Paramagnetic resonance with conduction electrons in deformed lithium alloys

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Paramagnetic resonance with conduction electrons is used to investigate lithium alloys slightly doped with Zn, Cd, Al, Bi, and Sn. A dislocation contribution to the spin scattering of the conduction electrons is observed. The thermodynamic parameters of the dissolution of Al, Bi, and Sn in lithium and the binding energy of Sn and Al atoms with dislocations in the lithium are determined.

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INTRODUCTION

Spin-lattice relaxation of conduction electrons in metals is determined by various interactions. The principal relaxation mechanism in a pure metal is the modulation of the spin-orbit and Coulomb interactions by thermal lattice vibrations; the temperature dependence of the conduction-electron paramagnetic resonance (CEPR) is then described by a law similar to the Bloch-Gruneisen law for the electric resistance.[1] Introduction of dopants leads to a supplementary contribution to the CEPR line width, proportional to the dopant cross section, with the spin-scattering cross section independent of temperature (see, e.g., Ref. 2). An investigation of CEPR in a number of pure metals has shown that the presence of dislocations also leads to spin-lattice relaxation of the conduction electrons.[3]

Up to now the samples used in experiments on spin scattering of conduction electrons in metals contained only one type of defect (a definite type of dopant or dislocation). We report here detailed investigations of the singularities of spin resonance with conduction electrons in plastically deformed metallic alloys. The host metal was chosen to be lithium. It is known from studies of paramagnetic resonance with conduction electrons[4] that in lithium, under conditions of assured observation of the resonance signal (at line widths less than 100 G), that Mg, Zn, Ga, Ag, Cd, In, Au, Hg, Tl, and Pb have good solubility, Al, Pd, Sn and Pt have limited solubility, and the remaining metals are insoluble. Our trial measurements have shown that reliable reproducibility of CEPR results can be achieved if the temperature at which the alloy was produced is higher than the melting points of the individual components. The heating of lithium-based alloys to temperatures above 800 K entails a possible strong contamination of the samples by the crucible material. We chose therefore for our investigations the volatile dopants Zn, Cd, Al, Sn, and Bi, which have low enough melting points and are representatives of each of the indicated metal groups. The density of these dopants was adjusted in the course of the investigations. It was established that in an alloy with a dopant density less than the maximum solubility the line width is independent of temperature. The dislocation contribution is then observable only if the width of the resonance line is low and the dopant contributes to effective pinning of the dislocations, a high density of which can be achieved by plastic deformation of the sample. The line-width temperature dependence observed in a number of alloys made it possible to plot solubility curves for Al, Bi, and Sn in Li, to observe the effect of dislocations on the solubility of these elements, as well as to determine the binding energies of the Al and Sn with the dislocations in Li.[1)]

I. EXPERIMENTAL RESULTS

§1. Sample preparation

The host alloy was Li having 99.99 wt % purity, with room/helium temperature resistance ratio $R_{300}/R_{4.2} = 550$. The CEPR line width in the initial lithium, due mainly to residual impurities, was 2.5 G and was independent of temperature up to the melting point. This made it possible to separate unambiguously in the course of the measurements the contribution made to the resonance-line width by the volatile dopants. We prepared for the investigations nine binary lithium alloys with contents: 10^{-2} at. % Zn, $2\cdot10^{-3}$ at. % Cd, $2\cdot10^{-4}$ at. % Sn, $1.1\cdot10^{-2}$ at. % Sn, $1.6\cdot10^{-2}$ at. % Sn, $2\cdot10^{-2}$ at. % Sn, $2.9\cdot10^{-2}$ at. % Bi, 1.0 at. % Al, and 1.6 at. % Al. The purities of the dopants were: Zn – 99.99, Cd – 99.99, Al – 99.99, Bi – 99.9 and Sn—99.99%. The crucible material was niobium (99.7%) which is known[6] to be insoluble in Li up to ~800 °C.

Samples with impurity content less than 10^{-2} at.% were prepared by diluting in the lithium alloys of intermediate concentration (~0.5 at.%). Plates measuring 10×3×3 mm were cut from the obtained ingots and were thinned between steel rollers to thicknesses 0.5–1 mm. It must be noted that our experimental results offer evidence that the very cutting of the sample needed for the measurements produces in the metal a surface layer that contributes to the CEPR signal the same dislocation density as does a deformation (rolling) by 600–700%. It was therefore impossible to produce by our procedure samples with different dislocation densities.

