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Refractive index dispersion and Raman scattering spectra of a germanium single crystal enriched with ^{70}Ge isotope

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The spectral dependence of the refractive index (RI) of a germanium single crystal enriched in the isotope ^{70}Ge ($>$ 99.92 at.%) in the spectral range $5500-500\,\text{cm}^{-1}$ ($1.8-20\,\mu$m) was measured. The data obtained are compared with the results for single crystals of other germanium isotopes (^{72}Ge, ^{73}Ge, ^{74}Ge, ^{76}Ge). In the entire studied range, the refractive index values of ^{70}Ge are higher than for heavier isotopes. This confirms the previously identified trend towards an increase in the RI with a decrease in the atomic mass of germanium isotopes. The Raman spectra of first- and second-order light scattering of a ^{70}Ge single crystal at a temperature of 293 K.

Keywords: elementary semiconductors, isotopic effects, interference refractometry, refractive index, Raman spectroscopy.

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Introduction

Thanks to the wide optical transmission range $1.8-23\,\mu$m, ow optical losses, and high refractive index (RI) (~ 4) monocrystalline germanium finds application in infrared optics as optical elements for spectroscopy, such as filters, windows, mirrors, components of night vision devices, and others. Doped germanium crystals are used for manufacturing infrared radiation detectors.

Waveguides and sensors made of germanium are employed to address challenges in modern infrared photonics [1–3]. The refractive index is one of the fundamental characteristics of mid-infrared materials, determining their optical properties and applications. The dispersion of the refractive index depends on the matrix composition, atomic structure, temperature, and other parameters. Various techniques are used to measure the spectral dependence of germanium's refractive index with natural isotopic composition [4–9]. One of the most promising methods is interference refractometry [10–13], which has high precision and is applicable over a wide wavelength range, allowing its use in studying isotopic effects in high-purity crystalline semiconductors. This was demonstrated in our previous works measuring refractive indices of monocrystals of stable isotopes of silicon and germanium [14–16].

The development of the hydride method facilitated the production of individual germanium isotopes with high chemical and isotopic purity [17]. Studies on monocrystals of germanium isotopes showed that changes in isotopic composition significantly affect thermal conductivity, thermoelectric power, spin interactions, phonon spectra of infrared transmission, and Raman scattering spectra [18–24]. In [16] data were obtained on the spectral dependence of the refractive index of monocrystals of germanium isotopes ^{72}Ge,^{73}Ge,^{74}Ge and ^{76}Ge. It was found that the refractive index decreases with increasing atomic mass of the isotope. Recently, high-purity monocrystalline samples of the lightest isotope ^{70}Ge were obtained by the hydride method [25]. Studying its properties and comparing them with previously obtained results for other germanium isotopes is of interest.

The aim of this work was to study the spectral dependence of the refractive index and Raman scattering spectra of monocrystals of the ^{70}Ge isotope.

Experimental Section

Germanium enriched in the ^{70}Ge isotope was obtained by the hydride method. The monocrystal was grown by the Czochralski method using the technique described in [17]. Samples of isotopically enriched germanium had p-type conductivity. The specific electrical resistance at room temperature was $\sim 50\,\Omega\cdot$cm. The isotopic composition of the monocrystal is presented in Table 1. The content of chemical impurities did not exceed $10^{-5}-10^{-6}$ mass.%

For spectroscopic measurements, three polished plane-parallel plates were prepared from the monocrystal ^{70}Ge. Surface quality was assessed by observing interference fringes in reflection between the samples and a reference sample in optical contact. Samples for refractive index measurements had surface flatness better than 1/10 of a

Table 1. Isotopic composition of the monocrystal ^{70}Ge

Atomic Mass of Isotope	C, at.%	Δ, at.%
70	99.92597	0.00040
72	0.00669	0.00009
73	0.00007	0.00002
74	0.0672	0.0040
76	< 0.00001	–

Note: C — isotope concentration, Δ — isotope concentration measurement uncertainty.

