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Micro-Raman investigation of $KGd(WO_4)_2$ single crystals triply-doped with $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ ions

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ABSTRACT

The paper presents the first investigations of $KGd(WO_4)_2$ single crystals triply-doped with $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ ions by micro-Raman spectroscopy. No significant influence of doping ions on the Raman bands position was observed for investigated crystals at room temperature. The highest values of wavenumbers of Raman bands were observed for $Pr^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. The low-temperature dependences of the frequency of selected modes located at about 900, 807, 766, 746, 436, 348 and 342 cm^{-1}, assigned to the stretching and bending vibrations of WO_4^{2-} tetrahedra and WOW and WOOW oxygen bridges, were analyzed in the 77–293 K temperature range. Some anomalies in the spectral parameters of these modes are observed at about 145 K for all investigated crystals. The results were discussed in terms of the local distortion of WO_4^{2-} tetrahedra and deformation of WOW and WOOW oxygen bridge bonds vibrations in low-temperature range on the basis of the mismatch of the ionic radii of Pr^{3+}, Er^{3+}, Ho^{3+}, Tm^{3+} and Yb^{3+} ions in the cationic layers.

1. Introduction

The rare earth triply-doped solid state materials showing infrared to visible frequency up-conversion are very promising candidates for the visible lasers with multicolor or white luminescence after NIR excitation [1–4]. The efficiency of the up-conversion process depends on the physical properties of the host medium and the concentration of the doping ions. The optical and spectroscopic properties of many lanthanide doped materials are currently intensively studied, using different experimental techniques [5–10]. Up to now there are only a few reports about visible up-conversion emission in the rare earth triply-doped crystals [11–14]. $KGd(WO_4)_2$ single crystal is an efficient laser host for the stimulated up-conversion emission of the rare earth ions [15]. This crystal can be easily doped with rare earth ions as Gd^{3+} ions can be substituted by other lanthanides. In the present work we report the vibrational properties of triply-doped $KGd(WO_4)_2$ (hereafter KGW) single crystals, doped with combination of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ ions studied by means of Raman spectroscopy. According to our best knowledge, there are no reports on the vibrational studies of KGW single crystals triply-doped with lanthanide ions such as Pr^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} or Yb^{3+}. In the proposed crystals the Yb^{3+} ions have been used as an energy sensitizer ion and the up-conversion luminescence originates from the Pr^{3+}, Ho^{3+}, Er^{3+}, and Tm^{3+} co-doped ions, which play a role of the activator ions, giving the up-conversion emission of red, green and blue colours. The potential applications of the investigated crystals in optoelectronic devices need the high quality materials with an excellent physical properties. The vibrational studies of the KGW host lattice are particularly important due to its role in transfer of excitation energy between lanthanide ions in energy conversion process.

KGW crystals belong to the monoclinic structure with $C2/c \equiv C_{2h}^6$ space group symmetry with four formula units in the unit cell ($Z = 4$) at room temperature. The lattice cell parameters were found to be: $a = 10.652$ Å, $b = 10.374$ Å, $c = 7.582$ Å, $\beta = 130.80^\circ$ [16]. The structure of KGW is formed by chains of $W_2O_8^{4-}$ ions arranged along the c–axis and connected one to another by oxygen atoms (WOW oxygen bridge bonds). The dimers $W_2O_8^{4-}$ are formed by two WO_4^{2-} ions, which are connected by four W–O bonds (WOOW double oxygen bridge bonds). Along the b axis the tungsten – oxygen layers are alternate with the cationic layers created by K^+ and Gd^{3+} ions [5]. The slight local distortion of WO_4^{2-} tetrahedra at room temperature is present in KGW crystals probably due to the random distribution K^+ and Gd^{3+} ions, which occupy the same crystallographic positions in crystal lattice [17]. In the rare earth ions doped KGW crystals the Pr^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} or Yb^{3+} ions replaces the Gd^{3+} ions in their positions. For the doping concentration used in this work the crystal structure of triply-doped KGW is preserved. However, the slight change in the cell parameters can be the consequence of the different ionic radii of Pr^{3+}, Gd^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} or Yb^{3+} ions equal to 1.126, 1.053, 1.015, 1.004, 0.994 and 0.985 Å,

