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Publication Date

1993-11-10



Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials Sciences Division

Submitted to Philosophical Magazine

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November 1993



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**ISOTOPICALLY CONTROLLED GERMANIUM: A NEW
MEDIUM FOR THE STUDY OF CARRIER SCATTERING BY
NEUTRAL IMPURITIES**

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NOVEMBER 1993

This work was supported in part by the Laboratory Directed Research and Development Program of the Lawrence Berkeley Laboratory through the U.S. Department of Energy under Contract No. DE-AC03-76SF00098. One of us (H.D.F.) acknowledges financial support from U.S. National Science Foundation Grant DMR-91 15856 and from the Alexander-von-Humboldt-Stiftung (Bonn/Germany).

**Isotopically Controlled Germanium:
A New Medium for the Study of Carrier Scattering by Neutral Impurities**

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ABSTRACT

The Hall-mobility of carriers in isotopically pure Ge crystals, doped by neutron transmutation (NTD) was measured over a wide temperature range. Our experimental data allow a quantitative comparison with theory. We find that at temperatures below 20K the mobility is limited by neutral impurity scattering and it can be quantitatively described by the theory of McGill and Baron.

1. INTRODUCTION

There has been a dramatic surge in interest in the effects of the isotopic composition on the physical properties of semiconductors. Manuel Cardona, to whom this paper is dedicated for the occasion of his 60th birthday, has stimulated and performed a variety of studies aimed to clarify how the vibrational, electronic, and optical properties of Ge are affected by the isotopic composition [Cardona *et al.* 1993(a), 1993(b), Fuchs *et al.* 1991, 1992(a), (b), 1993(a), (b), Etchegoin *et al.* 1992, 1993]. Currently, the results of such studies are being used to tailor materials properties for specific purposes and applications. Even though most of this work was done only in the last four years and many experiments are still underway, the investigation of isotope effects has a long and successful history in fields other than that

of semiconductors. In fact, some techniques that have been developed in previous years primarily for the defense industry (e.g., the separation of isotopes) are now being used in the newly developing research of the isotopic control of semiconductors. In this context, M. Cardona likes to quote the Biblical prophecy "...they shall beat their swords into plowshares and their spears into sickles..." [Isaia 2:4, 701 BC].

The tremendous potential of isotopically controlled materials in *solid state physics* was first realized by many researchers, when the isotope effect was used to show that superconductivity results from an interaction of the charge carriers with the lattice vibrations [Fröhlich 1950]. This is viewed as one of the main experimental findings which led to the BCS-Theory [Bardeen, Cooper and Schrieffer 1957]. Since then, the substitution of isotopes in a material has proven to be a powerful tool for identification purposes in numerous experiments. Because only the mass of the atom is changed (and to first order not the electronic or chemical properties) when one isotope is replaced by another, mass-sensitive measuring techniques can be used to identify the origin of many of the observed properties. A typical example for this is the isotope shift in local vibrational modes (e.g., replacement of H by D, or ^{16}O by ^{18}O) [e.g., Bosomworth *et al.* 1970, Pajot *et al.* 1987].

The first report on isotopically controlled *semiconductors* goes back to 1958, when Geballe *et al.* found that the thermal conductivity of enriched ^{74}Ge at 15 K was 4 times higher than that of natural Ge [Geballe and Hull 1958]. Many years later, isotopically enriched ^{12}C -diamond was found to be the "*world's most efficient heat conductor*" [Anthony *et al.* 1990]. Even though diamond in its natural form contains already 98.9% of the isotope ^{12}C , the room temperature thermal conductivity of diamond with 99.93% ^{12}C is enhanced by 50%. Such isotopically enriched diamond crystals have been used very recently as monochromators for synchrotron radiation, where the heating (and the subsequent broadening of the spectrum) is a persistent problem due to the high incident radiation density [Berman *et al.* 1993]. Isotopically enriched ^{13}C also holds other world

records: it is the "*hardest known terrestrial material*" [Ramdas *et al.* 1993] having the "*largest atomic density of any known solid*" [Holloway *et al.* 1991].

The lattice spacing of a crystal depends on the mass of the constituent atoms (through their mass-dependent vibrational frequencies). Substitution of a lighter isotope by a heavier one leads to a small decrease of the lattice spacing. This was confirmed experimentally for isotopically enriched ^{74}Ge [Buschert *et al.* 1988] and for ^{13}C -diamond [Holloway *et al.* 1991].

