A NOVEL SINGLE STEP LAPPING AND CHEMO-MECHANICAL POLISHING SCHEME FOR ANTIMONIDE BASED SEMICONDUCTORS USING 1 \( \mu m \) AGGLOMERATE FREE ALUMINA SLURRY

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ABSTRACT

This paper presents a novel approach for a single step lapping and chemical-mechanical polishing of antimonide-based III-V compounds using agglomerate-free alumina slurries. Relatively high removal rates and minimal scratching have been obtained using agglomerate slurries. The effects of slurry preparation cycle on the slurry properties and chemo-mechanical polishing results are discussed.

INTRODUCTION

Traditionally, wafer polishing of elemental and compound semiconductors is performed in multiple steps. In the first step, the sawed wafer is made parallel on both sides using a large diameter abrasive (15-20 \( \mu m \)) on a hard pad. Subsequently, the highly parallel wafer is polished with decreasingly lower abrasive size particles (down to 0.01 \( \mu m \)) on medium hardness pads and soft pads. This strategy is well established for most of the elemental and binary compounds such as Si, Ge, GaAs, InP etc [1,2]. For relatively soft or fragile compounds such as III-V antimonides, II-VI’s and recently developed ternary and quaternary III-V substrates [3-5], usage of large diameter lapping abrasives introduces permanent cracks in the wafers. Especially for the ternary and quaternary alloys, large lattice stress exists [6] and the wafers easily crack during the lapping process. Availability of high quality ternary and quaternary substrates in large scale will open up new avenues for devices employing novel band gap engineering strategies. Until recently, bulk ternary and quaternary substrates could not be grown without cracks. Growth of crack-free ternaries GaInSb, GaInAs and quaternary GaInAsSb crystals have now been made possible using novel approaches [3-5,7] and the feasibility for large scale production has been demonstrated. However, commercial development of ternary and quaternary substrate technology not only depends on the improvements in the crystal growth techniques, but also on the substrate slicing and polishing procedures. Substrate preparation issues have been addressed in this paper.

Initial work on lapping and polishing GaInSb poly- and single crystalline materials using the conventional procedures (for elemental and binary compounds) led to excessive breakage of wafers. Multi-component alloys are in general fragile and care should be taken during the wafer preparation process. Cracking of wafers usually takes place when high mechanical force is applied during polishing and/or by using large diameter abrasive particles (\( > 5 \mu m \) in this case). The mechanical cracks are generated at the surface and then rapidly propagate to the entire bulk of the wafer. In this work, it has been demonstrated that the cracking can be avoided by using smaller abrasive particles (\( \sim 1 \mu m \)) for lapping. However, the lapping and polishing times are increased enormously unless agglomerate-free slurries are used. Prolonged lapping and polishing cycles can also lead to mechanical damage of the wafers. This paper presents a novel approach for preparing damage free antimonide-based III-V
substrates of GaSb and GaInSb using the Baikolox CR-series of agglomerate-free alumina slurries. Rapid removal of material in a damage-free fashion has been evidenced with slurries containing abrasive particles of 1 μm and less. The surface finish obtained using these slurries is close to that of device grade silicon. The high removal rate exhibited by these slurries is attributed to the agglomerate-free nature resulting from special preparation procedures.

EXPERIMENTAL DETAILS

Single- and poly-crystalline wafers of GaSb and Ga1-xInxSb (0.05 < x < 0.25) were sliced from in-house grown bulk crystals [3-5] using a Princeton Scientific wire saw (model WS-22) with boron carbide (14 μm abrasives)-glycerine slurry. The sliced wafers exhibited the usual saw ridges, but no mechanical cracks. GaSb was used as a base-line comparison for the ternary substrates. For lapping and polishing, the samples were attached to a holder using crystal bond (wax) from MR Semicon Inc. A variety of alumina based slurries have been evaluated along with several different types of soft and hard pads (such as Chemo-Tex, Nylon, Velvet from South Bay Technology and Rodel IC-1400). Buehler Micro-polish and Malvern Multipol polishing systems were used. In the Buehler Micropolish unit, the slurry is contained in a shallow enclosure with a fixed pad. The sample holder is attached to an arm which performs an oscillatory rotation. In the Multipol unit, the sample rotates along with the pad (in the opposite direction) while the slurry is continuously dispensed. Typical sample rotation speed employed in this work varied in the range of 20 – 50 rpm. Between subsequent polishing steps, the sample along with the holder was cleaned in de-ionized water, kept inside an ultrasonic bath and dried with nitrogen gun. After polishing, the wafers were inspected under optical microscopy. Atomic Force Microscopy (AFM) measurements have been used to evaluate the micro-roughness of the polished surfaces. The AFM scans were performed using a Nanospec AFM system in the tapping mode. In most of the cases, the scratches mentioned here are not seen with optical microscopy.

