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Research Article

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SEM, EDXRF and SHG efficiency of Co(II) Doped Lead Iodate Crystals Grown by Gel Technique

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ABSTRACT

Lead iodate crystals doped by cobalt were grown in silica gel method. Grown crystals were characterized by scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDAX), X-ray fluorescence spectroscopy (EDXRF) and Non linear optical (NLO) property. SEM pictures shows that no morphological or structural changes take place due to doping. EDXRF and EDAX confirms that Co(II) is intercalated in lead iodate crystal. The emission of green radiation from the samples ascertains the NLO property of the crystals.

Key words: doping, SEM, EDAX, EDXRF and second harmonic generation.

INTRODUCTION

Now-a-days the search for new non-linear optical (NLO) materials is an important task because of their practical applications in harmonic generation (Frequency doubling), amplitude and phase modulation, laser technology, switching and other signal processing devices. Most of the organic NLO crystals usually have poor mechanical and thermal properties and are susceptible for damage during processing even though they have large NLO efficiency. Purely inorganic NLO materials have excellent mechanical and thermal properties [1]. Iodates have important electro-optical properties [2, 3] because of the un-bond electron pair of iodine atoms in $(IO_3)^-$ anions [4] so most of the iodates exhibit prominent NLO behavior. From the literature survey, it was found that, not a single report on the growth and characterization of cobalt doped lead iodate crystal is available, with these aspects in the mind; it has been decided to grow and study the cobalt doped lead iodate crystals in view of search for new materials with high optical nonlinearities.

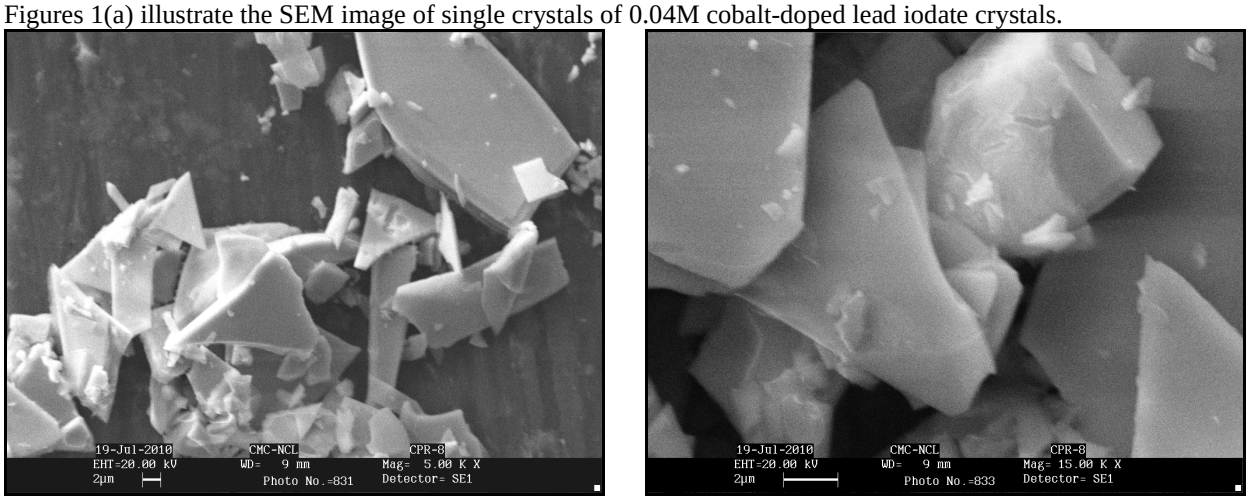
EXPERIMENTAL SECTION

Cobalt-doped lead iodate crystals were grown in silica gel using single diffusion technique. The concentration of cobalt dopant was 0.04M. Its growth and XRD analysis is already reported [5].