Another fundamental quantity which is affected by the isotopic constitution of a semiconductor crystal is its electronic band gaps. Changes in the lattice spacing (see above) as well as the coupling of the carriers to the mass-dependent lattice vibrations (electron-phonon coupling) lead to increased band gaps when the average isotopic mass is increased [Cardona *et al.* 1993(a), Zollner *et al.* 1991]. Even though the isotopic masses are spread only by about 10% in Ge and C resulting in band gap shifts of $\approx 0.3\%$, various measurements on diamond and Germanium were able to experimentally verify the theoretically expected shifts [Collins *et al.* 1990, Zollner *et al.* 1991, Davies *et al.* 1992, 1993, Etchegoin *et al.* 1992, 1993]. The shifts of the *indirect* band gaps of diamond and Ge were observed with photoluminescence (PL) measurements. The mass dependence of the *direct* band gap of Ge has been determined from infrared absorption measurements [Agekyan *et al.* 1989]. Spectroscopic ellipsometry [Zollner *et al.* 1991, Etchegoin *et al.* 1992, 1993] allowed an accurate analysis of the isotope shift of the so-called E_1 -gap of Ge, i.e., where the conduction and valence bands run parallel over a large portion of the Brillouin zone in the [111] direction [Cardona 1969].

The isotopic composition affects the lattice vibrations of a crystal (phonons) in three different respects: (i) The *average isotopic mass* M leads to a phonon frequency proportional to $M^{-1/2}$ (shift of about 4% of the total phonon energy for Ge and C). (ii) The *isotopic disorder* contributes to an additional k -dependent phonon selfenergy shift (ca. 0.5%). (iii) The *anharmonicity* results also in an additional frequency shift proportional to

$M^{-1/2}$, but it amounts to max. 0.06%. Detailed studies of the lattice dynamics were performed in the group of M. Cardona for Ge [Fuchs *et al.* 1991, 1992(a), (b), 1993(a), (b), Etchegoin *et al.* 1993] and by Hass *et al.* for diamond [Hass *et al.* 1992]. As opposed to "classical" alloys (e.g., Si/Ge), the scattering potential due to the mass defects in isotopically mixed Ge or C (isotopic mass fluctuations) is not strong enough to localize the vibrations.

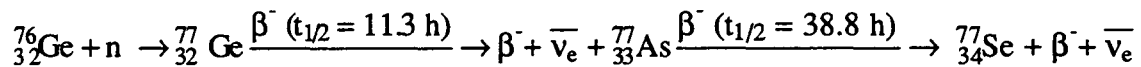
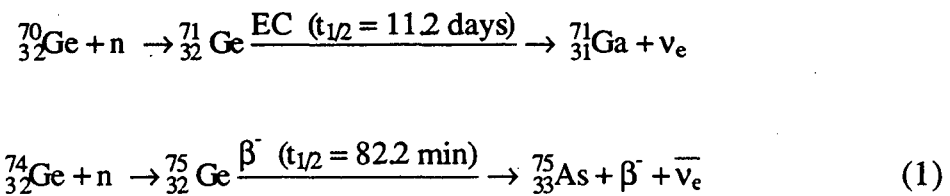
The frequency of a localized vibrational mode (LVM) of an impurity (e.g., O in Ge or Si) in a crystal is affected by the neighboring atoms. In an isotopically mixed crystal, there exist a multitude of LVMs because of all the different combinations of isotopic masses surrounding a given impurity. Various *isotopically enriched* Ge and diamond crystals have been used to unambiguously identify the different lines in the *mixed* crystals [Collins *et al.* 1988, Ramdas *et al.* 1993].

The strong absorption of neutrons by ^{113}Cd makes neutron diffraction measurements of the phonon dispersion of natural CdTe impossible. A special CdTe-crystal depleted of the isotope ^{113}Cd was grown for this purpose [Rowe *et al.* 1974]. Another example for difficulties arising due to the property of certain isotopes is the EPR study of As impurities in Ge. The nuclear spin of ^{73}Ge ($I=9/2$) interacts with the nuclear spin of As, leading to a strong line broadening in the EPR spectra in natural Ge. Wilson therefore used an isotopically enriched ^{74}Ge sample depleted of ^{73}Ge to obtain high-resolution EPR spectra [Wilson 1964].