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respectively [18]. Moreover, the difference in ionic radii of Gd^{3+} and doping ions can influence a local distortion of WO_4^{2-} tetrahedra and deformation of single WOW and double WOOW oxygen bridge bonds. The low-temperature investigations of the vibrational properties of mentioned above rare earth triply-doped systems were also undertaken by Raman spectroscopy.

2. Experimental

The monoclinic triply-doped KGW crystals were obtained by Top Seeded Solution Growth method [16]. The microscopic investigations of KGW crystals were carried out for oriented samples of sizes 5 mm × 4 mm × 3 mm with edges parallel to the axes of the reference system (*xyz*). The reference system (*xyz*) was defined by the following convention: $x||a$, $y||b$ and $z||c^*$, respectively. In the used notation *a*, *b* and *c* denote the crystallographic axes of the monoclinic system and c^* denotes the axis perpendicular to the *ab* plane. The orientation of the samples of investigated crystals was performed by the Laue method and analytical use of the stereographic Wulff net [19]. The doping concentration of Pr^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} and Yb^{3+} ions was 3 at.% for all investigated crystals. The Raman investigations were performed in back-scattering geometry using Renishaw InVia Raman microscope equipped with confocal DM 2500 Leica optical microscope, a thermoelectrically (TE)-cooled RenCam CCD detector and Ar^+ ion laser working at 488 nm wavelength. The polarized Raman spectra were recorded in single scan with 10 s exposure time of CCD detector. The applied power of the laser beam before focusing, was less than 1 mW. The diffraction-limited optical spatial resolution for the used 50× LWD objective for excitation wavelength 488 nm calculated from Abbe's equation is approximately equal 0.8 μm [20]. An Edge filter was used to stray Rayleigh light rejection. The instrumental resolution was better than 2 cm^{-1}. The position of Raman peaks was calibrated before collecting the data using Si sample as an internal standard. The polarized Raman spectra were recorded in the spectral range 70–1100 cm^{-1}, covering the region of internal and external vibrations for $z(xx)z$ and $z(xy)z$ scattering geometries. The temperature measurements were performed using Linkam THMS 600 cooling/heating stage in temperature range 77–293 K with temperature stability 0.1 K. In order to obtain the band parameters of temperature-dependent Raman spectra a fitting procedure was applied, considering the Bose–Einstein population factor.

3. Results

The room-temperature polarized in $z(xx)z$ and $z(xy)z$ Raman spectra of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals detected in the spectral range 70–1100 cm^{-1} are presented in Fig. 1.

