

Local vibrational mode spectroscopy of dimer and other oxygen-related defects in irradiated and thermally annealed germanium

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Oxygen doped Ge has been irradiated with 2-MeV electrons and isochronally annealed between 80 and 440 °C. In total 64 local vibrational mode bands from oxygen-related defects have been observed in the infrared absorption spectrum, 23 of the bands belonging to thermal donors. Two bands at 621.6 and 669.1 cm^{-1} (5 K) are assigned to respectively VO^0 and VO^- , implying that the stretching mode of the A center in Ge is charge state dependent, similarly to the A center in Si. Bands at 525.5, 780.3, and 817.9 cm^{-1} (5 K) are assigned to three modes of the oxygen dimer. The wave-number positions are in excellent agreement with corresponding modes in Si and with published *ab initio* calculations for the dimer in Ge. The irradiation results in an enhanced formation of dimers and thermal donors. The annealing data of the dimer and the early thermal donor bands, support the model of the dimer being the first step in the oxygen agglomeration sequence of thermal donor formation. Preliminary results are also obtained concerning Ge-isotope splitting of some of the absorption bands.

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I. INTRODUCTION

During the last few years the interest for early oxygen agglomeration centers in silicon has focused on the oxygen dimer.¹⁻⁶ Being the simplest oxygen cluster, the dimer is considered as the precursor of the oxygen thermal donors (TD's) in silicon, which have been extensively investigated. It is thought that the dimer also represents the fast diffusing species needed to explain the TD formation kinetics. As an electrically inactive cluster, the dimer became experimentally accessible when local vibrational mode (LVM) absorption attributed to the defect was discovered.¹⁻⁴ Owing to enhanced dimer formation during irradiation also weaker LVM bands were observed, resulting in four well-established dimer bands.^{2,5} Several papers have been published on the calculation of stable dimer configurations.⁵⁻⁸ Today the staggered dimer, consisting of two Si-O-Si interstitial oxygen units sharing one Si, appears as the preferential model (the same model would also apply to the oxygen dimer in germanium).⁶ The latter calculations predict five LVM frequencies in Si and four in Ge.

In Ge, where oxygen related defects have been less extensively studied than in Si, a striking similarity with Si has been found for all oxygen related defects identified so far, e.g., interstitial oxygen (O_i), TD's (including bistability), VO and precipitates (see Ref. 9 for an overview). Recently LVM absorption from the TD's has been observed in Ge as well.¹⁰⁻¹² On that occasion we have drawn the attention to two weak LVM bands at 780 and 818 cm^{-1} , which always appear together with similar amplitude in oxygen doped Ge, submitted to a dispersion treatment followed by a quench to room temperature.¹⁰ In similar samples also small concentrations of TD1 and TD2 are traced in the far-infrared electronic absorption spectrum. These observations point to the formation of relatively small oxygen clusters during rapid cooling from high temperature and it was thought that the peaks at 780 and 818 cm^{-1} were good candidates for the dimer. This

assignment seemed substantiated by the almost perfect scaling from the position of the 1012 and 1060 cm^{-1} dimer bands in Si.¹⁰ A tentative attribution of the 780 cm^{-1} absorption, appearing in irradiated oxygen doped Ge, to centers with two oxygens was also made by Whan¹³ and Klechko *et al.*¹⁴

In this paper we report on the investigation of the LVM absorption spectrum of irradiated and isochronally annealed oxygen doped Ge. The primary purpose of the experiments was to increase the assumed LVM absorption from the dimers and to follow the evolution of the absorption into the temperature range of early TD formation. An increased dimer concentration would also favor identification of other (possibly weaker) dimer bands; LVM bands of the dimer in Ge are predicted by Coutinho *et al.*⁶ at 466, 517, 749, and 784 cm^{-1} . In addition we wanted to re-investigate the old infrared results of Whan¹³ and Becker and Corelli¹⁵ using better spectral resolution and extending the wave-number range below 600 cm^{-1} .

