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Ultraviolet to infrared refractive indices of tetragonal double tungstate and double molybdate laser crystals

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Abstract Sellmeier parameters of thirteen tetragonal (space group $I\bar{4}$) double tungstate and double molybdate laser crystals with $M^+T^{3+}(X^{6+}O_4)_2$ composition have been calculated using the room temperature refractive indices determined from the ultraviolet band gap of the crystals to $\lambda \approx 2$ µm. All considered crystals are uniaxial but only crystals with Bi in their composition show a significant birefringence ($\Delta n > 10^{-2}$). The refractive index value increases for the sequence $T^{3+} =$ La, Y, Gd, Lu, and Bi independently of the M^+ and X^{6+} cation pair. Implications for the design of laser waveguides and laser pulse dispersion are discussed.

1 Introduction

Lanthanide (Ln) doped tetragonal double tungstate (DW) and double molybdate (DMo) single crystals, with chemical formula $M^+T^{3+}(X^{6+}O_4)_2$ (X = W or Mo, respectively), briefly MTX, and $I\bar{4}$ crystallographic space group, are currently studied for optical gain media in ultrafast (fs) mode-locked laser systems: Yb-doped $NaY(WO_4)_2$ crystals provided pulses as short as 53 fs at $\lambda = 1.035$ µm (with 91 mW of average power and 96 MHz of repetition rate) [1], and laser pulses of 191 fs (with 82 mW of average power and 144 MHz of repetition rate) were shown at $\lambda = 2.060$ µm using Tm-Ho codoped $NaY(WO_4)_2$ crystals [2]. Moreover, they are also considered as gain media in cryogenic lasers to accommodate the full emission bandwidth of the diode lasers used for optical pumping [3]. In both applications, the relevant physical property used is the large optical bandwidth of the electronic transitions of Ln^{3+} ions in this crystal class. Such property is related to the crystallographic disorder of M and T ions over two crystal sites [4, 5].

This tetragonal phase, closely related to the scheelite $I4_1/a$ phase of the parent $CaWO_4$ crystal, is often observed for DW and DMo crystals quenched to room temperature from their melting or decomposition temperatures. However, the presence of polymorphic transformations at temperatures below the melting or decomposition points limits the number of crystal compositions that can be grown from its melt and successfully cooled to room temperature. A comprehensive review of the phases of DW and DMo compounds can be found in [6]. So far, most of the characterization work has been conducted on Na-based tetragonal DW crystals, but the number of transparent NaTW crystal laser hosts free of polymorphic transformations is limited to T = Y, La, Gd, Lu, Bi. Li- and Na-based DMo (T = Y, La, Gd, Lu, Bi) as well as $KLa(MoO_4)_2$ are transparent crystals also free of polymorphic transformations; they have melting points lower than DWs and the optical cross sections of Ln^{3+} in some of them are larger than in isostructural DWs, therefore, they are receiving increasing attention for the above mentioned purposes, despite of the presence of color centers in as-grown crystals of some compositions grown by the Czochralski (Cz) method.

The linear refractive index (n) is a fundamental parameter required for spectroscopic calculations and laser applications. In particular, it is required in the Judd–Ofelt analysis of the radiative properties of Ln^{3+} ions, for the calculation of the nonlinear and thermal refractive index contributions, for the design of antireflection coatings and optical waveguide structures, for the calculation of athermal laser propagation directions, etc. So far, we have provided the dispersion of n in the visible and near infrared (up-to $\lambda \approx 1$ µm) for some few crystal compositions, namely for

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NaTW (T = Y [1], La [7], Gd [8], Lu [9], and Bi [10]), for MBiMo (M = Li [11] and Na [10]) and for Yb:NaYMo [12]. Recent interest for Er, Tm, and Ho lasers in these hosts requires the knowledge of the refractive index at λ ≈1.6 and ≈2 µm. Such results have not been provided in previous studies, therefore, in the literature most often n is approximated to a constant value obtained from the extrapolation of visible results when available. This work presents a comprehensive study of the refractive index dispersion from the ultraviolet absorption crystal band edge to λ ≈ 2 µm in thirteen isostructural tetragonal DW and DMo crystals.