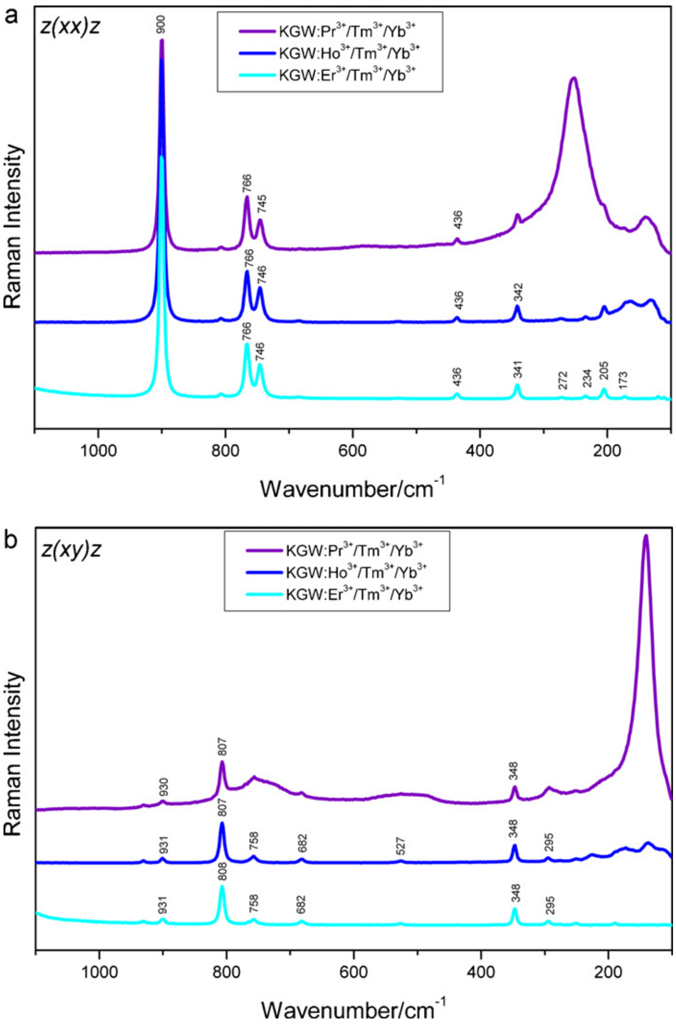

Fig. 1. Room temperature Raman spectra of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals for (a) $z(xx)z$ and (b) $z(xy)z$ polarization.

The detailed assignment of the Raman active modes for the doubly-doped with Er^{3+} and Yb^{3+} ions KGW crystals and series of single-doped with Nd^{3+}, Eu^{3+}, Er^{3+} and Ho^{3+} ions KGW crystals were presented in our previous papers [21,22]. In tungstate crystals the WO_4^{2-} tetrahedra are the most active molecular groups giving most of the bands in the Raman spectra. However, the Raman spectra are more complicated due to some bands activated by the WOW and WOOW oxygen bridge bonds vibrations. As it can be seen from Fig. 1 there are some Raman bands in spectra characteristic for tungstate crystals for all investigated crystals. The positions of bands have been determined and their wavenumbers are given for each crystal. Moreover, Raman spectra contain some broad luminescence bands, excited with 488 nm wavelength, which are characteristic for rare earth ions excitation in crystalline lattice of KGW. Particularly, the strong luminescence bands below 400 cm^{-1} for $z(xx)z$ geometry and relatively low-intensity, broad bands in almost whole region up to 950 cm^{-1} for $z(xy)z$ configuration were detected in spectra of $Pr^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. These luminescence bands disturbed the analysis of Raman peaks and the determination of their spectral parameters are even impossible in some cases. Because of this the analysis of Raman spectra were limited to the range of internal vibrational motions above 260 cm^{-1}. The dominant in this spectral range, sharp peaks at about 900 cm^{-1}, detected in $z(xx)z$ polarization, are attributed to symmetric stretching vibrations of the WO_4^{2-} tetrahedra. The polarized $z(xy)z$ modes located at about 931 and 807 cm^{-1} are assigned as the coupling stretching vibration of WO_4^{2-} and WOW. The bands positioned at about 756 and 746 cm^{-1} detected in $z(xy)z$ and $z(xx)z$ polarization, respectively are attributed to coupling stretching vibration of WO_4^{2-} and WOOW. The bands located at about 766, 682 and 527 cm^{-1} are assigned as the stretching vibrations of the WOOW oxygen bridge bonds. The deformation bending vibrations of WO_4^{2-} tetrahedra are recorded at 342 and 348 cm^{-1} in $z(xx)z$ and $z(xy)z$ polarization, respectively. The bending vibrations of WOW at 436 cm^{-1} and bending coupling vibrations of WO_4^{2-} and WOOW at 296 cm^{-1} are recorded in $z(xy)z$ polarization. Below 260 cm^{-1} the lattice modes are detected in Raman spectra. The coupling between the vibrations of different molecular groups in the KGW crystal lattice modified by doping rare earth ions leads to the change in the structure of some vibrational modes. However, as it can be also seen from Fig. 1 the concentration of rare earth doping used in KGW crystals does not significantly change the position and the relative intensity of Raman bands. Some slight differences in the values of wavenumbers for the corresponding Raman bands in $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals are below 1 cm^{-1}. The highest values of wavenumbers of Raman bands were observed for $Pr^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. In this crystal the highest average ionic radius of doping ions occurs, due to the highest ionic radius of Pr^{3+} ions, which substituted the Gd^{3+} ions in crystalline lattice. Thus, the inter-atomic distances are slightly shorter, which results in higher frequency vibrations and shift of wavenumbers of some Raman

