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Structural Investigation and Scintillation Properties of Cd$_{1-x}$Zn$_x$WO$_4$

Solid Solution Single Crystals

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Abstract

The strategy for Cd$_{1-x}$Zn$_x$WO$_4$ ($x < 0.5$) solid solution growth was successfully achieved by the Czochralski method. Polycrystalline samples and crystals of the Cd$_{1-x}$Zn$_x$WO$_4$ system were studied using X-ray diffraction technique. Furthermore, the scintillation properties for Cd$_{1-x}$Zn$_x$WO$_4$ ($x < 0.5$) crystals were investigated. It was found that diffraction peaks of the polycrystalline Cd$_{1-x}$Zn$_x$WO$_4$ ($x = 0.21.0$) 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$_x$WO$_4$ 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$_x$WO$_4$ 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$_x$WO$_4$ 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 $\text{Ba}_{1-x}\text{Sr}_x\text{B}_2\text{O}_4$ ($x = 0.006$-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 $\alpha$-BBO crystal. Therefore, some solid solution crystals have been synthesized and grown to investigate the change of structure and properties such as $\text{A}_x\text{B}_{1-x}\text{WO}_4$ ($\text{A, B} = \text{Ca, Sr, Ba, Mg, Mn, Zn, Ni, Pb}$), $\text{Cd}_{1-x}\text{Zn}_x\text{S}$, $\text{Pb}_{1-x}\text{Sr}_x\text{TiO}_3$, $\text{La}_{1-x}\text{Pr}_x\text{GaO}_3$, $\text{Al}_{2-x}\text{In}_x(\text{WO}_4)_3$, $\text{Pb}(\text{MoO}_4)_3(\text{WO}_4)_{1-x}$. 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. $\text{CdWO}_4$ and $\text{ZnWO}_4$ 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. Cell parameters and ion radius of $\text{CdWO}_4$ are near to those of $\text{ZnWO}_4$ and they can form a complete series of solid solution. In addition, the typical macrodefects of $\text{CdWO}_4$ single crystal are pores and cracking due to deviation from stationarity and the presence of gas-forming impurities from the melt. 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$_x$WO$_4$ 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$_x$WO$_4$ (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$_x$WO$_4$ solid solution with (x < 0.5) were grown by the Czochralski technique. Considering the actual scintillation properties, the Cd$_{1-x}$Zn$_x$WO$_4$ crystals with x > 0.5 are absent. The structural evolution, optical and scintillation properties for Cd$_{1-x}$Zn$_x$WO$_4$ crystals were investigated.

**Experimental**

**Crystal growth.** Polycrystalline materials of Cd$_{1-x}$Zn$_x$WO$_4$ 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 $\geq$ 99.9%) and WO$_3$ (purity $\geq$ 99.9%). First, the starting materials were dried at 200 °C 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 °C for 10h and slowly cooled to room temperature in furnace, and then white powder was obtained.

Single crystals of Cd$_{1-x}$Zn$_x$WO$_4$ (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 $\Phi$ 60 $\times$ 39 mm$^3$. The Ir
crucible and an Ir-Ir40%Rh thermocouple was placed in an insulated Al₂O₃ 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 CdWO₄ single crystal which was cut along the [010] direction. The temperature was raised until the polycrystalline materials melt at a rate of 200 °C/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 °C/h. Single crystals of Cd₁ₓZnₓWO₄ (x < 0.5) were obtained, as shown in Figure 1.

Figure 1. Single crystals of Cd₁ₓZnₓWO₄: (a) CdWO₄; (b) Cd₀.943Zn₀.057WO₄; (c) Cd₀.887Zn₀.113WO₄; (d) Cd₀.584Zn₀.416WO₄.

Measurements. The Cd₁ₓZnₓWO₄ 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, CdₓZn₁₋ₓWO₄ (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$_x$WO$_4$ crystals were performed using Jana 2006 program.$^{23}$ Refined lattice parameters of Cd$_x$Zn$_{1-x}$WO$_4$ crystals at room temperature and Zinc content of Cd$_x$Zn$_{1-x}$WO$_4$ (0 < x < 0.5) wafers were listed in Table 1.

For scintillation measurements, the substrates of Cd$_{1-x}$Zn$_x$WO$_4$ single crystals (x < 0.5) with dimensions 10×8×0.8 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$_x$WO$_4$ 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 $\gamma$-ray source was used to excite the sample. The $\gamma$-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$_x$WO$_4$ crystals in which high quality crystals could be obtained.

