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Study of magnon mode characteristics in solid oxygen by high resolution spectroscopy

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Abstract

The higher energetic magnon at 27 cm^{-1} was systematically investigated by far-IR-absorption and Raman scattering in $\alpha\text{-O}_2$ ($T = 11\text{--}24\text{ K}$). The softening of the magnon frequency—described in mean field theory—can be explained by a weakening of the exchange interaction due to thermal expansion and by a loss of magnetic order due to thermal fluctuations. Bandwidth as a function of temperature can be fitted by a power law, with a zero bandwidth at $T = 0\text{ K}$ ($\Gamma = aT^{4.4}$). Dephasing processes via magnon–magnon scattering are the only meaningful relaxation mechanisms of this magnon mode. The integrated band intensity, normalized to our low temperature value, directly follows the spin–spin correlation function and describes the long range magnetic order parameter in $\alpha\text{-O}_2$. The spectral characteristics, band frequency and band intensity (ω and I) prove that the $\alpha\text{--}\beta$ phase transition (at $T_{\alpha\beta} = 23.8\text{ K}$) is not purely magnetic.

1. Introduction

The oxygen molecule possesses a non-zero electronic spin $S = 1$ in its electronic ground state. Therefore, solid oxygen exhibits both the properties of a molecular crystal and a magnetic material. Due to this unusual combination of properties, solid oxygen is an object of fundamental interest for experimental and theoretical studies.

At ambient pressure, oxygen exists in three crystallographic low temperature modifications [1]. The most interest is attracted by the low-temperature α -phase of oxygen (existing at temperatures $T \leq 23.8\text{ K}$), since the transition to this phase is accompanied by antiferromagnetic spin ordering. Therefore, besides the usual phonon, libron and exciton states typical for molecular crystals, spin waves, exciton–magnon and bi-exciton states (typical for other antiferromagnetic insulators) are also present in the spectra of α -oxygen. Since the binding energy of molecular crystals is mostly determined by weak van-der-Waals forces, the

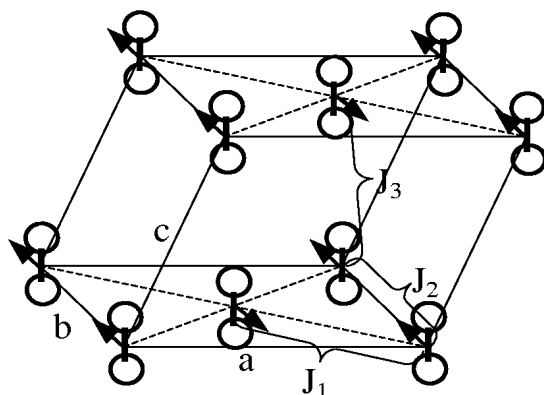


Figure 1. Details of the crystallographic and magnetic structure of α -oxygen from figures in [1] and [2]. The lattice parameters are $a = 5.375 \text{ \AA}$, $b = 3.425 \text{ \AA}$, $c = 4.242 \text{ \AA}$. The monoclinic angle is $\beta = 117.78^\circ$. The distance of $\text{O}_2\text{-O}_2$ within the plane (3.187 and 3.425 \AA) and out of plane (4.187 \AA) are relevant here, as well as the respective exchange interaction constants $J_1 > J_2$ or J_3 .

magnetic exchange interaction—in the case of $\alpha\text{-O}_2$ —makes a considerable contribution to the total crystal energy (about 10%, while normally the magnetic interaction contribution to the total energy is at most about 1% [3]). Consequently, experimental and theoretical studies on spectra of elementary excitations in solid oxygen produce significant difficulties since one has to deal here with coupling of translational, librational and magnetic degrees of freedom.

The α -phase of oxygen (see figure 1) is orientationally ordered and possesses a monoclinic base-centred lattice (space group $C2/m$) with one molecule per primitive structural unit cell [1]. The molecular axes in α -oxygen are collinear and perpendicular to the (001) layers and the distance between nearest neighbour molecules within the basal plane is substantially smaller than that between molecules in adjacent layers. According to neutron diffraction data, $\alpha\text{-O}_2$ is a two-sublattice collinear antiferromagnet with two magnetically non-equivalent molecules per unit cell [4].

