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Development of KCT-300 TL Pellet with High Sensitivity and High Mechanical Strength

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KCT-300 (KAERI CaSO4:Dy TL detector with a P-compound of 7.8 wt%) developed at KAERI, appeared to have a higher radiation sensitivity than a commercialized Teflon embedded $CaSO_4$:Dy TL pellet. However, it was mechanically weak and fragile. Therefore, we try to improve its mechanical strength by increasing the P-compound concentration. It was difficult because of surface ruggedness problem in the TL pellet after sintering process. We could finally succeed in improving the mechanical strength of KCT-300 with high P-compound concentration by adding a thermal treatment process in the TL pellet fabrication process. This paper presents the method and result to improve the mechanical strength of KCT, which can be effectively used in the personal and environmental radiation monitoring.

KEYWORDS : thermoluminescence dosimeter, $CaSO_4$:Dy phosphor, Teflon powder, KCT-300, sintering

I. Introduction

$CaSO_4$:Dy thermoluminescence dosimeter (TLD) has been widely used as a personal or environmental dosimeter because of its high sensitivity to radiation. It is not possible to make solid detectors (or pellets) of only $CaSO_4$:Dy TL phosphor without any binding material. So, sintered pellets made from a mixture of $CaSO_4$:Dy TL phosphor and Teflon have been widely used until now. However, due to the reduction of $CaSO_4$:Dy phosphor content in TL pellets (15~20 wt%), the TL sensitivity was correspondingly reduced, and moreover the re-usability of this Teflon pellet was not good because of its color changing. Therefore, researches for finding good $CaSO_4$:Dy TL detectors by mixing $CaSO_4$:Dy TL phosphor with different binding materials have been carried out to effectively use this TL detectors[1)2)3)4)5)]. KCT-300 (KAERI CaSO4:Dy TL detector) is an another kind of $CaSO_4$:Dy TL detector which was recently developed at KAERI[6)], in which small amounts of $NH_4H_2PO_4$ have been embedded as a binding material. Its TL sensitivity turned out to be higher than the other $CaSO_4$:Dy TL detector. However, it was mechanically weak and fragile. The mechanical strength of KCT-300 could be improved by increasing $NH_4H_2PO_4$ content, but its TL sensitivity decreased and the surface of KCT-300 turned out rugged after sintering. Various efforts have been done to improve the mechanical strength of KCT-300 without losing its sensitivity. Finally, we have found an optimal method to solve this problem. This paper presents the method and result to improve the mechanical strength of KCT-300 by adding high temperature treatment process before fabrication into a final $CaSO_4$:Dy TL detectors.

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II. Materials and Methods

1. Fabrication of KCT-300

The conventional fabrication process of KCT-300 is shown in **Figure 1**. KCT-300 has been obtained after cold pressing of a homogeneous mixture of $NH_4H_2PO_4$ powder as binding material and $CaSO_4$:Dy TL phosphor (grain size: 63-100 ㎛). With increasing $NH_4H_2PO_4$ content, mechanical strength of the TL pellet accordingly increased. However it was not suitable for dosimeter because the surface ruggedness problem which. The reason of ruggedness problem seemed to be caused by the evaporation of NH_3 and H_2O during sintering the TL pellet. Therefore, in order to avoid this problem, a pre-heat treatment of this powder mixture before cold pressing has been adopted expecting the NH_3 and H_2O in $NH_4H_2PO_4$ compound to be evaporated during this pre-heat treatment process. The obtained result is shown in **Figure 5**.

The content of $NH_4H_2PO_4$ powder mixed with $CaSO_4$:Dy TL phosphor has been varied from 7.8 wt% to 20 wt%, 25 wt%, 33 wt%, 50 wt% and 66 wt% with a range of pre-heat treatment temperature from 200℃ to 500 ℃. At the result, the KCT-300 TL pellets made from this pre-heat treatment at 400℃ for 10 min did not have any surface ruggedness problem after sintering up to 33 wt% of $NH_4H_2PO_4$ powder content. In the range between 200℃ and 350℃, surface ruggedness have been reduced somewhat but it is not still suitable for dosimeter application. And at 500℃, mechanical strength of the TL pellets became weaker than the pre-heat treatment TL pellets at 400℃. Therefore, the optimum condition for pre-heat treatment temperature to produce KCT-300 has been estimated to be 400℃ for 10 min. **Figure 2** shows the new fabrication process of KCT-300 TL pellets.