2 Experimental details

DW and DMo single crystals have been grown either by the Cz or by the top seeded solution growth (TSSG) methods. Details of the growth procedures can be found in our previous works [7–12]. It is worth to mention that in some cases Cz-grown crystals are chemically reduced during the growth process; this is particularly noticeable in some DMo, for instance, $NaGd(MoO_4)_2$. Such chemical reduction introduces lattice defects that absorb light in a broad range of the visible spectra. In these cases, the crystals were either grown by the TSSG method or annealed in air (aa) to avoid any influence of defects on our measurements (refer to Table 1). Due to crystal availability, in some few cases Ln-doped crystals were used, selecting the lowest Ln concentration available.

According to the tetragonal crystallographic structure, the DW and DMo crystals here considered are optically uniaxial, with the optic axis of the indicatrix parallel to the crystallographic c-axis. The ordinary, n_o, and extraordinary, n_e, refractive indices were determined independently by the minimum deviation angle method [13]. For each crystal composition, two prisms were cut with the orientation required to propagate the electric field component of the vertically polarized light parallel to the a or c crystal axes, respectively. The prism angle and the beam deviation angle were determined with an Ealing goniometer. Several optical laser sources have been used to cover the visible and infrared spectral range: Nitrogen laser (0.337 µm) pumped dye lasers (0.35–0.7 µm), Ar laser (0.3345, 0.351, 0.363, 0.458, 0.4658, 0.4727, 0.4765, 0.488, 0.496, 0.5017, 0.514 µm), He–Ne laser (0.6328 µm), Ti-sapphire laser (0.650–1 µm), and Master Oscillator/Power Oscillator (Quanta-Ray) optical parametric laser system (0.85–2 µm). All measurements were performed at room temperature without any specific control of the sample temperature, therefore, the crystal temperature is estimated as 25 ± 5 °C. The largest n uncertainties arise from the crystal orientation accuracy (±2°) and from orientation deviations of the prism lateral faces during polishing. The calculated n uncertainty is lower than ±0.005 in all cases.

3 Results and discussion

Figure 1 shows the dispersion of n for two representative $LiT(MoO_4)_2$ crystal compositions. The results have been fit to an infrared-corrected Sellmeier law,

$$n^2 = A + \frac{B\lambda^2}{\lambda^2 - C^2} - D\lambda^2. \tag{1}$$

The C parameter corresponds to the wavelength of the crystal energy band gap, however, measurements or calculations of the band gap of these crystals are not available, for instance, optical absorption measurements performed with thin (thickness ≥50 µm) crystals do not provide a realistic value of the band gap and excitation spectra of the intrinsic photoluminescence are affected by the presence of optically active defects. Therefore, C was treated as a fitting free parameter. Table 1 summarizes the parameter value sets obtained from the best fit in each crystal.

Some few comments can be drawn from the present results.

Although C was treated as a fitting free parameter, the values obtained from the fit are consistent with previous information of the band gap evolution in tetragonal DW and DMo crystals: (i) The C values obtained for NaYW are smaller than the optical absorption edge measured at 5 K, i.e., 0.293 µm [1]. Similar differences are found for NaGdW or NaLuW crystals with 5 K optical absorption edges at 0.295 µm [8], and <0.288 µm [9], respectively. Moreover, the maximum of the intrinsic photoluminescence of DW and DMo crystals with Bi appears at shorter wavelength, shifted by 25–50 nm with respect to the absorption edge [10]. (ii) The difference of the 5 K optical absorption edge for both polarizations is lower than 10 nm, accordingly the difference in o and e C values is most often within this range. (iii) DMo crystals and crystals with Bi in their composition have C values larger than those observed in DWs. This corresponds to the shift of the optical absorption edge to larger wavelength, i.e., the 5 K absorption edge of NaBiW, NaBiMo, and LiGdMo are <0.353 µm [10], <0.400 µm [10], and 0.340 µm, respectively.