**Results and discussions**

As shown in Figure 1, Cd$_{1-x}$Zn$_x$WO$_4$ ($x < 0.2$) crystals were obtained with slight inclusions, cracks and coloration on the bottom. As to slight cracks of Cd$_{1-x}$Zn$_x$WO$_4$ crystals, control of growth conditions is responsible. Obvious cracks of Cd$_{1-x}$Zn$_x$WO$_4$ ($x = 0.416$) crystal on the bottom may be ascribed to enough mixture of zinc and increasing diameter. The Cd$_{1-x}$Zn$_x$WO$_4$ crystals have different shapes and lengths due to different control of diameter and growth time. In particular, Cd$_{1-x}$Zn$_x$WO$_4$ ($x = 0.416$) 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$_x$WO$_4$ ($x = 0, 0.1, 0.3, 0.5, 0.7, 0.9, 1.0$) at room temperature.

Figure 3. X-ray diffraction patterns of Cd$_{1-x}$Zn$_x$WO$_4$ 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$_x$Zn$_{1-x}$WO$_4$ crystals as a function of composition, which showed that ZnWO$_4$ and CdWO$_4$ could form a complete series of solid solution.\textsuperscript{18,19} Polycrystalline Cd$_x$Zn$_{1-x}$WO$_4$ samples are prepared and measured. Powder X-ray diffraction patterns of polycrystalline Cd$_{1-x}$Zn$_x$WO$_4$ specimen are shown in Figure 2. As shown in Figure 2, all the diffraction peaks of polycrystalline Cd$_{1-x}$Zn$_x$WO$_4$ 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$_x$WO$_4$ 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$_x$WO$_4$ 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$_x$WO$_4$ (Figure 2).
Table 1. Refined lattice parameters and zinc content for Cd$_{1-x}$Zn$_x$WO$_4$ crystals.

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

Figure 4. Refined lattice parameters of Cd$_{1-x}$Zn$_x$WO$_4$ 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$_x$WO$_4$ 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$_x$WO$_4$ single crystals was confirmed. Figure 4 shows the variation of lattice parameters of Cd$_x$Zn$_{1-x}$WO$_4$ crystals. Refined lattice parameters of CdWO$_4$ are $a = 5.0160$ Å, $b = 5.8391$ Å, $c = 5.0662$ Å and $\beta = 91.512^\circ$, which are close to the standard data ($a = 5.029$ Å, $b = 5.860$ Å, $c = 5.071$ Å and $\beta = 91.51^\circ$). 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$_4$. 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_{x}WO_{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_{x}WO_{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_{x}WO_{4} crystals were exactly measured. As shown in Figure 5, the absorption edges of Cd_{1-x}Zn_{x}WO_{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_{x}WO_{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 ($\lambda_{ex} = 316$ nm) spectra of Cd$_{1-x}$Zn$_x$WO$_4$ crystals: (a) CdWO$_4$; (b) Cd$_{0.945}$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$_x$WO$_4$ crystals with $x < 0.5$ are shown in Figure 6. It can be seen that Cd$_{1-x}$Zn$_x$WO$_4$ 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 $[\text{W-O}_4]^{2-}$. $^{24}$ Zn$^{2+}$ has a stronger polarization effect on luminescent group $[\text{W-O}_4]^{2-}$ 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$_x$WO$_4$ 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$_x$WO$_4$ 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 CdWO₄ (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 Cd₁₋ₓZnₓWO₄ crystals with different zinc content are also presented in Figure 7. The energy resolution of CdWO₄ crystal is 7.8 %, which agrees with the observation of other research. 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 Cd₁₋ₓZnₓWO₄ 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 Cd₁₋ₓZnₓWO₄ crystals. Cd₁₋ₓZnₓWO₄ (x < 0.1) crystals exhibit excellent scintillation properties after performance comparison. It is obvious that the scintillation properties of Cd₁₋ₓZnₓWO₄ (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 Cd₁₋ₓZnₓWO₄ 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 °C to 1257 °C. Therefore, Zn doping in small quantity is effective to improve crystal growth and satisfy excellent scintillation properties.

Conclusions

In summary, Cd₁₋ₓZnₓWO₄ (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 Cd₁₋ₓZnₓWO₄
(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$_x$WO$_4$ 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$_x$WO$_4$ 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.

Acknowledgments

This work is partially supported by the National Natural Science Foundation of China (No. 21171102), the Expert Project of Key Basic Research of the Ministry of Science and Technology of China (No. 2011CB612306), the Natural Science Foundation of Ningbo City (No. 2013A610141), the Climb Project of Young Leaders (pd2013089), Foundation of Ningbo University (zj1120), and the K. C. Wong MagnaFund in Ningbo University.

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Crystals of Cd$_{1-x}$Zn$_x$WO$_4$ (x < 0.5) were grown by Czochralski technique. The structural investigation, scintillation properties of Cd$_{1-x}$Zn$_x$WO$_4$ samples were investigated.