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Pre-evaluation method for the spectroscopic properties of YAG bulk materials by sol–gel synthetic YAG powder

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Abstract

We propose a convenient method for pre-evaluating the spectroscopic properties of luminescent impurity doped YAG by sol–gel synthetic powder. Three synthetic methods, the sol–gel, normal-strike, and reverse-strike methods, are examined for their ability as a predictor and the sol–gel method proved the most effective of the three in experiments on crystallinity, particle size, and spectroscopic properties. The sol–gel Nd:YAG powder agrees well with that of a single crystal or ceramic YAG regarding the luminescent spectrum, the intensity, and the lifetime, even though the powder sintered at a low temperature of 1100 °C in comparison with the YAG crystal melting point of 1970 °C. These facts suggest that the sol–gel synthetic powder is the most effective and the easiest technique for evaluating spectroscopic properties of bulk YAG materials before fabricating the final materials.

Keywords: A. Powder: chemical preparation; A. Sol–gel processes; C. Optical properties

1. Introduction

The fabrication techniques for transparent ceramics have been actively developed. Many reports [1–7] on isotropic crystals such as YAG ($Y_3Al_5O_{12}$) and ceramic (polycrystal), which has the same optical characteristic as a single Nd:YAG crystal [8–10] have attracted great interest in various fields. The following points are the advantages of transparent ceramics. First, the growth temperature of 1750 °C [1] is lower than the melting temperature (1970 °C) [11,12] of the single crystal because a fine, uniform particle was used as the raw material powder. Second, since the sintered ceramic is free from defects caused in the melt–crystal interface when a single crystal is grown by the Czochralski method [13–16] a large product can be obtained. Third, the production period is shorter than that of the single crystal growth. The fourth point concerns the capability of a great variety of functional impurity dopants. This point is very attractive in advanced materials research, especially laser or luminescent materials. In the case of doped-YAG ceramics, a high concentration of a doped element, a new element, or co-doped ceramics is also possible. Finally, the fabrication of a composite material with undoped and doped parts is also possible.

Although the production period of ceramics is shorter than that of crystals, obtaining good quality optical ceramics is still very difficult. This is because the fabrication process of ceramics consists of several steps, which are the synthesis of powder, molding, binder removal, and sintering, and each step has its own process difficulty. Therefore, if we can evaluate the luminescent properties of sintered ceramics by synthesized ceramic powder before sintering the final ceramics, this would be convenient and significant for luminescent materials research, such as laser media.

The precursors of YAG ceramics were mainly prepared by solution syntheses. Various synthesis methods have been reported, including coprecipitation, homogeneous precipitation, sol–gel, and reversed micelle [17–23]. The form and particle-size-distribution depend on each synthesis approach; that means each particle may have its own characteristics. Therefore, it is important to verify which powder synthetic

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methods and conditions are appropriate to estimate the optical properties of bulk ceramics.

In this report, we have examined the pre-evaluation ability of synthetic luminescent impurity doped YAG powder for final crystalline or ceramics YAG. To make sure which synthetic procedure is suitable for predicting final ceramics properties, non-doped, Cr or Nd:YAG powder were synthesized by the sol–gel method and the two coprecipitation methods, reverse-strike and normal-strike, and then the crystallinity, particle size, and spectroscopic properties are evaluated by X-ray diffraction (XRD) pattern , scanning electron microscope (SEM), particle-size-distribution analyzer, and spectrophotometer. According to the experiment, the sol–gel synthesis powder showed better performance on the reproducibility of single crystal or ceramics Nd:YAG spectroscopic properties derived from good crystallinity and mono-distribution of particle size than the other synthetic processes. This pre-evaluation method with synthesized sol–gel powder will help us to evaluate YAG ceramics by avoiding the difficulty of making final bulk samples.

2. Experimental

2.1. Fabrication method of YAG powders

No-doped YAG, Cr:YAG, and Nd:YAG powders were produced by three methods: (a) sol–gel, (b) normal-strike, and (c) reverse-strike. The doped elements and doped concentration are listed in Table 1. The particle properties were evaluated on the no-doped YAG (samples 1–3). The dependence of the luminescent intensity on the concentration of the doped element was studied with Cr:YAG powders (samples 4–10). Then a comparison of luminescent spectrum, intensity, and lifetime with the powder, the single crystal, and ceramic was performed with Nd:YAG (11–13 in Table 1). A single crystal of 0.6 mol%-Nd:YAG was available from the II-VI Japan Corporation, and ceramic of 0.6 mol%-Nd:YAG was obtained from Konoshima Chemical Co., Ltd. The single crystal and ceramic samples were 10 mm in diameter and 1.7 mm in thickness.