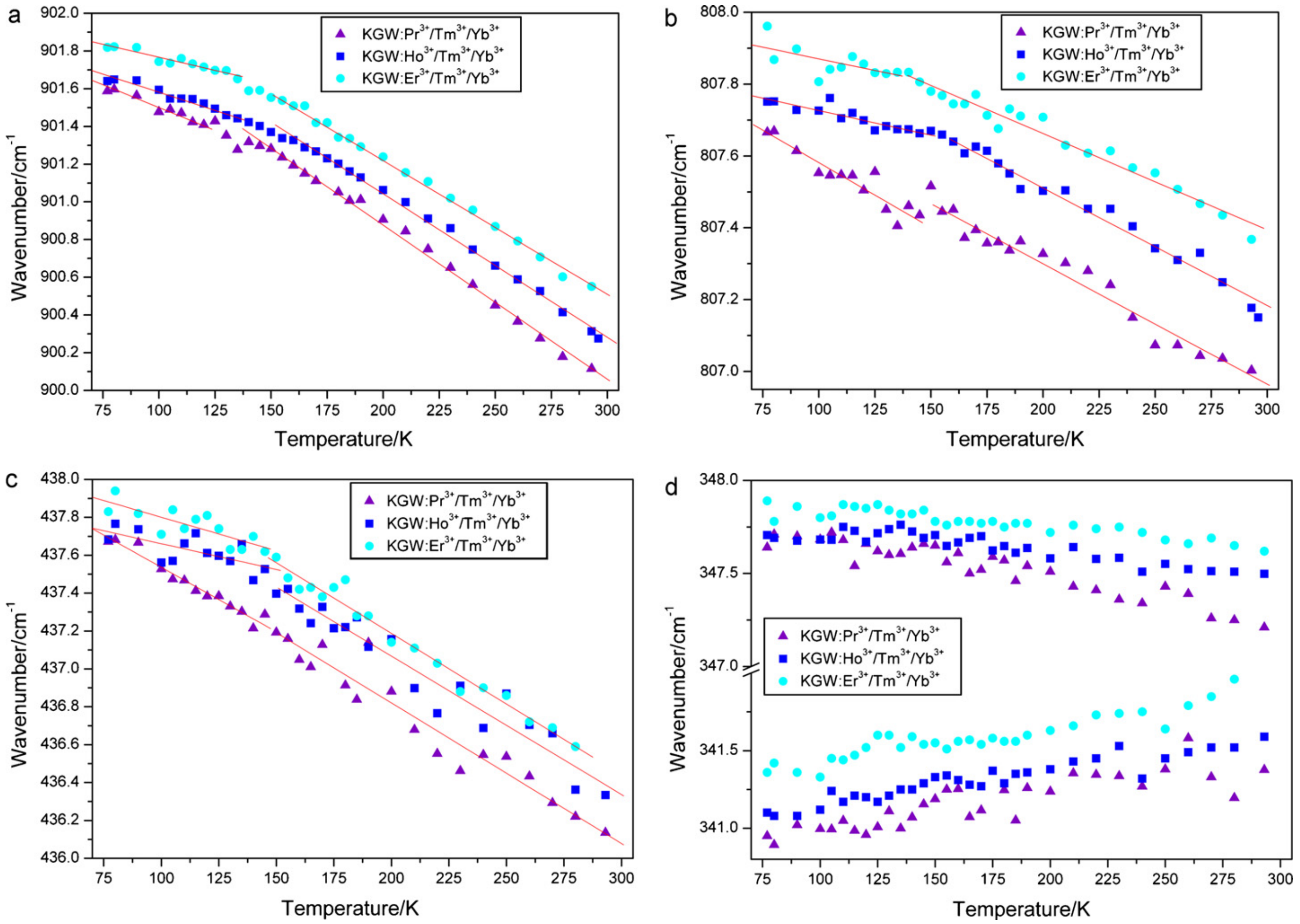

Fig. 2. The low-temperature dependences of vibrational modes at (a) 900 cm^{-1} (b) 807 cm^{-1} (c) 436 cm^{-1} (d) 348 and 342 cm^{-1} of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals.

bands of $Pr^{3+}/Tm^{3+}/Yb^{3+}$ in comparison to $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals.

To determine the influence of temperature on the investigated crystals the spectral parameters of selected modes located at about 900, 807, 766, 746, 436, 348 and 342 cm^{-1} were analyzed in the 77–293 K temperature range for all crystals. The low-temperature dependences of the frequency of these modes are presented in Figs. 2 and 3. As it can be seen the similar influence of temperature on all investigated crystals was observed and the temperature dependences of analyzed modes have very similar character. The frequency of the bands at about 900 cm^{-1}, related with symmetric stretching vibrations of the WO_4^{2-} tetrahedra, changes linearly from 293 to 77 K. However, we observed evidently the change of slope near 145 K. The detailed analyzes allowed us to determine that the anomaly is slightly above 145 K for $Pr^{3+}/Tm^{3+}/Yb^{3+}$ and below 145 K for $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. Similarly, the frequency of the bands at about 805 cm^{-1} attributed to coupling stretching vibrations of WO_4^{2-} tetrahedra and WOW bridge bonds, changes linearly from 293 to 77 K, with some anomaly at about 145 K. This is probably connected with the local distortion