ABSTRACT

Ceramic laser materials have come a long way since the first demonstration of lasing in 1964. Improvements in powder synthesis and ceramic sintering as well as novel ideas have led to notable achievements. These include the first Nd:YAG ceramic laser in 1995, breaking the 1 KW mark in 2002 and then the remarkable demonstration of more than 100 KW output power from a YAG ceramic laser system in 2009. Additional developments have included highly doped microchip lasers, ultrashort pulse lasers, novel materials such as sesquioxides, fluoride ceramic lasers, selenide ceramic lasers in the 2 to 3 µm region, composite ceramic lasers for better thermal management, and single crystal lasers derived from polycrystalline ceramics. This paper highlights some of these notable achievements.

Keywords: ceramics, laser materials, 100 KW, microchip lasers, ultrashort pulse, ceramic composites, non-oxide ceramics.

1. INTRODUCTION

Scaling solid state lasers to higher power requires crystalline materials with high thermal conductivity. Table 1 shows a list of cubic crystalline materials along with their thermal conductivities and other physical properties [1]. If we use YAG (Y₃Al₅O₁₂) as the benchmark, then there are several materials with higher thermal conductivities. While MgO and MgAl₂O₄ (spinel) have higher thermal conductivities, they cannot be easily doped with rare earth ions. On the other hand, the sesquioxides (Y₂O₃, Sc₂O₃, Lu₂O₃) can be doped with rare earth ions and furthermore, possess higher thermal conductivities than YAG, and certainly much higher than glass (Figure 1) [2]. While the thermal conductivity typically decreases with dopant concentration, Lu₂O₃ is the exception due to reduced phonon scattering (figure 1). This becomes a candidate material for lasers utilizing high dopant concentrations, such as thin disk or microchip lasers. Soules [1] has identified a thermal shock figure of merit, \( R_T \), where:

\[
R_T = \frac{1 - \nu}{\kappa K_{fr}} / aE
\]

and \( \nu \) is Poisson’s ratio, \( \kappa \) is the thermal conductivity, \( K_{fr} \) is the fracture toughness, \( a \) is the expansion coefficient and \( E \) is Young’s Modulus. Soules [1] suggested using the fracture toughness instead of the strength to eliminate the large variations that are typically observed for strength values. The last column in table 1 lists the values for \( R_T \) based on available data and highlights the superior performance of the sesquioxides (assumes similar performance for Sc₂O₃, Lu₂O₃ and Y₂O₃) and YAG compared to the other materials.

2. PREPARATION OF CERAMICS

While the sesquioxides and YAG have excellent properties, these crystals are difficult to grow in large sizes and with high dopant concentrations using traditional high temperature melting. This is attributed to a combination of problems including compositional variations, crucible interactions, phase transitions, poor rare earth solubility. All these limit size, complexity and yield. Fortunately, the ceramization process is a low temperature route for making transparent polycrystalline ceramics (figure 2). In this process, powder is converted into a fully dense and transparent polycrystalline...
ceramic material at approximately 65% of the melting point, thereby avoiding the high temperature issues associated with traditional crystal growing. The polycrystalline ceramic material looks like a single crystal, but consists of grains ranging in size from a few microns to hundreds of microns (depending on conditions) and grain boundaries that separate the grains. If the grains and grain boundaries are clean, free from pores and impurities, the ceramic can possess high transparency. This process enables higher rare earth doping, uniformity, scalability to large sizes and complex shapes, as well as making the material tougher and stronger. So, traditional limitations can be overcome with polycrystalline ceramics.

