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Growth, structure, spectral properties and crystal-field analysis of monoclinic Nd:$YNbO_4$ single crystal

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ABSTRACT

A Nd:$YNbO_4$ single crystal was successfully grown by Czochralski (Cz) method, its structural and spectroscopic properties were investigated. The X-ray rocking curve of the (010) diffraction face of Nd:$YNbO_4$ crystal was measured, the full width at half maximum (FWHM) of this diffraction peak is 0.05°, which indicates a high crystalline quality of the as-grown crystal. Its lattice parameters, atomic coordinates and so on were obtained by Rietveld refinement to X-ray diffraction data. According to the Archimedes drainage method, the crystal density of Nd:$YNbO_4$ is calculated to be 5.4 g/cm^3. The Mohr's hardness value along (010) face was determined to be 6.0. The transmission spectrum along (010) face at room temperature was recorded and the excitation and emission spectra at 8 K were measured. Photoluminescence peaks of Nd:$YNbO_4$ were assigned, and the crystal-field splitting of Nd^{3+} in $YNbO_4$ was obtained. The fluorescence lifetime of the $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition of Nd^{3+} in $YNbO_4$ is fitted to be 152 μs These spectroscopic and energy splitting data give an important reference for the research of laser property of Nd:$YNbO_4$ crystal.

1. Introduction

In recent years, laser diode pumped solid-state lasers based on Nd-doped crystals have attracted great attention for the applications in the medical treatment, industrial processing, and optical communications due to their high level of stability, high efficiency and compactness [1–4]. Nd-doped vanadate crystals have been proved to be a kind of high efficient laser medium with relatively high thermal conductivity. Nd:YVO_4 as one of the oldest researched vanadate crystal have been commercialized for polarizer and low pumping power laser in these years [5,6]. Nd:$LuVO_4$ and Nd:$GdVO_4$ exhibit better thermal properties than Nd:YVO_4, which are expected to be used in moderate power lasers [7,8]. However, vanadate crystals tend to component volatility and form color centers during the Cz growth process, which indicates that it difficult to obtain high-quality and large-size crystal by Cz method [9]. Nowadays, rear earth doped tantalite materials as a novel laser hosts are being gradually researched, because they have stable physical and chemical properties and can be grown by Cz method. For example, a Nd:$GdYTaO_4$ single crystal was successfully grown by Peng for the first time, which shows excellent spectroscopic properties and the laser performance at 1066 nm was also performed [10]. The physical and spectroscopic properties of Yb, Ho:$GdYTaO_4$ crystal was researched by Dou [11]. However, there are few reports about the rare earth doped niobate crystals, to the best of our knowledge. $YNbO_4$ has three types of structure [12,13]: high temperature tetragonal phase (T-type), low temperature monoclinic phase (M-type, with space group of $I2/a$), and monoclinic phase (M′-type, with space group of P2/a). In M-type, Nb atom coordinates with four O atoms to form a distorted tetrahedron whereas in M′-type Nb atom and six O atoms form a distorted octahedron. In Nd:$YNbO_4$ single crystal, Nd^{3+} substitutes for Y^{3+} with C_2 symmetry. The low symmetry of Nd^{3+} in $YNbO_4$ is beneficial to relaxing the parity-forbidden rule and realizing polarized laser output [10,14]. Compared with the other low symmetry laser crystals grown by Cz method, Nd:$YNbO_4$ has some advantages [15,16]. For example, there is no component volatility in Cz growth processing of Nd:$YNbO_4$, besides, the melting point of $YNbO_4$ crystal is lower than that of orthosilicates. Thus, larger-size and higher-quality Nd:$YNbO_4$ crystal can be easily obtained using Cz method.

In addition, the researches of niobates mainly focus on the rare earth doped phosphors. And niobate family ($ReNbO_4$, Re=Y, Gd, La) have been proved to be inspiring luminescent materials. However, the researches on rare earth doped niobate crystals are rarely reported, to the best of our knowledge [17–19]. Therefore, Nd:$YNbO_4$ crystal is chosen to be laser host and its structure and luminescence properties