of WO_4^{2-} tetrahedra due to the mismatch of the ionic radii of Pr^{3+}, Er^{3+}, Ho^{3+}, Tm^{3+} or Yb^{3+} ions in the neighboring cationic layers. As a consequence we observe more rapid decrease of frequency of selected modes due to the contraction of W–O bonds in WO_4^{2-} tetrahedra and WOW bridges during cooling the samples from 293 to about 145 K than below 145 K. Probably, at this temperature the inter-molecular distances slightly changes and stronger distortion of WO_4^{2-} tetrahedra occurs. The further slow lowering of frequency of analyzed bands, from about 145 to 77 K, is a consequence of limited vibrations of more rigid, distorted WO_4^{2-} tetrahedra. The low-temperature dependences of the frequency of selected modes located at 436 cm^{-1}, assigned as bending vibrations of WOW, are also nonlinear with some anomaly at about 145 K (Fig. 2c) observed for $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. Moreover, the temperature dependences of deformation bending vibrations of WO_4^{2-} tetrahedra at 348 and 342 cm^{-1} show opposite behavior on cooling investigated crystals from 293 down to 77 K. As shown in Fig. 2d the wavenumber of 348 cm^{-1} bands slightly increases, while the 342 cm^{-1} bands decreases with lowering temperature. This anomalous shift of the vibrational frequency of 342 cm^{-1} bands with lowering temperature is due to the slight dislocation of oxygen atoms in WO_4^{2-}, which lead to the described above stronger distortion of WO_4^{2-} tetrahedra in low temperature range.

