Synthesis and characterization of flower-like Al$_2$O$_3$:C for optically stimulated luminescence (OSL) dosimeter

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Abstract

Growth in radiation diagnosis and therapy drives personal radiation dosimeters for physical and radiological protection control. Nevertheless, OSL (optically stimulated luminescence) personal dosimeters in Thailand are all imported. In this work, the particulate and flower-like structure of carbon-doped alpha aluminum oxide polycrystal (α-Al$_2$O$_3$:C) with the various carbon dopants (0.1% and 1%) were studied as a detector in the OSL personal dosimeter. The particulate powder was prepared by alumina powder (295 nm). The flower-like structure α-Al$_2$O$_3$:C was prepared by boehmite precursors, synthesizing from aluminum sulfate via the low-cost and low-temperature hydrothermal method and sucrose as carbon sources. The crystalline structure was identified by X-ray diffraction (XRD). The chemical properties and oxygen vacancies were investigated by Fourier-transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS). The size of as-prepared flower-like structure α-Al$_2$O$_3$: C was 2–3 μm confirmed by scanning electron microscopy (SEM). The alumina precursors and carbon dopants affected electron trapping states, morphologies, and OSL outputs. This finding successfully proved that the flower-like α-Al$_2$O$_3$ derived from boehmite precursor exhibits excellent OSL sensitivities to beta irradiation (better than that of the particulate power of α-Al$_2$O$_3$: C) and responded linearly in the range of 0–80 kGy.

1. Introduction

According to International Basic Safety Standards for Protection against Ionizing Radiation and the Safety of Radiation Sources (The International Atomic Energy Agency, IAEA), the radiation monitoring equipment must be provided to the operator and supervised not to receive radiation over the dose limit [1]. Therefore, the personal dosimeter is a significant device for personnel and those involved in the application of radiation sources (radiation therapists, patients, and researchers). The development of radiation diagnosis and treatment in the past decade also raises the demand for accurate personal dosimeter worldwide. However, the market share of personal dosimeter is limited by major players from developed countries: Landauer Inc., Mirion Technologies Inc., Thermo Fisher Scientific, Fuji Electric Co. Ltd and ATOMTEX Fouards Srl Ridzwan et al reported that for middle-income Asian countries, medical radiation workers could not fully access personal dosimeters due to the financial factor and slow procurement from international companies [2]. For Thailand, all dosimeters and reading systems are currently used all imported. Thailand Institute of nuclear technology (public organization) can only provide the OSL (optically stimulated luminescence) personal dosimeters reading, recording, and reporting for legal proceedings. Therefore, self-production in developing and undeveloped countries is a suitable solution to increase personal dosimeter uses.
The crystal detector inside the OSL dosimeter can store the absorbed ionizing radiation as trapped electrons and holes (figure 1(a)). The trapped electrons are de-trapped by light stimulation with the desired wavelength and obtaining OSL signal from the recombination of the trapped electron at the recombination center [3, 4]. Furthermore, re-reading and re-analysis of radiation stored in the OSL dosimeter can be achieved by adjusting the suitable stimulation laser to the suitable power. Carbon doped aluminum oxide (Al₂O₃:C) is commonly used as a phosphor detector in OSL personal dosimeters. The carbon dopants substitute the oxygen atoms, promoting oxygen vacancies that act as luminescence centers [3, 4]. The oxygen vacancies trapping one and two electrons are namely F⁺-center and F-center, respectively, as shown in figure 1(b).

Single crystal of α-Al₂O₃:C is commonly used as an OSL detector [3–6]. For example, the OSL dosimeter detectors from Landauer Inc. (a key player in the personal dosimeter world market) are produced from crushing α-Al₂O₃:C single crystal to improve the homogeneity of detectors [3–6]. The α-Al₂O₃:C single-crystal can be grown by several methods: Czochralski (Cz) method [7], temperature gradient technique (TGT) [6], and vertical gradient freezing (VGF) [8]. However, the highly reducing atmosphere in graphite presence, elevated temperatures (> 2000 °C), and expertise are required [3, 4]. These are obstacles for developing and undeveloped countries for their self-production.