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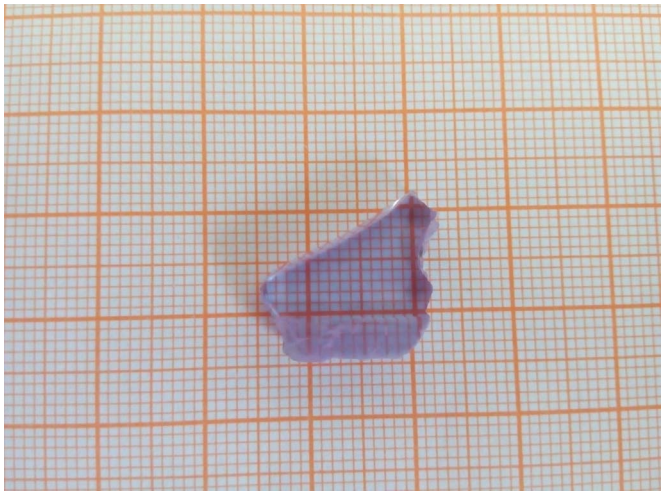

Fig. 1. The crystal slice of Nd:$YNbO_4$ which was cut and polished perpendicular to the *b* axis.

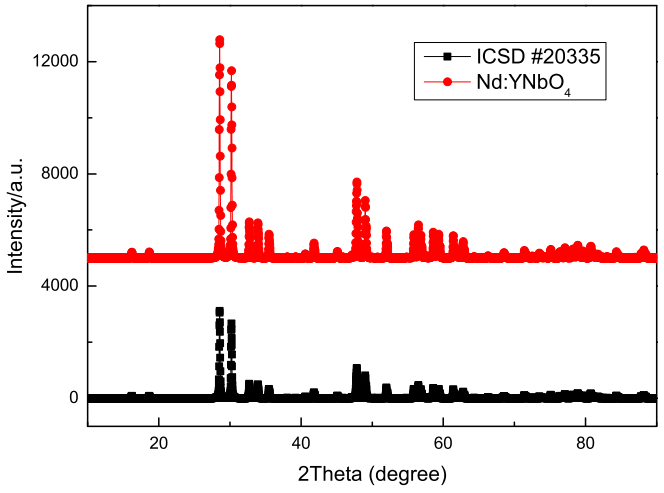

Fig. 2. XRD diffraction patterns of Nd:$YNbO_4$ crystal and $YNbO_4$ standard patterns (ICSD # 20335).

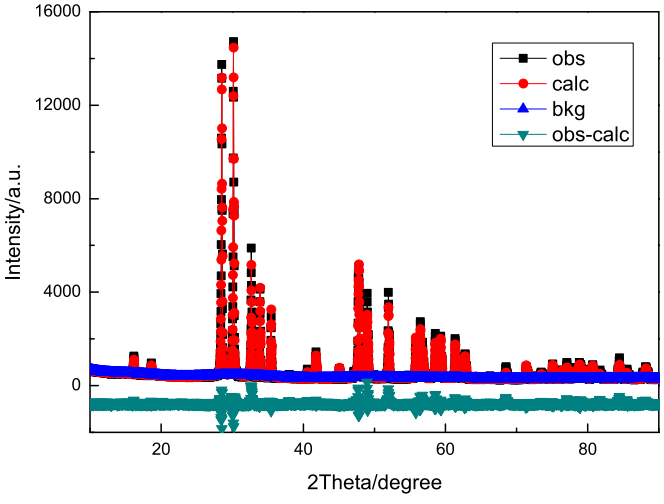

Fig. 3. Rietveld refinement to the X-ray diffraction of Nd:$YNbO_4$ crystal (obs: the experimental data; calc: the calculated pattern; bkg: the background curve; obs-calc: the difference curve).

was investigated in this work, in order to evaluate its laser performance.

Table 1
Structural parameters of Nd:$YNbO_4$ crystal, which are obtained by Rietveld refinement to X-ray diffraction data.

Atoms	X	Y	Z	Uiso
Nd and Y	0	0.6207	0	0.25
Nb	0	0.1442	0	0.25
O1	0.2454	0.0554	0.248	0.3259
O2	0.2841	0.29	0.301	0.2981
Lattice	A=7.041 Å, B=10.952 Å, C=5.301 Å; α=γ=90°, β=134.07°			
R factor	Rp=6.7%, Rwp=8.75%			