2. Determination of P-compound content

With increasing $NH_4H_2PO_4$ content, mechanical strength of the KCT-300 increased accordingly but TL sensitivity decreased. However, even after such a reduction, TL sensitivity in KCT-300 remained quite high. KCT-300 appeared to have sensitivity six times as high as the Teledyne Teflon pellets. Experiments to improve the mechanical strength of KCT-300 have been conducted by varying $NH_4H_2PO_4$ content in the KCT-300.

The ratio of $NH_4H_2PO_4$ powder to $CaSO_4$:Dy TL phosphor has been changed from 7.8 wt% to 20 wt%, 25 wt%, 33 wt%, 50 wt% and 66 wt%. **Figure 3** shows the TL sensitivity of KCT-300 with varying the $NH_4H_2PO_4$ content from 7.8 wt% to 66 wt% after optimum pre-heat treatment for 10 min at 400℃. As seen in **Figure 3**, The TL intensity is reduced rapidly from above 25 wt% of $NH_4H_2PO_4$ contents. Between 20 wt% and 25 wt% of $NH_4H_2PO_4$ content, both mechanical strength and TL intensity appeared to be stable. Therefore, 25 wt% of $NH_4H_2PO_4$ in $CaSO_4$ TL phosphor have been chosen to be an optimum content to produce KCT-300.