It is well known that two magnon modes exist in α -oxygen at the centre of the Brillouin zone and are either IR or Raman active in the magnetic dipole approximation. Both of them have been detected in far-infrared absorption spectra at $\sim 27 \text{ cm}^{-1}$ [5–8] and at $\sim 6.4 \text{ cm}^{-1}$ [6, 7]. The magnetic nature of these excitations was proven by the absence of an isotopical frequency shift for the high-frequency mode [6] and by the dependence of their frequencies on applied magnetic fields [6, 7]. In Raman scattering experiments [9], only the high-frequency mode was detected at 27.2 cm^{-1} at $T = 4.3 \text{ K}$ and the missing isotope shift was also confirmed.

The temperature dependence of the high frequency magnon was studied by several groups with similar results (wavenumber accuracy $\pm 0.5 \text{ cm}^{-1}$) [5, 7, 8]. None of these authors discussed quantitatively aspects such as the magnetic order parameter, mode relaxation or magnon–libron interaction. Only Meier *et al* [7] discussed the softening of the high-frequency magnon mode and stated that the librational states become more populated with increasing temperature, with a consequent lowering of exchange interaction J due to librational excitations, implying a softening of magnon frequency. Since most of these studies were performed below 10 K, librational modes are neither significantly populated ($\omega(B_g) \sim 43 \text{ cm}^{-1}$, $\omega(A_g) \sim 80 \text{ cm}^{-1}$) nor do they possess a significant root-mean-square torsional amplitude ($\langle \tilde{\Theta}^2 \rangle \approx 9^\circ$) [10]. In our opinion, a proper explanation for the decrease of magnon mode frequency with increasing temperature is still missing. It should be noted

that previous studies [6–9] were performed either with low resolution ($\sim 1 \text{ cm}^{-1}$) or with large wavenumber inaccuracy ($\pm 0.5 \text{ cm}^{-1}$). Therefore, both magnon mode characteristics—band frequency $\omega(T)$ and bandwidth $\Gamma(T)$ —could not be discussed quantitatively. In addition, most spectra in the literature were so weak that it was not possible to apply a real band shape analysis (Gaussian part: inhomogeneous broadening + apparatus function, Lorentzian part: homogenous broadening). Finally, there are no statements about band intensities $I(T)$ as a function of temperature, especially close to the α – β phase transition (antiferromagnetic order to short-range magnetic order). The low frequency magnon mode at $\sim 6 \text{ cm}^{-1}$ was previously observed twice in far IR studies [6, 7] and only at $T < 5 \text{ K}$. However, the method was no Raman scattering and gave no further spectroscopic characterization of band frequency $\omega(T)$, bandwidth $\Gamma(T)$ and band intensity $I(T)$.

Therefore, we performed systematic high resolution far-infrared and Raman scattering studies of α -oxygen on optically perfect samples between 10 and 25 K at equilibrium vapour pressure.

2. Experimental details and data

In our experiments we used an oxygen gas of 99.998% purity. Samples were grown from the liquid phase in a brass cell with quartz windows (aperture 10 mm). Our technique for growth of large polycrystalline samples for optical investigations is described in detail elsewhere [11]. Briefly, every cooling process from gas to α -phase was performed extremely slowly (0.5 – 1 K h^{-1}), the solid sample was annealed slightly below the crystallization point ($\Delta T \sim 0.1$ – 0.5 K) for about 10–20 h. A substantially slower cooling rate (0.05 K h^{-1}) was applied through each phase transition region (liquid $\rightarrow \gamma$, $\gamma \rightarrow \beta$, $\beta \rightarrow \alpha$). Two samples with thickness 5 and 6.7 mm were studied by IR spectroscopy and one sample with thickness 4 mm by Raman scattering during heating and cooling cycles. The sample temperature was determined by a calibrated Si-diode with an accuracy of $< 0.1 \text{ K}$ attached to the optical cell.

FTIR spectra were recorded in the far-infrared region by a Fourier transform spectrometer (Bruker IFS 120 HR) equipped with a Hg lamp, $75 \mu\text{m}$ -Mylar beam splitter and liquid He-cooled Ge-bolometer (resolution up to 0.05 cm^{-1}). Ordinary Raman spectra were excited by an Ar⁺-laser with 200–300 mW on the sample. As Raman configuration we used a quasi-back-scatter arrangement with a $\sim 30^\circ$ angle of incidence. The spectra were registered by a triple spectrometer in conjunction with a CCD camera (Jobin Yvon T64000; resolution 0.8 cm^{-1} and wavenumber accuracy of $\pm 0.2 \text{ cm}^{-1}$).