Isotopic Ge superlattices (SL), i.e., layered structures where each layer consists of one particular isotope, were proposed by Haller (1990) and have been grown very recently by Schorer *et al.* (1993). They offer the possibility to experimentally study the influence of the dimensionality on the confinement of vibrations which has theoretically been predicted [Cardona *et al.* 1993(b), Fuchs *et al.* 1993(b)]. Isotopic SLs are also very useful to study the interdiffusion of atoms in thin-layered structures. An investigation of the selfdiffusion

of Ga in a $^{69}\text{GaAs}/^{71}\text{GaAs}$ SL was only partially successful, unfortunately, because native defects from the heavily doped substrate penetrated the layers [Tan *et al.* 1992].

Isotopic SLs display an extraordinary property which raised hope for technological applications: the layers can be selectively doped *after* the growth with neutron transmutation doping (NTD) [Haller 1990]. Certain isotopes of Ge decay into another chemical element after exposure to a flux of thermal neutrons. Three Germanium isotopes transmute into shallow *acceptors* (Ga), shallow *donors* (As), and *double donors* (Se):



The isotopes ^{72}Ge and ^{73}Ge are transmuted into the stable ^{73}Ge and ^{74}Ge , respectively. Modulation doped devices and many other applications are in reach with this method.

NTD is not only of interest for *isotopic SLs*. It is also used on a large scale for the production of doped high-power high-voltage Silicon rectifiers and SCRs [Schnoller *et al.* 1976]. In the case of Si, the isotope ^{30}Si transmutes into P-donors after neutron capture. Controlling the ratio of ^{70}Ge and ^{74}Ge *in bulk* Ge-crystals allows a "fine tuning" of the majority as well as the minority carrier concentration. Currently, this is the best method to vary the free carrier concentration independently from the compensation ratio. As opposed to other doping methods, NTD yields a very homogenous, perfectly random distribution of the dopants [Itoh *et al.* 1993(b)]. Thus, isotopically controlled crystals offer a unique possibility to systematically study the scattering mechanisms of the charge carriers in Ge.

At temperatures above about 80 K, the mean free path of free holes or electrons is limited by scattering from lattice vibrations (phonons) [Brown and Bray 1962]. At lower

temperatures, the occupation number of the phonons is so low that the ionized impurities in the crystal become the dominant scattering centers for the carriers [Chattopadhyay and Queisser 1981]. At even lower temperatures ($<20\text{K}$) the thermal energy is too small to excite all the carriers into the valence or conduction band, i.e., the free carrier concentration $n(T)$ is reduced and the impurity bands are filled. This leads to a reduction of the number of ionized impurities N_i to $N_i = n(T) + 2N_{min}$, where N_{min} is the minority carrier concentration. If $N_{min} \approx N_{maj}$ (heavily compensated) then N_i is appreciably larger than the neutral impurity concentration $N_n = (N_{maj} - N_{min}) - n(T)$ even at lowest temperatures. The scattering of carriers from neutral impurities can therefore usually be neglected in compensated materials. In low-compensated crystals, on the other hand, the freezeout of the carriers neutralizes the previously ionized impurities. In such materials, scattering from neutral impurities is the limiting process for the carrier mobility at very low temperatures.

Many authors have studied lattice scattering and ionized impurity scattering using both experimental and theoretical approaches. We refer the reader to the literature [Chattopadhyay and Queisser 1981]. Neutral impurity scattering, on the other hand, has received much less attention. Some *theories* have been developed by Erginsoy (1950), Ansel'm (1953), Sclar (1956), and McGill (1975). But because of experimental difficulties only little reliable *experimental data* are available for Ge [Schwartz *et al.* 1961, Temkin *et al.* 1962, Fukai *et al.* 1964, Otsuka *et al.* 1964, Blakemore 1980] and Si [Sclar 1956, Norton *et al.* 1972, Baron *et al.* 1976]. Our unique set of isotopically controlled NTD Ge crystals with their low compensation ($N_{min}/N_{maj} \approx 0.01$) allow a first systematic study of the temperature dependence of the neutral impurity scattering of *p*-type and *n*-type Ge.

2. EXPERIMENTAL DETAILS

The growth and characterization of the unique isotopically enriched Ge single crystals has been reported in detail elsewhere [Itoh *et al.* 1993 (a)]. The electrically active impurity

concentration in all crystals before the NTD was of the order of 10^{12}cm^{-3} . Thin slices ($\approx 1\text{mm}$ thick) of the ingots were irradiated with thermal neutrons for the NTD. The n -type crystals were obtained through NTD of isotopically enriched ^{74}Ge , the p -type crystals correspondingly from NTD of isotopically enriched ^{70}Ge . The neutron cross section for the neutron capture of the isotopes for our irradiation condition were determined to be $\sigma_c(^{70}\text{Ge})=2.5(5)\times 10^{-24}\text{ cm}^2$ and $\sigma_c(^{74}\text{Ge}) = 0.6(1)\times 10^{-24}\text{ cm}^2$ by Itoh *et al.* (1993(b)). To remove structural defects due to the unintentional irradiation with fast neutrons, all samples had to be thermally annealed at 650°C for 10 seconds in a rapid thermal annealer [AG Associates].