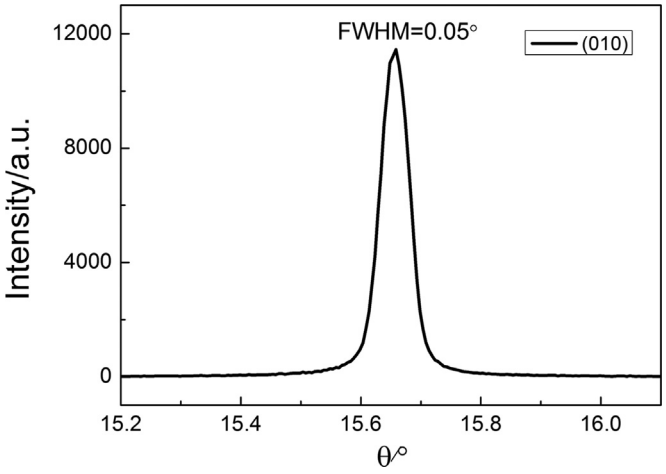

Fig. 4. X-ray Rocking curves of the (010) diffraction face of Nd:$YNbO_4$ crystal.

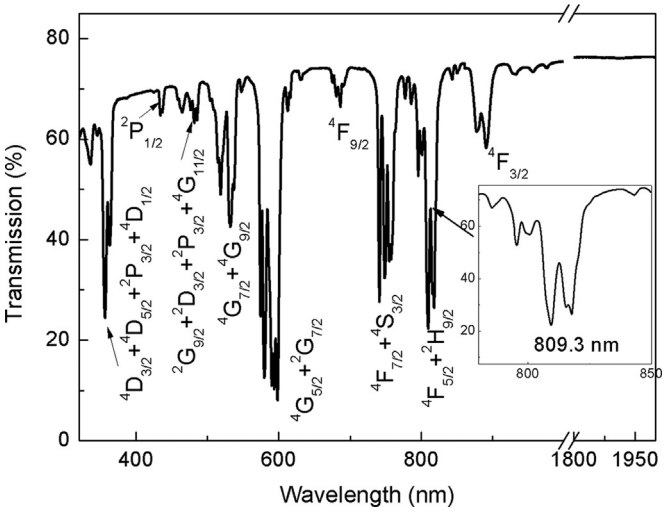

Fig. 5. Transmission spectrum of Nd:$YNbO_4$ crystal.

2. Experimental details

2.1. Crystal growth

Using the Cz method, 1 at% Nd-doped $YNbO_4$ crystal was grown successfully with a JGD-60 furnace (26th institute of CETC, China), with an automatic diameter controlled (ADC) growth system. The compounds Y_2O_3 (5N), Nb_2O_5 (4N), and Nd_2O_3 (5N) were used as raw materials. The oxides were weighed according to the chemical formula $Nd_{0.01}Y_{0.99}NbO_4$. After being mixed thoroughly and pressed into disks, the mixtures were loaded into iridium (Ir) crucible (60 mm in diameter, 45 mm in height). A $YNbO_4$ single crystal was used as the seed. The crystal was grown in N_2 atmosphere in order to prevent the iridium crucible from oxidization. The pulling rate was 0.5–1.5 mm/h

