CrystEngComm

Accepted Manuscript

ROYAL SOCIETY OF CHEMISTRY

www.rsc.org/crystengcomm

Synthesis, Growth, Defects and Laser Action of Yb: $Y_{0.71}Lu_{0.29}VO_4$ Crystals

Degao Zhong [1,2*], *Bing Teng* [1,2*], *Lifeng Cao* [1,2], *Shiming Zhang* [1,2], *Fei You*[1,2], *Bingtao Zhang* [1,2], *Jianhong Li* [1,3], *Xueping Tian* [1,2], *Chao Wang*[1,2], *Yuyi Li* [1,2], *Linxiang He*[4], *Romano A. Rupp*[5,6]

[1] College of Physics, Qingdao University, Qingdao, 266071, China

[2] Key Laboratory of Photonics Materials and Technology in Universities of Shandong (Qingdao University) , Qingdao, 266071, China

[3] CRYSTECH Inc., 266107, Qingdao, China

[4] Department of Physics and Materials Science, City University of Hong Kong, Kowloon, Hong Kong

[5] TEDA Applied Physics School, Nankai University, Tianjin 300457, China

[6] Faculty of Physics, Vienna University, A-1090 Wien, European Union

Abstract

Polycrystalline materials of $REVO_4$ (RE=Y, Lu, Yb) were synthesized by liquid-phase reaction. An Yb doped Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal was grown by Czochralski method. Dislocations and low angle boundaries were revealed by chemical etching and transmission electron microscope (TEM). The continuous-wave (cw) laser action was achieved in the range of 1020.7–1029.0nm by using a high-power diode laser as the pump source. cw laser output power of 1.09W with a slope efficiency of 21.6% was demonstrated for this 0.5 at. % Yb-doped Yb:$Y_{0.79}Lu_{0.21}VO_4$ mixed crystal.

Keywords: Czochralski method; liquid-phase reaction; laser crystal; crystal defects

Introduction

Yb-doped single vanadates, such as Yb:YVO_4, Yb:$GdVO_4$, and Yb:$LuVO_4$, have been identified as promising materials for diode-pumped solid-state lasers due to their good laser properties and high chemical

---

*Corresponding author's e-mail address: qduzhdg@163.com; 5108tb@163.com; Tel: 86-10-85953405; Fax: 86-10-85955977.

stability[1-5]. Since YVO_4, $LuVO_4$ and $GdVO_4$ crystals possess the same crystal structure and close melting points, mixed vanadates of $Y_xLu_{1-x}VO_4$, $Y_xGd_{1-x}VO_4$ and $Lu_xGd_{1-x}VO_4$ crystals can be produced by Czochralski method. Consequently, mixed Y, Lu and Gd vanadates were also tested as Nd or Yb host materials with the aim to improve laser performance [6-8]. Research on Yb: $Gd_{0.64}Y_{0.36}VO_4$ crystal showed that the mixed crystal demonstrated efficient cw and mode-locked laser operation [9]. Moreover, Liu et.al. [10] have conducted a comprehensive investigation to compare laser performance of isostructural Yb-doped vanadates, including the three crystals Yb: YVO_4, Yb: $GdVO_4$, and Yb: $LuVO_4$ and two mixed ones with compositional disorder, namely $Yb_{0.007}$:$Y_{0.407}Gd_{0.586}VO_4$ and $Yb_{0.015}$: $Lu_{0.52}Gd_{0.465}VO_4$. In comparison with the three ordered vanadates, the mixed crystals turned out to be inferior for power scaling. In a disordered mixed crystal host the local crystal field of the activation ions Yb may be variable. The random distribution of the Lu, Gd or Y ions neighboring the Yb activation ions will lead to inhomogeneous broadening of the spectral lines in the mixed crystal and influence the laser action. However, the knowledge of fundamental properties of Yb-doped mixed vanadates are still far less sufficient, and much work needs to be conducted on the improvement of the crystal quality, the optimization of the Yb doping level and the mixed proportion of Y and Lu [10].

Based on a summary of published literature, we found that only mixed crystals of Yb: $Y_xGd_{1-x}VO_4$ and Yb: $Lu_xGd_{1-x}VO_4$ have been grown and studied. Information on growth, crystal defects and laser action of Yb: $Y_xLu_{1-x}VO_4$ is still lacking. The Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal is an isomorph of Yb:YVO_4 and Yb:$LuVO_4$. It is anticipated that substituting a fraction of Lu ions with Y ions may produce mixed crystal with even more promising laser properties. It is well known that the main difficulty in the applications of rare earth vanadates is obtaining crystals with low concentration of defects. Crystal defects, such as dislocations, low-angle grain boundaries and inclusions, have been revealed in ordered crystals of Nd:YVO_4 and Nd:$GdVO_4$ crystals by X-ray topography, TEM and chemical etching [11-14]. However, crystal defects in mixed rare earth orthovanadates have seldomly been investigated. In this paper, we try to shed some light on the crystal defects and laser properties of Yb: $Y_xLu_{1-x}VO_4$ crystals.

Pure raw materials are important for obtain the high-quality Yb:$Y_xLu_{1-x}VO_4$ crystals. In general, polycrystalline materials of $REVO_4$ (RE=Yb, Lu and Y) are synthesized by means of solid- and liquid-phase reactions [14, 15]. However, it is difficult to achieve highly pure polycrystalline materials of $ReVO_4$ by solid-phase reaction. In order to obtain pure raw materials, we synthesized the polycrystalline materials of $REVO_4$ (RE=Y, Yb, Lu) separately using liquid-phase reaction. A Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal was grown by Czochralski method and studied.