II. EXPERIMENTAL DETAILS

The oxygen doped Ge has before irradiation been submitted to a 5-min dispersion at 900 °C followed by a quench to room temperature (RT). The interstitial oxygen concentration measured after the quench was $[\text{O}_i] = 2.9 \times 10^{17} \text{ cm}^{-3}$ as determined from the amplitude of the 856- cm^{-1} band (RT), using the conversion factor $[\text{O}_i] = \alpha_{\text{max}} \times (5 \times 10^{16}) \text{ cm}^{-2}$ with α_{max} the amplitude in cm^{-1} .¹⁶ After the quench the Ge was *n*-type with resistivity $\rho = 3.3 \text{ } \Omega \text{ cm}$. The Ge was irradiated with 2-MeV electrons to a fluence of $5 \times 10^{17} \text{ cm}^{-2}$, the temperature during irradiation was kept close to RT. Isochronal anneals of 20 min in the temperature range 80–440 °C (increments of 40 °C) were performed in flowing argon. The infrared spectra were recorded with a Bruker IFS66v FT-IR instrument at RT, 80 and 6 K using a nominal resolution of 1 and 0.25 cm^{-1} . The electron concentration *n* was monitored by resistivity and Hall-effect measurements at RT.

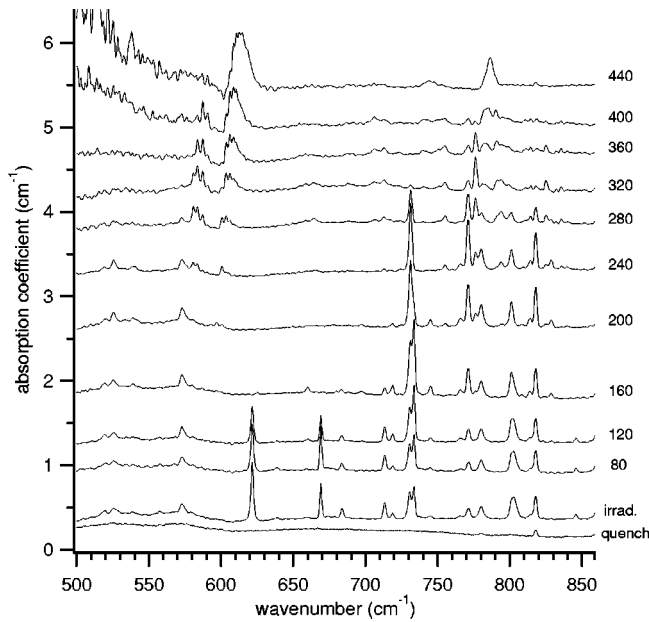


FIG. 1. Infrared absorption spectra of oxygen doped Ge after different successive treatments. Numbers to the right of the spectra indicate the annealing temperature in °C. The spectra are vertically shifted, lattice absorption has been subtracted. Measurements at 5 K and resolution 1.0 cm^{-1} .

III. EXPERIMENTAL RESULTS AND DISCUSSION

The evolution of the absorption spectrum due to the different treatments (quenched, irradiated, and annealed) is summarized in Fig. 1. The wave-number range corresponds with the LVM range below the O_i peak at 862.3 cm^{-1} . In the quenched sample two small peaks are seen at 780.3 and 817.9 cm^{-1} in agreement with our earlier experiments.¹⁰ The number of peaks appearing after irradiation and during annealing is overwhelming: on the whole we found up to 64 peaks; the positions are given in Table I except those of LVM's belonging to thermal donor defects which are summarized separately in Table II. Some of the bands displayed in Fig. 1 turn out to be composite when 0.25-cm^{-1} resolution is used; Table I gives the position of the resolved components. After the irradiation the O_i peak has decreased to 89% of its original amplitude and it decreases further in the later annealing stages until 74% is left after the 440°C anneal. A Ge reference sample without oxygen doping showed no LVM peaks after irradiation and annealing. The observations above suggest that most (if not all) of the LVM peaks observed are due to defects containing at least one oxygen atom. In the later annealing stages, the absorption evolves into the LVM thermal donor bands designated previously as “600,” “740,” and “780,” which referred to their average position.¹⁰ An interesting observation is that the TD bands start to develop at lower temperatures in comparison with unirradiated samples. Note that the faint peak at 817.9 cm^{-1} in the 440°C spectrum is due to $^{18}\text{O}_i$ and should not be confused with the peak appearing at the same position after quench or irradiation.

