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High pressure Raman scattering study on the phase stability of $LuVO_4$

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ABSTRACT

High pressure Raman spectroscopic investigations have been carried out on rare earth orthovanadate $LuVO_4$ upto 26 GPa. Changes in the Raman spectrum around 8 GPa across the reported zircon to scheelite transition are investigated in detail and compared with those observed in other vanadates. Co-existence of the zircon and scheelite phases is observed over a pressure range of about 8–13 GPa. The zircon to scheelite transition is irreversible upon pressure release. Subtle changes are observed in the Raman spectrum above 16 GPa which could be related to scheelite ↔ fergusonite transition. Pressure dependencies of the Raman active modes in the zircon and the scheelite phases are reported.

1. Introduction

Compounds of the family ABX_4 are widely used as solid state scintillator materials, laser host materials, in opto-electronic devices, etc. So there is a renewed interest in the study of the phase transition behavior of these systems. Some of the extensively investigated systems of this family are zircon [1], orthovanadates [2,3], chromates [4], phosphates [5], fluorides [6], orthotungstates [7,8], and molybdates [9]. Most of these compounds crystallize either in zircon [space group $I4_1/amd$, $Z=4$] or scheelite structure [space group $I4_1/a$, $Z=4$] at ambient conditions. The rare-earth orthovanadates RVO_4 ($R=$ rare-earth elements, Sc and Y) crystallize in zircon structure at ambient conditions. These compounds are attractive from the point of technological applications owing to their properties such as large refractive indices, non-linear coefficients, birefringence and effectively no IR absorption from ~2.5 to 15 μm [10]. Recently, $LuVO_4$ has been found to be a promising material for self-Raman laser converters [11]. The high pressure properties of this series of compounds are interesting since at moderate pressures (~8 GPa) the zircon structure transforms to a denser scheelite-type phase [2–4,12,13] irreversibly whereas at lower temperatures ~40 K some of the RVO_4 members transform to a lower symmetry structure via a cooperative Jahn–Teller transition [14]. Upon further pressurization, many of the scheelite structured compounds are reported to transform to a lower symmetry monoclinic phase [15,16].

The synchrotron-based ADXRD investigations on the high pressure behavior of $LuVO_4$ revealed the existence of zircon to scheelite transition at 8 GPa [17]. Interestingly, while lattice dynamical calculations in $LuVO_4$ suggested that there are no dynamical instabilities in the scheelite phase upto 100 GPa, ADXRD results reported phase transition to a monoclinic *M*-fergusonite ($I2/a$) structure above 16 GPa [17]. There have been several theoretical and experimental efforts to identify the post-scheelite structures, which, in many cases is a monoclinic phase *M*-fergusonite ($I2/a$), wolframite ($P2/c$), *M′*-fergusonite ($P2_1/c$), $LaTaO_4$, $BaWO_4$-II ($P2_1/n$), etc. Based on analysis of spontaneous strain in the monoclinic fergusonite phase, scheelite ↔ monoclinic transitions in orthotungstates are inferred to be second order phase transitions [18]. The associated volume change across the transition is very small, often ~0.5% [7,19]. This also makes it very difficult to distinguish between different monoclinic phases like wolframite, fergusonite, etc. [7,20,21]; for example, there were controversies in assigning the high pressure phase of $CaWO_4$ [7,21,22]. It is now suggested that, of the different possible post-scheelite monoclinic structures, wolframite structure is favoured under non-hydrostatic conditions compared to fergusonite structure in $CaWO_4$ [7]. Due to the subtle nature of the transition, it is often difficult to detect the post-scheelite transition and even more difficult to identify the space group of the high pressure phase. Many in-situ techniques have been used such as photoluminescence spectroscopy [23,24], X-ray absorption near edge structure (XANES) at high pressure to identify the post-scheelite transition [7,8]. Ab initio lattice dynamical [25–27] and density functional calculations [28] have been used to predict the post-scheelite phases. Since Raman spectroscopy is more sensitive to local structural changes, it has often proved useful in identifying

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subtle phase transitions. Study of scheelite–fergusonite transition in $R\mathrm{VO}_4$ by Raman spectroscopy is very rare. In the present work, we have carried out Raman spectroscopic investigations on LuVO_4 at high pressure upto 26 GPa with a view to obtain insight into the nature of post-scheelite phases.