In the present work powdered sample of lead iodate crystals were examined by using LEICA S440 SEM instrument at the National Chemical Laboratory, Pune. An elemental composition were carried out to know the chemical composition of the elements using FEI quanta 200 3D EDAX instrument at National Chemical Laboratory, Pune. The elemental composition of the grown crystals was also verified with X-ray fluorescence spectrometer. EDXRF was carried out using Horiba XGT-7200 instrument at Gemmological Institute of India, Mumbai. Kurtz and Perry powder SHG test [6] was carried out at the Department of Inorganic and Physical Chemistry, Indian Institute of Science (IISC), Bangalore.

RESULTS AND DISCUSSION

Scanning electron microscopy (SEM):
Figures 1(a) illustrate the SEM image of single crystals of 0.04M cobalt-doped lead iodate crystals.



(a) **(b)**

Figure 1: (a) SEM picture of 0.04M Co-doped lead iodate crystals and (b) magnified SEM picture

All SEM images shows plate like crystal morphology and crystals are grown by layer deposition. Thick and thin layers are seen in figures. The individual plates of samples are flat and the plates with the sharp edges were observed. On some plates further plate like growth was observed. Higher magnification SEM images are shown in figures 1(b) shows more clearly layer structure of doped crystals [7]. The SEM images of lead iodate crystals are reported [8]. It was found that no morphological or structural changes take place due to doping.

Energy dispersive X-ray spectroscopy (EDAX):
Figures 2 shows EDAX spectrum of cobalt doped lead iodate doped crystals. The peaks show the presence of lead, iodine, oxygen and cobalt in the doped crystals. This is a clear indication of presence of the cobalt dopant in the crystals. Table 1 shows the elemental and atomic percentage of the elements Pb, I, O and Co in the doped crystals. It was observed that atomic % of Pb, I and O are in good agreement with stoichiometrically expected atomic % 11.11, 22.22 and 66.66 respectively.

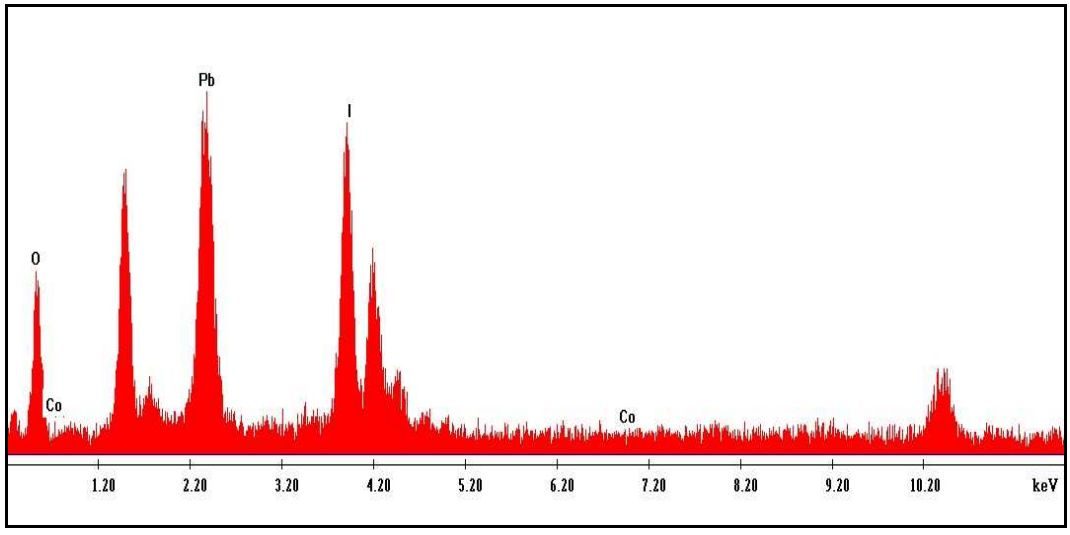


Figure 2: Energy dispersive spectrum of 0.04M Co doped lead iodate crystal

Energy dispersive X-ray fluorescence (EDXRF):
XRF was performed at the selected region of sample. The result is given in Table 2. From the result, it is concluded that there is little deviation in weight % and atomic % of the grown crystals from the theoretically calculated values.