Newton fringe ($\lambda_{1/4} = 544\,\text{nm}$), parallelism better than $1''$ and surface quality grade 60/40. Sample thicknesses were 0.85074, 1.25386, and 1.54781 mm. Accurate thickness measurement is crucial, as it contributes primarily to the uncertainty in refractive index determination by interference refractometry.

To ensure refractive index determination accuracy up to $1 \cdot 10^{-4}$, sample thicknesses were measured with a GD-30 holographic gauge with an accuracy of $0.05\,\mu$m.

Interference spectra were recorded using a vacuum-operated infrared Fourier spectrometer IFS-125HR, equipped with highly sensitive cooled MCT and InSb detectors, covering wavenumber range $8000 - 400\,\text{cm}^{-1}$ with resolution 0.1 at $20.0\,^\circ$C (293 K). Sample temperature was controlled within 0.1 K during transmission and thickness measurements the sample temperature was maintained at $20.1 \pm 0.05^\circ$C.

To minimize measurement errors of transmission spectra, the angular aperture of the beam was minimized in the spectrometer's sample compartment to ensure nearly parallel rays incident on the sample. The measurement procedure followed interference refractometry techniques described in [12,13], which used an identically configured IFS-113v spectrometer (Bruker). This methodology includes investigation of the modulation dependence of transmission on the beam's angular aperture for thin plate samples. It was shown in [13] that within $\pm 1 \cdot 10^{-4}$ the calculated RIs for the spectra of samples with a thickness of ~ 1 mm measured at different apertures, coincide, and the relative error $\Delta n/n$ of the RI calculation at an aperture with an angle of 2° 6° 10° is $-1.37 \cdot 10^{-5}$ $-1.12 \cdot 10^{-4}$ $-3.16 \cdot 10^{-4}$ respectively.

Spectra in this study were recorded using a 1.5 mm aperture diaphragm and an additional diaphragm before the sample. Additional beam diaphragming made it possible to reduce the angular aperture in the waist region, where the sample is placed, to $\sim 1^\circ$. The aperture size was chosen balancing signal-to-noise, since reducing aperture further reduces irradiance on the sample, negatively affecting spectrum quality. With aperture angles below ~ 0.5 and 1° and sample thicknesses of 1 mm, contribution of beam divergence to effective thickness did not exceed thickness measurement errors of ~ 0.05 and $0.15\,\mu$m respectively, ensuring necessary refractive index accuracy.

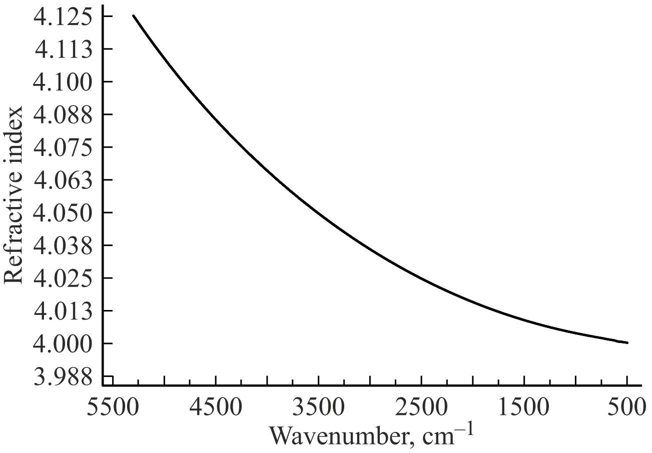

Figure 1. Spectral dependence of the refractive index of monocrystalline ^{70}Ge, averaged over three samples $T = 293$ K.

Raman spectra were obtained using the INTEGRA Spectra system (NT-MDT, Russia) at room temperature in backscattering geometry. Excitation was by a semiconductor laser with 473 nm wavelength. Raman spectrum detection used a cooled ANDOR CCD camera in the range $150 - 1000\,\text{cm}^{-1}$ with $0.7\,\text{cm}^{-1}$ step.