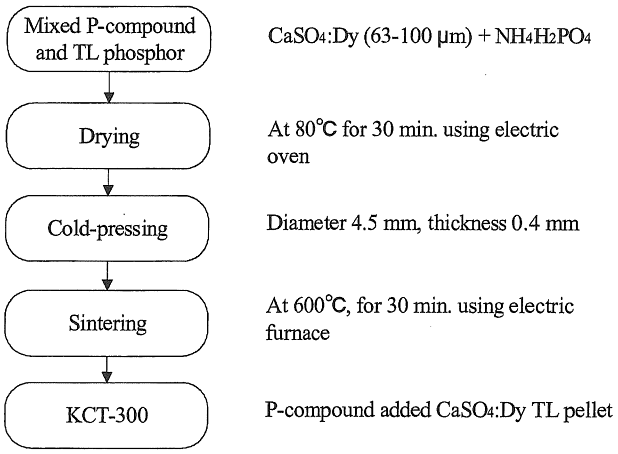

Figure 1 Conventional fabrication process of KCT-300

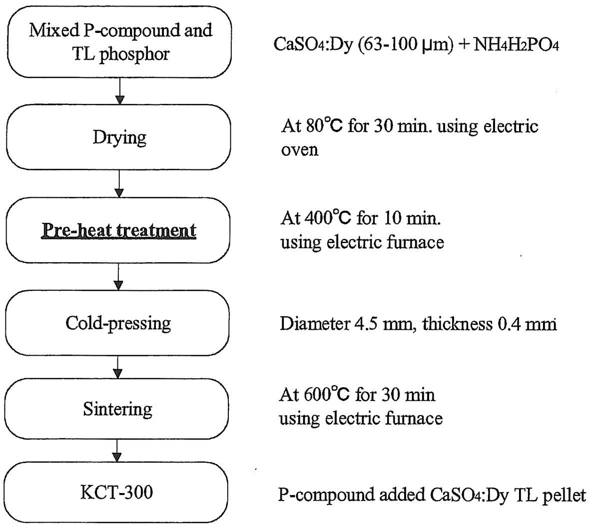

Figure 2 New fabrication process of KCT-300

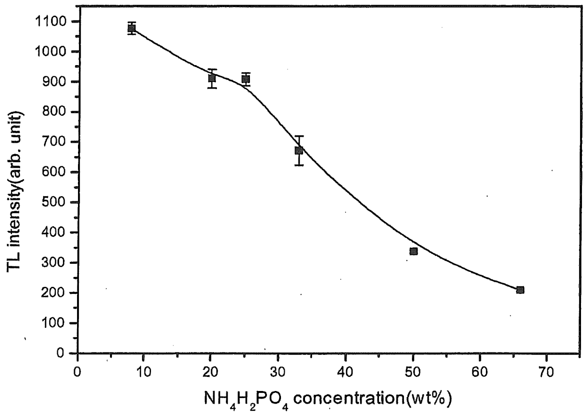

Figure 3 The TL sensitivities with increasing $NH_4H_2PO_4$ content after pre-heat treatment (400℃, 10min)

3. Determination of sintering condition

A pre-heated mixture of $CaSO_4$:Dy TL phosphor and $NH_4H_2PO_4$ (optimum content of 25 wt%) were cold-pressed (12 MPa) and sintered to produce a solid pellet (dimension : radius 4.5 mm, thickness 0.4 mm, weight 14 mg). KCT-300 pellets were irradiated in ^{90}Sr-^{90}Y reference beta source in laboratory. The intensity of KCT-300 TL detectors was measured with a Teledyne System 310 TLD reader. The heating cycle consists of a step-type with pre-heating at 135℃ and a linear heating up to 280℃. **Figure 4** shows the variation of TL intensity of main and low temperature peak with increasing sintering temperature from 300℃ to 800℃. TL intensity of main peak is very low up to 400℃ and increases with increasing sintering temperature to 700℃. TL intensity of main peak is highest at 700℃ but the intensity of low peak also increases. Therefore, an optimum sintering temperature and time were decided to be 600℃ for 30 min where the ratio of the TL intensity of the main peak to that of the low peak is maximum as shown in **Figure 4**.

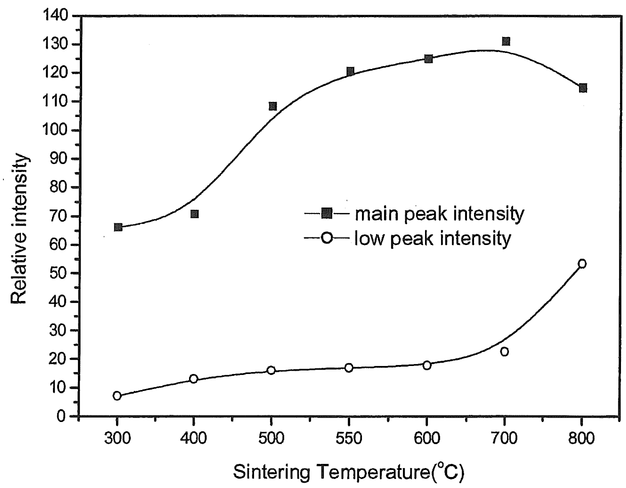

Figure 4 Dependence of the TL intensity on the sintering temperature

Two kinds of KCT-300 manufactured by the conventional process (left one) and the new process (right one) are shown in **Figure 5**. Each KCT-300 has same 25 wt% content of $NH_4H_2PO_4$ and is sintered at 600℃ for 10 min. As seen in **Figure 5**, the surface ruggedness problem in TL pellet made from a conventional fabrication process (**Figure 1**) is now resolved by applying a pre-heat treatment process before cold-pressing and sintering to make a final KCT-300.

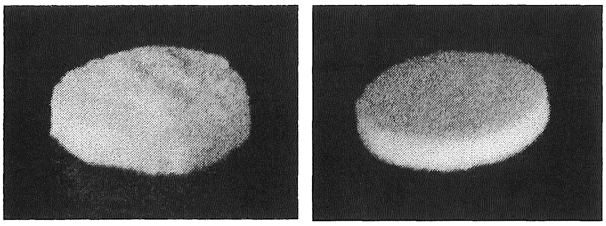

Figure 5 Comparison of surface of two kinds of KCT-300 manufactured by the conventional process (left one) and the new process (right one)

