

# An Overview on Yb-Doped Transparent Polycrystalline Sesquioxide Laser Ceramics

Angela Pirri <sup>1</sup>, Guido Toci <sup>1</sup>, Barbara Patrizi, and Matteo Vannini

**Abstract**—This paper is devoted to summarizing and discussing the scientific progress achieved during the past decade in diode-pumped ytterbium-doped solid-state lasers. The output radiation centered on the 1- $\mu\text{m}$  region is of particular interest for the well-known applications of previously developed  $\text{Nd}^{3+}$  lasers. Several hosts have been studied and tested in recent years. Due to the enormous number of papers dealing with ytterbium,  $\text{Yb}^{3+}$ , doped hosts, we will limit our report here to materials based on transparent polycrystalline sesquioxide laser ceramics, such as  $\text{Lu}_2\text{O}_3$ ,  $\text{Sc}_2\text{O}_3$ , and  $\text{Y}_2\text{O}_3$ . The reason is basically due to the wider emission band of ytterbium-doped sesquioxides as compared with more commonly used hosts such as YAG. The higher thermal conductivity with respect to hosts such as  $\text{CaF}_2$  makes them very interesting for applications requiring both good thermal conductivity and a wide fluorescence emission band, as well as for short pulse generation at high average power levels. Furthermore, the replacement of trivalent Yb in sesquioxides does not require any charge compensation (as is the for instance case of the replacement of divalent Ca in  $\text{CaF}_2$ ), which makes their fabrication easier, in particular at high doping levels.

**Index Terms**—Solid-state ceramic laser, laser ceramics, sesquioxide laser ceramics.

## I. INTRODUCTION

THE growing interest in the fabrication of cubic sesquioxide laser ceramics derives mainly from the requisite for innovative and efficient diode-pumped solid-state laser systems characterized by emission in the 1- $\mu\text{m}$  region. Basically, a laser system consists of a resonator, a pumping system, and a gain material. Although each of these plays an important role, the heart of a laser system is its gain material. High average output power, short pulses, and selected emission wavelengths can be achieved only if the host and the dopant are carefully chosen.

Indeed, the most important laser characteristics arise from the interplay between the spectroscopic and optical characteristics of hosts, as well as from the physical and chemical properties of dopants.

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A. Pirri is with the Istituto di Fisica Applicata “Carrara,” Consiglio Nazionale delle Ricerche, Sesto Fiorentino (Fi) 50019, Italy (e-mail: a.pirri@ifac.cnr.it).

G. Toci and M. Vannini are with the Istituto Nazionale di Ottica, Consiglio Nazionale delle Ricerche, Sesto Fiorentino (Fi) 50019, Italy (e-mail: guido.toci@ino.it; matteo.vannini@ino.it).

B. Patrizi is with the Istituto Nazionale di Ottica, Consiglio Nazionale delle Ricerche, Firenze 50125, Italy (e-mail: barbara.patrizi@ino.it).

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An ideal gain material should have excellent thermo-mechanical and optical properties, i.e., high thermal conductivity (which should be independent from the doping levels) to improve the heat removal, a low expansion coefficient, high hardness, and a low dependence of the linear refractive index on the temperature. Moreover, a low quantum defect and a low phonon energy are both desirable, because they contribute to reducing, respectively, the thermal load in the sample and the probability that non-radiative processes take place. Last but not least, materials should be free of excited absorption states, up-conversion, and quenching effects [1], [2]. In the infrared emission region,  $\text{Yb}^{3+}$ -doped sesquioxide laser ceramics such as  $\text{Lu}_2\text{O}_3$ ,  $\text{Sc}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  are currently under investigation, because they comply with some of the most important requirements and can in fact pave the way to a new generation of diode-pumped solid-state laser systems.

At the present state of the art, the maximum output power in CW regime, 174 W with an efficiency of 54%, has been measured by Kitajama *et al.* in  $\text{Yb}:\text{Lu}_2\text{O}_3$  ceramic thin-disk lasers [3]. As far as the generation of short pulses is concerned, Tokurakawa *et al.* have obtained laser pulses with a duration of 53 fs and an average power of 1 W by using a diode-pumped mode-locked laser based on  $\text{Yb}^{3+}:\text{Sc}_2\text{O}_3$  and  $\text{Yb}^{3+}:\text{Y}_2\text{O}_3$  multi-gain ceramic [4].

