segment to correct for the thermal drift. Finally, the indenter is completely unloaded. A 50Hz oscillation frequency was used. Prior to the measurements, the machine was calibrated using a triangular shaped diamond Berkovich tip on fused silica.

Results

The surface morphology was investigated using a Nanoscope Dimension™ 3100 AFM by Veeco. The RMS roughness values of AlN surface are plotted in Fig.1 as a function of H-implant fluence. As a general trend, it emerges from this analysis that AlN surface becomes rougher with an increase in H-fluence. This issue is very critical as it can impact the quality of the bonding of H-implanted wafers.

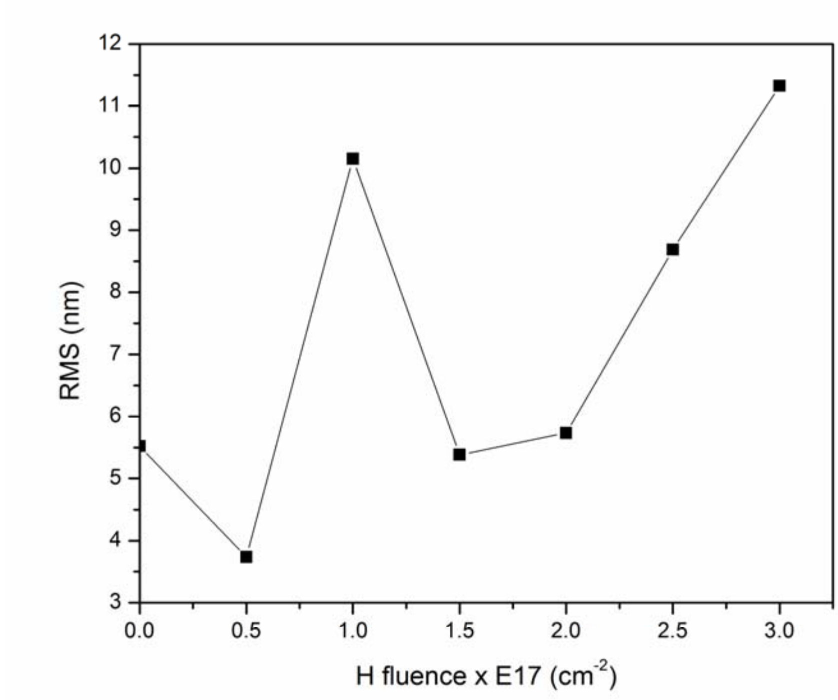

Figure 1. The evolution of the RMS roughness of AlN from AFM measurements as a function of H ion fluence. A scan area of 5μm×5μm was used for each sample.

Figure 2 shows the load-depth curve of a 1000 nm indent on an AlN sample that received a fluence of 0.5×10^{17} H cm^{-2}. No pop-in, no sink-in nor phase induced transformation can be seen in Fig. 2. This is also confirmed by the AFM scan of an impression of the Berkovich indent in Fig. 3. The peak of the 50 keV H implantation in the AlN samples was found to be around 350 nm. After indentation, the elastic modulus and hardness were plotted at selected indentation depths as shown in Fig. 4. No clear trend was observed in the elastic modulus as the H-fluence was varied. However, there is an increase in hardness as the H-fluence is varied. The AlN hardness increased from 18GPa for the virgin sample to ~25GPa for the highest fluence of 3×10^{17} H cm^{-2}. Our measurements for the virgin AlN sample are very close to literature values of nanoindentation of AlN (4). The highest value of the hardness was found to be around the H implantation region. Higher H-implantation doses effectively render the AlN material more brittle. As the samples are implanted with higher H-fluences, the amount of H ion implantation induced lattice damage increases resulting in higher values of hardness as can be seen in Fig. 4. As the indentation depth is increased beyond the H implantation region, the hardness values decrease and merge to the virgin sample's lower hardness value as depicted in Fig. 4.