2. Experimental details

The compound LuVO_4 was prepared by solid state reaction route, heating appropriate amounts of dried $\mathrm{Lu_2O_3}$ (99%) and $\mathrm{V_2O_5}$ (99%) mixed in 1:1 molar weight at 800 °C for 18 h followed by regrinding and heating at 975–1000 °C for 24 h. Samples obtained after second heat treatment were characterized by powder X-ray diffraction. The phase purity of compound synthesized was confirmed from the powder XRD pattern; no detectable intensity corresponding to the unreacted $\mathrm{Lu_2O_3}$ or $\mathrm{V_2O_5}$ was observed. Rietveld refinement of the pattern yielded the cell parameters of the zircon phase to be $a = 7.0230$ Å, $c = 6.2305$ Å [29], which are in close agreement with the reported values [30].

Raman scattering measurements at high pressure were carried out using a Mao–Bell-type diamond anvil cell (DAC), with 400 μm diameter culet diamonds and hardened steel gasket. A 4:1 methanol–ethanol mixture was used as pressure transmitting medium. Pressure inside the cell was measured using the standard ruby fluorescence technique [31]. Raman spectrum of unoriented LuVO_4 from inside the DAC was excited using 532 nm line of power ~15 mW. Scattered light was analyzed using a home built 0.9 m single monochromator [32], coupled with a super notch filter and detected by a cooled CCD (Andor Technology). Entrance slit was kept at 50 μm, which gives a spectral band pass of 3 $\mathrm{cm^{-1}}$.

3. Results and discussion

In the zircon phase, group theory predicts 12 distinct Raman active modes $2A_{1g}+4B_{1g}+B_{2g}+5E_g$ in LuVO_4. Raman spectra at ambient conditions agreed well with that reported [33]. Of the 9 distinct Raman bands observed in the present work, the highest frequency mode at 899 $\mathrm{cm^{-1}}$ is associated with the symmetric stretching and the modes at 845 and 825 $\mathrm{cm^{-1}}$ are asymmetric stretching modes [33] of the VO_4 unit. The modes in the range 300–500 $\mathrm{cm^{-1}}$ correspond to the VO_4 bending vibrations, and those below 270 $\mathrm{cm^{-1}}$ are the external modes. The Raman spectra of LuVO_4 at different pressures are shown in Fig. 1. Two of the external modes at 159 (E_g) and 261 $\mathrm{cm^{-1}}$ (B_{1g}) exhibit softening with pressure. All the other modes increase in frequency with increasing pressure. Around 8 GPa, discontinuous changes are noted in the Raman spectra. New set of modes appear in the bending mode region above 8 GPa. Two strong modes at around 390 $\mathrm{cm^{-1}}$ (at 10 GPa) are due to symmetric bending of the VO_4 unit [3]. The high frequency band (440–500 $\mathrm{cm^{-1}}$ at 10 GPa), corresponding to the asymmetric bending mode [3] has two components. However the intensity of one of them was too weak to be followed at high pressures. New modes appear in the external mode region also. In the V–O stretching region, intensity of the symmetric stretching mode at around 899 $\mathrm{cm^{-1}}$ of zircon phase starts reducing, with new modes appearing at lower frequencies as shown in Fig. 1. Changes observed in Raman spectra around 8 GPa closely resemble those reported across zircon–scheelite transition in YVO_4 [2], TbVO_4, and DyVO_4 [3]. Group theoretical analysis predicts $3A_9+5B_g+5E_g$ Raman active modes for the scheelite phase of which seven are internal modes: three stretching vibrations of the VO_4 tetrahedra (A_g, E_g and B_g) and four bending vibrations A_g, $2B_g$ and E_g. Additionally, six external modes are predicted of which two are librations (A_g and E_g) and four are translations ($2B_g$, and $2E_g$). At high pressures, eleven out of the thirteen expected Raman active modes in the scheelite phase could be observed with measurable intensity. In the scheelite phase, symmetry of the modes is identified by comparing the spectrum of the scheelite phase recovered at ambient conditions with that reported in other orthovanadates [2,3].