Table 1 Values of elemental content of $Pb(IO_3)_2$: Co crystals

Elements	Experimental values	
	Weight/%	Atomic/%
Pb M	38.97	11.69
I L	43.39	21.25
O K	17.12	66.50
Co K	0.53	0.55

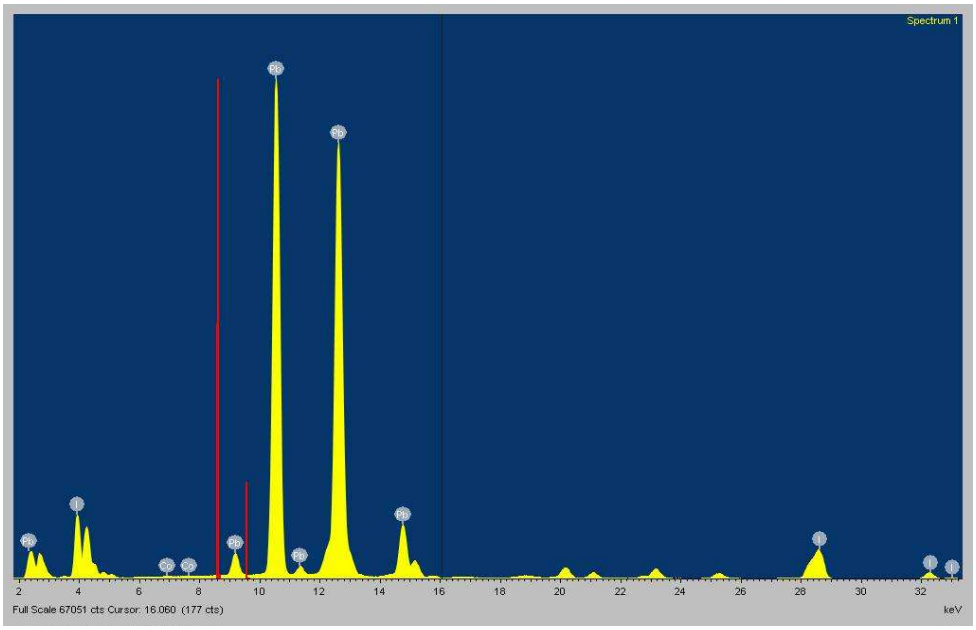


Figure 3: EDXRF spectrum recorded for cobalt-doped lead iodate crystal

Figure 3 shows the XRF spectra of 0.04M cobalt-doped lead iodate crystals. The spectrum shows strong peaks of Pb and I which indicates the presence of Pb and I in the sample. The weak peak of Co shows that very little amount of cobalt is doped in the crystals lead iodate. The content of oxygen cannot be measured accurately because of its low atomic mass.

Table 2 Elemental compositions of 0.04M Co doped lead iodate crystal

Elements	Line	Experimental values		Theoretically calculated values		Intensity [cps/mA]
		Mass[%]	Atomic[%]	Mass[%]	Atomic[%]	
I	K	44.34	21.88	45.567	22.22	3010.63
Pb	L	38.95	11.98	37.199	11.11	24605.53
Co	K	0.67	0.72	--	--	26.42
O		16.04	65.42	17.234	66.66	--

Non linear optical (NLO) property:

Cobalt-doped lead iodate crystals were irradiated by an incident radiation (1064 nm) from Q-switched quanta PROLAB 170 Nd: YAG laser of pulse width 10 ns and pulse energy of 2 mJ / pulse. KDP was used for calibrating the SHG intensity. Second harmonic signals (532 nm) of 20 mV obtained through KDP while 4.1mV and 2.3 mV were obtained through undoped, 0.04M Cobalt-doped lead iodate crystals respectively. Thus, the SHG efficiency of doped crystals decreases due to the dopant. The emission of green radiation from the samples ascertains the SHG property of these crystals.

CONCLUSION

In conclusion, SEM pictures shows plate like morphology of the grown crystals and no morphological or structural changes take place due to cobalt doping. The incorporation of Co^{2+} in the crystalline matrix of lead iodate crystals is

well confirmed by EDXRF and EDAX. NLO studies reveal that doped crystals exhibits SHG (frequency doubling) property but its SHG efficiency decreases cobalt dopant.

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