We have performed temperature dependent Hall measurements on four different p -type and two different n -type Ge crystals. Their Hall-mobility obtained from the conductivity and free carrier concentration data (listed in Table I) are displayed in Fig.1. A magnetic field of 3kG was used, i.e., for the temperature range of interest for the neutral impurity scattering the high-field limit $\mu B \gg 1$ is satisfied and the Hall mobility can be equated with the drift mobility. The physical mechanisms responsible for the limitations of the carrier mobility in the different temperature ranges will be discussed in the next section.

3. RESULTS AND DISCUSSION

We analyzed the mobility data of Fig.1 in terms of scattering of the carriers from phonons (μ_1), ionized impurities (μ_i) and neutral impurities (μ_n), assuming Mathießen's rule [Mathießen and Vogt 1864]

$$\frac{1}{\mu} = \frac{1}{\mu_1} + \frac{1}{\mu_i} + \frac{1}{\mu_n} \quad (2)$$

A more detailed and complete analysis will be published elsewhere [Itoh *et al.* 1993 (c)]. In this paper, we want to emphasize the relative contributions of the different scattering mechanisms at various temperatures. Thus, to extract the neutral impurity scattering contribution we subtracted $(1/\mu_l + 1/\mu_i)$ from the measured $1/\mu$.

The lattice contribution $1/\mu_l$ was obtained by a least square fit to the measured mobility in the higher temperature region. Scattering from acoustic phonons should lead to a $T^{-3/2}$ dependence of μ_l . However, empirically the temperature dependence was previously found to be $T^{-1.66}$ for electrons and $T^{-2.33}$ for holes due to additional scattering from optical modes and the particular structure of the energy bands [Morin and Maita 1954].

The ionized impurity contribution $1/\mu_i$ was calculated within the approximation of Brooks and Herring BH [Brooks 1955], which neglects the energy dependence of the relaxation time. It is well-known that the BH result generally overestimates the observed mobility, and many possible corrections have been suggested to improve this approximation under various conditions [Chattopadhyay and Queisser 1981]. We corrected the BH mobility by using an over-all scaling factor (0.4 for *p*-type Ge, 0.68 for *n*-type Ge) which was adjusted so as to fit the measured mobility in the 30-70K temperature range.

The relative contributions of phonon scattering ($1/\mu_l$), ionized impurity scattering ($1/\mu_i$), and the resulting neutral impurity scattering ($1/\mu - 1/\mu_l - 1/\mu_i$) are plotted in Fig.2 (Ge:Ga #2). For $T > 80\text{K}$, phonon scattering is the dominant scattering mechanism. Comparing Fig.1 and 2, it becomes clear that the "dip" in the carrier mobility around 50K is caused by scattering from *ionized impurities*, which dominate the scattering of the carriers between 20K and 80K. The flattening and saturation of the mobilities below about 20K originates from *neutral impurity* scattering, which can only be observed in crystals with very high crystalline quality and low compensation, like our isotopically enriched NTD Ge crystals.

Figure 3 shows the relative strength of the scattering from the ionized and the neutral impurities. There is only a relatively small temperature region in which the scattering from the neutral impurities dominates. This range extends to higher temperatures as the free carrier concentration is increased. The calculated "transition temperatures" above which the ionized impurities are the main scattering centers [Dzhakeli and Kachlishvili 1984] compare very well with our experimental results (see inset of Fig.3).

Finally we compare our results with previous calculations of the mobility limited by the scattering from neutral impurities. Most models have used the analogy with the elastic scattering of electrons from neutral hydrogen by appropriately scaling the Bohr radius and the ground state binding energy [Erginsoy 1950, Ansel'm 1953, Sclar 1956, McGill 1975]. Ansel'm, Sclar, and McGill modified Erginsoy's temperature-independent mobility by considering "resonant" scattering in analogy with the weakly bound H^- system. Within the accuracy and temperature range of our experiment, their corrections are identical; they deviate from each other for very low temperatures, however [McGill *et al.* 1975]. Figure 4 compares our experimental results with the theoretical predictions. Note the excellent overall agreement, considering the simplicity of our analysis. The difference between Erginsoy's calculation and the ones by Sclar and McGill *et al.* is of the same order of magnitude as the uncertainty of our results. Nevertheless, our data seem to support the trend of an increasing mobility with increasing temperature. Compared to the temperature dependence of the lattice (μ_l) or the ionized impurity scattering (μ_i), however, the neutral impurity scattering limited mobility (μ_n) is "almost" temperature independent.