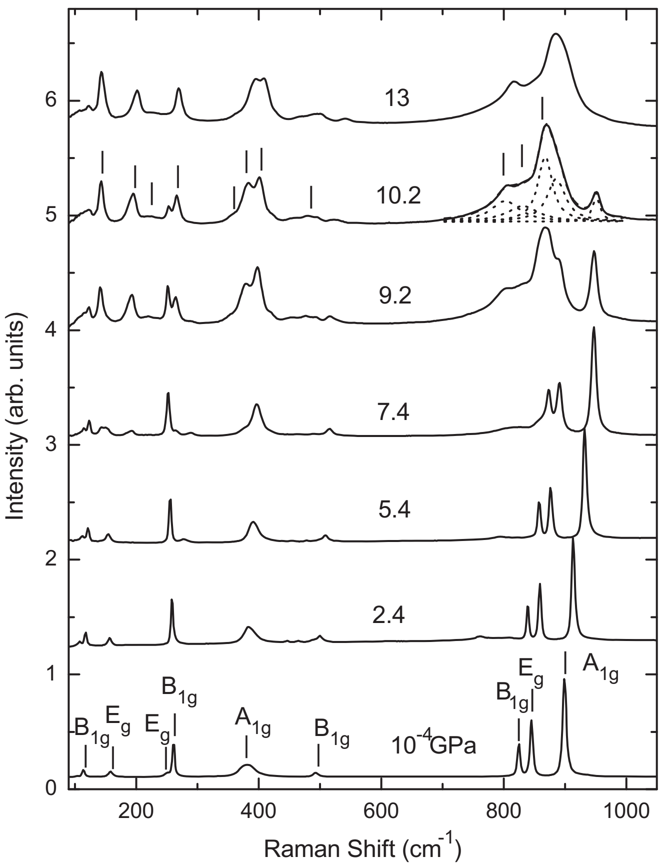

Fig. 1. Raman spectra of LuVO_4 as a function of pressure. In the Raman spectrum at ambient pressure, the symmetry of the modes in the zircon phase are marked. Note the abrupt changes in the Raman spectra above 7.4 GPa. The bars marked in the spectrum at 10.2 GPa are the peaks due to scheelite phase which were followed at higher pressures. The V–O stretching region in the Raman spectra at 10.2 GPa, is fitted to five Lorentzians (shown by dotted lines), three of which are due to scheelite phase and the two are due to zircon phase.

Co-existence of phases is noted over a large pressure range. At around 10.2 GPa, the V–O stretching region consists of five bands of which three bands are of the scheelite phase and two of the high frequencies bands are due to the zircon phase as shown in Fig. 1. Earlier Raman spectroscopic studies [2] on YVO_4 also observed discontinuous decrease in V–O stretching frequencies across the zircon to scheelite transition which was understood as arising from increase in V–O bond length or charge transfer. Recent X-ray diffraction studies in YVO_4 [16] showed an increase in V–O bond length across the zircon to scheelite transition that explains the decrease in V–O stretching frequency across the transition. In LuVO_4, X-ray diffraction studies [17], however, did not show any indications for increase in V–O bond length. On the other hand, the Lu–V distances were found to increase significantly [17] leading to large changes in the interatomic interaction across the zircon–scheelite transition. Table 1 summarizes the Raman mode frequencies observed in the zircon and the scheelite phases. Zircon to scheelite transition in LuVO_4 is irreversible and

Table 1
Ambient pressure phonon frequencies, their pressure derivatives, and mode Gruneissen parameters for various modes of $LuVO_4$.

Zircon phase				Scheelite phase		
Symmetry (zircon phase)	Frequency (cm^{-1})	$d\omega/dP$ ($cm^{-1}\,GPa^{-1}$)	γ_Z	Frequency (cm^{-1})	$d\omega/dP$ ($cm^{-1}\,GPa^{-1}$)	γ_S
B_{1g}	115	1.12(1)	1.43	139	0.38(16)	0.53
E_g	159	−0.85(2)	−0.79	180	1.57(25)	1.69
E_g	249	5.41(19)	3.19	197	2.51(16)	2.47
B_{1g}	261	−1.18(7)	−0.66	256	1.02(1)	0.77
A_{1g}	378	2.36(3)	0.92	331	2.35(20)	1.38
B_{1g}	492	3.06(1)	0.91	349	3.41(30)	1.27
B_{1g}	825	6.31(25)	1.12	376	2.46(19)	1.26
E_g	845	6.01(24)	1.04	440	4.0(4)	1.76
A_{1g}	899	6.31(24)	1.03	779	2.6(3)	1.1
				787	4.4(7)	1.08
				853	1.9(5)	0.43

The mode assignments in zircon phase are taken from Ref. [33]. Bulk modulus $B = 147$ GPa for zircon phase, $B = 194$ GPa for scheelite phase, from Ref. [17]. Note that two of the external modes in zircon phase have negative mode Grüneissen parameter. Frequencies in the scheelite phase are the values extrapolated to ambient pressure.

the scheelite phase could be quenched at ambient conditions just as in the case of other orthovanadates [2,3].

