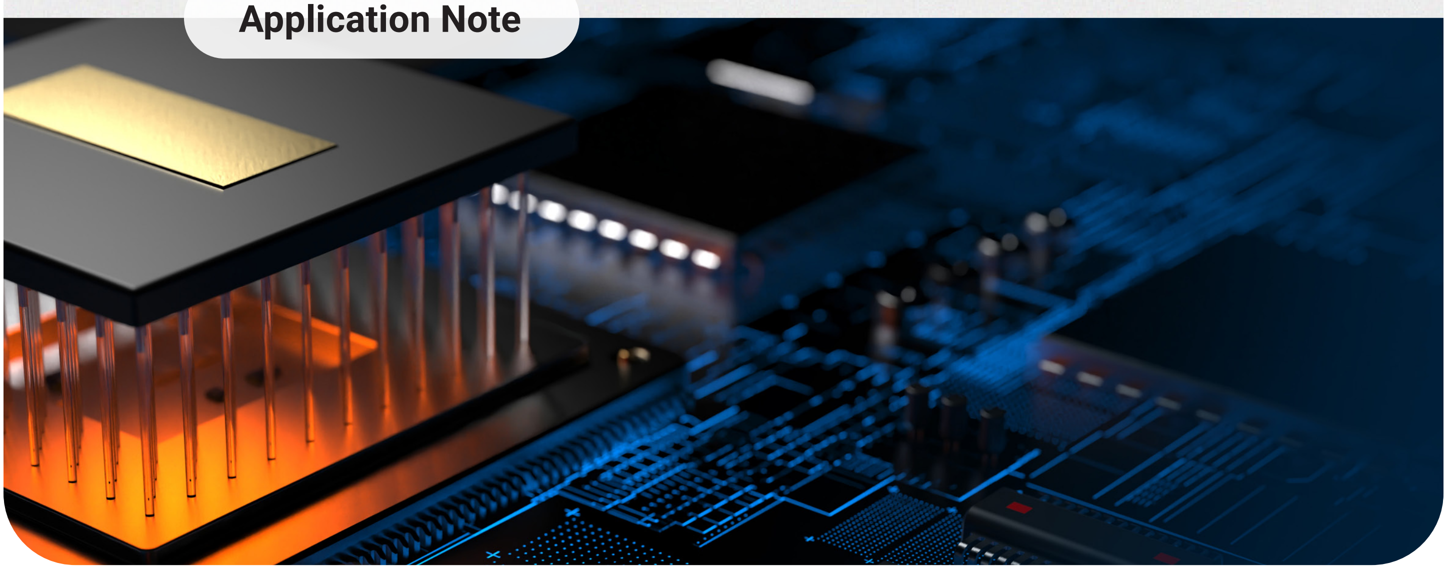
Real-Time Monitoring of Nanoparticle Synthesis in Semiconductor Processes using UV-Vis Spectroscopy

Application Note



KEYWORDS

- Metallic nanoparticles
- Nanoparticle synthesis
- Ocean HR high resolution spectrometer

TECHNIQUES

- Absorbance

APPLICATIONS

- Semiconductor processing
- Reaction analysis

Do you work with semiconductor processes where chemical reactions need to be monitored and controlled in real-time? The historical bottle- neck for process analysis typically has been the need to extract fluids and evaluate them away from the process, and then use that information to adjust some process parameters.

Often this is too slow to catch certain reactions or conversions before they can cause even larger problems down the line, so there is significant benefit to monitoring these fluids directly in-process and in real-time.

The beauty of miniature modular spectroscopy is that the measurement can be taken to the sample rather than the reverse of this. Modern broadband spectrometers report full spectra at millisecond speeds, providing instant insight into the status or condition of critical process fluids. Coupled with modern computers and analysis algorithms, this allows for rapid decision-making without users missing a beat. Ultimately, this insight can translate into massive savings in production costs and off-spec prod- ucts by catching problems before they become problems.

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Nanoparticles and Chemical Reaction Analysis

Here we look at a delicate chemical reaction relevant to the semiconductor industry, specifically the synthesis of metallic nanoparticles, and more specifically, gold nanoparticles.

As with many metallic nanoparticles the precursor here is a metal salt diluted in aqueous solution. By bringing this solution to a vortexed boil and introducing a reducing agent we will see the rapid formation of nanoparticles. Those with experience in this area have deve oped a “good eye” for which color indicates a proper reaction and final particle size, but a broadband spectrometer can precisely quantify this “color condition” and allow adjustment of parameters such as temperature, dilution, and reducing agent on the fly (Figure 1).