EFFECT OF FORCED CONVECTION ON SLURRY PROPERTIES

The hydrodynamic fluid motion during slurry preparation has been found to influence slurry viscosity and agglomeration. For the same weight content of abrasives, slurries prepared using different mixing schemes possess significantly different characteristics. In the present study, water based alumina slurries with particle sizes ranging from 0.05 – 1 μm has been prepared by mixing powder abrasives with water. High shear mixing during slurry preparation has been found to be critical for avoiding agglomeration [8]. Slurries prepared using this scheme exhibited higher viscosity (for the same weight percentage of the abrasive particles) and no decantation (slurry settling) after several months. On the other hand, the same slurries prepared using simple mixing showed agglomeration and abrasive separation within a few hours of preparation and possess low viscosity. The weight percentage of abrasive in the slurries was in the range of 10 – 20%. It is worth mentioning that the viscosity and suspension properties of the water based alumina slurries could be altered by addition of glycerine.

Several batches of agglomerate-free alumina slurries (the Baikolox CR-series) with abrasive sizes of 1, 0.3 and 0.05 μm were investigated. The abrasive weight percentages in these slurries were in the range of 10 – 14%. These high viscosity slurries contain some suspending agent along with glycerine, water and abrasive. The slurries remain suspended for a relative long period of time (few weeks to several months). They were used without any further modification in our experiments. The agglomerate-free nature of these slurries and
superior suspension properties result from the intricate slurry preparation procedures including high shear mixing [9].

POLISHING EXPERIMENTS AND RESULTS

The slurry properties have been found to influence the removal rate and micro-scratching. The removal rates for the agglomerate free slurry was found to be at least four times higher than the same slurry containing agglomerates. The high removal rates eliminates the need for lapping using large abrasive particles (12 – 30 μm), which gives rise to deep scratching and sub-surface damages and hence is of utmost importance for fragile alloys such as GaInSb and GaInAsSb. Typical removal rates with the Baikalox 1 μm slurry was found to be similar to that obtained using an agglomerated alumina slurry with 12 – 14 μm abrasives. Apart from the high removal rates, the agglomerate-free 1 μm slurry gives rise to a mirror smooth high quality surface as opposed to the usual lapping slurries. Hence, the number of polishing steps to get the final device grade surface reduces considerably. In our experiments, we have been able to reduce the total number of lapping and polishing steps to merely two: the first step was performed using the Baikalox 1 μm slurry followed by the final step with either a colloidal alumina or silica slurry with 0.02 – 0.05 μm abrasives. The entire process of lapping and polishing is thus simplified and yields superior result compared to the conventional multi-step process.

With the agglomerate-free slurries, the removal rates are less dependent on the pad material. In fact, softer pad such as velvet polished at a faster rate than some of the hard pad like Rodel IC-1400. The influence of pad material and structure has been found to be critical in maintaining flatness. The Hard pads such as Chemo-Tex and IC-1400, are suitable for superior flatness; however, they lead to high density of micro-scratches as shown in the AFM image in Fig. 1. This has been solved by employing a final short polishing step on soft pads like velvet or nylon, which eliminates all microscopic scratches and is essential for obtaining defect free surfaces with atomic flatness. The polishing cycle on the soft pad needs to be short in order to maintain wafer flatness. Typical polishing times on velocity pad were in the range of 2 – 5 minutes. The AFM image of GaSb single crystal polished using the IC-1400 pad followed by velvet (with the Baikalox 1 μm alumina slurry) is shown in Fig. 2. The RMS roughness is close to that obtained with an elemental semiconductor such as silicon.

