

Development of Phosphorus-compound $\text{CaSO}_4\text{:Dy(KCT-300)}$ TL Pellets

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Abstract

$\text{CaSO}_4\text{:Dy}$ thermoluminescence dosimeter (TLD) is widely used as a personal or environmental dosimeter because of its high sensitivity to radiation. There are many methods to make pellets from the TL phosphors[1-5]. Sintered pellets were made from a mixture of $\text{CaSO}_4\text{:Dy}$ phosphor and Teflon powder is the most common method. But this method has disadvantage that $\text{CaSO}_4\text{:Dy}$ pellet does not have very high sensitivity because of large amounts of Teflon in pellets. This paper described development of a new type of $\text{CaSO}_4\text{:Dy}$ pellets by using P-compounds as a bonding material (KCT-300), and compared the TL sensitivity with that of the commercialized Teledyne $\text{CaSO}_4\text{:Dy}$ pellets. Sensitivity of a new developed KCT-300 shows about 6 times than Teledyne ones, and can be used to measure very low radiation dose.

Key Words : $\text{CaSO}_4\text{:Dy}$ TL material sensitivity, phosphorous, binding material.

1. Introduction

TL sensitivity of $\text{CaSO}_4\text{:Dy}$ TL phosphors is very high. However the problem exists of preparing this material in the form of sintered pellets in which TL sensitivity of the original phosphor would stay unchanged. It is not possible to make solid detectors of only $\text{CaSO}_4\text{:Dy}$ TL phosphor without any binding material. Until now widely used sintered pellets, which were made from a mixture of $\text{CaSO}_4\text{:Dy}$ TL phosphor and Teflon. But, due to

reduction in content of $\text{CaSO}_4\text{:Dy}$ TL phosphor in pellets(15~20wt%) the TL sensitivity of pellets was reduced correspondingly. To variously use the $\text{CaSO}_4\text{:Dy}$ TL phosphor, research for preparing detectors by mixing $\text{CaSO}_4\text{:Dy}$ TL phosphor with different binding substances has been conducted[2-4]. However, TL intensity of all detectors is not improved. Presently, M.Prokic has developed a $\text{CaSO}_4\text{:Dy}$ TL pellets using a multi-component inorganic binding materials in small amounts, and improved it's TL sensitivity[5]. But, the information

about the binding materials was not disclosed. In this paper, presented that manufacturing process of Phosphorous-compounds $\text{CaSO}_4:\text{Dy}$ pellets as new binding material and relative TL sensitivity.

2. Materials and Experimental Procedures

2.1. Preparation of $\text{CaSO}_4:\text{Dy}$ TL Phosphor

$\text{CaSO}_4:\text{Dy}$ (Dy 0.1mol%) phosphor was prepared by following the method of Yamashita et al[6]. The $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and H_2SO_4 (two reagent are extra pure) are produced by Oriental Chemical Industries. Dy_2O_3 (0.0373g) as an activator was dissolved in a dilute aq. solution of sulfuric acid (10 ml) and poured into a concentrated sulfuric acid containing flask, followed by adding with $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (23.615g). The flask was heated at 320°C , and thus concentrated sulfuric acid was distilled, whereby crystalline $\text{CaSO}_4:\text{Dy}$ was formed on inner walls of the flask. The crystals separated from the walls were washed several times with distilled water. This crystals were dried at about 200°C . Such powders were sintered at 700°C for 1h, and then sieved. Following the above process, we obtained the crystal powder with suitable grain size ($63\text{--}100\mu\text{m}$). Fig. 1 is showed the manufacturing process of $\text{CaSO}_4:\text{Dy}$ TL phosphors. And Fig. 2 is showed comparison of

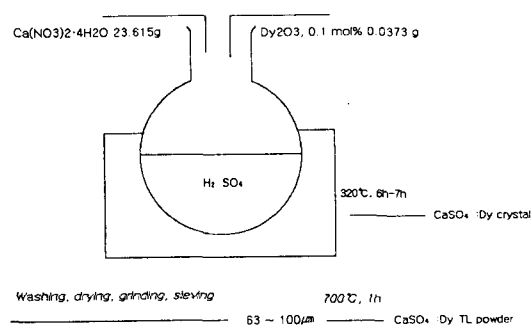


Fig. 1. Manufacturing Process of $\text{CaSO}_4:\text{Dy}$ TL Phosphors

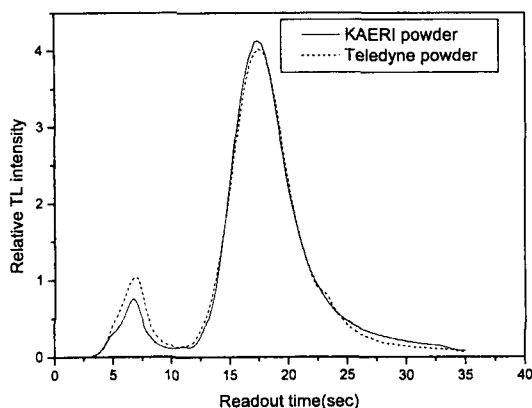


Fig. 2. Comparison of Glow Curve of $\text{CaSO}_4:\text{Dy}$ Phosphors Manufactured by KAERI and Teledyne

glow curve of $\text{CaSO}_4:\text{Dy}$ phosphors manufactured by KAERI and Teledyne. As can be seen in Fig. 2, sensitivity of KAERI $\text{CaSO}_4:\text{Dy}$ phosphors is equal or better to that of Teledyne $\text{CaSO}_4:\text{Dy}$ phosphors.