The Al₂O₃:C polycrystal is also a promising phosphor according to ease of production and cost efficiency [9]. Several alternative routes to synthesize α-Al₂O₃ polycrystals have been explored, such as sol-gel method, heat treatment of Al₂O₃ powder in the presence of graphite [9], and thermal decomposition of aluminum precursors including alumina alum (2NH₄Al(SO₄)₂·12H₂O) [10], ammonium aluminum carbonate hydroxide (AACH) [11, 12] and boehmite (AlOOH) [13, 14]. Fontainha et al reported that α-Al₂O₃:C polycrystals showed high sensitivity to x-ray and gamma radiation in the dose range of 0.02–500 mGy [9]. Polo et al reported the OSL response of sol-gel α-Al₂O₃ with different concentrations of impurities recently [15]. The linear response was in the beta dose of 5 mGy to 1 Gy (90Sr/90Y). The same group research also studied the signal fading of sol-gel α-Al₂O₃. It showed that the time-dependent fading was followed the exponential function (720 h experiments), and the signal lost 13 % after one week [16].

Flower-like structure Al₂O₃ are created by combining Al₂O₃ nano-flakes, leading to a high total specific area [17]. It was utilized in several applications, such as adsorbents [18, 19] and catalyst supports [19, 20]. Wang et al reported that using the flower-like Al₂O₃ as Fe-based catalyst support can enhance Fe dispersion leading to the superior catalyst for oxidative dehydrogenation of ethylbenzene with CO₂ [18]. Besides, the composites of flower-like Al₂O₃ and NiS₂ represented excellent performances for hydrogen evolution reaction due to high electrochemical stability from none aggregation and tight bonding of NiS₂ and the flower-like Al₂O₃ [19]. The morphologies of Al₂O₃ affect the physicochemical, optical and electrical properties [20, 21]. Although the synthesis of flower-like Al₂O₃ has already been well-known, only a few previous types of research reported the OSL dosimetry properties of flower-like Al₂O₃ structures. The hydrothermal method was reported as high-efficiency, simple, low-cost flower-like α-Al₂O₃ polycrystal [18–20, 22]. Also, it has a strong potential to be fabricated for commercial purposes.

Therefore, in this finding, the radiation response of flower-like Al₂O₃:C was investigated to prove the potential of using them as a radiation dosimeter detector. An effect of carbon dopants on their properties was also studied. Furthermore, the physicochemical, optical, and OSL properties of flower-like α-Al₂O₃:C polycrystals were compared with α-Al₂O₃:C polycrystal prepared by alumina powder activated carbon. Low cost and simple hydrothermal method (adapted from [18–20, 22]) was used to synthesize boehmite precursor
(AlOOH) from low-cost starting materials (aluminum sulfate sucrose). The annealing of precursors was done at 1200 °C under N2 atmosphere for phase transformation to α-Al2O3: C.

2. Materials and methods

Alumina powder (99.7%, Beijing Dingsheng Special Ceramics Manufacture Co, Ltd.) and activated carbon (AC) powder (Gelon Lib Group) were used as raw materials for α-Al2O3:C fabrication. In addition, aluminum sulfate octadecahydrate (Al2(SO4)3·18H2O, 97% Kemaus), urea (CO(NH2)2, 99.5% Merck), sucrose (C12H22O11, 99.5% Ajax Finechem), and deionized water (DI, Milli-Q 18.2 MΩ) was used as the starting materials for the synthesis of boehmite (AlOOH) as alumina precursor. Commercial Al2O3 : C OSL detectors were taken from the InLight-LDR Model 2 (Landauer Inc. USA) as shown in figure S1 (available online at stacks.iop.org/MRX/8/096202/mmedia) (supporting document).

In this work, two alumina precursors (alumina powder and boehmite) were used. For the first approach, the alumina powder was mixed homogeneously with 0.1 and 1 wt% of AC by vortex mixer (MIXER UZUSIO VTX-3000L) at 3000 rpm. Next, the powder mixture was placed into the alumina crucible and placed in an oven at 120 °C for 2–4 h with a heating/cooling rate of 5 °C min⁻¹ under the 10⁻³ mbar vacuum in the tubular furnace (CARBOLITE GERO30–3000). The as-received samples were named Al series: UAl, 01Al, and 1Al for undoped Al2O3, 0.1 wt% carbon-doped Al2O3, and 1 wt% carbon-doped Al2O3, respectively.