The effect of slurry agglomeration on the surface characteristics of Ga0.85In0.15Sb is shown in Figs. 3 and 4. These wafers were polished using agglomerated (Fig. 3) and agglomerate-free (Fig. 4) 1 μm alumina slurries. The pads used were Rodel IC-1400 followed by velvet (as in Fig. 2). Multiple deep scratches could be seen in the AFM image of Fig. 3. Moreover, the removal rate was found to be at least 2-3 times slower than what was observed with the agglomerate-free slurry (Baikalox).

In the present study, device grade atomically flat surfaces of both GaSb and GaInSb have been obtained using an optimized three step lapping and polishing cycle. In the first step, the agglomerate-free 1 μm alumina slurry was used to remove slicing ridges on the wafer until a flat surface is obtained. This step is usually performed on a rigid pad such as Rodel IC-1400 or Nylon. Typical lapping times depends on the non-uniformity of the wafer thickness. In this study, the lapping time was in the range of 30 – 60 minutes. The lapped wafer exhibit a mirror smooth surface, but possess multiple fine scratches. To eliminate the fine scratches, a short polishing cycle (~ 5 minutes) is performed using the same 1 μm slurry on a velvet pad. After this step, no visible scratches could be seen on the wafer surface. However, shallow scratches could be still seen in the AFM images (similar to that in Fig. 2). To eliminate the
FIG. 1 AFM image of GaSb polished with Baikowski-Universal Photonics alumina (1 μm) slurry on Rodel IC-1400 pad

RMS Roughness: 2.62 nm

FIG. 2 AFM image of GaSb polished with Baikowski-Universal Photonics alumina (1 μm) slurry on Rodel IC-1400 followed by velvet pads

RMS Roughness: 0.25 nm
FIG. 3 AFM image of Ga$_{0.85}$In$_{0.15}$Sb polished using an agglomerated alumina ($1 \, \mu m$) slurry on Rodel IC-1400 followed by velvet pads.

RMS Roughness: 1.6 nm

FIG. 4 AFM image of Ga$_{0.85}$In$_{0.15}$Sb polished using Baikowski-Universal Photonics alumina ($1 \, \mu m$) slurry on Rodel IC-1400 followed by velvet pads.

RMS Roughness: 0.1 nm
fine scratches, we explored a variety of water and oil based colloidal silica and alumina slurries with pH in the range of 3.5 to 10.5. The abrasive particle sizes in these slurries were in the range of 0.02 – 0.05 μm. The slurries with high pH such as Glanzox (colloidal silica with pH: 10.5) was found to be suitable for achieving an atomically smooth surface with no scratches. The final step is usually performed for 5 minutes on a velvet pad.

The lapping and polishing strategy developed in this work is quite general and applicable to other multi-component alloys. Similar results have been obtained in preliminary work on Chemical Mechanical Planarization of Liquid Phase Epitaxial grown AlGaAsSb/GaSb and GaInAsSb bulk substrates polishing experiments using agglomerate-free diamond based and alumina based slurries, respectively.

Post CMP cleaning is critical in obtaining atomically flat surfaces. For blanket wafer surfaces, brush cleaning is widely used [2]. From AFM analysis of polished wafer surfaces, it has been observed that some of the fine abraded or slurry particles do not detach even after brush cleaning. A simple way of cleaning the polished surfaces using DOWFAX surfactant on a velvet pad has been found to be effective [8].

SUMMARY

A new technique for lapping and polishing fragile and soft multi-component semiconductor alloys has been developed using agglomerate-free slurries. Damage free substrate preparation of GaInSb has been possible using this methodology. Atomically flat surface of GaInSb has been achieved by using agglomerate-free polishing slurries. It has been found that good slurry dispersion eliminates scratches and sub-surface damage, while increasing the removal rate drastically. High shear mixing has been found to be critical for obtaining agglomerate free slurries.

REFERENCES