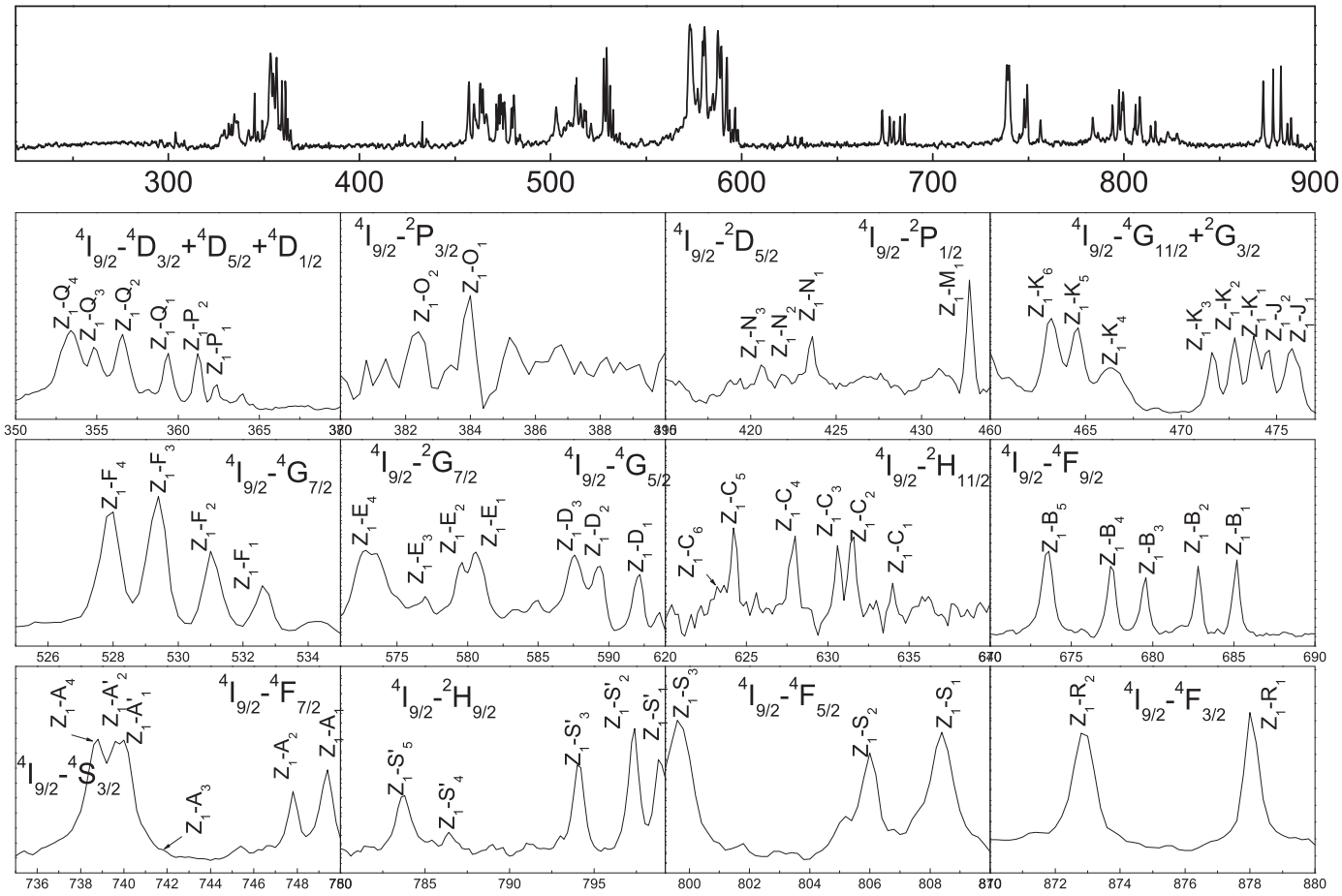

Fig. 6. Excitation spectrum and transition assignment of Nd:$YNbO_4$ at 8 K (λem=1066 nm).

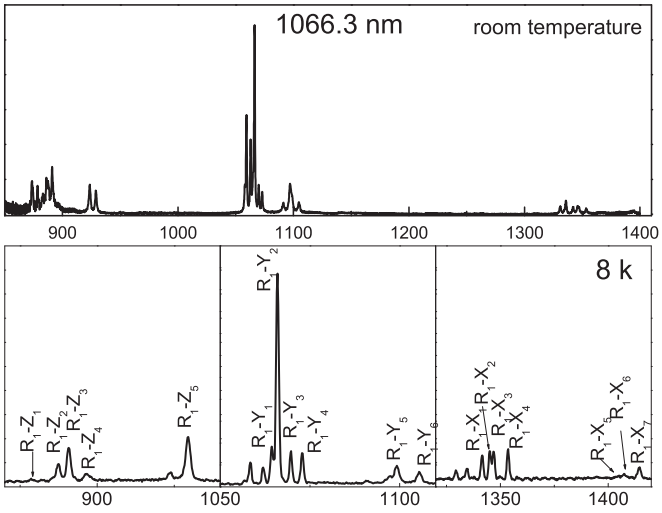

Fig. 7. Emission spectrum and transition assignment of Nd:$YNbO_4$ under 808 nm excitation at 8 K and room temperature.