TABLE I. Wave-number position (cm^{-1}) at 5, 80, and 300 K of peaks observed in oxygen-doped Ge either after quench, irradiation or isochronal annealing. Positions of thermal donor bands are reported in Table II. Labels in the column “Defect” refer to the annealing behavior of the peaks (Figs. 2–5).

Defect	5 K	80 K	300 K	Defect	5 K	80 K	300 K
	519.6	518.9		V	771.2	770.2	767.0
IV	525.5	525.0	518.8	VI	776.4	776.1	772.8
	538.9	538.9		IV	780.3	780.1	775.9
IV	572.9	572.4	568.1	IV	801.2	800.7	796.8
I	621.6	620.0	611.5	II	802.9	802.4	796.8
	625.2	625.2			805.4	804.9	796.8
	638.6	636.7		III	808.3	807.8	
	659.9	658.9			813.2	813.7	806.5
	664.0	664.0		V	814.5	813.7	806.5
I	669.1	667.2	657.0	IV	817.9	817.5	813.7
II	683.8	683.8	679.3	VI	825.4	824.5	817.7
	697.0	697.0		V	828.8	825.2	
II	713.3	712.9	709.1	VII	832.4	831.9	
III	718.8	717.9	715.4	VII	835.7	834.8	
	725.0	725.0			839.9	839.5	
III	730.7	730.2	(726.7)	I	846.0	845.5	
V	731.4	730.4	(723.9)		855.6		
III	733.6			O_i	862.3	861.3	855.6
III	745.2	744.0	(735.4)	VII	877.3	876.2	
VII	755.1	754.7	751.8	VII	905.4		
IV	766.0	765.6	763.7				

A. Comparison with earlier studies

Our observations are in general agreement with early similar studies by Whan¹³ and Becker and Corelli.¹⁵ A major difference, however, is that the present spectra are recorded with better resolution allowing to resolve closely spaced peaks. According to their different annealing these components belong to different defects, probably explaining irregularities in the corresponding annealing curves of Refs. 13 and 15. This is, e.g., the case for the 780- and 819-cm^{-1} bands, which in our spectra are resolved into components at 776.1 and 780.1 cm^{-1} and 813.7 , 817.5 , and 824.5 cm^{-1} , respectively, all positions corresponding with 80-K measurement temperature. Another difference is the detailed observation of

TABLE II. Wave-number position (cm^{-1}) at 5 K and assignment of resolved vibrational absorption bands belonging to TD's in Ge. DD: shallow double donor configuration. N: low-energy configuration.

	TD1	TD2	TD3	TD4	TD5	TD6	TD7	TD8	TD9
DD	593.0	597.0	600.6	603.5	606.1	608.6	610.6	612.5	614.5
		774.5	777.6	780.4	782.7	785.1			
N	581.0	583.6	587.4	590.6					
		712.9	706.0	706.0					
	794.1	791.9	790.5	790.5					

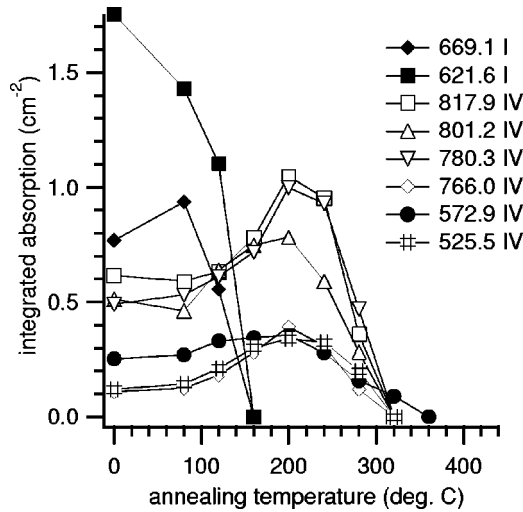


FIG. 2. Isochronal annealing curves of LVM absorption from radiation-induced oxygen related defects in Ge, groups I and IV. The modes are indicated by their wave-number position at 5 K followed by the group number.

sequentially appearing LVM's of TD species in three spectral ranges which in the spectra of the early papers are unobserved or present as broad features. More recently Klechko *et al.*¹⁴ also published infrared spectra measured at RT of electron-irradiated oxygen doped Ge. The as-irradiated spectrum corresponds with our RT spectrum after the 160–200 °C anneals, in agreement with the higher irradiation temperature in Ref. 14 and the absence of the 620-cm⁻¹ band due to the A center (VO), which disappears at rather low annealing temperature. The peak observed in our spectra at 667.2 cm⁻¹ (80 K) anneals out together with the 620-cm⁻¹ peak (80 K) from the A center.