Table 1
Sample list of produced powders, single YAG crystal, and ceramic YAG specimens (SG: sol–gel method, N: normal-strike method, R: reverse-strike method).

Sample no.	Concentration in YAG		Fabrication method
	Nd_2O_3	Cr_2O_3	
To check the crystallinity and particle distribution			
1	0.0	0.0	SG
2	0.0	0.0	N
3	0.0	0.0	R
To check the luminescence properties of Cr:YAG			
4	0.0	0.5	SG, N, R
5	0.0	1.0	
6	0.0	2.0	
7	0.0	3.0	
8	0.0	4.0	
9	0.0	5.0	
10	0.0	6.0	
To check the reproducibility of Nd:YAG spectroscopic properties			
11	0.6	0.0	SG
12 (standard)	0.6	0.0	Bulk ceramics
13 (standard)	0.6	0.0	Single crystal

2.1.1. Sol–gel method

The reagents used were $Al[O(CH_3)CHC_2H_5]_3$ (aluminum tris-sec-butoxide: 97%), $Y(CH_3COO)_3{\cdot}4H_2O$ (yttrium acetate tetraydrate: 99.99%), $Cr(CH_3COO)_3{\cdot}xH_2O$ (chromium acetate hydrate), $Nd(CH_3COO)_3{\cdot}H_2O$ (neodymium acetate nomohydrate: 99.9%), and anhydrous 2-propanol. Ultra high purity water of resistivity 13 MΩ/cm or more was also used.

An Al tris-sec-butoxide-2-propanol solution and an aqueous solution of Y acetate and Cr acetate (or Nd acetate) were prepared based on the stoichiometry of YAG. The ratio of Al tris-sec-butoxide-2-propanol and water was adjusted at a molar ratio of 1:100. The Al tris-sec-butoxide-2-propanol solution was added into aqueous solutions of Y acetate and Cr acetate (or Nd acetate) heated at 80 °C and stirred. The operation was done under an N_2 atmosphere so that the Al tris-sec-butoxide would not hydrolyze before the reaction. The mixture became a transparent gel in about 60 min.

The transparent gel was dried in an oven with an N_2 gas flow at 80 °C for 12 h and then kept at 130 °C for 24 h. Since the dried gel became a hydroxide precursor of the YAG crystal, it was strongly agglomerated. An agate mortar and pestle were used for grinding. Then the powder was heated at 800 °C for 100 h in an air atmosphere to remove the remaining organic impurity in the precursor. After that, it was sintered at 900, 1000, 1100, 1200, 1300, and 1400 °C in an air atmosphere for 4 h, and the temperature-raising rate was 150 °C/h.

2.1.2. Normal-strike method

The used reagents were $AlCl_3{\cdot}6H_2O$ (99.9%), $YCl_3{\cdot}6H_2O$ (99.9%), $CrCl_3{\cdot}6H_2O$ (99.995%), and NH_4HCO_3 (99.0%). In the synthesis and the washing of the precursor, ultra-pure water of resistivity 13 MΩ/cm or more was used. A 0.5 mol/l-aqueous solution of Al, Y, and Cr chlorides was prepared with a stoichiometric ratio. 0.2 M-H_2SO_4 was added to the aqueous solution of Al, Y, and the Cr to keep the pH of the aqueous solution below 2, and then the NH_4HCO_3 aqueous solution was added into the mixture and stirred. Precipitation began to appear over a 3.6 pH of the mixed solution. The NH_4HCO_3 aqueous solution was dropped into the solution until the pH value of the mixed solution reached 6.0. Afterwards, it was constantly stirred at 35 °C for 3 days.

The precipitate was washed with ultra-pure water using a centrifugal separator and a homogenizer. Washing was repeated until the ion concentration in the rinse solution decreased to 100 ppm (0.1 mS/cm) or less. The ion concentration was measured with conductivity and a dissolved solids tester DIST2 (HANNA Instruments). To reduce the residual water that causes strong agglomeration between particles, ethanol was substituted for the residual water.

