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Structural Investigation and Scintillation Properties of $Cd_{1-x}Zn_xWO_4$ Solid Solution Single Crystals

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Abstract

The strategy for $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) solid solution growth was successfully achieved by the Czochralski method. Polycrystalline samples and crystals of the $Cd_{1-x}Zn_xWO_4$ system were studied using X-ray diffraction technique. Furthermore, the scintillation properties for $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) crystals were investigated. It was found that diffraction peaks of the polycrystalline $Cd_{1-x}Zn_xWO_4$ (x = 0-1.0) system shift toward higher 2θ angle with increasing level of Zn substitution. A linear relation between the zinc composition x and the lattice parameters for $Cd_{1-x}Zn_xWO_4$ crystals existed. With increasing zinc content x, the lattice parameters a, b, c decrease, and the volume of the unit cell decrease. The shift of lattice parameter presents a trend from $CdWO_4$ to $ZnWO_4$. Energy dispersive X-ray (EDAX) analysis was carried out in order to confirm the composition of $Cd_{1-x}Zn_xWO_4$ single crystals ($x < 0.5$). The optical measurements revealed that the solid solution crystals had a high optical transmittance about 65 % at the range of 350-600 nm, and the absorption edges of $Cd_{1-x}Zn_xWO_4$ crystals exhibit the increasing peaks from 317.4 nm to 321.5 nm respectively. The emission peaks showed a red shift from 465.4 nm to 501.2 nm with the increasing content of zinc. Furthermore, it was observed that the light output (L.O.) decrease and energy resolution (E.R.) increase with the increase of zinc content.

Introduction

Special attention has been paid to solid solution due to the possibility of fine control of structure and combination of advantageous properties from the pure components. As reported, single crystal of $Ba_{1-x}Sr_xB_2O_4$ (x = 0.006-0.13) solid solution successfully grown by Czochralski technique exhibits a high transmittance of 87% and high laser damage threshold of 1.2 GW/cm^2, which can be used as a novel ultraviolet birefringent crystal to replace the α-BBO crystal.[1] Therefore, some solid solution crystals have been synthesized and grown to investigate the change of structure and properties such as $A_xB_{1-x}WO_4$ (A, B = Ca, Sr, Ba, Mg, Mn, Zn, Ni, Pb),[2] $Cd_{1-x}Zn_xS$,[3] $Pb_{1-x}Sr_xTiO_3$,[4] $La_{1-x}Pr_xGaO_3$,[5] $Al_{2-x}In_x(WO_4)_3$,[6] $Pb(MoO_4)_x(WO_4)_{1-x}$.[7-9] However, it is still worthy of thorough research on the changes of crystal structure and properties with the chemical composition variation.

Tungstates scintillation crystals belong to self-activated phosphor and have been the subject of constant attention and investigation for their applications such as nuclear physics, medical application, and high energy physics.[10-13] $CdWO_4$ and $ZnWO_4$ crystallize in a wolframite-type structure with the monoclinic $P_{2/c}$ space group, which exhibit the advantages of high density, high luminous efficiency, powerful anti-irradiation properties and low cost.[14-17] Cell parameters and ion radius of $CdWO_4$ are near to those of $ZnWO_4$ and they can form a complete series of solid solution.[18,19] In addition, the typical macrodefects of $CdWO_4$ single crystal are pores and cracking due to deviation from stationarity and the presence of gas-forming impurities from the melt.[20] Another problem is gas-forming CdO from melt, which will cause

environmental pollution. For $ZnWO_4$ single crystal, high-quality inclusion-free $ZnWO_4$ crystals have been grown by a modified Czochralski technique.[21] Compared with $CdWO_4$, the limitation of $ZnWO_4$ crystal is relative low light output and coloration.[22] To the best of our knowledge, mixed $Cd_{1-x}Zn_xWO_4$ crystals can combine advantageous performance from the pure components. Therefore, the aim of this work is to grow single crystals of $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) solid solution through Czochralski technique to investigate the variation of structure and evolution of crystal performance. In the paper, single crystals of $Cd_{1-x}Zn_xWO_4$ solid solution with ($x < 0.5$) were grown by the Czochralski technique. Considering the actual scintillation properties, the $Cd_{1-x}Zn_xWO_4$ crystals with $x > 0.5$ are absent. The structural evolution, optical and scintillation properties for $Cd_{1-x}Zn_xWO_4$ crystals were investigated.