The results obtained in crystals with nonnegligible dopant concentrations, i.e., 7.5 at% Tm:2.5 at% Yb:LiGdMo, 6.1 at% Yb:NaYMo, and 5 at% Nd:KLaMo, must be taken with some precaution. It was previously observed that the extraordinary refractive index of NaGdW increases by doping with 20 at% of Yb [8]. The influence of most common laser lanthanides (Nd, Ho, Er, Yb, and Tm) on the refractive index of a transparent MTX crystal is yet not clear; either refractive index measurements with higher resolution or the analysis of crystals with larger concentration of impurities, even stoichiometric in the laser active lanthanide, would be required to achieve a clear conclusion and to help in the future design of optical waveguide lasers developed in these crystals.

Table 1 Parameters of the Sellmeier fit (A, B, C, and D) of the room temperature ordinary (o) and extraordinary (e) refractive indices (n) of tetragonal DW and DMo crystals. Growth method (GM): Top seeded solution growth (TSSG), Czochralski (Cz), and modified Czochralski (m-Cz). Refractive indices and their birefringence ($\Delta n = n_e - n_o$) at the emission wavelengths typical of Yb^{3+} ($\lambda = 1.06$ μm), Er^{3+} ($\lambda = 1.6$ μm), and Tm^{3+} ($\lambda = 2$ μm) lasers. Group velocity dispersion (GVD) at $\lambda = 1.06$ μm

Crystal	GM	Pol	A	B	C (μm)	D (μm^{-2})	n, 1.06 μm	n, 1.6 μm	n, 2 μm	GVD (fs^2/mm)
$NaY(WO_4)_2$	Cz	o	2.521	1.214	0.1856	0.0130	1.939	1.928	1.919	105
		e	2.484	1.270	0.1803	0.0171	1.942	1.930	1.923	98
		Δn					+0.003	+0.002	+0.004	
$NaLa(WO_4)_2$	Cz	o	2.209	1.413	0.1801	0.0062	1.912	1.900	1.899	125
		e	2.126	1.488	0.1801	0.0091	1.910	1.899	1.895	129
		Δn					−0.002	−0.001	−0.004	
$NaGd(WO_4)_2$	Cz	o	2.230	1.507	0.1821	0.0055	1.943	1.935	1.931	135
		e	2.260	1.479	0.1870	0.0050	1.945	1.936	1.932	142
		Δn					+0.002	+0.001	+0.001	
$NaLu(WO_4)_2$	TSSG	o	1.491	2.327	0.1500	0.0078	1.964	1.954	1.949	134
		e	1.529	2.276	0.1510	0.0075	1.961	1.951	1.947	133
		Δn					−0.003	−0.003	−0.002	
$NaBi(WO_4)_2$	Cz	o	2.591	1.937	0.2464	0.014	2.150	2.131	2.122	312
		e	2.719	1.585	0.2377	0.013	2.091	2.075	2.067	239
		Δn					−0.059	−0.056	−0.055	
$LiLa(MoO_4)_2$: 0.5 at% Yb	Cz	o	2.216	1.608	0.1996	0.0130	1.967	1.953	1.946	168
		e	2.666	1.150	0.2313	0.0075	1.966	1.955	1.950	175
		Δn					−0.001	+0.002	+0.004	
$LiGd(MoO_4)_2$: 7.5 at% Yb,2.5 at% Tm	Cz	o	2.078	1.947	0.1979	0.0110	2.021	2.007	2.000	199
		e	1.994	1.988	0.1983	0.0061	2.012	1.999	1.994	211
		Δn					−0.009	−0.008	−0.006	
$LiBi(MoO_4)_2$	Cz	o	3.081	2.022	0.2963	0.0180	2.292	2.265	2.253	484
		e	3.283	1.491	0.2827	0.0200	2.206	2.184	2.174	323
		Δn					−0.086	−0.081	−0.079	
$NaY(MoO_4)_2$: 6.1 at% Yb	TSSG	o	2.507	1.377	0.2128	0.0160	1.981	1.967	1.961	162
		e	2.549	1.338	0.2150	0.0100	1.983	1.971	1.965	168
		Δn					+0.002	+0.004	+0.004	
$NaLa(MoO_4)_2$	Cz[aa]	o	2.227	1.540	0.1987	0.009	1.953	1.941	1.936	165
		e	2.230	1.528	0.2035	0.010	1.951	1.938	1.932	172
		Δn					−0.002	−0.003	−0.004	
$NaGd(MoO_4)_2$	TSSG	o	2.155	1.715	0.1998	0.012	1.980	1.966	1.960	181
		e	2.658	1.256	0.2231	0.016	1.989	1.974	1.967	164
		Δn					+0.009	+0.008	+0.007	
$NaBi(MoO_4)_2$	Cz	o	2.837	1.997	0.2836	0.0199	2.228	2.202	2.190	437
		e	3.134	1.367	0.2814	0.0090	2.144	2.126	2.120	310
		Δn					−0.084	−0.076	−0.070	
$KLa(MoO_4)_2$: 5 at% Nd in melt	m-Cz	o	1.605	2.025	0.1731	0.0079	1.917	1.906	1.901	163
		e	1.849	1.751	0.1851	0.0074	1.910	1.899	1.894	165
		Δn					−0.007	−0.007	−0.007	

