Scientific Journal of Impact Factor (SJIF): 4.72

e-ISSN (O): 2348-4470
p-ISSN (P): 2348-6406

International Journal of Advance Engineering and Research Development

Volume 4, Issue 11, November -2017

INVESTIGATION OF LUMINESCENCE STUDIES ON Tm^{3+} DOPED KBr SINGLECRYSTALS

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ABSTRACT:- *Tm^{3+} doped KBr single crystals have been grown from melted by using a Bridgemanstockbarger technique. The characterizations are Optical absorption-transmittance, Photoluminescence [PL], Thermo luminescence [TL], Powder X-Ray diffraction [PXRD], Laser Raman spectroscopy and Scanning Electron Microscopy [SEM]. The optical absorption - transmittance study confirms the suitability of the grown crystals for optical device applications. UV- Vis transmittance spectrum was recorded in the range 190-1100nm. PL emission wavelength was observed around at 451nm blue emission with 350nm excitation.This451nmattributed tothe $^1G_4 \rightarrow {}^3H_6$ transition. The grown crystals have been subjected to PXRD studies to determine the lattice parameters have been calculated and are presented. It is observed that crystallizes is in Hexagonal system with space group Fm3m. SEM micrograph analysis, the particle size ranges from few microns to 5µm. Trap parameters for the TL glow curve were calculated by Chen's peak shape method. TL emission confirms the participation of Thulium ions in the recombination process in host material .*

Keywords: *Optical absorption, PL, TL, Laser Raman, PXRD, SEM*

1. INTRODUCTION

Luminescence properties of trivalent rare earth ion (Tm^{3+}) is doped with the alkali halide (KBr) and due to their optical applications such as optical storage systems, color displays and medical have been earlier reported work on many researchers[1,2]. Tm^{3+} ions has twelve electrons filled in the 4f shell ($4f^{12}$) and it is very useful for the electron transition process[3]. The most commonly used access in Rare Earth(Tm^{3+}) is doped light emitting diodes (LEDs) is the blue light luminescence, accommodate a broadband emission in the visible region for PL process[4,5]. Blue (or) other colored light can also be obtained via PL process in lanthanide doped materials[6,7], and exhibits great applications in color displays, high density optical data storage and reading, optical communications and biomedical diagnostics[8-13],etc. Their capacity incorporates high concentrations of rare earth (RE) ions and also makes them suitable candidates for expansion of activate photonic devices[14]. However, the choice of the host material is extremely important for the advance of efficient PL due to Optical devices based on RE doped crystals. In the emission spectra of the rare earth ions the different host materials, an important role belongs to the electron interaction, which determines the redistribution of PL process. As well as the Tm^{3+} doped KBr single crystals is famous dosimetric material used for measurement of ionizing radiation. Thulium ions have several advantages which have high absorption cross section and broad band absorption in the 190-1100nm spectral range. The blue emission band of trivalent thulium(Tm^{3+}) ions wavelength located around at 451nm as reported in earlier work[15]. Alkali halides due to their understandable structure have made the discerning of many properties of insulators possible, particularly with regard to calculation of defect parameters and ionic conduction etc. Alkali halides have a broad range of lattice parameters, which makes it possible to absorb impurity ions of different sizes and valence states in the lattices[16-19]. In the present work, namely Tm^{3+} doped KBr single crystals were grown by Bridgeman Stockbarger technique. The grown crystals were characterized by Optical absorption, Photoluminescence [PL], Thermoluminescence [TL], Laser Raman spectroscopy, Scanning Electron Microscope (SEM), Powder X-Ray diffraction(PXRD).

2. EXPERIMENTAL DETAIL

Single crystals of pure KBr (99.99% purity) and thulium doped KBr (99.99% purity) were grown using the Bridgman Stockbarger technique. Thulium was added in the form of thulium fluoride (Aldrich 99.99% purity).