Experimental

Crystal growth. Polycrystalline materials of $Cd_{1-x}Zn_xWO_4$ with x = 0, 0.1, 0.3, 0.5, 0.7, 0.9 and 1.0 were synthesized through the conventional solid state reaction from the reagents of CdO (purity 99.95%)，ZnO (purity ≥ 99.9%) and WO_3 (purity ≥ 99.9%). First, the starting materials were dried at 200 □ for 4h and mixed in stoichiometry ratio. Then, the mixture was ground manually in a corundum mortar for 3h. At last, the mixture was calcined at 1000 □ for 10h and slowly cooled to room temperature in furnace, and then white powder was obtained.

Single crystals of $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) were grown from melts in a 2.5 kHz frequency furnace in N_2 atmosphere using the traditional Czochralski method. The synthesized raw materials were placed in an Ir crucible of Φ 60 × 39 mm^3. The Ir

crucible and an Ir-Ir40%Rh thermocouple was placed in an insulated Al_2O_3 ceramics. To reduce the axial temperature gradient and prevent cracking of the crystals, an insulated enclosure was placed above the crucible. All the single crystals were grown by the seeds of pure $CdWO_4$ single crystal which was cut along the [010] direction. The temperature was raised until the polycrystalline materials melt at a rate of 200 ℃ /h and all the melting condition and growth of crystals were seen by the observation window. The pulling rate of crystal was 1.0-2.0 mm/h and rotation speed was 15-20 r/min. After growth, the crystal was cooled down to room temperature at the 30 □/h. Single crystals of $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) were obtained, as shown in Figure 1.

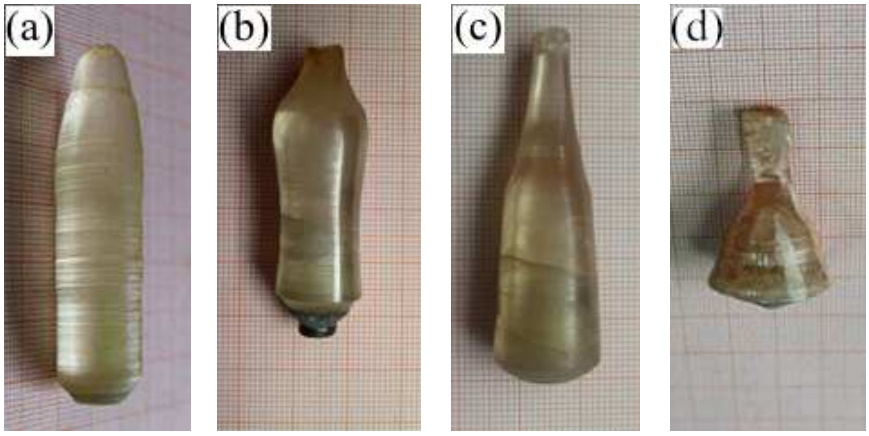

Figure 1. Single crystals of $Cd_{1-x}Zn_xWO_4$: (a) $CdWO_4$; (b) $Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$.

Measurements. The $Cd_{1-x}Zn_xWO_4$ single crystals were grown from nominal melt compositions, which are obviously different from the obtained crystals compositions due to gas-forming CdO from melt. Therefore, $Cd_xZn_{1-x}WO_4$ ($0 < x <$

0.5) wafers without macroscopic defects were selected for energy dispersive X-ray (EDAX) analysis which were performed at HITACHI SU-70 equipment using an accelerating voltage of 15kV. The phase identification and structural characterization were conducted using powder X-ray diffraction (XRD, Bruker D8 Focus diffraction with nickel-filtered Cu Kα radiation). Rough Rietveld refinements of the diffraction data of $Cd_{1-x}Zn_xWO_4$ crystals were performed using Jana 2006 program.[23] Refined lattice parameters of $Cd_xZn_{1-x}WO_4$ crystals at room temperature and Zinc content of $Cd_xZn_{1-x}WO_4$ ($0 < x < 0.5$) wafers were listed in Table 1.