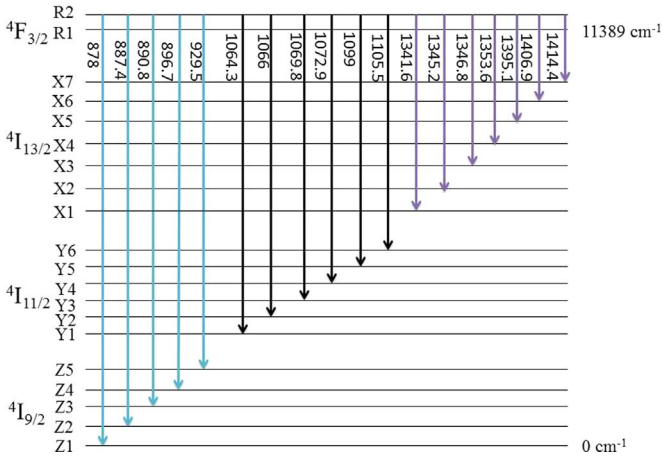

Fig. 8. A schematic diagram of the transitions $^4F_{3/2}$→$^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ of Nd^{3+} in $YNbO_4$.

and the rotation speed was 5.0–15.0 rpm. After growth, the crystal was cooled down to room temperature at a speed of 30–50 ℃/h. Generally, the optical and physical properties should be characterized individually along three crystallographic axes for monoclinic system. However, unfortunately, we have only succeeded in determining the crystallographic *b* axis as no big crystal was obtained, up to now. But the study in this work is still of significance and reproducibility for a new crystal.

2.2. Characterization

A Philips X'pert PRO X-ray powder diffractometer equipped with Cu Kα radiation was used to measure the structure of Nd:$YNbO_4$ crystal at room temperature. A scan step of 0.02° was applied to record the patterns in the 2θ range of 10–90°. X-ray rocking curve was collected by X'pert Pro MPD diffractometer equipped with a Hybrid Kα1monochromatorat room temperature. A HV-1000A micro hardness tester was employer to measure the hardness of the as-grown crystal. A Perkin-Elmer UV–VIS–NIR lambda-950 spectrophotometer was employed to measure the transmission spectrum of Nd:$YNbO_4$ crystal in

Table 2
Energy levels of Nd^{3+} in $YNbO_4$.

$^{2S+1}L_J$		E (cm^{-1})	$^{2S+1}L_J$		E (cm^{-1})
$^4I_{9/2}$	Z_1	0		C_4	15,926
	Z_2	120		C_5	16,019
	Z_3	163		C_6	16,044
	Z_4	237	$^4G_{5/2}$	D_1	16,804
	Z_5	631		D_2	16,964
$^4I_{11/2}$	Y_1	1993		D_3	17,020
	Y_2	2008	$^2G_{7/2}$	E_1	17,224
	Y_3	2041		E_2	17,250
	Y_4	2068		E_3	17,331
	Y_5	2290		E_4	17,455
	Y_6	2343	$^4G_{7/2}$	F_1	18,774
$^4I_{13/2}$	X_1	3935		F_2	18,830
	X_2	3955		F_3	18,890
	X_3	3964		F_4	18,942
	X_4	4001	$^2D_{3/2}$	J_1	21,018
	X_5	4221		J_2	21,074
	X_6	4281	$^4G_{11/2}$	K_1	21,105
	X_7	4319		K_2	21,151
$^4F_{3/2}$	R_1	11,389		K_3	21,201
	R_2	11,456		K_4	21,444
$^4F_{5/2}$	S_1	12,370		K_5	21,526
	S_2	12,407		K_6	21,589
	S_3	12,505	$^2P_{1/2}$	M_1	23,105
$^2H_{9/2}$	S'_1	12,518	$^2D_{5/2}$	N_1	23,611
	S'_2	12,542		N_2	23,702
	S'_3	12,593		N_3	23,772
	S'_4	12,716	$^2P_{3/2}$	O_1	26,048
	S'_5	12,759		O_2	26,156
$^4F_{7/2}$	A_1	13,344	$^4D_{3/2}$	P_1	27,598
	A_2	13,373		P_2	27,682
	A_3	13,481	$^4D_{5/2}$	Q_1	27,827
	A_4	13,537		Q_2	28,044
$^4S_{3/2}$	A'_1	13,512		Q_3	28,174
	A'_2	13,521	$^4D_{1/2}$	Q_4	28,301
$^4F_{9/2}$	B_1	14,595			
	B_2	14,645			
	B_3	14,716			
	B_4	14,761			
	B_5	14,847			
$^2H_{11/2}$	C_1	15,773			
	C_2	15,835			
	C_3	15,857			