## II. $\text{Yb}^{3+}$ ION

$\text{Yb}^{3+}$  is the ion of the penultimate of the rare earths with absorption and emission peaks in the infrared region. It has a relatively simple energy level scheme. The two manifolds, i.e.,  $^2\text{F}_{5/2}$  and  $^2\text{F}_{7/2}$ , are split respectively into three and four sub-levels, with energies that depend on the strength of the Stark’s effect, which in turn is determined by the host properties, the typology of the activators and the doping level. Due to this relatively simple energy level scheme, ytterbium-doped media give origin to a quasi three-level laser system that has a broad absorption band suitable for laser-diode pumping and broad emission bands. A closeness between the two cited bands enables the Yb ion to show a relatively small quantum defect ( $\sim 10\%$ ) that contributes to reducing the thermal load. Furthermore, the small diameter of  $\text{Yb}^{3+}$ , which is comparable to those of  $\text{Lu}^{3+}$ ,  $\text{Sc}^{3+}$  and  $\text{Y}^{3+}$ , makes possible a heavy doping of the sesquioxides (and of other hosts) without a significant distortion of the host lattice. It is worth noting that the long lifetime of the  $^2\text{F}_{5/2}$  sub-levels is another advantage, in that it permits efficient energy storage and lowers the pump power required to reach the lasing

threshold. Nowadays its main drawback, namely the reabsorption of laser emission by thermally-populated lower laser levels (which increases the lasing threshold), is easily overcome by using high-efficiency pumping systems and a moderate cooling of the gain media.

### III. FABRICATION TECHNIQUES AND RESULTS

The first attempts at developing sesquioxide materials involved single crystals, and several growth techniques were contrived such as the Czochralski [5] and the Bridgman methods [6], the Micro-Pulling Down ( $\mu$ -PD), the Verneuil method [7], [8], the laser-heated pedestal growth (LHPG) [9], [10], the cold-container method or skull-melting [2], and the flux method [4]. Due to its required equipment, each of the methods cited above allows for growing crystals up to a specific dimension (for instance, the Czochralski crystals are usually smaller than the Bridgman ones [11]); however, the first two methods are the ones most employed worldwide in laboratories.

In the case of sesquioxides, which are characterized by high melting points (2490 °C in  $\text{Lu}_2\text{O}_3$ , 2485 °C in  $\text{Sc}_2\text{O}_3$ , 2425 °C in  $\text{Y}_2\text{O}_3$ ) and by phase transition points lower [12] than the melting points, the Czochralski and the Bridgman methods were applied, albeit with some adjustments [11], [13]. There were two mandatory issues: the use of crucibles made of rhenium, (which has a melting point as high as 3180 °C) and equipment capable of avoiding any interaction between the melting and the crucible itself. At the same time, it was necessary to isolate the growth system from the growth process. Despite this, only small-size laser crystals have been produced. *Fornasiero et al.* obtained crystals with a maximum diameter of 10 mm and a thickness ranging from 3 mm to 6 mm by using a rhenium crucible surrounded by inert gases or a reducing mixture with 10–15 vol% of  $\text{H}_2$  [11]. *Peters et al.* have grown  $\text{Sc}_2\text{O}_3$  crystals by using the heat exchanger method (HEM) [13]. Recently, by applying the  $\mu$ -PD method [14] it was possible to grow  $\text{Lu}_2\text{O}_3$  and  $\text{Sc}_2\text{O}_3$  crystal rods that had diameters ranging from 1 mm to 5 mm [15], [16]. It is worth noting that the  $\mu$ -PD method permits growing single crystals by employing a small amount of raw material, which represents a great advantage since lutetium is very expensive. Moreover, it is possible to grow a crystal rapidly and then check its spectroscopic properties.

An alternative pathway is currently represented by transparent polycrystalline ceramics. Their fabrication process makes it possible to obtain samples with a uniform dopant distribution, high levels of doping and excellent thermo-mechanical and optical properties. For instance, the fracture toughness of ceramics is found to be higher than that of a single-crystal [17]–[21]. In  $\text{Y}_2\text{O}_3$  and YAG crystals, a comparable value was measured, i.e.,  $2 \text{ MPa} \cdot \text{m}^{1/2}$  and  $2.2 \text{ MPa} \cdot \text{m}^{1/2}$  [22], respectively. It is interesting to note that grain size,  $d$ , affects the ceramic strength,  $\sigma$ , in accordance with the Hall-Petch equation ( $\sigma \propto 1/d^{1/2}$ ). Smaller grain size thus implies higher values in ceramic strength. In the case of sesquioxides, the sintering temperature is around 1700 °C and, indeed, their fabrication is easier than that of the single crystal. Nowadays, samples having large sizes (e.g., centimeters) and good optical quality are available.

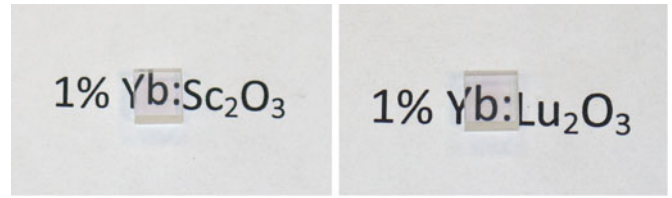


Fig. 1. 1 at.%  $\text{Yb}^{3+}$  doped  $\text{Sc}_2\text{O}_3$  and  $\text{Lu}_2\text{O}_3$  ceramic samples produced by Konoshima Chemical Ltd., Japan. Size of the samples is  $5 \times 5 \times 3 \text{ mm}^3$ .