For scintillation measurements, the substrates of $Cd_{1-x}Zn_xWO_4$ single crystals ($x < 0.5$) with dimensions $10\times8\times0.8$ mm^3 were prepared by accurate cleaving along the crystallographic axes. Transmittance, emission peak, energy resolution and light output are important parameters for the crystal scintillation properties. Excellent scintillators have the advantages of high transmittance, certain emission peak in the UV-VIS region, high energy resolution and high light output. The transmission spectra of solid solution single crystals were measured by TU-1901 spectrophotometer over the wavelength range of 200-600 nm. The emission spectra of solid solution single crystals were analyzed by HITACHI F-4500 spectrophotometer using a Xenon lamp as the exciting source. All the emission spectra were measured with an excitation wavelength of 316 nm. Furthermore, all solid solution crystals were polished and processed into rectangular shape with the same thickness of 2 mm. Light output (L.O.) measurements were carried out using a HAMAMATSU R2059 PMT with bialkali cathode. The $Cd_{1-x}Zn_xWO_4$ crystals were coupled to PMT on one end

with Dow Corning 200 fluid, while all other faces were wrapped with two layers of Tyvek paper to increase the collected light. A collimated γ-ray source was used to excite the sample. The γ-ray peak was obtained by simple Gaussian fit, and was used to determine light output by calibration of single photoelectron peak. All the samples were prepared from the top position of $Cd_{1-x}Zn_xWO_4$ crystals in which high quality crystals could be obtained.

Results and discussions

As shown in Figure 1, $Cd_{1-x}Zn_xWO_4$ ($x < 0.2$) crystals were obtained with slight inclusions, cracks and coloration on the bottom. As to slight cracks of $Cd_{1-x}Zn_xWO_4$ crystals, control of growth conditions is responsible. Obvious cracks of $Cd_{1-x}Zn_xWO_4$ ($x = 0.416$) crystal on the bottom may be ascribed to enough mixture of zinc and increasing diameter. The $Cd_{1-x}Zn_xWO_4$ crystals have different shapes and lengths due to different control of diameter and growth time. In particular, $Cd_{1-x}Zn_xWO_4$ ($x = 0.416$) crystal is of a brown color which is the same as $ZnWO_4$. The impurities and defects of oxygen vacancies were regarded as the main reason for the coloration.[22]

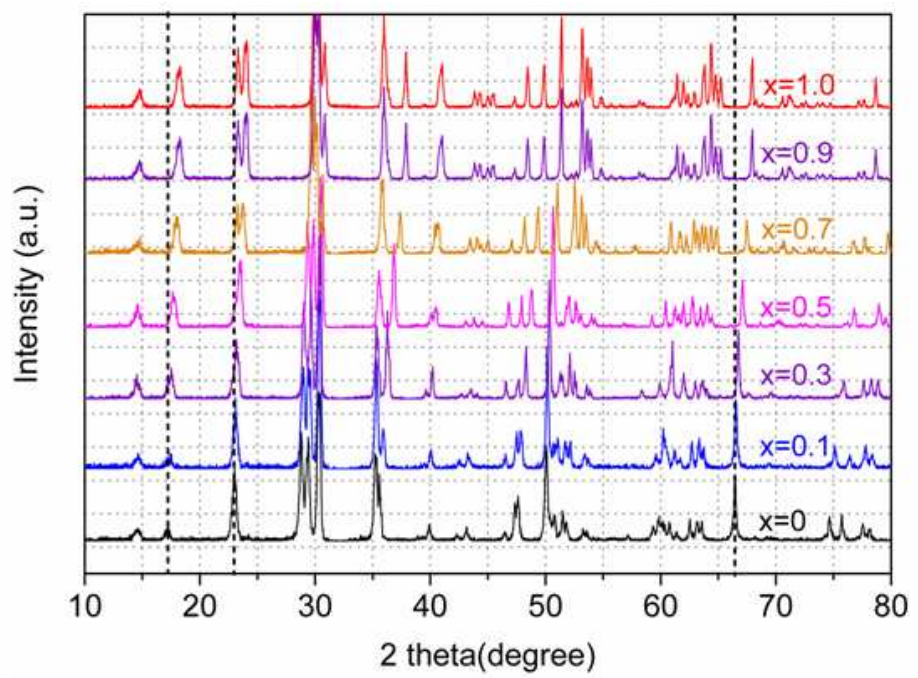

Figure 2. X-ray diffraction patterns of polycrystalline $Cd_{1-x}Zn_xWO_4$ (x = 0, 0.1, 0.3, 0.5, 0.7, 0.9, 1.0) at room temperature.