The crystals were grown in three different concentrations of the impurity 1%, 3% and 5% by weight. Samples of size approximately $5\times5\times1mm^3$ were used for all except the PL studies. For PL $5\times5\times3mm^3$ samples were used. The results of the three concentrations were similar and hence only the results pertaining to a thulium concentration of 3% by weight are presented and discussed. The absorption and transmittancespectra were recorded using a Perkin Elmer Lambda 35UV–Vis spectrophotometer in the region 190–1100nm. PL spectra were recorded at room temperature using Perkin Elmer LS 55 Luminescence spectrometer in the region 200–900 nm with a spectral width of 5 nm. TL emission was recorded using a Perkin Elmer LS 55 with the excitation slit being closed. The phase of the PXRD crystals was confirmed by X-Ray diffractometer using CuKα radiation. The microstructure of the grown crystalswas studied using Scanning Electron Microscopy SEM-JEOL-JSM-5610LV model. The Laser Raman Spectroscopy were recorded at ReinsaawInvia spectrometer model (λ_L=700nm). Before TL experiment the crystals were annealed at 400°C for half an hour and then quenched to room temperature to ensure homogeneous distribution of impurity and to remove any storage effect.

3. OPTICAL ABSORPTION

The optical absorption spectra of Tm^{3+} doped KBr single crystals is as shown in Fig.1. curve (a), the 260 and 329nm absorption band is due to the exciton absorption of the host matrix [20]. The sharp absorption peaks are 357nm, 463nm, 688nm, 696nm, 696nm, 774nm and 793nm which are attributed to the transitions of $^3H_6\rightarrow{}^1D_2$,$^3H_6\rightarrow{}^1G_4$, $^3H_6\rightarrow{}^3F_2$, $^3H_6\rightarrow{}^1D_2$ and $^3H_6\rightarrow{}^3F_3$ respectively with in $4f^{12}$ electronic configuration of Tm^{3+} ions [21].

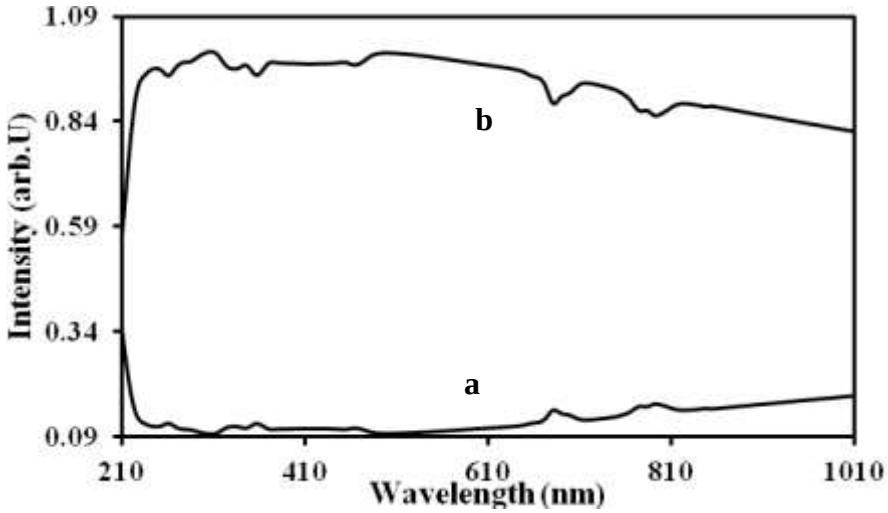

Fig.1 Curve(a) Optical Absorption , Curve (b) Transmittance Spectrum of Tm^{3+} doped KBr Single Crystals

This absorption lines are depended weakly on the host, since the 4f electrons are effectively shield by the filled SS and SP orbitals of Tm^{3+} ions. The optical transmittance of KBr: Tm^{3+} single crystals is shown in Fig1 Curve (b). It was recorded in the region 190- 1100nm using LAMBDA-35 UV-Vis spectrometer, which includes near UV, visible and far IR regions. This transmittance wavelengths are matched the absorption wavelengths, so the result was the grown crystal that is fully transparent and it is very useful for optical device applications.