2. Experimental

2.1 Preparation of the polycrystalline materials

$YbVO_4$, YVO_4, and $LuVO_4$ were synthesized by chemical precipitation method in aqueous solution liquid-phase reaction [14-16] from the starting powders RE_2O_3 (99.9999%, RE=Yb, Lu and Y) and NH_4VO_3 (99.5%). Firstly, a NH_4VO_3 solution was prepared by dissolving NH_4VO_3 powder in double-distilled water at nearly 100°C. Extraneous solids and colloidal particles were removed by filters with a pore diameter of 0.22 µm. $RE(NO_3)_3$ solutions were prepared by dissolving RE_2O_3 in nitric acid (6mol/L). Secondly, NH_4VO_3 and $RE(NO_3)_3$ solutions were diluted by hot double-distilled water (about 80°C) and mixed together in a flask equipped with a stirrer, and the mixed solution adjusted to pH 7.0 by using ammonia. Then a great amount of white precipitation of $REVO_4$ emerges in mixed solution. The chemical reaction equations are as follows:

$$RE_2O_3+6H^+=2RE^{3+}+3H_2O,$$

$$VO_3^-+RE^{3+}+2NH_3\cdot H_2O= REVO_4\downarrow+2NH_4^++ H_2O.$$

Thirdly, the $REVO_4$ precipitations were released from solution and washed three times using ethanol (G.R. purity) and secondary distilled water, respectively. The $REVO_4$ polycrystalline materials were dried and sintered at 800-1300 ºC for 8h in air and finally the white $REVO_4$ powder was harvested.

2.2 Crystal growth of Yb: $Y_{0.71}Lu_{0.29}VO_4$ mixed crystal

The starting composition of the mixture was M_{YbVO4}: M_{YVO4}: M_{LuVO4}=1:215:60 (by mass). Generally the chemical composition is not identical in the feeding powder mixture and in the grown crystal. And the actual composition of the grown crystal should be determined by accurate measurement after crystal growth. Single crystals were grown by the Czochralski method using a 2.5 kHz radio medium-frequency generator heating an iridium (Ir) crucible containing polycrystalline material. The size of the Ir crucible is 55 mm in diameter and 40 mm in height. The crystal was grown in an atmosphere of nitrogen containing ~1% oxygen by volume (The exist of small amount of oxygen can reduce the oxygen vacancy defects in grown crystal, and Ir crucible can also be protected from oxidation). Rectangular single crystal bars of pureYVO_4 (a-axis), with the dimensions of 4×4×30 mm^3, were used as seeds. The pulling rate was about 1-2mm/h after the size of crystal diameter reached a value of about 20mm. The crystal was rotated at rates of 10-15 rpm during growth. After growth, the crystal was lifted above the surface of the melt, and annealed in-situ at five temperature ranges. After higher temperature annealing treatment, the stresses that were introduced into crystal by the temperature fluctuations during crystal growth could be reduced.

$$\sim 1400°\mathrm{C} \xrightarrow{15°\mathrm{C/h}} 1200°\mathrm{C} \xrightarrow{18°\mathrm{C/h}} 1000°\mathrm{C} \xrightarrow{40°\mathrm{C/h}} 600°\mathrm{C} \xrightarrow{60°\mathrm{C/h}} 300°\mathrm{C} \xrightarrow{100°\mathrm{C/h}} Room-temperature$$

Inductively coupled plasma atomic emission spectrometer (ICP-AES) (Ultima2, Jobin Yvon Corp. France) analysis method was used to determine the concentrations of Yb, Y, and Lu in the Yb: $Y_xLu_{1-x}VO_4$ mixed crystals. The results show that the Yb concentration in mixed crystal is about 0.5 at%, and x is 0.71.

2.3 XRD and FT-IR spectra characterization of Yb:$Y_{0.79}Lu_{0.21}VO_4$ crystal

X-ray powder diffraction was used to study the crystal structure of the raw materials and of the grown crystal at room temperature. It was performed with a D/Max-rA (Rigaku, Japan) automatic X-ray diffractometer using nickel-filtered Cu Kα irradiation. The ground fine powders were scanned in steps of 1 °/min over a 2θ range of 10–80°. FT-IR spectra of Yb:$Y_{0.79}Lu_{0.21}VO_4$ was recorded on a Nicolet 5700 Spectrometer.

2.4 Characterization of crystal defects

(001) and (100) slices with a thickness of about 200μm were cut from a Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal and polished to serve as samples for chemical etching. A mixture $V_{\text{concentrated hydrochloric acid (36\%)}}$:$V_{\text{hydrofluoric acid (47\%)}}$ =10:1 (by volume) was selected as etchant of the (001) slices while a mixture $V_{\text{concentrated hydrochloric acid (36\%)}}$:$V_{H2O}$ =2:3 was selected as etchant for the (100) slices. The detailed etching conditions for different facets are listed in table 1. The etch pits were imaged with a BX51 Olympus optical microscopy. Conventional TEM specimen preparation methods were used, perforation being achieved using a solution of $V_{\text{concentrated sulfuric acid (98\%)}}$:$V_{\text{concentrated phosphoric acid (85\%)}}$ =1:1 (by volume) at 400 K [11]. A Final cleaning was performed by ion milling using 4.5 keV Ar^+ for 1h. The samples were analyzed using a JEM-2100F TEM operated at 200kV.

2.5 cw laser performance of Yb: $Y_{0.71}Lu_{0.29}VO_4$ mixed crystal

Samples were cut and polished to serve for laser performance measurement. In order to study the cw laser performance of Yb: $Y_{0.71}Lu_{0.29}VO_4$ mixed crystal, a simple plano-concave resonator was employed. The plane mirror was coated highly reflecting for 1030-1200 nm and highly transmitting for □820-990 nm. As output coupler, a concave mirror of 25mm radius-of-curvature was utilized, with transmission T=1% for the lasing wavelength around 1030 nm. An uncoated Yb: $Y_{0.71}Lu_{0.29}$ VO_4 crystal with the size of 3×3×2 mm^3 was fixed in a water-cooled copper holder and placed close to the plane mirror inside the cavity. The cavity length was about 25 mm. The pump source was a high brightness fiber-coupled diode (fiber core diameter of 200 μm and NA of 0.22) emitting unpolarized radiation at 974–986 nm depending on the output level [10].