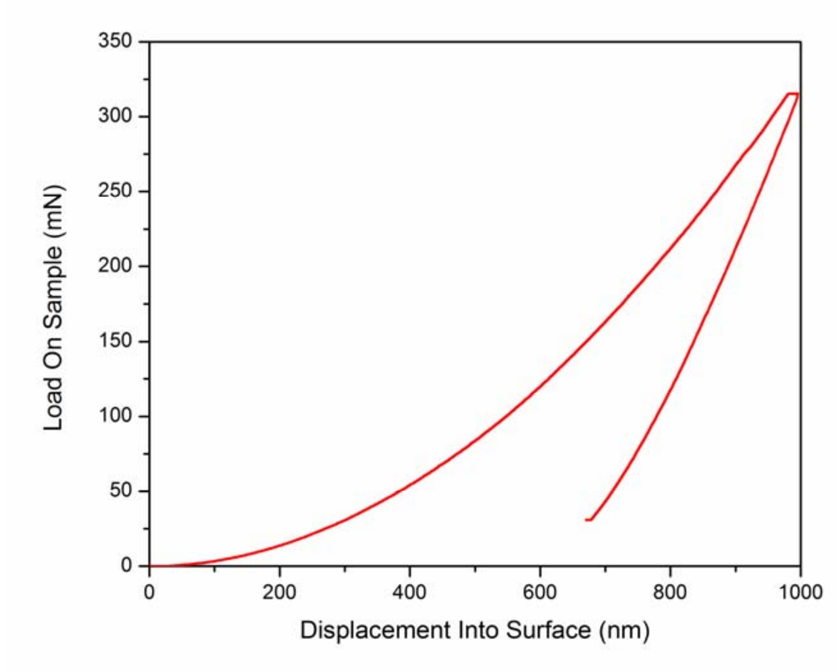

Figure 2. Load-depth curve of a 1000 nm indent on AlN implanted by H at a fluence of 0.5×10^{17} cm^{-2} at 50 keV.

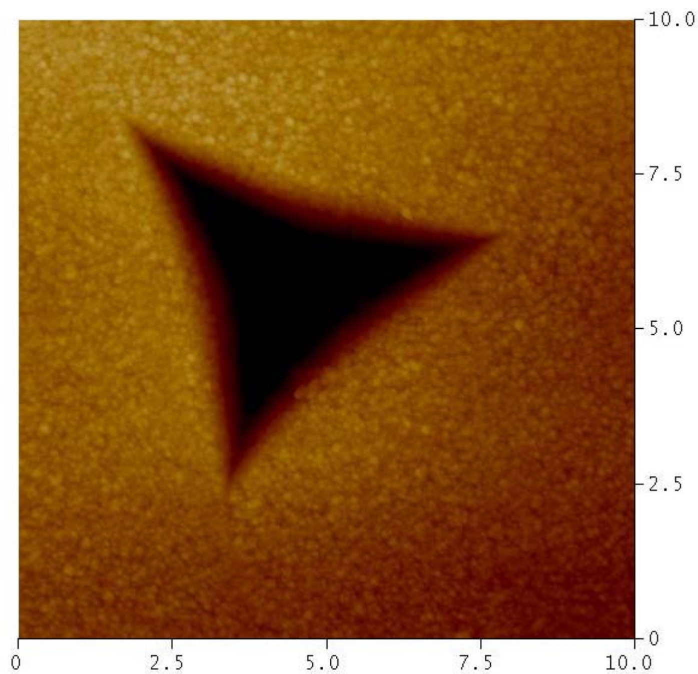

Figure 3. AFM scan of 1000 nm indent Berkovich indent on AlN implanted by H at a fluence of 0.5×10^{17} cm^{-2} at 50 keV.

