

## Electron Energy Loss Spectroscopy of thermochromic $V_2O_5$

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The vanadium pentoxide is a thermochromic oxide that has been extensively studied for its applications in different scientific areas. It has been used as a catalyst in organic synthesis[1], in electronics to fabricate electrochemical storage devices among others. The vanadium pentoxide has recently found applications as a cathode material in Li-Ion batteries[2],.

The purpose of our research is to study the changes in electronic structure of commercial  $V_2O_5$  during the metal-insulator transition, via Electron Energy Loss Spectroscopy (EELS) in both low energy-loss and high energy-loss regions.

The structural characterization was carried out by Differential Scanning Calorimetry (DSC) and Transmission Electron Microscopy (TEM). The characterization of the electronic structure was performed by using the EELS technique. From the low energy-loss part of the spectrum, we obtained the optical properties of the material via the complex dielectric function. From the high energy-loss region, we observed changes in the ELNES of  $V_2O_5$  during the phase change .

EELS spectra were performed in a Gatan Parallel Electron Energy Loss Spectrometer (PEELS model 766) attached to a Philips CM-200 transmission electron microscope. Spectra were acquired in diffraction mode with 0.2 eV per channel dispersion, an aperture of 2 mm and a collection semi-angle of about 2.7 mrad. The resolution of the spectra was determined by measuring the full width at half maximum (FWHM) of the zero loss peak and this was typically close to 1.5 eV, when the TEM was operated at 200 kV.

Phase change was induced by placing the sample in a heating sample holder. We were able to verify, through changes in the diffraction patterns before and after heating, the phase change of vanadium pentoxide. Figs. 1 and 2 show the changes of the diffraction patterns before and after heating, where it is seen that a phase change has been taken place.

Figure 3 shows the energy loss function  $\text{Im}(-1/\epsilon)$ , before and after the heating. It is observed that a peak appearing around 10 eV in the insulator phase at room temperature, has disappeared in the metallic phase at 300°C . The very marked difference observed between the two spectra, indicates a change in the electronic structure of the material during the phase change. Figure 4 shows the imaginary part of dielectric function, which gives us information about interband transitions, where peaks indicate the energies at which absorption has occurred.

The V  $L_{2,3}$  and O K ionization edges are shown in Figure 5, where it is observed a shift in the peak at 532 eV before the heating to 524. Fig. 6 shows results from DSC, in the interval between 25°C and 400°C, resulting in an exothermic reaction at 224°C.

[1]Mohd Azri Ab Rani et al. ,ISSN1450-216X Vol.24 No3(2008)428-432

[2] Jeffrey W. Fergus, Journal of Power Sources 195 (2010) 939–954.

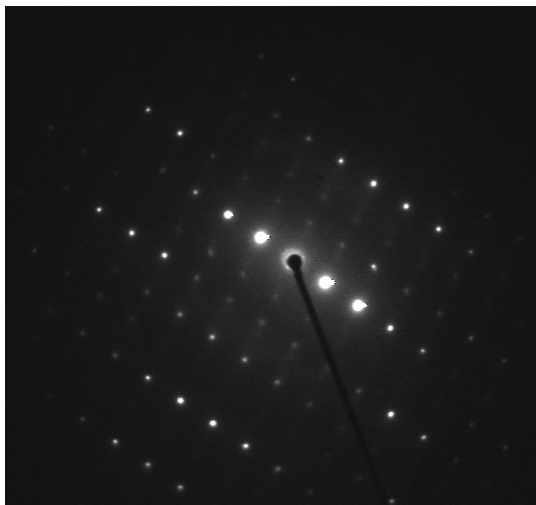


Figure 1. Diffraction pattern of single crystal  $V_2O_5$  at room temperature.

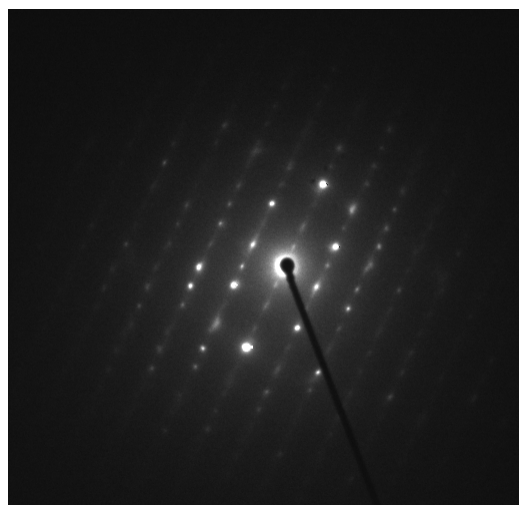


Figure 2. Diffraction pattern of single crystal  $V_2O_5$  at 300°C.