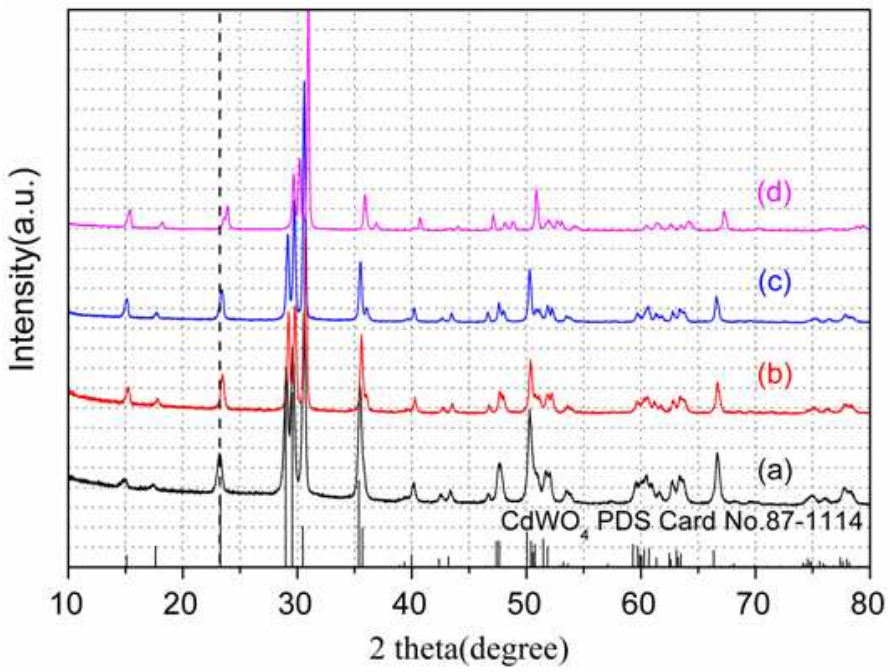

Figure 3. X-ray diffraction patterns of $Cd_{1-x}Zn_xWO_4$ crystals: (a) $CdWO_4$; (b)

$Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$.

Morell and Dahlborg investigated the variation of cell parameters of $Cd_xZn_{1-x}WO_4$ crystals as a function of composition, which showed that $ZnWO_4$ and $CdWO_4$ could form a complete series of solid solution.[18,19] Polycrystalline $Cd_xZn_{1-x}WO_4$ samples are prepared and measured. Powder X-ray diffraction patterns of polycrystalline $Cd_{1-x}Zn_xWO_4$ specimen are shown in Figure 2. As shown in Figure 2, all the diffraction peaks of polycrystalline $Cd_{1-x}Zn_xWO_4$ with x = 0 and x = 1 are in good agreement with pure $CdWO_4$ and $ZnWO_4$ respectively. No other impurity peaks are detected indicating that the samples are single phase $CdWO_4$ and $ZnWO_4$. Meanwhile, sharp and intense peaks demonstrate that well-crystallized $CdWO_4$ and $ZnWO_4$ sample are synthesized after high temperature treatment. Consequently, we expect that homogeneous polycrystalline raw materials of $Cd_{1-x}Zn_xWO_4$ are obtained. In addition, it is obvious that the corresponding peaks of X-ray diffraction patterns of solid solution shift toward higher 2θ angle with increasing level of Zn substitution. Shifts of atomic displacement within the unit cell are responsible for the change of diffraction patterns. X-ray diffraction patterns of $Cd_{1-x}Zn_xWO_4$ crystals with different compositions are shown in Figure 3. As shown in Figure 3, X-ray diffraction data of $CdWO_4$ single crystal agree well with the pure monoclinic phase structure of $CdWO_4$ (JCPDS file No. 87-1114). Obviously, diffraction peaks of solid solution shift toward higher 2θ angles with increasing content of zinc. This trend is similar with the shift of polycrystalline $Cd_{1-x}Zn_xWO_4$ (Figure 2).

Table 1. Refined lattice parameters and zinc content for $Cd_{1-x}Zn_xWO_4$ crystals.