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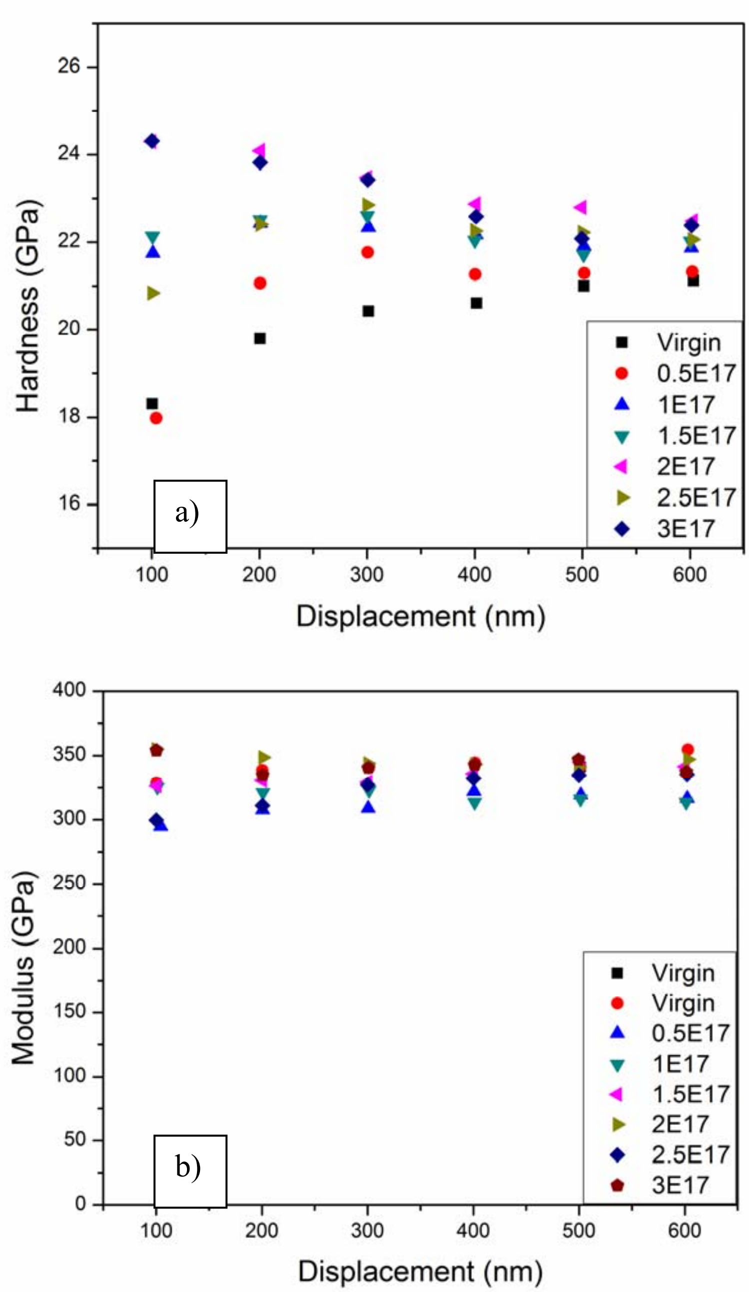

Figure 4. a) Depth profile of the hardness of AlN implanted at different H-fluences. b) Depth profile of the elastic modulus of AlN implanted at different H-fluences. For the sake of comparison, the corresponding profiles in virgin AlN are also shown.

Figure 5 shows the plot of the hardness and elastic modulus of the critical H-fluence for AlN layer splitting of 1×10^{17}cm^{-2} at 50 keV for various annealing temperatures ranging from 300 ^{o}C to 600 ^{o}C for 5 min anneal duration. From Fig. 5, the hardness increases upon annealing to a maximum value at an annealing temperature of 500^{o}C, but then the hardness drops for higher annealing at 600^{o}C. The same trend was observed in the subcritical H-fluence of 0.5 x10^{17}cm^{-2} and supercritical H-fluence of 2×10^{17}cm^{-2}. During annealing, the AlN samples appear to experience work hardening.

