IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Growth And Characterization Of Pure Polycrystalline Nacl

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Keywords: Transparent NaCl poly crystals, Optical energy gap, refractive index, Emission & excitation study.

Abstract. In these investigations, sufficiently large transparent poly crystals, free from micro-cracks were grown in a ceramic crucible using a simple low-cost chemical method of growing from the melt by rapid cooling. The X.R.D., D.T.A., T.G.A., refractive index and Luminescence study showed excitation peak at 244nm and emission peak at 360 nm which supports the formation of NaCl poly crystals. It is observed that in grown poly crystals NaCl, hygroscopic character has been reduced more over the micro hardness appears to have been increased. The work suggests to quantify the optical band gap of the grown NaCl polycrystal and its comparison with the reference standard to test the insulating property of the material. The value of optical energy gap is calculated & found to be 4.821eV & 3.288eV respectively for excitation and emission luminescence.

Introduction

Introduction: Alkali halides are those stable compounds which are formed by appropriate chemical combination of atoms from the first group IA namely Li, Na, K, Rb & Cs and corresponding halogens such as F, Cl, Br or I from the periodic table. Till date alkali halides are preferred by material researchers for their simple structure and property of easy to reproduce at the research as well as at commercial level. In 1946 Seitz remarked "In the field of material science, alkali halides are the most interesting crystals as they have continuously yielded to persistent investigation and have gradually provided better and better understanding of the most interesting properties of all solids". The statement remains relevant even today [1]. The alkali halides crystallize either in the NaCl (sodium chloride) structure or in the Cs Cl (cesium chloride) structure. All the alkali halides have congruent melting points and therefore their crystals can be formed from their melts. The alkali halide crystals with Na CI structure have a beautiful cleavage along the (100) plane whereas those with Cs CI structure do not exhibit cleavage [2]. These compounds are the often commercially significant sources of these metals and halides. The best known of these compounds is sodium chloride, table salt [3]. Owing to the simple cubic structure, the alkali halides have been largely studies, which played a crucial role in progress of crystal physics [4]. Among all single crystals, scintillator crystals have gained a lot of attention, and many attempts have been made to grow their single crystals with highly potential for huge applications such as medical imaging, dosimetry, nuclear detectors and homeland security [5]. Till date several techniques have been developed to grow single crystals, and Czochralski method (CZ) is one of the most common but very costly used method to grow high-quality single crystals using top seed solution growth (TSSG) [6-10]. The alkali metal halides exist as colorless crystalline solids, although as finely ground powders appear white. They melt at high temperature, usually several hundred degrees to colorless liquids. Their high melting point suggest their high lattice energies. All twenty stable alkali metal halides are known; the unstable ones are not known, with the exception of sodium astatine, because of the great instability and rarity of astatine and francium. The most well-known of the twenty is certainly sodium chloride, otherwise known as common salt. They are all white ionic crystalline solids that have high melting points [11]. Most alkali metal halides crystallize with the fcc lattices. In this structure both the metals and halides feature octahedral coordination geometry, in which each ion has a coordination number of six, similar to the structure of ferroelectric potassium niobate at room temperature.[12] The knowledge of the structure and chemical bonding in alkali halides has made them the suitable and favorite systems both for experimental and theoretical studies for verifying theories. Alkali halides were the first materials of choice in many pioneering experiments. Potassium chloride was the first crystal used by Laue in his famous experiments on X-ray diffraction and NaCl was one of the crystals which Bragg chose for structural analysis. Alkali halides are the convenient systems for scaling of properties. The lattice constant has been used to scale a large number of properties like elastic constants, hardness, colour center parameters like concentration of defects, Debye's temperature, etc. Thus, whether it was testing of theories or trying new experiments, the alkali halides have always played a leading role as model [12]. Yokota etal etal[13] have grown and investigated luminescent properties of pure and Ce, Pr doped NaCl single crystals grown by the modified micro-pulling-down method. For pure NaCl and Pr: NaCl crystals, the transmittance spectra indicated almost more than 60% in the wavelength from 200 to 800 nm and an absorption of Ce3+ ion was observed in the transmittance spectrum of Ce: Na Cl crystal. The emission spectrum originated from Ce3+ 5d–4f transition appeared around 300 nm in the photoluminescence spectrum and the decay time was 19.7 ns.Single crystals of NaCl doped with Ca and Mn have been grown and irradiated G. Sánchez-Mejorada etal [14] with ionizing radiations. The induction of defects has been correlated to the increase in the intensity of the thermo luminescent glow curve as a function of doses. The glow curves intensity as a function of doses shows the potential candidate for dosimeters. Moreover, study of optical properties of these crystals when irradiated with gamma rays have shown their potential as a good detector and optical store memory devices. Thermoluminescence and F centers of manganese doped Pure, manganese doped NaCl and NaCl-KCl crystals were grown by Czochralski method by L. Somera etal [15] and were investigated after irradiated by γ -radiations. Authors are able to conclude that the Optical bleaching with 470 nm light confirms successfully the F centers participation in the Thermo Luminescence process of Mn doped sodium chloride crystals. The existence of multiple traps generated by the manganese impurity is supported by the thermal bleaching and deconvolution of the glow curves.