[aa] Air annealed

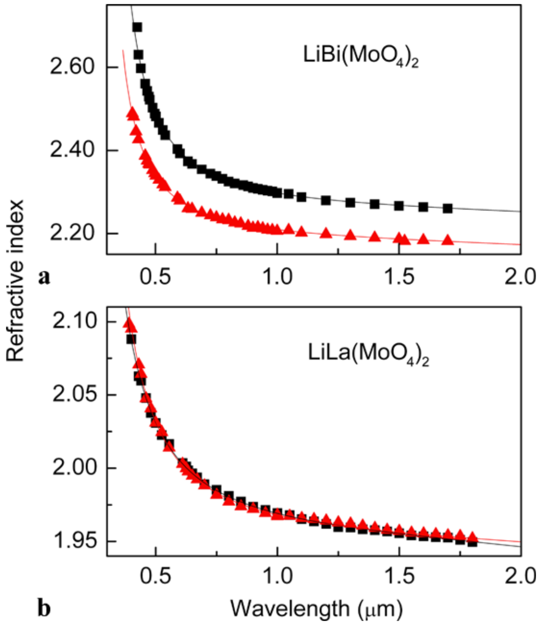

Fig. 1 Spectral dispersion of the ordinary (■, *black squares*) and extraordinary (▲, *red triangles*) refractive indices of negative uniaxial $LiBi(MoO_4)_2$ (**a**) and the isotropic-like 0.5 at% $Yb:LiLa(MoO_4)_2$ (**b**) Czochralski grown crystals. The lines are the corresponding fits to the infrared-corrected Sellmeier law

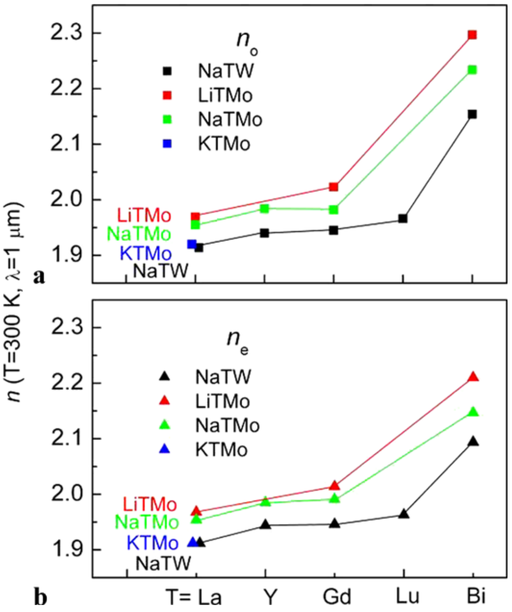

Fig. 2 Evolution of the room temperature ordinary (**a**) and extraordinary (**b**) refractive index at $\lambda = 1$ μm (*symbols*) of tetragonal MTX crystals. The *lines* are only a visual help

Tetragonal DW and DMo crystals have low birefringence ($\Delta n \approx 10^{-3}$) with the significant exception of Bi-based compounds ($5.5 \times 10^{-2} \leq \Delta n \leq 9 \times 10^{-2}$). A high polarizability associated to the lone $6s^2$ electron pair of Bi^{3+} [14] is the likely reason of this difference. In fact, Bi^{3+} doping in gadolinium-gallium garnets was used to increase the refractive index in channel-waveguide lasers [15]. Bi-based DW and DMo crystals are uniaxial negative, but for MTX crystals with T ≠ Bi the birefringence is of the same order or lower than the uncertainty of our measurements, therefore, from the practical point of view the latter crystals can be considered as near to optically isotropic.