Results and discussion

Dispersion of the Refractive Index

To study the spectral dependence of the refractive index of germanium samples enriched with the 70 isotope, infrared transmission spectra were recorded by interference refractometry. To calculate the spectral dependence of the refractive index using interference refractometry, the technique described in [11–16] was used, in which from frequencies of interference peaks ν_m n transmission spectra of plane-parallel germanium plates, the order of interference maximum m, is determined, then from expression (1), the refractive index $n(\nu_m)$ dependence is calculated over the entire measured spectral range of the interference spectrum:

$$2hn(\nu_m)\nu_m = m, \tag{1}$$

where m is interference order („maximum number"), h -is plate thickness in centimeters cm, $n(\nu_m)$ is refractive index at corresponding frequency, ν_m — interference maximum frequency in cm^{-1}.

Over the entire spectral range of interference spectra, positions of maxima of interference were determined at 2200 peaks with accuracy up to $0.001\,\text{cm}^{-1}$, each peak containing at least 20 experimental points. Averaging was performed over three samples.

For the first time, data on the dispersion of the refractive index of monocrystalline ^{70}Ge at 293 K in a wide range of wavenumbers, presented in Fig. 1. The spectral range for determining the dispersion of the RI and calculating

Table 2. Values of the approximation coefficients of the dispersion dependence of the refractive index of the ^{70}Ge sample in the spectral range 1.94–20 μm

Coefficient	Value
A0	3.992280
B1	$2.873970 \cdot 10^{-5}$
B2	$-4.185230 \cdot 10^{-8}$
B3	$4.086530 \cdot 10^{-11}$
B4	$-2.246000 \cdot 10^{-14}$
B5	$7.867070 \cdot 10^{-18}$
B6	$-1.755640 \cdot 10^{-21}$
B7	$2.417000 \cdot 10^{-25}$
B8	$-1.869460 \cdot 10^{-29}$
B9	$6.223960 \cdot 10^{-34}$

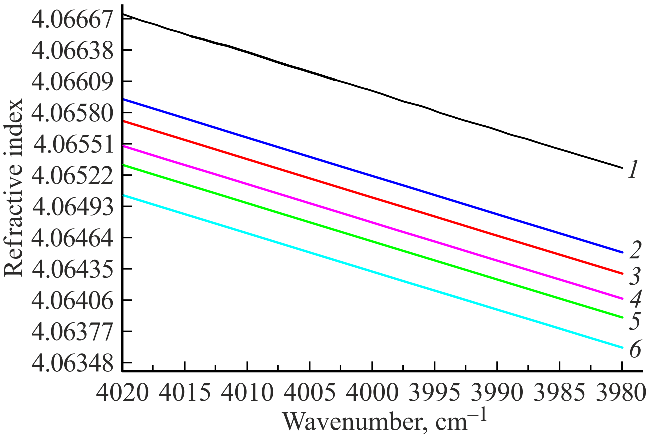

Figure 2. Dispersion dependence of the refractive index of high-purity isotopes of monocrystalline germanium in a narrow range of wavenumbers. Comparison of ^{70}Ge data with previously obtained data in [15]: *1* — ^{76}Ge, *2* — ^{74}Ge, *3* — ^{73}Ge, *4* — ^{nat}Ge, *5* — ^{72}Ge, *6* — ^{70}Ge.

the approximating dependence was $5300 - 450\,\text{cm}^{-1}$ or $1.89 - 22.22\,\mu$m.

The experimental data for germanium enriched with the ^{70}Ge isotope shown in Figure 1 were approximated using 9th-degree Cauchy polynomial regression [26] (2)

$$n(\nu) = A_0 + \Sigma_{(i=1)}^{9} B^i \nu, \tag{2}$$

where $i = 1, 2 \cdots 9$; ν is a wavenumber, $\nu = 10^4/\lambda$ (cm^{-1}).

The approximation coefficients for the polynomial that best fits the experimental results in the wavelength range $1.94 - 20\,\mu$m are listed in Table 2. The maximum value of the root-mean-square deviation R for all approximation ranges was $8.5 \cdot 10^{-6}$.