B. Annealing behavior of various LVM bands

The annealing behavior of the most intense peaks has been summarized in Figs. 2–5. The data in the graphs at 0 °C correspond with as-irradiated material. On the whole

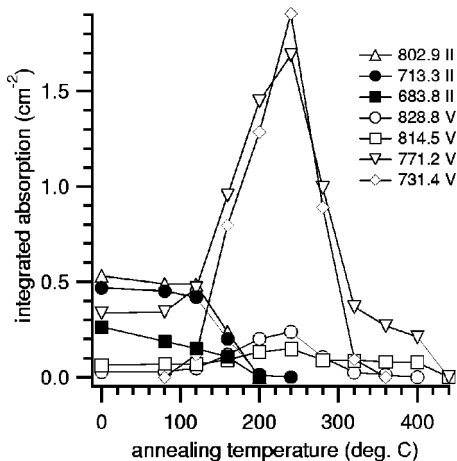


FIG. 3. Isochronal annealing curves of LVM absorption from radiation-induced oxygen related defects in Ge, groups II and V.

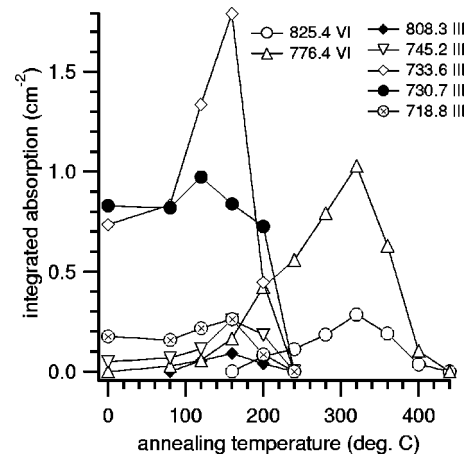


FIG. 4. Isochronal annealing curves of LVM absorption from radiation-induced oxygen related defects in Ge, groups III and VI.

we cataloged 28 of the 64 bands into seven groups, each group displaying a distinct annealing behavior and temperature range of appearing; the TD bands are cataloged separately. We labeled the groups as I,II, . . . ,VII, group I being the first to anneal out, group VII the last. The data plotted are the integrated absorption of Gaussian curves fitted to the LVM bands. Especially in the case of composite peaks this proved to be the best method. The sequential annealing of the groups is indicative of the formation of oxygen-containing defects becoming more complex with increasing annealing temperature. A difference with low temperature irradiation¹³ is that the RT irradiation already results in the formation of several species (groups I–IV); they may nevertheless be assumed to represent relatively simple complexes. In this paper we will concentrate on groups I and IV and on the TD bands.

1. Group I: A center (VO)

The most prominent LVM band in the as-irradiated Ge is the 621.6-cm⁻¹ peak, attributed before by Whan¹³ to the A center or vacancy-oxygen complex. This attribution followed

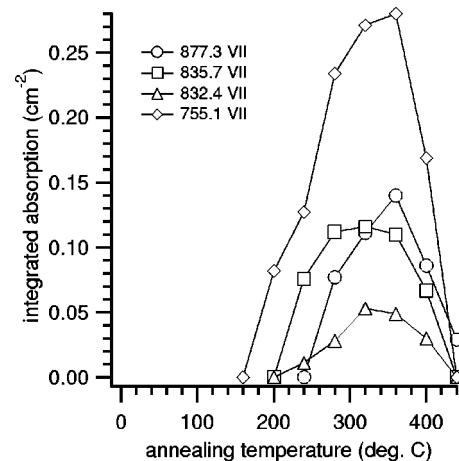


FIG. 5. Isochronal annealing curves of LVM absorption from radiation-induced oxygen related defects in Ge, group VII.

