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Thermo-Gravimetric Analysis on differently Annealed Cadmium Oxide

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Krishan Chandra Verma
Assistant Professor,
Dept. of Physics
Veerbhumi Govt. P.G. College,
Mahoba, U.P., India

Abstract

Thermo-Gravimetric Analysis (TGA) has been carried out for the first time for differently fired Cadmium Oxide (99.99+% pure) samples. Weight loss at particular temperatures for the as-supplied samples and no weight loss for the 800 ^{0}C fired sample together with RBS indicate O-loss from the former sample on heating.

Keywords: TGA, Cadmium Oxide, Non-stoichiometry, RBS, Thermal Properties.
PACS No. : 71.55, GS, 72.70, -a, 05.70, -a, 72.20, -i.

Introduction

Better conducting cadmium oxide should be better for use as Transparent Conducting Oxide (TCO) window in solar cells [1]. It has other potential applications in electronic and related devices. Pallets made from high purity (99.99+%) powder (brown) procured from Aldrich (USA) showed a room temperature resistivity of ~ 43 mΩcm. These have been fired [2] in air at different temperatures like 800 ^{0}C for a sufficiently long time of 36 hours, resulting in samples, being denoted by names like CdO_800. Decrease of room temperature resistivity [3], to ~ 10 mΩcm for CdO_400 (coffe brown) and to only ~2.2 mΩcm for CdO_800 (black), is large and not understood properly. This led us to undertake Thermo-Gravimetric Analysis (TGA) for differently fired cadmium oxide, probing the gain or loss in the sample mass (on heating) as an indicator of changes inside the sample. Apart form our earlier DTA-TGA in [4] on only one kind of less pure cadmium oxide, presents series of TGA appear to be the first such reporting.

Experimental outline

The set-up for TGA has two (delicate) weighing-balance-arms with pans to load the sample one and a standard in the other. These are housed within a small furnace .TGA records the difference in masses put on the two pans, as the temperature is varied. A decrease in mass at certain temperatures for a metal oxide may be due to decomposition and oxygen loss [5]. The standard in TGA should not change in mass, but help elimination of instrumental or background effects in the differential measurement [6]. Still, extra care of blank runs (Fig.1) have been taken in this work to estimate the remaining possible errors.

All TGA runs (Fig. 1 &2) have been taken at a slow heating rate of at 5 ^{0}C /min, as fast runs may fail to show all the minor features of mass change with three factors can contribute to the difference the two results can be included to be supporting each other to the extent [7] that 800 ^{0}C firing results in significant O-loss from Cadmium Oxide and the O-loss is likely to be to the tune of 19 atomic % [8].

Result and Discussion

TGA is good for comparing losses at different temperatures and for different samples:

Table: Summary of the findings of TGA up to ~ 815 ^{0}C on differently fired Cadmium Oxide.

CdO_as-supplied	Weight loss starts at 173 ^{0}C, steep loss up to 212 ^{0}C	Loss at 245 ^{0}C to 312 ^{0}C, is least steep	312 ^{0}C to 388 ^{0}C loss is fairly steep
CdO_800	No loss for weigh, appears to be a small again continuously up to the end		

The mass loss for ~ 30 mg as-supplied sample (to be called ‘‘CdO_as-supplied”) can be seen to be ~ 1.5 mg. This supports our RBS finding that in comparison to the as-supplied sample, 800∘C fired sample

(CdO_800) has less oxygen [9]. It is most interesting that on heating the highly O-deficient CdO_800 sample in the TGA apparatus, there is no more O-loss from the sample (Fig. 2) This gain (of ~100µg per 700 ^{0}C) is just larger than changes in the blank runs (also in Fig.2) indicating the background , but of the same order. So further Chemical reaction, like oxidation of a metallic sample, adding atoms from the environment to the sample at a certain temperature region will show up as an increase of mass in the TGA record [10]. A decrease in mass at certain temperatures, possible for a metal oxide decomposing into the metal and oxygen for example, is in general due to loss of one or more components. The standard should not change in mass, but help elimination of instrumental errors or the background effect in the differential measurement. Still, extra care needs to be taken to estimate the possible errors from these effects. TGA observation of mass changes in different cadmium oxide on heating and the temperatures needed to activate these changes should help understand the above mentioned effects of annealing [11].

Figure 1. Thermo-Gravimetric Analysis (TGA), by heating at the rate of at 5∘C/min, shown redcuction of the mass of the as-supplied Cadmium Oxide (Aldrich) i.e. loss of atoms from the composed.Its derivate (DTG) is given in the lower graph.