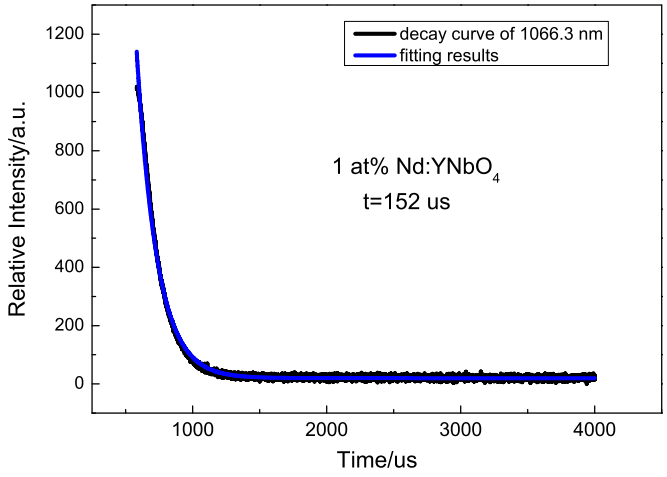

Fig. 9. Fluorescence decay curves of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+} in $Nd:YNbO_4$.

the wavelength range of 320–2000 nm, the slice for measurement was cut and polished perpendicular to *b* axis, as shown in Fig. 1. The fluorescence spectra of $Nd:YNbO_4$ were recorded by a Edinburgh FLSP920 spectrometer with an excitation source of Xenon lamp. The transmission spectrum was measured at room temperature and the fluorescence spectrum was recorded at 8 K temperature..

Table 3
The comparison of $Nd:YNbO_4$ crystal with other Nd^{3+}-doped crystals.

Crystals	FWHM (808 nM)	σ_α (10^{-20} cm^2) (808 nm)	τ (μs)	Ref.
$Nd:YNbO_4$	7	8.9	152	This work
$Nd:GdTaO_4$	6	5.1	178	[9]
Nd:YAG	2	8.3	230	[10]
Nd:LYSO	5	6.1	226	[25]

3. Results and discussion

3.1. Crystal structure and quality

Crystal density and hardness are important physical parameters for investigating a new crystal. The density of $Nd:YNbO_4$ crystal is calculated to be 5.4 g/cm^3 by using Archimedes drainage method, which is very close to the theoretical density calculated by the lattice constant (5.43 g/cm^3). The Vickers hardness measurement was repeated five times and an average was taken. The values determined on (010) faces was 720 HV. The Mohr's hardness was obtained to be 6.0 by using formula $H_M=0.675(H_V)^{1/3}$ [20] The XRD diffraction patterns of $Nd:YNbO_4$ crystal and $YNbO_4$ standard patterns (ICSD # 20335) are shown in Fig. 2. The position and shape of the diffraction peaks of the sample can be well consistent with the $YNbO_4$ standard card, which indicates that the as-grown crystal have the same structure with the M-type $YNbO_4$. The structure of $Nd:YNbO_4$ was refined by using the general structure analysis system (GSAS) software [21], according to the XRD data. Using the structure data of $YNbO_4$ (ICSD # 20335) as initial values, the Rietveld refinement results of $Nd:YNbO_4$ are shown in Fig. 3 and Table 1. The residuals Rp and Rwp are 6.7% and 8.8%, respectively, which are both less than 10%, indicating the refinement results reliable. The X-ray Rocking curves of the (010) diffraction face of $Nd:YNbO_4$ crystal is shown in Fig. 4. The diffraction peak shows a symmetric shape without split and the full width at half maximum (FWHM) is 0.05°, which indicates a high crystalline quality of the as-grown crystal....

3.2. Transmission spectrum

The transmission spectrum of $Nd:YNbO_4$ crystal in the range of 320–2000 nm at room temperature is shown in Fig. 5. There is a strong absorption band centered at 809.3 nm. The FWHM of this band is 7 nm and the transmission rate of this absorption is 22.2%. In transmission spectrum, the maximum transmission rate of the sample is 76.3%. According to the refractive index of $Nd:YNbO_4$ reported in Ref. [22], the absorption coefficients of $Nd:YNbO_4$ crystal can be calculated using the following formula

$$\alpha=-\frac{1}{d}\ln\frac{-(1-R)^2+\sqrt{(1-R)^4+4R^2T^2}}{2R^2T} \quad (1)$$

Where *d* is the wafer thickness, *T* is the transmission ratio, *R* is the reflectivity and $R=(n-1)^2/(n+1)^2$, in which *n* is the refractive index. The absorption coefficient at 809.3 nm is calculated to be 6.0 cm^{-1}. The absorption cross section σ_α can be calculated by $\sigma_\alpha=\alpha(\lambda)/N$, where α is the absorption coefficient and *N* is the concentration of Nd^{3+} in $Nd:YNbO_4$ crystal. The segregation coefficient of Nd ion is approximately 0.6 [23]. Therefore, the absorption cross section σ_α of 1 at% Nd-doped $YNbO_4$ is calculated to be 8.9×10^{-20} cm^2..