3. Results and discussion

Fig. 1 shows the XRD patterns of YVO_4 powers sintered at various temperatures for 8h in air. According to Committee on Power Diffraction Standards (JCPDS) card 17-0341, YVO_4 has a tetragonal zircon structure ($ZrSiO_4$, space group $D_{4h}^{19} - I_{41}/amd$) and its unit cell parameters are a=b= 7.119 Å, c = 6.289 Å. As shown in Fig. 1, all of the diffraction peaks of the obtained powers are in good agreement with tetragonal crystalline phase YVO_4 (JCPDS 17-0341). No additional peaks are found. The peak position did not change with the change of sintering temperatures, but the diffraction peaks became sharp and the intensity increased dramatically at 1200 ºC, indicating good crystallinity at this temperature.

Fig. 2 shows SEM micrographs of pure YVO_4 powders obtained at various sintering temperatures for 8h in air. From the pictures we can observe that the average sizes of particles increases with sintering temperature. YVO_4 crystal has a tetragonal structure and it usually grows in natural morphology with prism and pyramidal sectors just as clearly shown in the inset of top left corner in fig.2. From the pictures in fig.2, we can see that YVO_4 powders clearly take the shape of prism plus pyramid when the sintering temperature is up to 1200 ºC. This indicates that YVO_4 powders are well crystallized at 1200 ºC, which is in good agreement with the XRD result.

Fig.3 presents the XRD pattern of Yb: $Y_{0.71}Lu_{0.29}VO_4$. As shown in Fig. 3, it exhibits a XRD pattern similar to those of the tetragonal YVO_4 (JCPDS data No. 17–0341) and $LuVO_4$ (JCPDS data No. 17–0880), which belong to the same tetragonal zircon structure. The corresponding peak locations in the XRD pattern of Yb: $Y_{0.71}Lu_{0.29}VO_4$ shift gradually between those of the YVO_4 and $LuPO_4$. It is believed that the differences of ionic radius between Y^{3+} and Lu^{3+} should account for the small shift of the peak locations in the XRD patterns of the Yb:$Y_{0.71}Lu_{0.29}VO_4$.

Fig. 4 shows the photo of sliced samples of Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal with the size of 3×3×2 mm^3(b×c×a). The laser performance of yttrium lutetium mixed vanadate was studied using these 2 mm thick a-cut crystals. Shown in Fig.5a is the laser emission spectrum, recorded at an absorbed pump power of 3.51 W for crystal Yb: $Y_{0.71}Lu_{0.29}VO_4$. As can be seen from Fig.5a, free running laser oscillation takes place at many different wavelengths, covering a range of 1020.7–1029.0nm. Fig. 5b shows the output power as a function of absorbed pump power for output coupling of T=1%, obtained with the 2-mm-thick a-cut Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal. The output power scales almost linearly in the power range of P_{abs}>3.0W. The slope efficiency of laser operation was measured to be 21.6%, and a maximum output power of 1.09 W was obtained at P_{abs}=6.99W, resulting in an optical-to-optical efficiency of 15.7%. Laser thresholds for T=1% was estimated to be 1.21 W of absorbed pump power. Table2 presents the Parameters of the cw laser performance of different Yb-doped vanadate mixed crystals, obtained in this work and orther literatures. As can be seen from Table2, though a maximum output power of 1.09 W was achieved, the slope efficiency is still low. This indicated that there were local areas with serious defects in mixed crystals and the crystal quality needed to be further improved.

Fig. 6 compares FT-IR spectra of Yb: YVO_4 and Yb:$Y_{0.79}Lu_{0.21}VO_4$ crystals. The peaks related to Yb:$Y_{0.79}Lu_{0.21}VO_4$ appear to be very similar with Yb: YVO_4. A strong and broad absorption peak centered at 822cm^{-1} and a weak peak centered at 451cm^{-1} have appeared, which are attributed to the vibration modes of the V–O bond and the Y(Yb, Lu)–O bond, respectively [18,19]. The peaks attributed to the O–H stretching vibration and H–O–H bending vibrational modes are due to absorption of moisture on the surface of the crystal powders. By comparing FT-IR spectra of Yb: YVO_4 and Yb:$Y_{0.79}Lu_{0.21}VO_4$ crystals, there was no notable spectral shift in the peak position of M–O bond. The small shift in the peak position of V-O bond towards the lower wavenumber side of Yb:$Y_{0.79}Lu_{0.21}VO_4$ might be due to the influence of Lu in the mixed crystal. Furthermore, the FT-IR spectral measurement certifies the pure vanadate phase formation, agreeing well with the obtained XRD results.

Fig.7 presents etching micrographs of (100) and (001) slices. From Fig.7a and Fig.7b, etching pits on the (001) faces were observed to be square, unlike those on the (100) faces which took the shape of rectangle. But the corresponding edges of each etching pit were parallel to each other whether on (100) or (001) face.

Usually, these etching pits are considered as outcrop of dislocation lines. The shape of the etching pits is determined by the lattice structure and symmetry [20]. The interesting phenomenon is that several arrays of dislocation pits are observed on the (001) face and some arrays of dislocation pits twist and intersect each other, as shown in fig.7c and 7d. The same phenomena had also been observed in Nd:GdVO$_4$ [14,17] and Nd:YVO$_4$ crystals[21]. Generally, the formation of arrays of dislocation pits is related to low angle grain boundaries in grown crystals. The corresponding dislocation models of crystal grain boundaries have been established by W.T.Read and W. Shockley [22]. It is believed that grain boundaries of the form considered may permit intercrystalline slip and may act as stress raisers for the regeneration of dislocations.

Chemical etching, TEM and X-ray topographic studies on Nd:YVO$_4$ and Nd:GdVO$_4$ crystals revealed that dislocations were primarily located at low-angle grain boundaries [12,14]. As shown in fig.8, we also observed periodic arrays of dislocations in Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$ crystals by bright field TEM image. It has been suggested that low-angle grain boundaries form due to migration of dislocations introduced during crystal growth and cooling process [20]. Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$, YVO$_4$ and GdVO$_4$ possess the same tetragonal zircon structure. Fig.9 shows the 3D structure of Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$ crystal viewed along the c-axis and b-axis. As can be seen from Fig.9, the (100) face has high atomic density and large interplanar distances. The atomic bonding forces are strong within the (100) faces but weak between (100) faces. Consequently, the crystal can easily be cleaved along the (100) face. And low-angle grain boundaries nearly parallel to the (100)-face occur easily in bulk crystals grown along the *a*-axis. Moreover, the difference of the ionic radius between Y^{3+} and Lu^{3+} can also induce lattice mismatches and stresses in mixed crystals. Therefore, several arrays of dislocation pits are observed on (001) face. All these analyses show that there were still lots of dislocation defects and a certain amount of grain boundaries in grown Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$ crystals. If the crystal quality is further improved, the laser performance will be upgraded.