The peaks at about 766 and 746 cm^{-1} are assigned to the stretching vibration of WOOW oxygen bridges and coupling stretching vibrations WO_4^{2-} tetrahedra. During decreasing the temperature from 293 to 77 K the modes at about 746 cm^{-1} shift towards higher frequencies, while the bands at about 766 cm^{-1} are shifted to lower frequencies, similarly for all investigated crystals (Fig. 3a). The anomalous shift of the frequency of the 766 cm^{-1} bands with lowering temperature is due to the elongation of W–O bonds in the

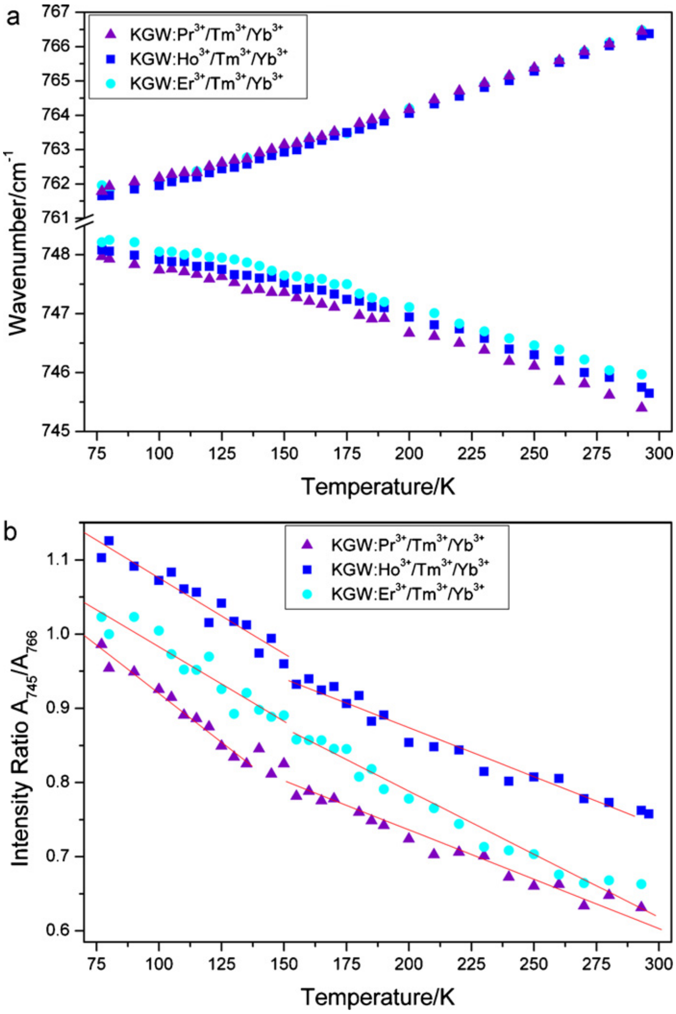

Fig. 3. The low-temperature dependences of stretching vibrational modes of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals: wavenumber of (a) 746 and 766 cm^{-1} modes and (b) integral intensity ratio A_{746}/A_{766} of 746 and 766 cm^{-1} modes.

WOOW, which results from the strengthening of intermolecular interaction between the adjacent WO_4^{2-} ions.

The intensity ratio A_{746}/A_{766}, which is the ratio of integral intensity of 746 and 766 cm^{-1} bands presents the relative activity of stretching vibrations of WO_4^{2-} tetrahedra and the WOOW molecular groups. The temperature dependences of the ratio A_{746}/A_{766} show different slope below and above the temperature at about 145 K (Fig. 3b). This results from the higher contribution of stretching vibrations of WO_4^{2-} tetrahedra than WOOW groups in the higher temperatures than below 145 K. Below this temperature the stretching vibrations of the more rigid and distorted WO_4^{2-} tetrahedra are less active than the stretching vibrations of W–O bonds in WOOW oxygen bridges. It should be noted that such anomalies in low-temperature range were not observed for 900, 766, 754, 743, 684, 526, 371, 348, 342, 206 and 88 cm^{-1} Raman bands for undoped KGW crystals. Moreover, the frequencies of most of these selected modes shift towards higher frequencies excluding the frequency of 766 cm^{-1} mode, which decreases with lowering temperature from 293 down to 77 K. Similar behavior was also observed for single doped with Er^{3+}, Eu^{3+}, Ho^{3+} or Nd^{3+} KGW crystals (for low concentration below 3%), whereas for doubly doped with Yb^{3+} and Er^{3+} ions KGW crystals we observed some anomalies of vibrational properties in low-temperature range [21]. Moreover, the low-temperature Brillouin scattering measurements show also some anomaly (in Brillouin shift and integral intensity values) of the longitudinal acoustic phonons [101] propagating in doubly doped KGW: Er^{3+}/Yb^{3+} crystals [10].

