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V L Ginzburg and the development of experimental work on high-temperature superconductivity at LPI: 'iron superconductors'

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1. Introduction

One day in 2006, one of the authors of this paper (VMP) received a surprise phone call with an exciting proposal. The caller was V L Ginzburg, and the proposal was to undertake research into high-temperature superconductivity (HTSC) to develop superconductors with the critical temperature above room temperature: a worthwhile effort, Ginzburg convincingly explained, because it was of exceptional practical importance and because no theoretical reason was known to forbid room-temperature superconductivity (RTSC).

What does changing the subject mean for an experimentalist? First, the scale of the proposed research ruled out small group work and required the effort of most, if not all, of the laboratory, thus necessitating that the personnel be freshly trained and undergraduate programs be set up to prepare specialists in the new field. Second, the project needed to be financed and equipment and materials had to be purchased. Finally, it was necessary to find funds for refitting the building for different experimental work and to develop a redesign project for the existing building, as a whole and in parts, to optimize the operation of the new equipment. It was not until after three years [1] of these types of concerns that the first experiments were carried out to synthesize and study

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high-temperature superconductors. Ginzburg showed keen interest in how the work was progressing and was constantly briefed on it [2]. This talk is, in fact, a regular progress report which should but alas cannot be submitted to Ginzburg.

At about the same time, in 2008, a new class of high-temperature superconductors, based on iron arsenides and iron selenides rather than cuprate compounds, was discovered [3–6], providing a logical starting point for the new experimental project. Because 'broad front' research was planned — and this with no experience in fields such as material science and analytical diagnostics — the cooperation of the Lebedev Physical Institute, Russian Academy of Sciences (LPI), with research bodies possessing the necessary experience and technology was absolutely necessary. Accordingly, a number of laboratories of Moscow State University's chemical and physical departments became involved in the project, as did the Institute for High Pressure Physics (HPPI), Russian Academy of Sciences. The first physical results on 'iron' superconductors from the collaboration of the LPI high-temperature superconductivity department with these institutions are summarized in this talk.

2. Briefly on the properties of 'iron' superconductors

There are some similarities and numerous differences found in comparing the properties of cuprate oxides and the new, 'iron' superconductors [7]. To date, a number of types of such materials have been synthesized and studied, which are classified as 1111 (50 K), 122 (40 K), 111 (18 K), 11 (8 K), and 22438 (40 K). Their typical representatives are *RE* FeAsO(F) (where *RE* = Sm, La, Dy, Eu, Th, Gd, and so on) [8, 9], $Ba(K)Fe_2As_2$ [10 – 12], LiFeAs [13, 14], FeSe(Te) [15], and $Fe_2As_2Ca_4(Sc,Ti)_3O_8$ [16]. Detailed state-of-the-art reviews of the subject were given in Refs [7, 17–19]. Similarly to the cuprates, the new compounds are layered, with layers of Fe, where electrons condense into superconducting pairs, spatially separated from oxygen layers that supply charge carriers when the composition deviates from the stoichiometry.

Unlike the cuprates, stoichiometric (undoped) FeAs materials are not insulators above T_c but show band conduction, and hence are metals, if poor ones. As the temperature is decreased, they undergo a structural transition from tetragonal to orthorhombic spin ordering (at $T \approx 150$ K) and then, at an even lower temperature ≈ 130 K, a magnetic transition leading to the antiferromagnetic (AF) spin ordering in the Fe sublattice. The anomalously strong magnetostructural coupling — or, more precisely, the coupling between the spin state of Fe and the lattice structure (the displacement of As atoms) — was discovered by comparing inelastic neutron scattering data [20] for the normal state with the calculated phonon spectrum [21, 22]. It turned out that the calculated and measured [20] positions of peaks in the phonon spectrum were in marked disagreement (up to 14%), which could not be removed unless the magnetic moment of Fe was taken into account. For the same reason, the calculated positions of As atoms in the lattice turn out to be strongly dependent on the magnetic moment of Fe atoms, and the calculated lattice constant along the *c*-axis is 10% (!) less than measured if the calculations ignore the magnetic moment of Fe.