4. Conclusions

Optimal conditions to synthesis the raw materials of YVO$_4$ using liquid-phase reaction were discussed. A new mixed laser crystal of composition Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$ was grown by Czochralski method. Dislocations and low angle grain boundaries were revealed by chemical etching. cw laser output power of 1.09W with a slope efficiency of 21.6% was demonstrated for a 0.5 at. % Yb-doped Yb:Y$_{0.79}$Lu$_{0.21}$VO$_4$ mixed crystal.

In a disordered mixed crystal host, the local field of the Yb^{3+} activation ions can be changed, which may benefit the spectra and laser action of mixed crystal and bring better performance. At the same time, lattice mismatches and stresses are more likely to arise due to the difference of the ionic radius between Y^{3+} and Lu^{3+}, which may result in defects formation such as dislocations and low angle grain boundaries. Nevertheless, we believe that Yb:$Y_xLu_{1-x}VO_4$ will be a promising laser material if more effort is devoted to improve the crystal quality.

Acknowledgements

Financial support from the National Natural Science Foundation of China (Grant No.51172111, No. 11204148, No.11374170 and No. 60978023) and promotive research fund for excellent young and middle-aged scientists of Shandong Province (Grant No. BS2013CL024) are gratefully acknowledged.

References

[1] V. E. Kisel, A. E. Troshin, N. A. Tolstik, V. G. Shcherbitsky, N. V. Kuleshov, V. N. Matrosov, T. A. Matrosova, M. I. Kupchenko, Opt.Lett., 2004, **29**, 2491.

[2] C. Kränkel, D. Fagundes-Peters, S. T. Fredrich, J. Johannsen, M. Mond, G. Huber, M. Bernhagen, R. Uecker, Appl. Phys. B, 2004, **79**, 543.

[3] A. A. Lagatsky, A. R. Sarmani, C. T. A. Brown, W. Sibbett, V. E. Kisel, A. G. Selivanov, I. A. Denisov, A. E. Troshin, K. V. Yumashev, N. V. Kuleshov, V. N. Matrosov, T. A. Matrosova, and M. I. Kupchenko, Opt.Lett., 2005, **30**, 3234.

[4] T. Jensen , V. G. Ostroumov, J.-P. Meyn, G. Huber, A. I. Zagumennyi, I. A. Shcherbakov, Spectroscopic Characterization and Laser Performance of Diode-Laser-Pumped Nd: GdVO4, Appl. Phys. B, 1994, **58**, 373.

[5] J.H. Liu, X. Mateos, H.J. Zhang, J.Y. Wang, M.H. Jiang, U. Griebner, V. Petrov, Opt.Lett., 2005, **30**, 3162.

[6] S.V. Garnov, S.A. Solokhin, E.D. Obraztsova, A.S. Lobach,P.A. Obraztsov,A.I. Chernov, V.V. Bukin, A.A. Sirotkin, Y.D. Zagumennyi, Y.D. Zavartsev, S.A. Kutovoi and I.A. Shcherbakov, Laser Physics Letters, 2007, **4**, 648.

[7] H. H. Yu, H. J Zhang, Z. P. Wang, J. Y. Wang, Y. G. Yu, D.Y. Tang, G.Q. Xie, H. Luo, M. H. Jiang, Opt. Express, 2009, **17**, 3264.

[8] B.Zhao, N. F. Zhuang, T. Li, F. Y. Guo, Z. Zhuo, J. Ye, J.Z. Chen, J. Cryst. Growth, 2011, **335**, 115.

[9] V.E. Kisel, N.A. Tolstik, A.E. Troshin, N.V. Kuleshov, V.N. Matrosov, T.A. Matrosova, M.I. Kupchenko, F. Brunner, R. Paschotta, F. Morier-genoud, U. Keller, Appl. Phys. B, 2006, **85**, 581.

[10] J. H. Liu, W.J. Han, H. J. Zhang, X. Mateos, V. Petrov, IEEE J. Quantum Elect., 2009, **45**, 807.

[11] D.E. Eakins, J.B. LeBret, M.G. Norton, D.F. Bahr, J. Cryst. Growth, 2004, **266**, 411.

[12] E. Wierzbicka, A. Malinowska, K. Wieteska, W. Wierzchowski, M. Lefeld-Sosnowska, M. Swirkowicz, T. Lukasiewicz, C. Paulmann, ACTA Phys. Pol. A, 2012, **121**, 906.

[13] W. Paszkowicz, P.Romanowski, J.Bak-Misiuk, W.Wierzchowski, K.Wieteska, W.Graeff, R.J. Iwanowski, M.H.Heinonen, O.Ermakova, H.Dabkowska, Radiat. Phys. Chem., 2011, **80**, 1001.

[14] L.J. Qin, X.L. Meng, J.G. Zhang, L. Zhu, H.J. Zhang,B.C. Xu, H.D. Jiang, J. Cryst. Growth, 2002, **242**, 183.

[15] L.Z. Zhang, G.F. Wang, S. K. Lin, J. Cryst. Growth, 2002, **241**, 325.

[16]J.Z. Chen, F.Y. Guo, N.F. Zhuang, J.M. Lan,X.L. Hu, S.K. Gao, J. Cryst. Growth, 2002, **243**, 450.

[17] Y.G. Yu, H.J. Zhang, J.Y. Wang, X.B. Hu, H.H. Yu, Investigation on Defects in Yb:GdVO4 Crystal, J. Cryst. Growth, 2007, **25**, 494.

[18] H.X. Peng, G.X. Liu, X.T. Dong, J.X.Wang, J. Xu, W.S. Yu, J. Alloy. Compd., 2011, **509**, 6930.