The sample for the CEPR measurement was sealed in a quartz ampoule filled with mineral oil that was thoroughly dehydrated and outgassed beforehand by prolonged vacuum pumping and simultaneous heating. The entire sample preparation was performed in a container filled with high-purity helium gas. The entire alloy preparation procedure was checked out for "pure" lithium. The CEPR measurements have shown that the sample is not noticeably contaminated

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in any stage of preparation, and the employed method of sealing the samples in a measurement ampoule permits repeated heating of the sample to temperatures on the order of $T_{\text{melt}} = 459$ K without changing the width of the resonance line. The samples for the electron-microscopy experiments were obtained by rolling the alloys to a thickness 10–15 μm, which was subsequently decreased to ~ 1 μm by etching in a 50% solution of acetic acid in chloroform.

The electric resistance was measured on samples of the same kind as used for the CEPR investigations.

§2. CEPR measurements

The CEPR measurements were made with a BER-418s 3-cm-band spectrometer in the temperature range 400–470 K. The line shape in all the investigated samples had the asymmetric form usual for bulk metals; the g factor and the resonance-line width $\Delta H = (T_2)^{-1}$(γ is the gyromagnetic ratio, T_2 is the time of the spin-lattice relaxation of the conduction electrons) were determined by the standard procedure used in Ref. 7. The g factor of all the investigated samples was found to be 2.002 ± 0.001.

§3. Temperature dependence of CEPR line width

From the viewpoint of the line width measurements, all the alloys investigated can be divided into two groups. One contains the alloys in which the line width does not change with time. A feature of the samples of this group is that the line width is independent of temperature. The group includes "pure" Li with $\Delta H = (2.5 \pm 0.2)$ G (see Fig. 1), Li:Zn (10^{-2} at. %) with $\Delta H = (27 \pm 1.4)$ G, Li:Cd (2×10^{-3} at. %) with $\Delta H = (42 \pm 2)$ G, and Li:Sn (2×10^{-4} at. %) with $\Delta H = (4.7 \pm 0.3)$ G. In the latter, in contrast to the Li, Li:Zn and Li:Cd samples, in which the line width did not change after melting down and returning to room temperature, the line width decreased to $\Delta H = (3.5 \pm 0.2)$ G after melting down.

The second group includes alloys in which the line width depends on temperature. The time of establishment of an equilibrium line width is determined by the temperature, and in the alloy Li:Sn ($> 10^{-3}$ at. %) also by the prior history of the sample (by the type of heat treatment, if any). Figure 2 shows as an example the time dependence of the line width for the alloy Li:Sn (1.6×10–3 at. %). In the alloys of this group we observed furthermore a time dependence of ΔH. Figures 1, 3, and 4 show by way of example typical temperature dependences of the line widths in the samples Li:Bi, Li:Al, and Li:Sn.[27]

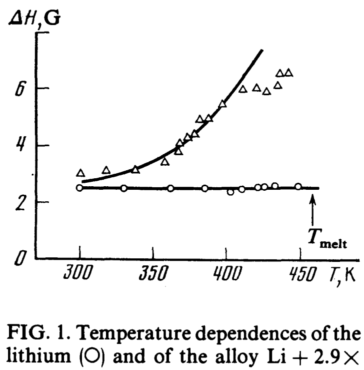

FIG. 1. Temperature dependences of the line widths of samples of "pure" lithium (○) and of the alloy Li + 2.9×10^{-2} at. % Bi (△) prepared 24 hours prior to measurement. The solid line for Bi is the result of calculation by Eq. (1).

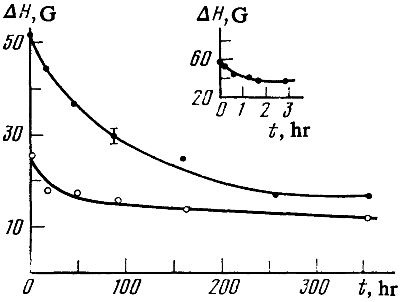

FIG. 2. Time dependence of the line width of the alloy Li + 1.6×10^{-2} at. % Sn quenched to room temperature from 445 (●) and 460 K (○). In the upper right corner is shown the time dependence of ΔH of the same alloy quenched from $T = 445$ to $T = 400$ K.

After repeated melting down of Li:Al and Li:Bi the measured temperature dependence agreed in the main with the variation of ΔH prior to the melting. A distinguishing feature of the samples of Li:Sn ($> 10^{-3}$ at. %) is that the temperature dependence of their line widths is irreversible. Thus, after raising the temperature to some value above 400 K and returning to room temperature, the line width becomes larger than the initial value and remains for a rather long time (see, e.g., Fig. 4) equal to the value at the peak point. If the temperature is raised again, the line width first decreases slightly, and then starts to increase at the peak point and duplicates the temperature dependence of the line width obtained for the sample with rising temperature. Plots a and b of Fig. 4 were obtained by cooling from 414 and 445 K, respectively. With increase of the peak temperature, the value of ΔH at $T = 300$ K increases, with a saturation value $\Delta H = 55$G at peak temperatures close to the melting point. After melting down the temperature dependence of the width of the resonance line changes somewhat (see Fig. 4) and becomes reversible.