For the second approach, the boehmite (AlOOH) was synthesized by the hydrothermal method. Three grams of aluminum sulfate, one gram of urea, and sucrose (0.1 and 1 wt%) were mixed thoroughly with 30 ml of DI water. The mixture was then poured into a Teflon-lined autoclave and placed in an oven at 120 °C for 2 h. The obtained precipitate was filtered and repeatedly washed with DI water. Finally, the synthesized boehmite powder was dried at 50 °C overnight before annealing at the same condition as described previously. The as-received samples were named B series: UB, 01B, and 1B for undoped Al2O3, 0.1 wt% carbon-doped Al2O3, and 1 wt% carbon-doped Al2O3, respectively.

The crystal structure of as-received samples was investigated by x-ray diffractometer (XRD, Bruker D8 advance), using copper Kα (λ = 1.54056 Å) with 30 kV and 30 mA. The morphology and particle sizes were studied by Scanning electron microscope (Tescan Vega 3) and dynamic light scattering (DLS, Malvern zetasizer Ultra). ATR-IR spectra were obtained by a Bruker (model Tensor 27) spectrometer in a range of 4000–650 cm⁻¹. The solid-state diffuse reflectance technique (DRS) was performed on Shimadzu UV-2600 in a range of 200–1100 nm. Surface chemical properties were examined on x-ray Photoelectron Spectroscopy (AXIS Ultra DLD, Shimadzu). The OSL properties were investigated using Lexsyg Smart OSL/TL reader for beta irradiation. The ⁸⁷Sr source was used for beta irradiation in the range of 0–80 kGy with a dose rate of 100 mGy/s.

3. Results and discussion

3.1. Physicochemical properties of Al2O3:C polycrystal

An average particle size of alumina powder (DLS technique) from Beijing Dingsheng Special Ceramics Manufacture Co, Ltd. was approximately 295 nm and became considerably larger after annealing. The average particle size of alumina powder (99.7%, Beijing Dingsheng Special Ceramics Manufacture Co, Ltd.) was approximately 295 nm and became considerably larger after annealing. The average particle size of alumina powder (99.7%, Beijing Dingsheng Special Ceramics Manufacture Co, Ltd.) was approximately 295 nm and became considerably larger after annealing. The average particle size of alumina powder (99.7%, Beijing Dingsheng Special Ceramics Manufacture Co, Ltd.) was approximately 295 nm and became considerably larger after annealing.

X-ray diffraction analysis was used to monitor the phase transformation of Al2O3. For Al2O3:C prepared by alumina powder (Al series), figure 3(a) shows the XRD pattern of UAl before annealing that exhibited the mixture of theta (θ) and alpha (α) Al2O3 phases. The characteristic peaks of θ-Al2O3 were interpreted as ICDD-PDF 00–010–0173, while those of α-Al2O3 were ICDD-PDF 00–042–1468. After annealing for 2 h, the θ-Al2O3 completely transformed to the α-Al2O3, as shown in figure 3(b). Notably, the larger crystallite sizes of Al2O3 were observed after annealing as the peak width was significantly narrower. After carbon doping, the XRD pattern of 01Al (figure 3(c) and 1Al (figure 3(d)) was similar to the UAl (figure 3(b)).

Furthermore, the XPS technique was used to observe the chemical composition and also the chemical environment within the obtaining samples. For the Al series, figure 4 shows the high-resolution narrow scan of Al2p, O1s, and C1s for the Al series. The Al2p line of UAl represents the two deconvoluted spectra at 74.00 and 75.46 eV, which are assigned to the major phase of the α-Al2O3 and the trace remaining of the θ-Al2O3, respectively [25]. For 01Al and 1Al, there is no evidence of θ-Al2O3, and the α-Al2O3 peak is at ∼74.2 eV. Notably, the binding energy (BE) of Al-O (74.00 eV) shifts to higher BE after carbon doping, indicating the formation of oxygen vacancies (Ov). From the O1s deconvolution spectra (figure 4), it can be classified as lattice...
oxygen (LO) and none lattice oxygen (NLO). The NLO peak area of UAl, 01Al and 1Al were 11.4, 19.8, and 22.7 %, respectively. This increase in the NLO species after carbon doping is in good agreement with the Al2p shifted to higher BE. The amount of NLO was related to oxygen defects in oxide materials [26]. Therefore, it implies that higher carbon doping concentrations increase the oxygen-vacancies formation. For the C1s signals of all three samples, the prominent peak was located at 284.7–285.2 eV, as shown in figure 4. Notably, 01Al and 1Al exhibit the peak at ∼284 eV resulting from the sp2 carbon dopants. In contrast, there is no evidence of this carbon species in the undoped Al2O3. The other peaks at the higher BE can be assigned to C-O (∼286 eV), C=O (∼287 eV), and O−C=O (∼289 eV), respectively.