Figure 2 illustrates the temperature evolution of magnon spectra ($\omega \sim 27 \text{ cm}^{-1}$) by (a) IR absorption and (b) Raman scattering, measured for the first time over the whole temperature range of α -O₂. Both types of spectra demonstrate the same overall temperature dependence: during heating, band frequency decreases and bandwidth increases. The band intensity rapidly decreases and disappears completely after the α – β phase transition. The inset in figure 2(a) shows the magnon band shape at $T = 15 \text{ K}$, characterized by a much smaller bandwidth than previously cited (about 1 – 1.5 cm^{-1} at $T \sim 4$ – 6 K [5–8]; 0.1 cm^{-1} at $T = 11 \text{ K}$ in our case) and by a purely Lorentzian profile. Both bandwidth and band shape indicate the high quality of our samples.

No further infrared absorptions related to translations in the α -phase were observed in the spectral range up to 90 cm^{-1} , contrary to the spectroscopic results of [8] and [12]. Our result is in accordance with group theory: Jansen [13] pointed out that the optical phonons in α -O₂ do not couple to magnons. Consequently, the optical phonons will be neither IR nor Raman active. Therefore, we think that the authors of [8] and [12] monitored additional infrared absorptions due to bad crystal quality or due to impurities. These additional far-IR absorption

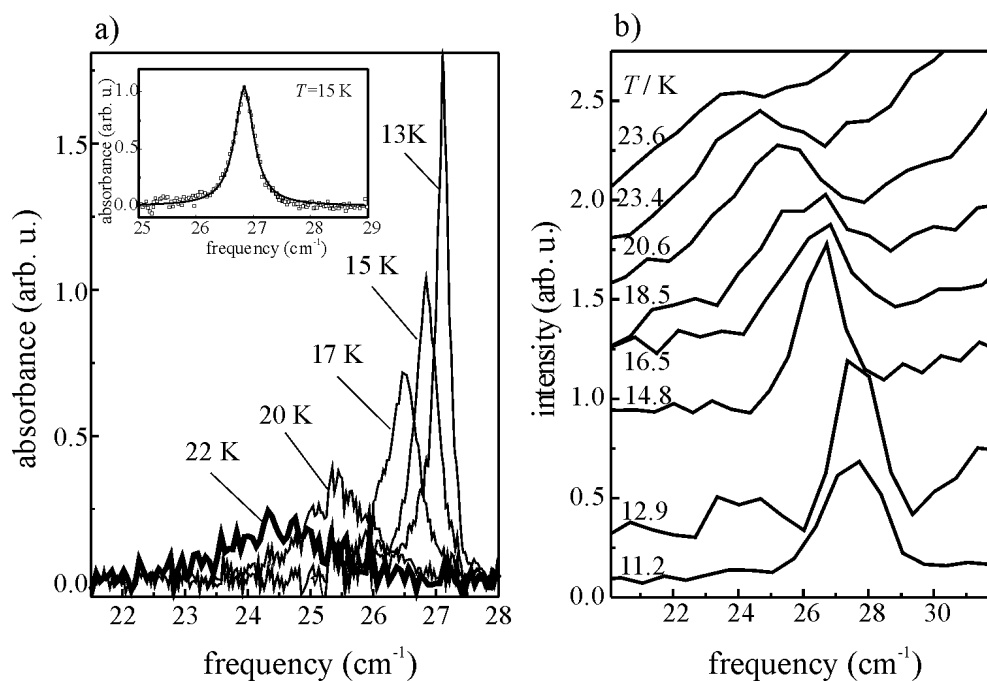


Figure 2. Temperature evolution of (a) infrared absorption and (b) Raman scattering spectra. Inset in (a) shows the magnon band at $T = 15$ K with a bandwidth of ~ 0.5 cm^{-1} and pure Lorentzian band shape (solid curve).

bands were only observed in their samples produced by gas condensation, whereas they were missing in samples grown from the liquid phase in closed optical cells (see [7] and present work).

3. Discussion

Raman and FTIR spectra were used to determine the band frequency, bandwidth and band intensity of the magnon mode at 27 cm^{-1} , which will now be discussed separately.

