In this study, the development of post-chemical mechanical polishing (CMP) protocols for cleaning abrasive nanoparticles from In$_{0.53}$Ga$_{0.47}$As surfaces was systematically analyzed. Abrasive silica nanoparticles (130 and 289 nm) were intentionally deposited onto InGaAs surfaces. Various concentration ratios of chemical etchants such as HCl and H$_2$O$_2$ were used to control material loss and surface oxides of InGaAs. The optimal concentration ratio of the HCl/H$_2$O$_2$ cleaning solution exhibited 40% particle removal efficiency (PRE). Application of megasonic (MS) cleaning improved the PRE to 80%. To prevent particle re-contamination, ammonium dodecyl sulfate (ADS) was used as an anionic surfactant to modify surface charge in the InGaAs substrate. Addition of surfactant further improved the PRE to over 96%. Optimal cleaning of InGaAs surfaces was achieved with a combination of HCl/H$_2$O$_2$, surfactant, and MS.

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The surface oxidation of InGaAs was examined before and after surface etching by X-ray photoelectron spectroscopy (XPS, Sigma Probe, Thermo, UK). The XPS experiment was performed in a UHV multipurpose surface analysis system operating at base pressures of $<10^{-9}$ mbar. The photoelectron spectra were excited by an Al $K_{\alpha}$ (1486.6 eV) anode operating at a constant power of 100 W (15 kV and 10 mA). During spectral acquisition, the constant analyzer energy (CAE) mode was employed at pass energy of 30 eV and a step of 0.1 eV. Based on the XPS results, the optimal concentration of HCl/H$_2$O$_2$ solution was chosen to minimize the oxidation on the InGaAs surface.

For the particle cleaning experiment, silica particles (130 nm or 289 nm) were suspended in DIW and deposited on the InGaAs substrate to intentionally contaminate the surface. 100 $\mu$L of the particle suspension was spread over the substrate surface, and after a 5-min interval, the substrate was dried using nitrogen gas. These contaminated coupons were aged for 6 hours in a class 10 clean room and then particle cleaning was performed using the optimized concentration of HCl/H$_2$O$_2$ solution. To further enhance the cleaning efficiency, a small amount of ADS was added to the HCl/H$_2$O$_2$ solution to control the zeta potential and etch rate. The concentration of ADS was varied from 0.001 to 0.1 mM to keep solutions well under the critical micelle concentration of ADS of 6.7 mM. Zeta potential (ELS-Z, Otsuka Electronics Co, Japan) was measured to understand the interaction between the InGaAs surface and the silica particles with and without ADS in acidic solutions. This instrument has a platinum alloy electrode to measure zeta potential, so it is immune to the issues peroxide based solutions. A batch megasonic cleaning process was also employed to increase the 130-nm silica particle removal efficiency by using megasonic power of 600 W and a frequency of 1 MHz.

The InGaAs surface contamination by silica nanoparticles was measured in two different ways. Samples with 289-nm silica particles before and after nanoparticle contamination and post-cleaning were analyzed using a dark field optical microscope (LV-100D, Nikon, Japan) with a Hg lamp of 130 W power to determine the particle count. This optical microscope tool consists of an X-Y auto stage (ProScan II, PRIOR Scientific, USA) and object lenses (× 5, 10, 20, 50 and 100) with particle image analysis software (Image-pro Plus, Media Cybernetics, UK). This configuration made it possible to count the particles at the same position before and after cleaning. The wafer with 130-nm silica particle contaminants at different stages of the experiment was analyzed by FE-SEM (MIRA3, TESCAN, Czech) with particle image analysis software (Image J, 1.52e, National Institute of Health, USA). Once particle counts were obtained for the samples of either 130- or 289-nm silica particles, the PRE was calculated according to

\[
\text{PRE} \, (\%) = \frac{n_i + n_b - n_f}{n_i + n_b} \times 100 \, (\%) \quad [2]
\]

where $n_i$ is the initial particle count of the as-received wafer, $n_b$ is the particle count of the intentionally contaminated wafer (using either 130- or 289-nm silica particles), and $n_f$ is the particle count after the cleaning experiment.