TABLE III. Comparison of experimental positions (cm^{-1}) of LVM bands due to oxygen defects in Ge and Si (low-temperature values). Values in the last column give the ratio of Si to Ge positions. Positions within brackets indicate bands for which assignment to the dimer is doubtful.

	Ge	Si	Factor
O_i	862.5	1136	1.32
	817.9 (801.2)	1059.8 ^a	1.30
O_2 (dimer)	780.3 766.0 (572.9)	1012.4 ^a	1.30
	525.5	690.1 ^a 555.8 ^a	1.31
VO^0	621.6	835.8 ^b	1.34
VO^-	669.1	885.3 ^b	1.32

^aReference 2.

^bReference 17.

from the comparison of the annealing behavior with that of the EPR signal of the negatively charged A center observed by Baldwin.¹⁸ A second prominent peak with very similar behavior (Figs. 1 and 2) which is reported here is observed at 669.1 cm^{-1} . A similar situation exists in silicon where two LVM bands from the A center at 836 and 885 cm^{-1} may be observed simultaneously depending on the resistivity, respectively belonging to the neutral and negative charge state of the VO acceptor.¹⁷ From our electrical data we calculate that the Fermi level is situated at about $E_C - 0.25 \text{ eV}$ during the annealing stages at which the two peaks are observed. If we compare this with the position of the VO -acceptor level in Ge (Refs. 19–21) at $E_C - (0.23-0.27) \text{ eV}$, it seems reasonable to conclude that in our case both charge states of the VO acceptor are simultaneously present. The reason that the 669.1-cm^{-1} band was not observed earlier may then be due to differences in sample doping or irradiation conditions.

The similarity with the Si case is further substantiated if the relative positions of the peaks in both semiconductors are compared with that of the corresponding LVM bands of O_i . As can be seen in Table III, the scaling factor applying to the ν_3 mode of O_i equals 1.32, the corresponding factors for the bands assigned to the two charges states of VO being in very close agreement with this value (a similar remark regarding the 621-cm^{-1} band was also made by Coutinho *et al.*⁶). It seems reasonable from the above to assign the 621.6- and 669.1-cm^{-1} peaks to the antisymmetric stretching LVM of the VO^0 and VO^- center, respectively. The results indicate that in Ge a similar upward shift of the mode occurs between the neutral and negative charge state as it is the case in Si, contrary to theoretical calculations predicting almost no shift for Ge⁶ (it should be noted that the authors of Ref. 6 indicate that the calculated shift for Ge is probably underestimated). We will return to the 669.1-cm^{-1} band when discussing fine structure observed at better resolution.

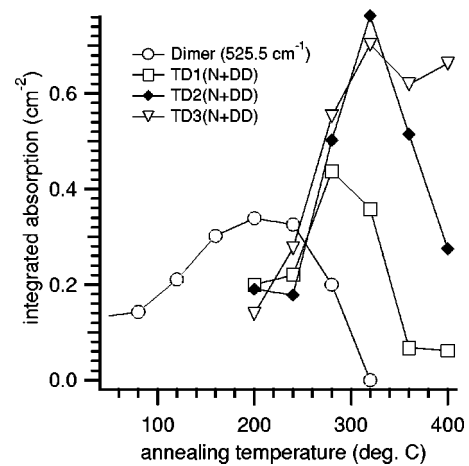


FIG. 6. Sequential isochronal annealing of dimer and early thermal donor absorption.