Transmission electron microscopy, X-ray diffraction and characterization of Cu Cl quantum dots (QDs) grown in NaCl single crystals were reported by Miyajima etal [16] via optical measurements. Their results can help to reveal the growth mechanism of semiconductor QDs embedded in a crystallite matrix. In addition, this work will play an important role in progressing the study of optical phenomena originating from assembled semiconductor QDs.

Vinogradov etal[17] reports a significant increase of the bulk damage threshold in the case of interaction of CO2 laser radiation pulses with ultrapure NaCl and KCl crystals grown in a reactive atmosphere was observed on introduction of divalent metal ions Ca and Pb in concentrations of 10-5-10-6 mol/mol. In this study a physical model was developed to account for the observed dependence on the basis of an analogy between a system of colloidal particles and F centers in a crystal and a liquid-vapor system.

Explosive phenomena in heavily irradiated NaCl [18] can be initiated during irradiation or afterwards when samples are heated to temperatures between 100 and 250 degrees C.

EPR and optical absorption studies of X-irradiated cobalt doped sodium chloride crystals were studied by S. C. Jain etal.[19]. The results combined with those of dielectric loss and optical studies show that X-irradiation of Co doped crystals produces new centers, labelled as S centers, which produce a dielectric loss peak, a decrease in electrical conductivity, an optical band at 210 nm and the EPR line. Possible models of the S centers are discussed.

B.Wang etal [20] reports that NaCl single crystal templates are water-soluble, environmentally-friendly and uniform in both geometry and size, as such are ideal for preparing high quality hollow nano/micro structures. The new approach may have the potential to replace the conventional hard or soft template approaches. Their work has revealed the formation mechanism of nano/micron NaCl crystals with different sizes and geometries.

The Structure and Liquid Flow Effect of Melt during NaCl Crystal Growth is reported by Y.Li etal [21] they found that the shape of a solid–liquid interface will change with the rotation rate or diameter increase. These studies may provide theoretical support to a further study on crystal growth, other alkali halide, and alkaline earth metal halide melt. The present work has been intended to achieve a sharp peak in luminescence by modifying the growth process , reduce hygroscopic character and grow seed crystals which may be used for variety of applications.

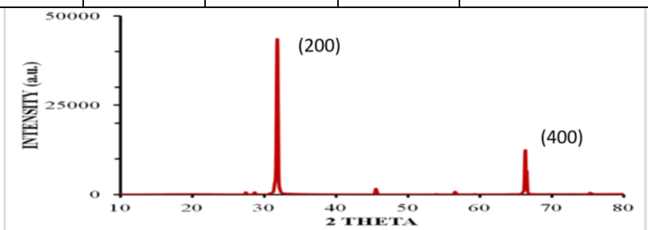
Experimental

The powder of NaCl (A.R. grade 99.99 %) was used for the growth of NaCl polycrystals. The starting material was heated in a ceramic crucible around 1000˚C, for a soaking period of three hours, using a super canthel wire wound furnace where temperature was maintained with ±10˚C accuracy. The melt was allowed to cool rapidly up to room temperature by making the electrical power off. Characterization methods namely X ray diffraction (XRD), Photoluminescence (PL), Differential Thermal Analysis (DTA), Thermogravimetric analysis (TGA), Refractive index (RI) were employed to investigate any deviations compared to the standard data available so far. The phase identification of the NaCl powder was performed by X-Ray Diffraction (XRD) on an X-Ray Diffract meter using Cu Kα radiation of wavelength 1.5405 Å. The angular accuracy was ±0.001˚and angular resolution was 0. 01˚.The 10 mg amount material was taken for XRD characterization. Powder XRD have been carried out for the NaCl polycrystalline. As shown in the Table 1, the (hkl) planes were determined by using a standard JCPDS card No.78-0571[22] to ascertain the quality of grown NaCl polycrystals in the laboratory. The PL spectra (excitation and emission) were recorded on a Hitachi 5301 fluorescence spectrophotometer. D.T.A.-T.G.A was made on PERKIN ELMER, USA model: Diamond TG/DTA, the heating rate was 10˚C/min from room temperature up to 1000˚C.The refractive index was determined by using model NAR-3T, CAT NO.1230 scale Refractive Brix, Measurement Range Refractive index (nD): 1.30000 to 1.71000 Brix: 0.00 to 95.00%, Measurement Accuracy Refractive index (nD): ±0.0001 Brix: ±0.05%.