Within a given M^+X^{6+} series, the refractive index increases in the sequence La < Y < Gd < Lu < Bi. However, the largest change is associated to the presence of Bi. Figure 2 illustrates this situation for $\lambda = 1$ μm. For the same MT pair, DMo crystals have larger refractive index than the corresponding DWs. Li-based DMos have larger refractive index than Na-based DMo crystals, and NaLaMo larger than KLaMo. These results can guide the future design of waveguided lasers in these crystals. First steps for the preparation of doped layers of DWs have been already made by using liquid phase epitaxy (LPE) to grow lattice matched layers on NaYW substrates [16].

The production of laser waveguides requires the creation of a region of high refractive index with respect to the surrounding media. This is often obtained by modifying the composition of the outer layer of the crystal, either by doping or by the growth of a new layer on the surface of a substrate. In the latter case, the partial substitution of constituent trivalent cations of monoclinic $KT(WO_4)_2$ (T = Y, Gd) crystals by Lu has been found an efficient way to increase the refractive index [17]. In the case of tetragonal DW and DMo, epitaxial layers can be grown efficiently by LPE, and thanks to the high miscibility and large number of isostructural compositions available there are several options for the modification of the refractive index. First option is connected to the La, Y, or Gd host ions substitution by Lu; intermediate refractive index modulations ($\delta n < 0.025$) can be obtained while the lattice mismatch between layer and substrate is expected to be moderate. A second option that would produce much higher refractive index modulation ($\delta n \leq 0.2$) is the substitution of trivalent rare earths (RE) by bismuth, however, this option could be limited by chemical stability of the molten flux and it is expected to introduce optical anisotropy and larger lattice mismatches between the substrate and the grown layer (lattice parameters of Bi-based DW and DMo crystals can be found in [11] while those of RE-based DW and DMo crystals can be found in [1, 6, 8, 9] among others). The substitution of the alkali metal may induce small to intermediate index changes ($\delta n < 0.015$–$\delta n < 0.04$), n decreasing as the alkali metal size increases but is basically limited to DMo crystals, because most of Li, K, and Cs-based DWs exhibit polymorphism and the tetragonal phase is not stable at room temperature. Finally, the partial substitution of tungsten by molybdenum would induce an intermediate index modulation ($\delta n \leq 0.05$) and low lattice mismatch. This possibility seems feasible since $NaY(W_{1-y}Mo_yO_4)_2$ ($y = 0.2$) crystals have been grown by the Cz method [18], but the proper flux

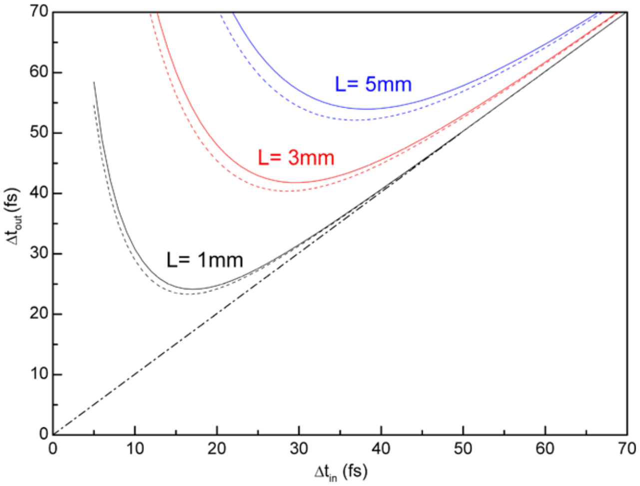

Fig. 3 Time pulse broadening after several propagation lengths in a $NaY(WO_4)_2$ crystal for a beam polarized parallel to the *c*-crystal axis (*dashed line*) and perpendicular to it (*solid line*). The *dot-dashed line* is a visual help

and LPE growth conditions must be carefully searched because the low melting point of Mo-based fluxes promotes the dissolution of corresponding DW phases.