3.1. Magnon frequency and thermal expansion

The temperature dependence of the frequency of the band maximum is plotted in figure 3, in agreement with data of earlier studies. Our data are characterized by low scattering from the general trend (broken curve in figure 3 as a guide to the eye) and are reproducible during cooling/heating cycles. Both qualities confirm the thermodynamic stability of our samples and the high accuracy of our measurements.

A comparison of the relative frequency shift of different elementary excitations with temperature (12–23.8 K) shows the following trend: for the magnon mode, the relative shift $\Delta\omega/\omega$ is $\sim 14\%$, for librions $\sim 6\%$ [14], for the vibron $\sim 0\%$ [14], and for the exciton ($\Sigma \rightarrow \Sigma$) $\sim 0.5\%$ [15]. We think that all these temperature dependent frequency shifts are merely due to thermal expansion. However, each of the mode energies are defined by appropriate potential energies, upon which geometrical parameters have a different influence.

In the literature, Blocker *et al* [5] reported the magnon frequency between 2 and 20 K and induced in the same diagram $\omega(T)$ at 24 K from their earlier data determined by another method.

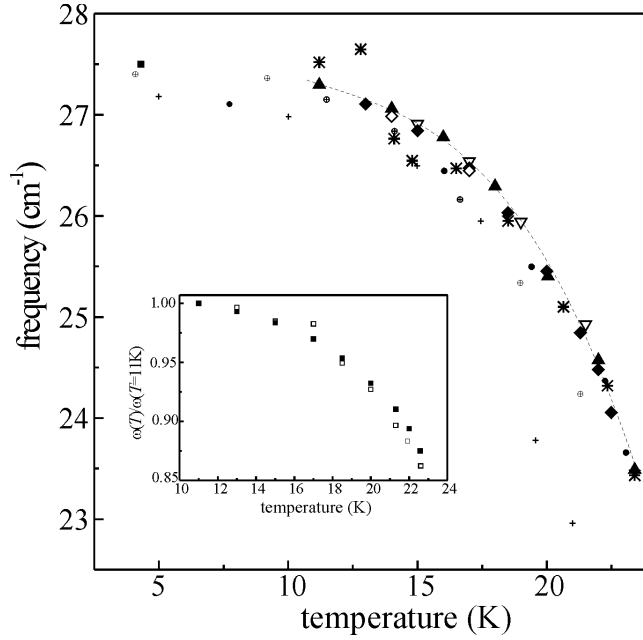


Figure 3. Temperature dependence of magnon frequency: ▲—warming, ▼—cooling of sample 1 (IR, thickness 6.7 mm); ◆—warming, ◇—cooling of sample 2 (IR, thickness 5 mm); *—our Raman data; all other symbols represent literature data: Raman: ■—[9]; IR: +—[5], ●—[7], ⊕—[8]. The error bars of our IR data are smaller than the symbols used. The broken curve is a guide to the eye. Inset shows the relative frequency shift due to temperature variation from our experiments (■) in comparison to theoretical values (□).

Their magnon shift is constant until 10 K and falls off smoothly with increasing temperature to about 85% of its low temperature value before falling rapidly to zero at the α - β transition. The authors related the experimental magnon shift to a decreasing effective anisotropic energy with increasing temperature. Meier *et al* [7] found a similar softening of magnon frequency to the data here, from $\sim 27.2 \text{ cm}^{-1}$ at 11 K to 23.5 cm^{-1} near the α - β phase transition. According to the neutron scattering data of Meier and Helmholtz [16], the spins remain strongly ordered up to the phase transition temperature. Therefore, this magnon frequency softening is not due to spin disorder in the sense that a certain Néel temperature is approached. Meier *et al* [7] suggested that a lowering of exchange integral J due to librational excitations implies a decrease of the magnon frequency. In our opinion, the energy of librational excitations in α -O₂ is about 60 K, i.e. much higher than sample temperatures (< 24 K). In addition the root-mean-square torsional amplitude does not change significantly: from $T = 10$ K ($\tilde{\Theta} = 9.1^\circ$) to 22 K ($\tilde{\Theta} = 9.6^\circ$) [10]. According to Meier [17] this does not affect the exchange integral ($\Delta J \leq 3\%$ in this temperature range).