A deviation from stoichiometry, due either to an induced oxygen deficiency or to oxygen being in part substituted by fluorine, suppresses the antiferromagnetic ordering of the Fe sublattice and gives rise to a superconducting (SC) state [7, 17, 18, 19, 23, 24]. The facts that the magnetic and structural transitions have their temperatures close to the critical temperature $T_c \approx 50$ K and that magnetostructural coupling is anomalously strong suggest a dominant role of phonons and possibly of spin fluctuations in superconducting pairing [25–28].

The key issues addressed in the study of these materials include the mechanisms of various types of doping, the carrier pairing mechanism, the order parameter symmetry, the quasiparticle energy spectrum, the possible existence of a pseudogap state, and the superconducting gap values. The following intriguing properties of this class of superconductors have stimulated great interest in their study: the possibility (currently being discussed) of an unusual order parameter symmetry, an unusually strong coupling between spin fluctuations and phonons, the emergence of an SC state regardless of the Mott insulator (in contrast to cuprates), the competition between AF ordering and SC pairing, and the existence of spin ordering of rare earth elements in the SC phase below T_c [23].

The essential point is that the isotope effect due to the substitution of ^{16}O by the ^{18}O isotope turned out to be much weaker that the iron isotope effect (i.e., substitution of ^{58}Fe for ^{56}Fe), confirming that electron pairing predominantly occurs in Fe layers. The isotope effect exponent $\alpha = 0.4$ is close to the standard Bardeen–Cooper–Schrieffer (BCS) theory prediction 0.5. Band structure calculations show that the total density of states at the Fermi level is formed from 3d atomic states of Fe [29, 30] and that the critical temperature correlates with the density of states [30] (evidence of superconducting pairing assisted by phonons in these compounds).

Knight shift measurements in 122 and 1111 compounds [31–33] show conclusively that the superconducting pairing is singlet and hence the coordinate wave function of the condensate must be antisymmetric. For the superconducting gap, s and d symmetries are possible. Data from different experiments are still inconsistent and can be interpreted as favoring multiband superconductivity with either the s^{++} [34] or $s^{\pm}$ [32, 33, 35] or d symmetry (see Refs [7, 17] for a detailed review of available experimental data); this problem remains unsolved experimentally. If experimentally confirmed, the theoretically proposed $s^{\pm}$ symmetry [25–28] would imply the existence of a previously unencountered type of multiband superconductivity, with the sign of the order parameter different for two different condensates, at the Γ and M points. It has also been predicted theoretically that for the order parameter with this type of symmetry, the superconducting condensate can coexist with antiferromagnetic order [36].

The magnitude Δ and the structure of the superconducting gap are closely related to the pairing mechanism. ARPES measurements, even when performed at the temperature 0.3 K, do not yet provide a sufficient (≈ 0.01 meV) resolution to reveal the fine structure of the superconducting gap. In addition, ARPES spectra are difficult to interpret because measuring photoemission involves only a thin near-surface layer of the material and because the surface itself undergoes a rearrangement [37] that changes the spectrum of the near-surface layer. As a result, the information on the gap comes almost exclusively from microcontact spectroscopy in either the tunneling (T) or the Andreev reflection (AR) regime. It turns out, however, that different experiments give different results [38–41], even when using similar measurement methods and performed on similar 1111-class materials (including the most studied compound, SmO(F)FeAs). Such differences may be not only quantitative but also qualitative: some experiments report on the d-wave symmetry, on single-band superconductivity, and, finally, on two-band superconductivity.

These experimental inconsistencies are partly due to the fact that the quaternary 1111 compounds are difficult to synthesize: they are currently available almost exclusively in a polycrystalline form and are usually not homogeneous. Moreover, in a number of experiments, surfaces not cleaned in advance or not cleaved under high-vacuum cryogenic conditions were studied by microcontact spectroscopy.

3. Sample synthesis and preparation

In recent collaborative work with LPI, Moscow State University (MSU), and the Institute of High Pressure Physics, type-1111 HTSC compounds CdFeAsO(F), DyFeAsO(F), CeFeAsO(F), and EuFeAsO(F) were synthesized, for which microcontact spectroscopic studies in the superconducting state were carried out for the first time, the presence of two superconducting gaps in the superconducting state was revealed, and the gap values were measured. The maximum critical temperature, $T_c = 52 - 53$ K, was observed for $GdFeAsO_{1-x}F_x$ samples with the doping level $x = 0.12$ [42].