Table 1. Properties of some cubic single crystal materials, including laser glass (LG-750) (adapted from T. Soules [1]).

| Material          | \(\kappa\) (W/m K) | \(\alpha\) (ppm/ K) | \(E\) (GPa) | \(\nu\) | \(K_{lc}\) (MPa m\(^{1/2}\)) | \(R_T\) (10\(^{-20}\) m\(^2\)/W) |
|-------------------|---------------------|---------------------|-------------|-------|-------------------------------|--------------------------------|
| MgO               | 60                  | 11.9                | 294         | 0.186 | 2                             | 28                             |
| MgAl\(_2\)O\(_4\) | 19                  | 5.95                | 258         | 0.2   | 2                             | 20                             |
| Y\(_2\)O\(_3\)   | 13.6                | 7.4                 | 173         | 0.307 | 2                             | 14                             |
| Sc\(_2\)O\(_3\)  | 16.5                | 6.7                 | --          | --    | --                            | --                             |
| Lu\(_2\)O\(_3\)  | 12.5                | 5.5                 | --          | --    | --                            | --                             |
| Gd\(_2\)Ga\(_5\)O\(_12\) | 7.4            | 7.5                 | 255         | 0.28  | 1.2                           | 3.8                            |
| Gd\(_3\)Sc\(_2\)Ga\(_5\)O\(_12\) | 4.9           | 7.3                 | 210         | 0.28  | 1.2                           | 2.7                            |
| Y\(_3\)Al\(_5\)O\(_12\) | 10.8           | 6.1                 | 282         | 0.28  | 2.2                           | 9.9                            |
| Lu\(_3\)Al\(_5\)O\(_12\) | 8.3             | 6.0                 | --          | --    | --                            | --                             |
| CaF\(_2\)        | 9.2                 | 19.6                | 110         | 0.25  | 0.5                           | 1.6                            |
| SrF\(_2\)        | 9.9                 | 19.0                | 90          | 0.25  | --                            | --                             |
| BaF\(_2\)        | 12.1                | 21.1                | 53          | 0.343 | --                            | --                             |
| LG-750 glass      | 0.6                 | 13.2                | 50          | 0.26  | 0.45                          | 0.30                           |

Fig. 1 Thermal conductivity versus dopant concentration for crystals (adapted from R. Gaume 2002 [2]).
Figure 2. Ceramization process for converting powder into a transparent ceramic.

Figure 3 highlights two major approaches used to make transparent ceramic laser materials from powder, namely process A and B. Process A relies on hot pressing of the powder, usually in a graphite furnace, at pressures up to 5,000 psi at elevated temperatures. The product is usually 90 to 99+% dense and then further hot isostatically pressed (HIP) to full density and transparency at similar temperatures but using argon gas pressure of up to 30,000 psi to collapse any residual pores. The alternative process B, utilizes cold forming techniques such as cold isostatic pressing (or slip casting, tape casting, extrusion, etc) to make a green body usually with the addition of binders and surfactants. The green body density is typically in the range of 40-60%. Thereafter, the sample is heated in air to burn off the organics and then vacuum sintered at elevated temperatures (sometimes in the presence of hydrogen gas) to give a density of 90 to 99+%, followed by HIP to give full density and transparency. In theory it should be possible to provide full density and transparency at the end of the hot press or vacuum sintering steps in process A and B, respectively, but this is rarely done. Most groups in the literature use process B, although we will describe the use of process B, later in the paper, to make laser quality Yb:Lu₂O₃ ceramics.
3. PROPERTIES

Since a ceramic material is obviously different from a single crystal due to the presence of grains and grain boundaries, it is important to determine their impact on some specific properties. The three most important properties are optical scattering, mechanical strength and laser damage threshold. As we shall see, these properties are comparable to the single crystals, if not indeed better for the ceramics.

Quarles [3] measured the scattering loss of high quality Nd:YAG ceramic and demonstrated that the optical scattering was in fact lower than that obtained in the single crystal (figure 4). He attributed this to the high uniformity of the rare earth ions in the powder being maintained in the ceramics.

![Figure 4](image_url)  
Fig. 4 Demonstration of lower scattering loss in ceramic Nd:YAG (Quarles [3]).

More recently, Feldman et al. [4] observed that the strength of ceramic YAG was about 1.4x higher than for single crystal YAG. Similar results have been observed by others and are listed in table 2 and can be attributed to the increased fracture toughness of the ceramic [5,6,7].

| Reference         | Single crystal | Poly-crystalline | Poly- vs Single Crystal |
|-------------------|----------------|------------------|-------------------------|
| Feldman [4]       | 243            | 345              | 1.4x                    |
| Maziex et al [5]  | 236            | 306              | 1.3x                    |
| R. Gentilman [6]  | 252            | 378              | 1.5x                    |
| G. Quarles [7]    | 222            | 287              | 1.3x                    |

Table 2. Strength of ceramics compared with single crystals.