In the alloy containing 1.1×10^{-2} at. % Sn the observed temperature dependence in the temperature region

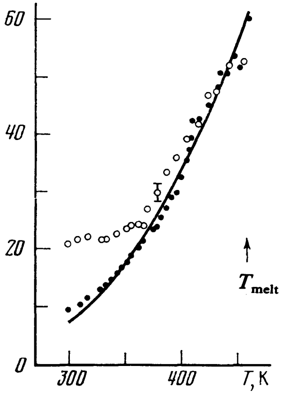

FIG. 3. Temperature dependence of the line width of the alloy Li + 1.6 at. % Al before melting (●) and after melting (○). Solid line— calculation by Eq. (2).

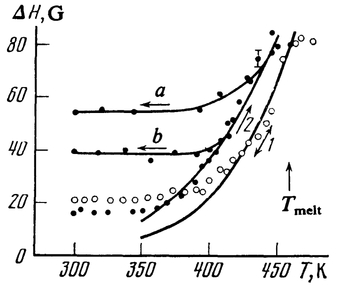

FIG. 4. Temperature dependence of the line width of the alloy Li + 1.6×10^{-2} at. % Sn before melting (●) and after melting (○); *a* and *b*—return of sample to the melting point from 414 and 445 K respectively. Solid lines 1 and 2—results of calculation by Eqs. (1) and (2), respectively.

above 440 K agrees with the corresponding dependence for the sample Li + 1.6×10^{-3} at. % Sn. With further increase of temperature, the line width did not change and remained equal to 65 G.

That the effects observed in Li:Sn are not due to surface oxidation is attested by electric-conductivity data and by the results of investigating the samples by transmission electron microscopy.

The behavior of the residual resistivity of an Li:Sn sample containing 1.6×10^{-2} at. % Sn was found to be similar at $T = 4.2$ K to the behavior of the CEPR line width. After rapidly heating the sample to $T = 445$ K, the residual resistivity increased from $\rho = 4.2\cdot10^{-8}$ Ω·cm to $\rho = 6.9\cdot10^{-8}$ Ω·cm; the two values decreased with time at approximately the same rates as ΔH.

Using a Tesla BS-500 electron microscope with cathode voltage 60 kV and magnification 26000 we observed in a sample having a tin density 1.6×10^{-2} at. % a fast dislocation motion found not to be due to local heating of the sample by the electron beam. For some samples we observed diffraction rings due to the small amount of the oxide Li_{20} on the surface. After rapidly heating the sample to a near-melting temperature we observed an immobile dislocation grid. Since the photographs did not contain distinct extinction contours, it was impossible to estimate the dislocation density.

II. DISCUSSION OF RESULTS

An investigation[8] of CEPR in colloidal samples of high-purity lithium has established that the intrinsic contribution (and apparently the contribution from equilibrium vacancies) to the resonant-line width in the temperature interval from 4.2 K to the melting point does not exceed 0.05 G. This means that the line width $\Delta H > 2$ G observed in the investigated samples is governed mainly by the spin relaxation of the conduction electrons on various defect types. It can be assumed that the main contribution to the scattering of the conduction electrons is made by impurities, dislocations, and extraneous-phase particles that can exist in alloys in which the dopant has limited solubility.

In the preparation procedure used by us strong deformation of the samples for the CEPR measurements is unavoidable. When the temperature dependence is investigated, raising the temperature can anneal the dislocations, and this should decrease the corresponding contribution to the CEPR line width. In doped samples, however, this process may require a rather long time, owing to dislocation pinning by the impurity atoms. The dislocation density can be greatly decreased by melting down the sample.

§1. Impurity solubility in lithium

As already noted, the line width due to the scattering of the conduction electrons by the impurities is proportional to the density of the impurities contained in the solid solution. One can thus expect the impurity part of the line width for alloys containing dopants in amounts not exceeding the equilibrium solubility[3)] to be independent of temperature. This case is realized in "pure" lithium and in alloys of Li:Zn and Li:Cd at impurity densities up to 10^{-2} at. %, i.e., at $\Delta H \ll 100$ G, when the CEPR is reliably observed. The available data on the solubility of Zn and Cd in Li agree with these results.

