BRIEF REPORTS AND COMMENTS

This section is intended for the publication of (1) brief reports which do not require the formal structure of regular journal articles, and (2) comments on items previously published in the journal.

Surface roughness-induced artifacts in secondary ion mass spectrometry depth profiling and a simple technique to smooth the surface

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We report on secondary ion mass spectrometry (SIMS) depth profile artifacts induced by surface roughness. The formation of a $TiSi_2$ film at 800 °C on a boron doping superlattice (DSL) of Si results in a rough (22.0 nm root mean square) interface between the film and Si DSL. After chemically etching off the $TiSi_2$ film, SIMS information is collected while sputtering through the surface of the Si DSL. The resulting depth profiles are irreproducible due to the initial surface roughness. By chemo-mechanically polishing the Si prior to SIMS analysis, we smooth the surface and the resulting depth profiles are then consistent and easily explained by current diffusion theory.

We have recently reported on the diffusion of B and Sb in Si doping superlattices (DSLs) after the formation a $TiSi_2$ film.[1] In this report we show that sputtering through a Si surface roughened by a $TiSi_2$ film, while collecting secondary ion mass spectrometry (SIMS) information, produces artifacts in the depth profiles of dopants. A simple chemo-mechanical polish (CMP) of the Si can smooth the surface, as shown by atomic force microscopy (AFM), SIMS, and transmission electron microscopy (TEM), resulting in accurate and consistent depth profiles. This technique has been demonstrated in silicided poly silicon contacts, but SIMS resolution was still limited by the polysilicon.[2]

Doping superlattices were grown by low temperature molecular beam epitaxy (LT MBE).[3] They consisted of B doping spikes with 10 nm widths, and peak centers spaced 100 nm apart. Each B spike had a concentration of $1.3\times10^{19}/cm^3$. The shallowest spike was capped with 50 nm of Si. Prior to Ti deposition, all samples were cleaned in TCA, acetone, methanol, DI water, 10:1 buffered oxide etch, and DI water. The samples were loaded into a sputtering chamber which was pumped down to a base pressure of 2×10^{-9} Torr. The chamber was backfilled with argon to a pressure of 2×10^{-3} Torr and 30 nm of 99.995% purity Ti was sputtered onto the surface. The samples were annealed in forming gas (85% N_2+15% H_2) at 800 or 840 °C for 1 h. The resulting $TiSi_2$ film was removed by etching in dilute HF (25%) for 6 min. The cross sectional TEM (XTEM) measurements were performed at 200 keV with the incident beam parallel to the [110] zone axis using bright field images. Tapping mode was used in AFM measurements, with a resolution of 0.01 nm. Dopant concentration profiles were obtained by a Physical Electronics PHI 6300 quadrapole secondary ion mass spectrometer (SIMS) with 2 keV O_2^+ ions for B profiling (B^+ ion). The sputtering rate was approximately 0.37 nm/s and the incident angle was 60°.

Figure 1 shows the Si surface after the $TiSi_2$ has been etched off. The sample annealed at 800 °C has a root mean square (rms) roughness of 22.0 nm. The rough surface comes from $TiSi_2$ grains faceting to present low interfacial energy planes to Si.[4] The resultant SIMS depth profiles of the B DSL from this sample are shown in Fig. 2(a) as compared to the sample before the film was deposited and annealed. The annealed SIMS depth profiles are from three different areas on the same sample. Note that some Si is consumed in the $TiSi_2$ film and is subsequently lost when the $TiSi_2$ is etched off. A more detailed profile of just the third doping spike is shown in Fig. 2(b). The three profiles are obviously inconsistent and the integrated counts of the dopants do not agree. If we CMP the underlying Si surface after the $TiSi_2$ film has been etched but before SIMS profiling, the Si surface is now smooth (Fig. 3) with rms roughness of 0.1 nm. From a comparison with Fig. 2(a), about 140 nm of Si has been removed by the polishing process.

Using this procedure, many samples that had rough interfaces were polished before SIMS depth profiling.[1,5] All produced consistent and repeatable depth profiles of the dopants (Fig. 4). Note that the shallower slope on the deeper edge (as measured from the surface) of the doping spikes is not due to

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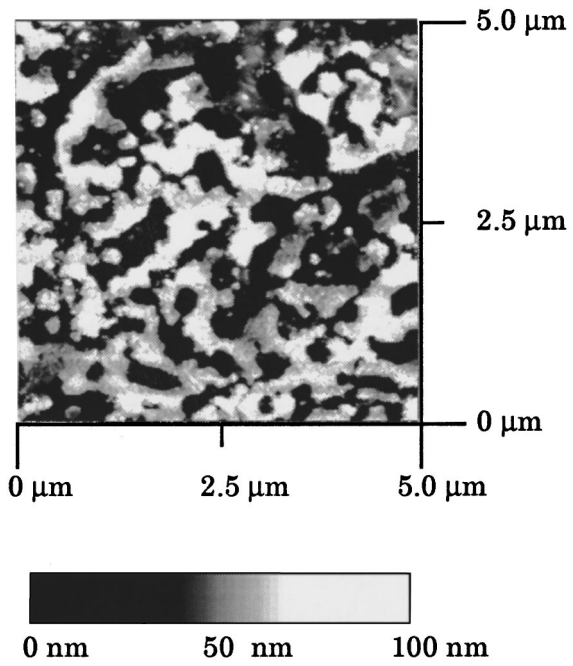

FIG. 1. Atomic force microscope micrograph of the underlying Si surface after chemical removal of the $TiSi_2$ film.