In ceramics, it is also well known that the strength is inversely related to the square of the grain size by the Hall-Petch equation; strength \( \alpha \frac{1}{\text{grain size}^{1/2}} \). Therefore, reducing the grain size will further increase the strength. Additionally, elimination of impurities and pores form the grain boundaries will improve strength and optical performance.

Ueda et al. [8] have shown that the laser damage threshold for both rare earth ion doped and undoped YAG ceramics are comparable to the single crystal, if not better (figure 5). So, it would appear that ceramics, if fabricated correctly, possess excellent optical and mechanical properties.
4. HISTORY OF CERAMIC LASER MATERIALS

While the physical and optical properties of ceramic YAG have improved so that it is now comparable, if not better, than single crystal YAG, the earlier ceramic lasers were of inferior quality and not necessarily made from YAG. The following highlights some key milestones in the development and demonstration of lasing using ceramic materials.

(i) 1964 – The 1st Ceramic Laser
Hatch et al. [9] were the first to demonstrate lasing in a ceramic, in this case Dy\(^{2+}\):CaF\(_2\). The ceramic was made by vacuum melting the tri-fluorides, grinding the product into a powder of 150 µm particle size, hot pressing the powder in vacuum and finally reducing the product to Dy\(^{2+}\) using 0.25 MeV x-rays. The ceramic product contained relatively large grains of 150 µm, implying no grain growth, and lased at liquid nitrogen temperature upon flash lamp pumping with a threshold of 24.6 J. CaO scattering centers were identified at the grain boundaries which contributed to 2% scattering loss in the visible and subsequently limited the laser performance.

(ii) 1973 – 2nd Ceramic Laser
It took about another 9 years for the second demonstration of lasing using a ceramic [10]. This was based on 1%Nd\(_2\)O\(_3\) doped Yttralox (10%ThO\(_2\)-89%Y\(_2\)O\(_3\)), whereby the ThO\(_2\) was used to control grain growth. Greskovich and Chernoch [10] synthesized submicron powder (\(<0.1\) µm) using co-precipitation of oxalates and then sintered the powders under hydrogen gas at 2170°C. The ceramic had large sized grains (130 µm) and high scattering loss of 5 to 7 cm\(^{-1}\) attributed primarily to index inhomogeneity since the pore volume was relatively low (1 ppm) and the pores were only 1 µm. Despite this, the flashlamp pumped ceramic lased with a slope efficiency of ~0.1%.

(iii) 1995 – 1st Ceramic YAG Laser
Ikesue et al. [11] were the first to demonstrate lasing in YAG ceramic doped with 1.1 atomic percent Nd. They synthesized pure, submicron oxide powders (Y\(_2\)O\(_3\) – 60 nm, Al\(_2\)O\(_3\) – 400 nm, and Nd\(_2\)O\(_3\) - 500 nm) with <100 ppm-wt impurity content and performed a thorough analysis of the densification dynamics via vacuum sintering. Several recommendations came out their work. Examples include the use of 320 ppm SiO\(_2\) sintering aid, ball milling with high purity alumina balls, spray drying the powders, and sintering at 1700°C to get full densification and complete conversion of the starting oxides into the YAG phase (Y\(_3\)Al\(_5\)O\(_{12}\)) via the intermediate Y\(_4\)Al\(_2\)O\(_9\) and YAlO\(_3\) phases. They also recommended rapid quenching to prevent impurity phase segregation at the grain boundaries. Their process is often called “Reactive Sintering” since they started with the individual oxides and converted them to YAG during sintering. The ceramic grain size was ~50 µm, with pores less than 5 µm in diameter, a total pore volume estimated at 200 ppm and measured scattering loss of 0.9%/cm. CW lasing was observed at 1.06 µm with a slope efficiency of 28% using diode...
pumping at 808 nm. The efficiency was similar to the value obtained for their single crystal sample. The ceramic also possessed very similar physical, mechanical and optical properties to the single crystal sample.