4. CONCLUSIONS

We have measured the Hall mobility of a unique set of isotopically pure, NTD doped Ge crystals with excellent crystalline quality and compensation ratios as low as 1%. The temperature dependence of neutral impurity scattering has been analyzed and found to be in excellent agreement with previously published theoretical predictions. Our knowledge of

the properties of isotopically enriched NTD bulk crystals will lay the foundation for future work on low dimensional, layered structures which might open a new field of exciting fundamental physical studies as well as promising applications.

ACKNOWLEDGMENTS

We would like to thank W. Walukiewicz for many valuable discussions, W.L. Hansen for help with the crystal growth, J. W. Farmer for the neutron irradiation of our crystals, and V. I. Ozhogin for providing us with the isotopically enriched raw material. This work was supported in part by the Laboratory Directed Research and Development Program of the Lawrence Berkeley Laboratory through the U.S. Department of Energy under Contract No. DE-AC03-76SF00098. One of us (H.D.F.) acknowledges financial support from US NSF Grant DMR-91 15856 and from the Alexander-von-Humboldt-Stiftung (Bonn/Germany).

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Figure Captions

Fig.1: Temperature dependence of the carrier mobility of *p*-type (a) and *n*-type (b) NTD Germanium.

Fig.2: Temperature dependence of the relative contributions to the mobility. Note that the mobility is dominated by neutral impurity scattering below 20K (Ge:Ga #2 crystal).

Fig. 3: Ratio of ionized and neutral impurity scattering, μ_i/μ_n . The "transition temperature" above which the *ionized* impurity scattering dominates over the *neutral* impurity scattering increases with increasing carrier concentrations. The calculated data of the inset are taken from Dzakeli and Kachlishvili (1984).

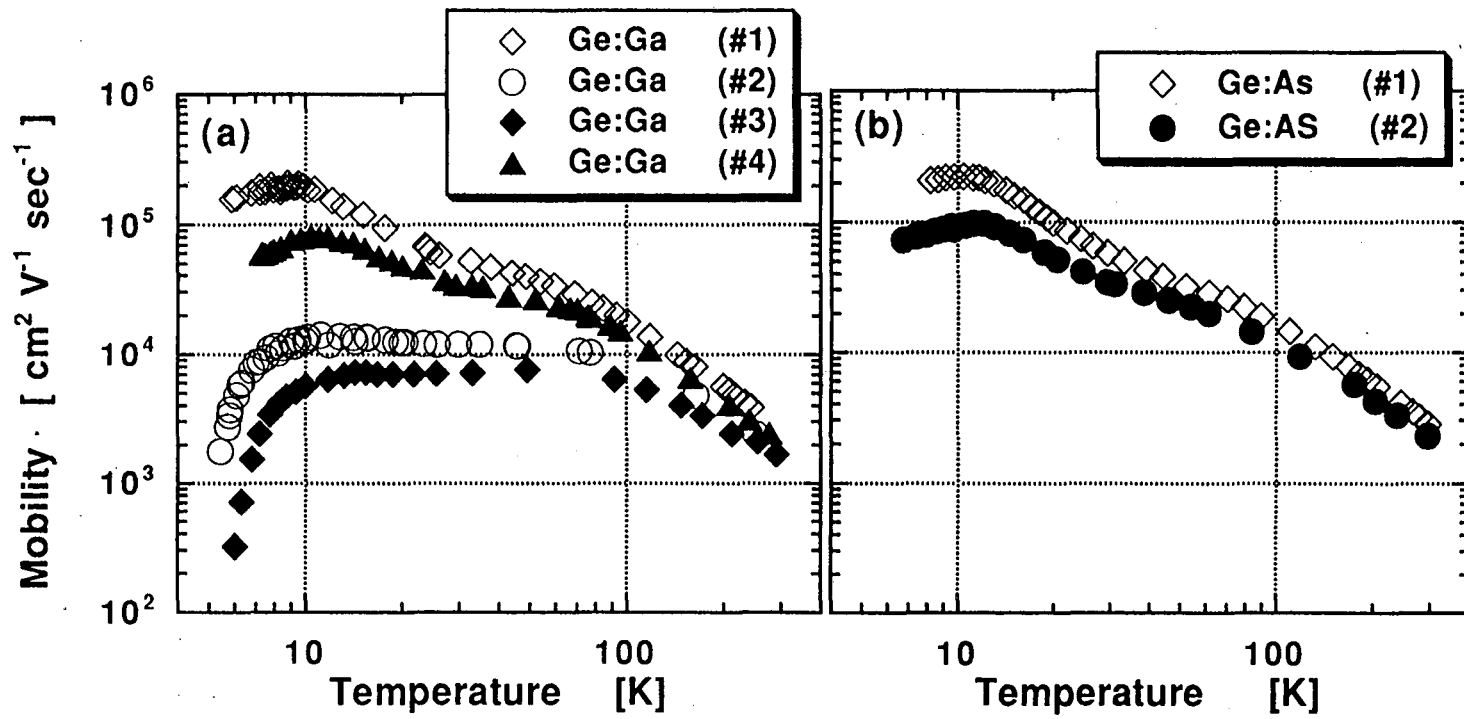
Fig.4: Neutral impurity scattering limited mobility ($\mu_n N_n$): comparison of our experimental results with previous calculations.

