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Thermoluminescence Performance of Carbon-doped Aluminium Oxide for Dose Measurement by Various Preparation Methods

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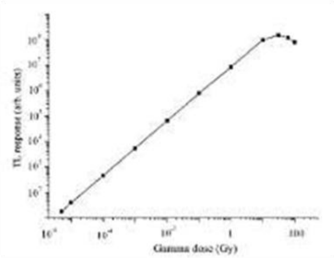
Graphical abstract



Abstract

Thermoluminescent dosimeter (TLD) of carbon-doped aluminium oxide (α-Al_2O_3:C) produced in the form of single crystals show high sensitivity to ionizing radiation (about 40-60 times higher than TLD-100 (LiF:Mg,Ti)). The present article offers a review of the materials preparation and corresponding thermoluminescence (TL) properties of α-Al_2O_3:C subjected to various types of ionizing radiations. Different methods of α-Al_2O_3:C preparation in form of single crystal and thin films are reviewed. The development of methods of preparation is based on the approaches that involve the evaluation of the luminescence light yield in TL process. Most of the methods used were suitable, but each of these methods has their advantages and disadvantages depending on the required form of materials. Considering the results presented by various authors, possible better method of material preparation is proposed. The potential alternative fabrication technique of α-Al_2O_3:C thin film by using radio-frequency magnetron sputtering is briefly discussed.

Keywords: TLD; carbon-doped aluminium oxide; TL process; luminescence light yield; radio-frequency magnetron sputtering

Abstrak

Aluminium oksida yang diaktifkan dengan karbon (α-Al_2O_3:C) digunakan sebagai bahan termopendarcahaya dalam dosemeter termopendarcahaya (TLD). α-Al_2O_3:C dalam bentuk kristal tunggal menunjukkan tahap sensitiviti yang tinggi terhadap sinaran mengion (kira-kira 40-60 kali lebih tinggi daripada TLD-100 (LiF: Mg,Ti)). Artikel ini membentangkan kajian tentang cara penyediaan α-Al_2O_3:C dan ciri-ciri termopendarcahayanya apabila terkena pelbagai jenis sinaran mengion. Kaedah penyediaan bahan ini dalam bentuk kristal tunggal dan filem nipis yang berbeza telah dikajikan. Pembangunan kaedah penyediaan adalah berdasarkan pendekatan yang melibatkan jumlah kuantiti pendarcahaya yang dipancarkan oleh bahan termopendarcahaya dalam proses TL. Kebanyakan kaedah penyediaan yang diaplikasikan adalah sesuai, namum demikian kaedah-kaedah tersebut masih mempunyai kelebihan dan kekurangan masing-masing bergantung kepada bentuk sample yang diperlukan. Merujuk kepada keputusan yang telah dibentangkan oleh penulis-penulis dari seluruh dunia, kaedah penyediaan bahan yang mungkin lebih baik akan dicadangkan. Justeru, cara alternatif yang berpotensi untuk menghasilkan α-Al_2O_3:C dalam bentuk filem nipis dengan menggunakan teknik magnetron sputtering berfrekuensi gelombang radio akan dibincangkan secara ringkas.

Kata kunci: TLD; aluminium oksida yang diaktifkan dengan karbon; proses TL; kuantiti pendarcahaya; magnetron sputtering berfrekuensi gelombang radio

■1.0 INTRODUCTION

Thermoluminescence dosimeters are used primarily to detect and monitor the amount of exposure to radiation in order to keep a person within safe level especially for medical purpose. Thermoluminescent dosimeters were not used extensively until the 1960s when TLD badges became more popular. Instead of reading the optical density (blackness) of a film, as is done with film badges, the amount of light released versus the heating of the individual pieces of thermoluminescent material is measured. The glow curve produced by this process is then related to the radiation exposure. In year of 1957, the dosimetric properties of aluminium oxide (Al_2O_3) were first described by Rieke & Daniel [1] with a later investigation of its TLD behavior by McDougall & Rudin in 1970 [2]. To have better performance in dosimetric field, Al_2O_3 is always doped with impurities that induce many different types of trapping centers exist at which charged particles produced by ionizing radiation can be trapped.