Polycrystalline samples of $GdFeAsO_{1-x}(F_x)$ were prepared by high-pressure synthesis [42, 43]. The chips of high-purity (99%) Gd and As, and powders of FeF_3, Fe, and Fe_2O_3 (99.9%) were used as starting materials. The precursors GdAs and FeF_3 produced in the first stage were mixed in a nominal proportion with Fe and Fe_2O_3 and pressed into pellets 3 mm in diameter and 3 mm in height. These were then put into a boron nitride crucible and a synthesis process was carried at about 50 kbar and at 1350 °C for 60 min.

Figure 1 presents the results of quantitative X-ray analysis of the powder diffraction data ($CuK_{\alpha 1}$-radiation, reflection geometry) obtained with Bruker-D8 Advance diffractometer using the Rietveld full-profile refinement method. The X-ray diffraction patterns indicate that the materials studied have a fine polycrystalline structure dominated by the 1111-type phase and contains an admixture of secondary phases (in particular, FeAs and Gd_2O_3). The primary phase, GdFeAsO(F), has the space group symmetry $P4/nmm$ and unit cell constants $a = 3.8982(3)$ Å and $c = 8.4059(9)$ Å. Because admixture phases decrease in content inward from the surface, the surface layer was polished away. The subsequent elemental analysis of sample surfaces using the JSM-700IFA scanning microscope with an EDX (Energy-Dispersive X-ray) extension shows that the Gd-excess regions were distributed randomly in the form of grains about 1 μm in size. Figure 2 demonstrates the results of the local elemental composition analysis over a region $\approx 1\ \mu m^2$ in area ('spectrum 1'). The table shows the percentage of each of Gd, Fe, and As in five randomly chosen regions of the sample (the oxygen content failed to be reliably measured) and average values over an area of 1.175 mm^2.

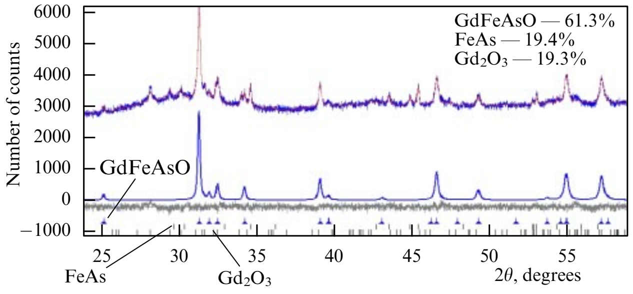

Figure 1. Measured profile (upper curve), refined results, and peak positions of the Gd-1111 phase (middle curve) and the difference between the measured and fitted spectra (lower curve).

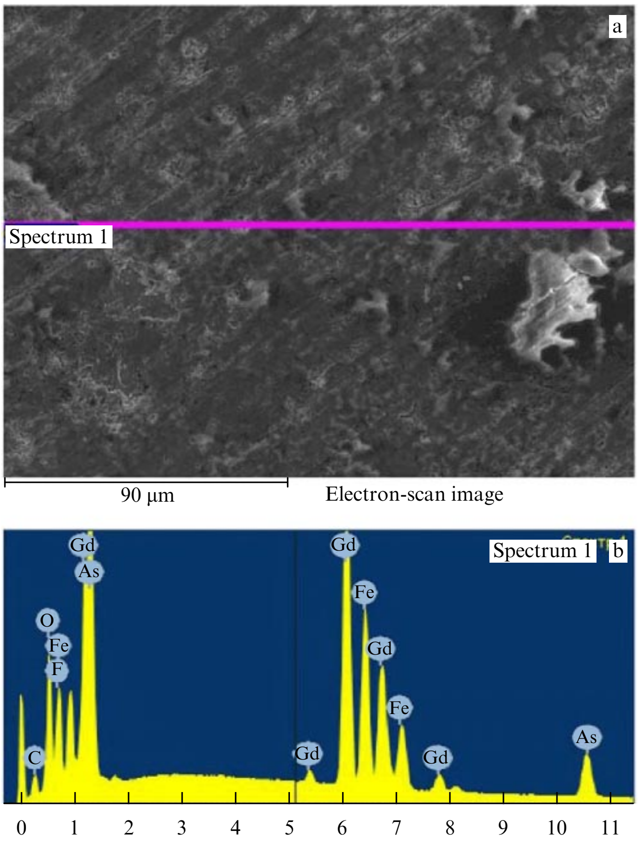

Figure 2. (a) Scanning electron microscope image of the sample surface and (b) the local spectrum of the characteristic lines of elements for the surface region designated as 'Spectrum 1.' Abscissa: energy in keV.