To analyse the temperature dependence of the magnon frequency let us recall that, in the mean field approximation, the antiferromagnetic resonance (AFMR) mode frequency in α -O₂ at the centre of the Brillouin zone ($\vec{k} = 0$) is given by (e.g. equation (30) in [2]):

$$\omega(T) \sim \sqrt{4AJ(r(T))(\sigma(T))^2}, \quad (1)$$

where A is the anisotropy constant, σ the sublattice magnetization, and J is the exchange field: $J = n_1 J_1 - n_2 J_2 + n_3 J_3$, here n_1, n_2, n_3 are numbers of the nearest, next-nearest and next-next-nearest neighbours ($n_1 = 4, n_2 = 2, n_3 = 4$; see figure 1) and $J_1 > J_2$ or J_3 .

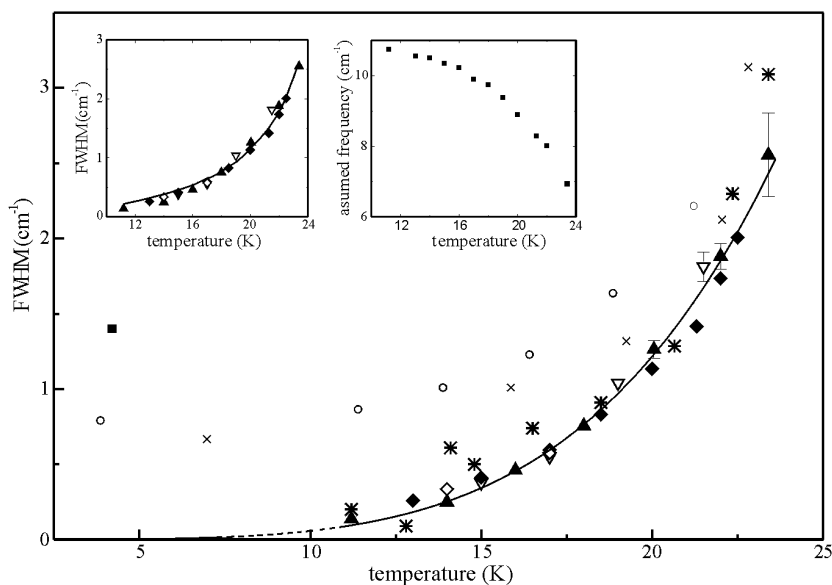


Figure 4. Temperature dependence of magnon bandwidth: \blacktriangle —warming, ∇ —cooling of sample 1 (IR, thickness 6.7 mm); \blacklozenge —warming, \diamond —cooling of sample 2 (IR, thickness 5 mm); $*$ —our Raman data; all other symbols represent literature data: Raman: \blacksquare —[9]; IR: \times —[7] \circ —[8]. Solid curve is the power fit function $\Gamma \sim T^{4.4}$ of our data, extrapolated towards $T = 0$ K (broken curve). Error bars of our IR data are smaller than the symbols. Inset top left shows the modelling of our experimental data by a pure magnon dephasing process; inset top right assumes a temperature dependence of the low energy magnon, averaged over the whole Brillouin zone.

Since $J(r)$, $r(T)$, and $\sigma(T)$ are known, we can try to model the temperature dependence of the magnon shift only by thermal expansion. We used an exponential dependence of exchange interaction on intermolecular distance by Sumarokov *et al* [18]: $J(r) = J_0 \exp[-\alpha(r - r_0)]$ (where $J_0 = 19.6 \text{ cm}^{-1}$, $\alpha = 3.765$, $r_0 = 3.187 \text{ \AA}$). Structural data—i.e. temperature dependent lattice constants from x-ray studies—were taken from Krupskii *et al* [1] and data on sublattice magnetization from neutron scattering data by Stephens and Majkrzak [19]. The inset in figure 3 shows good agreement of our IR data (closed symbols) with the theoretical predictions (open symbols).

Therefore, we conclude that the magnon softening with increasing temperature in $\alpha\text{-O}_2$ can be explained by a weakening of the exchange interaction due to thermal expansion of the crystal lattice and by a loss of magnetic order due to thermal fluctuations.