2.2. Preparation of $\text{CaSO}_4:\text{Dy}$ TL Pellets

2.2.1. Determination of p-compounds Contents

Before manufacturing of embedded P-compounds $\text{CaSO}_4:\text{Dy}$ pellets, must be determined optimum P-compounds contents, as considering mechanical strength and TL sensitivity. Sintering pellets were obtained a homogeneous mixture of P-compounds from 5~30mol% and $\text{CaSO}_4:\text{Dy}$ TL phosphor. And measured its TL sensitivity; the results were showed at Fig.3. The sharp of glow curve of embedded P-compounds $\text{CaSO}_4:\text{Dy}$ pellets is very similarly to sharp of glow curve of $\text{CaSO}_4:\text{Dy}$ TL phosphors, regardless of P-compounds concentrate. With increased the P-compounds concentration, mechanical strength is increased, but TL sensitivity is decreased. So, determined that optimum P-compounds concentrate is

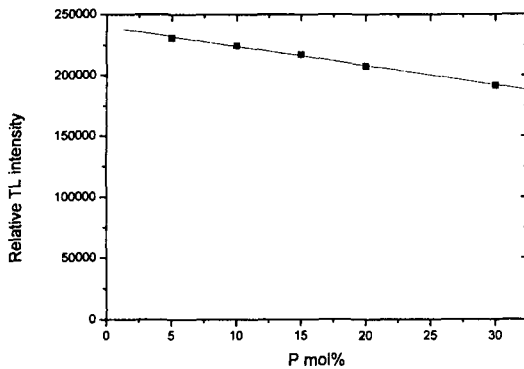


Fig. 3. TL Sensitivity of Pellets with P-compounds Concentrate

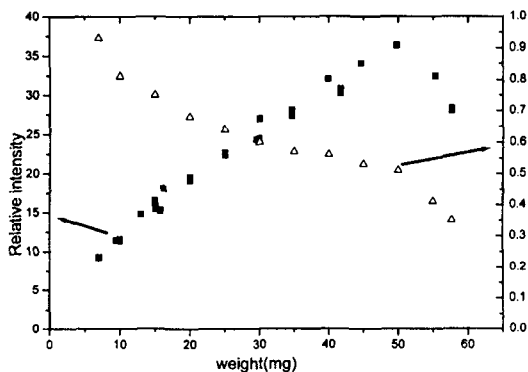


Fig. 4. TL Intensity of Embedded P-compounds $\text{CaSO}_4:\text{Dy}$ (P-compounds concentrate is 10mol%) with Weight
(Δ : Relative TL Sensitivity to Sensitivity of $\text{CaSO}_4:\text{Dy}$ TL Phosphors 10mg)

10~20mol%. This concentrate is corresponding to 5~10% of total pellet weight.

2.2.2. Determination of Weight

Fig. 4 is showed the result of TL intensity of embedded P-compounds $\text{CaSO}_4:\text{Dy}$ (P-compounds concentrate is 10mol%) with weight. TL intensity increased up to the weight 50mg. But it is not necessary too thickness (thickness is proportion weight), because of TL intensity of pellets is very

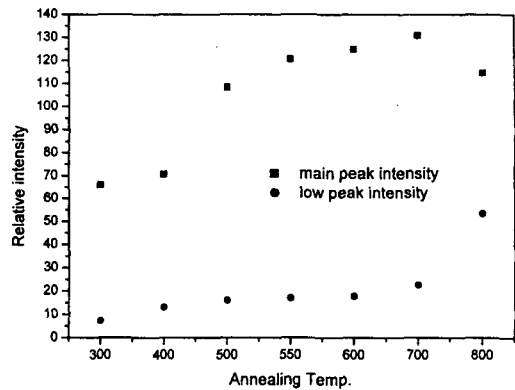


Fig. 5. Changing of TL Intensity with Annealing Temp

high. And if the thickness was very thin, pellets are fragile. Hence, weight of pellets optimized at 20 ~ 30mg (0.65~1.0mm thick.).