As confirmed by magnetic measurements [42], a bulk superconducting phase is present in synthesized samples. Shown in Fig. 2 is the temperature dependence of the resistance $R(T)$ for the approximately optimal composition $x = 0.12$ for a number of magnetic field values; the sharp peak of the derivative dR/dT (Fig. 3b) indicates the narrow width

Table. Percentage content of Fe, As, and Gd in different regions of the sample surface (in atomic percent).

Sample regions	Fe	As	Gd
2	11.74	11.36	20.55
4	14.64	15.43	19.16
5	16.11	15.29	17.19
6	16.17	13.57	18.70
7	14.44	15.30	18.12
Average content over the area 350 × 500 μm^2	14.1±2.2	15.5±1.9	18±11

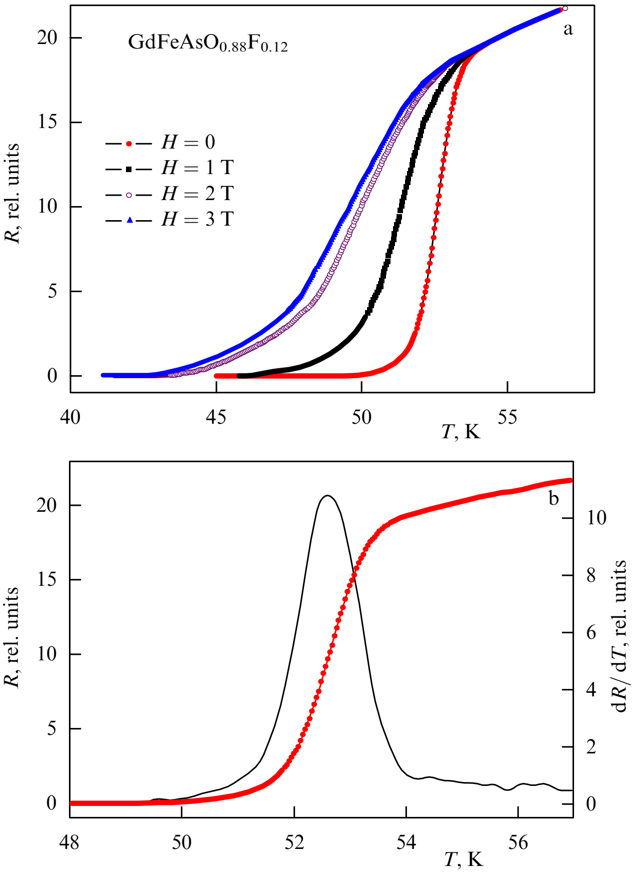

Figure 3. (a) Temperature dependence of resistance for three magnetic field values for a GdFeAsO(F) sample of an approximately optimal composition [37]. (b) Derivative $\mathrm{d}R(T)/\mathrm{d}T$ for $H=0$.

of the superconducting transition. The superconducting transition temperature $T_c \approx 52.5$ K, determined from the maximum of the derivative $\mathrm{d}R(T)/\mathrm{d}T$, is slightly less than the maximum value $T_c = 55.4$ K that has been achieved for this class of compounds (specifically, for SmFeAsO(F) [8]). In a magnetic field, the superconducting transition is broadened and T_c decreases monotonically (Fig. 3a), a behavior typical of FeAs superconductors [7, 17, 43]. Measurements [42] of $T_c(H)$ yielded the estimate $|\mathrm{d}H_{c2}/\mathrm{d}T| \approx 3$ T K^{-1}, comparable to the optimally doped superconductor SmFeAsO(F) [43] with close values of T_c. Using the above value of the derivative, the critical field is estimated as $H_{c2} \sim 130$ T, implying good prospects for high magnetic field applications of FeAs superconductors.

Along with polycrystalline 1111-system samples, we also studied variably doped single crystals of the 122 system; in these, the optimal doping T_c reached a value about 34 K.

