ISSN (Print) : 2320 – 3765
ISSN (Online): 2278 – 8875

International Journal of Advanced Research in Electrical, Electronics and Instrumentation Engineering

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 2, February 2015

Optical Structural Electronically and Band Gap Determination of Newly Grown Semi-conducting Copper Sulphide Crystal Using Gel Method

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ABSTRACT: In modern research growth of newer material copper sulphide is a need today for electronically and electrically active devices. A structural optical and Band gap determination signifies its characterisation, the band gap of these crystal exhibits variety of applications in semiconductor and in electronics .A crystal of copper sulphide shows purely semiconducting properties which are grown using hydrogen sulphide gas. A spectrum sensing advances Applications using X-ray diffraction.

KEYWORDS: Copper, Band gap, Spectrum, Characterisation.

I.INTRODUCTION

A characterization of growing crystal was performed using structural analysis and thermal Analysis. The Non linear optical properties of various copper crystals is very important .In the recent year this prominent NLO behavior is studied by growing iodated of alkali transition and alkaline earth material. it must be alkaline earth material. The band gap of copper sulphide crystal exhibits variety of applications in semiconductor and in electronics .A crystal of copper sulphide shows purely semiconducting properties which are grown using hydrogen sulphide gas. A spectrum of these are discussed in this paper. It is also reported that sulphide are semiconductor in nature the synthesis of sulphide are complicated because of relative low vapour pressure of the sulphur ,neverth less successively more attention is being turns toward investigating sulphide of various materials .There is no need to explain the role of semiconductor in the development of present electronic industries and semiconductor device for e.g. Zink sulphide (ZnS) used for lenses and other optical devices lead sulphide used in infra, red sensors ; cadmium sulphide [cds] which is used in photocell. Iodated, iodides and sulphide are taken it basic material copper. The growth of newer Engineering Semiconducting material in the form of crystal is widely useful in different technologies. Some grown crystals having optical, piezoelectric and NLO behavior. Copper sulphide crystals are insoluble in water which is decomposing before melting point. In the work reported have the determination of the optical band gap of the sulphide crystal, its structural and thermal behaviour explained and calculated. Copper sulphide is a metallic conductor due to the incomplete occupancy of the sulphur, the determination of properties of copper sulphides is generally monovalent copper compounds. Research on thin film solar cells with copper sulphide doped in iron, indium, cadmium, bismuth is a great interest today.

II. LITERATURE SURVEY

Today Crystal have became the base of modern technology in all aspects & ever increasing the demand of crystals in the variety of field in science & technology. The work of growth & the study of copper iodated crystal have been taken in the laboratory. The advances in the material science & solid state devices depend on the availability of good quality crystals. Gel technique is considered more useful at room temp. & has an inexpensive Process

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III. EXPERIMENTAL PROCEDURE

A copper sulphide crystal are grown using chemical reaction method. Using this method gel of copper chloride formed and water containing H2S gas filled with subsequent nucleation and the crystal growth of copper sulphide takes place.

IV.PREPARATION OF GEL

Initially different concentration solution of sodium Meta silicate taken for e.g. 10gm,12gm ,14 gm, 16 gm, 18 gm, 21gm, 22gm in distilled water to get 250cc solution. The solution is constantly stirred and then filtered by Dr Watts filter paper. It is then kept in to an airtight bottle free from dust and contamination. Density of the solution was measured using specific gravity bottle. A solution of different molarities prepared by adding proper amount of chemicals to the double distilled water for copper nitrate, copper chloride, Hydrogen sulphide gas, acetic acid and sodium meta silicate. A gel formation of mineral or organic acid takes place with a mixing of sodium meta silicate solution. It forms process of polymerization in the mixture of solution or resultant solution. In the present work, various concentration of acetic acid and those of sodium meta silicate were tried for optimum condition with different concentrations of Hydrogen Sulphide gas solution