Many efforts have been made to improve the fabrication process, starting from the quality of the powders (this is closely connected with the raw materials), as well as the preparation of the powders themselves and the sintering process, which are crucial aspects in reaching the goal of improvement. Nanocrystalline powders can be fabricated by means of combustion synthesis [23], [24], the sol gel processing [25], the hydrothermal method [26], the emulsion synthesis, and the co-precipitation in aqueous medium [27]–[29] as well as by laser ablation [30]. The influence of the powder processing on the sintering of 1 at.%  $\text{Yb}:\text{Sc}_2\text{O}_3$  ceramics has been demonstrated by *Bravo et al.*, who prepared the powders using the solid state reaction process and co-precipitation method, and then, compared their performances [31].

As far as the sintering process is concerned, several techniques are currently being applied, e.g., vacuum sintering [32], [33], vacuum sintering followed by hot isostatic pressing [34], hot pressing (HP) [35], hot pressing and hot isostatic pressing (HP-HIP) [36], spark plasma sintering (SPS) [37], [38], and pressure-less sintering under flowing  $\text{H}_2$  atmosphere [39]. As for the growth techniques of single crystals, the various sintering processes impose severe limitations on the dimension, uniformity, shape of the sample, and production time. For instance, the SPS method permits the fabrication of ceramic samples in a relatively short time (about one sample per day), but at the same time their sizes are very limited. It is interesting to note that the vacuum sintering temperatures of each material, which have been reported in the literature by various authors, usually show a fairly narrow interval of variation, one that ranges from 1700 °C to 1850 °C [40], while the HIP temperatures show a much broader range, i.e., from 1300 °C [41] to 1850 °C [34].

Lastly, many authors have demonstrated that the appropriate mixing between powder fabrication methods and sintering processes is crucial to achieving the greatest optical transparency in ceramics. To date, the best results have been obtained by using powders fabricated in laboratories [34], [42], [43] and then subjected to vacuum sintering and hot isostatic pressing or hot pressing [44]. Photos of 1 at.%  $\text{Yb}^{3+}$  doped  $\text{Sc}_2\text{O}_3$  and  $\text{Lu}_2\text{O}_3$  ceramic samples produced by Konoshima Chemical Ltd., Japan can be viewed in Fig. 1.

### IV. THERMAL CONDUCTIVITY

Dealing with the thermal effects that may occur inside the gain material of a high-power laser is extremely important, because these can seriously affect laser performance, leading, for instance, to lesser efficiency, lower power extraction, and spatial

distortion of the laser beam. Hosts with high thermal conductivity are preferred because they minimize the thermal gradients that lead to spatial beam distortion and to thermal birefringence. Among these, sesquioxides play a key role as undoped matrices, because they are characterized by high thermal conductivity values. In undoped crystals, values of thermal conductivity as high as 12.2 W/mK for  $\text{Lu}_2\text{O}_3$  [6], 15.5 W/mK for  $\text{Sc}_2\text{O}_3$  [6] and 13.6 W/mK for  $\text{Y}_2\text{O}_3$  [6], [12] have been measured. These values are considerably higher than those for other hosts (e.g., a value of 9.71 W/mK has been reported for  $\text{CaF}_2$  [11]).

A theoretical model developed in order to describe thermal conductivity in undoped and doped crystals, and which still remains valid for transparent ceramic, can be found in [45]. Thermal conductivity depends on many parameters, the most important of which are the temperature experienced by the sample (which increases during the pumping process) and the doping levels. Furthermore, in the case of ceramics, it also depends on the grain size of the microcrystals. Larger doping concentrations of activators act as defects that modify the host lattice, especially when the atomic weight and the radius of the dopant ions are different as compared to ones that have been replaced [46]. Similar atomic weights reduce the probability that a phonon scattering will take place [45], while similar radii make possible higher levels of doping, thus preserving the initial values of the thermal conductivity. In 3 *at.%*  $\text{Yb:Sc}_2\text{O}_3$  and  $\text{Yb:Y}_2\text{O}_3$  crystals, a substantial decrease has been found in the thermal conductivity: i.e., 7.7 W/mK and 6.6 W/mK, respectively [12]. In any case, these values remain high when compared with other hosts.