### Results and Discussion

Various concentrations of HCl from 0.001 M to 0.05 M and H$_2$O$_2$ from 0 M to 0.01 M were prepared and pH value of each solution is reported in Table I. All solutions of HCl are at acidic pH and no significant changes in pH values were observed with addition of peroxide to the solutions. Etch rate experiments were performed at these concentrations and using positive PR-patterned InGaAs coupons. The etch depth of InGaAs was measured using AFM and the etch rate (Figure 1) was calculated from the etch depth and time. No significant etching occurred when InGaAs was exposed to HCl solution without H$_2$O$_2$.

| HCl(mM)/H$_2$O$_2$(mM) | 0.001 | 0.005 | 0.01 | 0.05 |
|------------------------|-------|-------|------|------|
| 0                      | 3.44  | 2.67  | 2.34 | 1.60 |
| 0.001                  | 3.45  | 2.66  | 2.34 | 1.61 |
| 0.005                  | 3.46  | 2.66  | 2.33 | 1.61 |
| 0.01                   | 3.44  | 2.69  | 2.35 | 1.60 |

This result confirms that HCl alone cannot etch the InGaAs surface without a chemical oxidizer. Two etching regimes can be distinguished at all HCl/H$_2$O$_2$ conditions. The etch rate increases rapidly when the ratio of HCl to H$_2$O$_2$ concentration increases to 1:1. However, as this ratio increases beyond 1:1, the etch rate of InGaAs decreased. This phenomenon can be explained by the reaction between Cl and OH groups on the InGaAs surface. In the presence of H$_2$O$_2$, the InGaAs surface is terminated by OH groups. If the HCl concentration is higher than that of H$_2$O$_2$, the OH groups on the surface of InGaAs are replaced by Cl$^-$ through nucleophilic attack and it forms stabilized bond with InGaAs.\(^{16,18}\) D.H. van Dorp et al. tested etching of InGaAs surface using HCl and HCl/H$_2$O$_2$ solution and mentioned that at high HCl concentration the surface OH groups are replaced due to nucleophilic attack by Cl$^-$ on the group III and V elements.\(^{18}\) The weaker electronegativity of Cl with respect to O leads to a stabilization of the back bond.\(^{18}\) Hence, the stabilized surface decreases the etch rate of InGaAs with high concentration of HCl than H$_2$O$_2$.

To examine the effect of HCl/H$_2$O$_2$ etching on the surface oxide composition, the InGaAs substrates were analyzed using XPS. The binding energies of each element and the oxide composition of InGaAs before and after HCl/H$_2$O$_2$ treatment are given in Table II. Figure 2 shows the XPS spectra of InGaAs etched with two concentrations of HCl (0.1M, 0.001M) with 0.01M H$_2$O$_2$ for 1 minute. The oxide composition of as-received InGaAs was taken as a reference (Table I). Indium is active in forming the oxide in the form of In$_2$O$_3$.\(^{18}\) The oxide composition (In$3d_{5/2}$ peak) was increased at low concentration HCl (0.001M) etching compared to the reference because the oxidation rate is higher than etching in the presence of high concentration of H$_2$O$_2$. And it can be seen that etching with a high concentration of HCl (0.1M) reduced the oxide content. In this condition, oxidized surface of InGaAs was replaced by Cl$^-$ through nucleophilic attack and stabilize the surface. Hence, it was confirmed that the higher concentration of HCl compared to H$_2$O$_2$ reduces oxide formation.

The results of etch rate and XPS analysis, suggest that a relatively high concentration of HCl (0.05 M) and low concentration of H$_2$O$_2$ (0.005 M) is optimal for InGaAs cleaning to achieve the minimum material loss with controlled oxide formation. As mentioned earlier, HCl/H$_2$O$_2$ solutions are preferred etchants of InGaAs due to the low
Table II. Binding energy and oxide composition of InGaAs before and after HCl and H$_2$O$_2$ etching.

| Element          | Chemical state | Binding energy (eV) | InGaAs (as received,%) | HCl$>$H$_2$O$_2$ treated (%) | HCl$<$H$_2$O$_2$ treated (%) |
|------------------|----------------|--------------------|------------------------|-------------------------------|------------------------------|
| Indium (In3d$5/2$) | In$_2$O$_3$    | 445.0              | 21.28                  | 33.54                         | 18.79                        |
|                  |                | 452.0              |                        |                               |                              |
| Gallium (Ga3d$5/2$) | Ga$_2$O$_3$   | 20.5               | 23.43                  | 25.25                         | 21.53                        |
| Arsenic (As3d$5/2$) | As$_2$O$_5$   | 44.1               | 33.80                  | 34.47                         | 31.01                        |

Figure 2. 3d core level XPS spectra of InGaAs surface at different conditions (a) as received (b) treated with lower concentration of HCl than H$_2$O$_2$, and (c) treated with higher concentration of HCl than H$_2$O$_2$. The solid (black) lines are XPS experimental data and the dashed lines (blue and red) are numerical fitting results.