4. PHOTOLUMINESCENCE

The photoluminescence is the most direct way to make light from a solid material. Rare earth based luminescent materials have considerable particle applications in almost optical device involving the artificial production of light[22-24].

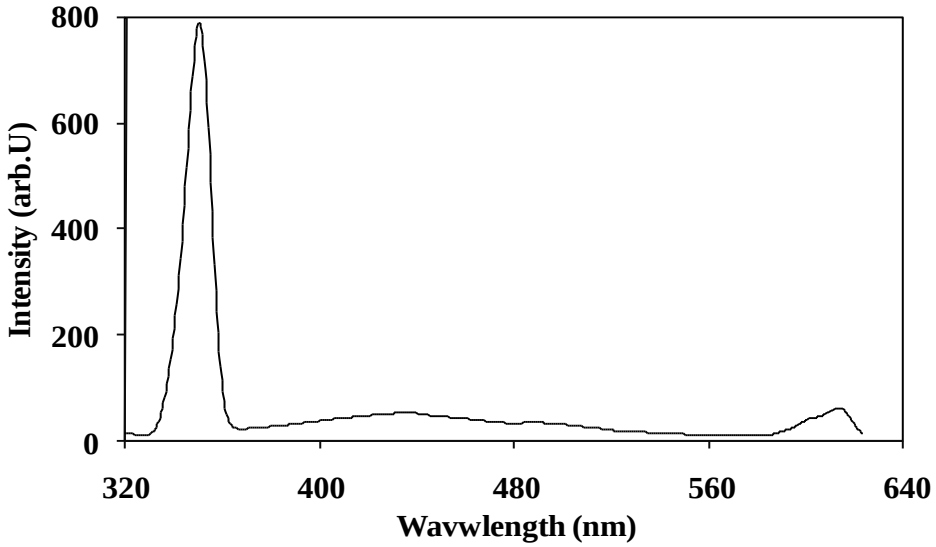

Fig.2 Excitation spectrum of KBr:Tm^{3+} Emission at 450nm

Fig.2 shows the PL excitation spectrum of Tm doped KBr crystals with emission wavelength at 450nm. The excitation bandwavelength consists of one sharp peak centered at 351nm which is in the visible region and assigned due to the intraconfigurational 4f shell transitions from the ground to excited state levels of Tm^{3+}ion. Weak band centered at 444 and 618nm. Recently studies on $KYbNWO_4O_2$: Tm^{3+}have identified the rare Tm^{3+}ion related 4f -4f transition [25], Tm^{3+} with $4f^{12}$ electronic configuration has complicated energy levels, so it is excitation spectrum containing of broad peaks due to 4f-4f transition of Tm^{3+}ions.