Fig. 6  Results of the first Nd:YAG ceramic laser (after [11]).

(iv) **2001 – Nd:YAG Ceramic Laser using Precipitated Powder**
J. Lu et al. [12] diode pumped a 1%Nd:YAG ceramic and demonstrated lasing at 1064 nm with an output power of 72 W and slope efficiency of 24.8%. This result was obtained using submicron 1%Nd:YAG powder that was synthesized by Konoshima Chemical Co. via a co-precipitation process followed by calcination, ball milling, slip casting and vacuum sintering. Their ceramic had a small grain size with 1 nm grain boundaries, a pore volume of only 1ppm and birefringence similar to a single crystal.

(v) **2002 – Breaking the 1 KW Output Power Barrier**
In 2002, a group led by Ueda, in collaboration with Toshiba and Konoshima Co., achieved a milestone by demonstrating an output power of 1.46 KW using a Nd:YAG ceramic [13]. The slope efficiency was 42% and only slightly lower than the 49% obtained for a single crystal (figure 7). The rod was 8 mm in diameter and 203 mm long. The high quality of the rod was attributed to improvements made in powder synthesis and sintering.

Fig. 7  Results for first Nd:YAG laser to break 1 KW output power (after [13]).

(vi) **Microchip Lasers**
J. Dong et al. [14] have studied heavily Yb-doped YAG ceramics. For example, they demonstrated a slope efficiency of 52% for 1-mm-thick YAG ceramic doped with 20 atomic percent ytterbium ions. Heavy-doped Yb:YAG ceramic is more suitable for a thin disk laser than a single-crystal with the same Yb³⁺-ion lasants. They have also improved upon this and demonstrated up to 61% efficiency. In another example, they demonstrated a decrease in efficiency with increasing Yb content, albeit a 10% doped sample had an efficiency of 85% (figure 8).
(vi) Ultrashort Pulse Lasers

Ikesue et al. [15] demonstrated one of the earliest mode locked lasers using a Nd:YSAG ceramic with a pulsewidth of 10 ps. The slope efficiency was 44.4% with 610 mW output power. More recently, Tokurakawa et al [16] demonstrated 68 fs pulses and 540 mW average power output using a Yb:Sc$_2$O$_3$ ceramic with approximately 1.8 at% Yb doping. They went on to demonstrate even shorter pulses of 53 fs by combining two ceramic laser materials in front of each other. In this case, a 1.5 mm thick Yb:Y$_2$O$_3$ ceramic containing 2.5 at% Yb was placed behind the Yb:Sc$_2$O$_3$ ceramic. Non-linear gain and spectral broadening (figure 9a) led to a pulsewidth of 53 fs (figure 9b) with ~1 W average power output.

![Figure 8](image)

**Fig. 8** Highly doped Yb:YAG ceramic lasers (after [14]).

(vii) Non-oxide Based Ceramic Lasers

Fluoride ceramic lasers have been demonstrated based on active color centers and rare earth ion doping [17,18]. Basiev et al. [17] converted LiF single crystals into a submicron-grained ceramic via hot pressing at 600°C. Subsequent irradiation with 21 MeV electrons produced F$_2$ color centers which exhibit a broad absorption in the 800 - 1000 nm region and also a broad emission spectrum in the 1000 - 1300 nm region. Diode pumping at 967 nm led to lasing at 1.117 µm with up to 26% slope efficiency and about 3 mW output power. This value was higher than the 18% obtained for the single crystal.

Basiev et al. [18] also demonstrated pulsed lasing in 5%-Yb:0.65CaF$_2$-0.35SrF$_2$ ceramics made by hot pressing single crystals. They noted that the fracture toughness was increased by 75%. The ceramics lased with about 45% slope efficiency which was comparable to the single crystal value of 50%. More than 1.5 W output power was obtained (figure 10).