In the scheelite phase, all the modes increase in frequency with pressure. This is in agreement with lattice dynamical calculations, which did not show any soft mode in the scheelite phase [17]. This behavior is unlike in some other ABO_4 compounds such as tungstates wherein a low frequency (B_g) mode (which involves the translation of the BO_4 tetrahedron in the c-direction [34]) or E_g mode in $YLiF_4$ [35] softens in the scheelite phase. While B_g mode softens across transition to M-fergusonite phase, E_g mode softens across transition to M' -fergusonite phase ($P2_1/c$). In the present studies, a low frequency mode (139 cm^{-1} at ambient pressure) in the scheelite phase has very less pressure dependence. On further pressurization, subtle changes in the Raman spectra are noted above 16 GPa across the reported scheelite to fergusonite transition [17]. There is an overall change in the lineshape of the V–O stretching mode. The broad mode around 820 cm^{-1} develops asymmetry on the low frequency side around 770 cm^{-1} above 16 GPa, as shown in Fig. 2. This mode belongs to E_g symmetry in the scheelite phase and is expected to split in the monoclinic fergusonite phase [25]. Pressure dependencies of some of the modes change considerably above 16 GPa as shown in Fig. 3. The $d\omega/dP$ of the 349 cm^{-1} mode of scheelite phase decreases from a value of 3.41 to 1.27 $cm^{-1}\,GPa^{-1}$ above 16 GPa. There is a marked change in the pressure dependencies of the mode frequencies of the V–O stretching modes also (Fig. 3). The evolution of Raman spectra is monotonous above this pressure upto 26 GPa, the highest pressure reached in the present studies.

The pressure dependence of the low frequency mode at 139 cm^{-1} increases from 0.37 to 1.8 $cm^{-1}\,GPa^{-1}$ around 16 GPa. Pressure dependence of the translational mode changing significantly across the scheelite–fergusonite transition has been reported in many of the tungstate/molybdates. In $CaWO_4$, it changes from a weak negative value to a positive value in fergusonite phase. In fact, softening of the translational mode is considered as a signature of the zone center instability in scheelite phase [36]. Pressure dependencies of the mode frequencies in $LuVO_4$ are tabulated in Table 1 and mode Grüneissen parameters are calculated for the zircon and scheelite phases. Pressure dependencies are obtained from linear fit to the data in the pressure range $0 \leq P \leq 7.5$ GPa for the zircon phase and $8.2\,\text{GPa} \leq P \leq 16$ GPa for the scheelite phase. The bulk modulus and its derivative reported in [17], obtained by fitting the experimental pressure–volume data to Birch–Murnaghan Equation of State: $B = 147$ GPa and $B' = 4.3$ for zircon phase; $B = 194$ GPa and $B' = 5.3$ for the scheelite phase—were used for calculating the mode Grüneissen parameters. Fig. 4 shows the Raman spectra of $LuVO_4$ in the pressure reducing cycle. It may be noted from the figure that $LuVO_4$ reverses to scheelite phase around 16 GPa without any detectable hysteresis and remains in scheelite phase when the pressure is released.

