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Recent advances in transparent/translucent polycrystalline Sialon ceramics

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The transparent polycrystalline ceramics have acquired an interest in different fields such as transparent armor windows, cutting tools, IR windows, wear and scratch resistant parts (watches, bearings, and displays), and synthetic opals and rubies. In recent years, the transparent ceramics are applied in lasers and high power solid state lighting as a luminescent material. Different types of transparent polycrystalline ceramics were reported for different applications. However, there are few transparent ceramics that can withstand the aggressive environment. Recently, some studies on optical properties of Sialon ceramics show that the Sialon ceramics would be suitable for the optical applications. Sialon ceramics are known for their excellent mechanical and thermal properties and applied as a structural ceramics. The Sialon ceramics have all those potentials that can overcome the mechanical and thermal limitations of existing transparent ceramics where a very high grade of transparency is not required. The reported Sialon ceramics are IR transparent and visibly translucent. The translucent Sialon ceramics can be applied as a phosphor ceramic plate (PCP) for high power solid state lighting (SSL). However, the fabrication and production processes are costly due to the high temperature synthesis and complex processing techniques. In this short review, we will discuss the recent trend, processing techniques, properties, applications, and prospects of transparent Sialon ceramics.

Keywords: Sialon ceramics, sintering, physical properties, transparency, phosphors.

УДК: 535, 548

Последние достижения в области прозрачной/полупрозрачной поликристаллической сиалоновой керамики

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Интерес к прозрачной поликристаллической керамике появляется в различных областях, таких как прозрачные бронированные окна, режущие инструменты, инфракрасные окна, износостойкие и стойкие к царапинам детали (часы, подшипники и дисплеи), а также синтетические опалы и рубины. В последние годы прозрачная керамика применяется в лазерах и высокомощном твердотельном освещении в качестве люминесцентного материала. Различные типы прозрачной поликристаллической керамики были описаны для различных применений. Однако мало прозрачной керамики, способной противостоять агрессивной среде. В последнее время некоторые исследования оптических свойств сиалоновой керамики показывают, что сиалоновая керамика подходит для оптических применений. Сиалоновая керамика известна своими превосходными механическими и термическими свойствами и применяется в качестве конструкционной керамики. Сиалоновая керамика обладает всеми теми возможностями, которые могут преодолеть механические и термические ограничения существующих прозрачных керамик, для которых не требуется очень высокий уровень прозрачности. Сиалоновая керамика прозрачна для инфракрасного излучения и визуально прозрачна. Полупрозрачная сиалоновая керамика может применяться в качестве фосфорно-керамической пластины (ФКП) для мощного твердотельного освещения (ТТО). Однако процессы изготовления и производства являются дорогостоящими из-за высокотемпературного синтеза и сложных технологий обработки. В этом кратком обзоре мы обсудим последние тенденции, методы обработки, свойства, области применения и перспективы прозрачной сиалоновой керамики.

Ключевые слова: сиалоновая керамика, спекание, физические свойства, прозрачность, люминофоры.

1. Background

High performance materials are of demand in different sectors such as energy (electrical and electronics), environment, medicals (bio-implants), high temperatures (insulators and sensors), optical, and aerospace [1,2]. In the past, the ceramics were limited to white wares but recently, ceramics found applications in above mentioned fields due to their excellent thermal, mechanical, and optical properties. Traditionally, the glasses are known to be a good optical material and used extensively from household to advanced applications. However, new optical materials are required to fulfill the need of advancing technology where glasses are not applicable. Even though the single crystals were introduced as advanced optical materials, but their higher cost and complex production techniques made the material unaffordable. As compared to single crystals, the polycrystalline transparent ceramics are cheaper and show similar mechanical, chemical and thermal stability as well as can be produced in complex shapes and are not size limited. Polycrystalline ceramics having partial or total transparency had great value in different types of applications.

Recently, many types of transparent ceramics were developed and used commercially in different fields. Some of the known transparent ceramics are Al_2O_3, AlON, YAG, rare earth oxides (Re_2O_3), and Spinel ($MgAl_2O_4$) [2–5]. Most of these transparent ceramics are oxide and having ionic bonding with a cubic crystal structure. These transparent materials are becoming more and more important for applications in which materials are subject to extremely high mechanical and thermal stress. However, there is always a need for new optical materials with excellent thermal and mechanical properties. Sialon ceramics are least studied material in the field of optics despite their excellent mechanical and thermal properties.