4. Measuring the specific heat jump at the superconducting transition

The specific heat jump at the superconducting transition has been measured in the single crystals of 122 compounds $Ba_{1-x}K_xFe_2As_2$, $Ba(Fe_{1-x}Co_x)_2As_2$, and $Ba(Fe_{1-x}Ni_x)_2As_2$, varying in the type and level of doping. In ordinary superconductors, measurements of the jump provide information on the electron density of states at the Fermi level, the electron–electron coupling constant, and the volume of the superconducting phase. In the BCS model, the jump in the electronic specific heat is given by $\Delta C = 1.43\gamma T_c$, where γ is the electronic specific heat coefficient. This relation holds well for low-temperature superconductors.

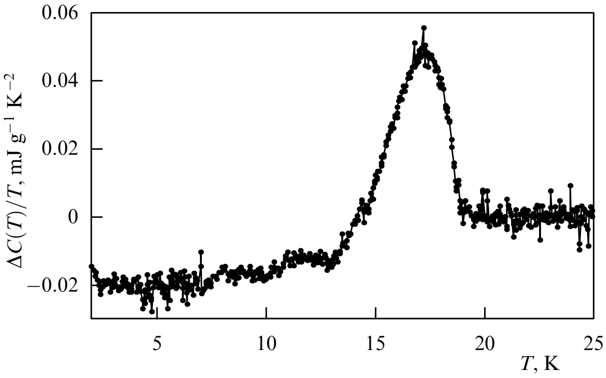

Figure 4. $\Delta C(T)/T = C(T, H=0)/T - C(T, H=9\ \mathrm{T})/T$ is plotted for an electron-doped sample of $Ba(Fe_{1-x}Ni_x)As_2$ [44].

For high-temperature superconductors, the situation is much more complex. In particular, underdoped phases of HTSC cuprates show no specific heat jump at the superconducting transition point at all [44]. Still, such samples can achieve the critical temperature above 60 K. Importantly, because such samples are structurally perfect, there is no reason to speak of their crystal inhomogeneity as a possible mechanism behind the blurring of the transition.

In [44], specific heat was measured in magnetic fields from 0 to 9 T to determine the jump $\Delta C/T_c$ at $T = T_c$. The field $H = 9$ T shifted the transition to lower temperatures, and the magnitude of the jump was estimated from the difference curve $\Delta C(T)/T = C(T, H=0)/T - C(T, H=9\ \mathrm{T})/T$ (Fig. 4).

An important result of that work was to establish an empirical relation between the transition temperature T_c and the magnitude of the specific heat jump in 122 iron pnictides. It was found that the specific heat jump is given by $\Delta C/T_c \propto T_c^2$, showing that it is determined only by the transition temperature T_c and not by whether the material is hole-doped or electron-doped or by the dopant concentration (Fig. 5). This agrees well with the results in Ref. [45]. The data obtained lend indirect support to the assumption that similarly to the cuprates, the 122 iron pnictides have both the specific heat jump and T_c determined by a single parameter. The reason for this unusual ΔC versus T_c dependence is not yet clear. It is only to be hoped that future

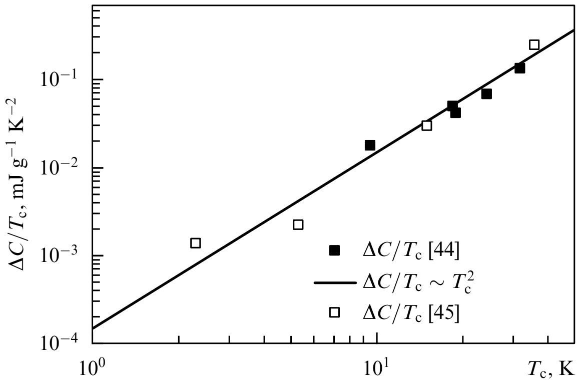

Figure 5. Specific heat jump $\Delta C/T_c$ as a function of T_c for type 122 iron pnictides. Black squares: results in Ref. [44], white squares: data in Ref. [45].

research will elucidate the physical mechanism behind this relation.

