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THE GROWTH AND THE COMPOSITIONAL ANALYSIS OF Nd^{3+}-DOPED YAG CRYSTAL

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ABSTRACT

The Nd:YAG crystal with the dimension of 65 mm in length and 30 mm in diameter has successfully been grown by Czochralski technique in an argon gases atmosphere. Their crystalline parameters are then characterized using the X-Ray Diffraction (XRD) technique. The percentage of each element has been determined using the Energy Dispersive X-ray Analysis (EDX). The calculation on the lattice parameters confirmed that the sample is purely cubic Nd:YAG structure. Meanwhile, the Nd^{3+} dopant concentration is observed to vary from (0.06 – 0.12) atomic% depending on position from the neck of the crystal.

INTRODUCTION

The Czochralski technique or crystal pulling has become the best method for the growth of many bulk oxide materials. This technique through various modifications has become the dominant process used in industry today for the production of semiconductor and oxide single crystals [1]. Materials such as silicon, sapphire (Al_2O_3), GaP, GaAs, InP, $Gd_3Ga_5O_{12}$, Nd:$Y_3Al_5O_{12}$, germanium, and $LiNbO_3$ are normally grown today by this technique [1].

YAG has a cubic crystal lattice system. Incorporated of Nd^{3+} into $Y_3Al_5O_3$ garnet reveals the substituted of Nd^{3+} into eight coordinate of Y^{3+}. This substitution does not affect the crystal structure because on the ionic radius of both Nd^{3+} (1.12 Å) and Y^{3+} (1.01 Å) almost the same [2]. Nd:YAG is usually used in monocrystalline form, fabricated with Czochralski growth method, but there is also ceramic (polycrystalline) Nd:YAG available in high quality and in large sizes. Nd:YAG crystals is ordinarily produced with Nd^{3+} concentrations from 0.18 at% to 1.1at% for applications in all types of solid-state lasers systems [3]. The Nd^{3+} concentration in Nd:YAG crystal normally varies along the grown boules. The concentration is low on the top but slightly increases at the bottom of the boules.

EXPERIMENT PROCEDURE

The Nd^{3+}-doped YAG system was prepared by melting technique in iridium crucible under pure argon gases atmosphere. These samples were prepared from certified

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reagent grades Y_2O_3 (99.99% purity, Al_2O_3 (99.99% purity) and 4.5 atomic% of Nd_2O_3 (99.99% purity). By using Czochralski technique, the iridium crucible (5.6 cm diameter, 5.6 cm high) is placed at the centre of coil position which surrounded with zirconia insulator in order to protect the high radiation in the furnace as shown in Figure 1. Alumina was mounted around the system to decrease the radial temperature gradient in the melt and the seed used along the <111> direction. The pulling and rotation rate used are 0.75mm/h and 15 rpm respectively. After the growth completed, the crystal was cooled for 60 hours. The grown sample was then cut into three parts with the thickness 3 mm. These samples will undergo grinding and polishing using alumina suspension.

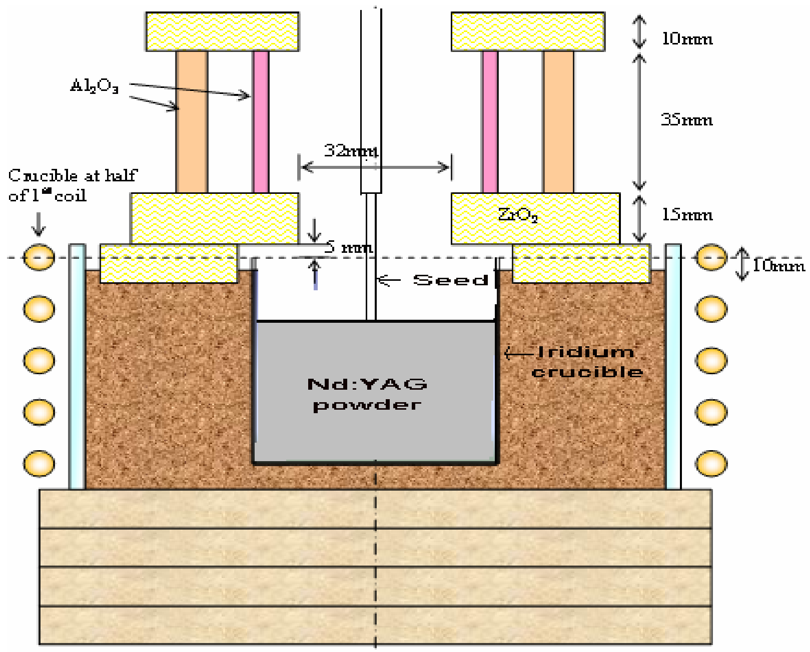

Figure 1: The schematic diagram of Nd:YAG crystal growth using the Czochralski technique

The sample was examined using a Bruker D5005 diffractometer to identify the structure, using 40 kv-50mA power with nikel filter copper radiation (λ=1.5406 Å). Energy Dispersive X-ray Analysis (EDX) was used to determine the actual elements content of Nd:YAG crystal.

RESULT AND DISCUSSION

The Nd:YAG crystal has successfully been grown using czochralski technique. It has a weight of 63 grams and has a length 65 mm and 33 mm in full diameter. These parameters are very much depending on the growth setting parameters. Figure 2 shows the actual Nd:YAG crystal which has been grown by this method. It is observed that the crystal is visually light purple but it gradually become the dark purple as the content of Nd^{3+} is higher. This is due to the changes in valence state of Nd^{3+} in the crystal. The crack is found at the bottom part of the crystal. It might be due to the

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jerking out effect during the pulling of the crystal from the melt.