2. Sialon ceramics

Si_3N_4 exists in two structural modifications: trigonal α-Si_3N_4 and hexagonal β-Si_3N_4, both of which are built up of $Si(N_4)$ tetrahedral with space groups $P3_1c$ and $P6_3/m$, respectively. In 1971, Oyama and Kamagaito discovered the solid solution in β-Si_3N_4 based on substitution of Al-O for Si-N in β-Si_3N_4 and named as β-Sialon [6]. Similarly, Jack et al. [7] in 1976 discovered the α-Sialon. α-Sialon and β-Sialon are the two common phases studied widely, which are isostructure to α-Si_3N_4 phase and β-Si_3N_4, respectively [7–8]. The partial substitution of Si and N with Al and O, respectively in β-Si_3N_4, gives β-Sialon structure. The insertion of metal cation in interstitial sites of α-Si_3N_4 structure gives α-Sialon with partial substitution of Si and N with Al and O, respectively having a formula of $M_x^{v+}Si_{12-m-n}Al_{m+n}O_nN_{16-n}$, where $x = m/v$ and M is one of the metal cations (Li^+, Mg^{++}, Ca^{++} and most of the lanthanide ions (Ln^{3+})) [8]. Similarly, the β-Sialon is represented as β-$Si_{6-z}Al_zO_zN_{8-z}$, where the value of z ranges from $0 \leq z \geq 4$. Even though the α-Sialon has more complex crystal structure and composition than β-Sialon, the α-Sialon shows more potential in the field of optics.

The α-Si_3N_4 structure is considered to be closely related to that of β-Si_3N_4, consisting of alternate basal layers of β-Si_3N_4 and a mirror image of β-Si_3N_4. As a result, there are two large interstitial sites at 2/3, 1/3, 3/8 and 1/3, 2/3, 7/8 in α-Si_3N_4. These sites can be occupied by metal cations having an ionic radius smaller than 0.1 nm. The value of m and n is taken to synthesize the α-Sialon ceramics according to the following formula [8]:

$$\frac{1}{3}(12-m-n)\mathrm{Si_3N_4} + \frac{1}{3}(4m+3)\mathrm{AlN} + \left(\frac{m}{2\nu}\right)\mathrm{Me_2O_\nu} + \\ + \frac{1}{6}(2n-m)\,\mathrm{Al_2O_3} \rightarrow \alpha-\mathrm{M}_{m/\nu}\mathrm{Si}_{12-m-n}\mathrm{Al}_{m+n}\mathrm{O}_n\mathrm{N}_{16-n},$$

where ν is the valency of M and electroneutrality requires $x = m/\nu$. Here, $x \leq 2$ and m(Al-N), n(Al-O) replace (m+n)(Si-N) in each unit cell of Si_3N_4.

The unit cell parameters change very small with the substitution of Al-O bonds for Si-N bonds in the β-Sialon structure, because the bond lengths of Si-N (1.74 Å) and Al-O (1.75 Å) are very similar; whereas the substitution of Al-N bonds for Si-N bonds in α-Sialon results in a considerable unit cell size expansion, due to the bond length difference between Al-N (1.87 Å) and Si-N (1.74 Å).

Sialon ceramics are sintered with sintering additives forming liquid phase at high temperature that allows densification through diffusion-solution-reprecipitation [8,9]. The precursor Si_3N_4 has covalent bonding and does not melt at high temperature but dissociates into Si and N. Therefore, to get fully densified Sialon ceramics, the metal oxide additives such as MgO, Y_2O_3, CaO, Ln_2O_3, and Al_2O_3 with common AlN are added and sintered at a high temperature above 1800°C. The problem associated with the liquid phase sintering is the formation of glassy phases at the grain boundaries. Therefore, careful composition design is made with considering the content of SiO_2 and Al_2O_3 on the surface of Si_3N_4 and AlN, respectively, to minimize the grain boundary phase.