Recently, there are a lots of efforts have been directed towards the improvement of its sensitivity via introduction of various dopants like Si,Ti [3], Mg and Y [4], Cr and Ni [5]. In this review, it is only focus on carbon-doped aluminium oxide (α-Al2O3:C) as the TL material. Based on previous research done by Akselrod *et al.* in year 1993, α-Al2O3:C phosphor has thermoluminescence (TL) sensitivity 40 to 60 times higher than TLD-100 and its emission at 410-420 nm coincides with the region of most favorable response to the photomultiplier tubes [6]. Other advantageous properties of α-Al2O3:C, as linearity in a wide range dose, simple glow curve, low fading, good reproducibility, mechanical resistance and relative low atomic number.

The presence of impurities in a material is important for the thermoluminescene process. High luminescence sensitivity in carbon-doped aluminum oxide can be achieved with high concentration of dosimetric trapping centers. The dosimetric traps in this material are the result of oxygen vacancy centers in the crystal called F and F+ centers. Yang *et al*. (2008) reported that introduction of carbon into Al2O3 will cause the two-valent carbon ions replace the three-valent cations of Al, which leads to the formation of hole trapping centers during the growth process [7]. They observed that the F+ centers' absorption band intensity increases with increasing carbon content in the crystal, which testifies to the fact that F+ centers are formed as charge compensators to heterovalent impurity C2+ ions. Most likely, the F-centers are part of aggregate defects made up of oxygen vacancies and impurities present in crystals. In short, when a material is exposed to ionizing radiation, part of the absorbed energy is stored in the metastable energy levels of its electronic bands. Adding some impurities or causing defects in the lattice structure or in some other way may form local energy levels or traps in a material. Part of the stored energy may later be released as visible light by heating the material. This phenomenon is called thermoluminescence (TL).

Thermoluminescence dosimeter materials presently in use are inorganic crystalline materials and are referred as phosphors due to their ability to emit visible light radiation when suitably excited [8]. They are available in a variety of forms, including powders, compressed chip, Treflon-impregnated disks, single crystals, and thin films. Conventionally, TLD phosphor is fabricated utilizing various methods such as crystal growth technique, electrochemical oxidation [9,10], sol-gel technique [11], ion beam implantation [12] and combustion synthesis. In this review, we will focus on the performance of all fabrication techniques of α- Al2O3:C in form of crystal and thin film. This is due to high sensitivity has been attributed to oxygen vacancy centers produced during the material preparation. Thus, the good TL properties of the materials are always depending on the defects created and methods fabrication that used.

2.0 ATTRACTIVE THERMOLUMINESCENCE CHARACTERISTICS OF α-Al2O3:C

The latest spike of interest in α-Al2O3 (sapphire) is easy to explain taking into account the optical, chemical and thermal stability under irradiation and the availability of well established, high productivity and low cost crystal growth technology. Incorporation of element carbon into α-Al2O3 to increase its dosimetric sensitivity had created a new era in application of α- Al2O3 despite of conventional existing application such as mechanical, optical and micro-mechanical applications. This has been proven by a brilliant research group after they proposed a technology to increase the anion deficiency in the crystals by growing them under strongly reducing conditions [13, 14, 15]. In the research, they concluded aluminium oxide doped with carbon was ranked as the most sensitive material in TL dosimetry.

In 2007, Kortov. V had done a review on the studies and application of thermoluminescence dosimetric material. In the paper, he stated some main requirements must be imposed on materials for TLD to have optimum performance in assessing accurate absorbed dose [16]. α-Al2O3:C possess good characteristic of TL material as (a) wide range of linear dependence between luminescence intensity and absorbed dose from 10-7 to 10 Gy, (b) high sensitivity in which a high TL signal per unit absorbed dose will be obtained (approximately 40-60 times greater than LiF: Mg, Ti), (c) independency of the TL response on the incident radiation, (d) low fading during storage in the dark (less than 5% per year), (e) simple TL glow curve with TL peaks at 190ºC, and (f) mechanically strong, chemically inert and radiation resistant.