![Figure 9](image)

**Fig. 9** Combining a 2.5%-Yb:Y$_2$O$_3$ ceramic behind a 1.8%-Yb:Sc$_2$O$_3$ ceramic in a laser cavity to demonstrate (a) spectral broadening from nonlinear gain and (b) pulsed lasing with 53 fs pulses (after [16]).
Laser has also been observed from chalcogenide ceramics [19,20]. For example, Gallian et al. [19] prepared ceramic Cr\textsuperscript{2+}:ZnSe using two techniques. One method utilized hot pressing mixtures of the powders (HPC), including 1% CrSe, and the other method involved thermally diffusing the CrSe into CVD ZnSe (CTD). The samples were pumped at 1.91 µm using a Raman shifted Nd:YAG pulsed laser. Both processes produced ceramics that lased at 2.4 µm, but the highest slope efficiencies of 10% were obtained for the thermally diffused samples compared with 5% for the hot pressed samples (figure 11a). In fact, this technology has matured reasonably fast such that a 15 W CW laser at 2.8 µm is now commercially available from IPG [20]. The Cr:ZnSe ceramic laser is tunable from 2.05 to 2.8 µm and pumped with an Er-silica fiber CW laser and (figure 11b).

(viii) Composite Ceramic Lasers
Ikesue et al. [21, 22] demonstrated the fabrication of laser quality composite ceramics. Initial samples consisted of 1%Nd:YAG ceramics attached to undoped YAG layers as shown in figure 12 [21], made using a layer by layer approach. The ceramic composites lased with similar efficiencies (~50%) highlighting the good quality of the samples.
Figure 13a shows the graded dopant profile of Nd ions in YAG samples stacked side by side before sintering. After sintering the stack fuses together and produces a smooth graded profile of the Nd ions that provides superior thermal management, thereby significantly reducing thermal gradients during laser operation (figure 13b) [22].

Messing [23] used a tape casting process to make a composite rod that lased with 25% slope efficiency and with almost 2 W output power (figure 14). The ceramics were made by vacuum sintering of cast and stacked tapes containing the oxide powders. Binder burnout was performed prior to sintering.
(ix) Single Crystal Lasers from Ceramics Lasers

Ikesue et al. [21] characterized the grain growth dynamics in YAG as a function of temperature and identified abnormal grain growth, also called exaggerated grain growth, above about 1730°C. Above this temperature, the grains can grow at very fast rates (up to 2 mm/hr). By placing a seed crystal in contact with the surface of a polished Nd:YAG ceramic, he was able to eliminate the grain boundaries, so that it appeared as though the crystal grew into the ceramic, without compromising the Nd uniformity. Basically, grain boundary migration was faster than diffusion. He went on to demonstrate this using higher doping levels of Nd (3.6 at%) (figure 15a). In one example, the laser slope efficiency increased after conversion to a single crystal (figure 15b).

5. HIGH POWER LASERS: TOWARDS 100 KW AND BEYOND

There have been some significant achievements that have led to the output power to increase from 1 KW and break the 100 KW mark. W. P. Latham et al. [24] reported 6.5 KW output power from a thin disk laser based on a 200 µm thick 9%-Yb:YAG ceramic active medium with a 1 mm thick undoped YAG cap to mitigate thermal loading (figure 16). Several similar thin disk lasers were combined to generate a total of >25 KW output [25].
Three other groups have developed high power slab based lasers as shown in figure 17 using ceramics from Konoshima Corp. For example, Lawrence Livermore Labs. [26] developed the heat capacity laser using Nd:YAG, Northrop Grumman Corp. [27] developed the end-pumped Yb:YAG slab laser, and Textron [28] developed the Thinzag (or zigzag) Nd:YAG laser.