As far as  $\text{Y}_2\text{O}_3$  ceramics are concerned, *Fan et al.* have measured a value of 13 W/mK in an undoped sample, and have clearly demonstrated that temperature variation influences its thermal behavior: in fact, in the range from 90 K to 290 K, the thermal conductivity decreased by a factor of four [47]. The experimental data were confirmed by *Tokurakawa et al.*, who tested both the undoped  $\text{Y}_2\text{O}_3$  and the doped 2 *at.%*  $\text{Yb}^{3+}$ , which were fabricated by means of vacuum sintering [48]. They found 12.6 W/mK and 7.9 W/mK, respectively, thus confirming the role played by doping levels on thermal properties. An accurate study reporting the optimum doping level, which preserves a reasonable level of thermal conductivity, can be found in [5]. In this paper, the best performing samples have a concentration of  $\text{Yb}^{3+}$  that ranges between 5.0 *at.%* and 7.0 *at.%*. Furthermore, the experiment showed that, especially in highly-doped ceramics, an increase in the temperature generates an increase in the absorption coefficient and in the fluorescence lifetime. Similar results were achieved by comparing the data obtained at cryogenic and room temperatures with 10 *at.%*  $\text{Yb:Y}_2\text{O}_3$ , 1 *at.%*  $\text{Yb:Lu}_2\text{O}_3$  and 1 *at.%*  $\text{Yb:Sc}_2\text{O}_3$  ceramics [6]. Samples activated with low dopant concentrations become strategic for amplification purposes. In high-power laser systems, the principal goal is the amplification of high-energy pulses at high repetition rates by keeping the heat generated during the pumping process at the lowest possible level. This is of particular importance for large-scale laser systems such as the ones studied in the HIPER project [49] in Europe, in the MERCURY (Nd:Glass) in the USA, and in the LUCIA (Yb:YAG) in France. The Gaumé model for thermal conductivity [45] has been

confirmed in lutetia ceramics by *Rand et al.*, who measured with 10 *at.%*  $\text{Yb:Lu}_2\text{O}_3$  a value of 11.1 W/mK [7], which is comparable with the data reported in [6]. This result was also found to be almost independent in the  $\text{Yb}^{3+}$  concentration. The influence of grain size on the results could be observed by comparing the previous results with the values obtained in 1 *at.%*  $\text{Yb:Lu}_2\text{O}_3$ , i.e., 8.0 W/mK, which is lower than expected.

It is important to emphasize that, in the literature, the thermal conductivity values measured in ceramics are sometimes contradictory. This observation is addressed to the complexity of the measurement itself, as well as to the difficulty in comparing data obtained by using different methods, i.e., the laser flash or temperature wave method, moreover, the grain size plays an important role [8], [10]. For instance, in 3 *at.%*  $\text{Yb:Lu}_2\text{O}_3$  ceramic *Nakao et al.* found 13.9 W/mK by using the laser flash method [9], while *Tokurakawa* measured a value of 10.9 W/mK in the undoped matrix by means of the temperature wave method [50]. In any case, a comparison between ceramics, even ones having the same composition, is useful only if the grain sizes are comparable [8]–[10], [50].

## V. LASER OSCILLATIONS

As reported above,  $\text{Yb}^{3+}$ -doped  $\text{Lu}_2\text{O}_3$ ,  $\text{Sc}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  transparent ceramics were obtained by employing several fabrication techniques. High optical quality rods, slabs and thin-disks with different concentration of activators, from 1 *at.%* to 10 *at.%* of  $\text{Yb}^{3+}$ , have been used as laser gain materials in high-performance laser systems. Depending on the host, laser oscillations at around 1030 nm, 1040 nm, 1078 nm and 1094 nm have been demonstrated in both CW and *quasi*-CW operational modes. Moreover, their broad emission bands have been found to be suitable for the generation of fast pulses. In most cases, the mode-locking condition was achieved by means of a semiconductor saturable absorber mirror (SESAM). However, although sesquioxides demonstrate emission spectra that correspond to pulses of up to 200 fs, assuming a transform-limited  $\text{sech}^2$  pulse, this limit has been overcome thanks to their high non-linear refractive index, which may trigger non-linear effects, e.g., self-phase modulation and Kerr lensing. Indeed, sub-100 fs pulses have been generated. The non-linear refractive index in undoped matrices of  $\text{Lu}_2\text{O}_3$ ,  $\text{Sc}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$  was  $3.96 \times 10^{-13}$  esu [52],  $5.32 \times 10^{-13}$  esu [51] and  $5.79 \times 10^{-13}$  esu [51], respectively.

An *excursus* to the main results is presented here as follows.

### A. $\text{Yb}^{3+}:\text{Lu}_2\text{O}_3$

The first laser emission with 3 *at.%*  $\text{Yb}^{3+}$  ceramic prepared with vacuum sintering and nanotechnology fabricated by Konoshima Chemical Co. [53], [54] was obtained by *Takaichi et al.* [55]. In CW operation mode, it delivered 0.95 W at 1079 nm with a slope efficiency of 53%, while at 1035 nm the output power was 0.7 W with a slope efficiency of 36%. One year later, the continuous tunability of lutetia in two bands, from 1033.8 nm to 1038.5 nm and from 1067.3 nm to 1081.3 nm, was demonstrated. The maximum output power obtained was around 270 mW. The tunable cavity was built by inserting a SF10 glass prism while the ceramic was pumped by a fiber coupled laser

diode at 976 nm (zero-phonon line). The measurements were performed at room temperature [56].