V. SINGLE DIFFUSION TECHNIQUE

In actual procedure, 5cc of 2N acetic acid was taken in a small beaker, to which sodium Meta silicate solution of density 1.04 gm/cc was added drop wise from burette with constant stirring performed with the help of magnetic stirrer, till pH of the mixture reaches a value 4.4. A pH meter HANNA instrument of digital pocket sized is used for this purpose. A 5cc of copper chloride solution of concentration 0.4M was added with constant stirring in mixture of acetic acid and sodium meta silicate solution .A continuous stirring process required to avoids excessive ion concentration which otherwise causes premature local gelling and makes the final medium inhomogeneous and turbid .The pH of the mixture was maintained at 4.4 Number of attempts were tried for optimum condition for appropriate range of pH values and allowed gel to obtain crystals of copper sulphide. The gel setting time required for the silica gel solution of pH greater than 4.5was short, it is observed that the mixture of solution with pH value less than 4.2 required quite greater number of days, however in the pH range 4.2 to 4.4 there is appropriate waiting in gelation time. Room temperature and atmospheric effect also plays an important role on gelation, aging that is evaporation of water molecules form on surface of gel. To perform these experiment borosil glass test tubes of diameter 2.5cm and height 25cm was used as crystallizing vessels. This mixture was then transferred to the test tube, a mouth of test tube closed using cotton plug. which is used to avoid contamination and dust affecting from atmosphere. The gel setting time was 12 to13days .This completely set gel was left for aging for 4days.i.e.96 hour's to120 hours .It is also observed that the aging of gel reduces the diameter of the capillaries in gel so that reaction can be controlled. H2S gas dissolved in distilled water was used as supernatant.

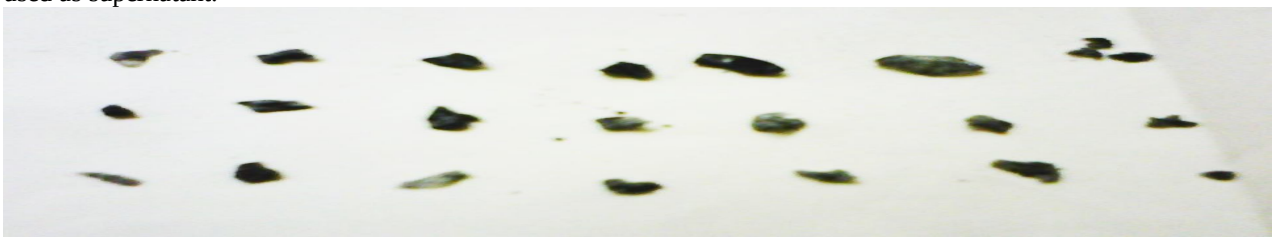

Fig. 1 Number of grown crystals of copper sulphide

VI. EFFECT OF VARIOUS PARAMETERS ON CRYSTAL GROWTH OF COPPER SULPHIDE

It is necessary to study the effect of various parameters on crystal growth which is mainly depends on gel cell size, and cell size is influenced by gel density etc. Hence, these parameters have profound influence on nucleation density, growth rate habit and quality of crystals. Concentration of reactants is also important.

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VII. EFFECT OF GEL DENSITY

The gels of different densities were obtained by mixing sodium Meta silicate solutions of specific gravity 1.02 to 1.08 with 2N acetic acid, keeping pH value constant. It was observed that transparency of the gel decreased with increase of gel density gels with higher densities required less setting time of gel compared to the gels with lower densities. It may be noted that well defined and transparent crystals were obtained with sodium Meta silicate solution of density 1.04gm/cc. On the other hand, gels with densities below 1.04 gm/cc required longer time to set and still gels were not stable .Density of 1.02 gm/cc was the lower practical limit .The effect of gel densities on the quality of crystals as shown in table 5.5. The variation of gelation time with gel density shown in figure5.8 .It is observed that the gelation time decreases with increase in gel density .Table 1 shows the effect of density on number of nuclei formed. A greater gel density implies smaller pore size and poor communication among the pores and thus decreasing the nucleation density. Bechhold et al showed that diffusion coefficient becomes distinctly smaller as gel densities increased. There is no evidence that the diffusion constant of small atoms was greatly influenced by the silica gel density as long as the density is low. Thus, the diffusion constant is not greatly influenced by the presence of dilute gel.