Result and Discussion

Table (1). X- Ray Diffraction data of polycrystalline NaCl

2θ	I	IR	h	k	l	Lattice parameters a˚ A
28.7	670	1.5	1	1	1	5.3835
31.86	43559	100	2	0	0	5.6136
45.58	1624	3.7	2	2	0	5.6237
53.96	175	0.4	3	1	1	5.6314
66.36	12458	28.6	4	0	0	5.6301
75.34	506	1.1	4	2	0	5.6363
84.02	459	1	4	2	2	5.6382



Fig(1).X-Ray Diffract graph for pure polycrystalline NaCl.

The X- Ray Diffraction data Table 1 and Fig 1 shows similar characteristics as that of standard NaCl crystal. Lattice constant (a) =5.6136˚A corresponding to maximum intensity and (100) plane and (a)= 5.6382˚A corresponding to maximum angle is nearly equal to the referred lattice constant (a)= 5.63978˚A [23].

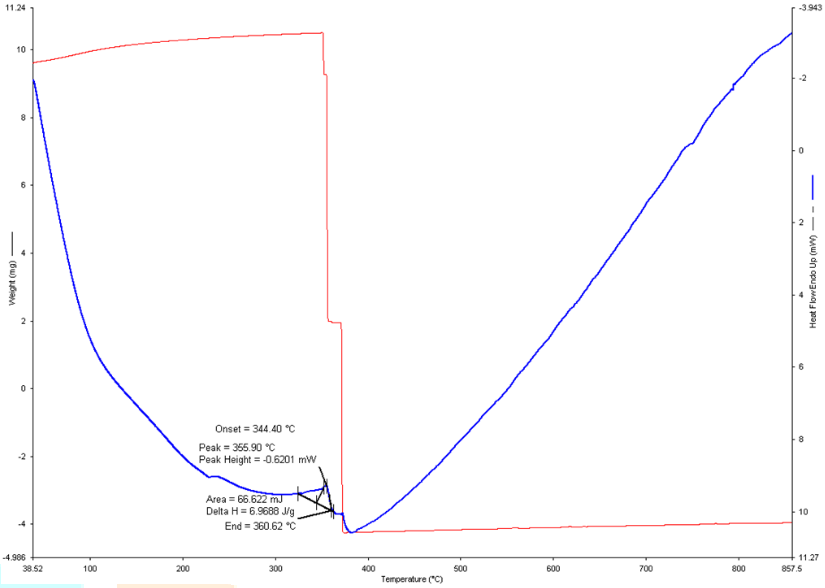


Fig (2). DTA and TGA variations

From DTA-TGA Fig (2). study we observe a peak at 355.90˚C this may be due to the structural changes, however in our case sharp endotherm is not observed. Hence the melting point could not be ascertained T.G. A indicates that the grown NaCl polycrystals is quite stable. The weight loss in NaCl is not observed after 400˚C.NaCl has not decomposed its weight remain constant and NaCl remain as sodium metal.

Table (2). Refractive Index data of Polycrystalline NaCl

Sr. No.	Sample	Refractive Index		Reference
		Our Data	Standard data	
1	NaCl Powder	1.346	1.5442	G.Y. Rajurkar[24]
2	NaCl Polycrystal	1.345	1.5442	B.H Gurnani [25]