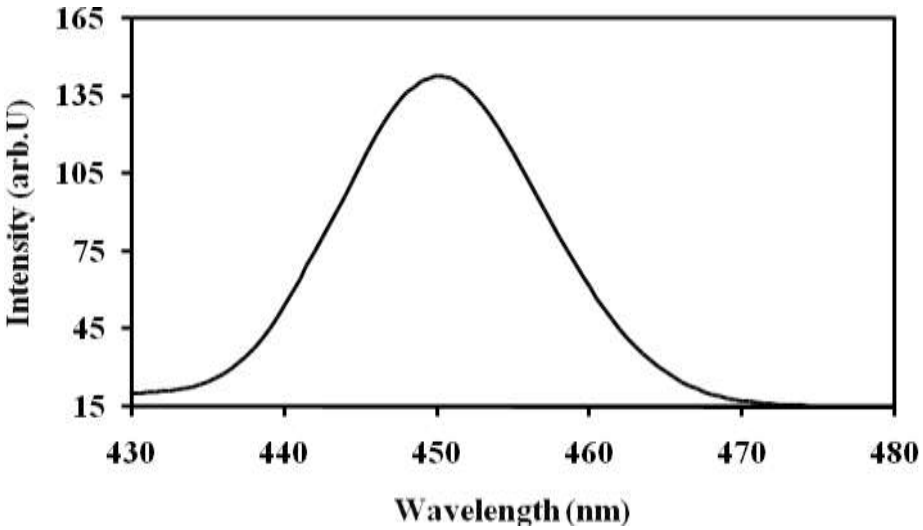

Fig.3 Emission Spectrum of KBr:Tm^{3+} for excitation at 350 nm

Fig.3. shows the PL emission spectrum of Tm^{3+} doped KBr single crystals with excitation wavelength at 350nm and exists the single broad emission band centered at 451nm, in the blue region. The Tm^{3+} ion in G emits a 451nm photon and returns to the H, through the $^1G_4 \rightarrow {}^3H_6$ transition [26]. In general,Tm^{3+} ion have complicated energy level schemes, due to the strong deviation from the 4f electronic configuration, and thus, through many paths, the relaxation of the excited states of Tm^{3+} ions can take place, giving rise to Ultraviolet, Visible, and Infrared emission with moderate intensity [27-29]. Among these emissions, in this case the Ultraviolet emission possesses the stronger intensity with high color purity, which may be used as blue light emitting process. Therefore, KBr:Tm^{3+} was prepared and investigated. In this investigation indicates the KBr host doped Tm^{3+} ions were exists an excellent luminescent property for optical device applications.

5. THERMOLUMINESCENCE GLOW CURVE

The TL glowcurve of KBr:Tm^{3+} crystals is shown in Fig.4. The grown crystal before taking TL were annealed at 400°C and then irradiated at a γ-ray dose of 5GY. TL was taken out soon after the irradiation to ignore the circumstance of error.

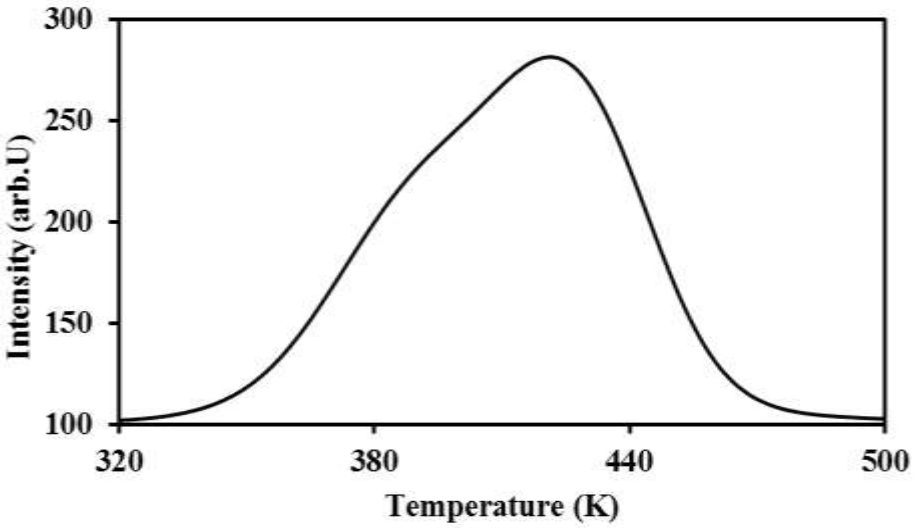

Fig.4 Thermoluminescence Spectrum of KBr:Tm^{3+} Crystals after γ– ray irradiation for 1-hour

The TL was observed up to 600K at a heating rate of 120°C/min. the glow curve reveals one clear and well distinguished peak at 422K. The TL intensity of this sample is lowcompared with the standard thermoluminescencedosimetry(TLD) material and therefore does not have any specific application in dosimetry. The TL glow curve are very useful to analyze the nature of traps present in the material and also gives us information about the energy absorbed by the material during irradiation. The kinetic parameters such as activation energy(E) and frequency factor(s) gives information about defect centers cause TL in the material[30]. So, we calculated these parameters of the prepared sample. The dosimetry properties of the TL materials mainly depend on the kinetic parameters of the glow peak. Considering this aspect, the parameters were calculated using Chen's peak shape method. This method is one of the broadly used technique to determine the kinetic parameters of the glow peak of TL materials.