TEST TUBE NO.	ACETIC ACID 2N (CC)	COPPER CHLORIDE INCORPORATED IN GEL 1M	DENSITY OF GEL (GM/CC)	GEL SETTING TIME (DAYS)	OBSERVATIONS
1	5	5	1.02	15	Gel is not stable
2	5	5	1.03	13	Long time required to set gel
3	5	5	1.04	12	Few crowded crystals
4	5	5	1.05	10	Number of crystals decreases different shaped crystals exist
5	5	5	1.06	08	Less transparent
6	5	5	1.07	07	Not well defined crystals

Tab.1 Effect of gel density on setting time (pH=4.4, feed solution H2S dissolved in distilled water)

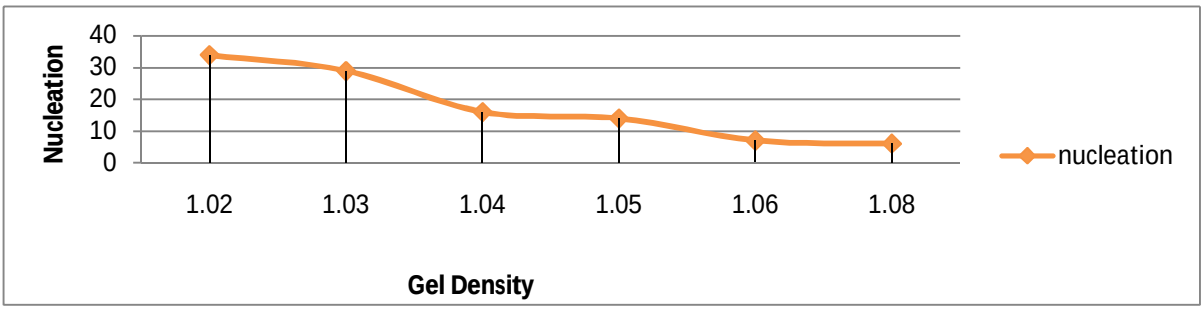

Fig. 2 Nucleation with gel density

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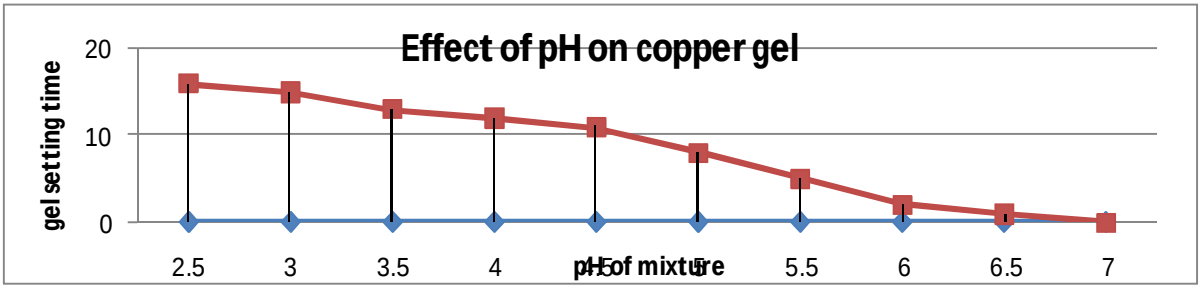

Fig. 3 Plot of pH against gel setting time in days References

VIII. EFFECT OF GEL AGING

Gel aging plays an effective role on the growth of copper sulphide crystals .To investigate the effect of aging on gels, gel of same pH and density were allowed to age for various periods before adding the feed solution over a set gel containing copper chloride. Supernatant of constant molarities was then added as a feed solution over the set gel. It was found that number of copper sulphide crystal decreases with increase in aging of gel. Aging of gel decreases the pore size as well as diffusion and nucleation density. More aging causes more amount of water evaporation out of the gel. The effect of water evaporation should be considered before and after the formation of gel framework. Before the gel is set the evaporation of water causes an increase in gel density which in turn decreases the diffusivity of reactive sulphide ions in the gel, thereby decreasing the number of nucleation sites. Table-2.shows the effect of aging time on number and the quality of crystal . In present work, aging of 120 hours was found suitable because it makes gel neither dry or brittle nor fragile. The aim of reduction in nucleation centres can also be achieved. Hence aging period of 120 hours is the optimum condition for the growth of good quality crystals.

IX. X-RAY DIFFRACTION (XRD)

X-ray diffractogram is useful in the analysis of crystal structure, d-values, cell parameters, unit cell volume and lattice system etc. can be evaluated using X-ray diffractogram. When the high frequency electromagnetic waves are selected to have wavelength comparable to the interplaner spacing of the crystals, they are diffracted according to the physical laws. The inter planer spacing (d) can be calculated to four digits and even more significant figures by measuring the diffraction angles. This, in turn, can be used to determine cell parameters and the system to which the sample under study belongs, etc. the reflecting planes in crystal h, k, l values can be calculated.