The precipitate was dried at 80 °C for 12 h and followed by 130 °C for 24 h. The aggregation of the particle–particle was weak, so the aggregation was easily crushed with an alumina

mortar and pestle. The dried YAG precursor was a carbonate. The organic impurity was removed in the same way as the sol–gel method.

2.1.3. Reverse-strike method

The reagents used were the same as in the case of the normal-strike method. Al, Y, and the Cr aqueous solutions were prepared based on stoichiometry. The total ion concentration was 0.5 mol/l.

Al, Y, and the Cr aqueous solutions were dropped into a 2.5 M NH_4HCO_3 aqueous solution initially adjusted with ammonia to a pH of 8 and stirred at 35 °C. Precipitation occurred immediately. After the precipitation, the operation was identical to the above normal-strike method.

2.2. Evaluation of powder characteristics

YAP ($YAlO_3$ and 1870 °C melting point [24,25]) and YAM ($Y_4Al_2O_9$ and 1930 °C melting point [24,25]) are allotropes of YAG (1970 °C melting point). The degree of crystallization and phases of the synthesized powders were examined by X-ray diffraction (XRD: Rigaku RINT2500). The measurement conditions were as follows: Cu Kα/50 kV/300 mA, 0.010° in sampling width, 4.000°/min in scan speed, and 25.000–40.000° in the scan range. The distribution of the particle size was measured by a laser-scattering particle-size-distribution analyzer, HORIBA LA-920 with He–Ne laser (wavelength: 632.8 nm), and the morphologies of the powder were observed by a scanning electron microscope, HITACHI S-800. The concentration of each element in the synthesis solution was measured by inductively coupled plasma spectroscopy (ICP: Perkin-Elmar Inc. Optima 3000XL). The spectroscopic properties (luminescence spectrum, intensity, and lifetime) were measured by a high-resolution photoluminescence spectrophotometer JASCO SS-25. The measurement specimens were prepared by mixing the powders and a 1 wt.%-polyvinyl alcohol aqueous solution and applying on slide glasses and drying.

3. Results

3.1. X-ray diffraction

The measured X-ray diffraction patterns of samples 1–3 at several sintering temperatures are shown in Fig. 1(a)–(c), respectively. For the sol–gel method (sample 1), clear peaks of YAG crystal have already appeared at 800 °C, suggesting that crystallization started before 800 °C. The other phases, YAP or YAM, were not observed in any sintering temperature. These peaks agree well with the diffraction peak of YAG [26]. For the normal-strike (sample 2) and the reverse-strike (sample 3) methods, a definite peak began to appear at 1000 °C. The YAM peaks [27] were found under 1100 °C in the normal-strike method. In the reverse-strike method, YAM peaks still remained at 1400 °C.

The relationship between sintering temperature and the integrated area of the main peak at $2\theta = 33.3°$ is shown in Fig. 2 for samples 1–3. The integral values of sol–gel powder were higher than the other two synthetic methods, and these are relatively constant for the sintering temperature. For the normal-strike and reverse-strike methods, both integral values increased with increasing temperature. And then the values for the normal-strike method were roughly constant at 1100 °C and reached the sol–gel values. On the other hand, the values for the reverse-strike method still increased with increasing temperature after 1100 °C; therefore, the higher sintering temperature would be requested to reach the constant value. From here on in this report, the sintering temperatures are set at 1100 °C for sol–gel and at 1200 °C for the others.