Improvements in the fabrication techniques enabled *Sanghera et al.* to synthesize 10 at.%  $\text{Yb}^{3+}:\text{Lu}_2\text{O}_3$  by using the HP sintering process [36], [57]. They measured at 1080 nm an output power above 16 W with a slope efficiency of 74%. The ceramic was pumped in *quasi*-CW with a duty factor of 50% and a repetition rate of 127 Hz.

The ceramic with the lowest doping level, i.e., 1 at.%, fabricated by Konoshima Chemical Co., was tested by *Pirri et al.* [58]. At 1032.5 nm the laser delivered 1.8 W with a slope efficiency of 45%. The ceramic was placed in a V-shaped resonator and pumped at 968 nm by a laser diode coupled with a 100- $\mu\text{m}$  diameter fiber in *quasi*-CW regime (duty factor 20%, 10 Hz), while the cavity was tuned by substituting the output coupler mirror with a gold grating set in Littrow's configuration. This made it possible to explore, for the first time, the continuous tuning range from 1000 nm to 1045 nm.

As explained previously, the replacing of  $\text{Lu}^{3+}$  ion with  $\text{Yb}^{3+}$  only slightly affects the thermal conductivity of activated lutetia. In fact, it is an interesting material for thin disk laser systems in which a high concentration of doping is required in thin samples (200  $\div$  300  $\mu\text{m}$  thickness). In 2014, a CW laser operation of thin-disk lasers based on  $\text{Yb}:\text{Lu}_2\text{O}_3$  ceramics was demonstrated. A 300- $\mu\text{m}$  thickness disk doped with 3 at.%  $\text{Yb}^{3+}$  was used, pumped at 976 nm by a Volume Bragg Grating (VBG) locked fiber coupled laser diode. The laser delivered 45.1 W at 1032 nm with a slope efficiency of 60.1% [9]. A significant power and efficiency scaling was achieved with a thinner disk of 150  $\mu\text{m}$ , doped with 3 at.%  $\text{Yb}^{3+}$  [3]. In the CW operation, the laser delivered 174 W of output power with a slope efficiency of 54%. The maximum pump density was 5.5  $\text{kW}/\text{cm}^2$ .

As regards the possibility of generating short pulses, the fluorescence spectrum showed two main peaks placed around 1032 nm and 1079 nm [60], [61] with a FWHM of 13  $\div$  15 nm that made it possible to generate a pulse duration of about 100 fs at the Fourier limit. The first demonstration of a diode-pumped passively mode-locked 3 at.%  $\text{Yb}:\text{Lu}_2\text{O}_3$  ceramic laser was obtained by *Tokurakawa et al.* [59]. They measured a pulse duration of 357 fs at 1033.5 nm with a maximum average power of 352 mW and a repetition rate of 97 MHz.

This first result was improved by exploiting the high non-linear refractive index of the host matrix. *Tokurakawa et al.* used a combination of  $\text{Yb}:\text{Lu}_2\text{O}_3$  and an undoped  $\text{Y}_2\text{O}_3$  to build up a laser system based on Kerr-lens mode-locking that generated 65 fs pulses at 1032 nm with an average power of 320 mW [61]. To the best of our knowledge, this is still the shortest pulse generated by a laser system based on lutetia ceramic. However, to be even more precise we should note that *Paradis et al.* recently measured 35 fs of pulse duration in  $\text{Yb}:\text{Lu}_2\text{O}_3$  thin-disk crystal, and until now this represents the shortest pulse obtained with lutetia [62].

### B. $\text{Yb}^{3+}:\text{Sc}_2\text{O}_3$

The first laser emission of 2.5 at.%  $\text{Yb}:\text{Sc}_2\text{O}_3$  at room temperature was observed in 2003 [63]. The sample was fabricated

by Konoshima Chemical Co. using the nanocrystalline technology and the non-press vacuum sintering technique [53], [54]. The sample was pumped at 940 nm using a laser diode coupled with a 100- $\mu\text{m}$  core diameter fiber. A CW laser emission at 1041 nm and 1094 nm was demonstrated. At the shorter wavelength, the laser delivered 420 mW of output power with an efficiency of 9%. The sample was further characterized by measuring the refractive index, i.e., 1.96 at 1064 nm, and the fluorescence lifetime of the upper state of the laser transition revealed a value of 0.80 ms.