[19] H. Wang, M. Yu, C.K. Lin, J. Lin, J. Colloid. Interf. SCI, 2006, **300**, 176.

[20] K. Sangwal, I. Owczarek, J. Cryst. Growth, 1993, **129**, 648.

[21] J.B. Lebret, M. G. Norton, D.F. Bahr, D.P. Field, K.G. Lynn, J. Mater. SCI, 2005, **40**, 3347.

[22] W.T.Read, W. Shockley, Phys. Rev., 1950, **78**, 275.

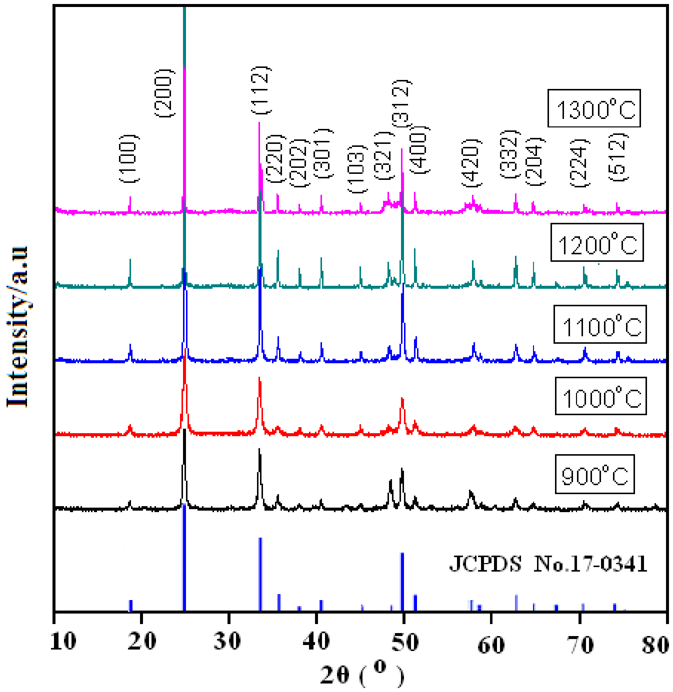


Fig. 1 XRD patterns of YVO_4 powers sintered at various temperatures

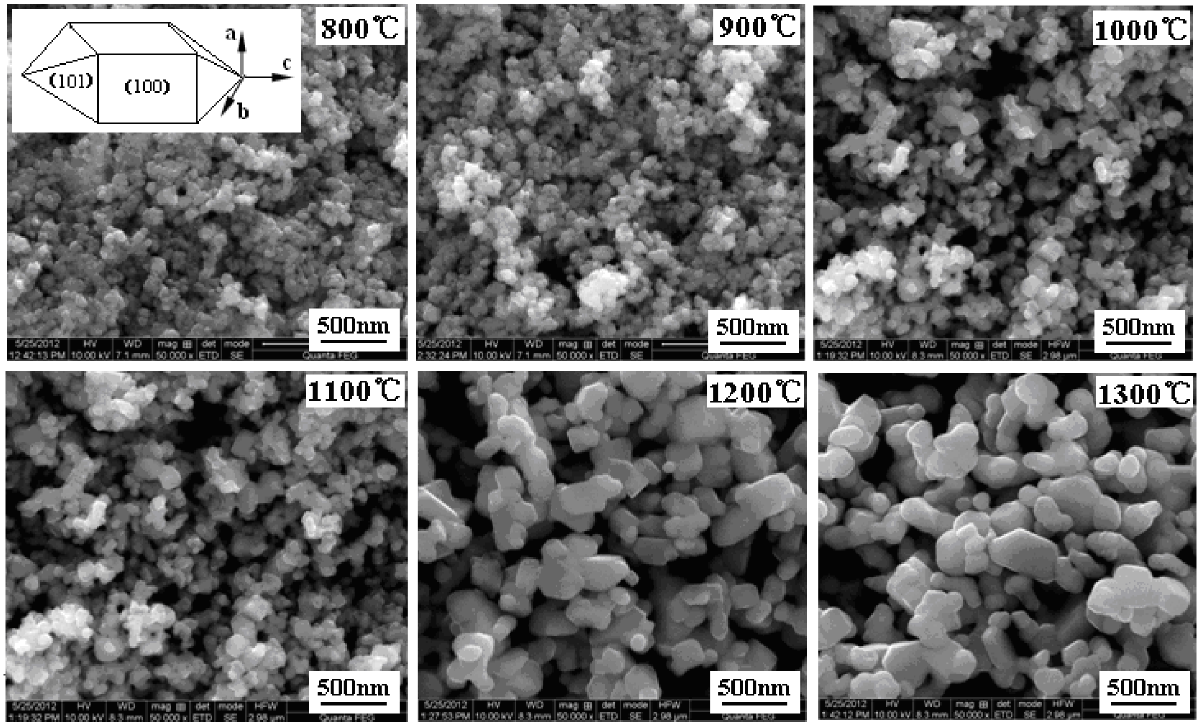


Fig. 2 SEM micrographs of pure YVO_4 powders obtained at various sintering temperatures