5. Measuring the characteristics of a ‘superconductor–normal metal–superconductor’ (SNS) microcontact

The microcontact spectroscopy studies of Gd-1111 either used the multiple Andreev reflection spectroscopy [46] of individual ‘superconductor–constriction–superconductor’ microjunctions [47] (with the constriction acting as the normal metal) or relied on the intrinsic multiple Andreev reflection spectroscopy of stacked contacts, which often arise due to the presence of steps and terraces on pure cryogenic cleaves of a crystal. In these studies, synthesized pellet-shaped samples were cut into thin rectangular plates ($2 \times 1 \times 0.1$ mm^3), which were mounted on the microjunction spectrometer stage. The spectrometer holder was subjected to controlled precision bending at the temperature 4.2 K to create a crack in the sample. The details of this ‘break junction’ measuring technique can be found in Refs [48, 49].

Figure 6 shows the measured current–voltage characteristic $I(V)$ and its derivatives $\mathrm{d}I(V)/\mathrm{d}V$ and $\mathrm{d}^2I(V)/\mathrm{d}V^2$ for a single SNS microcontact on a polycrystalline sample of $\mathrm{GdO_{0.88}F_{0.12}FeAs}$ [50]. The symmetric $I(V)$ characteristic seen in the figure is typical of ‘pure’ SNS microcontacts with a moderate excess current [47, 51]. The differential conductance $\mathrm{d}I(V)/\mathrm{d}V$ exhibits a series of dips, at $V = 22$, 11, and 5 mV. In the case of multiple Andreev reflection, dips should appear at the SNS contact voltages $V_n = 2\Delta/en$ with integer $n = 1, 2, \ldots$. Therefore, the first two features can be associated with $n = 1, 2$ and the local value of the superconducting gap can be estimated as $2\Delta = 22$ meV. For a two-gap superconductor, it can be expected that there are two independent subharmonic sequences corresponding to the large (Δ_{L}) and small (Δ_{S}) gaps in the spectrum. Supporting this view is the fact that the feature at $V \approx 5$ meV in Fig. 6 does not correspond to the expected voltage 7.3 meV for the third harmonic from the large gap and hence indicates the presence of a smaller gap ≈ 2.5 meV.

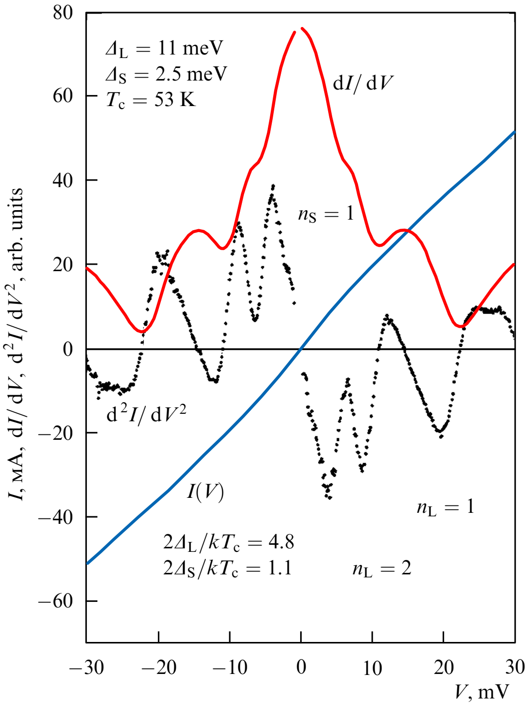

Figure 6. Current–voltage characteristic $I(V)$ and its derivatives $\mathrm{d}I(V)/\mathrm{d}V$ and $\mathrm{d}^2I(V)/\mathrm{d}V^2$ for a single SNS contact at $T = 4.2$ K for a $\mathrm{GdO_{0.88}(F_{0.12})FeAs}$ sample [50].

