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Porous SiO_2 and TiO_2 Thin Films with Gold and NiO Nanoparticles for Optically Based Gas Sensors

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SUMMARY

Thin films of SiO_2 and TiO_2 prepared by sol-gel methods have been doped with gold and nickel oxide nanoparticles (NPs) using *in-situ* and *ex-situ* techniques, and used as platforms for optical gas sensors for CO and H_2 detection. Optical gas sensors of this type require an elevated operating temperature and measurements made in the temperature range 200 °C to 350 °C showed a reversible gas-induced variation of absorption spectra in visible and near IR wavelengths for both CO and H_2. The variation in absorbance depends on the wavelength, film annealing temperature and composition and porosity of the composite film matrix. Results demonstrate the possibility of selectively detecting the H_2 and CO species in this case.

EXPERIMENTAL

The nanocomposite films of SiO_2 and TiO_2 with Au and NiO nanoparticles have been prepared by mixing sol-gel solutions containing appropriately selected precursors (1,2) with either $HAuCl_4.3H_2O$ or a gold colloidal suspension. Thin films of the composites with a nominal Au-NP concentration between 5 and 8wt% were deposited by spinning onto a SiO_2 glass substrate, and then annealed at temperatures in the 300°C-700°C range for 30 minutes in air. Films with uniform thicknesses from 20-200 nm have been deposited. Structural characterization was done using XRD, SEM, EDX, TEM and XPS techniques. Optical absorbance sensor data were obtained for a series of annealed samples over visible and near IR wavelengths, ($350 < \lambda < 850$nm), in a custom built gas flow cell which permitted the film composite to be in a controlled temperature and gas environment. The films were exposed to CO and H_2 concentrations of between 10ppm and 1% by volume in air and absorbance spectra were measured in both scanning and temporal (fixed λ) modes to determine sensor functionality.

RESULTS & DISCUSSION

All the films prepared have uniformly distributed components of Au and/or NiO in the SiO_2 or TiO_2 porous matrix. For the SiO_2/NiO-Au films which were annealed at 700°C, the NiO-Au nanoparticles have a two-fold structure which forms epitaxially on Au(111)-NiO(200) planes, as shown in Fig. 1.

The optical absorbance data of Fig. 2 for SiO_2 with Au-NiO, shows the change in absorbance that occurs when the film is exposed to either 1% H_2 or 1% CO. The principal features of these difference spectra demonstrate that there is a wavelength dependence of maximum and minimum ($Abs_{Air} - Abs_{Gas}$) values, and that specific wavelengths can be selected to exclude the optical absorption change for one of these species versus the other. Shown in Fig. 3 is the temporal absorbance response of the SiO_2 with Au-NiO film at $\lambda = 640$nm for a sequence of gas exposures, including in the centre section ($16 < t < 28$ mins), a combined exposure to CO and H_2, where the effect due to CO ($20 < t < 22$ mins), is close to eliminated, thus displaying selectivity to H_2 at this wavelength.

Data for the optical sensor response of other SiO_2 and TiO_2 nanocomposites with Au and NiO will be discussed in the overall presentation.

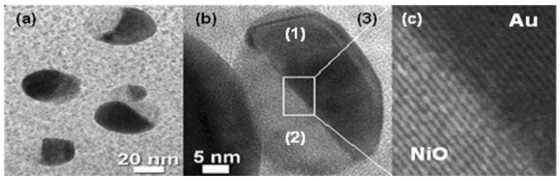

Figure 1. Thin film showing (a) the distribution of NiO-Au in a porous SiO_2 matrix. The NiO and Au nanoparticles are, (b), two-fold and, (c) epitaxial on NiO(200)/Au(111) planes.

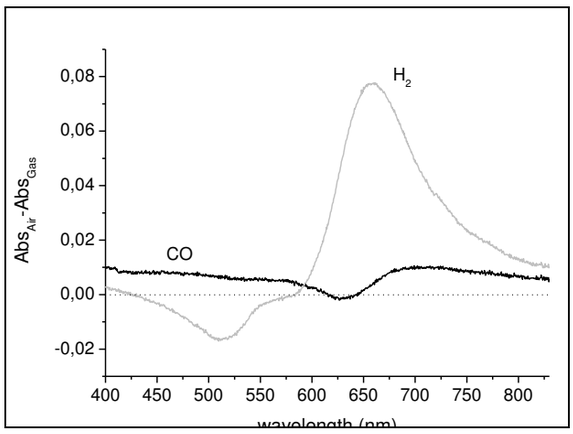

Figure 2. Difference absorption spectra ($Abs_{Air} - Abs_{Gas}$) for SiO_2 with Au-NiO nanoparticles when exposed to either 1% H_2 or 1% CO in air at T=300°C.

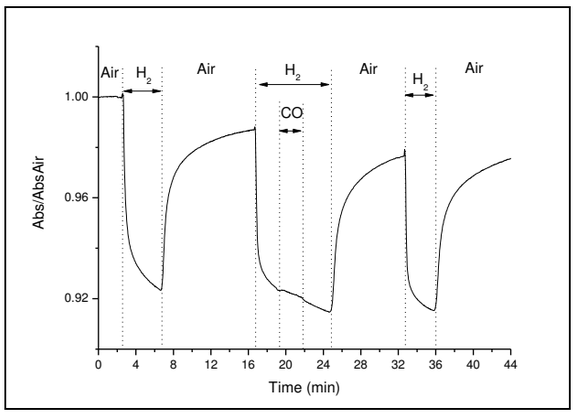

Figure 3. SiO_2 with Au-NiO nanoparticle film. Relative absorbance change at $\lambda = 640$nm and T=300°C for sequential exposure to Air, 1% H_2 and 1% CO. Selectivity to H_2 detection is demonstrated.

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