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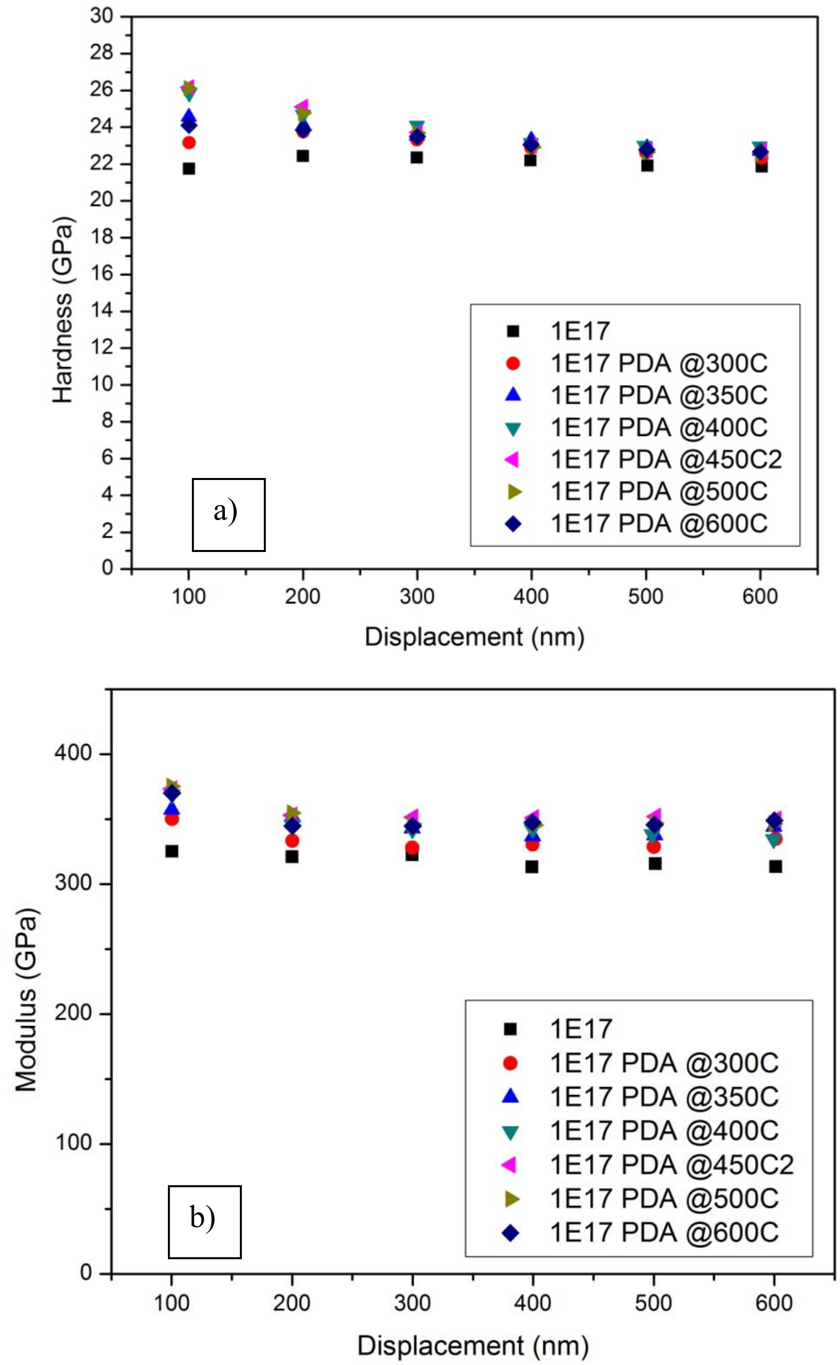

Figure 5. a) Depth profile of the hardness of AlN implanted at 1 × 10^{17} H/cm^2 and annealed at different temperatures. b) Depth profile of the elastic modulus of AlN implanted at 1 × 10^{17} H/cm^2 and annealed at different temperatures. For the sake of comparison, the corresponding profiles in as-implanted AlN are also shown.

As the annealing temperature is increased, the ion implanted hydrogen in the AlN layer is subject to complex point defect reactions with the defect clusters. The result is a measurable increase in hardness value shown Fig.5, which is indicative of an increase in brittleness. The hardness of the H implanted region of the AlN sample increases with annealing temperature until a critical temperature is reached at 500 ^{o}C. However, further increase to higher annealing temperatures causes a drop in hardness to lower values. This drop in hardness is attributed to loss of hydrogen by out diffusion at elevated temperatures and possibly due to relaxation of internal strain. The standard deviation of the elastic modulus measurements is ±7.62 GPa and that of the hardness measurements is ±0.829 GPa

Conclusions

Epitaxial AlN samples were implanted with various H-fluences for wafer bonding and layer splitting by ion-cut technology. The mechanical properties were carefully analyzed by nanoindentation. Our measurements revealed a dependency of the hardness on the implanted H-fluence and the annealing temperature. We successfully established a relationship between thermal treatments and the mechanical properties. The hardness of AlN increases with annealing temperature rendering the material more brittle. This study provides us with unprecedented insights in the mechanical behavior of H-implanted AlN throughout subsurface micro cracking leading to thin layer transfer.

Reference

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