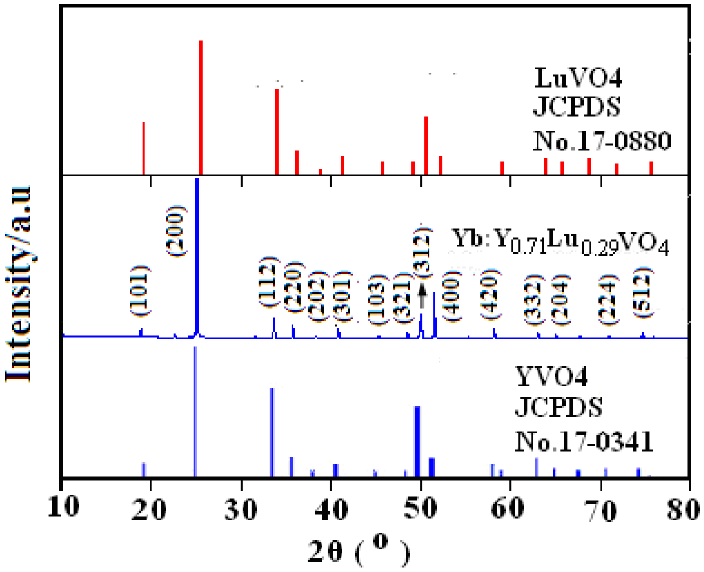


Fig.3 XRD pattern of Yb: $Y_{0.71}Lu_{0.29}VO_4$

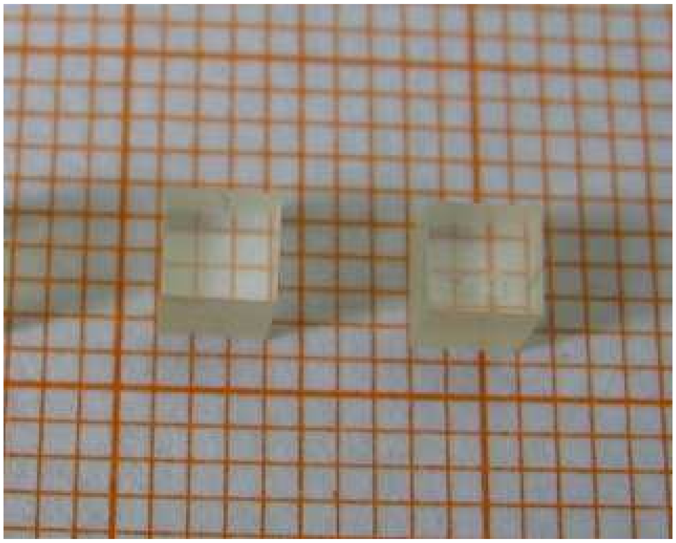
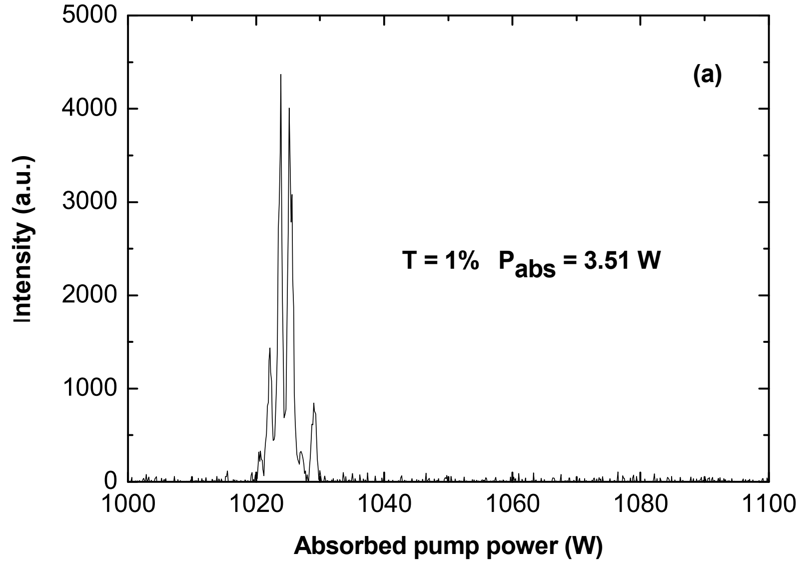


Fig.4 Sliced samples of Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal


(a)
T = 1% P_{abs} = 3.51 W
Intensity (a.u.)
0
1000
2000
3000
4000
5000
1000
1020
1040
1060
1080
1100
Absorbed pump power (W)

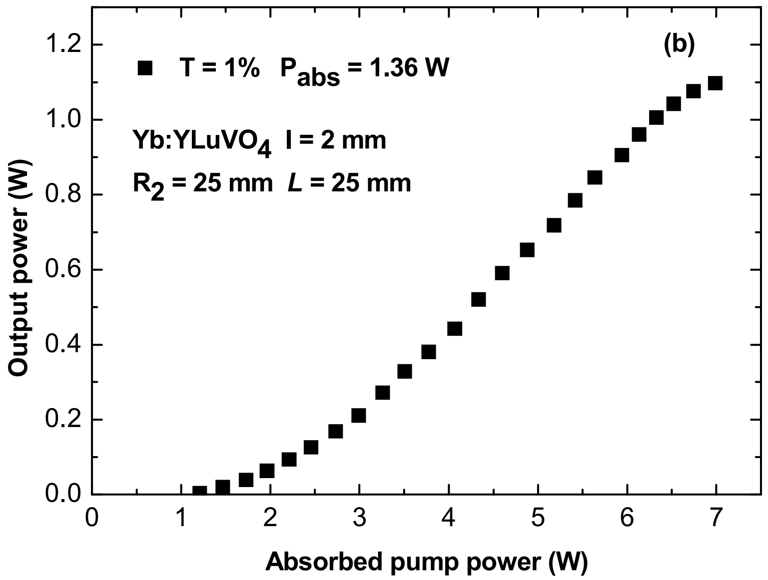

(b)
■ T = 1% P_{abs} = 1.36 W
Yb:YLuVO$_4$ l = 2 mm
R_2 = 25 mm L = 25 mm
Output power (W)
0.0
0.2
0.4
0.6
0.8
1.0
1.2
0
1
2
3
4
5
6
7
Absorbed pump power (W)

Fig. 5 The laser performance obtained with a 2 mm thick a-cut Yb: $Y_{0.71}Lu_{0.29}VO_4$ crystal.