3.0 FABRICATION METHODS OF α-Al2O3:C

Since α-Al2O3:C has emerged as a TL material for radiation dosimetry, there are many preparation techniques have been applied to produce α-Al2O3:C especially in crystal form. Conventionally, α-Al2O3:C utilizes Czochralsky or Venuil crystal growth technique as its fabrication method. This technique involves crystal growth from melting temperature (2050 ºC) and carried out in the highly reducing conditions in the presence of graphite. There are pros and corns of this method. The dosimetric characteristics are very depends on the growth parameters in which a slight change in growth condition will affect the formation of traps and distribution of defects. The conventional method of carbon incorporation is limited by the fact that doping and crystal growth occur simultaneously at higher temperature because carbon incorporation cannot be controlled precisely into the molten mass from where the crystal is grown, thus the consequent generation of defects is hard to control. Besides conventional fabrication method, different fabrication methods of α-Al2O3:C as shown in table 1 that have been conducted in thermoluminescent dosimetry are reviewed.

Table 1 A review of different fabrication methods of α-Al2O3:C in form of single crystal and thin film that have been conducted in thermoluminescent dosimetry

Materials	Preparation methods	Form of materials	Type of exposure	Response	Authors
α-Al2O3:C	Vacuum-assisted Post-growth Thermal Impurification Technique	Single crystal	Sr-90 / Y-90 (50 μGy - 1 Gy)	41 times higher than TLD-100	Kulkarni, M.S. *et.al* (2005)
α-Al2O3:C	Temperature Gradient Technique	Single crystal	Sr-90 / Y-90 (5 mGy - 10 Gy)	40-60 times higher than TLD-100	Xinbo, Y. *et.al* (2008)
Al2O3:Tb , Si, Eu	Combustion Synthesis	Single crystal	Co-60 (100 mGy - 70 Gy)	5000 times higher than the undoped Al2O3.	Barros, V.S.M. *et.al* (2008)
α-Al2O3:C	Electrochemical Anodizing	Nanoporous	Co-60 (200 mGy - 1000 mGy)	-	Barros, V.S.M. *et.al* (2007)
Al2O3	Laser Ablation	Nano-sized Thin Film (Amorphous)	Sr-90 / Y-90 (2.5 Gy - 20 Gy)	-	Villarreal-Barajasa, J. E. *et.al* (2002)

According to Kulkarnia *et al.* (2005) [17], an alternative preparation method of α-Al2O3:C by vacuum-assisted post-growth thermal impurification technique was introduced. This technique was applied based on the disadvantages brought by a forementioned conventional crystal growth technique. In this technique, single crystal α-Al2O3:C (10×10 mm2; 0.4 mm thick) was heated at temperatures ranging from 1100 ºC to 1500 ºC in the vacuum (~1.33×10-4 Pa) in the presence of graphite. The temperature of the furnace was controlled to within ±1 ºC using a temperature controller of the type Eurotherm 2416. Two well- defined glow peaks at 56ºC and 191 ºC were obtained in the TL readout. The TL sensitivity of the sample is found to be 41 times higher than the TLD-100. This fabrication method has an advantage over the conventional method in term of involving temperature which is substantially lower than the melting point of α-Al2O3 (2047 ºC). Other than that, the extent of defect creation can be varied by changing the process temperature and time.

Xinbo. Y and his research group did another attempt on using temperature gradient technique (TGT) to produce highly sensitive TL crystal α-Al2O3:C in year 2008. According to the research, TGT is a simple directional solidification technique, which has been used for the growth of high temperature crystals by Shanghai Institute of Optics and Fine Mechanics for many years. In TGT technique, α-Al2O3:C crystal was grown in a tapered molybdenum crucible. The TGT furnace was heated at 1827 ºC for several hours to eliminate surface impurities so as to minimize the environmental contamination. Then the furnace was loaded for the growth process, evacuated to 10-3 Pa, heated to 2076 ºC, and kept 5×10-6 to 10 Gy and saturation at about 30 Gy. However, α- Al2O3:C crystal could not be irradiated at < 5×10-6 Gy as limited by the experimental conditions as shown in the Figure 1.