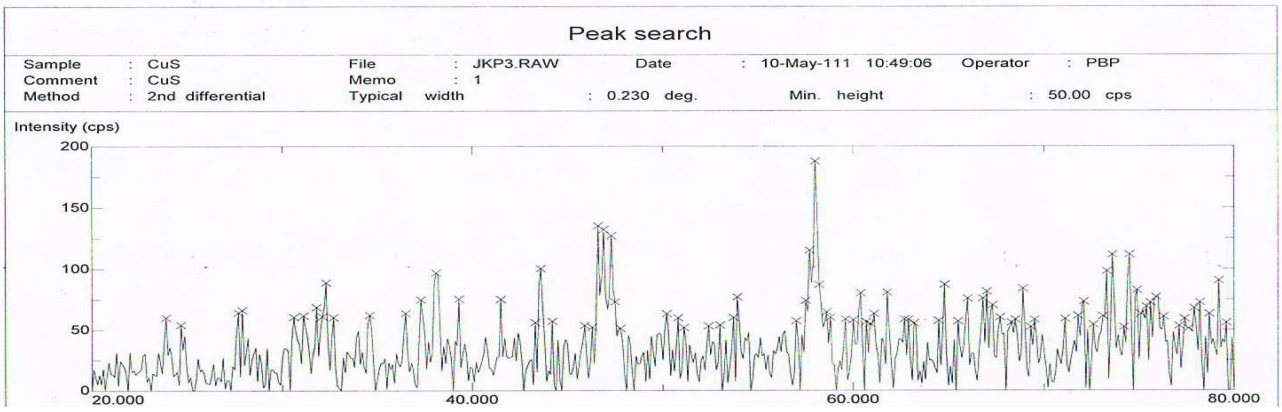

Fig 4. X-ray Diffractogram of copper sulphide

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ISSN (Online): 2278 – 8875

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X-ray diffractogram of gel grown crystal of copper sulphide was recorded using Minislex model ,Japan with Cukα radiation of wave length 1.5408°.A and scanning speed of 10 0/minute. A copper target and nickel filter were used From the powder diffractogram on data of copper sulphide which shows twenty different peaks and corresponding d values and (h k l) values were computed by using computer program POWD [An interactive powder diffraction data interpretation and indexing program] The recorded X-ray diffractogram is as shown in fig. 4. The present work describes the characterization of copper sulphide crystals by Following Techniques. the X-ray and Thermal behaviour of samples were practically performed at Jalgaon in North Maharashtra University Maharashtra.

Peak No.	2theta	FWHM	d-value	Intensity	I/I0 ratio	Hkl
1	23.900	0.118	3.7200	60	32	110
2	24.700	0.118	3.6013	54	29	200
3	27.700	0.235	3.2177	64	35	101
4	27.900	0.118	3.1951	66	36	011
5	30.600	0.235	2.9190	61	33	
6	31.100	0.471	2.8732	62	33	-301
7	31.800	0.118	2.8116	69	37	-202
8	32.100	0.235	2.7860	61	33	
9	32.300	0.118	2.7692	89	48	210
10	32.700	0.235	2.7362	60	33	-102
11	34.600	0.353	2.5902	62	34	111
12	36.500	0.235	2.4596	64	34	-302
13	37.300	0.353	2.4087	75	40	300
14	38.100	0.353	2.3599	97	52	002
15	39.300	0.118	2.2906	75	41	-112
16	41.500	0.118	2.1741	75	41	020
17	43.300	0.118	2.0878	56	30	-402
18	43.600	0.235	2.0741	101	54	012
19	44.200	0.118	2.0473	57	31	211
20	45.900	0.118	1.9754	54	29	021

Tab. 2 Powder Diffraction Data for copper sulphide crystals with calculated (h k l)

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These values are computed using computer programmed, POWD is as shown in the table 5.8. From POWD it found the lattice parameter of unit cell satisfy condition a#b#c and α=β=90 and γ#90.so unit cell structure is monoclinic. Calculated unit cell lattice parameter of the copper sulphide crystal.

X. BAND GAP DETERMINATION - UV-VIS SPECTROPHOTOMETRY

The optical property of copper sulphide crystal can be studied using UV-VIS spectrophotometer. A fine powdered form of copper sulphide crystal was used as sample .The reflection and absorption spectra of copper sulphide crystals have been recorded over the wavelength range 200 to 700mm using a UV-2450 spectrophotometer of SHIMADZU scientific instruments at the room temperature. The experiment was carried out in the research laboratory of the physics department at Pratap College, Amalner Maharashtra.