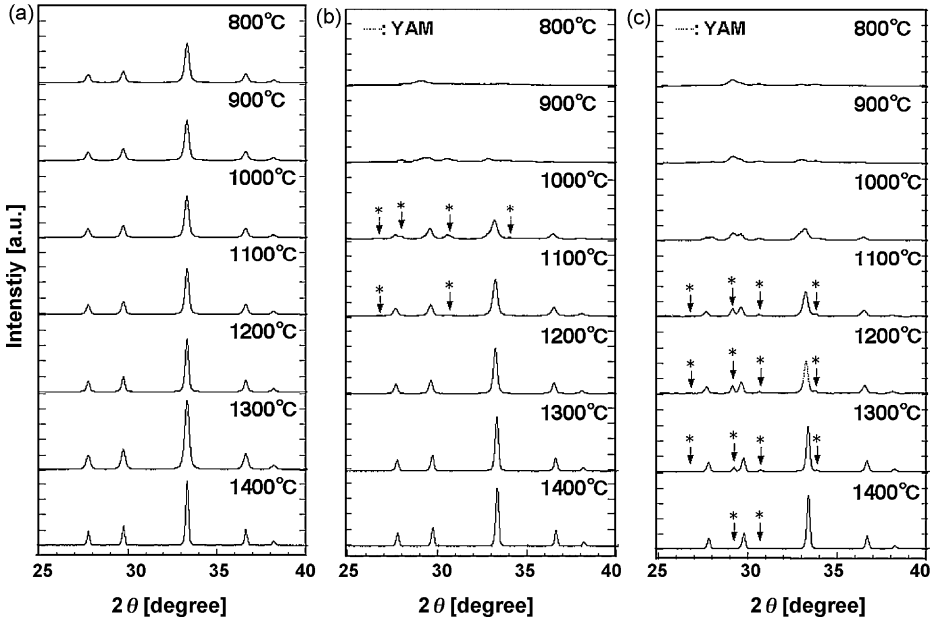

Fig. 1. X-ray diffraction patterns of YAG powder prepared by (a) sol–gel method, (b) normal-strike method, and (c) reverse-strike method as a function of sintering temperature. Asterisks show YAM peaks (ICDD #14-0475).

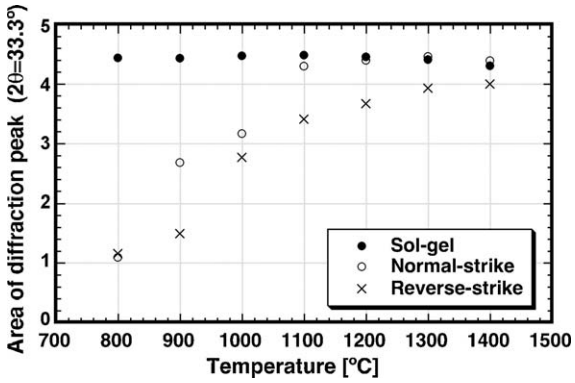

Fig. 2. Area of diffraction peak ($2\theta = 33.3^{\circ}$) for YAG measured by X-ray diffraction pattern as a function of sintering temperature.

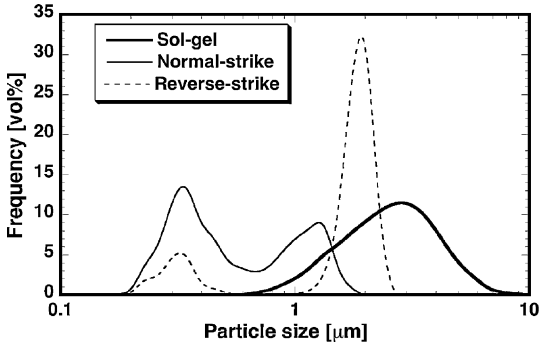

Fig. 3. Distribution of particle size of YAG powder measured by laser-scattering particle-size-distribution analyzer.

3.2. Size distribution and electron microscopic images

The particle-size-distribution was checked for samples 1–3 (Fig. 3). The particle-size-distribution of the sol–gel powder was observed as a single broad dispersion from 1 to 10 μm in Fig. 3 (bold solid line). On the other hand, the normal- and reverse-strike powders show two peak distributions. The smaller distributions of the primary particles are about 0.2 μm in both the normal-strike and reverse-strike, and the larger distributions of the secondary particles were 2 and 1.5 μm, respectively.

The SEM images are shown in Fig. 4. Sol–gel, normal-strike and reverse-strike method particles correspond to (a1–3), (b1–3), and (c1–3), respectively. For the sol–gel method, the size ranges from 1 to 10 μm and agrees with the results of the particle-size-distribution in Fig. 3. It is considered that the primary particles of the precursor were very small as shown in Fig. 4(a3). Since the primary particles had many hydroxyl groups originating from $Al(OH)_3$ or $Y(OH)_3$, the condensation and crystallization of the YAG primary powder could begin at low temperature; then, the primary particles became hard agglomerates, as shown in Fig. 4(a2) [28].