Figure 2: The actual Nd:YAG crystal grown using the Czochralski technique.

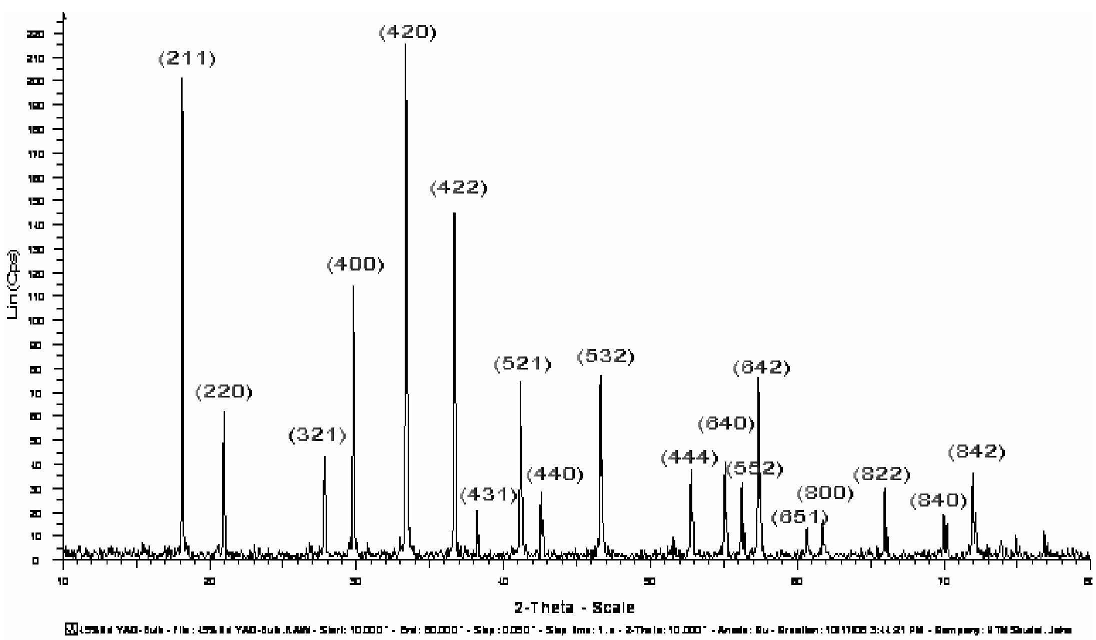

Figure 3: X-Ray diffraction pattern of 4.5 atomic% Nd doped Nd:YAG crystal

Figure 3 shows the X-Ray diffraction pattern of 4.5 atomic% Nd^{3+}- doped YAG crystal. The pattern reveals that the sample was in crystalline phase similar to those of reference

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in ICSD (Inorganic Crystal Structure Database) standards [4]. The intensities of the reflection for some crystal planes, together with their Miller indices and distances between the reflection planes are shown in Table 1. The respective references are also inserted. From the Table 1, it can be deduced that the lattice parameter is about 12.01 Å which is in agreement with a published data by A. Golubovic *et. al.*[6].

Table 1: The spacing of lattice planes (d), Miller indices (hkl) and intensities for the Nd:YAG crystal and their respective references [4].

2θ (angle)	2θ (angle) [4]	d (Å)	d (Å) [4]	Miller Indices (hkl)	Intensity (%)	Intensity (%) [4]
18.152	18.068	4.88313	4.90551	211	93.4	34
20.921	20.893	4.24269	4.24830	220	28.7	7
27.826	27.756	3.20349	3.21141	221	19.9	18
29.810	29.715	2.99466	3.00400	400	53.1	24
33.343	33.319	2.68498	2.68686	420	100	100
36.641	36.606	2.45056	2.45276	422	67.3	20
38.199	38.158	2.35411	2.35653	431	9.3	5
41.160	41.111	2.19131	2.19381	521	34.5	21
42.628	42.524	2.1192	2.12415	440	7.5	5
46.620	46.553	1.9466	1.94925	532	35.6	22
51.605	51.542	1.76965	1.77166	631	4.4	2
52.854	52.735	1.73074	1.73436	444	17.5	16
55.121	55.067	1.66479	1.66632	640	19	29
56.270	56.208	1.63351	1.63517	552	14.8	7
57.453	57.333	1.60266	1.60571	642	35.2	25

Table 2 shows the percentage content of elements for respective parts of the Nd:YAG crystal. It shows that the percentage of the element seem to increase from the top part to the bottom part of crystal by vary from 0.06 to 0.12 atomic% Nd. These changes might be due to distribution coefficient, κ of the solid solution during the crystal growth process [1]. The control of the concentration level and composition with respect to the length of the crystal is extremely important because it will affect the optical properties of the crystal.

Table 2: The percentage of element for top, middle and bottom parts of Nd:YAG crystal.

Element	Top (±0.01 atomic%)	Middle (±0.01 atomic%)	Bottom (±0.01 atomic%)
Oxygen (O)	59.96	59.96	59.93
Aluminium (Al)	24.98	24.98	24.97
Yttrium (Y)	14.99	14.99	14.97
Neodymium (Nd)	0.06	0.065	0.12

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CONCLUSION

From the above discussion, it can be concluded that the Nd:YAG crystal has successfully been grown using the Czochralski technique. The crystal has a lattice parameter of 12.01Å. The Nd:YAG crystal obtained is found to compose of 0.06 to 0.12 atomic% Nd^{3+} depending on the position of the crystal.

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