Cd/Zn in the initial solution (at. ratio)	a/Å	b/Å	c/Å	β/°	V/Å^3	Cd/Zn in the crystal (EDAX analysis)
1/0	5.0160	5.8391	5.0662	91.512	148.3	
0.9/0.1	4.9976	5.8389	5.0557	91.490	147.5	0.943/0.057
0.8/0.2	4.9728	5.8314	5.0417	91.493	146.2	0.887/0.113
0.5/0.5	4.8792	5.7847	4.9953	91.367	141.0	0.584/0.416

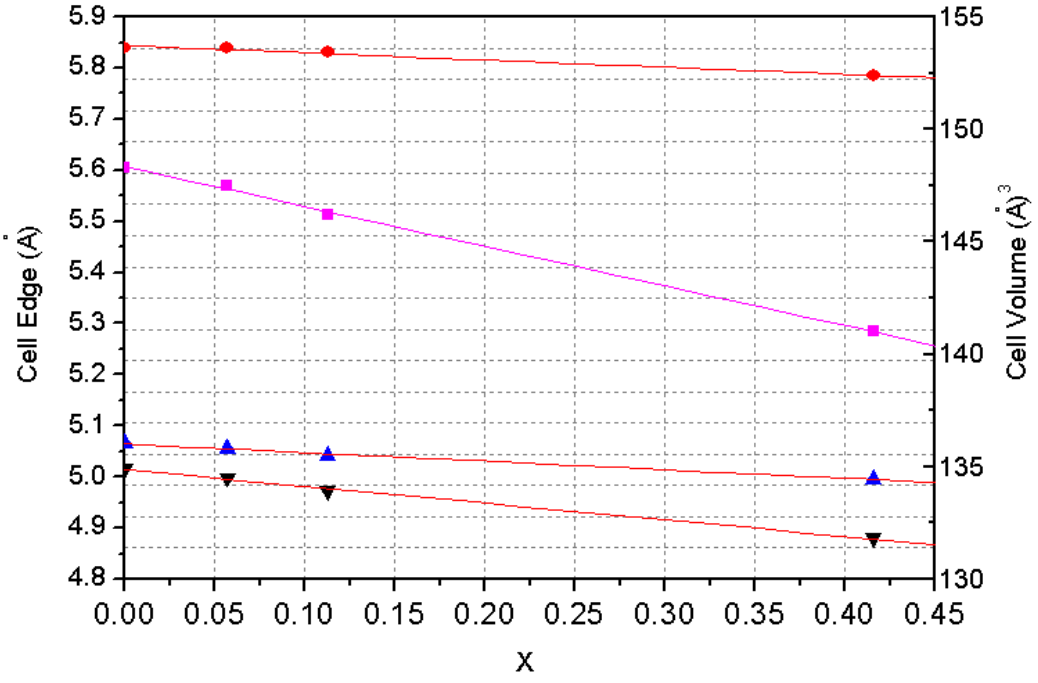

Figure 4. Refined lattice parameters of $Cd_{1-x}Zn_xWO_4$ crystals: (a) $CdWO_4$; (b) $Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$, ●, b; ▲, c; ▼, a; ■, cell volume.

Rough Rietveld refinements of the diffraction data of $Cd_{1-x}Zn_xWO_4$ crystals were performed to show structural evolution of cell parameters with the increasing level of Zn substitution of Cd. In addition, the measurements show that Zn/Cd ratio deviated

substantially from that of the initial melt (Table 1). Therefore, the composition of $Cd_{1-x}Zn_xWO_4$ single crystals was confirmed. Figure 4 shows the variation of lattice parameters of $Cd_xZn_{1-x}WO_4$ crystals. Refined lattice parameters of $CdWO_4$ are a = 5.0160 Å, b = 5.8391 Å, c = 5.0662 Å and β = 91.512°, which are close to the standard data (a = 5.029 Å, b = 5.860 Å, c = 5.071 Å and β = 91.51°). It is evident that lattice parameters a, b, c change linearly with a linear correlation coefficient of 0.999, 0.990 and 0.997, respectively. Meanwhile, the volume of unit cell has a linear decrease with the increase of zinc content and its linear correlation coefficient is 0.999. The evolution of lattice parameters is mainly ascribed to the decreasing angle of W-W-W and decreasing distance of W-O in the WO_6 octahedra when going from $CdWO_4$ to $ZnWO_4$.[19] This is in accord with the decreasing ionic radius of six-coordinated Cd^{2+} and Zn^{2+}, 0.95 and 0.74 Å.