The particle sizes prepared by the normal- and reverse-strike methods were estimated to be about 0.2 μm from the SEM images (b2 and c2). The size of the primary particles prepared by the normal- and reverse-strike methods was about 50 nm from Fig. 4(b3 and c3). These are larger than the sol–gel method shown in Fig. 4(a3). The second peaks (around 1–2 μm) of the normal- and reverse-strike methods in Fig. 3 are considered the secondary particle that aggregates the primary particles.

3.3. Dependence of luminescent intensity on synthetic methods

The dependencies of emission intensity on Cr^{3+} ion concentration for the Cr-doped YAG powder produced by the sol–gel, normal-, and reverse-strike methods were studied. The samples are listed in Table 1 (samples 4–10). Luminescent intensity was detected at 708 nm with excitation at 590 nm monochromatic light using SS-25. These wavelengths, 708 and 590 nm, correspond to the emission and excitation peaks of Cr^{3+} ion, respectively. The results are shown in Fig. 5. The luminescent intensity of the sol–gel powder increased at first, reached the maximum at 3 mol% concentration, and then gradually decreased with an increasing concentration of Cr^{3+} ion. The tendency was the same in the other two synthetic methods. The luminescent intensity of the sol–gel powder was the strongest of the three methods, followed by the normal-strike and reverse-strike methods. It is considered that this result depends on the crystallinity of the synthesis powders.

3.4. Reproducibility for sol–gel powder on luminescent properties

According to Section 3.3, luminescent properties of sol–gel powder are superior to the other ones. In this section, we test the reproducibility of sol–gel powder for bulk ceramics or single crystal in spectroscopic properties. The samples of 11–13 were used. The excitation spectra detected at 1064 nm are shown in Fig. 6. The excitation spectrum of the sol–gel powder agreed well with both the spectra of the crystal and the ceramics. Luminescent spectra excited at 808 nm are also shown in Fig. 7. The luminescent spectra of the sol–gel powder almost agreed with both the spectra of the crystal and the ceramics, but the luminescent spectral width is slightly wider than those of the crystal and the ceramics. It may be affected by some quantum size effect due to the nano-size of the YAG powder in Fig. 4(a3). The luminescent intensity and the lifetime for three samples are shown in Table 2. The luminescent properties of single crystal and transparent ceramics have been reported to be almost equivalent [8–10]. The measured values of the emission intensities and the lifetimes were nearly equal among the three samples. Therefore, it is considered that the sol–gel YAG

Table 2
Luminescence intensities and lifetimes from Nd doped sol–gel powder, single crystal, and ceramics at 1064 nm excited by 808 nm light.

Samples	Intensity (mV)	Lifetime (μs)
Sol–gel powder	1.35	244
Bulk ceramics	1.32	237
Single crystal	1.41	229