For the calculation of these parameters the following shape parameters were to be evaluated $\omega = T_2 - T_1$(total half intensity width), $\delta = T_2 - T_m$(high temperature half width) and $\tau = T_m - T_1$(low temperature half width). The order of kinetics can be evaluated from the geometric factor (μ_g) of the glow peak ($\mu = \delta/\omega$) and depends on the glow peak. The value ofμ_g for second order kinetics is 0.48 . The geometric factor is around below 0.5 and indicates that the crystal obeys second-order kinetics. The activation energy(E) and frequency factor(s) can be calculated from equations as was reported in earlier work[31]. The trap depth for 422K glow peak is 0.88eV, frequency factor for 422K glow peak were found to be 1.32×10^9 s^{-1}. From the data, the peak at low temperature (i.e) 422K is due to low energy traps. From this, we conclude that low temperature peak are related with shallow traps.

6. THERMOLUMINESCENCE EMISSION

TL emission of KBr:Tm^{3+} consist of two distinguishable emission peak at 440nm is Fig.5 (curve a) and 475nm is Fig.5 (curve b) corresponds to glow peak temperature at 422K. Among both emission band, the peak with high intensity peak 475K is overtake for deconvolution.The deconvolution formed by means of using software "Origin5.0". TL deconvoluted emission were carried out on KBr:Tm^{3+}single crystals between the wavelength 300-700nm.The deconvoluted TL emission spectrum consists of two well distinguished peaks at 442nnm and 503nm as shown in Fig.6. The PL emission band wavelength observed at 451nm assigned to the ($^1G_4 \rightarrow {}^3H_6$) transition of Tm^{3+}ions [26].