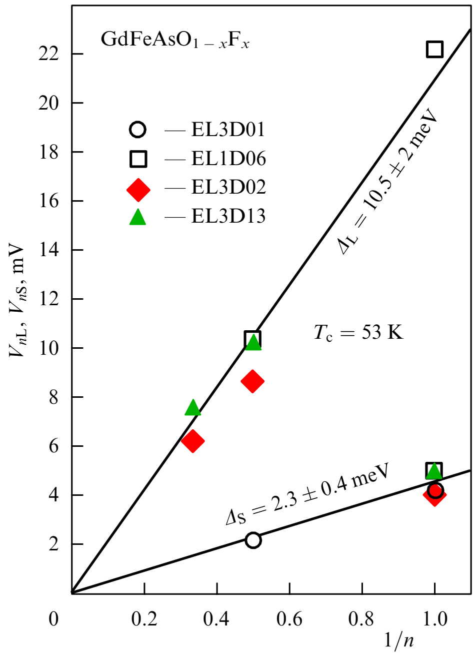

Figure 7. Measured characteristic voltages $V_n = 2\Delta_{\mathrm{L,S}}/en$ versus $1/n_{\mathrm{L,S}}$ for four SNS contacts prepared by the break-junction method at helium temperature [50].

By re-adjusting a contact in the same sample during the same low-temperature experiment, it was possible to observe various $I(V)$ characteristics with a series of features that correspond to a large or small gap, or even to both of them. Because of the fine crystalline structure of the sample, different microcontact representations gave uncorrelated gap values and characterized the local properties of the superconducting phase at different points. Figure 7 summarizes the results from a large number of microcontacts. It can be seen that the measured values of the voltages V_n fit fairly well into the two linear dependences on $1/n_{\mathrm{L,S}}$. The obtained data therefore suggest that superconducting $\mathrm{GdFeAsO_{0.88}(F_{0.12})}$ has two gaps with the energies $\Delta_{\mathrm{L}} = (10.5 \pm 2)$ and $\Delta_{\mathrm{S}} = (2.3 \pm 0.4)$ meV at $T = 4.2$ K.

Using the measured gap values and $T_{\mathrm{c}} = (52.5 \pm 1)$ K, we obtain an estimate for the ratio $2\Delta/k_{\mathrm{B}}T_{\mathrm{c}}$. For the large gap, it follows from our data that $2\Delta_{\mathrm{L}}/k_{\mathrm{B}}T_{\mathrm{c}} = (4.8 \pm 1.0)$, which is larger than the standard weak-coupling one-band BCS value 3.52; but this result is not inconsistent with the strong coupling regime in the BCS model. As regards the smaller gap, our measured ratio $2\Delta_{\mathrm{S}}/k_{\mathrm{B}}T_{\mathrm{c}}$ is ≈ 1, much less than the standard 3.52. This small value indicates that ‘weaker’ superconductivity at $T > T_{\mathrm{c}}^*$ (where T_{c}^* is the ‘intrinsic’ critical temperature of the weaker condensate in the absence of interband interaction) may be due to an internal reason, namely, the fact that two condensates in two regions of the Brillouin zone are close to one another in k-space, with the larger-gap condensate playing the ‘leadership’ role. It is commonly held that such a situation occurs, in particular, in $\mathrm{MgB_2}$ [48, 49] and $\mathrm{LaO_{0.9}F_{0.1}FeAs}$ [52].

The existence of a large superconducting gap on the scale of $2\Delta_{\rm L}/k_{\rm B}T_{\rm c} > 3.52$ in 1111 compounds *RE*OFeAs (*RE* = La, Sm, Nd) is confirmed by tunneling spectroscopy break-junction (BJ) measurements [53, 54], point contact Andreev reflection (PCAR) spectroscopy [38, 40, 55–60], scanning tunneling spectroscopy (STS) [39, 51], and angle-resolved photoelectron spectroscopy [61]. To date, the only reported measurement of the gap in Gd-1111 is that in Ref. [50]. Comparing gap measurements on Gd-1111 and other similar-$T_{\rm c}$ same-class superconductors (Sm-1111, Nd-1111, and Tb-1111) displays a fairly good agreement of the values of $2\Delta_{\rm L}/k_{\rm B}T_{\rm c}$ measured in Ref. [50] with STS data [39], BJ spectroscopy data [51], and with most PCAR measurements [40, 58–60]. There are, however, reports of gaps about twice as large as this [55, 56]. As regards the small gap, its published experimental values disagree even more strongly, by a factor of about three, as a comparison [50] showed. Moreover, a number of studies do not report the small gap at all, and Ref. [50] argues that a third gap, with an even smaller value about 1 meV, may exist. Therefore, both the symmetry of the order parameter and the width of the superconducting gaps in class *RE*-1111 superconductors remain questions for further experimental verification.

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