For the B series, figure 5(a) shows the XRD pattern of as-synthesized boehmite (AlOOH). The major phase is boehmite (ICDD-PDF 01–083–1505) with a trace amount of ammonium alunite (NH4Al3(SO4)2(OH)6)
After 2 h, UB presents the mixed phases of $\theta$-$\text{Al}_2\text{O}_3$ and $\alpha$-$\text{Al}_2\text{O}_3$, as shown in figure 5(b). Most of the $\theta$-$\text{Al}_2\text{O}_3$ transformed to $\alpha$-$\text{Al}_2\text{O}_3$ after 4 h. However, the trace amount of $\theta$-$\text{Al}_2\text{O}_3$ exists in 01B (figure 5(d)) and 1B (figure 5(e)) despite 4 h of annealing. Notably, the intensity of the $\theta$-$\text{Al}_2\text{O}_3$ phases increased as the carbon dopants increased.

The chemical functional group of as-synthesized boehmite precursor was investigated by FTIR. Figure 6(a) presents the IR spectra of boehmite precursors. The transmission bands at 3361 and 3273 cm$^{-1}$ can be assigned to the (Al)O-H asymmetric and symmetric stretching, respectively [13, 27]. The bands at 2922 and 2848 cm$^{-1}$ can be described as the N-H stretching from the occurrence of ammonium ion. The intense bands at 1648 and 1590 cm$^{-1}$ can be assigned to the O-H bending followed by N-H bending at 1468 and 1415 cm$^{-1}$. The SO$_4$$^2-$ vibrational bands show at 1315 and 1261 cm$^{-1}$. The bands at 1124 and 1018 cm$^{-1}$ can be assigned to the (Al)O-H bending, while the bands at 860 and 701 cm$^{-1}$ can be assigned to the vibrational mode of AlO$_6$. Moreover,
figures (b) and (c) show that after 2 and 4 h of annealing, UB consists of the N-H and O-H vibration signals. This result implies that there is a trace amount of ammonium alunite in UB. The morphology of the B series was also investigated by SEM. Figures 2(d)–(f) presents the SEM images of UB, 01B, and 1B. All samples displayed flower-like morphologies with particle sizes of 2–3 μm. This morphology evolution could originate from the combination of nanoflakes that interacted with one another via the O–H–O hydrogen bonding [13]. The flower-like α-Al2O3:C composite was synthesized via a hydrothermal method using sucrose, urea, and aluminum sulfate as reactants. Herein, sucrose plays a critical role in the formation of flower-like structures as the carbon dopant. The well-defined α-Al2O3:C can be obtained at 120 °C for 2 h. The formation process of the flower-like α-Al2O3:C crystal was proposed in Scheme 1. During the first hydrothermal state, aluminum sulfate reacts with urea solution to form Al(OH)3. A relatively strong electrostatic attraction between SO42− and Al3+ prevents the Al3+ and OH− from nucleating too fast [12]. Therefore, the crystallization process is relatively stable. After that, dehydration and aromatization of sucrose occur and convert Al(OH)3 to AlOOH@C. In this part, hydrogen bonds are formed between the carboxyl group of carbonaceous layers and the hydroxyl ions of AlOOH, restraining further nucleation and growth for controlling the flower-like structure [22]. Finally, at the annealing condition at 1200 °C for 2–4 h, the AlOOH@C precursor transforms to achieve the phase transition to the flower-like α-Al2O3:C [28].

Figure 6. FT-IR spectra of (a) boehmite precursor, UB after (b) 2-hour annealing and (c) 4-hour annealing.