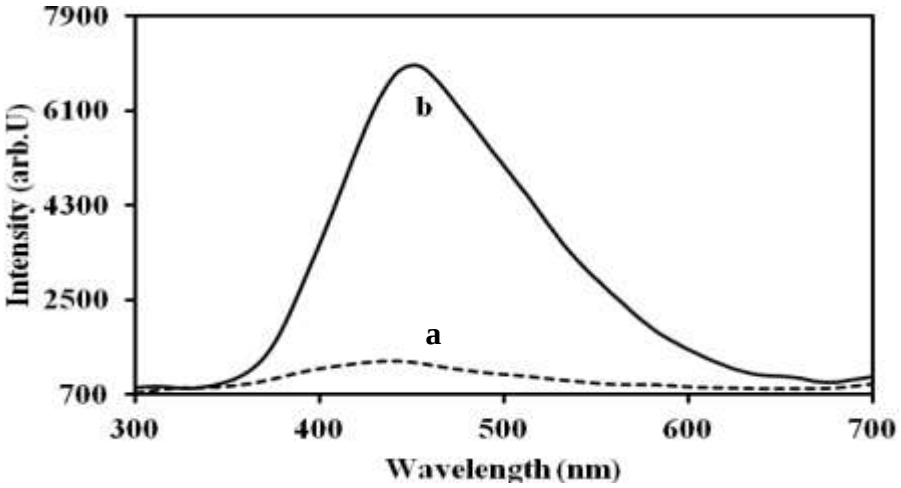

Fig.5 TL Emission spectrum of KBr:Tm^{3+} at (a) 440 K , (b) 475 K

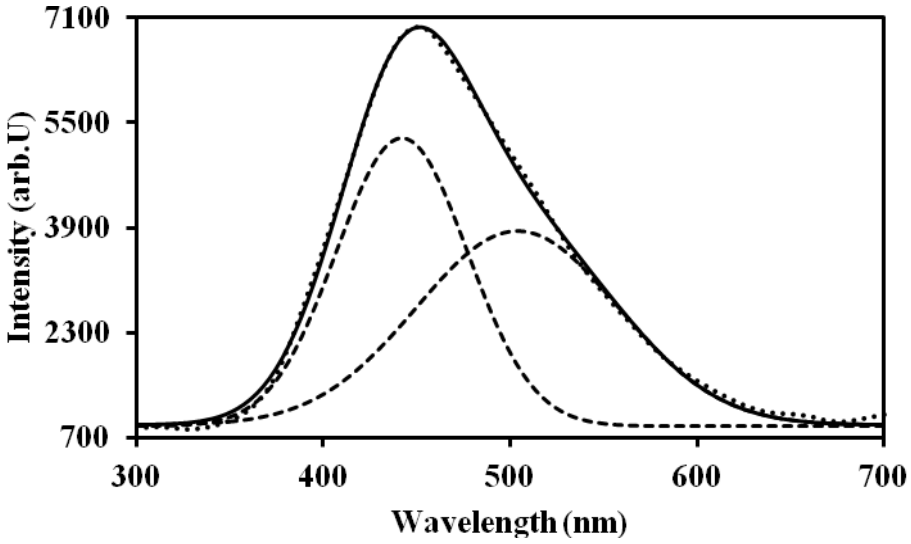

Fig.6 TL Emission Spectrum of KBr:Tm^{3+} Crystals for glow peak at 475 K

The TL and PL emission peaks more or less equal wavelength 444nm and 451 nm respectively.So this confirms that emission is only due to Tm^{3+}ions and involves the recombinationTm^{3+} electrons. TL emission process is due to thermal release of Tm^{3+}electrons and their consequent recombination with hole centers. The comparison of PL and TL emission band are shown in table.1

Table.1 Comparison of wavelength in Optical absorption, Photoluminescence and Deconvoluted TL emissionof KBr:Tm^{3+} single crystals

Optical absorption		**Photoluminescence**		**Thermoluminescence**
Name	**Absorption Wavelength (nm)**	**Excitation bands Wavelength (nm)**	**Emission bands Wavelength (nm)**	**Deconvolutedemission band in nm. Temperature at 475K**
KBr : Tm^{3+}	357 ($^3H_6 \rightarrow {}^1D_2$) 463 ($^3H_6 \rightarrow {}^1G_4$) 688 ($^3H_6 \rightarrow {}^3F_2$) 774 ($^3H_6 \rightarrow {}^1D_2$) 793 ($^3H_6 \rightarrow {}^3F_3$)	351 444 618	451 ($^1G_4 \rightarrow {}^3H_6$)	442 503

7. SCANNING ELECTRON MICROSCOPY

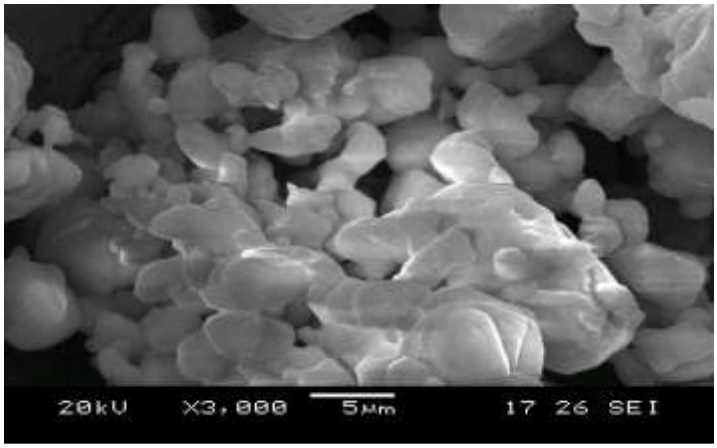

Fig.7 SEM image of KBr:Tm^{3+} Single crystals

The SEM micrograph of the Tm^{3+} doped KBr single crystals as shown in Fig.7. The grown crystals were subjected to x1000 magnification and accelerating voltage value of 20KV. The surface observed like a flower structure with agglomeration. The particle size is approximately few microns to 5µm for the grown crystals. The SEM micrograph of the grown crystals which find that the particles are irregular in shapes. So we can terminate the SEM results in good agreement with the result obtained from XRD results.

