

DSC and TGA on Diffently Fired Cadmium Oxide

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Abstract

Thermo-Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) experiments, backed by Rutherford Backscattering (RBS) characterizations, have been carried out on as-supplied cadmium oxide and cadmium oxide heat-treated at 800 °C for 36h. We find substantial loss of oxygen on heat treatment at high temperatures like 800 °C. This partly accounts for the large increase of the room temperature electrical conductivity due to heat treatments. Endotherms centered at two temperatures appear to correspond O-loss in two major steps. An exotherm at ~ 300 °C, midway between the two Endotherms, must correspond to atomic re-arrangement after O-loss at the lower step.

Keywords: DSC, TGA, RBS, diffently fired Cadmium Oxide

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Introduction

Doping of II-VI semiconductors like zinc oxide and cadmium oxide by impurities to generate p- or n-type carrier, control their concentration and tailor the band gaps is a very competitive field of research in view of their potential use [1,2] as electronic material and as a Transparent conducting oxide in solar cells. However, non-stoichiometry in the II-VI host, as observed [3,4] in Cd-O, has similar effect as doping by a third element. This self-doping must well documented, understood and taken into a third element. This self-doping must well documented, understood and taken into account before taking up any inpurity-doping project. This problem has been addressed in this work.

cadmium oxide is usually supplied by manufactures as light brown powder. It is the most ionic II-VI compound that should have high resistivity. Present samples of 99.99+% quoted purity, as supplied by Aldrich (USA), showed a fairly high room temperature resistivity of ~43 mΩcm. It is remarkable that after 800 °C firing of the as-supplied sample for 36 hours, we got black coloured semi-metallic sample of only ~2.2 mΩcm room temperature resistivity.

This resistivity reduction is large and not understood properly. However, Rutherford Backscattering (RBS) showed [3] lower O-content for the 800 °C fired sample. Here, we investigate the details of the weight loss (O-loss) by Thermo-Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). Our earlier DTA-TGA [5] was only one kind of cadmium oxide, and the sample was of lower purity.

Aim of study

Making a roadmap of compound type semiconductor including II-VI oxides has attracted attention owing to their potential application as in optoelectronics, laser diodes and solar cell.

And modified above oxides on basis of DSC & TGA investigation for potential application

Experimental Outline and Results

The runs (typically Figures 1 and 2) have been taken in a DTA/TGA 6300 unit and EXSTAR 6300 DSC unit, both from Seiko (Japan) on ~ 30 mg of the sample (above-mentioned Cademium Oxide from Aldrich) under flowing Ar gas and at a slow heating rate of at 5 °C /min, as fast runs may fail to, show all the minor features. The sample was taken in a light platinum crucible in one of the TGA or DSC pans. Another empty crucible on the other pan was the standard for the differential measurements.



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Fig. 1 Thermo-Gravimetric Analysis (TGA), By Heating at the Rate of At 5 °C/Min, Show Reduction of the Mass of the As-Supplied Cadmium Oxide (Aldrich)