Scheme 1. The formation of flower-like α-Al2O3:C polycrystal [28].
Figure 7 shows the XPS of α-Al2O3:C prepared from the boehmite precursor (B series). The fitted Al2p peaks of UB locate at 73.00 and 74.00 eV, which can be ascribed to Al-Al and α-Al2O3, respectively. After carbon doping, there is a devolution peak at ∼76.00 eV, indicating the formation of the θ-Al2O3. Hence, the α-Al2O3 was the majority phase mixed with a small amount of the θ-Al2O3. The oxygen species, as shown in figure 7, consisted of NLO and LO. The amount of NLO of UB, 01B, and 1B samples are 16%, 12%, and 41%, respectively. Similarly, the BE of α-Al2O3 shifts approximated 0.5 eV (1B) and only 0.02 eV (01B) to the higher BE. This behavior confirms an increase in oxygen vacancies after carbon doping. From fitted C1s, a signal of C=C (∼284.0 eV) only appeared in UB. However, after carbon doping, no evidence of C=C and Al-Al existed. It implies that carbon dopants are oxidized as a higher amount of C=O (∼286 eV), O–C=O (∼287 eV), and CO2 (∼289 eV) in the 01B and 1B.

3.2. Optical properties
The reported experimental band gap value for the α-Al2O3 is 8.8–9.0 eV. For γ-Al2O3 is 7.0–8.7 eV, and amorphous Al2O3 is 5.1–7.1 eV [7, 9, 23, 29–31]. The influential factors to the bandgap are synthesis methods, defects, and impurities [7, 9, 23, 29–31]. The Kubelka-Munk (K-M) plot determined the energy bands, which can be calculated by equation (1). Where the $F(R_{\infty})$ is the K-M, and the $R_{\infty}$ is the diffuse reflectance (DR) of the sample [32].

$$F(R_{\infty}) = \frac{(1 - R_{\infty})^2}{2R_{\infty}}$$

The DR was measured in the range of 200–800 nm (2–6 eV). The band energies were estimated from the intercept of the tangent. Figure 8(a) shows the K-M plots of UAl, 01Al, and 1Al. Two distinguish bands (4.80 and 5.00 eV) were observed in the UAl, which was significantly larger than 01Al and 1Al. The 01Al (3.57 eV) energy bands were located close to 1Al (3.62 eV). The results suggest that carbon doping affects the optical energy bands. It was reported that the oxygen vacancies were created due to carbon doping which acted as F centers [3–5]. Also, they are the essential ingredients for high luminescence sensitivity [3–5]. Figure 8(b) shows the K-M plot of UB, 01B, and 1B. The energy band are located close to 1Al (3.62 eV). The results suggest that carbon doping affects the optical energy bands. It was reported that the oxygen vacancies were created due to carbon doping which acted as F centers [3–5]. Also, they are the essential ingredients for high luminescence sensitivity [3–5]. Figure 8(c) shows the K-M plot of UB, 01B, and 1B. The energy band are located close to 1Al (3.62 eV). The results suggest that carbon doping affects the optical energy bands. It was reported that the oxygen vacancies were created due to carbon doping which acted as F centers [3–5]. Also, they are the essential ingredients for high luminescence sensitivity [3–5].

Figure 7. High resolution XPS narrow scan (solid blue line) and fitted spectra (solid black line) of (a) Al2p, (b) O1s and (c) C1s for UB, 01B, and 1B, respectively.
3.3. OSL properties

The OSL dose-response of the Al and B series was obtained by Lexsyng Smart reader. The samples were irradiated by beta particles in the dose range of 0 to 80 kGy (dose rate of 0.1 Gy/s). Consequently, an electron/hole are generated. Electrons move to trap states, as shown in figure 1(a). The trapped electrons are stimulated by blue LED (458 nm) and emit OSL signals after recombination at F or \( F^+ \)-centers in this experiment.