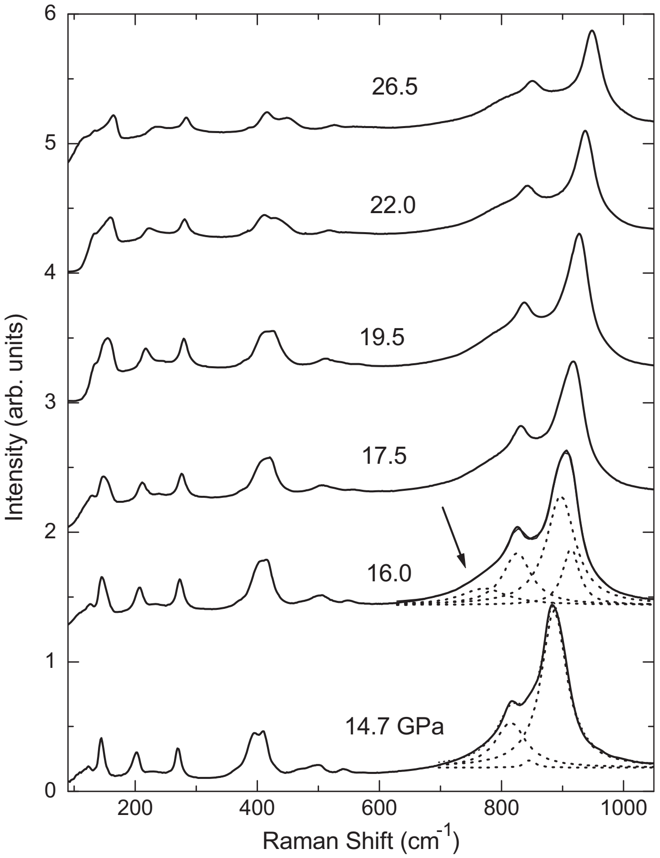

Fig. 2. Raman spectra of $LuVO_4$ at various high pressures. The V–O stretching region above 16 GPa can be fitted to four Lorentzians. Arrow mark indicates appearance of a new mode.

Scheelite ↔ fergusonite transition in an ABX_4 compound is considered as involving small displacements of A and B atoms from their high symmetry positions and relatively large changes in the X position [15]. As fergusonite structure is a distorted version of scheelite structure, at pressures close to transition

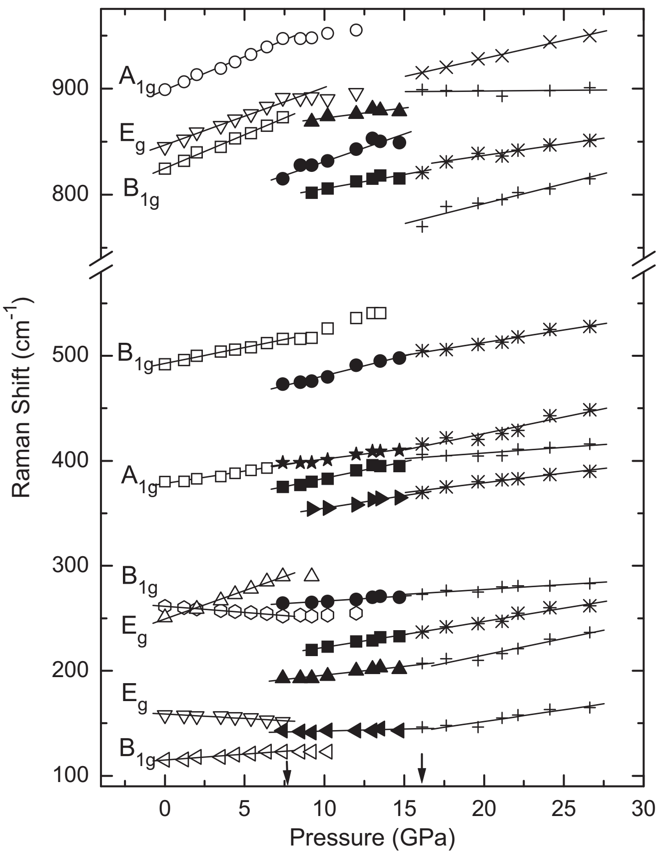

Fig. 3. Pressure dependencies of mode frequencies in $LuVO_4$. Symmetry of the modes in the zircon phase are labeled. Arrow marks indicate the transition pressures. Open symbols denote the zircon phase, closed symbols the scheelite phase and the remaining symbols denote fergusonite phase. Solid lines denote the linear fit of the data within a phase. Note the changes in the V–O stretching mode frequencies around 16 GPa.