3.2. Magnon bandwidth and relaxation processes

Figure 4 shows the magnon bandwidth as a function of temperature. Our Raman scattering and IR-absorption data are one order of magnitude smaller than those in the literature and show lower scatter. The true bandwidth was determined from Raman spectra (figure 2(b)) by subtracting the apparatus function determined by narrow Ne atomic lines. As temperature increases, the band broadens and follows a power law $\Gamma(T) \sim aT^n$ with $n = 4.4$ (solid curve in figure 4). For further discussion we select only one set of data which we gained during cooling/heating cycles by far-IR absorption (figure 4, inset top left). Our values at

higher temperatures (22–24 K) fit nicely into the overall development. It is known that in some cases the high temperature values close to a phase transition are lie distinctly higher than that expected by extrapolation of low temperature values using a T or T^2 dependence. These higher values are usually interpreted as an indication of a phase transition, but it is our opinion that this is not the case.

As already mentioned, the IR-absorption bands were purely Lorentzian in band shape (figure 2(a), inset). Bandwidth measurements as a function of temperature provide a simple and convenient method to identify the most important mechanisms responsible for the relaxation processes of elementary excitations. Several channels are known: inelastic scattering (depopulation), elastic scattering (dephasing) and scattering on defects [20]. According to this theory, the most prominent relaxation channel is the three-particle-down decay process. In the classical limit, this process is modelled by a linear dependence of bandwidth on temperature and leads to non-zero bandwidth at $T = 0$ K. In our case, we found a strong non-linear dependence ($\Gamma \sim aT^n$ with $n > 4$) and the extrapolation provides zero bandwidth at $T = 0$ K (see figure 4, solid and broken curve). Higher order processes (magnon decay to several magnon/phonons) would deliver a T^3 or T^4 dependence but are relatively unlikely in our case. Our data show no inhomogeneous broadening. Therefore we conclude that magnon relaxation in α -O₂ is driven by pure dephasing processes:

$$\Gamma = B_{\text{deph}} n_i (n_i + 1) \quad (2)$$

where B_{deph} is a weighted coupling constant and n_i the occupation number of the quasi-particle involved in this scattering process [20]. One may think that this magnon at $\sim 27 \text{ cm}^{-1}$ is scattered by phonons. However, if we choose a possible bath phonon $\omega_q = 20 \text{ cm}^{-1}$ and assume quadratic dependence ($\Gamma(T) \sim T^2$), an unexpectedly high magnon/phonon coupling constant $B \sim 13 \text{ cm}^{-1}$ is calculated. Neither the exponent nor the bath phonon (sample temperature < 20 K) or the coupling constant appears to us realistic.

Therefore, we think that only magnon–magnon scattering processes lead to the strong broadening of the magnon peak with temperature. The necessary low frequency magnon has an excitation energy of only 8.5 K [6, 7]. Theory of spin relaxation processes in magnetic crystals [21] shows that for magnon–magnon scattering at low temperatures the lifetime of spin excitations is proportional to T^4 , which is very close to our fit function $\Gamma = aT^{4.4}$. Total agreement is not expected because this theory was developed for isotropic magnets whereas the magnon behaviour in α -O₂ is characterized by strong magnetic anisotropy.

The inset in figure 4 (top left) shows the fit function (solid curve) to our experimental values based on $\Gamma(T) = B_{\text{deph}} n_i (n_i + 1)$, where $B_{\text{deph}} = 0.48 \text{ cm}^{-1}$ and a temperature dependence for the low frequency magnon $\omega(T) = 10.7 - \Delta\omega(T)$ (see figure 4 inset top right). Since the temperature dependence of the low frequency magnon is not known ($\omega = 6.4 \text{ cm}^{-1}$ only at $T = 1.6$ K) [6, 7] we used a similar temperature dependence for this magnon $\Delta\omega(T)$ as that for the high frequency magnon (cf figure 3). For the low energetic magnon mode, this assumption delivers a relative shift with temperature $\Delta\omega/\omega$ of about 35%: two times larger than for the higher energetic magnon. Further, the measured frequency is much lower in comparison with the assumed one (6.4 cm^{-1} at 1.6 K; $\sim 10.7 \text{ cm}^{-1}$ at 11 K). In spite of these two obvious discrepancies one has to keep in mind that the spectroscopically measured values are at $\vec{k} = 0$, whereas the modelled function $\omega(T)$ (inset figure 4 top right) is an average over the whole Brillouin zone.

Furthermore, for a more qualitative analysis of magnon mode relaxation processes in solid α -O₂, precise frequency data for the low energy magnon mode are required as a function of temperature and a knowledge of the magnon density of states is necessary.