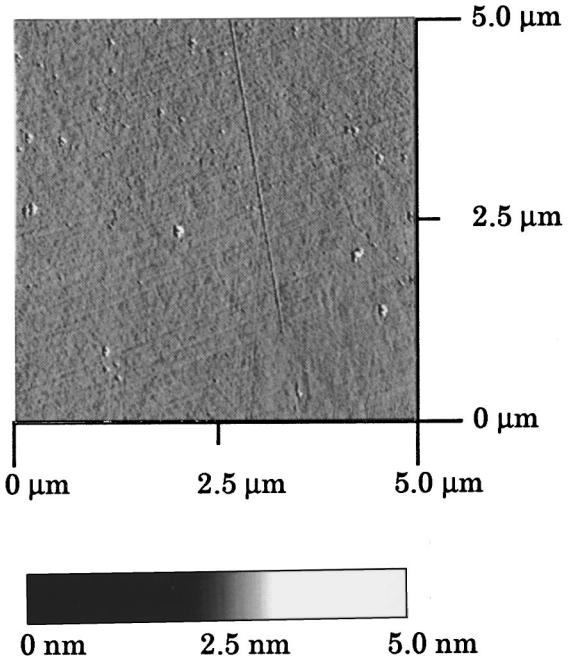

FIG. 3. Atomic force microscope micrograph of the underlying Si surface after chemical removal of the $TiSi_2$ film and chemo-mechanical polish.

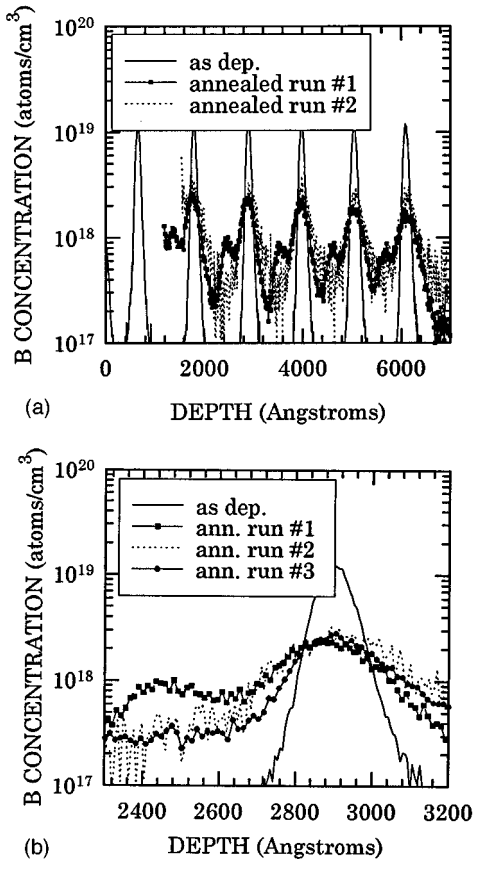

FIG. 2. (a) SIMS depth profiles of the silicided B doping superlattice after chemical removal of the $TiSi_2$ but without a chemo-mechanical polish of the surface. (b) Detailed graph of the third spike; three SIMS depth profiles were taken on different spots on the same sample.

surface topography but is a consequence of ion-mixing effects.[6,7]

Besides smoothing the surface, this CMP procedure can be used to remove Si from a wafer. The procedure was developed on samples that had a buried dislocation loop layer formed in a Cz Si (100) wafer (p type, 5–10 Ω cm) after an ion implant (1×10^{15} Ge^+ ions/cm^2 at 180 keV) and recrystallization anneal (800 °C for 1 h in flowing N_2). This loop layer is approximately 30 nm in width and is 240 nm deep [Fig. 5(a)]. The polishing agent was SYTON™ (South Bay Technology), a colloidal silica grit with an average particle size of 0.05 μm, dispersed on a felt pad. The wafer was diced into 20×20 mm samples. Samples were attached to a South Bay Technology Model 150 lapping fixture using crystal bond. A force of 3.2 N was exerted on the samples,

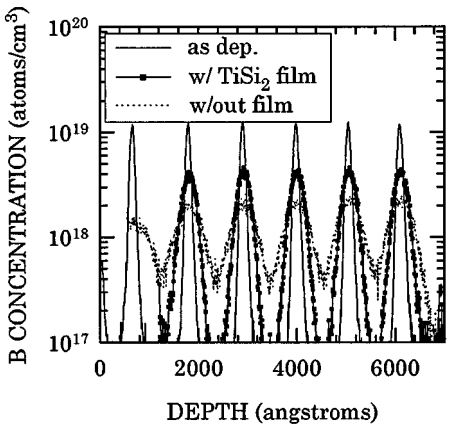

FIG. 4. SIMS depth profile of a silicided B doping superlattice annealed at 840 °C that has been CM polished after chemical removal of the $TiSi_2$ film.