Heavily doped ceramic, 10 at.%  $\text{Yb}^{3+}$ , was tested by *Casagrande et al.* [64]. A 1-mm thickness sample was end-pumped by a diode stack delivering *quasi*-CW pump pulses of a 600  $\mu\text{s}$  duration at 1 Hz. The maximum peak power was 2 KW. The experiment was performed at room temperature as well as at a cryogenic temperature (80 K), in order to study the reabsorption effects on both the efficiency and the wavelength of the laser. The authors demonstrated that the absorption and the emission cross sections increased by decreasing the temperature, while the wavelength of the laser oscillation did not shift from shorter to longer values. This behavior was justified by the fact that in scandia the third sublevel of the  ${}^2\text{F}_{7/2}$  manifold of  $\text{Yb}^{3+}$  is scarcely populated and more greatly separated from ground level than occurs in lutetia and yttria. Lastly, they found that the laser efficiency increased from 7% (at 280 K) to 26% (at 80 K) while the laser threshold decreased from 506 mJ to 141 mJ.

High efficiency and high-power laser emissions were obtained with 5 at.%  $\text{Yb}^{3+}$  ceramic fabricated by Konoshima Chemical Co. [65]. From the data it appears that the cryogenic temperature contributed to enhancing  $\text{Yb}$ -doped sesquioxides laser performance, as was theoretically expected. The sample was pumped in *quasi*-CW, while the measurements were performed at room and cryogenic temperatures. In the former case, the laser delivered 18 W of output power with a laser efficiency of 43%. At a cryogenic temperature (77 K), both the efficiency and the output power increased up to 70% and 40 W, respectively.

The doped scandia tunability range was verified in different experiments [58], [66], [67]. In the first experiment, the sample, which was obtained by means of vacuum sintering and the nanotechnology method doped with 2.5 at.%  $\text{Yb}^{3+}$ , was tested under CW pumping at 976 nm. The tunable cavity was obtained by placing a SF10 glass prism between the folding and the output coupler mirrors. Two continuous bands, from 1040.5 nm to 1042.6 nm and from 1090.5 nm to 1096.5 nm, were observed. A different approach was used in the latter experiment, during which the output coupler mirror of a V-shaped cavity was replaced by a gold-coated grating (1800 grooves/mm) used at the Littrow's configuration [58]. The measurements performed by this approach, which were carried out by pumping the sample at 968 nm in *quasi* CW mode at a repetition frequency of 10 Hz and with a pulse length of 20 ms, gave rise to a continuous tunability curve that ranged from 1010 to 1051.6 nm. With the non-tunable cavity, the laser delivered a maximum output power of 2.2 W at 1040.5 nm with a slope efficiency of 59%. Power and efficiency scaling were achieved by pumping at a wavelength of 933 nm.

In this case, the output power and the slope efficiency increased to 4.3 W and 70%, respectively, and a broader tunability ranging from 1005 nm to 1050.5 nm, was measured [59]. A tunability of up to 55 nm was achieved instead with 5 *at. %* Yb:Sc<sub>2</sub>O<sub>3</sub> fabricated by means of co-precipitation and vacuum sintering [68].

In Kerr-lens mode-locking regime, *Tokurakawa et al.* tested a ceramic with 2.5 *at. %* Yb<sup>3+</sup> doping which generated 92 fs pulses at 1042 nm with an average power of 850 mW [69]. The optical-to-optical efficiency was 21.9%. Shorter pulses, with a duration of 90 fs and with an average power of 160 mW, were also obtained at a laser oscillation of 1092 nm. To date, this remains the best result achieved with Yb<sup>3+</sup> doped scandium oxide.

### C. Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub>

The period between 2002 and 2003 was very productive for the development of laser systems based on transparent ceramics. Thanks to the use of a 3-mm-thick ceramic sample fabricated by Konoshima Chemical. Co., *Kong et al.* achieved the first laser oscillation in Yb<sup>3+</sup> doped yttrium oxide [70], [71]. The sample, doped with an 8 *at. %* Yb<sup>3+</sup> and water cooled (10 °C), was pumped at 937 nm in CW mode. At 1078 nm it delivered 0.75 W with a pump power of 11 W, thus achieving an efficiency of 12.6%. The pump laser threshold was found to be 4.7 W.

The progress in the optical quality of yttria ceramics enabled the same group to reach, in CW emission, an output power of up to 4.2 W with an efficiency of 29% at 1078 nm [72].

Shorter emission wavelengths, i.e., 1030 nm, for a TEM<sub>00</sub> mode, were demonstrated with a 2 *at. %* Yb<sup>3+</sup> sample obtained by means of a pressure-less fabrication method. The uncoated ceramic plate had a thickness of 500 μm and was pumped by a laser diode at 940 nm. The maximum output power and the efficiency were 0.6 W and 45%, respectively [2]. Emission at 1075 nm was demonstrated with the use of a 4 *at. %* Yb<sup>3+</sup> sample, the thickness of which was 0.8 mm. It delivered 1.4 W of output power in the TEM<sub>00</sub> mode with 1.9 W of absorbed pump. The efficiency was as high as 72%.

