Microstructure, luminescence, and dielectric properties of microwave-sintered Ce:LuAG nano-ceramics

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\textbf{ABSTRACT}

Herein, phase pure and highly crystalline Ce:LuAG nano-ceramics were fabricated using a novel, ultra-fast microwave sintering approach. The influence of microwave sintering on the microstructural, photoluminescence, and dielectric characteristics of Ce:LuAG nano-ceramic powders was examined. Microwave-assisted sintering of Ce:LuAG nano-ceramic powders yielded high crystallinity, low lattice strain, and reduced grain size. The process also improved the sintering kinetics and enhanced the surface diffusion between the grains, resulting in enhanced luminescence and dielectric properties. The Cole-Cole impedance plots showed single semicircular arcs, indicating non-Debye relaxation and a high dielectric constant in the microwave-sintered Ce:LuAG nano-ceramic and highlighting its potential for use in optoelectronics.

1. Introduction

Recently, multi-functional polycrystalline ceramics have been evaluated as potential frameworks and alternatives to metals and alloys in materials science \cite{1,2}. Inimitable properties including hardness, toughness, thermal stability, and abrasiveness make polycrystalline ceramics excellent for a wide range of applications \cite{3}. Especially, polycrystalline nano-ceramic materials with good heat transport phenomenon, optical firmness, and mechanical strength have potential applications in microelectronics and optoelectronics \cite{4}, including in dielectrics, piezoelectric materials, magneto-resistive, and superconductors \cite{5}. Particularly, lanthanide-doped polycrystalline garnet-based nano-ceramics (A\textsubscript{3}B\textsubscript{5}O\textsubscript{12}) have attracted remarkable interest in the field of optoelectronics due to their distinctive structural and luminescent properties. Rare-earth ions (Nd\textsuperscript{3+}, Ce\textsuperscript{3+}, P\textsuperscript{3+}, and Yb\textsuperscript{3+}) doped into LuAG and YAG superlative ceramic matrices are key materials for solid-state lasers, inorganic scintillators, and luminescent photonic systems \cite{6,7}. The exceptional lasing, scintillation, and luminescence mechanisms arise from the isotropic cubic structure, low birefringence effect, high density, creep resistance, and high chemical-radiation stability of these materials.

It was also reported that the dielectric ceramics exhibit good quality factors with low dielectric constants for microwave electronic devices such as optical resonators, filters, optical waveguides, and duplexers \cite{9}. Rare-earth-doped LuAG is a promising alternative to ferrite-based nano-ceramics for producing high dielectric performance and low energy dissipation super-capacitors, among other energy storage device applications \cite{10}. In recent literature, the dielectric properties of trivalent rare-earth-doped LuAG-based nano-ceramics have been scarcely reported. Therefore, this study aims to investigate the optical and dielectric properties of Ce:LuAG nano-ceramics for various multi-functional optoelectronic applications.

Traditionally, materials science researchers have used solid-state synthesis routes to prepare Ce:LuAG nano-ceramic powders. However, novel precipitation-assisted hydrothermal approach was adopted herein to synthesize Ce:LuAG nano-ceramic powders. This approach facilitates in homogeneity of a molecular precursor with controlled stoichiometry and good structural crystallinity to enhance the resulting optical luminescence and dielectric behaviors. In addition, the optical and dielectric behaviors of the as-prepared nano-ceramic powders depend on the preparation conditions and type of sintering approach. It has been reported that decreased integrated microwave sintering temperature and time can prevent abnormal grain-growth and defect center formation, which in turn, enhances the intrinsic properties of the...
fabricated materials [11]. Therefore, a novel microwave sintering technique was developed herein with significant modifications to the crystallographic textures and microstructure of the resulting materials with improved performance. The novel method offers rapid heating and cooling rates and shorter synthesis duration (activated by plasma) which, in turn, increases the driving force between the particle surfaces and induces grain boundary diffusion [12]. Ionic transport occurs across the grain boundaries which improves the electric conductivity through the spatial distribution of Ce$^{3+}$. Hence, the influence of conventional and microwave sintering approaches on the phase formation and microstructural modifications and their effects on optical luminescence and dielectric properties of Ce:LuAG nano-ceramics for the optoelectronic application was investigated herein.