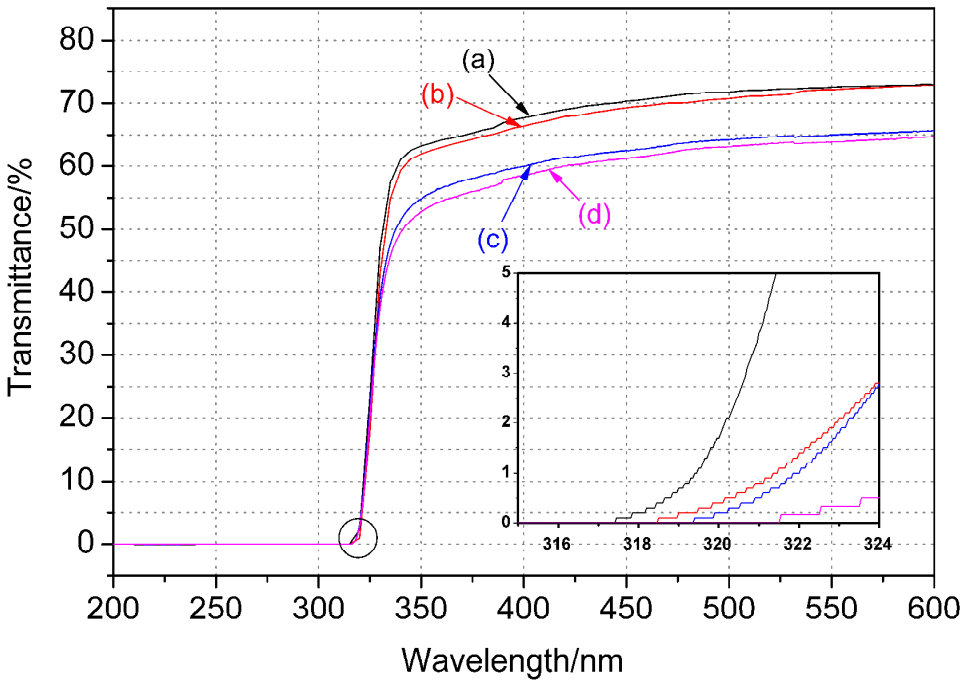

Figure 5. Transmission spectra of $Cd_{1-x}Zn_xWO_4$ crystals: (a) $CdWO_4$; (b) $Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$.

Figure 5 exhibits the transmission spectra of $Cd_{1-x}Zn_xWO_4$ crystals with $x < 0.5$. It can be seen that all the substrates of solid solution crystals have a high optical transmittance about 65% at the range of 350-600 nm. Obviously, the transmittance level in the visible region decrease with the increasing content of zinc. In addition, all the transmittance curves have a smooth platform and no any absorption peaks. The absorption edges of $Cd_{1-x}Zn_xWO_4$ crystals were exactly measured. As shown in Figure 5, the absorption edges of $Cd_{1-x}Zn_xWO_4$ crystals exhibit the increasing peaks at 317.4, 318.5, 319.4 and 321.5 nm respectively. It is obvious that the shift of the transmittance level and absorption edges of $Cd_{1-x}Zn_xWO_4$ crystals may probably be attributed to structural change of WO_6 octahedra in the crystals with the increasing level of Zn substitution of Cd. The W-W-W angle and distance of W-O decrease in the WO_6 octahedra when going from $CdWO_4$ to $ZnWO_4$.