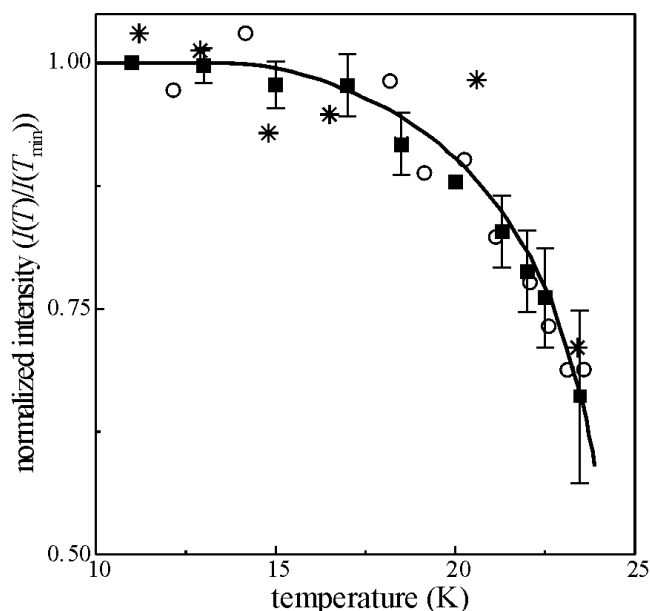


Figure 5. Integrated normalized intensity of magnon peak (■—IR, *—Raman) and intensity of the magnetic (101) diffraction peak by [19] (○). Solid curve shows the spin–spin correlation function $\langle S_i S_j \rangle$ in case of a 2D-Ising model (see [22]).

3.3. Band intensity and magnetic order parameter

Due to good crystal quality and modern spectroscopic technique we were able to analyse for the first time the temperature dependence of the intensity of the magnon band in solid α -O₂ up to the α - β phase transition temperature. Figure 5 shows the temperature dependence of integrated IR absorption intensity (squares) and of Raman scattering intensity (asterisks) normalized to the intensity at our lowest experimental temperature ($T = 11.2$ K). Our data are in good agreement with the intensity of the magnetic diffraction peak ($\bar{1}01$) from neutron scattering experiments in α -O₂ [19] (open circles in figure 5) which is proportional to the square of the sublattice magnetization, i.e. mainly the spin–spin correlation function. We conclude that intensity of the magnon band both in IR- and Raman-spectroscopy can be used as a measure of the long range magnetic order parameter in solid oxygen.

Following [19], the shape of the magnetization curve of α -oxygen is similar to that of the 2D Ising model [22] (solid curve in figure 5). Unfortunately, our far-IR and Raman data for α -O₂ only reached 22.5–23 K, not sufficiently close enough ($\Delta T/T_{\text{Néel}} \sim 10^{-2}$) to the magnetic transition to make any statements about the critical behaviour. However, an extrapolation of the spin–spin correlation function (2D Ising) $\langle S_i S_j \rangle \rightarrow 0$ implies that the magnetic order in α -O₂ would disappear at a critical temperature in the range ~ 26 –30 K. Therefore, we can conclude that the α - β phase transition ($T_{\alpha\beta} = 23.8$ K) is not a purely magnetic transition.

4. Conclusion

Using improved spectroscopic techniques and with improved crystal quality we performed Raman and IR spectroscopic studies on the higher energetic magnon mode and determined from spectra frequency, bandwidth and intensity as a function of temperature.

In contradiction to the literature we were able to model the magnon softening—described in mean field theory—by a weakening of the exchange integral due to thermal expansion and by a loss of magnetic order due to thermal fluctuations.

The bandwidth in our spectra was one order of magnitude smaller in comparison to earlier studies and was highly reproducible during heating/cooling cycles. To deduce possible mode relaxation mechanisms from $\Gamma(T)$ we discussed possible channels and selected magnon/magnon scattering process as the most probable one.

Band intensities as a function of temperature were determined for the first time and could be directly related to the long range magnetic order parameter.

Both spectral characteristics, band frequency and band intensity, especially in extrapolation towards a possible Néel temperature (i.e. loss of spin order) prove that the α – β phase transition at $T_{\alpha\beta} \sim 23.8$ K is not purely magnetic. Since we were able to draw conclusions about magnetic structures from fingerprints in optical spectra, we plan to extend this type of study to controversial results on low-pressure, low-temperature phases with possibly different magnetic and/or structural order: e.g. α' and δ' phases discovered by spectroscopic techniques [23] but not proved by structural studies [24].

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