Figure 3. InGaAs etching mechanism in various concentration ratios of HCl and H$_2$O$_2$ cleaning chemicals.
degree of anisotropy in their etching. Three types of etching mechanisms were proposed based on the concentration ratio of HCl and H$_2$O$_2$ as represented in Figure 3. The etching mechanism includes two major processes: first a self-limiting oxide is grown on the surface after which the oxide layer is removed selectively. The first etching step involves chemical hydroxylation by H$_2$O$_2$ on the InGaAs surface. It is generally believed that the formation of the first hydroxide layer at the surface is a slow step in the oxidation process as shown in Figure 3. At higher HCl than H$_2$O$_2$ concentration, the replacement of OH groups by Cl$^-$ ions on the polarized InGaAs surface is faster than the rupture of the back bond. The weaker electronegativity of Cl with respect to O leads to stabilization of the back bond, and hence the InGaAs etch rate is decreased. Further oxidation of the back bonds results in the formation of surface products that contain OH which enhances fast surface dissolution. At comparable HCl and H$_2$O$_2$ concentrations, the surface is mainly terminated with OH groups. The larger back bond destabilization results in a higher etch rate, and the dissolution rate of surface products is very fast. At lower HCl than H$_2$O$_2$ concentration, the dissolution rate is lower than the rate of oxide formation, because oxide dissolution can be enhanced only in the presence of sufficient HCl concentration.

A high concentration of HCl (0.05 M) and low concentration of H$_2$O$_2$ (0.001 M) was selected as the optimal cleaning solution for particle removal from InGaAs surfaces. Figure 4 shows the PRE as a function of time for 289-nm silica samples cleaned using the optimized HCl/H$_2$O$_2$ solution. It was observed that the PRE was increased with increased etching amount and cleaning time. PRE reached about 80% at the 3-min cleaning time, corresponding to 1.2-nm surface etching. However, particles were recontaminated on the surface and it was confirmed by comparing the change in positions of the particles before and after InGaAs particle cleaning using optical microscope measurement as shown in Figure 5. This recontamination may be due to the attractive force between the opposite zeta potentials of the silica particles and InGaAs surface.

Silica particle recontamination can be prevented by understanding the charge interaction between the particles and the substrate at various pH. This interaction can be controlled through the addition of ADS surfactant to the cleaning solution. The measurement of zeta potential of particle and substrate surface with and without ADS surfactant is shown in Figures 6 and 7. Figure 6 shows the decrease in negative zeta potential value of silica particle with increase in the pH, with no significant difference observed with or without ADS. This result indicates that the adsorbed surfactant on the particle surface does not significantly affect the zeta potential of the particles. Figure 7 shows the zeta potential values of InGaAs surface with and without ADS. The ADS addition to the cleaning solution decreased the zeta potential.
Figure 8 shows the effect of surfactant concentration on the etch rate of InGaAs in HCl/H$_2$O$_2$ cleaning solution. There was no significant difference in the etch rate when ADS was added from 0 to 0.001 mM in the HCl/H$_2$O$_2$ solution. The InGaAs etch rate decreased from 0.4 to 0.1 nm/min as the ADS concentration was increased from 0.001 to 0.005 mM, and remained constant at over 0.005 mM ADS. At concentrations higher than 0.005 mM ADS, the anionic surfactant is completely adsorbed on the InGaAs surface, which decreases the InGaAs etch amount. Therefore, the required minimum amount of ADS surfactant is 0.005 mM for effective cleaning of InGaAs surface, which will modify the zeta potential value of the substrate as well as decrease the etch depth.