2. Group IV: Oxygen dimer (O_2)

As pointed out in the Introduction, we have reasons to believe that the bands at 780.3 and 817.9 cm^{-1} detected in samples submitted to the oxygen dispersion quench may be due to the oxygen dimer. As seen in Figs. 1 and 2, the peaks become stronger during electron irradiation and their amplitudes grow further upon annealing towards $200 \text{ }^\circ\text{C}$, opening opportunities to detect possible other dimer-related bands. Initially the members of group IV were put together only on the bases of their similar annealing behavior (e.g., they are all present as irradiated, the maximum absorption occurs at about $200 \text{ }^\circ\text{C}$ and all members, except one, anneal out at $320 \text{ }^\circ\text{C}$). It is clear that the annealing curves of the 780.3- and 817.9-cm^{-1} bands are identical. From the four other peaks put in group IV, those at 525.5 and 766.0 cm^{-1} with about three times smaller amplitudes display annealing curves very similar to those of the two major peaks. The correlation with the two remaining bands seems, however, less obvious regarding the somewhat different curve or annealing temperature, so that we end up with four preferential candidates for dimer-related LVM bands. We have scaled the positions with those of established dimer bands in silicon in the same way as we did in the previous section for VO and the results are also included in Table III. It turns out that three of the four bands have a counterpart in Si with a scaling factor very close to the factor 1.32 of O_i , which may be considered a strong additional evidence for assignment to the oxygen dimer in Ge. No counterpart for the 766-cm^{-1} band seems to be present in Si, throwing doubt on its correlation with the dimer. A counterpart for the 555.8-cm^{-1} band in Si is expected to occur around 420 cm^{-1} in Ge, which is in a more noisy range due to lattice absorption, preventing the clear observation of faint peaks. In Fig. 6 the annealing curve for the dimer is plotted together with those for the first three TD's. Note that for the case of comparing integrated absorption (i.e., concentration) we have plotted the results of LVM's in a similar frequency range, i.e., the 525.5 cm^{-1} band for the dimer and the LVM lines for TD1–TD3 in the “600” range (see also the next section on TD bands). The sequential appearance of the dimer with that of TD1–TD3 is

obvious and supports the idea of the dimer being a precursor of the TD's. It also seems to support our earlier attribution of TD2 to a complex containing four oxygens¹⁰ implying that TD1, fitting between the dimer and TD2, would then probably contain three oxygens (it must be noted, however, that the validity of the latter conclusion depends on the accuracy of the O_i calibration factor used^{10,16}). When the experimental data are compared with the calculated values for the staggered dimer in Ge in Ref. 6, an excellent agreement is found for the upper three dimer modes (i.e., 525.5, 780.3, and 817.9 cm^{-1} vs 517, 749, and 784 cm^{-1}), the calculations predicting wave-number positions only 2–4 % smaller than the experimental values. This underestimation is almost identical as for the upper three dimer modes in Si.⁶ Finally our experiments confirm that unlike the situation in Si, the dimer LVM bands in Ge display a normal temperature shift.

3. Thermal donor bands

In the irradiated Ge, LVM absorption from the TD's becomes visible in the range around 600 cm^{-1} already after 20 min annealing at 200–240 °C. Even if a systematic comparison with the 350 °C isothermal anneal of our previous study of unirradiated Ge cannot be made,¹⁰ the observations indicate an enhanced TD formation after irradiation. A remarkable observation is that in Fig. 1 the TD modes from both bistable forms of TD1–TD4 appear simultaneously in the spectra, i.e., at 581.0–590.6 cm^{-1} for the neutral state (N) and at 593.0–603.5 cm^{-1} for the double donor state (DD). The reason why part of the early TD's appear as N and part as DD is not clear (since bistability was not the subject of the present study no special measures were undertaken with regard to the illumination conditions during cooling). For the annealing curves of Fig. 6, the integrated absorption from both forms was added. In total, LVM contributions from TD1–TD4(N) and TD1–TD9(DD) are observed in the “600” range. In Fig. 1, also the “740” and “780” TD bands are clearly seen in the 440 °C spectrum where most LVM bands of other centers have annealed out. Compared to the previous experiments, the present study allowed to better observe the LVM absorption from the earliest TD's including TD1. Apart from the confirmation of the TD1 positions in the “600” band reported in Ref. 11, this also allowed the observation of a band at 794.1 cm^{-1} which we attribute to TD1(N). The position adds nicely to the sequence of the TD2–TD4(N) modes around 790 cm^{-1} and the corresponding annealing curve is similar to that of the 581.0- cm^{-1} mode of TD1(N). The LVM positions of TD species which so far have been observed in ¹⁶O doped Ge are summarized in Table III.