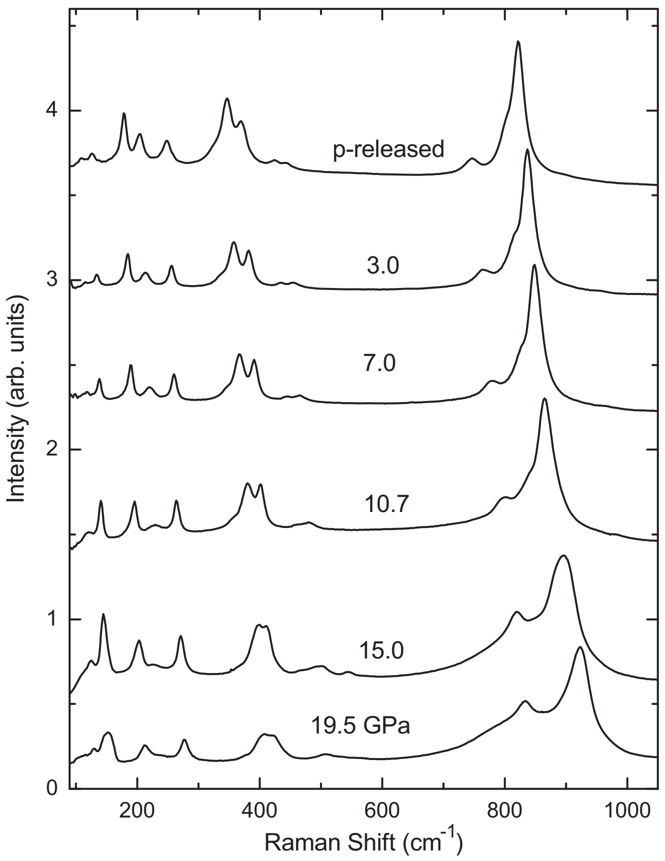

Fig. 4. Raman spectra of $LuVO_4$ in the pressure releasing cycle. Note the reversibility of scheelite to fergusonite transition and irreversibility of zircon to scheelite transition.

pressure, it is often difficult to identify the transition from structural studies. Since the coordination of vanadium in $LuVO_4$ in fergusonite phase is same as that in scheelite phase, changes expected in the frequencies of internal modes are small. Splitting of the doubly degenerate modes is generally expected across transition from tetragonal to monoclinic structure. In $LuVO_4$, we observe splitting of only V–O stretching mode of E_g symmetry across scheelite ↔ fergusonite transition. Based on a systematic study of phase transition in scheelite structured compounds of the family ABX_4, Errandonea and Manjón have pointed out that the transition pressure to fergusonite phase increases as the packing ratio of anionic BX_4 around the cations A increases [15,37]. They have formulated an empirical relation to estimate the transition pressure in a scheelite ABX_4 compound in terms of radius ratio BX_4/A [37]. This ratio for $LuVO_4$ from literature [38] is 1.81 that gives an upper estimate of transition pressure of about 13 GPa. This agrees well with the observed scheelite to fergusonite transition pressure of 16 GPa.

From Table 1, it is clear that the mode at 139 cm^{-1} in the scheelite phase has very less pressure dependence. By comparison with the modes of scheelite phase of $DyVO_4$ [3], this mode appears to be due to the translation of VO_4 tetrahedra. Its invariance under pressure indicates interaction between VO_4 tetrahedra remain unchanged or even weaken under pressure [39]. In tungstates, across the scheelite to fergusonite transition, a translational mode belonging to B_g symmetry softens in the scheelite phase; becomes A_g mode in fergusonite phase and thereafter it continues to harden [36]. It has been conjectured in [36], that the frequency of this translational mode in the scheelite phase (at ambient conditions) is related to the transition pressure. Errondonea et al. [36] have established a relation between the square of the soft mode frequency (ω) at ambient pressure and the transition pressure (P_t) for scheelite to fergusonite transition for tungstates and molybdates in accordance with the soft mode theory of second order transition. According to [36], for a given class of compounds, say tungstates, the ratio of square of ω to transition pressure P_t is a constant. In YVO_4, Wang et al. have identified the onset of scheelite to fergusonite transition at 26.5 GPa by ADXRD [16]. Recent high pressure Raman experiments [36] on YVO_4 have detected the frequency of the scheelite type B_g mode that softens to be at 166 cm^{-1} (at ambient pressure). In $LuVO_4$, the frequency of the relevant translational mode is 139 cm^{-1}. Extending this approach to the orthovanadates, to predict the transition pressure in other members of the class the transition pressure is estimated to be 18.5 GPa for $LuVO_4$. It is thus seen that for the family of vanadates also, there exists a linear relation between the square of the translational mode frequency and the transition pressure implying that the second order transition to monoclinic phase could be driven by a translation mode.

4. Conclusion

High pressure behavior of $LuVO_4$ is investigated upto 26 GPa using Raman spectroscopy. $LuVO_4$ transforms to scheelite phase above 8 GPa characterized by discontinuous changes in the Raman spectra. Co-existence of zircon and scheelite phases is observed upto 13 GPa. Mode Grüneisen parameters of $LuVO_4$ have been calculated in the zircon and scheelite phases. Subtle changes in the Raman spectra observed above 16 GPa are compatible with scheelite $LuVO_4$ transforming to monoclinic fergusonite phase as suggested by the ADXRD results in the literature. $LuVO_4$ remains in scheelite phase on releasing the pressure.

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