2. Synthesis and experimental details

2.1. Materials and methods

High-purity chemicals were purchased from Sigma-Aldrich (Korea) and used as precursors for the synthesis of Ce:LuAG nano-ceramic powders. Lu(NO$_3$)$_3$·6H$_2$O and Al(NO$_3$)$_3$·9H$_2$O precursors (3:5) were dissolved in 50 mL of deionized water and stirred for 15 min. Subsequently, 0.05 mol% of Ce(NO$_3$)$_3$·9H$_2$O was added as a dopant to maintain the stoichiometry (Ce$_{0.05}$Lu$_{2.95}$Al$_5$O$_{12}$). The solution was precipitated by adding AHF (0.8 M) in a reflux-condenser setup and heated at 90 °C for 6 h to initiate nucleation. The precipitant suspension was sonicated for 30 min and transferred to a Teflon sealed autoclave reactor and heated at 180 °C for 12 h. The white precipitates from the reactor and heated at 180 °C for 12 h. The white precipitates from the autoclave were dried at 60 °C for 24 h and finely ground for 10 min. The calcined Ce:LuAG powders were divided into two equal batches and uniaxially pressed (15T Hydraulic press, MTI Korea) into two pellets (8 mm × 1.5 mm). The second pellet was heated at 1100 °C for 4 h, and the first pellet was conventionally sintered (Mu$\nu$al, Korea) at 1100 °C for a shorter duration of 10 min. The second pellet was subjected to microwave sintering (CEM Phoenix Microwave Furnace, Korea) at 1100 °C for 10 min.

3. Results and discussion

3.1. Structural analysis

Fig. 1a shows the XRD patterns (X-ray diffractometer with Cu Kα radiation, Bruker, D8 ADVANCE, US) of the CS and MW sintered Ce:LuAG nano-ceramic powders prepared via the modified hydrothermal method. Trivalent Ce ions were incorporated into LuAG matrix, substituting Lu$^{3+}$ at the dodecahedral site of the garnet lattice ($D_2$ symmetry) [13]. The cubic phase was obtained at the sintering temperature of 1100 °C without Lu$_4$Al$_{12}$O$_{35}$/Lu$_2$Al$_{2}$O$_7$ phases. Standard powder cell refinement software was used to determine the crystallographic parameters of the CS and MW sintered Ce:LuAG nano-ceramic powders. The experimental XRD peaks were in agreement with the standard JCPDS card No: 73-1368 [13]. The MW sintered sample showed higher crystallinity than CS sample due to the higher in situ temperature and reduced sintering time made possible due to MW heating. The effect of conventional and microwave sintering on the structure of the nano-ceramic powders was described and the parameters listed in Table 1.

<table>
<thead>
<tr>
<th>S. No</th>
<th>Parameters</th>
<th>Conventional Sintering</th>
<th>Microwave Sintering</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Lattice parameters (Å)</td>
<td>11.92(3)</td>
<td>11.91(2)</td>
</tr>
<tr>
<td>2</td>
<td>Cell Volume (a$^3$)</td>
<td>1692</td>
<td>1686</td>
</tr>
<tr>
<td>3</td>
<td>Crystallite size (nm)</td>
<td>54</td>
<td>34</td>
</tr>
<tr>
<td>4</td>
<td>Lattice strain (ε)</td>
<td>0.112</td>
<td>0.078</td>
</tr>
</tbody>
</table>