Tables

Table I: Carrier concentrations of the Ge crystals used in this work

		$N_A - N_D$	N_D	$K=N_D/N_A$
<i>p</i> -type	Ge:Ga #1	3.1×10^{14}	3×10^{12}	9×10^{-3}
	Ge:Ga #2	7.7×10^{15}	9×10^{13}	1.2×10^{-2}
	Ge:Ga #3	1.7×10^{16}	2×10^{14}	1.2×10^{-2}
	Ge:Ga #4	1.0×10^{15}	1.2×10^{13}	1.2×10^{-2}
		$N_D - N_A$	N_A	$K=N_A/N_D$
<i>n</i> -type	Ge:As #1	3.5×10^{14}	8.5×10^{12}	2.4×10^{-2}
	Ge:As #2	1.2×10^{15}	1.2×10^{13}	1.0×10^{-2}

Figure 1.



70#7-314.KGD

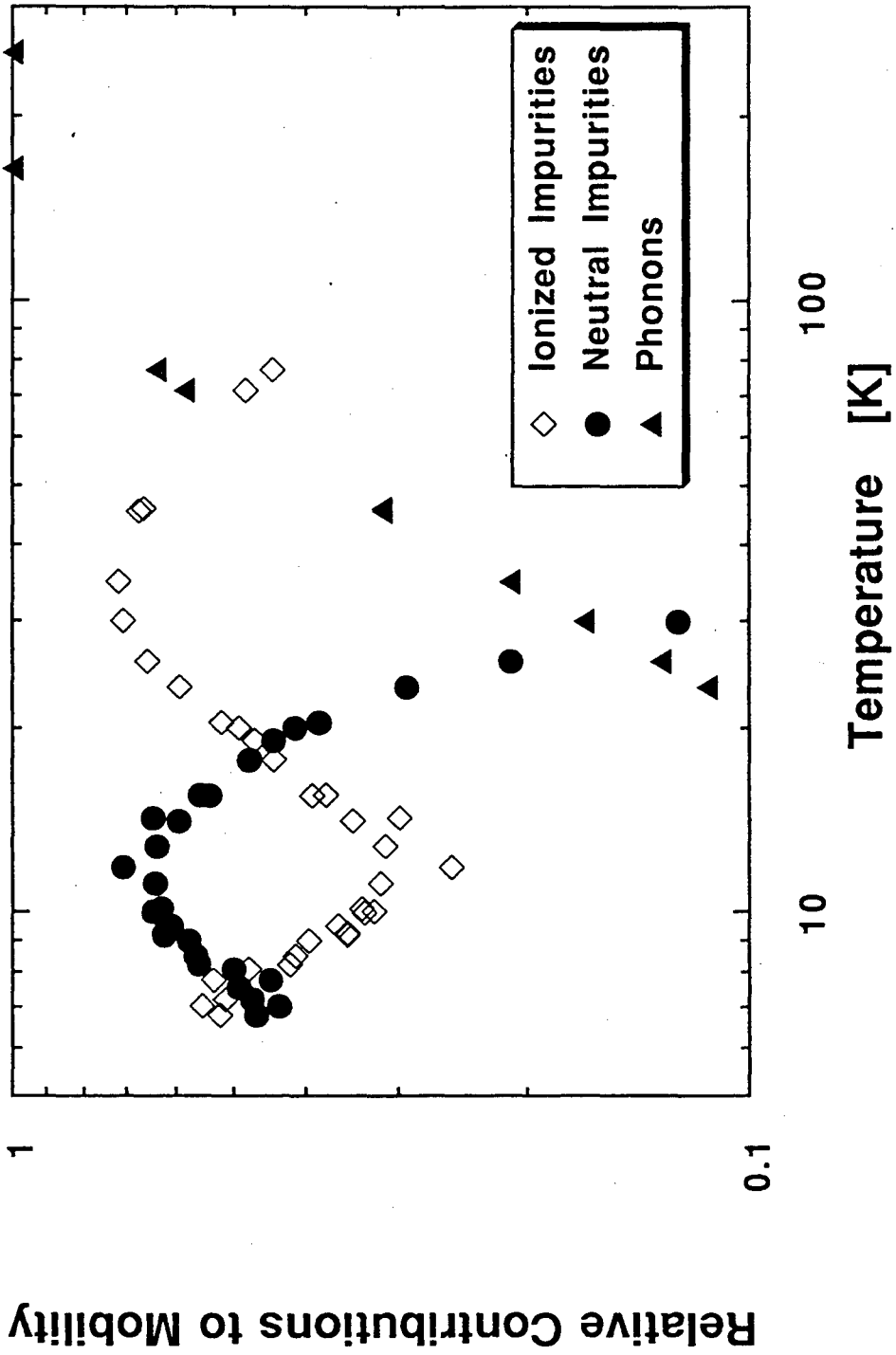


Figure 2.

Figure 3.