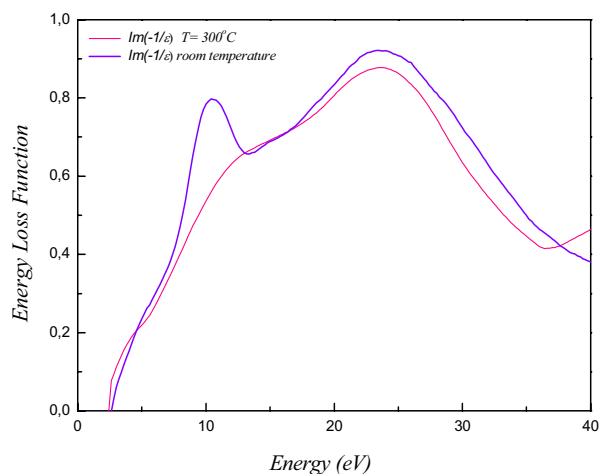


Figure 3. Energy Loss Function taken from  $V_2O_5$  single crystal.

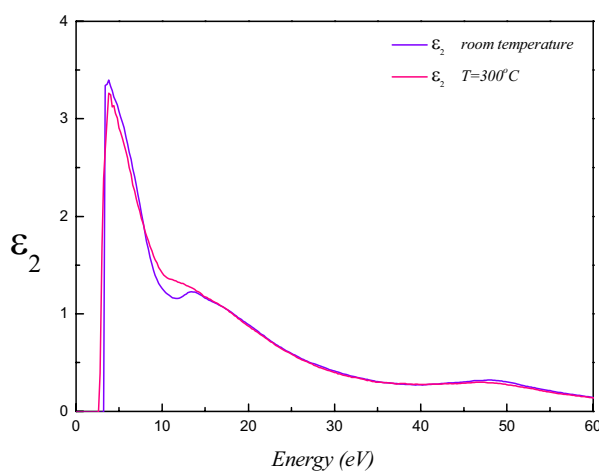


Figure 4. Imaginary part of the dielectric function taken from  $V_2O_5$  single crystal.

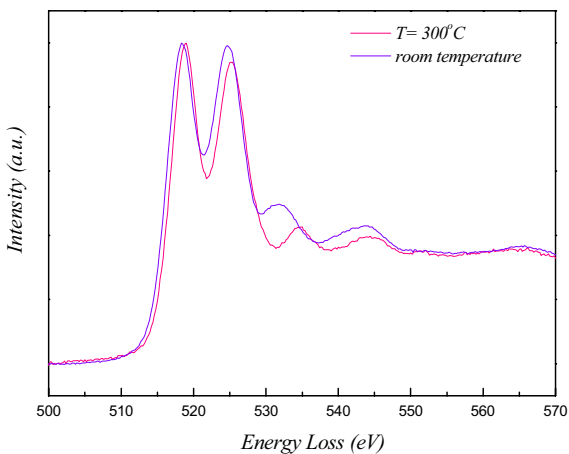


Figure 5. V L<sub>23</sub> and O K ionization edges, taken from  $V_2O_5$  single crystal.

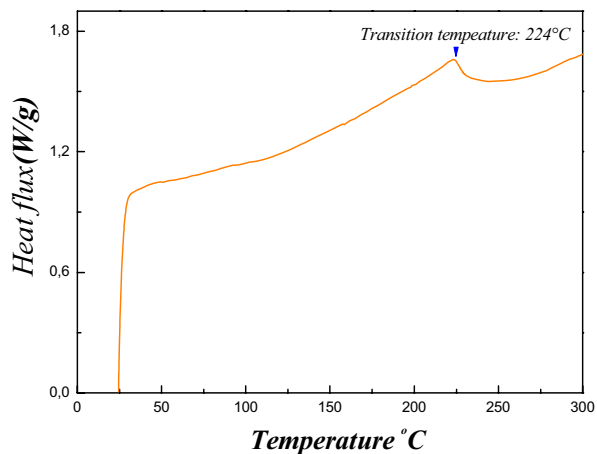


Figure 6. Differential Scanning Calorimetry of  $V_2O_5$ .