An output power of 9 W with 41% efficiency at 1078 nm and a pump threshold of 3.1 W were instead demonstrated by means of an 8 *at. %* Yb<sup>3+</sup> sample pumped at 937 nm with a fiber-coupled laser diode [73]. The 3-mm-long, antireflection coated ceramic was water-cooled at 0 °C. The authors reported on an interesting comparison between 8 *at. %* Yb-doped YAG crystal and Yb:Y<sub>2</sub>O<sub>3</sub> in which they found that a ceramic laser performs better, since it shows higher efficiency and greater output power thanks to its lower reabsorption losses at the same level of pump power.

Two years later in a new experiment, a 2 mm long, an 8 *at. %* Yb<sup>3+</sup> doped, antireflection coated ceramic was pumped on the zero phonon-line, at 976 nm, thus demonstrating an enhancement in the laser performance. In CW emission, a maximum output power of 1.74 W was achieved at 1078 nm, and 0.73 W at 1040 nm. The efficiency was 82.4% for the 1078 nm laser emission and 57.1% at 1040 nm [74]. The ceramic was water cooled at 5 °C.

A 10 *at. %* Yb<sup>3+</sup> ceramic that was developed by *Merkle et al.* was used for a spectroscopic and laser investigation at room and cryogenic temperature (77 K) [65]. The 1.67-mm-long sample was pumped at 939 nm in quasi-CW mode operation (2 Hz, 1 ms pump pulse duration) by means of a 400-μm core fiber-coupled laser-diode. The authors demonstrated that, at liquid nitrogen temperatures, the laser performance was strongly enhanced due to a reduction in the reabsorption at the laser wavelength; thus, the stronger 1030 nm emission peak reached the threshold prior to the peak at 1077 nm. The slope efficiency was 70%, while the laser energy threshold was 7 mJ. At room temperature, instead, the laser oscillated at 1077 nm, and the efficiency decreased to 64% while the threshold increased to 52 mJ. From 77 K to 300 K, the maximum output power was halved, i.e., it was reduced from 80 W to 40 W with a launched pump power of 120 W.

Recently, highly transparent 5 *at. %* Yb<sup>3+</sup> ceramic was fabricated by using a combination method of vacuum sintering and hot isostatic pressing. The ceramic slab was pumped at 940 nm by means of a fiber-coupled laser diode. *Wang et al.* demonstrated a laser oscillation at 1076 nm with 0.77 W of output power and an efficiency of 10.6% [33].

The first CW mode-locked emission of a 4 *at. %* Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> ceramic was demonstrated in 2003 [75]. The mode-locking was obtained by means of a SESAM. At a laser wavelength of 1076.5 nm, pulses as short as 615 fs were generated with a repetition rate of 98 MHz. The average power was 420 mW, while the pulse energy was 4.3 nJ, with 2.6 W of absorbed pump power. Several years later, *Tokurakawa et al.* demonstrated a diode-pumped passively mode-locked operation of 1.8 *at. %* Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> ceramic laser [69]. A SESAM was used also in this case. The aforesaid team measured pulses as short as 188 fs at 1038 nm, with an average output power of 220 mW and with an absorbed pump power of 2.4 W at 97 MHz. The thickness of the sample was 1.5 mm.

The wide emission spectral bandwidth, which shows two well-defined peaks around 1030 nm and 1076 nm, together with the large nonlinear refractive index as explained above, are two important features in the generation of fast pulses. The first self-mode-locked Yb-doped yttria ceramic laser was demonstrated in 2007 [76]. The 3-mm-long gain material was doped with 8 *at. %* Yb<sup>3+</sup> and pumped at 937 nm by means of a fiber-coupled laser diode. The water-cooled (7 °C) ceramic sample generated pulses having a duration of 1.1 ps at 1078 nm, with an average output power of 2.7 W at a repetition rate of 126 MHz. In this laser system, no saturable absorber was used, but the mode locking was achieved by the interplay of two effects, i.e., the Kerr self-focusing and the diffraction losses generated by the thermal lens aberration due to the non-uniform heat deposition which occurred during the pumping. In fact, because the magnitude of the diffraction loss was proportional to the laser-mode size, the diffraction loss in combination with the Kerr-lens focusing acted as a saturable absorber.

Shorter pulses and high average power, 547 fs and 7.4 W respectively, were achieved in a mode-locking laser operation at 1031 nm. The ceramic sample was fabricated using nanocrystalline and vacuum sintering technologies. The Yb<sup>3+</sup> doping level was 2 *at. %*, while the thickness of the disk was 400 μm

[48]. It was pumped at 975 nm by a fiber-coupled laser diode, while the laser mode-locking was achieved by using a SESAM.