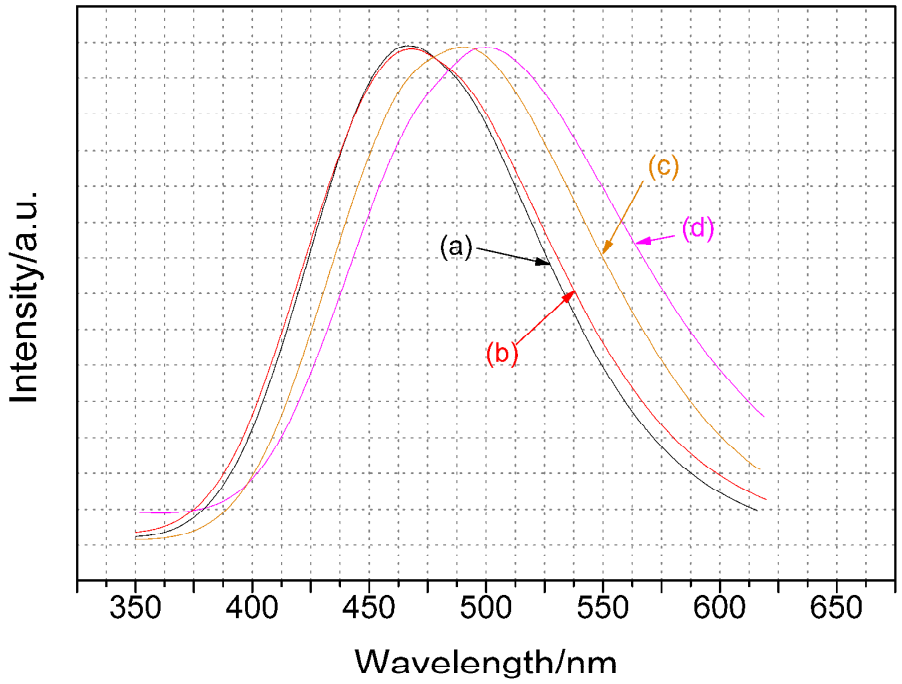

Figure 6. Luminescence (λ_{ex} = 316 nm) spectra of $Cd_{1-x}Zn_xWO_4$ crystals: (a) $CdWO_4$; (b) $Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$.

The emission spectra of $Cd_{1-x}Zn_xWO_4$ crystals with $x < 0.5$ are shown in Figure 6. It can be seen that $Cd_{1-x}Zn_xWO_4$ crystals exhibit a wide emission peak at 465.4, 467.4, 490.8 and 501.2 nm respectively. It is obvious that the emission peak shows a red shift with the increasing content of zinc. Fluorescence of tungstates derives from the transfer of electrons from 2p orbital of O to the empty 5d orbital of W in group $[W\text{-}O_4]^{2-}$.[24] Zn^{2+} has a stronger polarization effect on luminescent group $[W\text{-}O_4]^{2-}$ than Cd^{2+}. Therefore, the red shift may be attributed to the reduction of transfer energy from O to W with increasing level of Zn substitution of Cd.

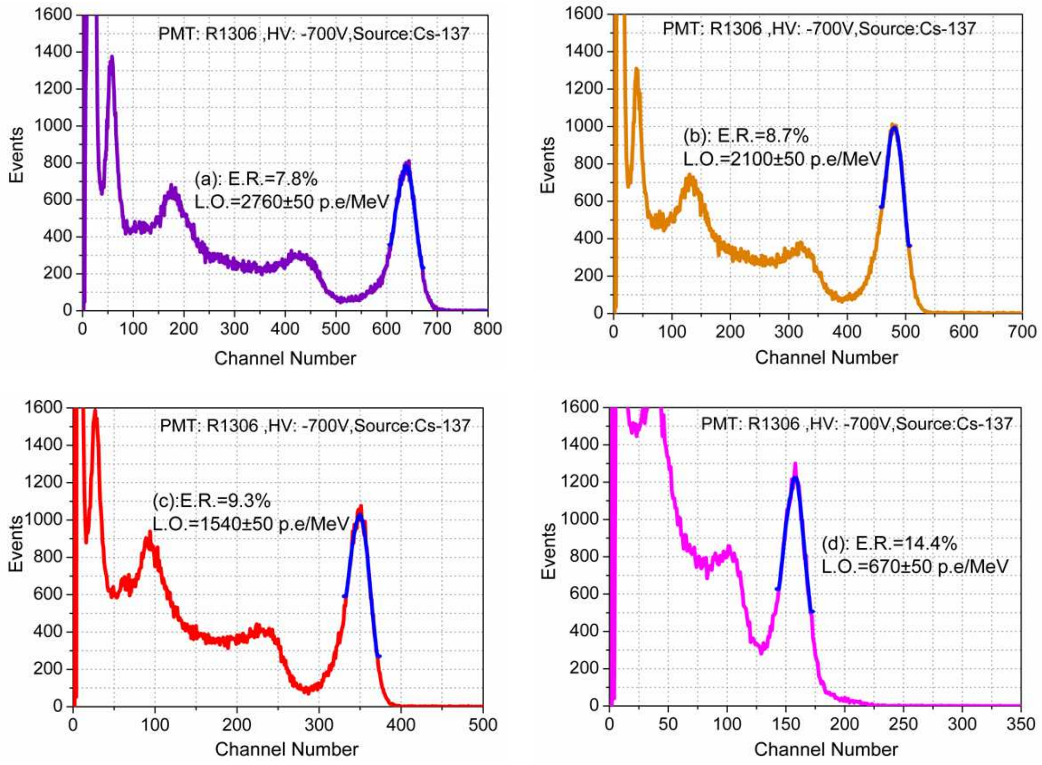

Figure 7. Light output and the FWHM energy resolution of $Cd_{1-x}Zn_xWO_4$ crystal: (a) $CdWO_4$; (b) $Cd_{0.943}Zn_{0.057}WO_4$; (c) $Cd_{0.887}Zn_{0.113}WO_4$; (d) $Cd_{0.584}Zn_{0.416}WO_4$ measured by a collimated ^{137}Cs source.