Fig. 1 shows the prevailing trend for the fabrication process of Sialon ceramics. Generally, the powders are mixed by the conventional balling process. At first the wet ball milling is applied where powders are mixed in ethanol with high purity silicon nitride balls for 24 h, and then after the powders are dried in the oven for 12 h. The dried powders are dry milled with silicon nitride balls for 12 h. The hot press

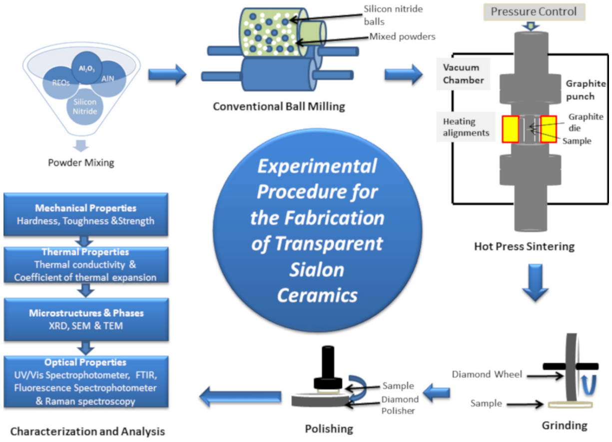


Fig. 1. (Color online) Schematic diagram showing the experimental procedure for the fabrication of transparent Sialon ceramics.

sintering technique is generally used to sinter the silicon nitride based ceramics. The h-BN is coated on the surface of the inner parts of graphite mold, die, and punch to occlude the carbon diffusion from the graphite. The high sintering temperature of 1700 –1950°C is used with a uniaxial pressure of 30 MPa in the presence of nitrogen gas [10 –12].

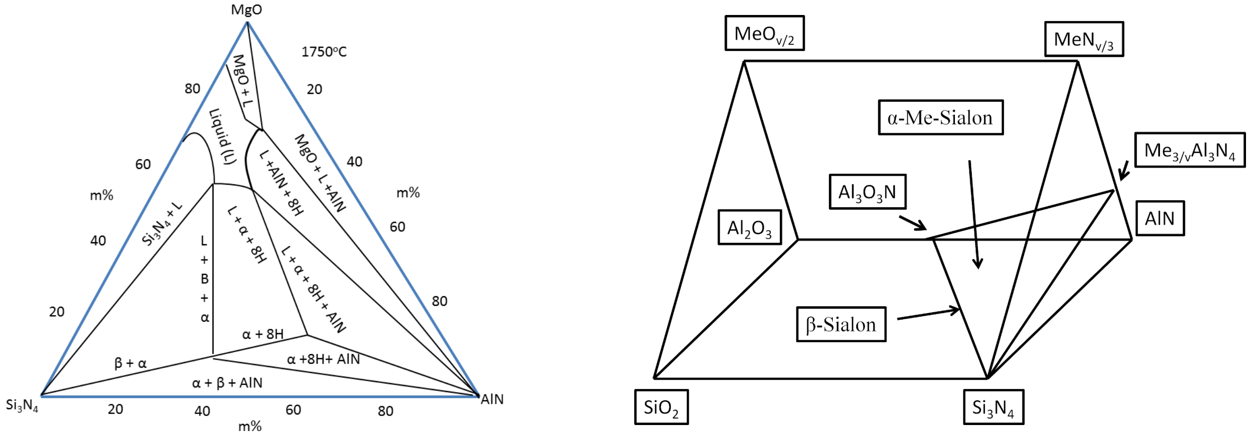
3. Transparent/translucent Sialon ceramics

In general, the transparent materials are those which can show undistorted clear image through it. However, in scientific definition, the material should exhibit high real inline transmission to be transparent [13]. Most of the transparent ceramics are cubic lattices and transparent regardless of their thickness. Being hexagonal crystal structure, the Sialon ceramics show birefringence and optical properties are not good as cubic crystal system ceramics. However, the optical properties can be enhanced by tailoring the grain size and structure in hexagonal systems, as reported for polycrystalline alumina [14]. The Sialon ceramics with high relative density and sub-micron grain size show higher transparency. High pressure sintering techniques can be applied to sinter the Sialon ceramics such as spark plasma sintering (SPS), hot press (HP), and hot isostatic pressing (HIP) to eliminate pores and to get higher relative density near to 100%.