The unit cell lattice parameter values reported in Table 1 were greater than the theoretical value of the un-doped LuAG ($a = 11.90$ Å, JCPDS No: 73-1368) due to the ionic radii discrepancy between Lu$^{3+}$ (0.84 Å) and Ce$^{3+}$ (1.03 Å) at the dodecahedral site [13,14]. The lattice parameter of the MW sintered Ce:LuAG nano-ceramic powder was slightly lower compared to the conventionally sintered Ce:LuAG, as indicated by the higher angle peak shifting of the (420) diffraction plane (Fig. 1b). The peak shift was induced by the lattice strain in the sample. The lattice strain (ε) was calculated from the Williamson-Hall (W–H) plot, which is shown in the inset of Fig. 1b. The lattice strains of the CS and MW sintered Ce:LuAG nano-ceramic powders were 0.112 and 0.078, respectively (Table 1). The reduced strain observed in the MW sintered sample is likely due to the fast heating and cooling phase, which reduced the sintering duration and thermal stress accumulated in the sample. The crystallite sizes (D) of the CS and MW sintered Ce:LuAG nano-ceramic powders were determined using Scherer’s formula as 65 and 37 nm, respectively. The FWHM for the (420) diffraction plane of the MW sintered sample was higher than that of the CS sample (Fig. 1b), confirming the reduced crystallite size. Hence, it could be inferred that the crystallite size and lattice strain can be effectively reduced with enhanced crystallinity via microwave sintering. This in turn has the potential to improve the luminescence of Ce:LuAG nano-ceramics for various light-emitting applications.
3.2. Microstructure analysis

Fig. 2a and b shows the FE-SEM surface morphology (Zeiss Sigma, FESEM, US) of the CS and MW sintered Ce:LuAG nano-ceramic particles synthesized using the hydrothermal method. Homogeneous and less-agglomerative nanoparticles were obtained with grain sizes of ~172 and ~80 nm measured from particle size distribution plot shown in Fig. 2c and d, respectively. The rapid heating considerably restricted the grain growth of the nanopowders during microwave sintering, leading to the reduced crystallite size, as indicated by the XRD results. The presence of Ce, Lu, Al, and O was confirmed by X-ray photoelectron spectroscopy analysis (Supporting Fig. S1). The binding energy peaks at 193.9, 72.3, 531, and 893.7 eV were respectively assigned to the Lu4d, Al2p, O1s, and Ce3d orbitals. The surface morphology of the conventionally sintered Ce:LuAG nanoparticles adopted an oblate shape compared to the microwave sintered sample. Because of the prolonged sintering duration, the nanoparticles exhibited inter-particulate necking due to surface diffusion in the direction of the higher heat flux, leading to an oblate spheroidal morphology [15]. For further insight into the effect of microwave sintering on the microstructural properties, the nano-ceramic powders were pelletized and examined by FE-SEM.

Fig. 3a and b shows the surface morphology of the CS and MW sintered Ce:LuAG nano-ceramic pellet samples. Abnormal grain growth, residual pores, and microvoids were clearly observed on the outer surface of the CS sintered nano-ceramic sample. However, in the MW sintered sample the grains were properly densified without micropore or residual void formation on the grain boundary junction [16]. Ikesue et al. [7] reported that clear grain boundaries without pores could reduce light scattering and improve optical transparency for a variety of applications. Microwave sintering also induced lattice vibrations which can increase the atomic diffusion between adjacent grains. Moreover, the heat gradient at the nano-ceramic particle surface was increased, inducing plastic deformation. As a result, particles with low interfacial energy undergo surface diffusion along the direction of the driving force, decreasing the inter-particle distance [12], as shown in Fig. 4.

3.3. Photoluminescence studies

The consequence of the sintering environment on the resulting luminescence behavior of Ce:LuAG nano-ceramic powders was investigated by optical photoluminescence spectroscopy (Gary Eclipse Fluorescence Spectrophotometer, Agilent Technologies, US). The conventional and microwave sintered Ce:LuAG nano-ceramic powders exhibited two asymmetric excitation bands at 350 (UV-region) and 450 nm (visible blue region; Fig. 5). The intense excitation peak at 450 nm was assigned to the 2F5/2 → 5d1 transition. The broad excitation band in the UV-region (350 nm) was ascribed to the 2F5/2 → 5d2 transition of Ce3+ [13]. Fig. 5 also shows the photoluminescence emission spectra of the conventional and microwave sintered Ce:LuAG nano-ceramic samples excited at 450 nm. A broad luminescence band almost covering the entire visible region from 480 to 650 nm was observed with the maximum intensity centered at 507 nm. The primary emission mechanism in Ce:LuAG matrix can be attributed to the spin-orbit coupling between the ground 4F7/2, 4F5/2 states and excited 4D3/2, 4D5/2 states of Ce3+ [13, 14], respectively (Supporting Information Fig. S2). From the excited state, the electrons lose energy and relax to the lowest 5d state via non-radiative transition. After this process, the de-excitation of electrons to the ground state 4f occurs via radiative emission.
This radiative photon emission process resulted in greenish-yellow emission at 507 nm.