The solid state heat capacity laser developed by Lawrence Livermore National Lab. (LLNL) used 0.3%-Nd:YAG slabs that were 10x10x2 cms. These were made by first slip casting two 1 cm thick slabs that were partially sintered, polished and then co-sintered together to increase the thickness to 2 cms. The plates were also co-sintered with 1 cm wide Sm:YAG strips around the edges to suppress ASE (figure 18). In 2006, they demonstrated lasing with 67 KW output power for < 1sec using diode pumping of 5 slabs. The slab quality was excellent, with average rms distortion at HeNe wavelength of $\lambda/30$ and surface rms ~0.5 nm.
Then in 2009 Northrop Grumman Corp. (NGC) demonstrated >100 KW output power [27], followed soon by Textron in 2010 [28]. The collective evolution of output power from YAG ceramics with time is shown in figure 19. This really highlights that the highest powers demonstrated to date originate from ceramics made by using co-precipitated powder at Konoshima Corp.

![Fig. 19 Evolution of laser output power versus year for YAG ceramics.](image)

### 6. SESQUIOXIDE LASERS AT NRL

Lu$_2$O$_3$ and the other sesquioxides (Sc$_2$O$_3$, Y$_2$O$_3$) possess higher thermal conductivities than YAG [29] which is an important property, especially for scaling to higher laser power [30]. Lu$_2$O$_3$ has a thermal conductivity that is predicted from fundamental principles to be almost insensitive to the Yb$^{3+}$ dopant concentration due to negligible phonon scattering, and measurements bear this out [29,31]. Consequently, Lu$_2$O$_3$ would be a desirable laser host material to investigate, especially at high rare earth dopant concentrations. Therefore, we have synthesized 10%-Yb$^{3+}$:Lu$_2$O$_3$ powder using co-precipitation followed by ball milling. Ceramics were made by hot pressing the powder at about 1600°C for 2 hours. The hot-pressed samples were transparent, with densities greater than 99% of theoretical. The samples were subsequently hot isostatically pressed (HIP) at about 1600°C for 2 hours under an Ar gas pressure of 30,000 psi to produce fully dense and transparent ceramics. We have demonstrated lasing with an efficiency of 74% at 1080 nm by pumping at 975 nm. Figure 20 shows the laser output power versus absorbed power showing a maximum output power of more than 16 W using a 5% output coupler. This represents the highest output power demonstrated to-date using any Yb$^{3+}$ doped Lu$_2$O$_3$ ceramic. We do observe a small roll-over in the output power at these high power levels indicating the presence of some thermal effects. When we compare the fluorescence lifetime results with values described in the literature for Yb-Lu$_2$O$_3$ single crystals, as shown in figure 21, we see that the values in the literature vary considerably [32,33,34]. This is a consequence of a plethora of effects including fluorescence trapping, impurities and concentration quenching. However, our results for the powder demonstrate that there is no quenching, even up to 10% Yb content. This is due to the high purity and uniformity of doping in the powder. The fluorescence lifetime of the ceramic is also comparable to the powder used to make it for up to 8% Yb doping. Thereafter, at 10% Yb doping level, the lifetime of the ceramic drops quickly indicating that there is concentration quenching, perhaps due to increased concentrations of Yb at the grain boundaries. Therefore, the conclusion is to keep the Yb content at 8% or lower for the current ceramics highlighted by grains in the range of 20-50 µm. It might be possible to use higher dopant concentration for ceramics with smaller grain sizes.
It is clearly evident that ceramic laser materials have come a long way since the first demonstration of lasing in 1964 using a Dy\textsuperscript{3+}:CaF\textsubscript{2} ceramic. It was not a rapid evolution, as noted by the fact that it took another 31 years to demonstrate lasing in ceramic Nd:YAG by Ikesue in 1995. Within another 7 years, improvements in powder synthesis and ceramic sintering enabled the 1 KW output power threshold to be broken in 2002, followed by another 7 years to break the 100 KW mark in 2009. There have been several other notable achievements that include highly doped microchip lasers, ultrashort pulse lasers, lasers made from different materials such as sesquioxides, fluorides, and selenides (e.g. for 2 to 3 µm region), composite ceramic lasers for better thermal management, and single crystal lasers derived from polycrystalline ceramics. We strongly believe that there will be many more notable achievements to follow.
8. ACKNOWLEDGEMENTS

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