4. Conclusions

We present results of micro-Raman investigation for triply-doped KGW crystals with compositions of $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ ions. The polarized Raman spectra were analyzed in the spectral range of internal vibrational motions from 260 to 1000 cm^{-1}. The Raman modes detected in this spectral range are assigned to the stretching and bending vibrations of WO_4^{2-} tetrahedra and WOW and WOOW oxygen bridges. It was found that the used concentration and combination of rare earth ions in the investigated KGW crystals does not significantly influence on the position and the relative intensity of Raman bands at room temperature. However, some slight differences in the values of wavenumbers for the corresponding Raman bands in $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals occur. The highest values of wavenumbers of Raman bands were observed for $Pr^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. In this crystal the highest average ionic radius of doping ions occurs, due to the highest ionic radius of Pr^{3+} ions, which substituted the Gd^{3+} ions in crystalline lattice. Thus, the inter-atomic distances are slightly shorter, which results in higher frequency vibrations and shift of wavenumbers of some Raman bands of $Pr^{3+}/Tm^{3+}/Yb^{3+}$ in comparison to $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. As it was mentioned earlier, the slight local distortion of WO_4^{2-} tetrahedra at room temperature is present in KGW crystals. This is due to the random distribution of K^{+} and Gd^{3+} ions, which occupy the same crystallographic positions in KGW crystal lattice. For the doped crystals stronger distortion probably occurs due to the mismatch of the ionic radii of Pr^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+} or Yb^{3+} ions replacing the Gd^{3+} ions in their positions in the neighboring cationic layers. During cooling down the crystals from 293 to 77 K the frequency of 900, 807, 746, 436, 348 and 296 cm^{-1} modes increases due to the contraction of W–O bonds in WO_4^{2-} tetrahedra, WOW and WOOW oxygen bridges, while the frequency of 766 cm^{-1} modes attributed to the stretching vibration of WOOW oxygen bridges, slightly decreases in this temperature range due to the elongation of W–O bonds. The anomalous shift to lower frequencies, with lowering temperature, was also observed for bands at 342 cm^{-1} assigned as deformation bending vibrations of WO_4^{2-}. This phenomena occurs due to the slight dislocation of oxygen atoms in WO_4^{2-}, which lead to stronger distortion of WO_4^{2-} tetrahedra in low temperature range. The low-temperature dependences of the frequency of selected modes located at 900, 807, 766, 746 and 436 cm^{-1} show some anomaly at about 145 K for $Pr^{3+}/Tm^{3+}/Yb^{3+}$, $Ho^{3+}/Tm^{3+}/Yb^{3+}$ and $Er^{3+}/Tm^{3+}/Yb^{3+}$ doped KGW crystals. This is probably connected with the dominant contribution of vibrations of WO_4^{2-} tetrahedra above 145 K temperature. At this temperature the inter-atomic distances slightly changes and stronger distortion of WO_4^{2-} tetrahedral occurs. As a consequence below the characteristic temperature 145 K more limited vibrations of more rigid, distorted WO_4^{2-} tetrahedral occur. The further investigations of described rare-earth triply-doped systems including IR spectroscopy and low-temperature X-ray measurements will be continued.

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