The emission intensity was higher in the microwave sintered nano-ceramic due to the improved crystallinity, reduced strain, and successful inclusion of Ce³⁺ in LuAG. The emission spectrum (Fig. 5, inset) was deconvoluted into two-component emissions (503 and 555 nm) by Gaussian fitting. These deconvoluted emission peaks showed that the Ce:LuAG phosphor can accommodate a broad spectral light regime, which can be tuned for various light-emitting diode applications. The CIE coordinate points of the CS and MW sintered Ce:LuAG nano-ceramics were extracted from the luminescence spectra using a CIE color chromaticity indexer (Fig. 5b). The (x, y) coordinate values of the CS and MW sintered nano-ceramics were (0.326, 0.576) and (0.372, 0.593), respectively. From the CIE color coordinate values, calculated color temperature (CCT) is also estimated [17] and the values are given in Table 2. The CCT and CIE results reveals a combination of blue, green and yellow emissions in the CS and MW sintered Ce:LuAG nano-ceramics. Hence, it is concluded that the fabricated nano-ceramic can be developed for luminescence tuning LED application. Based on the luminescence results, a decay lifetime experiment was conducted only for the strongly emitting microwave sintered Ce:LuAG nano-ceramics (Fig. 5c). The decay curve was fitted with two-exponential functions, showing decay lifetimes of 23 and 64 ns. The fast decay component arises from radiative luminescence recombination and the slower component from delayed non-radiative recombination of the Ce ions.

3.4. Impedance and dielectric studies

Impedance spectroscopy has been widely used to characterize the electrical behavior and interfacial charge transport kinetics associated with dielectric materials (1260A Impedance Gain-Phase Analyzer, Solartron Analytical, UK). It provides both resistive (real) and reactive
(imaginary) electric moduli for an applied potential over a wide frequency range from 1 MHz to 0.1 Hz. Fig. 6a shows the complex impedance plane (Cole-Cole) plots of the Ce:LuAG nano-ceramics which were post-heated via conventional sintering and microwave sintering. The prepared materials were pelletized and sandwiched between two platinum electrodes and the measurement was conducted to investigate the charge transport properties at an applied open circuit potential of 50 mV at RT. Single semicircle behavior was observed for both the CS and MW sintered samples.

 Generally, dielectric materials exhibit two semicircles in their Cole-Cole plots to distinguish the high and low-frequency grain boundary resistances. Herein, the single semicircle observed for both the CS and MW sintered samples confirmed the absence of grain boundary resistance, which has a negligible effect on the total charge transport resistance in nano-ceramics. The obtained single semicircle was fitted using an equivalent circuit model (inset of Fig. 6a) by connecting R and C in parallel and the semicircle arc interface on the real axis of the impedance provides series resistance (Rs) and grain resistance (Rg; summarized in Table 3) at the corresponding high and low-frequency regions. Here, Rs is the series resistance which contributes to the resistance of the platinum electrode and its interface with ceramic material. For the MW sintered sample, the Rs was low (374.3 Ω) compared to that of the CS sample (738.1 Ω). The poor interface of the CS ceramic on platinum electrode induced high contact resistance at the Ce:LuAG/Pt interface. The diameter of the semicircle of the MW sintered sample was smaller than that of the CS sample at an applied AC potential of 50 mV, indicated increased conducting behavior. The Rg (grain resistance) of the MW sintered sample was quite low (39.72 MΩ) compared to the CS material (455.8 MΩ). The rapid cooling effect in the MW sintered sample reduced the grain boundary contribution in the resulting nano-ceramic, leading to efficient charge transport between grain interfaces. In addition, reduced grain growth and interfacial surface diffusion between neighboring grains increased the charge carrier mobility and electron hopping rate between the grains. For the CS sample, microvoids and pores between the grains formed during the slow cooling process were responsible for the reduced electron hopping. Fig. 6b shows the bode phase plots of the CS and MW sintered samples, further confirming the non-Debye relaxation [18].