In 1982, the first translucent β-Sialon was reported [15]. After that, in the early 2000s, some works were published on transparent/translucent polycrystalline α-Sialon ceramics, and most of them are listed with mechanical and optical properties in Table 1. Kuang et al. [16] reported the Sialon ceramics fabricated with the composition of Si_3N_4, MgO and AlN, excluding Al_2O_3. Fig. 2a shows the phase diagram of Sialon ceramics sintered around 1750°C showing stability region of α-Sialon and β-Sialon phases [16]. The translucent Mg-stabilized α/β-Sialon having a composition of 88 wt.% of α-Si_3N_4, 9 wt.% of AlN and 3 wt.% of MgO was reported [17]. The additive alumina can be excluded where the value of $m = 2n$. Surprisingly, the duplex α/β-Sialon also shows good transparency in the NIR region even with β-Sialon phase. The polyhedral α and β-Sialon grains with tight grain boundaries with less intergranular phase show transparency in the Sialon system. Altering the ratio of α to β phase can give the desired physical properties for different applications. The duplex α/β-Sialon ceramics show excellent hardness and fracture toughness [11,18]. Introducing the small amount of additives (Ln_2O_3) up to 1 wt.% significantly enhances the duplex α-Sialon phase as high as 97% by volume and transparency but the light transmission was decreased as compared to duplex α/β-Sialon [18]. The results show that the addition of Ln_2O_3 gives a higher glassy phase with thick grain boundaries, which eventually degrades the optical transmission. To achieve higher light transmission, a higher α-Sialon phase with thin grain boundaries and less glassy phase should be present. Joshi et al. [12] fabricated the IR transparent Sialon with the higher transmission of 78% in the NIR region.

The Sialon ceramics are generally fabricated with alumina with other metal oxides as shown in phase diagram in Fig. 2b [8]. In contrast to Mg-α/β-Sialon, the alumina and other stabilizing cations (Ln^{3+}) addition give a higher α-Sialon phase with higher transparency. Jones et al. [10] reported the transparency as high as 70% in the visible region with Lu^{3+} doped α-Sialon ceramics having a composition of $m = n = 1.1$. This system gives single phase α-Sialon and equiaxed grain morphology with tight grain boundaries. Most of the rare earth cations absorb the UV and visible lights, but Lu^3+ has no color center and only acts as a stabilizing cation in α-Sialon. Before this study, Karunaratne et al. [19] and Mandal et al. [9] reported the translucent Sialon ceramics, but the transmission of the materials were not reported. The translucent α-Sialon reported by Su et al. [20] had transparency of 65.2% in NIR and 58.5% in the visible region with 20 GPa hardness and 5.1 MPa. $m^{1/2}$ fracture toughness.

Liu et al. [21] compared the optical properties of Y-α-Sialon ($m = n = 1$) sintered with hot press sintering and spark plasma sintering (SPS), and found that hot press sintered sample shows high transmission than spark plasma sintered sample. The dark colored sample was obtained with SPS and may be due to some carbon diffusion. Also, the grain growth was observed in spark plasma sintered sample and concludes that small grains with tight grain boundaries can enhance the light transmission visible region.



a b

Fig. 2. Schematic phase diagrams showing the stability region for α-Sialon and β-Sialon. Ternary system without alumina [16] (a), and Janecke prism for Sialon system with alumina [8] (b).

4. Functional transparent/translucent Sialon ceramics

Functionalizing the transparent ceramics as a luminescent material can have broad applications in the field of scintillators, lasers, lighting, etc. due to their excellent mechanical and thermal properties as well as chemical stability [22]. The conventional phosphors for solid state lighting (SSL) are powder mixed with resin, and their stability is in question for a long run of the chip due to their poor thermal conductivity. Therefore, the dissipation of heat from the LED chip is important to retain the color and life of wLED [23]. Remote phosphors such as glass phosphors are also not adequate for high brightness SSL due to their poor mechanical and thermal properties. The timeline of phosphors and transparent phosphors with luminous efficacy is given in Fig. 3 [24–31].

To overcome the limitations of powder and glass phosphors, the transparent polycrystalline YAG: Ce phosphor ceramic plate (PCP) was reported [27]. However, the thermal conductivity is low to be applied in high brightness SSL. To enhance the thermal conductivity, Al_2O_3-YAG: Ce composite PCP was reported with high thermal conductivity of 18.5 $Wm^{-1}K^{-1}$ [29]. The Sialon PCP may overcome the above mentioned limitations that limit the other PCPs for extreme high brightness lighting. The α-Sialon ceramics has thermal conductivity around 12–19 $Wm^{-1}K^{-1}$, which makes the material appropriate for extreme high brightness lighting [11,32].