2.2.3. Manufacturing and Sintering Condition

The TL phosphors (63-100 μm) were added with P-compounds ($\text{NH}_4\text{H}_2\text{PO}_4$) of 10mol%, and uniformly mixed with a little distil-water. The mixture was pulverized after had been dried. Pellets of mixture of $\text{CaSO}_4:\text{Dy}$ TL phosphor and P-compounds of 10mol%, weighting 25mg, each, with a 4.5mm diameter and a thickness of 0.8mm, were first cold-pressed and then sintered. Fig. 5 showed changing of TL sensitivity (main peak and low temperature peak) with annealing temperature (300°C ~ 800°C). It is up to 400°C, TL sensitivity is very low. And region from 500°C to 700°C, main peak higher with increasing of annealing temperature. At 700°C main peak is the highest but low peak is increased too. So optimum annealing condition fixed 600°C, 30min.

2.2.4. Sensitivity

The result of comparison KCT-300 (4.5mm dia., 0.8mm thick., 25mg weight) with TL phosphors (phosphors weight of the same pellets weight as

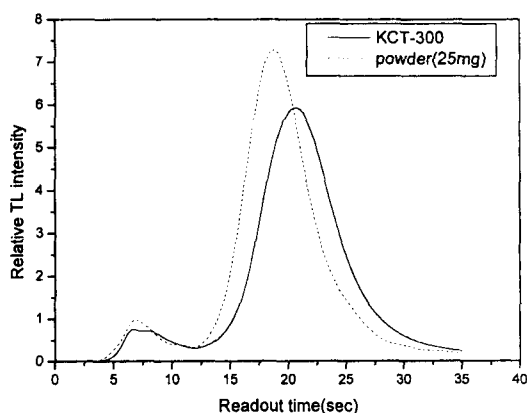


Fig. 6. Glow Curve of KCT-300 and TL Phosphors

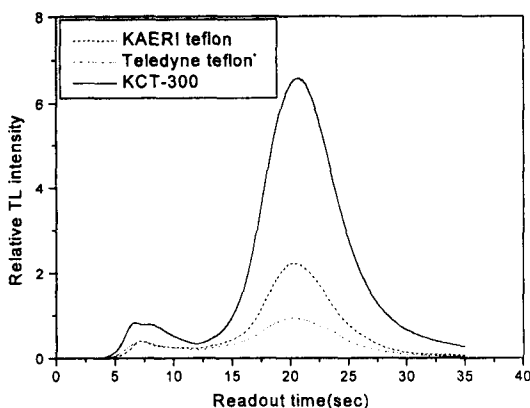


Fig. 7. Glow Curve of KCT-300 and Teflon Pellets(manufactured by KAERI and Teledyne)

25mg), sensitivity of KCT-300 corresponds to 90% of sensitivity of phosphors. Fig. 6 showed glow curve of pellets and TL phosphors. And Fig. 7 showed glow curve of pellets and Teflon pellets. In the case of Teflon pellets, it manufactured by KAERI and Teledyne. Sensitivity of KCT-300 is very higher than Teflon pellets. Pellets have sensitivity 3 times of KAERI Teflon pellets, and 6 times of Teledyne Teflon pellets.

3. Result

This paper presented fabrication of KCT-300 pellets and it's sensitivity. Optimum P-compounds

concentration of KCT-300 is 10 mol%, and cold-pressing. The pellets became solid detector after sintering at 600°C , 30min. And then pellets have sensitivity similar to TL phosphors. The sensitivity corresponds to about 6 times of Teflon pellet of Teledyne co. widely used. That is KCT-300 can be effectively used in personal dosimeter, especially it's have an advantage in low dose measurement.

Acknowledgments

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Reference

1. G.A.M.Webb, J.E.Dauch and G.Bodin. Operational evaluation of a new high sensitivity thermoluminescent dosimeter. *Health Phys.* 23, 89-94(1972).
2. S.S.Shastry, S.S.Shinde and R.C.Bhatt. Thermoluminescence response of $\text{CaSO}_4:\text{Dy}$ sintered pellets. *Int. J.Appl.Radiat.Isot* 31, 244-245(1980).
3. S.P.Morata, A.M.P.Gordon, E.N.D.Santos, L.Gomes, L.L.Campos, L.Prado, M.M.Vieira and V.N.Bapat. Development of a state dosimetry based on thermoluminescent CaSO_4 crystals. *Nucl. Instrum. Methods.* 200, 449-455(1982).
4. J.Azofin, A.Gutierrez and T. Niewiadomski. Performance tests of a $\text{CaSO}_4:\text{Dy}$ based thermoluminescence personal dosimeter. *Radiat. Prot. Dosim.* 17, 53-56(1986).
5. M.Prokic. Thermoluminescent characteristics of calcium sulphate solid detectors. *Radiat. Prot. Dosim.* 37, 271-274(1991).
6. T.Yamashita, N.Nacla, H. Onishi and S.Kitamura. Calcium sulphate activated by thulium or dysprosium for thermoluminescence dosimetry. *Health Phys.* 21, 295-300(1971).