3.3. Luminescence properties

3.3.1. Excitation spectrum of $Nd:YNbO_4$

The excitation spectrum of $Nd:YNbO_4$ at 8 K temperature is shown in Fig. 6. There are 14 excitation bands in Fig. 6, which are corresponding to the characteristic excitation transitions of Nd^{3+} from

the ground state ($^4I_{9/2}$) to different excitation states. According to the position of the excitation peaks, the transitions from the ground state to different excitation states were assigned in Fig. 6. The assigned results are very closed to those of Nd:$GdTaO_4$ which are also with monoclinic system [24]..

3.3.2. Emission spectrum of Nd:$YNbO_4$

The emission spectrum (λ_{ex}=808 nm) of Nd:$YNbO_4$ in the range of 850–1400 nm is shown in Fig. 7. The emission spectra are measured at 8 K and room temperature, and 8 K spectrum is more accurate than that of measured at room temperature. The characteristic emission peaks under 808 nm excitation are corresponding to the transitions of Nd^{3+} from $^4F_{3/2}$ level to $^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ levels, of which the emission peak at 1066.3 nm of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition is the strongest. Based on the room temperature emission spectrum, the emission cross section σ_{em} was estimated to be 29×10^{-20} cm^2 by using the Füchtbauer-Ladenburg (F-L) formula [25]. The emission peaks under 8 K are assigned. A schematic diagram of the transitions $^4F_{3/2}\rightarrow{}^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ of Nd^{3+} are shown in Fig. 8...

3.3.3. Crystal-field analysis

The excitation and emission spectra at 8 K were fitted by Lorenz function , which is usually used to identify the peaks location. Then the Stark energy levels of Nd^{3+} in $YNbO_4$ were calculated according to the fitted peaks position, the calculated results are shown in Table 2. All the obtained spectral and Stark energy levels have an important reference for the research and evaluation of the laser properties of Nd:$YNbO_4$ crystal.

3.3.4. Fluorescence decay curve of Nd:$YNbO_4$

The fluorescence decay curve of the $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition excited by 808 nm is shown in Fig. 9. The decay curve can be well fitted with a single exponential decay function, and the fluorescence lifetime is fitted to be 152 μs, which is longer than that of Nd:YVO_4 crystal [10]. The comparison of Nd:$YNbO_4$ crystal with other Nd^{3+}-doped crystals is shown in Table 3. As can be seen, Nd:$YNbO_4$ crystal shows good comprehensive performance. The small emission cross section and long fluorescence lifetime of Nd:$YNbO_4$ indicates that it has a high energy storage capacity. In a word, Nd:$YNbO_4$ is a promising material can be applied in Q-switched laser..

4. Conclusions

A Nd:$YNbO_4$ single crystal was grown successfully by Cz method. The density of the as-grown crystal is measured to be 5.4 g/cm^3 by using Archimedes method. The Mohr′s hardness was determined to be 5.7. The FWHM of the X-ray Rocking curve diffraction peak of (010) face is 0.05°, indicating a high crystalline quality of the as-grown crystal. The structure of Nd:$YNbO_4$ was refined using Rietvield refinement method and its lattice parameters and atomic coordinates were obtained. A maximum transmittance of Nd:$YNbO_4$ is 76.3% in transmission spectrum and the transmittance at 809.3 nm is 22.3%. The absorption coefficient of Nd:$YNbO_4$ crystal along (010) face at 809.3 nm is calculated to be 6.0 cm^{-1}. The excitation and emission peaks at 8 K were assigned and the crystal-field splitting energy levels of Nd^{3+} in $YNbO_4$ crystal were obtained. The fluorescence lifetime of the $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition of Nd^{3+} in Nd:$YNbO_4$ is fitted to be 152 μs. All of the results have an important reference for exploring the Nd:$YNbO_4$ as a novel laser crystal.

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