

## A study of nanostructured ZnS polymorph by synchrotron X-ray diffraction and atomic pair distribution function†

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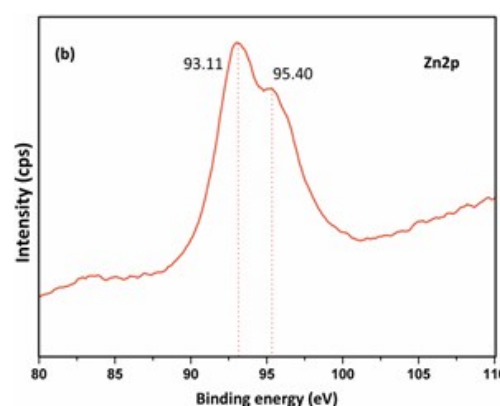
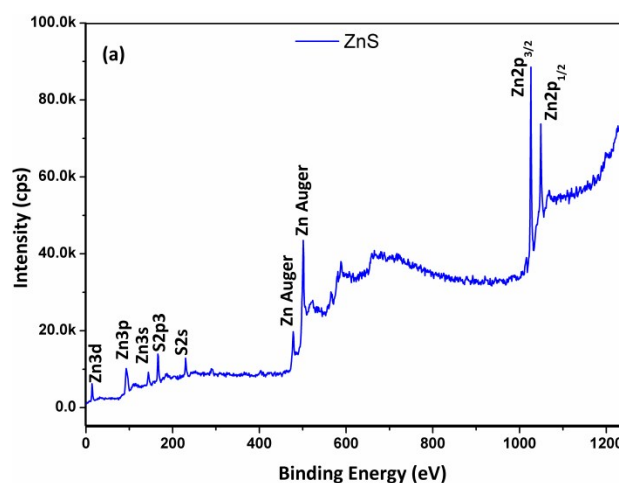
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The XPS data has been recorded using Al K-alpha (1486.61 eV) X-rays at 13 KV, 300 W. The data lists Intensity (counts/s) vs kinetic energy. The KE values can be converted to BE values ( $BE = 1486.61 - KE$ ). There can be charging in the samples; which can be corrected by referencing Carbon C1s at 284.6 eV. The peaks of carbon: C1s ref1 is recorded in the beginning of the scans, and C1s ref2 at the end of the scans. Checked the peak positions of these two peaks is same (plus/minus  $\sim 0.2$  eV). By taking average of these two peak positions C1s ref1 and C1s ref2 and subtract 284.6 eV from the average value to calculate the difference. This difference gives the peak shift due to charging. Subtracted the difference value from the BE values of the entire element's data. After subtraction the peaks will move towards lower BE side.

The XPS analysis of S2 samples scanned in the range of 0–1400 eV is shown in Fig. 1. The survey spectrum of Fig. 1a shows that Zn and S were present in the as synthesized S2 samples. No contaminant species were observed within the sensitivity range of the technique. The binding-energy (BE) data have been standardized to an energy scale that assumes adventitious carbon C1s 284.6 eV and recorded C1s around 290 eV then the charge correction factor is 5.4 eV. The binding energy values for the Zn3p Zn3d and S2s are as 87.745, 8.98 and 224.93 respectively. With further detailed examinations, the compositions Zn and S are consistent with stoichiometric ZnS free of other impurities such as carbon. This indicates that, to a certain degree, the concentration of S vacancies can be controlled by varying the amount of Zinc content in the samples<sup>1</sup>. The qualitative analysis from S2 sample XPS spectra of Zn2p and S1 sample S2p and EDAX spectra reveals that the atomic ratio of Zn to S is very close to 1:1 stoichiometrically. Fig. 2 depicts the typical energy-dispersive absorption X-ray spectroscopy (EDAX) spectra for ZnS S1 sample. From EDAX spectra the chemical analysis show that presence of Zn and S signals only,

indicating that the nanocube which are made up of zinc and sulphur atoms which outlines the high purity of the sample.



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†Electronic Supplementary Information (ESI) available: Details information is given in ESI about XPS and EDAX measurement.