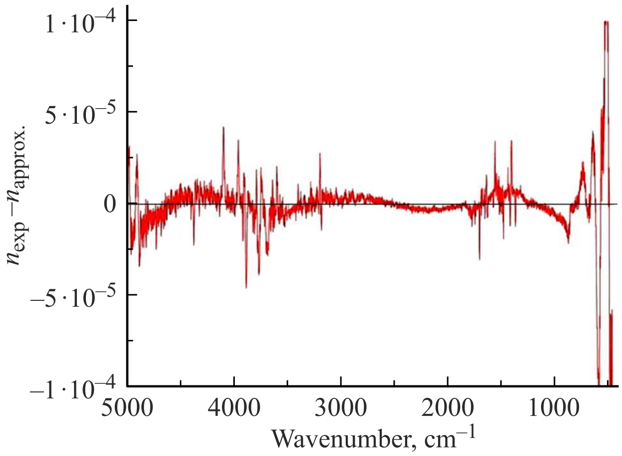

Figure 3. Spectral dependence of the difference between calculated n_{exp} and approximated $n_{\approx}$ refractive index values for ^{70}Ge.

In Figure 2, the spectral dependence of the refractive index of the monocrystal ^{70}Ge sample is presented in a narrow wavenumber interval 4200–3980 cm^{-1} far from the edges of electronic and phonon transitions of germanium. On the same graph, dispersion data for other isotopes obtained earlier in [16] are provided. The refractive index value for the ^{70}Ge ample was higher throughout the investigated spectral range. The results confirm the previously identified trend of increasing refractive index with decreasing average atomic mass of germanium isotopes.

Accuracy Analysis of Measurements

Figure 3 shows the spectral dependence of the difference between the calculated refractive index for the ^{70}Ge monocrystal and its approximated values.

It is seen that the deviation of the approximation from the calculated refractive index values is less than $5 \cdot 10^{-5}$ over a sufficiently wide spectral range. However, at wavenumbers less than 1000 cm^{-1} (wavelengths greater than 10 μm), the accuracy of refractive index determination decreases due to noise in transmission spectra (mainly caused by reduced intensity of the radiation source). The root-mean-square deviation of the refractive index did not exceed $3 \cdot 10^{-4}$, nd the relative error was $\varepsilon_n = 0.01\,\%$; with a confidence level of $\alpha = 0.95$.

In the interference refractometry method, the error in refractive index calculation in formula (3) consists of three components:

$$\Delta n = \sqrt{\left(\frac{\delta n}{\delta m}\Delta m\right)^2 + \left(\frac{\delta n}{\delta h}\Delta h\right)^2 + \left(\frac{\delta n}{\delta \nu}\Delta \nu\right)^2}, \tag{3}$$

where Δn — total refractive index determination error, $\Delta m \delta n/\delta m$ — error in determining the interference order $\Delta \nu \delta n/\Delta \nu$ — error in determining the frequency, $\Delta h \delta n/\delta h$ —error in determining sample thickness. Since the interference order number is determined absolutely

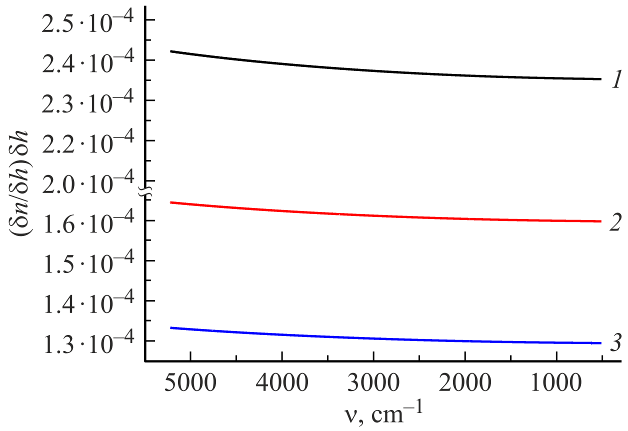

Figure 4. Spectral dependence of the refractive index error of ^{70}Ge, caused by sample thickness measurement error: $h = 0.085074$ cm (*1*), 0.125385 cm (*2*), 0.154781 cm (*3*). $\Delta h = 0.05\,\mu$m ($5 \cdot 10^{-6}$ cm) — thickness measurement uncertainty of the holographic gauge.