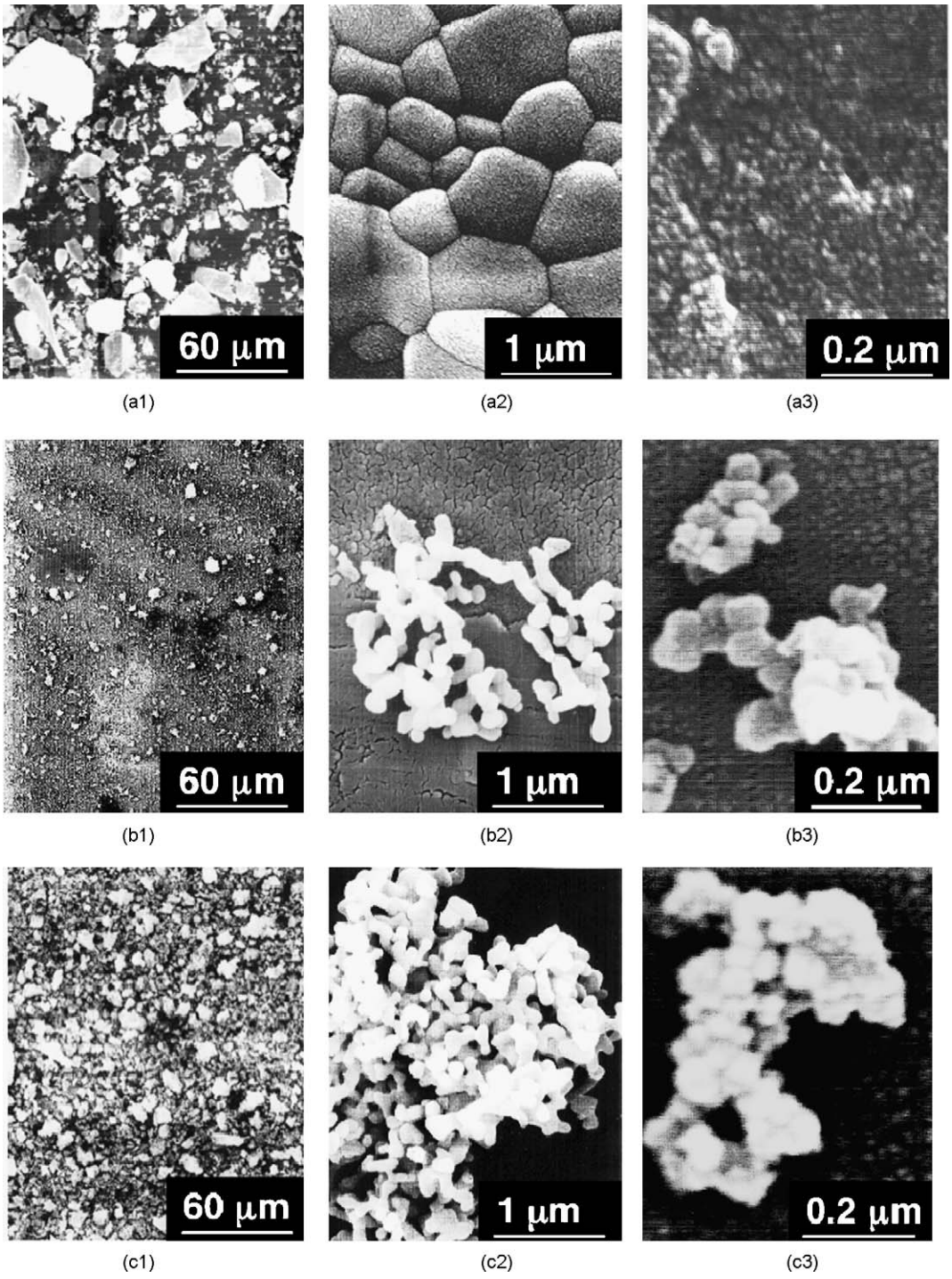

Fig. 4. SEM images of powders produced by (a1–a3) sol–gel, (b1–b3) normal-strike, and (c1–c3) reverse-strike methods.

powder has the potential to evaluate the spectroscopic properties of YAG ceramics before we get the final ceramic products.

4. Discussion

Our concern is to know which powder is the most suitable for reproducibility of luminescent properties of final YAG ones, and whether the spectroscopic properties of synthetic YAG powder can predict the spectroscopic properties of final products. Let us consider rearranging the results.

(1) Crystallinity

The crystallinity of the sol–gel powder is more favorable than the other synthetic powders because of the following:

(a) The XRD pattern of YAG crystal is clearly observed after 800 °C heat treatment though the melting temperature of YAG crystal is 1970 °C.

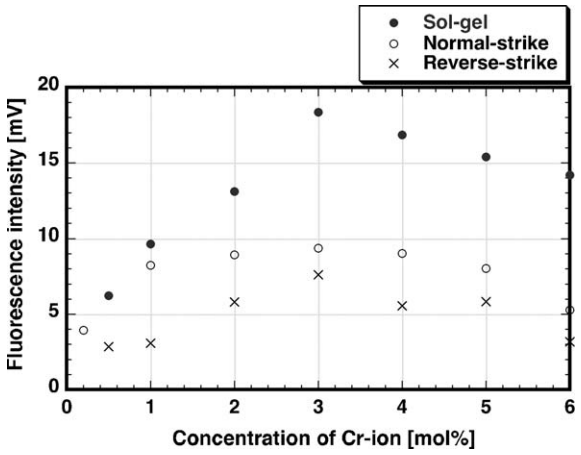

Fig. 5. Dependence of Cr:YAG powder luminescence on Cr ion concentration. Peak reflects concentration quenching of Cr ions. (Closed circle: sol–gel, open circle: normal-strike, crosses: reverse-strike.)