Although Sialon phosphor powder was reported decades ago [33], the reports on transparent/translucent Sialon PCP are few and new to the PCP field. In 2015, the first Sialon PCP for white lighting was reported [28]. The reported PCP was Mg stabilized α-Sialon: Eu^{2+} having wide excitation spectrum from UV to blue light, and broad emission from green to orange makes it suitable for white light application. The transparency was around 50% at 800 nm for 0.1 mm thickness. The main activator used was Eu^{2+}, having a

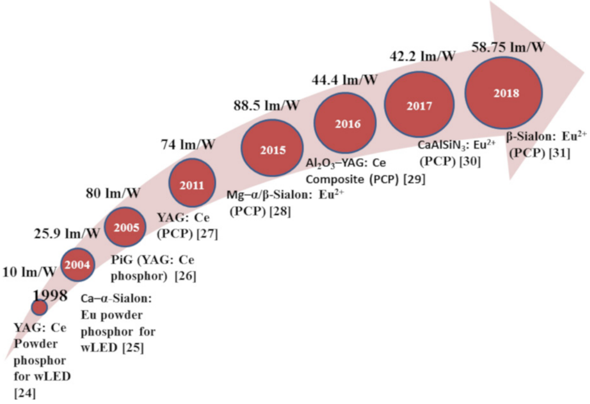


Fig. 3. Progress in lighting with blue light coupled yellow phosphor [24–31].

Table 1. Optical and mechanical properties of transparent/translucent Sialon ceramics.

Sialon Ceramics	Transmittance % (thickness) Vis = 800 nm NIR = 2500 nm	Excitation wavelength (nm)	Emission wavelength (nm)	Hardness (GPa)	Fracture toughness ($MPa.m^{1/2}$)	Ref.
β-Sialon	40% at 4500 nm (0.65 mm)					[15]
Y-α/β-Sialon	34% NIR (0.5 mm)			19.46	4.97	[34]
Lu-α-Sialon	72% Vis (0.5 mm)			19.27 ± 0.76	2.55 ± 0.11	[10]
Dy- α-Sialon	65% NIR (1.0 mm)			20.0	5.10	[20]
Dy, Y-α-Sialon	64% NIR (0.5 mm)					[35]
Gd-α-Sialon	65% NIR (0.7 mm)					[36]
Mg-α/β-Sialon	66% NIR (0.5 mm)			21.4 ± 0.3	6.7 ± 0.1	[17]
(Y, Yb)-α-Sialon	72% NIR (1.0 mm)			20.5 ± 0.2	4.0 ± 0.3	[37]
(Sc, Lu)-α-Sialon	60% NIR (0.5 mm)			20.4	5.2	[38]
Li-α-Sialon	57% NIR (0.5 mm)			20.1 ± 0.2	3.0 ± 0.1	[39]
Mg, Y-α/β-Sialon	65% NIR (0.5 mm)					[40]
La, Mg-α/β-Sialon	47% NIR (0.5 mm)			20.2 ± 0. 2	4.8 ± 0.2	[41]
Mg-α/β-Sialon: Ba^{2+}	78% NIR (0.5 mm)			21.4	5.4	[12]
Mg-α/β-Sialon: Eu^{2+}	50% Vis (0.1 mm)	450	570	20.93 ± 0.71	5.12 ± 0.30	[28]
Mg-α/β-Sialon: Gd^{3+}	30% Vis (0.15 mm)	280	470	19.40 ± 0.23	5.14 ± 0.60	[42]
Mg-α/β-Sialon: Pr^{3+}	35% Vis (0.15 mm)	270	618	19.16 ± 0.58	5.20 ± 0.30	[42]
(Mg,Yb)-α-Sialon	35% Vis (0.15 mm)	460	550			[43]
Mg, Er-α-Sialon	40% Vis (0.1 mm)	980	550			[44]
(Er, Yb)-α-Sialon	60% Vis (0.2 mm)	980	550	20.45 ± 0.18	5.44 ± 0.25	[45]
Er-α-Sialon	30% Vis (0.8 mm)	980	1530			[46]
(Ho, Er)-α-Sialon	30% Vis (0.5 mm)	980	660	20.54	5.59	[47]
(Tm, Er)-α-Sialon	40% Vis (0.2 mm)	980	678 (Vis) 803 (NIR)			[48]
β-Sialon: Eu^{2+}	5% Vis (0.1 mm)	460	552			[31]
Gd-α-Sialon: Eu^{2+}	45% Vis (0.1 mm)	460	590			[31]
Gd-α-Sialon: Pr^{3+}	50% Vis (0.1 mm)	460	510, 640			[31]