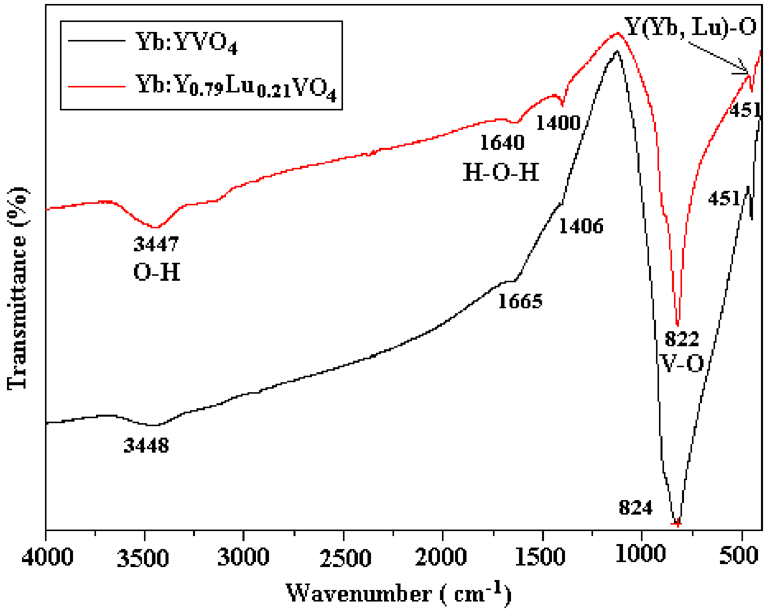


Fig. 6. FT-IR spectra of Yb: YVO_4 and Yb:$Y_{0.79}Lu_{0.21}VO_4$ crystals

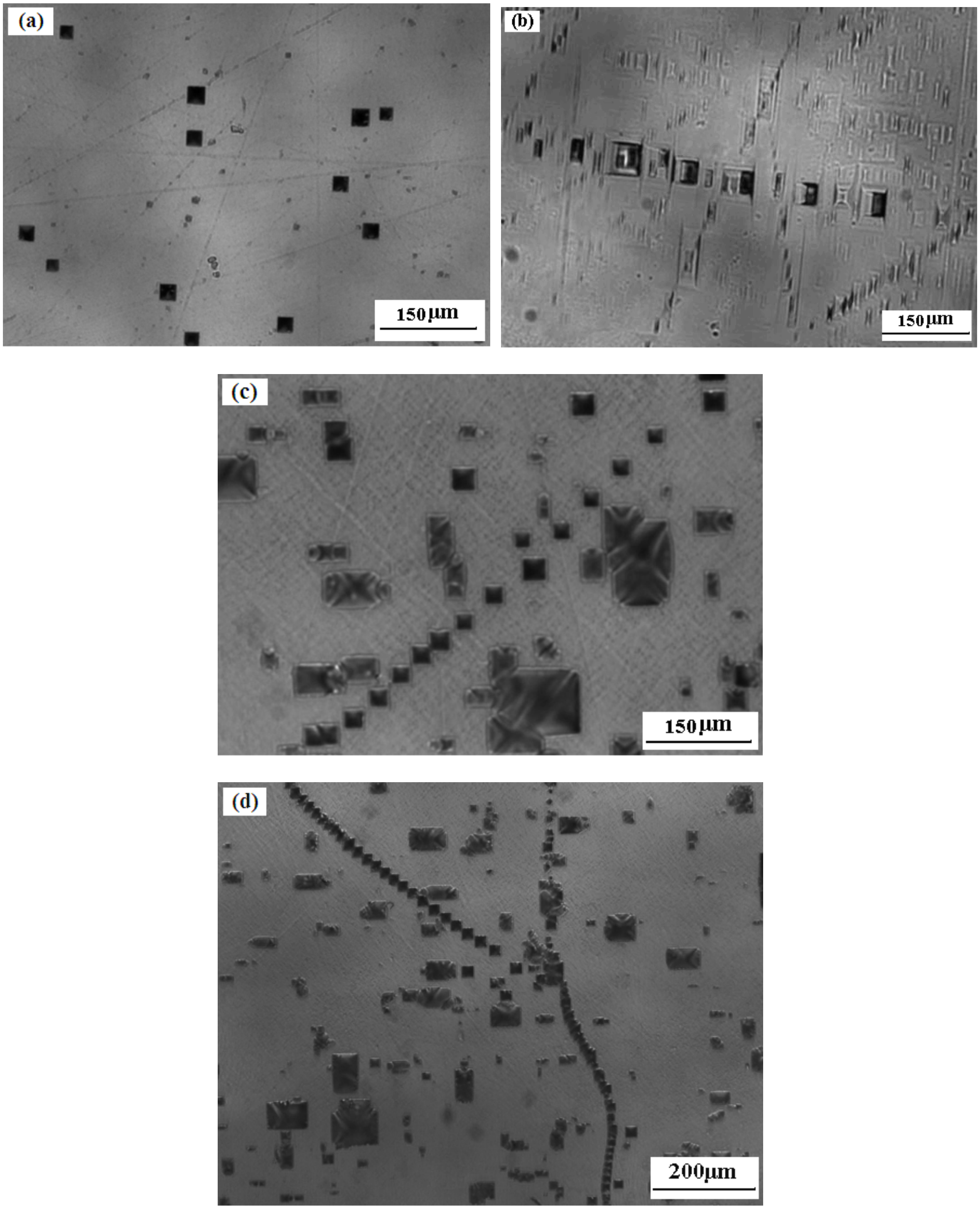


Fig.7 The etch figures on (001) and (100) planes. (a) Etch pit figure on (001) plane, etched for 20min; (b) etch pit figure on (100) plane, etched for 1 h; (c) array of dislocation pits on (001) plane, etched for 20min; (d) three intersecting of dislocation pits on (001) plane, etched for 20min.

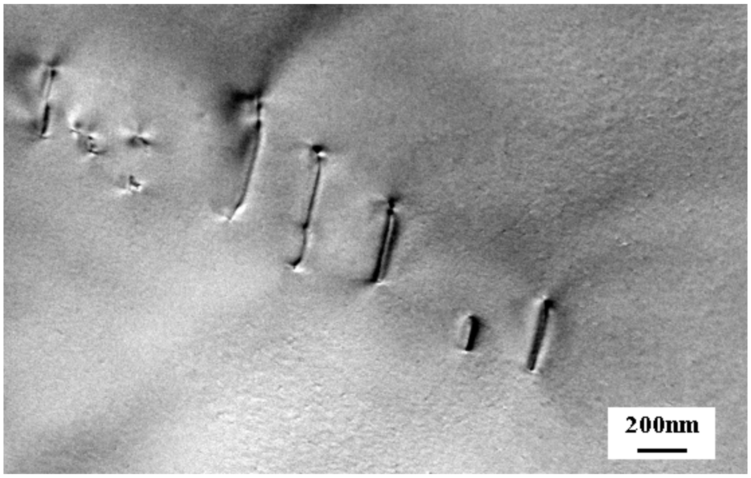


Fig. 8 Bright field TEM image of a periodic array of dislocations forming a low angle grain boundary.