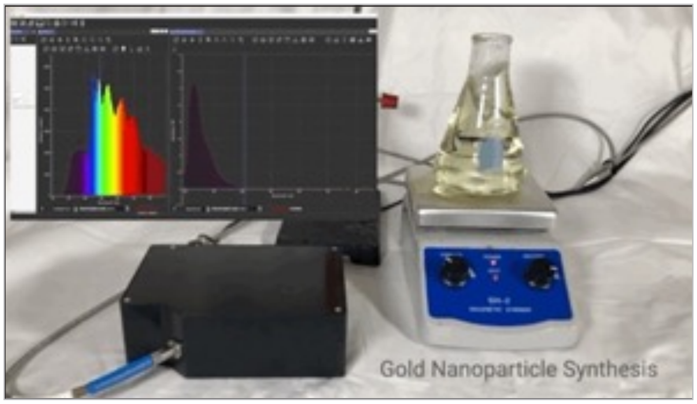

Figure 1. As nanoparticles form in a dilute metal salt solution, the color of the solution will change, indicative of particle size. A compact spec- trometer can precisely monitor this change for best results.

Experimental Setup and Results

A deuterium-halogen light source was used to provide analytical energies from the UV through the visible wavelengths, and the Ocean HR spectrometer platform allows sub-second spectral updates with customization around averaging and smoothing. There are several pieces of critical process information hidden within these dynamic spectra.

- Firstly, the concentration can be directly correlated to the extent of absorbance observed at the peak via Beer’s Law. This allows us to ensure quantitatively that the reaction has gone to completion and provided the expected yield of final product.
- Secondly, the specific wavelength where the peak occurs correlates to the average diameter of the resulting nanoparticles. This allows us to ensure qualitatively that the reaction produced the proper product without deviation from the desired particle size.
- Thirdly, the width or integral of this absorbance peak can be correlated to the distribution of other particle sizes that may be present, which again allows qualitative analysis of the final output. In Figure 2 we can see the slight peak shift over time from about 545 nm in the yellow trends down to 535 nm in the final red trends.

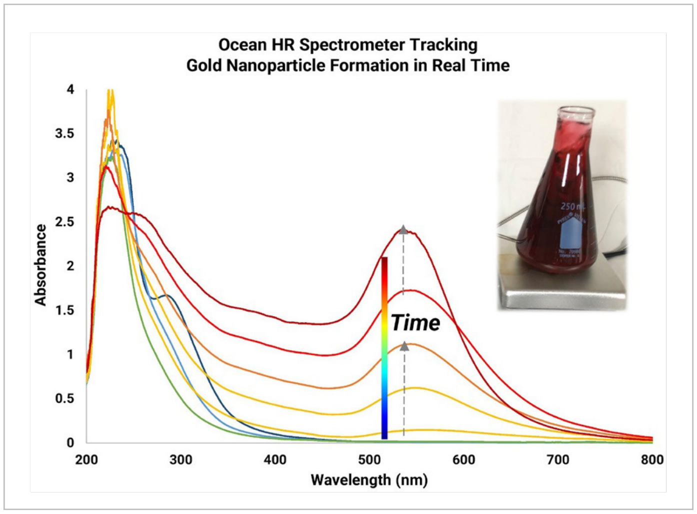

Figure 2. With a compact spectrometer, real-time absorbance measure- ments of nanoparticle solutions can provide a snapshot of the chemical reaction over time and by wavelength.

Summary

Having instant visibility for inorganic reaction processes can be the saving grace in scenarios where impurities may be present or some process parameter is a bit off. Nanoparticles and other nanostructures have been the fulcrum for rapid development of mobile devices with increasing power, and new green technologies such as directed self-assembly of block copolymers will need careful process analysis as they work to displace traditional top-down lithography approaches[1]. This continued development will also lead into more complex analysis of nanolayered materials where various types of semiconductors can tailor both near-and far-field optical properties[2].

Whatever direction you may be evolving your semiconductor processes, consider the benefits of real-time insight using rapid broadband spectroscopy to give you a competitive edge and peace of mind. Nanoparticles may be small, but in the world of mobile devices we know that when it comes to the little things there’s nothing bigger.

References

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2. Metallic Nanoshells with Semiconductor Cores: Optical Characteristics Modified by Core Medium Properties. Bardhan, Rizia, et al. s.l. : ACS Nano, 2010, Vol. 4. 10.1021/nn102035q.