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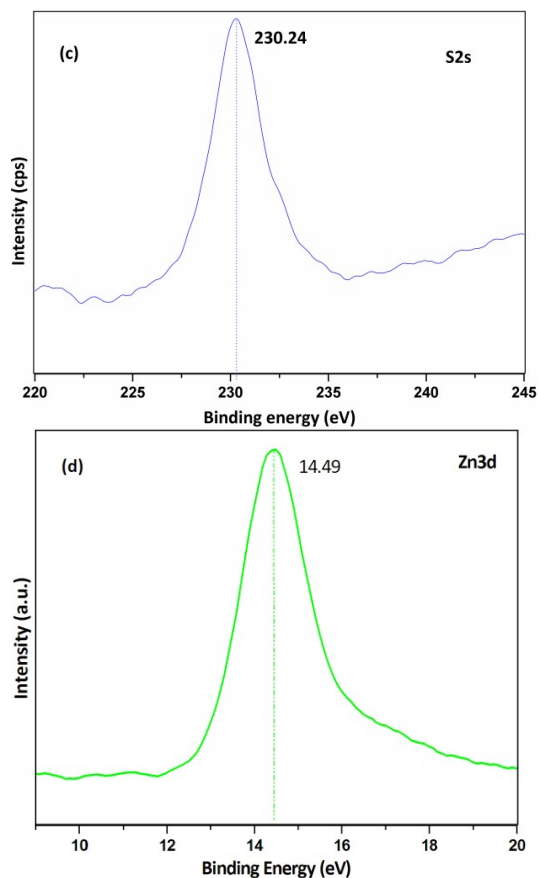


Fig. 1. The full survey spectrum for sample S2 is given in (a) whereas Zn2p in (b), S2s (c) and Zn3d (d) respectively.

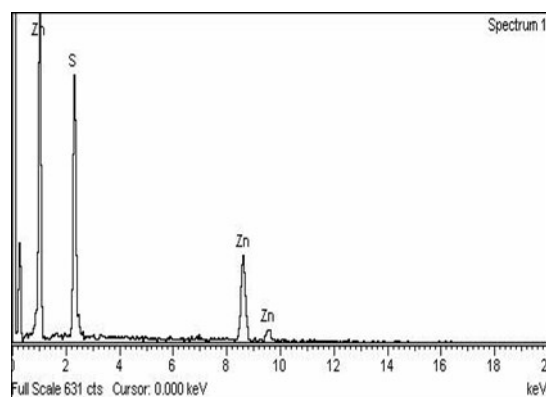


Fig. 2 The EDAX spectra for sample S1.

### FTIR Study

The FTIR spectra for as synthesized samples are shown in fig. 3. In

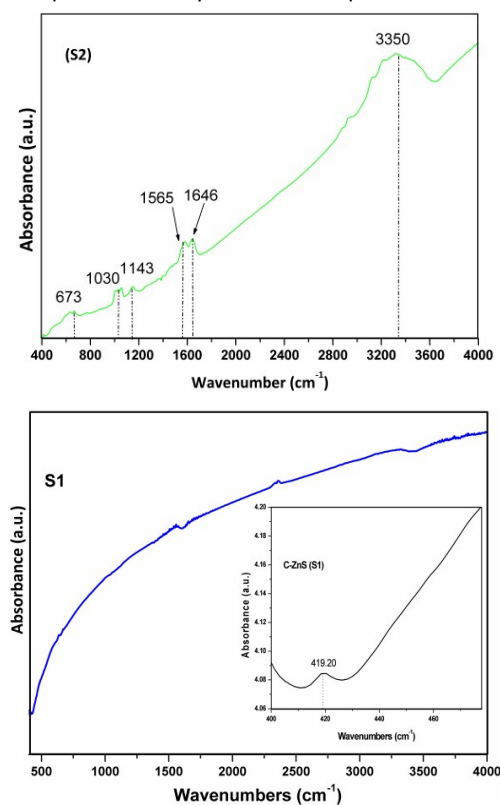


Fig. 3 FTIR spectra of samples S1 and S2.

fig. 3(S2) few peaks were observed at 400-4000  $\text{cm}^{-1}$  for S2. In the Fig. 3(S1, S2) the absorption peak around at 400-700  $\text{cm}^{-1}$  indicating Zn and S stretching<sup>1</sup>. The broad peak at 3350  $\text{cm}^{-1}$  for S2 was assigned to O-H stretching resulting from small quantity of absorbed water ( $\text{H}_2\text{O}$ ) on the samples. The peaks at 1050 to 1143  $\text{cm}^{-1}$  were observed due to the formation of microstructure in S2 samples.<sup>2, 4</sup> The bands at 1565 to 1646  $\text{cm}^{-1}$  are due to  $-\text{C}=\text{O}$  stretching mode arise from the absorption of atmospheric  $\text{CO}_2$ . The band values are in good agreement with the reported literature.<sup>1-4</sup>

### References

- 1 T. Y. Yesu, R. Anita, B. Kavita, *Int. J. Appl. Sci. Eng. Res.* 2012, **1**, 2.
- 2 S. Ummartyotin, N. Bunnak, J. Juntaro, M. Sain, H. Manuspiya, *Solid State Science*, 2012, **14**, 299-304.
- 3 A. Mandal, A. Dandapat and G. De, *Analyst*, 2012, **137**, 765.
- 4 M. Lu, L. Chen, W. Mai and Z. L. Wang, *Appl. Phys. Lett.*, 2008, **93**, 242503.