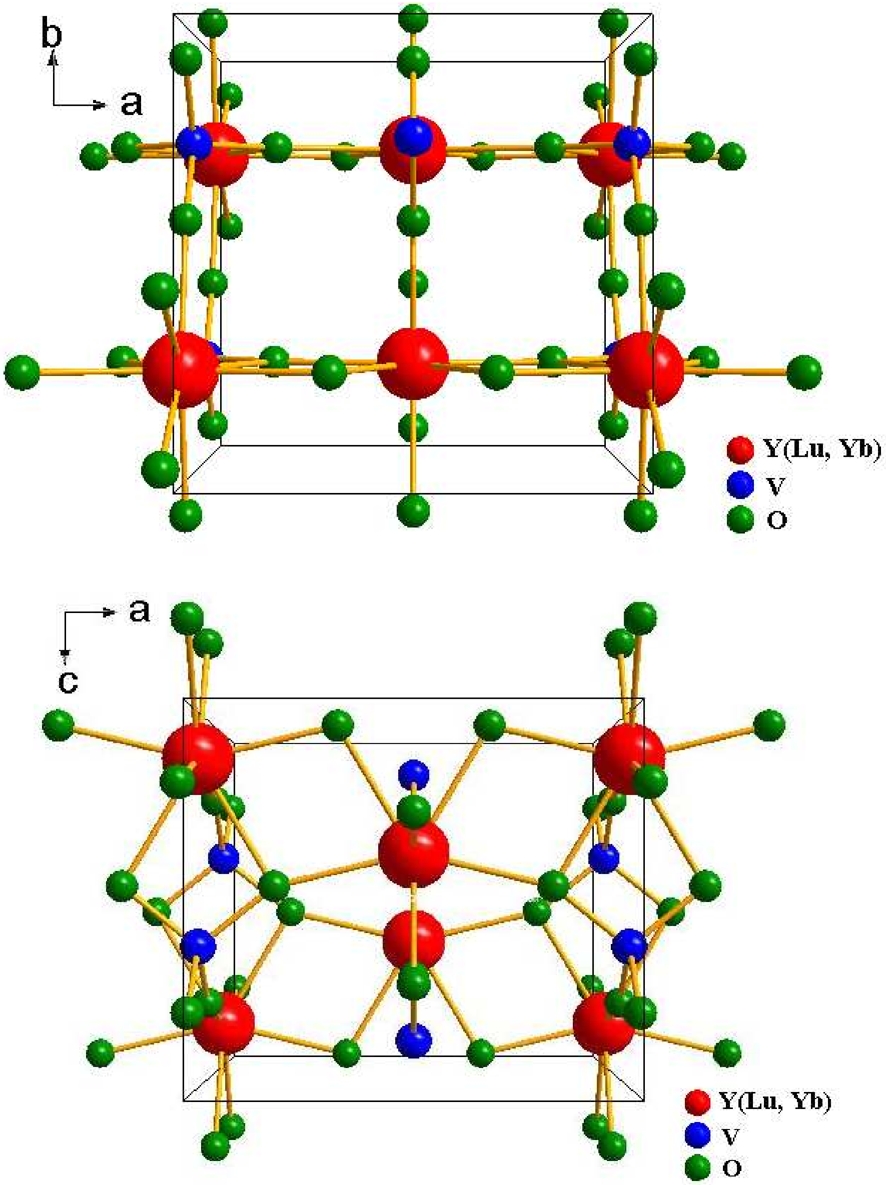


Fig. 9. View of the 3D structure of Yb:$Y_{0.79}Lu_{0.21}VO_4$ shown down the c-axis and b-axis.

Table 1 Etching conditions for various facets [17]

Facets	etching agent	Etching time (min)	Temperature
(100)	V_{HCl}:V_{H2O} =2:3 (volume ratio)	60	100ºC
(001)	V_{HCl}:V_{HF} =10:1 (volume ratio)	20	100ºC

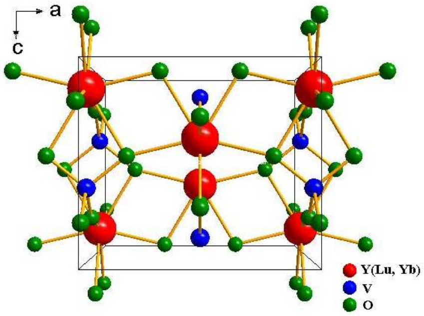
Table2 Parameters charaterizing the cw laser performance of different Yb-doped vanadate mixed crystals, obtained with the output coupler T=0.5 and 1%.

Crystal (a-cut, 2mm)	Maximal output power (W)	Slope efficiency	Laser thresholds (W)	Laser wavelengths (nm)
Yb(2.1 at.%):$Gd_{0.64}Y_{0.36}VO_4$ (T=1%) [9]	0.7	45%	1	-
Yb(0.25 at.%):$Y_{0.79}Lu_{0.21}VO_4$ (T=1%)	1.09	21.6%	1.21	1020-1029
$Yb_{0.007}$:$Y_{0.407}Gd_{0.586}$ VO_4 (T=1%) [10]	3.2	44%	0.6	1037-1044
$Yb_{0.015}$:$Lu_{0.52}$ $Gd_{0.465}$ VO_4 (T=0.5%) [10]	3.3	58%	2.2	1049-1055

For Table of Contents Only

Synthesis, Growth, Defects and Laser Action of Yb: $Y_{0.71}Lu_{0.29}VO_4$ Crystals

Degao Zhong, Bing Teng, Lifeng Cao , Shiming Zhang , Fei You, Bingtao Zhang , Jianhong Li, Xueping Tian, Chao Wang, Yuyi Li, Linxiang He, Romano A. Rupp



A new promising mixed laser crystal of composition Yb:$Y_{0.79}Lu_{0.21}VO_4$ was grown and realized continuous-wave laser output.