Fig. 7a and b shows the deviation of the real and imaginary parts of the impedance as a function of angular frequency. The magnitude of the real impedance was lower for the MW sintered sample compared to the CS sample at the low-frequency region, indicating increased conducting behavior. The Rs (grain resistance) of the MW sintered sample was quite low (39.72 MΩ) compared to the CS material (455.8 MΩ). The rapid cooling effect in the MW sintered sample reduced the grain boundary contribution in the resulting nano-ceramic, leading to efficient charge transport between grain interfaces. In addition, reduced grain growth and interfacial surface diffusion between neighboring grains increased the charge carrier mobility and electron hopping rate between the grains. For the CS sample, microvoids and pores between the grains formed during the slow cooling process were responsible for the reduced electron hopping. Fig. 6b shows the bode phase plots of the CS and MW sintered samples, further confirming the non-Debye relaxation [18].

Fig. 5. (a) Photoluminescence spectra of the CS and MW sintered Ce:LuAG nano-ceramic phosphor (b) Variations in CIE color coordinates and CCT of blue-green-yellow emitting Ce:LuAG nano-ceramics with respect to conventional and microwave sintering conditions (c) Decay lifetime of microwave sintered Ce:LuAG nano-ceramic phosphor. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

**Table 2**

<table>
<thead>
<tr>
<th>Sample</th>
<th>X</th>
<th>Y</th>
<th>CCT (K)</th>
<th>Emission color</th>
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<tr>
<td>CS</td>
<td>0.326</td>
<td>0.576</td>
<td>5623</td>
<td>Bluish Green</td>
</tr>
<tr>
<td>MW</td>
<td>0.372</td>
<td>0.593</td>
<td>4877</td>
<td>Yellowish Green</td>
</tr>
</tbody>
</table>

**Table 3**

<table>
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<tr>
<th>Sample</th>
<th>Rs (Ω)</th>
<th>Rg (MΩ)</th>
<th>C (x 10⁻¹¹ F)</th>
<th>χ² (x 10⁻⁴)</th>
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<tbody>
<tr>
<td>CS</td>
<td>738.1</td>
<td>455.8</td>
<td>5.328</td>
<td>1.77</td>
</tr>
<tr>
<td>MW</td>
<td>374.3</td>
<td>39.72</td>
<td>5.659</td>
<td>6.87</td>
</tr>
</tbody>
</table>

Fig. 6. (a) Cole-Cole plots of the CS and MW sintered nano-ceramics; (b) Bode phase plots.
CS sample shifted to a higher frequency for the MW sintered sample, indicating an increased hopping rate of localized charge carriers due to relaxation in the system.

To examine the polarization behavior, conducting properties, dielectric relaxation, $\varepsilon'$ (dielectric constant), and $\tan\delta$ (dielectric loss) plots were obtained (Fig. 7c and d). These parameters were calculated from the following equation using complex impedance plane plots [20],

$$
\varepsilon' = \frac{t}{\omega\varepsilon_0 Z_0^2} \frac{Z''}{Z''^2 + Z'^2}$$

$$
\varepsilon'' = \frac{t}{\omega\varepsilon_0 Z_0^2} \frac{Z'}{Z''^2 + Z'^2}$$

$$
tan\delta = \frac{\varepsilon''}{\varepsilon'}$$

where, $\varepsilon$ is the dielectric constant; $Z'$ and $Z''$ are the real and imaginary parts of impedance ($\Omega$), respectively; $A$ represents the area of the pelletized ceramic sample (50.24 mm$^2$); $t'$ is the sample thickness (1.5 mm); $\varepsilon_0$ denotes the permittivity of free space ($8.854 \times 10^{-12} \text{ m}^{-3} \text{ kg}^{-1} \text{ s}^4 \text{ A}^2$); $\omega$ is the angular frequency (Hz).