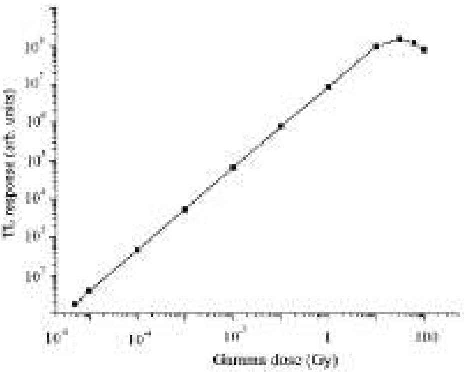


Figure 1 TL response of α-Al2O3:C crystal relative to gamma dose (Xinbo. Y *et al.* (2008))

Combustion synthesis (CS) is also one of a suitable method to prepare Al2O3 doped materials for TLD. Barros V.S.M. e*t al.* (2008) conducted a research based on preparing Al2O3 doped with rare-earth materials by using combustion synthesis. For this method, brief explanation is written because there is no detailsabout fabricated α-Al O :C crystal through CS method but itin the molten state for several hours. After the temperature field was stabilized, crystallization was started by slow cooling (-270.15 ºC/h) with a high precision temperature program controller.

Compared to Czochralsky method, TGT has a distinguishing feature that the solid-liquid interface is submerged beneath the melt surface and is surrounded by the high-temperature melt until the liquid is all gone [18]. Crystal growth is carried out under stable temperature gradients and the temperature field in the high- temperature melt is opposite to the gravitational field orientation which minimizes the convection effects. In this research, α- Al2O3:C crystal showed a single glow peak at 189ºC and a blue emission peak at 415 nm after irradiated with different dose of beta source. It also showed excellent linearity in dose range from might has good TL performance as stated in this research. In this method, the aluminium oxide doped materials were preparedby mixing stoichiometric amount of aluminium nitrate, urea and desired dopant nitrate. The mixture was put into a muffle furnace preheat at 500 ºC where it ignited spontaneously within few seconds. The resulting powder was pelletized and annealed at temperature ranging from 1000 ºC to 1400 ºC. In particular, the CS method is an excellent technique for preparing crystalline materials because of its low cost, high yield and the extreme facility to prepare samples with well-defined microstructure at low processing temperatures as low as 500 ºC and in short reaction times (∼s) [19]. On top of that, CS process is based on the use of the heat released from the redox chemical reaction, instead of the use of intensive high-temperature furnaces, to supply the energy necessary for the synthesis. The author observed that the Al2O3:Eu doped samples showed an isolated and well defined peak at around 200 ºC, which seems well suited for radiation dosimetry.

■4.0 NANO-SIZED α -AL2O3:C IN THERMOLUMINESCENCE MATERIAL

In the previous section, it is mentioned that α-Al2O3:C is produced in form of single crystal and require sophisticated laboratories. Currently, the importance of nano-materials in the field of luminescence, has been increased, especially, as they exhibit enhanced optical, electronic and structural properties. It is interesting to note that Kortov. V pointed out some opportunities arising in connection with the use of nano-sized materials in TLD in the part of future trend for TL materials in year of 2007. The statement was then supported by a research about an alternative route to synthesize nanoporous carbon doped aluminum oxide prepared through electrochemical oxidation of aluminum in organic acids with subsequent thermal treatment in the same year [20, 21]. In the method, thin films were obtained with a highly ordered pore distribution with diameter of the order of 50 nm, under constant voltage in organic acid solutions by using anodizing process of aluminium. The TL glow curve consists of first peak in 110 ºC region and second peak at 190 ºC when sample irradiated with a Co-60 gamma dose of 450 mGy. This result showed this method is a suitable fabrication method of TL material in nano-sized scale. However, its TL sensitivity is still under investigation.

The great discovery of nano-sized in TL material and corresponding dosimetric performances helps enhance the development of different thin film fabrication methods. In year of 2002, the main TL properties of amorphous aluminum oxide thin film which prepared by pulsed laser deposition with thickness as low as 300 nm was presented by Barajas, J.E.V. et.al [22]. A detailed description of this experimental and deposition procedure can refer to Ref. [23]. Pulsed laser deposition technique is a popular method to produce thin film materials owing to its advantages over other deposition technique. The advantages are use of small target, the conservation of the stoichiometry on the deposited film, easy handling of the technique and the feasibility to control the thickness of the thin film [24]. As the result of this research, TL glow curve exhibited two peaks at 95 ºC and 162 ºC for beta irradiation. It is also worth noting that for doses below 2.5 Gy, the TL response was very poor and more detailed characterization of the thin film as well as the effects in the thin film has to be investigated. Furthermore, there is so far no investigation done towards produced sample that irradiated by gamma irradiation.