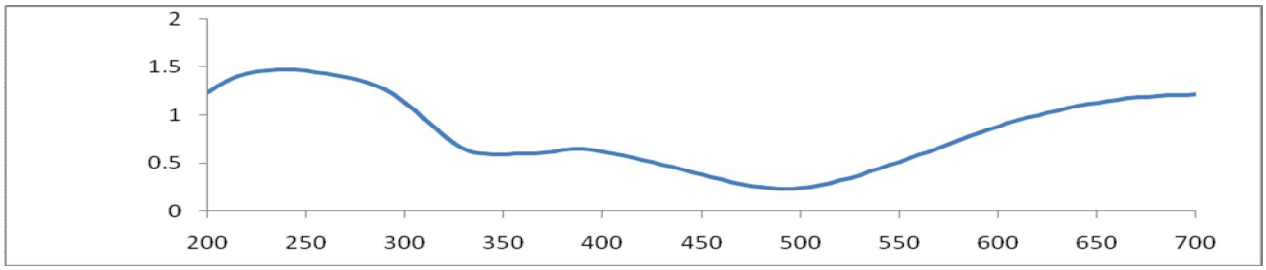

Fig. 5 UV-VIS Spectra of copper Sulphide

WAVELENGTH	ABSORB	λ	ABSORB
200	1.239	410	0.583
205	1.297	415	0.562
210	1.353	420	0.533
215	1.399	425	0.507
220	1.432	430	0.482
225	1.457	435	0.458
240	1.47	450	0.38
245	1.468	455	0.354
250	1.458	460	0.33
255	1.444	465	0.306
260	1.429	470	0.286
265	1.412	475	0.268
270	1.391	480	0.253
275	1.375	485	0.242
280	1.341	490	0.236
285	1.303	495	0.237
290	1.263	500	0.244
295	1.204	505	0.256
300	1.129	510	0.274
305	1.051	515	0.295
310	0.956	520	0.319
315	0.865	525	0.347
320	0.779	530	0.376
325	0.705	535	0.408
330	0.651	540	0.442
335	0.615	545	0.477

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340	0.599	550	0.512
345	0.591	555	0.55
350	0.592	560	0.586
355	0.599	565	0.624
360	0.604	570	0.663
365	0.605	575	0.7
370	0.612	580	0.737
375	0.624	585	0.775
380	0.641	590	0.811
385	0.652	595	0.846
390	0.649	600	0.88
395	0.637	605	0.913
400	0.621	610	0.943

Tab. 3 UV-VIS Spectrum result of copper Sulphide

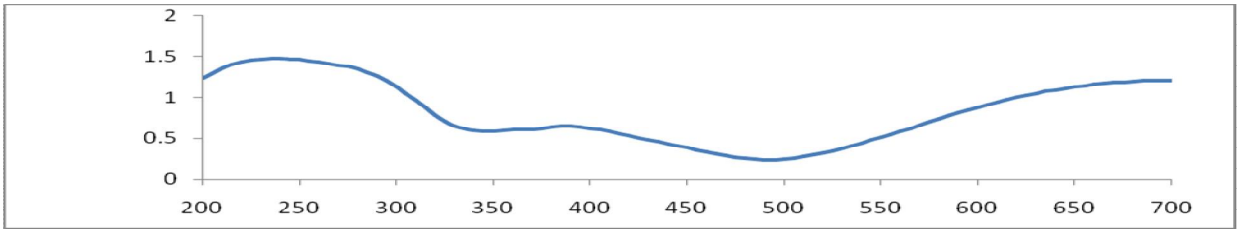

Fig. 6 Calculation diagram of copper Sulphide Band gap of copper sulphide=1240/340=3.64 EV
Band gap of copper Sulphide=3.64 EV

XI. CONCLUSION

The Crystal of Semiconductor Copper Sulphide Can Be Grown by Using Gel Technique .Single Diffusion Gel Growth Technique Is Suitable Copper Sulphide Crystals.Different Habits Of Copper Sulphide Crystals Can Be Obtained By Changing Different Parameters In Crystal Growth.Unit Cell Parameter Value And D Values Match Very With The Reported OnesThe Structure Of Copper Sulphide Is Monoclinic Confirmed By X-Ray Diffraction.
Fundamental Infra-Red Frequencies, Generally Observed In All Sulphide Compounds.A Band Gap Determination Shows Variety Of Applications In Electronics And Electrical Devices And For Engineering Materials.

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