Figure 9 presents continuous wave OSL (CW-OSL) curves (\(^{90}\)Sr beta irradiation) of Al and B series. The OSL signal decayed as an exponential function. The decay of sample irradiated at 80 kGy decayed significantly faster than that of 50, 30 and 10 kGy for Al and B series. Notably, B series decay rate was slower than that of Al series and had a considerable higher initial OSL signal. Yukihara \textit{et al.} reported the OSL properties of commercial OSL crystal Al\(_2\)O\(_3\):C (Landauer Inc, USA) \[34\]. The OSL decay rate of this crystal increased with beta dose in the range of 11.2–1120 Gy \[34\], which is in good agreement with our results. The beta dose-response of commercial OSL crystal was linearly up to 100 Gy and exhibited supralinear after that \[34\].

The beta dose-response curves of Al and B series were plotted in the range of 0–80 kGy. The data were fitted with a linear function. The coefficient of determination (R\(^2\)) is used to identify the data variation and the slope of the curves indicate the OSL sensitivity of samples to beta radiation. Figure 10(a) shows the OSL dose-response curves of UAl, 01Al, and 1Al. UAl shows the lowest sensitivity with a slope of 2.4362, and the R\(^2\) is 0.9785. As the presence of carbon dopants, 01Al exhibits the most excellent sensitivity with the slope of 7.6374 among 1Al and UAl and the linearity with the R\(^2\) of 0.9932. The possible explanation would be that carbon dopants could substitute oxygen atoms in the Al\(_2\)O\(_3\) crystal, leading to an increase of \( F^+ \)-center and OSL signal. The result is in good agreement with the XPS results that 01Al and 1Al had a more significant amount of NLO than UAl. The NLO are related to the number of oxygen vacancies, acting as the electron trapping states. Therefore, the 01Al response is more sensitive to beta particles than...
UAl. However, an excess of carbon dopants may destroy the crystal structure in that the saturated carbon has dissolved in Al2O3 crystals resulting in the drops of OSL signal [5, 6].

For the B series, figure 10(b) shows the OSL dose-response curves of UB, 01B, and 1B. Interestingly, the OSL sensitivities of the B series were around 50 times higher than that of the Al series. Notably, UB represents the best response to beta radiation, showing the most significant slope of 363.16 (figure 10(b)). However, the R2 of the B series’ dose-response (0.94–0.97) curve slightly deviates from one compared with the A series (0.93–0.99). From the dose-response results, Al and B series have an advantage over the commercials OSL crystal in that they exhibited the linear beta dose-response at high dose (10 kGy- 80 kGy), whereas the commercial OSL crystal deviated from the linear response at > 100 Gy [4, 34]. Therefore, it would suggest that the flower-like structure and particulate powder of Al2O3:C polycrystal could use in high dose dosimeter applications.

The OSL results suggest that the Al2O3 morphologies have a substantial effect on the OSL signal and beta dose-response. Electron and hole mobilities were reported that crucially depend on crystallite sizes and morphologies [9]. The possible explanation for the excellent OSL behaviors of the B series would be the resemblance of the UB trapping state to the commercial Al2O3:C detector as shown in the optical results (figures 8(a)–(c)). Also, the flower-like Al2O3:C polycrystal (B series) had larger surface areas and crystallite size than Al2O3:C particles (Al series). Furthermore, the suitable amount of oxygen vacancies can be another factor that impacted the OSL sensitivity. Although this finding confirmed that the flower-like Al2O3 polycrystal has...
excellent OSL sensitivity for beta particles at high doses, other radiation responses (gamma and x-ray), fading, and temperature dependence need to be further studied.

4. Conclusions

The OSL efficiency of two \(\alpha\)-Al\(_2\)O\(_3\):C morphologies (the flower-like structure and particulate powder) has been investigated. The carbon dopants (0.1 and 1 wt%) affect the amount of generated oxygen vacancies, confirmed by XPS. The linear response to beta particles, in the 0–80 kGy, presents in both morphologies. However, the flower-like \(\alpha\)-Al\(_2\)O\(_3\):C polycrystal shows approximately fifty times higher in OSL sensitivities than that of \(\alpha\)-Al\(_2\)O\(_3\):C particulate powder. The undoped \(\alpha\)-Al\(_2\)O\(_3\):C flower-like polycrystal exhibits the best OSL sensitivity due to the suitable amount of oxygen vacancies, similar band energy to commercial OSL, and morphology effects. The flower-like \(\alpha\)-Al\(_2\)O\(_3\) polycrystal could be a promising crystal detector for the OSL personal dosimeter.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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