8. LASER RAMAN SPECTROSCOPY

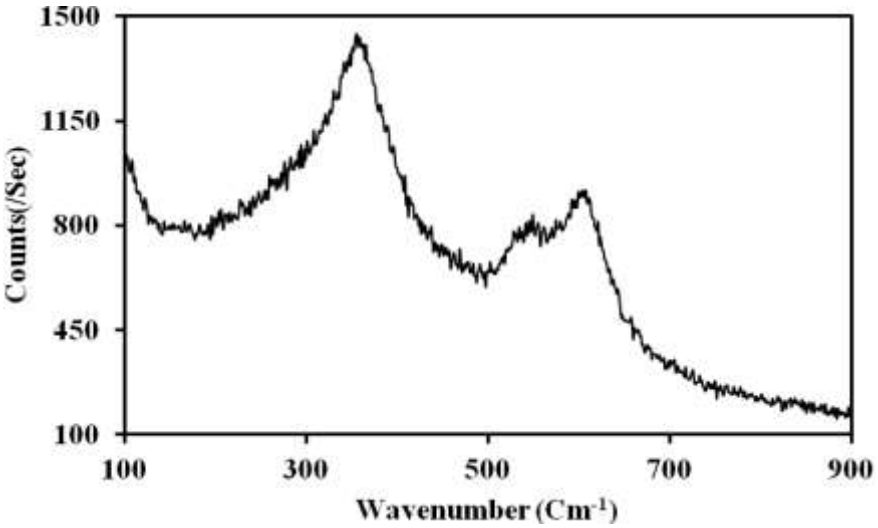

Fig.8 Laser Raman scattering spectrum of KBr:Tm^{3+} crystals

The Raman spectrum have using red laser excitation. The wavelength observed at room temperature as shown in Fig.8 for the Tm^{3+}doped KBr crystals. It composed of three main peaks of 355cm^{-1}, 549cm^{-1} and 607cm^{-1}. The earlier work reported as Raman modes have been observed frequencies around at 141cm^{-1} for Br_3^- (stretching vibration) and 216$cm^{-1}$for Br^-_2 type[32]. The excitation band wavelength observed around at 549cm^{-1} assigned to the first overtone of 355cm^{-1} mode. The lattice modes of the crystalline bromide observed at 549cm^{-1} and 607cm^{-1}superposed to the second order spectrum of the host. The second order scattering bands have been reported by other authors at 983cm^{-1}, 1030cm^{-+} and 1160cm^{-1}[33]. In the present work the band observed at 355cm^{-1} , 549cm^{-1} and 607cm^{-1} are assigned to second order scattering.