Measurement results of light output and energy resolution of $Cd_{1-x}Zn_xWO_4$ crystal ($x<0.5$) are presented in Figure 7(a)-(d). The light output of $CdWO_4$ is 2760±50 p.e./MeV. The absolute light yield for $CdWO_4$ is determined to be 20750±375 photons/MeV according to the emission weighted quantum efficiency[25, 26] of the detector which is 14 % and the light collection efficiency which is 95 %.[27] As shown from data, $Cd_{0.943}Zn_{0.057}WO_4$ crystal shows a relative low light output

(2100±50 p.e./MeV) as compared with $CdWO_4$ (2760±50 p.e./MeV). Obviously, the measurement value of light yield decreases with the increase of zinc content. The results of overall energy resolution measurements for $Cd_{1-x}Zn_xWO_4$ crystals with different zinc content are also presented in Figure 7. The energy resolution of $CdWO_4$ crystal is 7.8 %, which agrees with the observation of other research.[28] However, the measurement value of energy resolution increases with the increase of zinc content, with a linearity of 0.9988. In view of the same testing conditions, zinc content in $Cd_{1-x}Zn_xWO_4$ crystals is responsible for the variation trend of light output and energy resolution. It evidences that zinc content results in difference of the total number of photons emitted by $Cd_{1-x}Zn_xWO_4$ crystals. $Cd_{1-x}Zn_xWO_4$ ($x < 0.1$) crystals exhibit excellent scintillation properties after performance comparison. It is obvious that the scintillation properties of $Cd_{1-x}Zn_xWO_4$ ($0.1 < x < 0.5$) crystals are weaker with the increase of zinc content, especially the parameter of light output. In addition, in experiments of $Cd_{1-x}Zn_xWO_4$ crystals growth gas-forming CdO from melt decreases gradually with the increase of zinc content. Meanwhile, the melting and growth temperature decreases and the temperature can be estimated in the range from 1200 □ to 1257 □.[17] Therefore, Zn doping in small quantity is effective to improve crystal growth and satisfy excellent scintillation properties.

Conclusions

In summary, $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) solid solution crystals were successfully grown by the Czochralski method for the first time. X-ray diffraction measurement showed that the corresponding diffraction peaks of the polycrystalline $Cd_{1-x}Zn_xWO_4$

(x = 0-1.0) system shift toward higher 2θ angles with increasing level of Zn substitution. It's because a linear relation between the zinc composition x and the lattice parameters existed for $Cd_{1-x}Zn_xWO_4$ crystals. With increasing zinc content x, the lattice parameters a, b and c decrease and the volume of the unit cell decrease. The decreasing angle of W-W-W and decreasing distance of W-O in the WO_6 octahedra is responsible for the evolution of lattice parameters when going from $CdWO_4$ to $ZnWO_4$. The crystals have a different composition than the melt due to gas-forming CdO from melt and segregation coefficient between solid phase and liquid phase. Therefore, the composition of $Cd_{1-x}Zn_xWO_4$ single crystals ($x < 0.5$) was confirmed by energy dispersive X-ray analysis (EDAX). The optical measurements revealed that the solid solution crystals had a high optical transmittance about 65 % at the range of 350-600 nm, and the emission peaks showed a red shift from 465.4 nm to 501.2 nm with the increasing content of zinc. Furthermore, the light output changed from 2760±50 p.e./MeV to 670±50 p.e./MeV and energy resolution changed from 7.8 % to 14.4 %. The measurement value of light yield decreases corresponding to the increasing zinc content. Similarly, the value of energy resolution increases corresponding to the increase of zinc content with a linearity of 0.9988. The evolution of light output and energy resolution would be ascribed to a partial substitution of Cd by Zn. In view of crystal performance comparison and growth, Zn doping in small quantity is effective to improve crystal growth and satisfy excellent scintillation properties.

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Notes

The authors declare no competing financial interest.

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Table of contents entry

Crystals of $Cd_{1-x}Zn_xWO_4$ ($x < 0.5$) were grown by Czochralski technique. The structural investigation, scintillation properties of $Cd_{1-x}Zn_xWO_4$ samples were investigated.