HARRICK SCIENTIFIC PRODUCTS

No. 21139

OBSERVATION OF THERMOCHROMISM IN ZINC OXIDE BY UV-VISIBLE DIFFUSE REFLECTANCE SPECTROSCOPY

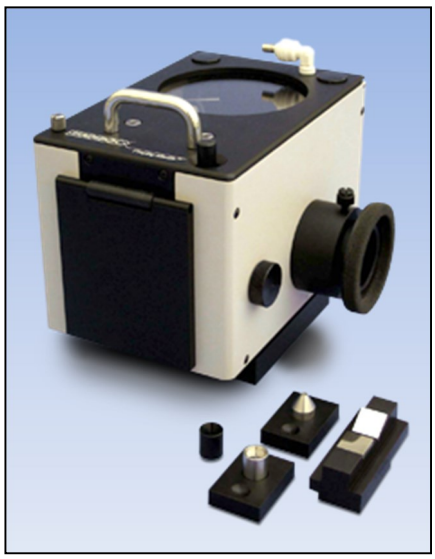

Figure 1. Harrick Praying Mantis UV-Vis Diffuse Reflectance Accessory.

Figure 2. Harrick High Temperature Reaction Chamber.

INTRODUCTION

Thermochromism is a phenomenon in which a material changes color upon a change in temperature. This indicates a shift in absorption bands in the UV-Vis region that can be observed via UV-Vis spectroscopy. Upon heating to temperatures 100 °C to 500 °C above room temperature, zinc oxide (ZnO) changes color from pure white to a pale yellow, becoming more yellow as temperature is increased. The cause of this change has been debated, and may be due to the incorporation of hydrogen[1], the loss of oxygen to the environment[2], a shift in the adsorption edge[3], or a combination of multiple effects, which may also depend on the length of time that the sample is heated.

The UV-Vis spectroscopic analysis of solid samples is most easily accomplished via specular or diffuse reflectance spectroscopy. For a rough powder such as ZnO, diffuse reflectance is the method of choice. This applications note details the use of the Harrick Praying Mantis™ diffuse reflection accessory in observing the shift in the visible absorption band of ZnO upon heating within the Harrick High Temperature Reaction Chamber.

EXPERIMENTAL

Zinc oxide (ZnO) was obtained from Loud Wolf Limited. The sample was loosely packed in the reaction chamber sample cup. The K-type thermocouple and heater of the reaction chamber were attached to the Harrick Temperature Controller to regulate the temperature of the chamber. The UV-Vis spectroscopic measurements were carried out on a double beam UV-Vis-NIR spectrometer with the Praying Mantis™ installed.

Spectra were recorded over the range 350 nm – 800 nm with the spectrometer in double beam mode with an attenuator (1.5 abs) in the reference beam, a 2.000 nm SBW and 1.100 nm data interval. The sample was initially heated to 100 °C and allowed to thermally equilibrate before a spectrum was recorded. This was then repeated at 200 °C, 300 °C, 400 °C, and 500 °C. Spectra were referenced to ZnO at room temperature, which does not exhibit any visible absorbance.

applications note

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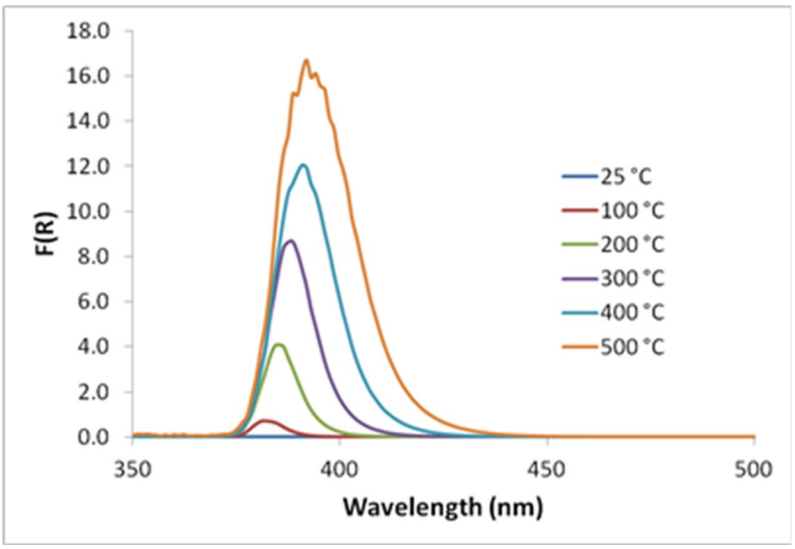

Figure 3. UV-Vis spectra of ZnO from room temperature to 500 °C.

RESULTS AND DISCUSSION

The UV-Vis spectra of ZnO over the temperature range 25 °C – 500 °C are shown in Figure 3. The spectra have been transformed by the Kubelka-Munk function, as is standard for diffuse reflection spectra. Ideally, the transformation makes the spectra proportional to concentration. It is clear that as the temperature rises, the absorption of visible light increases in both intensity and range of wavelengths. This corresponds with the observed increasing yellow color of the solid as temperature increases, as non-yellow light is absorbed, leaving the sample with its yellow color.

CONCLUSIONS

This work demonstrates the ability of the Harrick Praying Mantis™ and High Temperature Reaction Chamber to observe the UV-Vis spectroscopic changes of powders and rough solids at elevated temperatures.

REFERENCES

1. Prakoso, S.P.; Saleh, R. *World Journal of Condensed Matter Physics* **2011**, *1*, 130-136.
2. Wilberg, E.; Holleman, A.F. *Inorganic Chemistry* (2001), Elsevier.
3. Coogan, C.K.; Rees, A.L.G. *J. Chem. Phys.* **1952**, *20*, 1650-1651.