accurately, $\Delta m \delta n/\delta m = 0$. The greatest contribution to the refractive index determination error in this method is from the measurement error of sample thickness Δh. Figure 4 presents spectral dependencies of $\Delta h \delta n/\delta h$ for germanium samples of different thicknesses.

The refractive index measurement error of germanium $\Delta n = n\Delta h/h$ at unambiguous m values for all interference maxima when using one sample of about 1 mm thickness averages around $2 \cdot 10^{-4}$ (at $\Delta h = 0.05\,\mu$m). Using two or more samples reduces this error due to correlation of refractive index values measured for samples with different thicknesses over the entire measurement range. Our estimates show that the error in refractive index determination over the entire spectral range, due to uncertainty in sample thickness, did not exceed $1.4 \cdot 10^{-4}$.

Thus, based on data on the error in determining peak positions $\Delta\nu\delta n/\delta$, component $\sim 6 \cdot 10^{-5}$, similarly to previous work [16], the total refractive index determination error of ^{70}Ge by interference refractometry in our measurements did not exceed $2 \cdot 10^{-4}$.

Dependence of Refractive Index on Average Atomic Mass of Ge

Values of refractive index, calculated for several wavelengths in monocrystals enriched with stable isotopes ^{70}Ge, ^{72}Ge, ^{73}Ge, ^{74}Ge, ^{76}Ge, $^{\mathrm{nat}}$Ge, are presented in Table. 3.

The dispersion results of the refractive index in a wide wavelength range at 293 K obtained in this work for ^{70}Ge, complement previous studies of refractive indices of germanium monocrystal isotopes started in [15]. Values obtained for ^{70}Ge, allowed refinement of the approximation functions (1)–(3). It is seen that the dependence of measured refractive index values on the average atomic mass of the isotope at wavelengths 2.5, 5.0 and 10.0 μm shown in Figure 5 is nonlinear. A downward spectral shift of the refractive index dependence relative to the light isotope is observed (Figures 2 and 5).

Table 3. Refractive index of monocrystalline germanium enriched in ^{70}Ge, at wavelengths $2-10\,\mu$m, in comparison with data for other isotopes obtained previously [15]

Isotope	2 μm	2.5 μm	5 μm	10 μm
^{70}Ge	4.10890	4.06601	4.01577	4.00391
^{72}Ge	4.10797	4.06517	4.01502	4.00319
$^{\mathrm{Nat}}$Ge	4.10762	4.06501	4.01506	4.00325
^{73}Ge	4.10742	4.06479	4.01478	4.00295
^{74}Ge	4.10716	4.06461	4.01464	4.00281
^{76}Ge	4.10685	4.06433	4.01439	4.00258