## VI. CONCLUSIONS AND OUTLOOK

The production of high-optical-quality sesquioxide transparent ceramics can be considered to be one of the most important results achieved for the development of high average-power laser systems in recent years. This is due, first of all, to the specific properties of the sesquioxides themselves and, secondly, to the characteristics of ceramics: for instance, their lower fabrication temperature, which makes it possible to fabricate large size samples that are suitable for complex laser systems, including amplification chain systems. Among Yb-doped sesquioxides, lutetia is very interesting because of its good thermal conductivity value, which is almost independent of the doping level. Indeed, lutetia is ideal both for lasers based on thin-disk geometry, which require a high doping level of the thin disk gain material, and/or for microchip lasers. A thermal shock figure of merit for lutetia was calculated by Soules and reported in [77]. This host has a high non-linear refractive index that can favor the occurrence of non-linear effects, and in consequence it produces short pulses. The lutetia refractive index is in any case higher than those of scandia and yttria. The possibility of exploring a new class of host materials, resulting from the mixing of two or more sesquioxides together is very interesting. Each Yb-doped sesquioxide host shows a characteristic fluorescence spectrum, the position of the peaks, broadness, and so on. The combination of two or more of their fluorescence spectra may act to enlarge the gain bandwidth, thus leading to the generation of faster pulses. In this regard, the shortest pulse was obtained by Tokurakawa and co-workers [4], using a combination of 2.5 at.%  $\text{Yb}^{3+}:\text{Sc}_2\text{O}_3$  and 1.8 at.%  $\text{Yb}^{3+}:\text{Y}_2\text{O}_3$  ceramic as multi-gain material. The thicknesses of the two materials were 1 mm and 1.5 mm, respectively. The stable and self-starting mode-locked operation was achieved by means of a SESAM. Two different pulse durations of 66 fs (with a 1.5 W average power) and 53 fs (with a 1 W average power) were measured. The repetition rate in both cases was about 99 MHz.

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**Angela Pirri** received the M.S. degree in physics from the University of Pisa, Pisa, Italy in 2002, and the Ph.D. degree in experimental physics from the University of Siena, Siena, Italy, in 2005. From 2003 to 2004, she worked on nonlinear optics as a Marie Curie Fellow with the Laser Centre of Vrije Universiteit, Amsterdam, The Netherlands. From November 2005 to April 2008, she worked on the development, characterization, and applications of novel coherent sources in the extreme ultraviolet based on high-order laser harmonics with the European Laboratory for Non-Linear Spectroscopy, Florence, Italy. She was a "Visiting Scientist" with ETH Zurich, Zurich, Switzerland, and with the Laboratoire Matériaux et Phénomènes Quantiques, Université Paris Diderot—Paris 7, Paris, France. Since May 2008, she has been a Researcher with the Institute of Applied Physics, National Research Council, Florence, Italy, where she was working on solid-state laser based on transparent polycrystalline ceramics and crystals with emission in the infrared and visible regions. She is the author of more than 50 peer-reviewed journal papers and many international conference communications.



**Guido Toci** was born in 1966. He received the M.S. and Ph.D. degrees in physics from the University of Florence, Florence, Italy, in 1991 and 1996, respectively.

Since 1997, he has been a Researcher with the Italian National Research Council (CNR), first with the Institute of Quantum Electronics, then with the Institute of Applied Physics "Nello Carrara" (IFAC) (from 2001 to 2013). Since the end of 2013, he has been a Researcher with the National Institute of Optics-CNR (INO-CNR), Sesto Fiorentino, Italy. He is currently

the author of more than 75 papers on peer-reviewed journals and holds three patents. His research interests include new solid laser materials, in particular ceramics and crystals doped with Yb and Nd, and the development of solid-state lasers devices, as well as laser applications to environmental monitoring and industrial laser applications.



**Barbara Patrizi** received the Master's degree in molecular and biomedical sciences with the University of Perugia, Perugia, Italy, in 2009, and the Ph.D. degree in atomic and molecular spectroscopy from European Laboratory for Non-Linear Spectroscopy (LENS), Florence, Italy, in 2013. During the Ph.D. degree, she focused her research on the study of carbon monoxide recombination and structural dynamics in truncated hemoglobins by ultrafast pump-probe laser spectroscopy. During Postdoc, she worked with the Group of Ultrafast Spectroscopy, LENS, focusing

her activity on the chemical-physical characterization of organic molecules and genetically engineered light harvesting antennas for photovoltaic devices. Since December 2016, she has been a Researcher with the National Institute of Optics, National Research Council (CNR), Florence, Italy. Her research interests include development and characterization of laser ceramics materials, development and characterization of organic photonic crystals, CO recombination dynamics in cold-adapted truncated hemoglobins, and characterization of organic dyes for applications in LED technology.



**Matteo Vannini** was born on December 9, 1954. He received the "Laurea in Physics" on December 21, 1979. He worked in Frascati, Rome, Italy; Marseille France; and Los Alamos, NM, USA, where he worked mainly on excimer UV lasers. Since then, he has been with the University of New Mexico, Albuquerque, NM, USA, where he worked on nonlinear optics on the second-order cascade effect. He was a staff member with National Research Council (CNR), where he worked on solid-state laser. In particular, he worked on Ce: LICAF in the UV region and in the near infrared region with Yb-doped media. He is the author of more than 100 papers and several conference communications.