Fig. 7c and d show the dielectric constant and dielectric loss plot of the Ce:LuAG nanophosphors prepared under conventional and microwave sintering, respectively. Here, the dielectric constant is plotted against the angular frequency which exhibits a higher $\varepsilon'$ value for MW sintered sample compared to the CS sample. It should be noted that the $\varepsilon'$ value for both the CS and MW sintered samples decreased with increasing frequency, as is typical dielectric for garnet structures [21]. The di-electric constants ($\varepsilon'$) for the CS and MW sintered samples were 1369.35 and 6899.51, respectively, at 100 Hz. Thus, a 5-fold increase in the dielectric constant was observed for the MW sintered sample over that produced by the conventional sintering technique. The increased $\varepsilon'$ for the MW sintered sample was attributed to the interfacial surface diffusion between the grains which induced interfacial polarization as an additional contribution to the dielectric constant. The atoms at the surface interface of the grains were electrically active and the dipole moments changed with varying electric fields, especially at low frequencies [21]. The variation of dielectric loss with frequency is shown in Fig. 7d for the CS and MW sintered samples and the dielectric loss decreased with increasing frequency. Generally, $\tan\delta$ originates from the absorption current owing to the molecular transformation from one position to another, which involves dielectric loss. Herein, the formation of space charge polarization during the interfacial surface diffusion of grains absorbs the current and subsequently increases the dielectric loss in the MW sintered sample compared to that of the CS sample.

3.5. Thermal stability

Thermal stability parameters of the microwave sintered Ce:LuAG nano-ceramic are evaluated by performing temperature dependent luminescence experiment. An in-house temperature control system integrated cell with the spectrophotometer (power output 150 W, 500 V) is designed to investigate the thermal stability properties. The luminescence spectra are recorded at the excitation and emission wavelengths of 455 and 507 nm, respectively. The temperature dependent luminescence profile is recorded from 300 to 520 K, with the temperature measuring accuracy of ± 0.5 °C. The luminescence intensity of the microwave sintered Ce:LuAG nano-ceramic phosphor (Supporting Fig. S3) is observed to be stable at 425 K (93.7% of its original room temperature intensity). On increasing the integrated cell temperature, the emission intensity decreases drastically at 480 K and it shows 53.8% of its original intensity (Inset of Supporting Fig. S3). Such a strong decrease in the luminescence intensity with increase in the temperature is ascribed to a thermo-physical property called thermal quenching. In order to evaluate the thermal quenching behavior of the microwave sintered Ce:LuAG nano-ceramic, the integrated temperature dependent emission profile is plotted by Arrhenius equation [22].

$$
I_T = \frac{I_0}{1 + A_e(-E_d/kT)}
$$

Rearranging the equation in logarithmic form,
\[
\ln\left(\frac{I_i}{I_0}\right) - 1 = \ln C - \frac{E_a}{kT}
\]

where, \(I_i\) is the emission intensity monitored at different applied temperature \(T\), \(I_0\) is the initial emission intensity, \(k\) is the Boltzmann constant \((8.629 \times 10^{-5} \text{ eV K}^{-1})\), \(C\) is equation constant and \(E_a\) is the activation energy. The value of \(E_a\) is calculated from the linear slope of the logarithmic equation, \(\ln\left(\frac{I_i}{I_0}\right) - 1 \sim \frac{1}{T}\) (Supporting Fig. S4).

From the graph, the activation energy is calculated to be 0.265 eV, suggesting that the microwave sintered Ce:LuAG nano-ceramic phosphor can be employed as an active optical material for high power W-LEDs application.

4. Conclusion

In summary, Ce:LuAG nano-ceramic phosphors were synthesized using the hydrothermal method, and the effect of microwave and conventional sintering on crystallinity, morphology, PL, and impedance properties were extensively investigated. The microwave sintering significantly reduced the phase evolution duration, lattice strain, and crystallite size which enhanced the luminescence emission, with intense greenish-yellow luminescence observed at 507 nm. Formation of interfacial diffusion in the grains increased the electron hopping rate and enhanced the conductivity of the microwave sintered nanoceramics. A 5-fold increase in the dielectric constant was realized in the microwave sintered samples due to interfacial polarization during surface diffusion of the Ce:LuAG nano-ceramic grains.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ceramint.2020.07.186.

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