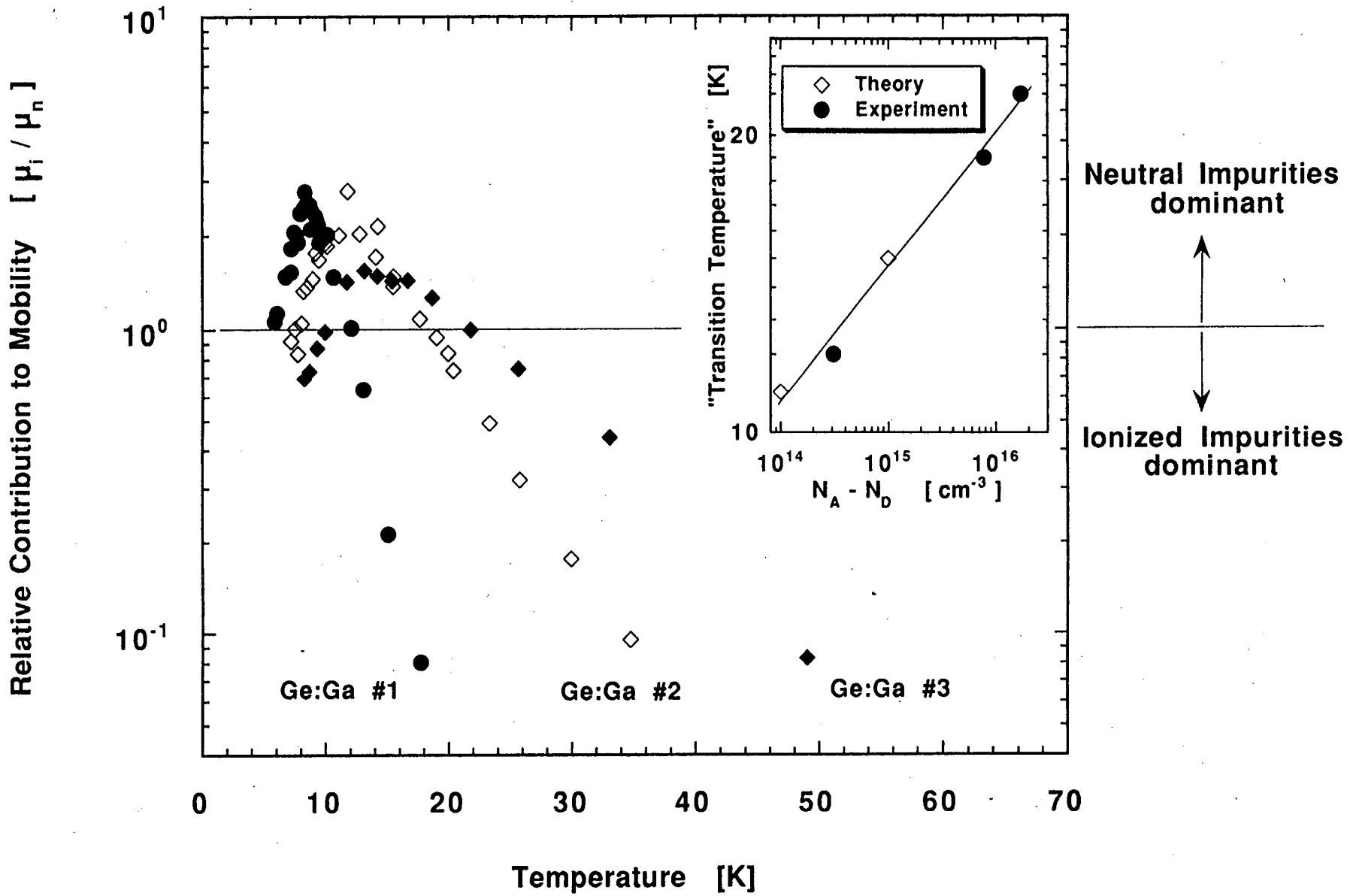
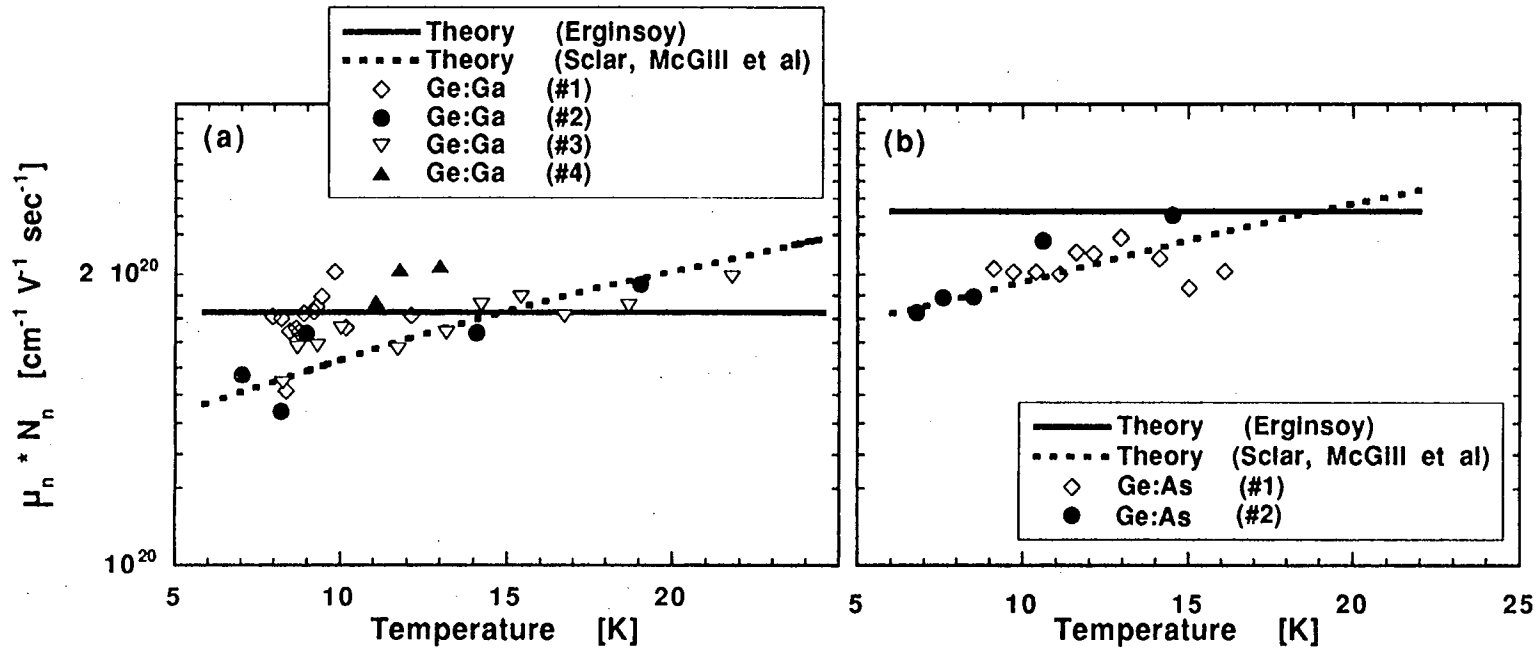


Figure 4.



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