Based on all of the disadvantages of fabrication techniques in preparing the TL material either in crystal or thin film form, they contribute to discover a more suitable method to produce the TL materials that applicable and sustainable in accessing dose absorbed for environmental and personal monitoring. An alternative method to prepare α-Al2O3:C thin film for dosimetric application is being proposed. Nanoscale thin films TL materials are suggested produced by using radio frequency (RF) magnetron sputtering method. Although this methodology is very rarely used in samples preparation, it may bring a new discovery to dosimetry field because the thin film properties can be controlled by using an appropriate selection of the deposition parameter which may improve the properties of recent TL detectors. This proposed method will be discussed further in next section.

■5.0 RADIO FREQUENCY (RF) MAGNETRON SPUTTERING

There are some thin film coating methods in the market nowadays include electron beam deposition, chemical vapor deposition (CVD), physical vapor deposition (PVD) or conversion plating. RF magnetron sputtering is grouped under the PVD. With a better understanding of the sputtering processes and development of RF sputtering, sputtering has become one of the most versatile techniques in thin film technology for preparing thin solids films of almost any material. Some of the advantages of sputtering as thin film preparation method over other thin film fabrication methods are (a) high uniformity of thickness of the deposited film, (b) good adhesion to substrate, (c) better reproducibility, (d) maintenance of the stoichiometry of the original target composition, and (e) relative simplicity of film thickness control [25].

Sputter deposition is basically a process in which ionized atoms are accelerated into a surface (sputter target) in order to eject atoms from the surface. The ejected atom can then be condensed onto a substrate to nucleate a thin film of the ejected atoms. In the 1970s, the development of magnetron source has created a significant advance to increase the efficiency of sputter tooling. The magnetron uses strong magnetic fields from the permanent magnet to keep secondary electron spatially confined in the vicinity of the target surface. Thus, greater ionization of sputter gas-atoms, denser plasma, and higher plasma currents and deposition rates are produced due to their residence time in the plasma is greatly lengthened.

In the other hand, RF sputtering is applicable for high melting materials or insulating targets such as oxides and nitrides. The typical radio frequency of 13.56 MHz is supplied to the electrodes in RF sputtering to generate an alternating current in the deposition chamber owing to the limitation of the DC diode apparatus to achieve high levels of gas ionization and sputtering of the cathode. This is done purposely to build up a negative self- bias on the target. In such a case the argon ions, Ar+ have a tendency to neutralize the target negative charge applied to the target and eventually the ions will not attracted to the target anymore (no sputtering takes place). To overcome this, an alternating current in RF is used rather than DC. Ions cannot follow this frequency (too heavy and slow), but electron do, thus building up a negative self-bias on the target. Similarly the Ar+ will be easily bombarded the target surface, removing particles as thin film. Sputtering a mixture of elements or compounds will not result in a change of composition in the target and thus the composition of the vapor phase will be the same as that of the target and remain the same during the deposition.

■6.0 CONCLUSION

It is shown that α-Al2O3:C is an excellent and popular TL materials despite of TLD-100 and widely used among various TL materials due to the abundance source of carbon as dopant on earth than other effective TL materials. Hence, many new physical and chemical methods of preparations have also been developed in the last two decades to look for most suitable fabrication methods of TL materials in order to produce a very effective TLD. It seem that α-Al2O3:C can be prepared in thin film of crystal form through various fabrication technique. However, there is no a perfect preparation method of this TL materials being discovered in getting the optimum TLD performance in assessing medium dose and high dose of various types of ionizing irradiation. At the end of this paper, I would like to suggest an alternative fabrication method of α-Al2O3:C thin film by using RF magnetron sputtering in order to have optimal light emission, linearity in a wide range of medium and high doses of ionizing radiation. Further investigations are in progress to examine the suitability of radio-frequency magnetron sputtering technique to become a potential fabrication method of α-Al2O3:C thin films by showing good TL properties.

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