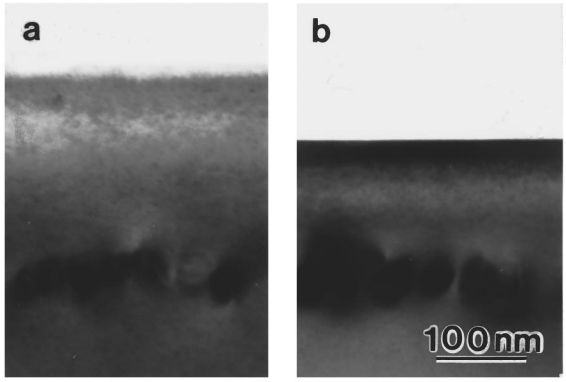

FIG. 5. XTEM micrographs of Si with a dislocation loop layer (a) before CM polishing and (b) after 7300 cm travel on the pad during CMP.

and the velocity was 0.2 m/s. Cross sectional TEM measurements from the center of the sample revealed how much Si had been removed by the position of the loop layer relative to the surface [Fig. 5(b)]. A plot of the removal rate is shown in Fig. 6. Atomic force microscopy plots on the polished surfaces are similar to Fig. 3, showing the surface remains smooth even with large amounts of polishing. An estimate of how evenly the Si was removed over the area of the sample can be made from the TEM measurements. If we assume the extreme case, that the material is removed in a ''wedge'' shape from the sample that has been polished the most (Fig. 6), we estimate the angle of the polished surface to be no greater than 2 s from the initial surface.

Ignoring the surface roughening, one could interpret the depth profiles in Fig. 2 to show an enhancement of 18× in the diffusion of B. A simple measure of the diffusion length at $3\times10^{18}/\text{cm}^2$ in Fig. 2(b) gives a $\sqrt{2Dt}$ value of 34 nm. This results in diffusivity estimate of 1.6×10^{-15} cm^2/s for the unpolished sample versus 8.9×10^{-17} cm^2/s that was measured in a sample annealed at 800 °C for 1 h without a $TiSi_2$ film.[1] Hence, ignoring the surface roughening would lead one to conclude erroneously that there is an enhancement of B diffusion in this case. The presence of the $TiSi_2$ film actually results in retarded B diffusion,[1] hence the sharper spikes in Fig. 4 in the silicided sample that has been analyzed after CMP. Since the apparent widening of the dopant spike due to the rough interface is temperature independent, ignoring the interface roughening would lead one to arbitrarily large enhancements by simply dropping the diffusion temperature. Since Sb has a smaller diffusion length in Si, this effect is even worse for diffusion studies involving Sb.

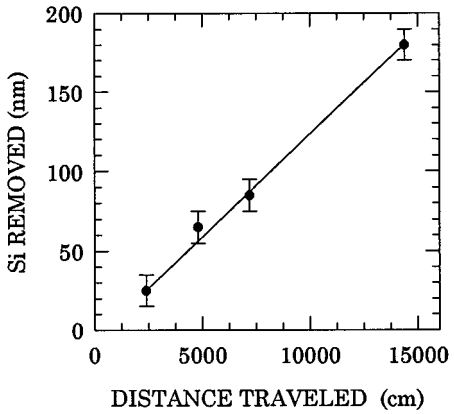

FIG. 6. Plot of the Si removed vs distance traveled on the polishing pad.

Indeed, a recent series of articles has claimed anomalous asymmetric diffusion of Sb in doping spikes after the formation of a Pd_2Si film at only 250 °C for 100 min.[8–10] A comment by Ronsheim and Tejwani[11] on the original article[8] suggested that the depth profile may have been the result of artifacts from either the roughened surface or from sputtering-induced roughness. Honeycutt and Rozgonyi[12] saw similar anomalous diffusion behavior of Sb in Si after formation of $TiSi_2$ film at 800 °C for 5 min, which lead them to conclude $D_A/D_A^* = 10^7$, where D_A is the diffusivity of Sb in Si with a $TiSi_2$ film and D_A^* is the diffusivity of Sb in Si without a $TiSi_2$ film. However, this measurement was later attributed to depth profile artifacts due to a rough interface and concluded to be erroneous.[13]

It is imperative that even minor amounts of interface roughness induced by the silicidation reaction be removed and the surface smoothed before SIMS depth profiling. This is true for any sputter depth profiling technique such as SIMS or Auger spectroscopy, or for a technique such as Rutherford backscattering, all of which rely on a uniform distance between the surface and the buried dopant that is being profiled. The results presented here, show clearly that sputtering artifacts, and not anomalous diffusion behavior, explain the results presented earlier.[8–10,12]

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