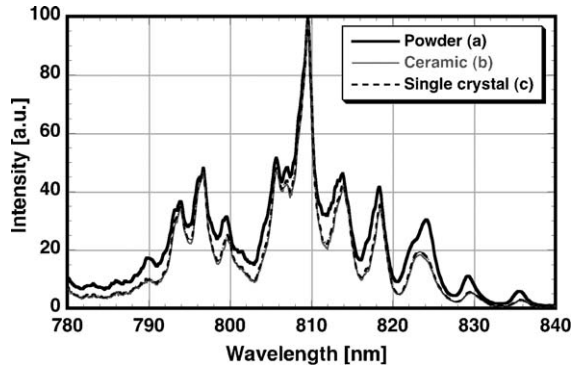

Fig. 6. Excitation spectra of luminescence at 1064 nm of (a) synthetic Nd:YAG powder, (b) ceramic, and (c) single crystal.

(b) The easiest aggregation leads to the largest segment size.
(c) Only single YAG phase is obtained at low temperature (800 °C∼); on the other hand, normal- or reverse-strike have residual phases of YAM and YAP.

(2) Luminescent intensity

The sol–gel power of Cr:YAG shows more intense luminescence than the other two synthetic powders. There is some mutuality between crystallinity and luminescent intensity.

(3) Reproducibility

The sol–gel powder, which has the best crystallinity and luminescent intensity among the three synthetic methods, shows good reproducibility of luminescent properties for final YAG products, such as a single crystal or ceramics.

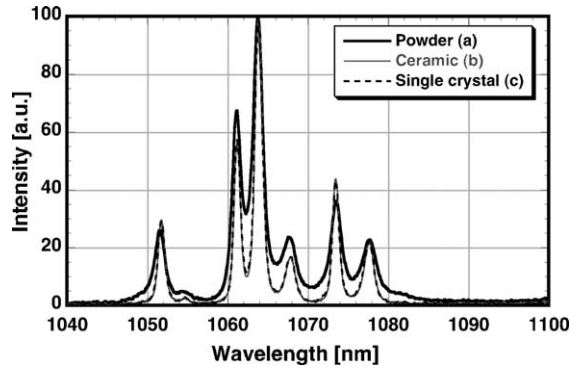

Fig. 7. Luminescence spectra at 1064 nm excited by 808 nm light of (a) synthetic Nd:YAG powder, (b) ceramic, and (c) single crystal.

As discussed above, it is considered that the sol–gel powder will work as a good predictor for spectroscopic properties of final YAG single crystal or ceramics. The advantages of this method are considered to be as follows:

(1) In pre-evaluation of spectroscopic properties, the luminescent impurity doped sol–gel powder will work as a predictor.
(2) The optimum process temperature for making sol–gel synthesis powder is 1100 °C. This method does not require high temperatures such as 1970 °C for single crystal growth or 1750 °C for ceramics. Therefore, the synthesis process is much easier than that of single crystal or ceramics.
(3) This method can avoid the difficulty of fabricating a bulk sample with crystal growth or ceramics technique.

This method could be significant for the evaluation and design of the optical property of a single crystal YAG and bulk YAG ceramics before making final optics. Moreover it seems that this method can be used not only for YAG but also for other oxide crystals, for instance, Y_2O_3, ZrO_2, TiO_2, and $MgAl_2O_4$.

5. Conclusions

The validity of an evaluation technique using a YAG powder produced by the sol–gel method was examined. The sol–gel YAG powder, which had the highest crystallinity compared with other coprecipitation methods, also showed the most intense luminescent emission of Cr:YAG. The mimetic performance for single crystal or ceramic YAG was also checked. The luminescent spectrum, intensity, and lifetime of the sol–gel Nd:YAG powder were equivalent to those of single crystal or ceramic YAG, even though the powder was sintered at a lower temperature (1100 °C) compared with the YAG crystal melting point of 1970 °C. These two facts suggest that sol–gel YAG powder is the most effective and easiest for evaluating functional bulk YAG optical materials before they have been completed. It is expected that this method is significant for the evaluation and design of optical properties not only of YAG but also of other oxides, for instance Y_2O_3, ZrO_2, TiO_2, and $MgAl_2O_4$.

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