

STUDY OF GERMANIUM OXIDE FORMATION BY INFRARED EMISSION SPECTROSCOPY

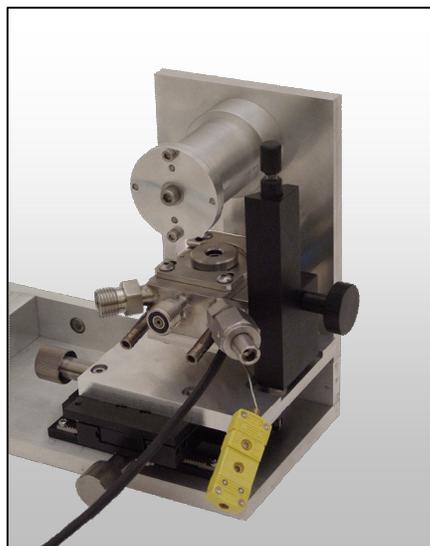


Figure 1. The Harrick Emission Accessory with the Harrick Heated Vacuum Chamber (HVC) being used as the sample holder.

INTRODUCTION

Infrared emission spectroscopy is a useful, if sometimes overlooked, spectroscopic technique with a number of unique advantages. One advantage is the ability to perform *in situ* heated experiments, that is, spectroscopic measurements can be made without interruption of processes occurring during the experiment. The Harrick Emission Accessory with the Harrick High Temperature Chamber (HVC) (see Figure 1) enables such experiments.

One application of this is the observation of thin layers forming on a substrate. Infrared emission is used here to observe

an oxide layer forming on a sample of germanium heated at 700 °C.

EXPERIMENTAL

Emission spectra were collected using the Harrick emission accessory on an emission port of an FTIR spectrometer equipped with an LN₂ cooled MCT detector. All spectra were signal averaged for 128 scans at a resolution of 8 cm⁻¹. The gain was set to 8, the aperture was set to 100% (fully open), and the spectral range was 4000-650 cm⁻¹. The germanium sample used was a fragment from an infrared-grade window, polished on both sides. The sample was placed directly on top of the HVC sample cup. The chamber was enclosed by its flat top window assembly, without a window installed, to stabilize the sample temperature. The sample was heated to 700 °C using the Harrick Temperature Controller. Single-beam spectra were taken every 2.5 minutes and ratioed (1:1) to a single-beam spectrum of an empty sample cup covered with a mask with a pinhole to let out the cavity radiation approximating a blackbody at that temperature (700 °C).

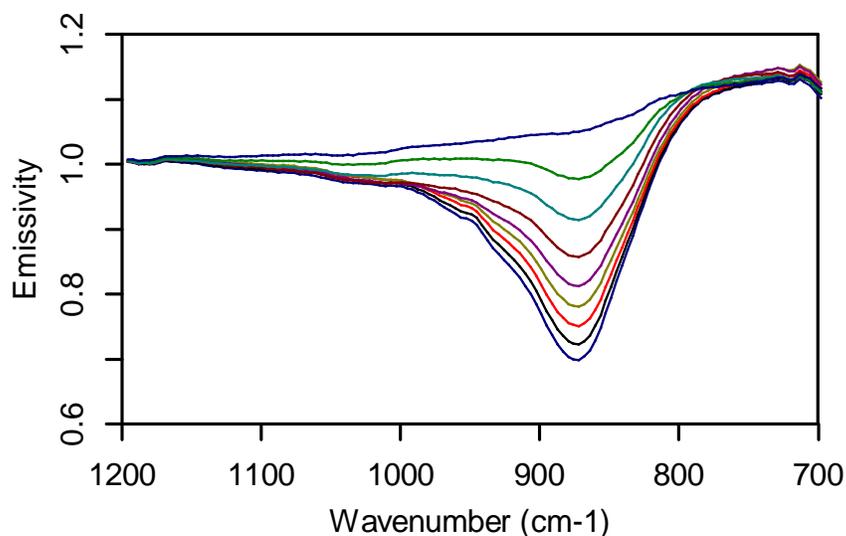


Figure 2. Spectra of germanium oxide taken at 700 °C in 2.5 minute intervals. As time elapses, the intensity of the peak increases.

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RESULTS AND DISCUSSION

The resulting spectra of the germanium sample over time at 700 °C are shown in Figure 2. The germanium sample held at 700 °C is being oxidized as it is exposed to the atmosphere, yielding the peak shown at around 870 cm^{-1} . As time progresses, the intensity of this peak increases as the amount of oxide increases.

Note that the bands in the spectra are downward, much like a transmission spectrum, rather than upward as should be expected from an emission spectrum. This is a result of examining a thick sample. The underlying Ge is emitting light which then transmits through the oxide coating on the surface. So the resulting spectra resemble transmission spectra.

Furthermore, the baseline has a slight positive slope which could result from a number of factors. The slope could be due to a temperature difference between the sample and the blackbody used as a background. Though the measured temperature was identical for both, it is possible that they were not at the same temperature due to differences in thermal conductivity differences. In addition, the temperature of the sample may have changed over time, as it was heated for an extended period of time. The

slope could also be due to spurious emission from the "blackbody" used for the background. A pinhole in polished 316 stainless steel disc was used. So there could be a contribution from the stainless steel, which does not even come close to a perfect reflector.

CONCLUSIONS

Infrared emission spectroscopy was used to observe the oxidation of a sample of germanium at 700 °C over time. As time elapsed and more germanium was oxidized, the intensity of the peak observed at around 870 cm^{-1} increased. Additional work is in progress to investigate some of the anomalies observed herein, but it is clear that infrared emission can be used to detect the formation of oxidative layers *in situ*.



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