C. Resolution of fine structure from Ge isotopes

At high spectral resolution the 862.3- cm^{-1} band due to the ν_3 antisymmetric stretching mode of O_i in Ge is resolved into a number of components^{22–24} corresponding with different ${}^n\text{Ge-O-}{}^m\text{Ge}$ combinations, n or m indicating the five stable isotopes ⁷⁰Ge, ⁷²Ge, ⁷³Ge, ⁷⁴Ge, or ⁷⁶Ge. At 0.25- cm^{-1} resolution (the best resolution used in the present study) this isotope splitting is reduced to only six major com-

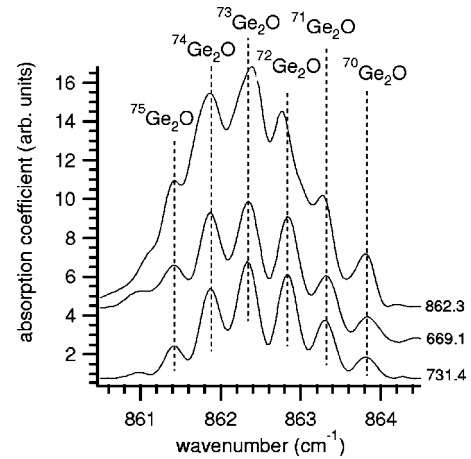


FIG. 7. Partially resolved splitting due to Ge isotopes at 0.25- cm^{-1} resolution. The wave-number scale corresponds with the top spectrum of O_i . The two lower spectra have been scaled to fit with the components of the upper spectrum.

ponents as can be seen in Fig. 7. Note that a label such as ⁷³Ge indicates a remnant from major components due to combinations with an average atomic mass of 73 such as ⁷²Ge-O-⁷⁴Ge or ⁷³Ge-O-⁷³Ge. Surprisingly the LVM peaks at 669.1 and 731.4 cm^{-1} exhibit a similar splitting as the O_i band. This may indicate that similarly to the O_i defect, the bands are due to defects containing one oxygen bounded to two equivalent Ge neighbors. This seems an interesting conclusion with respect to our attribution of the 669.1 cm^{-1} band to VO^- , even if the VO^0 band apparently does not display a similar fine structure at the resolution used. At present we have no candidate for the 731.4- cm^{-1} band. In any case these findings have to be confirmed by high-resolution experiments before definite conclusions may be drawn.

IV. CONCLUSION

The re-investigation of the infrared absorption spectrum of irradiation centers in oxygen doped Ge using the possibilities of fourier-transform spectroscopy, has resulted in interesting data. Using better resolution than before, several LVM bands could be separated into components belonging to different defect centers and additional weak bands were observed. In addition to the TD bands for which one mode could be added to the ones already known, this resulted in the observation of 41 modes due to defects probably containing at least one oxygen atom. According to the isochronal annealing data these should correspond to at least seven different centers with different complexity. Two modes annealing out at 160–200 °C have been attributed to respectively the neutral and negative charge state of the VO center: the mode at 621.6 cm^{-1} already known before and the one at 669.1 cm^{-1} reported here.

A major result concerns the observation of modes at 525.5, 780.9, and 817.9 cm^{-1} which are attributed here to the oxygen dimer. The positions are in excellent agreement with observed counterparts in Si and with *ab initio* calculations. The annealing curve of the dimer bands precedes the

sequence of annealing curves of the early thermal donor centers TD1–TD3, which seems to confirm the idea as the dimer being a precursor of TD formation. Whether the oxygen dimer in Ge would also represent a faster diffusing species than oxygen interstitials is, however, less obvious regarding the high diffusion coefficient of the latter in Ge.²⁵ In any case the present results open interesting possibilities for a kinetic study of early TD formation in Ge.

At present no firm conclusions are drawn regarding the identification of other LVM bands. The bands belonging to groups II and III probably also belong to relatively simple species, such as VO₂ or V₂O. Further conclusions are awaited until ¹⁸O doped Ge has been measured in similar experiments as presented here for ¹⁶O doped material. The

observations of corresponding isotopic shifts are even more important for the confirmation of the assignments made here concerning the VO and the dimer modes. Other experiments to be made are high-resolution measurements in order to investigate possible splittings from Ge isotopes which offer exciting possibilities as well.

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