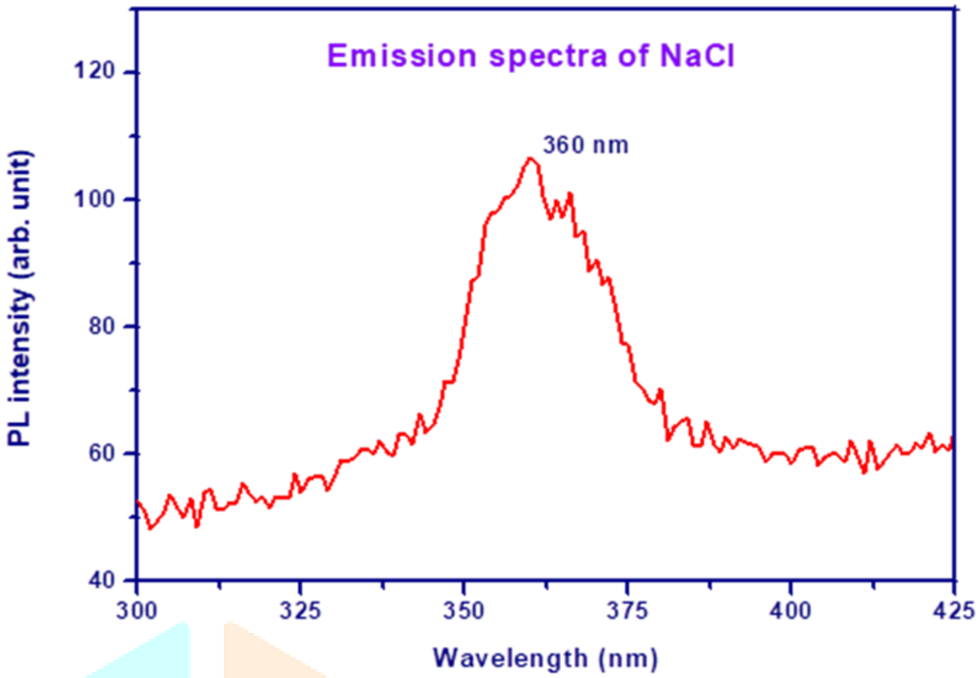


Fig (3) PL Emission spectra NaCl polycrystal

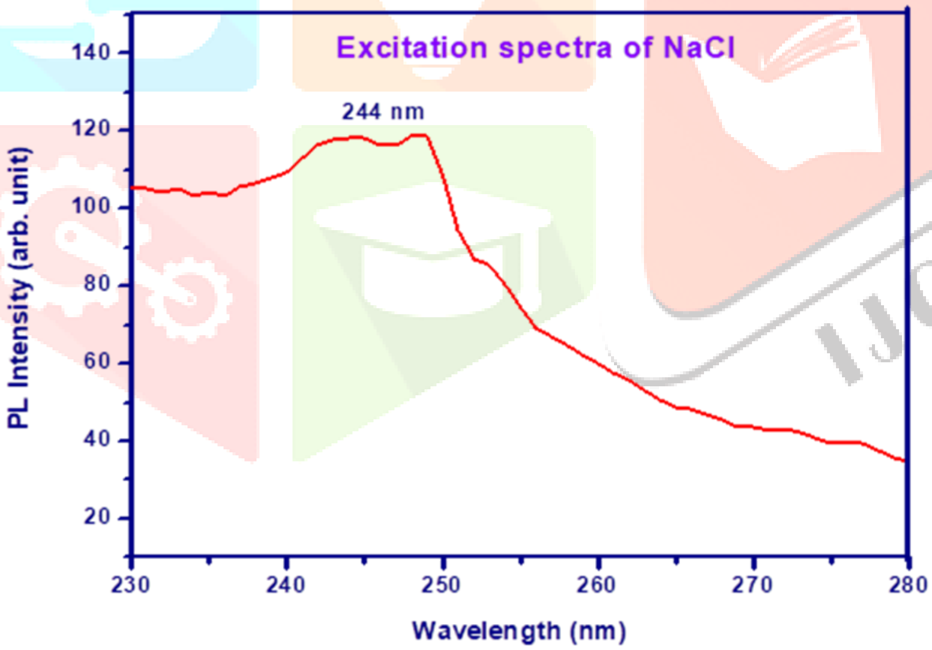


Fig (4) PL Excitation spectra NaCl polycrystal

The Photoluminescence spectra of polycrystalline NaCl forms exhibits violet phosphorescence, The Emission peak observed at 360 nm as shown in Fig. (3) and the Excitation peak is observed at 244nm as shown in Fig. (4). From the peak value in the emission spectra the value of the energy gap has been calculated. It is found to be 3.288 eV. The value confirms the insulating property of polycrystalline NaCl. More over the excitation spectra suggests the value of the optical band gap having value 4.821 eV for the excitation process. However, the value appears to be quite less in comparison the reported value.8.75eV [26] ,8.5eV [27].and 2.85eV [28] which suggests a decline in a dielectric character.

Conclusion

The X.R.D, D.T.A., T.G.A, Luminescence study, refractive index & activation energy calculations help us to conclude that the grown material was polycrystalline NaCl. The grown polycrystals can be effectively used as seed crystal for nucleation to grow large size crystals. They can also be used as a matrix for addition of nanocrystals as activators. Lastly can be explored as scintillators, dosimeters and nuclear detectors.

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