9. POWDER X-RAY DIFFRACTION SPECTRUM

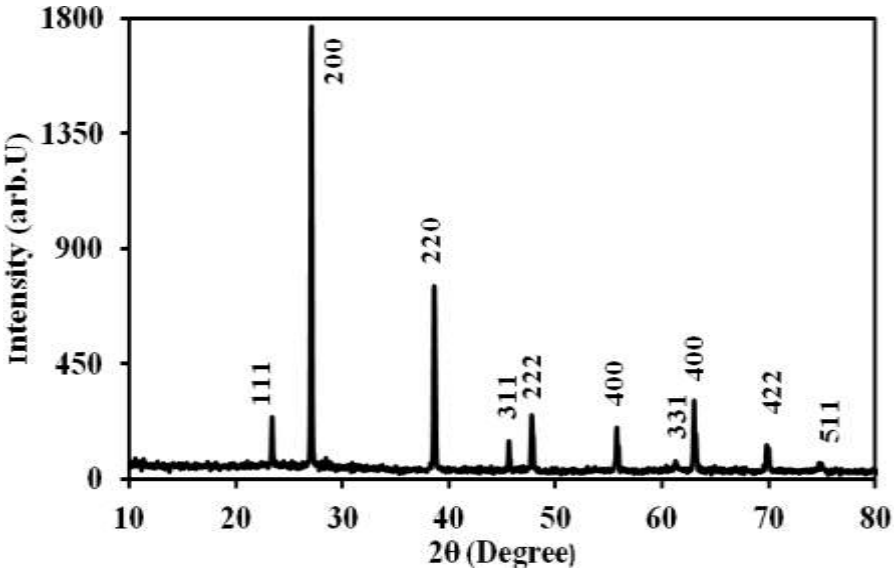

Fig.9 PXRD pattern of the KBr:Tm^{3+} Crystals

Fig.9 shows the powder x-ray diffraction pattern of the Tm^{3+} doped KBr single crystals used theCuKα radiation (1.5406Å). Hence the powder x-ray diffraction pattern of the grown crystals were used to calculate the crystalline structure and crystallite size. The grown crystals are good and well crystalline nature with the high intensity peak 2θ = 27º corresponding to the hkl diffraction plane [200]. This crystals have observed that the all diffractionspeaks compared with the standard data of pure KBr (JCPDS Card No:04-0531)[34]. It exhibits seven different peaks at2θvalues are 23.3°, 27°, 38.5°, 45.6°, 47.7°, 55.7° , 61.2°,62.9°, 69.9° and 75° corresponding to diffraction planes are (1,1,1), (2,0,0), (2,2,0), (3,1,1), (2,2,2), (4,0,0), (3,3,1), (4,0,0), (4,2,2) and (5,1,1) respectively. Thelattice parametersare a=b=7.95Å, c=8.84Åand α=β=γ=90°. It is observed crystalline structure in hexagonal system of pure KBr with the space group Fm3m.KBr can be observed indicating the dopant is well incorporated into the host lattice. So the grown sample obtained in a single phase and that doping with the small amount of Tm^{3+} will not induce any impurity phase.The average crystallite size of the grown crystal is 8.5µm. It was calculated byScherrer's formula D=kλ/βcosθ, Where k is the dimensionless shape factor indicate that the value of about 0.9, λ is the X-ray diffraction wavelength (λ=1.5406Å), θ is the diffraction angle of high intensity peak and β is the full width half maximum of high intensity peak[35].

CONCLUSION

The present paper KBr : Tm^{3+} is a good material for optical display devices. Optical absorption study reveals the transparency of the grown crystals. PL broad blueemission wavelength observed at 451nm in KBr:Tm^{3+} crystals, which is due to ($^1G_4 \rightarrow {}^3H_6$) transition of$Tm^{3+}$ ion. The results of a TL glow curve pattern of the grown crystals shows one prominent peak 422K. Furthermore, the kinetic parameters, i.e., activation energy (E), frequency factor(s) have been calculated.The TL emission peak confirms Tm^{3+} ion and it is involved in blue light emitting process. From the PXRD analysis, the calculated average crystallite size is 8.5µm. The laser raman scattering observed at 355cm^{-1}, 549cm^{-1} and 607$cm^{-1}$are assigned to second order scattering . SEM reveals that the microcrystalline morphology of the Tm^{3+} doped KBr single crystals is like a flower shape of particles and are various size from few microns to 5µm.

Acknowledgement

The authors great fully acknowledges UGC (RGNF) for providing financial support and UGC – DAE Consortium for scientific Research, IGCAR, Kalpakkam, India for providing experimental support.

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