4. Sensitivity of KCT-300

The glow curves of KCT-300 and $CaSO_4$:Dy Teflon pellets are shown in **Figure 6**. As seen in **Figure 6**, the sensitivity of KCT-300 is much higher than the $CaSO_4$:Dy Teflon pellets. A sensitivity of KCT-300 is 2.5 times than that of KAERI Teflon pellets[7] and five times as high as of Teledyne Teflon pellets. **Figure 7** shows glow curve of KCT-300 and India made $CaSO_4$:Dy pellet recently developed[8]. The sensitivity of KCT-300 is about 2.5 times as high as of $CaSO_4$:Dy TL pellet embedded with another kind inorganic binding materials, which was recently developed in India. A Time-Temperature Profile (TTP) for KCT-300 readout is as follow. KCT-300 is pre-heated at 135℃ for 4 sec (it is different from the pre-heat treatment of TL material in the fabrication process of KCT-300), and heated up to 280℃ with heating rate of 15℃/sec and then maintain 280℃ for 10 sec for annealing.

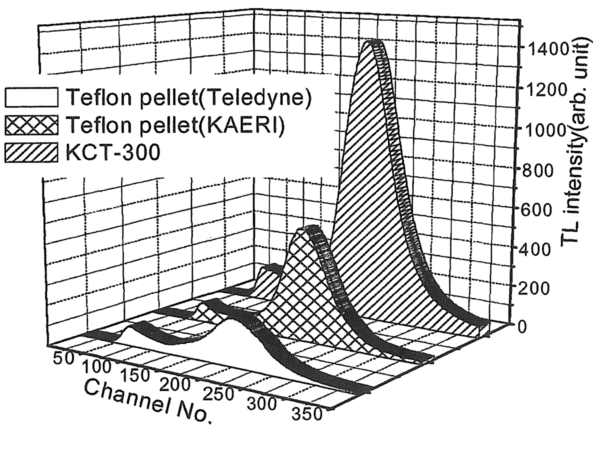

Figure 6 Glow curves of KCT-300 and $CaSO_4$:Dy Teflon pellets (Teflon pellets by KAERI and Teledyne)

III. Summary and Conclusion

The first kind of KCT-300 developed at KAERI which adopts a small amounts of $NH_4H_2PO_4$ as a binding material shows much higher sensitivity than any other $CaSO_4$:Dy TL detectors. However, its mechanical strength was not good and fragile. Various efforts have been done to improve the mechanical strength of KCT-300 without losing its sensitivity. In this paper, we have presented a method to improve the mechanical strength of KCT-300. The mechanical strength of KCT-300 has been improved by adding a new process of pre-heat treatment of the mixture of optimum 25 wt% of $NH_3H_2PO_4$ powder and $CaSO_4$:Dy TL phosphor at 400℃ for 10 min before cold pressing (12 MPa) to produce a TL pellet. An optimum sintering condition to produce a solid TL pellet (new KCT-300) was 600℃ for 30 min. The new KCT-300 turned out to have a high sensitivity as much as 5 times of Teledyne $CaSO_4$:Dy Teflon pellets and 2.5 times of India made $CaSO_4$:Dy TL pellet. Therefore, the new KCT-300 with high sensitivity and high mechanical strength could be effectively utilized in environmental and individual radiation monitoring.

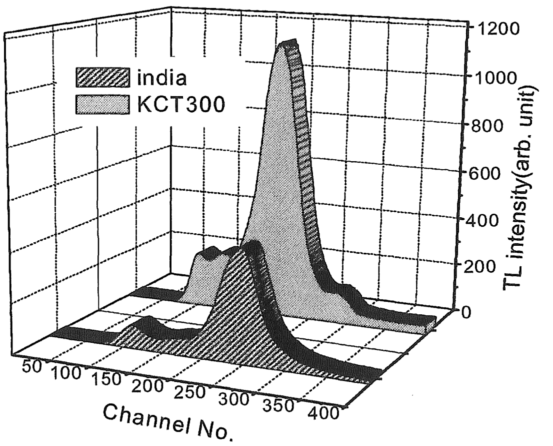

Figure 7 Glow curves of KCT-300 and newly developed India $CaSO_4$:Dy pellet

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