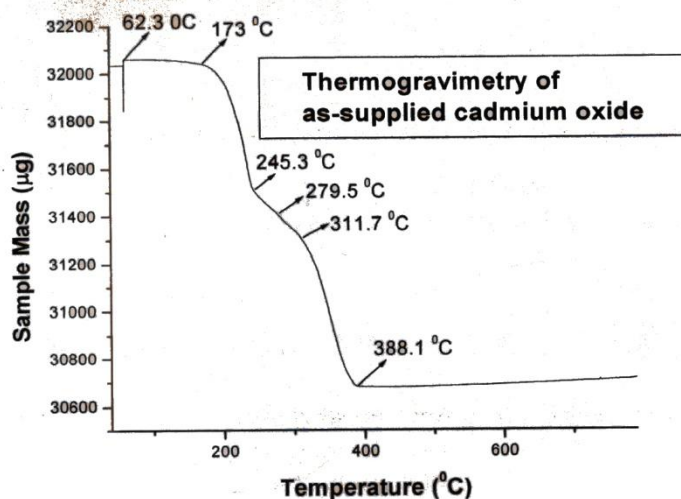
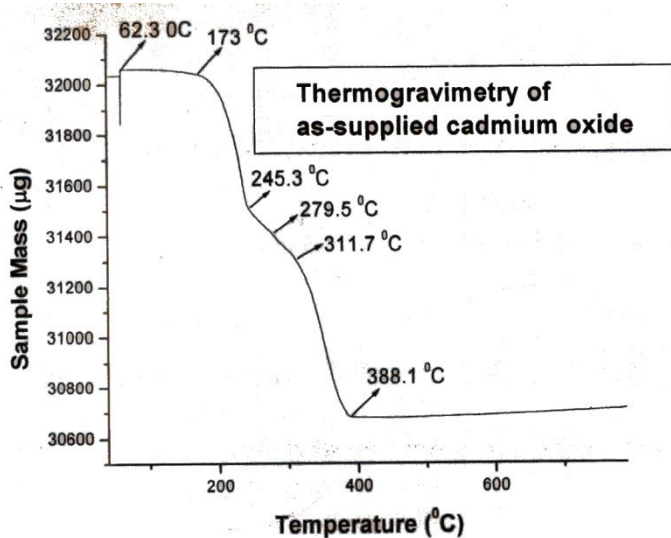


Fig. 2 Differential Scanning Calorimetry (DSC), By Heating at the Rate of At 5 °C/Min. of The As-Supplied Cadmium Oxide (Aldrich)



Discussion

TGA is Good for Comparing Mass Losses

Table: Summary of the findings of TGA up to ~815 °C on the differently fired Cadmium Oxide.

CdO_as-s	weight loss starts at 173 °C, steep loss up to 212 °C	Loss at 245 °C to 312 °C, is least steep	312 °C to 388 °C Loss is fairly steep
CdO_800	No loss of weight		

For the as-supplied sample, the two steps in TGA (Figure 1 and Table) match the endotherms in DSC (Figure 2). The mass loss for ~ 30 mg as-supplied sample (to be called "CdO_as-s") can be seen to be ~1.5mg. This implies removal of (Δn/n) =40 atomic % of the Oxygen, if all of the mass loss is due to O-loss. The lost mass includes some hydrocarbon impurities. This is in line with our qualitative RBS finding [3] of O-loss on heating. Since , the corrected heights for Cd-steps the RBS results for these

samples are proportional to the Cd-content [6,7], we now estimate that (Δn/n)=19 atomic %of excess Cd or (Δn/n)= 19%loss of oxygen after the 36 h firing at 800 , taking CdO_125 as the initial and 50:50 sample [8]. CdO_125 and as supplied CdO have been found to be practically same with respect to colour and resistivity [9], as the firing temperature of at 125 may not to be high enough to induce structural changes. However, it is expected to remove adsorbed impurities like moisture, if any. In that case,CdO_125

will show a lower mass loss [10]. But that ($\Delta n/n$)=19 atomic % represents O-loss more realistically.

Another interesting point is that on heating the highly O-deficient CdO₈₀₀ sample in the TGA apparatus, there is no more O-loss from the sample (Table).

Conclusion

As-supplied cadmium oxide starts losing weights (mass or material) at 173 °C. Next, it shows steep weight loss (Fig.1) up to 388 °C. RBS identified the lost species as oxygen. Understanding the DSC result allows us to go deeper into the 2-steps O-loss from the NaCl structure of cadmium oxide [11]. O-loss at round 237 °C is from random sites till an atomic re-arrangement takes place at around 270 °C to lead to more tightly bound lower potential energy structure or state [12]. So, some more O-loss from this state requires the higher temperature of 388 °C. This further explains the fact that there is no O-loss from the 800 °C.

Endnotes

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