A major issue for the stable mode-locked production of fs laser pulses is the control of the pulse chirping induced by the dispersion of the phase velocity upon pulse propagation through the crystal used as optical gain medium. This is described by the group velocity dispersion (GVD) given by [19]

$$\mathrm{GVD} = \frac{\lambda^3}{2\pi c^2}\left(\frac{d^2 n}{d\lambda^2}\right). \tag{2}$$

In principle, low GVD values are desirable for the generation of fs laser pulses.

Taking into account Eq. (1), the derivatives of the refractive index can be written as:

$$\frac{dn}{d\lambda} = -\frac{\lambda}{n}\left(\frac{\mathrm{BC}^2}{(\lambda^2-\mathrm{C}^2)^2} + \mathrm{D}\right), \tag{3}$$

$$\frac{d^2 n}{d\lambda^2} = -\frac{1}{n}\left(\left(\frac{dn}{d\lambda}\right)^2 - \frac{\mathrm{BC}^4 + 3\mathrm{BC}^2\lambda^2}{(\lambda^2-\mathrm{C}^2)^3} + \mathrm{D}\right), \tag{4}$$

therefore, for a given λ, GVD can be obtained from the Sellmeier coefficients. Table 1 includes the GVD values of DW and DMo crystals at the typical laser emission of Yb^{3+} or Nd^{3+} lasers, $\lambda = 1.06$ µm. It can be observed that DW crystals have GVD lower than DMo ones, the incorporation of Bi increases GVD very significantly and the variation of the alkali metal has not influence on the GVD value. It is interesting to remark that the lowest GVD values are obtained for NaYW, coincidently with the fact that under the same testing conditions the shortest mode-locked Yb^{3+} laser pulses were obtained in Yb:NaYW (67 fs), in comparison to NaYMo (91 fs) [12], NaLuW (90 fs) [9], or NaGdW (120 fs) [8].

To illustrate the potential broadening of a laser pulse after propagation through a NaYW crystal, Fig. 3 shows the pulse duration relationship between the incoming (Δt_{in}) and outgoing (Δt_{out}) pulses given by [19]

$$\Delta t_{\mathrm{out}} = \frac{\sqrt{\Delta t_{\mathrm{in}}^4 + 7.69 \times \mathrm{GVD}^2 \times L^2}}{\Delta t_{\mathrm{in}}}. \tag{5}$$

For the propagation length of $L = 1$ mm, a significant increase of the pulse duration is not expected for pulses longer than 50 fs. Yb-doped DW [1, 8, 9] and DMo [12, 20] mode-locked crystalline lasers have been demonstrated with Yb concentrations from 3 to 10 at% using samples with thickness in the range 1.5 to 3.5 mm. For such propagation lengths, the chirping of the laser pulse is more significant and proper GVD compensation is essential to achieve the shortest pulses.

4 Conclusions

The dispersion of the principal refractive indices of tetragonal double tungstate and double molybdate crystals has been determined at room temperature. These changes have been fit to the well known Sellmeier law with infrared correction and the corresponding parameters determined. From thirteen considered crystals, ten, Na(Y, La, Gd, Lu)W, Li(La, Gd)Mo, Na(Y, La, Gd)Mo, and KLaMo, are characterized by small absolute value of the birefringence, $10^{-3} < \Delta n < 10^{-2}$, being optically positive (Y, Gd) or negative (La, Lu). LiGdMo is also negative. Crystals including Bi^{3+} in the composition, NaBi(W, Mo) and LiBiMo, are optically negative and exhibit by an order of magnitude higher Δn, reaching 8.6×10^{-2} for LiBiMo. For a given combination of monovalent and hexavalent cations, the refractive index value increases in the sequence La, Y, Gd, Lu, and Bi of the trivalent cation of the crystal composition. These results are expected to help in the future design of optical waveguides in this crystal class and explain why NaYW is the crystal producing shortest ultrafast mode-locked laser pulses within this crystal class.

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