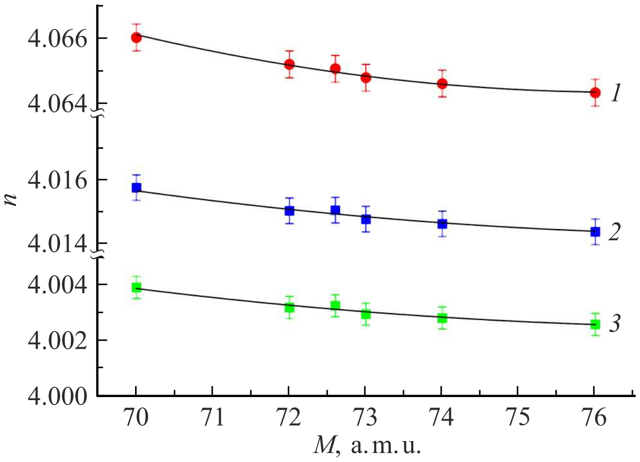

Figure 5. Dependence of refractive index value on average atomic mass of germanium at 2.5 μm (*1*), 5.0 μm (*2*) and 10.0 μm (*3*). Approximation curves: (*1*) $n = 4.28573 - 0.00577 \cdot M + 3.76075 \cdot 10^{-5} \cdot M^2$; (*2*) $n = 4.13573 - 0.0031 \cdot M + 1.97842 \cdot 10^{-5} \cdot M^2$; (*3*) $n = 4.12961 - 0.00326 \cdot M + 2.08513 \cdot 10^{-5} \cdot M^2$; M — average atomic mass in atomic mass units.

Comparative analysis with previously obtained data for germanium monocrystals of different isotopic composition showed that germanium enriched with ^{70}Ge, has the highest refractive index in the spectral range $1.94-20\,\mu$m at $T = 293$ K. The results confirm the trend of refractive index increasing with decreasing atomic mass of germanium isotopes.

The observed dependence of the refractive index is related to the average atomic mass of germanium by expression (4), which takes into account the classical oscillator lattice model with dipole interaction [27,28]. In this model, for a cubic lattice, neglecting absorption and spatial dispersion:

$$n^2(\omega) = 1 - \frac{\alpha E_g(M_i)}{\omega^2 - E_g^2(M_i)} - \frac{\tilde{\beta}\Omega_i}{\omega^2 - \Omega_i^2}, \qquad (4)$$

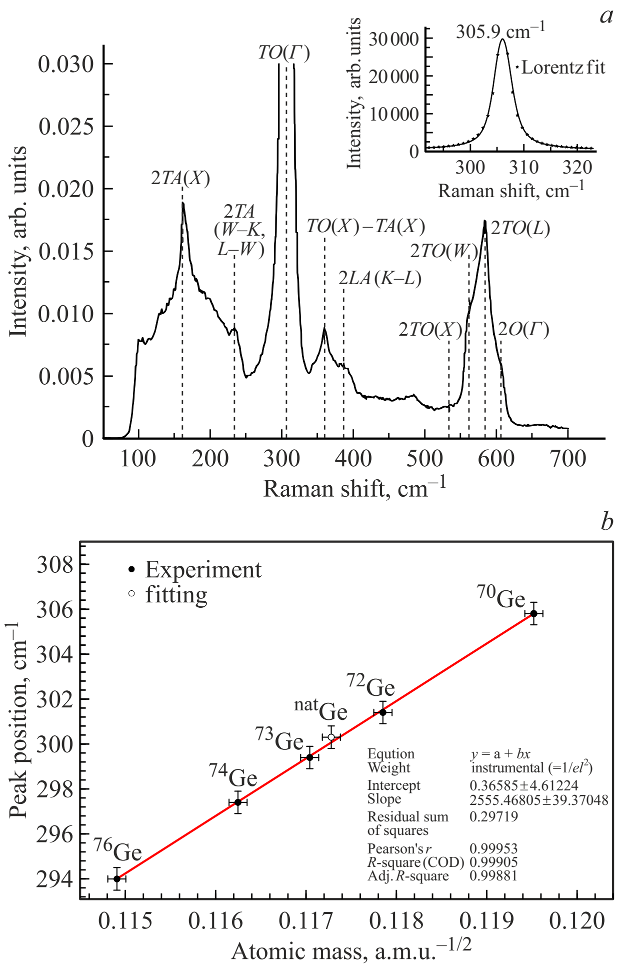

Figure 6. Raman scattering spectrum of monocrystal of germanium isotope^{70}Ge (*a*); Dependence of Raman band maximum on average atomic mass of germanium (*b*). Data for isotopes ^{72}Ge, ^{73}Ge, ^{74}Ge, ^{76}Ge and $^{\mathrm{nat}}$Ge taken from [16].

where E_g — bandgap width, M_i — atomic mass of isotope, Ω_i — phonon frequency, α and $\tilde{\beta}$ and model parameters describing two types of oscillators, corresponding respectively to the electronic and phonon edges of intrinsic absorption. Expressions for energies $E_g(T_i, M_i)$ and $\Omega_i(M_i)$ in the intrinsic absorption edge region of isotopically pure germanium crystal are given in [29–31].