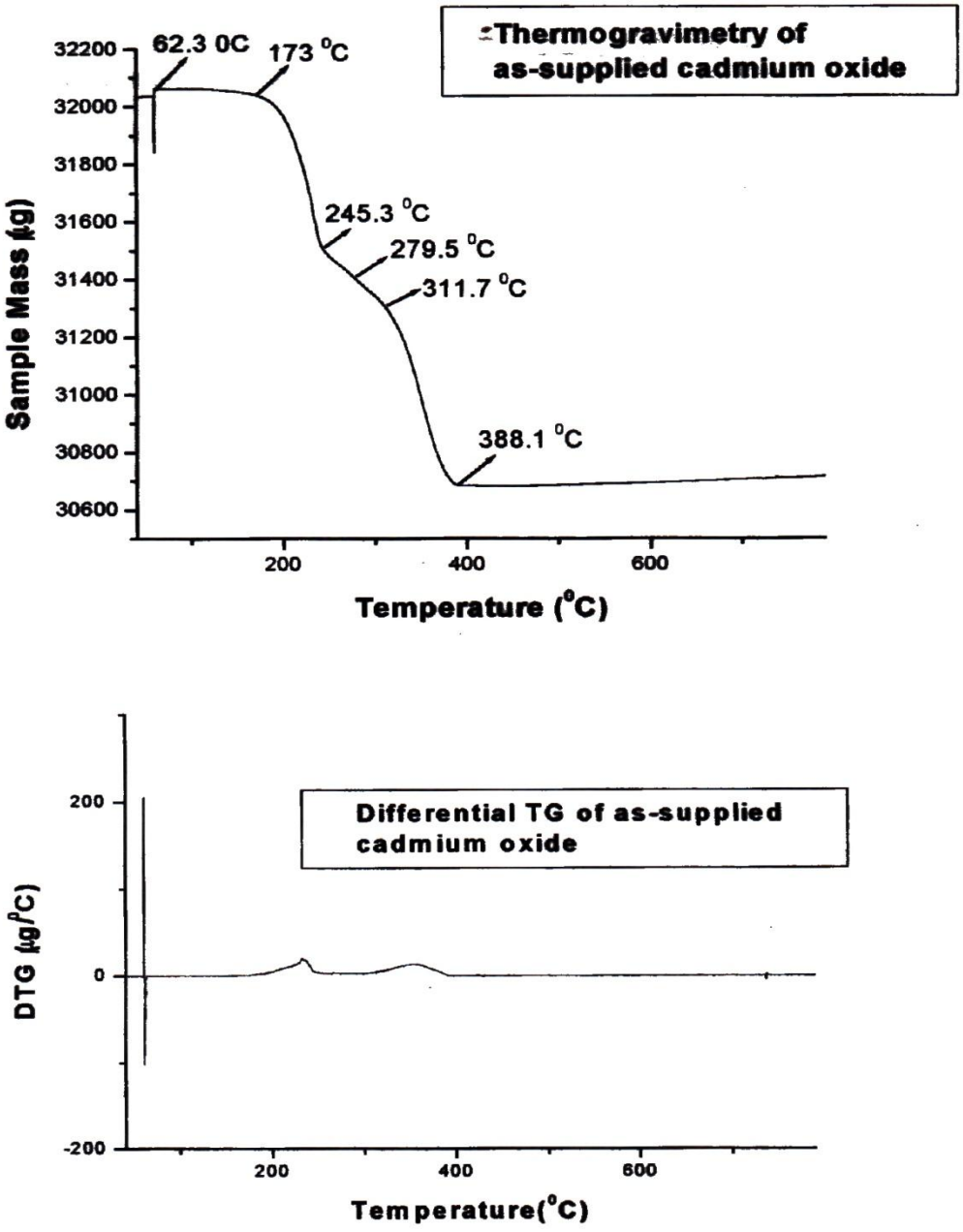
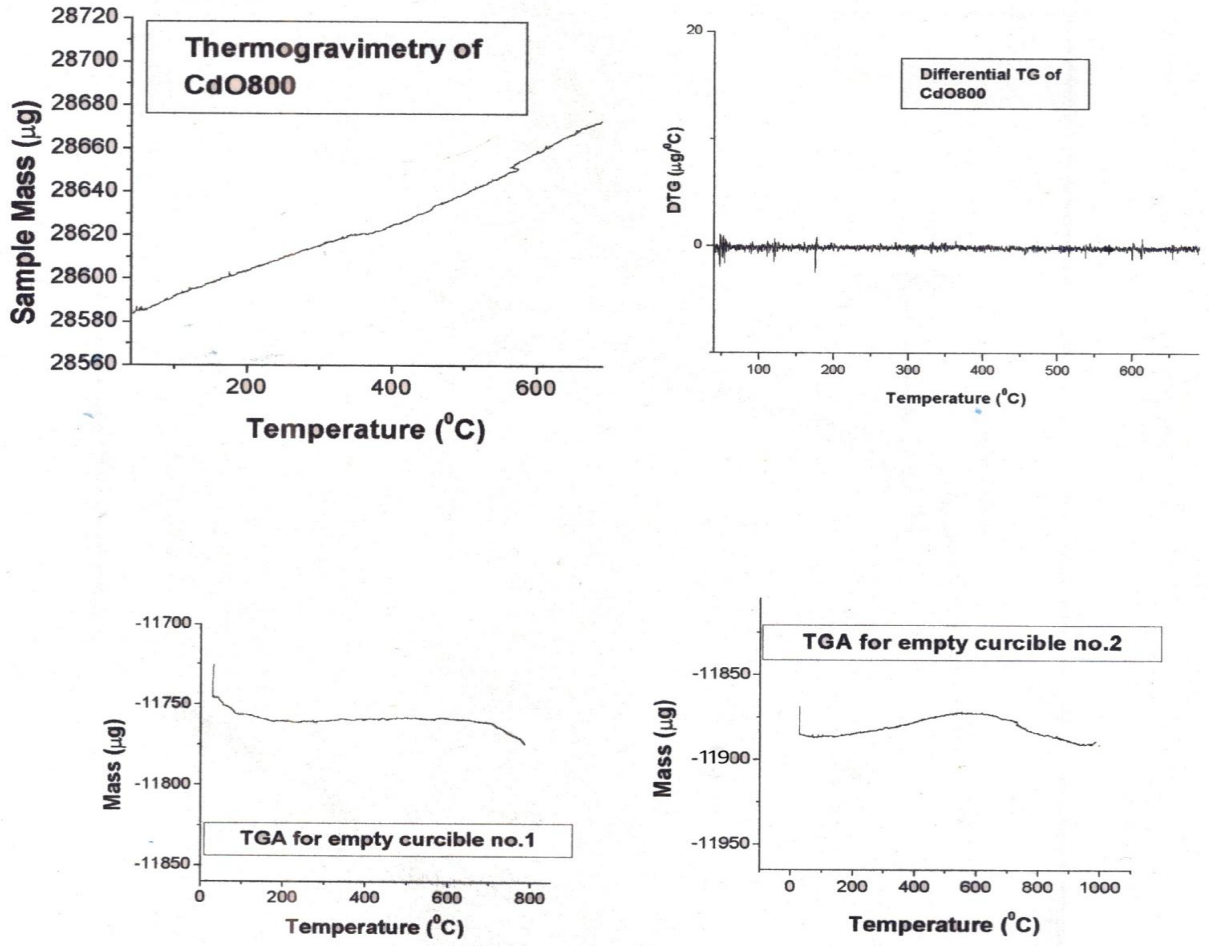


Fig. 2 . Thermo-Gravimetric (TG) result, by heating at the rate of 5 ^{0}C/min, for the 800 ^{0}C fired cadmium oxide (Aldrich) sample, its differential TG, and two background finding runs of.



Aim of The Study

Aim of present Study was to assess TGA analysis and electrical conductivity peculiarities in differently annealed cadmium oxides material. And other properties will be probed in feature. Because differently heat treated cadmium oxides material has a potential application in fuel cells, solar cells and batteries.

Conclusion

Our initial blank runs in Thermo-Gravimetric Analysis have shown the extent of small and non-systematic background variation possible in TGA result. Such blank runs defining the small but non-zero the background gravimetric fluctuations must be carried out before any serious TGA study. Our TGA result and backed by RBS characterization on Cadmium Oxide supplied by two different manufacturers show substantial loss of Oxygen on heat treatment at 800∘C.This has to give rise to an increase of electron concentration in the samples to maintain the charge balance [12]. This can explain partly the large increase of the room temperature electrical conductivity [13] due to heat treatments.

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