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Real-Time Molecular-Beam Epitaxy Flux Inspection Technique

A single-photon ionization time-of-flight mass spectrometric system allows for in-situ monitoring during semiconductor growth. 

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A technique for observing the gaseous species present during molecular-beam epitaxy of III-V, II-VI, and silicon semiconductors has been developed, utilizing single-photon laser ionization (SPLI) of gas phase species followed by time-of-flight mass spectrometric (TOF-MS) analysis of the resulting ions. The single-photon ionization probe results in greatly reduced fragmentation of the ions as compared to the more traditional electron-impact ionization sources standard in commercial quadrupole mass spectrometers. The reduced fragmentation greatly simplifies mass spectral interpretation. Additionally, this laser-based probe can interrogate both the species incident to and scattered from the semiconductor's substrate in real time during growth.

The apparatus is housed in an ultrahigh-vacuum chamber equipped with standard surface analysis instruments, including means for observing reflection high-energy electron diffraction (RHEED). This chamber is also equipped with molecular-beam effusive sources of both arsenic and gallium. A five-axis manipulator capable of radiative heating of the substrate positions the growth surface within the extraction region of the TOF-MS. This region is designed to allow unimpeded access of the effusive molecular beams and the RHEED electron beam to the sample, enabling probing of dynamical growth processes.

The laser beam passes near the center of the extraction region, in front—on the molecular-beam side—of, and parallel to, the substrate in a counterpropagating direction to the RHEED beam. The ions generated by the laser beam are then extracted up the flight tube of the TOF-MS and accelerated to a constant energy. The ions impinge on a focused mesh electron multiplier located at the end of the meter-long tube. Ions of different masses are distinguished based on their arrival times at the multiplier, following the ionizing laser pulse. The resulting mass spectra can be examined using a digital oscilloscope, or individual species can be followed in time through a computer interface.

The single-photon ionization probe laser utilizes 118-nm radiation, which provides sufficient energy (10.5 eV) to ionize, but not fragment, most of the species used in semiconductor epitaxy. This radiation is the ninth harmonic of the Nd:YAG laser and can be generated rather easily. The fundamental (1064 nm) output from a commercial Q-switched Nd:YAG laser is tripled to give 355-nm radiation using standard nonlinear optical techniques. The repetition rate of this laser is 10 Hz; an average laser pulse has approximately a 5-ns duration.

The third-harmonic radiation is then focused by a UV-grade fused-silica lens through a fused-silica window into a static gas cell containing a xenon-argon mixture. The 118-nm radiation is generated in this cell by a nonresonant tripling process. An LIF lens located in the cell then focuses the light through an LIF window and into the extraction region of the TOF-MS in the vacuum chamber, where the ionization occurs.

Typically about 35 mJ per pulse of the 355-nm radiation is focused into an approximately 25-percent mixture of xenon in argon at a total pressure of about 3200 Pa (about 24 Torr) in the gas cell, using a lens with a 50-cm focal length. This results in a conversion efficiency of approximately $1 \times 10^{-5}$, producing about 200 nJ per pulse of the 118-nm radiation in the extraction region to the TOF-MS. This quantity of ninth harmonic energy is sufficient to ionize and detect background molecules, such as As$_n$, in our system, at densities of less than $3 \times 10^{12}$ molecules/cm$^2$.

Species used in semiconductor epitaxy that have been detected thus far using this technique include Si, Ga, In, As$_n$, As$_2$, and As$_3$. Mass spectra of a molecular beam of As$_n$ recorded using the single-photon laser ionization time-of-flight technique show a greatly reduced level of fragmentation compared to multiphoton or electron impact ionization. For exam-
The A$_s^*$, A$_s^{-}$, and A$_s^{-}$ signals observed in the A$_s$ mass spectra obtained using the SPLI TOF-MS method are less than 0.1 percent (noise level), 0.4 percent, and less than 0.1 percent, respectively, of the A$_s^*$ signal. This can be compared with multiphoton ionization, using a focused beam of approximately 3 mJ per pulse of 266-nm radiation, the fourth harmonic of the Nd:YAG laser, of the same A$_s$ beam, which resulted in signal levels for the same species of approximately 40 percent (noise level), 415 percent, and 55 percent respectively of the A$_s^*$ signal.

The scattered and desorbed species present during epitaxial semiconductor growth can also be probed in real-time using this technique. For example, following the evolving arsenic fluxes during GaAs growth, the arsenic uptake can be observed during growth stages by noting the decreases in the A$_s^*$ and A$_s^{-}$ mass spectral signal levels. These uptakes correlate very well with the expected incorporation rates, which are based on the information obtained using the RHEED surface probe.

The SPLI TOF-MS technique's noninvasive probe allows almost continuous detection of the gas phase species present above the wafer's surface during the deposition process. The technique is capable of simultaneous measurement of multiple species in real-time, thus permitting possible feedback control of source fluxes. It can be utilized in situations where more traditional probes, such as RHEED, are not as useful, such as growth at high temperatures or growth on a rapidly rotating substrate. Work is currently in progress to extend the technique to use as a probe in dry-etch processes for silicon and in silicon molecular-beam epitaxy.

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