Using expression (4) and known literature values [32,33] for the bandgap and phonon frequency of monocrystals of ^{70}Ge and ^{76}Ge isotopes, estimates were made of the refractive index difference for these isotopes at wavelengths 2.5, 5 and 10 μm. The obtained estimates agree with the experimentally observed trend of increasing refractive index with decreasing isotope atomic mass. More detailed studies will be pursued in future works.

Raman Scattering Spectra

In the Raman scattering spectrum of monocrystal germanium isotope ^{70}Ge, a band $TO(\Gamma)$ corresponding to first-order scattering on optical phonons at the symmetry point Γ is observed (Fig. 6).

Table 4. Frequencies of Raman band maxima for isotope ^{70}Ge

Phonon *	Maximum position, cm^{-1}
$2TA(X)$	162.4
$2TA(W-K, L-W)$	233.7
$TO(\Gamma)$	305.7
$TO(X)+TA(X)$	358.7
$2LA(K-L)$	388.9
$2TO(W)$	559.2
$2TO(L)$	584.0
$2O(\Gamma)$	607.0

Note: *Phonon assignment according to [34].

The maximum of the fundamental TO(G), band, approximated by a Lorentz function, is observed at 305.9 cm^{-1} , as shown in the inset of Fig. 6, *a*. The frequency of the band maximum in the Raman spectrum of ^{70}Ge shifts to shorter wavelength region compared to heavier isotopes. The results agree with earlier obtained dependencies of Raman band maxima on the average atomic mass of germanium. The positions of the fundamental band peaks for all stable germanium isotopes are shown in Fig. 6, *b* depending on average atomic mass $M_i^{-1/2}$. The red line represents the best linear fit of phonon wavenumber experimental values for all monocrystals according to the relation: $\Omega_i = 2559 \cdot (M_i[\text{a.m.u.}])^{-1/2}\,\text{cm}^{-1}$, valid in the harmonic approximation.

The residual sum of squares (RSS) of the linear regression was minimal (0.07) for the dependence $\Omega_i(M_i^{-1/2})$ of germanium isotope samples. This confirms the high structural perfection of monocrystals obtained from the isotopes. A similar picture was observed for mono-isotopic silicon samples in [14,15].

The second-order Raman scattering spectra of the monocrystal enriched with the isotope ^{70}Ge were studied, related to photon scattering via two-phonon processes. The frequencies of the Raman band maxima in the ^{70}Ge sample are presented in Table 4.

As with the first-order band, in the second-order spectrum there is a shift of the Raman band maxima towards shorter wavelengths with decreasing atomic mass. The greatest ratio of the isotopic shift magnitude to the atomic mass change $\Delta\omega/\Delta M$ is observed in the high-frequency part of the spectrum.

Conclusion

For the first time, data on the spectral dependence of the refractive index of monocrystalline germanium enriched with the isotope ^{70}Ge at 293 K were obtained. Comparison with refractive index determination results for heavier germanium isotopes showed that ^{70}Ge has the highest refractive index in the spectral range $1.94-20\,\mu$m, confirming the previously observed trend of increasing refractive index with decreasing average atomic mass.

The Raman scattering spectra of the monocrystalline ^{70}Ge sample were studied at room temperature. The results allowed refinement of the previously obtained dependence of the first-order Raman scattering frequency on the average atomic mass in germanium monocrystals enriched with isotopes ^{70}Ge, ^{72}Ge, ^{73}Ge, ^{74}Ge and ^{76}Ge.

Tabular data on the frequencies of second-order lattice vibrations of ^{70}Ge were obtained.

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Conflict of interest

The authors declare that they have no conflict of interest.

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