4f-5d transition that gives broad emission centered at yellow region (570 nm) when excited by blue light (460 nm). The other lanthanide such as Yb^{2+}, Pr^{3+}, and Gd^{3+} doped Mg-α-Sialon with luminescence properties were also reported and listed in Table 1.

The translucent β-Sialon: Eu^{2+} PCP was also reported with greenish-yellow luminescence fabricated at high temperature (1900°C) [31]. The same composition as β-Sialon: Eu^{2+}, when sintered at low temperature (1700°C) shows orange luminescence with α/β composite phase as well as other unknown phases [31]. The large cationic size of Eu^{2+} cannot stabilize α-Sialon structure completely, and composite phases appeared at low temperature, and at a higher temperature, more stable β-Sialon phase was obtained. The co-doping of small cations (<0.1 nm) like Gd^{3+} along Eu^{2+} can well stabilize the α-Sialon structure, and dominant α-Sialon phase with higher transparency around 50% at 800 nm for 0.1 mm thickness was reported [31].

Transparency is one of the parameters for the phosphor plates, but total transparency is not necessary. The scattering centers in plate type phosphor would enhance the luminous efficacy according to Kim and Tang [49,50]. The scattering centers due to pores and secondary phase act as scattering centers, and these scattering centers help to absorb more incident light by the phosphor particles.

The upconversion luminescence properties in transparent Sialon ceramics were also reported and given in Table 1. Joshi et al. [44] reported the first upconversion luminescence for translucent (Mg, Er)-α-Sialon with green luminescence and IR downconversion luminescence at 1530 nm as excited by 980 nm laser. IR luminescence at 1530 nm is essential in the field of telecommunication for signal amplification. Later on, Bin et al. [45] reported the highly transparent (Er, Yb)-α-Sialon Sialon ceramics with 60% transparency at 800 nm for 0.2 mm thickness. In a later system, the MgO was excluded, and alumina was included to increase the content of sensitizer and activator to enhance the upconversion luminescence and also transparency. After that, more works were carried on upconversion luminescence with other lanthanides ($Ho^{3+}$and Tm^{3+}) [47,48]. The transparency in the NIR region was reported as high as 80% and shows NIR downconversion luminescence; therefore the transparent Sialon can be a suitable IR laser transparent ceramics [47]. The 2 μm wavelength range is called "eye-safe". Laser systems that operate in the "eye-safe" wavelength range have great market potential, especially in free space and medical applications where eye safety is very important. The Sialon ceramics are high temperature material and are stable at very high temperature, and this upconverting transparent Sialon ceramics can be used as optical temperature sensing.

5. Conclusions and future perspectives

So far, very few works are carried out on transparent Sialon ceramics regarding other polycrystalline transparent ceramics. The complex composition, tedious fabrication process, high temperature and pressure synthesis and not a cheap material regarding glass makes the material difficult to apply in general commercial applications. However, for advanced, high temperature, and harsh environment applications, the transparent Sialon ceramics can be a potential material to be studied further. For the optical properties, the α-Sialon phase is the crucial phase that has to be studied well concerning crystal structure, synthesis technology, stabilizing cations, grain boundaries, and glassy phases.

Recent advances in sintering technology and characterization tools offer efficient fabrication of transparent materials and understanding the material properties in-depth for further modifications in materials. Recently, the transparent nanoceramics with higher transparency almost 80% in the visible region were fabricated by a pressureless glass crystallization method using a CO_2 laser for heating the levitated samples [51,52]. This method is an alternative to time-consuming, expensive, and extreme pressure applied sintering methods. In the future, if transparent Sialon ceramics are fabricated with nano-size grains, the optical properties as well as mechanical properties can be enhanced.

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