Figure 9 shows the PRE of 130 and 289 nm silica particles cleaned via etching only and electrostatic repulsion only. The results show that 130 nm particles exhibited lower PRE than 289 nm particles when cleaned with HCl/H$_2$O$_2$ solution. On the other hand, the ADS solution alone shows no noticeable removal of the contaminant silica particles. This result is consistent with the known difficulty of removing silica nanoparticles with electrostatic repulsion alone. Therefore, to remove silica nanoparticles effectively from the InGaAs surface, both the etching solution and ADS are necessary. Figure 10 shows the PRE as a function of ADS concentration in HCl/H$_2$O$_2$ solution. The PRE increases dramatically with ADS concentration until 0.005 mM. Beyond 0.005 mM ADS, PRE continued to increase with ADS concentration, but at a lower rate. This dramatic change is due to the combined effect of surface etching and electrostatic repulsion between the surface and the particles. The zeta potential of the silica particles and InGaAs substrate was measured using the HCl/H$_2$O$_2$ cleaning solution with and without ADS concentration of 0.005 mM. The zeta potential of InGaAs substrate was changed from +8.9 to −4.8 mV and silica particles were changed slightly to −4.5 mV. Most of the 289-nm silica particles were removed (PRE 98%) in HCl/H$_2$O$_2$ with ADS (≥0.01 mM) solution. However, in the case of 130-nm silica particles, the PRE using the same cleaning solution is only around 85%. To increase the PRE of 130-nm silica particles, megasonic cleaning was adopted along with HCl/H$_2$O$_2$ with ADS cleaning solutions. Effective particle removal will therefore be achieved by using the combination of chemical interactions and physical force. Particle removal tests were performed using batch type megasonic cleaning.

Figure 11 shows the PRE results in various mixtures of HCl/H$_2$O$_2$ solutions with and without ADS. In the case of HCl/H$_2$O$_2$ with
megasonic cleaning, the PRE is higher than using HCl/H2O2 solution alone. The simultaneous chemical effect of HCl/H2O2 and ADS (0.005 mM) along with megasonic cleaning removes most of the 130-nm silica particles from the InGaAs surface, with the PRE reaching around 96%. When megasonic cleaning was compared to the results without megasonic cleaning, PRE was increased by approximately 20%. This result might be due to the combined effects of megasonic, surface etching and repulsive force induced by ADS.

Roughness is a primary factor in determining the bond between an InGaAs substrate and a film to be deposited upon it, because a rough surface is known to be a major cause of interfacial voids. Thus, an effective cleaning process for InGaAs must not significantly alter its surface roughness. The surface roughness of InGaAs before and after cleaning was analyzed using AFM and the results are shown in Figure 12. The as-received InGaAs substrate shows initial roughness of about 3.38 nm. Cleaning InGaAs under various conditions resulted in no significant change in surface roughness compared to the initial value because the etch depth was too small to significantly affect the surface roughness. Hence, the formulated cleaning solution with megasonic is a suitable cleaning method for InGaAs substrates.

Conclusions

In this study, a post-CMP cleaning solution was formulated using HCl/H2O2 solution containing ADS surfactant for effective InGaAs surface cleaning. Varying the concentrations of HCl and H2O2 had a significant impact on the etch rate of the InGaAs substrate. Through etch rate and XPS analysis it was found that solution with a high concentration of HCl and a low concentration of H2O2 is most suitable for cleaning InGaAs with minimized material loss and minimized surface oxide. ADS was used to the solution to improve PRE. ADS decreased the zeta potential of the surface of InGaAs to more negative values which electrostatically repulsed the silica particles in the acidic conditions used for cleaning. Maximal removal of the 289-nm silica particles was achieved via cleaning with > 0.01 mM ADS in HCl/H2O2. However, chemical means alone were not effective at removing 130-nm silica particles from the InGaAs surface. To remove the 130-nm particles effectively, physical force via megasonic cleaning was applied in addition to the cleaning solution. The mixture of HCl/H2O2 and ADS with megasonic cleaning produced a PRE of about 96%. Finally, the surface roughness was evaluated using AFM before and after cleaning process. These measurements confirmed that there was no significant change in the surface roughness of the InGaAs as a result of the cleaning processes employed. Hence, it is concluded that the formulated cleaning solution with megasonic cleaning can be a suitable cleaning solution for post-CMP InGaAs substrates.

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