In the past decades, increased human population and activities have introduced a large amount of pollutants into the environment. Various types of traditional analytical instruments were used for monitoring the emitted chemicals with low detection limit, high accuracy, and discrimination power. However, many of these methods are laboratory-based which require sample collection, transportation, extraction, and purification steps.

To make real-time on-site monitoring possible, miniaturized sensors with various integrated elements were developed. One of the most well-known strategies is to utilize nanostructured materials with enhanced sensing properties for those devices. For a majority of the current state of art devices, the synthesis of nanostructured materials and device integration are done separately, that is, “synthesis first and then integration” approach which involves two separate process steps. However, this approach comes with some disadvantages such as misalignment, contamination, as well as disconnection between nanomaterials and electrodes.

To overcome the aforementioned technical challenge, several synthesis methods were developed and validated for in-situ integration of nanostructured metal and metal oxide materials for environmental sensors in this work. The electroplating technique combined with photolithography was used to make the predefined metal electrodes. Then, with subsequent post-treatments, nanostructured metals and metal oxides could be produced in-situ and directly integrated in the electrodes without any extra transfer process steps.

In the development of a phosphate sensor, nanostructured Co and Co alloy electrodes were prepared by alloying and dealloying methods and a template (glass fiber filter paper and ZnO nanowires) assisted electroplating method. Both potentiometric and amperometric responses to phosphates in the concentration range of 10^-6 to 10^-2 M were obtained, showing an improvement in the detection limit and interference suppression. A sensing mechanism was proposed to elucidate the behavior of a Co electrode in aqueous solutions with varying pH conditions and optimum pH ranges for working devices were proposed.

For the flammable gas sensor development, the in-situ oxidation of Cu was utilized to form nanowires for sensing electrode fabrication. Multiple CuO nanowires were synthesized in-situ on the electroplated interdigitated Cu electrodes on a hotplate at 500 °C in air. The nanowires were successfully integrated as a sensing element into the device, forming bridges between two electrodes. The sensor’s behavior was characterized by a current-voltage measurement. Simple processing parameters could be utilized for controlling the electrode morphologies and determining the characteristics of contacts – Schottky or Ohmic at the electrode interface. A hypothesis was proposed to explain the transition phenomenon between Schottky and Ohmic contact modes, providing important baseline for future device design and fabrication. Finally, the fabricated sensor was tested for a flammable gas detection using saturated ethanol vapor at room temperature, which implicates a low power consumption gas sensor without elevating the sensor temperature unlike traditional gas sensors.