In freshly prepared alloys in which the doping component exceeds the equilibrium solubility, thermodynamic equilibrium sets in after a certain time lapse due to diffusion of the impurities from the solution into the dopant-rich phase, when the temperature of such alloys is raised the impurity should go over from the phase into the solution. It is known from the thermodynamics of the dissolution processes that the temperature dependence of the equilibrium density of impurities is given by (see, e.g., Ref. 10)

$$c_0=\exp(\Delta s/k)\exp(-\Delta h/kT), \tag{1}$$

where Δs is the decrease of the mixing entropy per atom from the ideal mixing entropy, Δh is the change of the enthalpy when one impurity atom goes into solution, and k is Boltzmann's constant. Since the CEPR line width is proportional to the impurity density in the solution, measurements of ΔH can yield unambiguously the density if one knows the parameter $d(\Delta H)/dc$ that characterizes the cross section for the conduction-electron scattering by the given impurity. In the alloys Li:Bi (2.9×10^{-2} at. %, Li:Al (1.6 at. %), and Li:Sn (1.6×10^{-2} at. %) the line width increases with rising temperature and reaches saturation near T_{melt}. At lower impurity density in the sample, the temperature dependence of ΔH saturates at lower temperatures and at smaller line widths. This seems to indicate that when the temperature reaches values at which ΔH ceases to depend on temperature, all the impurity contained in these alloys is already in the dissolved state, so that the value of $d(\Delta H)/dc$ can be determined. The second column of the table indicates these values for Bi, Al, and Sn in Li. It must be noted that from a comparison of the line widths at high and room temperatures it follows that the contribution of the dopant-rich phases to the spin scattering of the conduction electrons is not very substantial.

Expression (1) at the values of the constants $d(\Delta H)/dc$, $\Delta h/k$, and $\Delta s/k$ listed in the table can describe quite well the temperature dependences of ΔH in the alloys Li:Bi (Fig. 1), Li:Al (Fig. 3), and Li:Sn (Fig. 4), obtained after melting down the samples, at a sufficiently low dislocation density. The deviation from this law at $T < 380$ K is apparently due to the insufficiently rapid diffusion of the dopant atoms into the

TABLE I.

Sample	$d(\Delta H)/dc$ G/at.%	$\Delta s/k$	$\Delta h/k$, K	$c_0(T=300$ K), at.%	β/b exp eV	β/b teor, eV
Li + 1,6 at.% Al	36	$-0{,}08\pm0{,}08$	1840 ± 60	0,2	$\leqslant 0{,}19$	0,07
Li + $2{,}9\cdot10^{-2}$ at.% Bi	$1{,}2\cdot10^{2}$	$0{,}04\pm0{,}10$	3320 ± 150	$1{,}4\cdot10^{-3}$	–	–
Li + $1{,}6\cdot10^{-2}$ at.% Sn	$5{,}1\cdot10^{3}$	$0{,}0\pm0{,}3$	4060 ± 120	10^{-4}	0,34	0,07

matrix, so that thermodynamic equilibrium cannot be reached within the time between the measurements (~1 hr). The table lists also the room-temperature equilibrium solubilities of these impurities, which can be determined in accordance with Eq. (1) or after "aging" the samples at room temperature for one week. The values of $d(\Delta H)/dc$ listed in the table for Li:Al and Li:Sn are quite close to those obtained in Ref. 4. The data for Bi, in contrast to the opinion expressed in Ref. 4, give grounds for concluding that Bi has at room temperature at noticeable ($\sim 10^{-3}$ at. %) solubility in lithium.[4)] The value of $d(\Delta H)/dc$ is at the same time in qualitative agreement with the conclusions of Ref. 4 concerning the resonant character of the spin scattering of the conduction electrons by the impurities.

To conclude this section, we note that notwithstanding the existing analogy between the mechanisms of the spin and transport relaxations in metals, the data obtained by the CEPR method cannot be obtained in practice from resistivity measurements, since at high temperatures the electric properties are determined mainly by the phonon mechanism of conduction-electron scattering. Furthermore, only a joint analysis of the CEPR data and of the residual resistivity data permits a reliable determination of the change of the residual resistivity of lithium in which 1 at. % of impurity is dissolved. In particular, if the impurity density in the solution is determined from the CEPR line width, we obtain for Li:Sn $d\rho/dc = 5.5\cdot10^{-4}\ \Omega\cdot$cm. It should be noted that this property of the Li:Sn could not be determined even by most painstaking measurements of its resistivity.[12]

§2. Interaction of dislocations with impurities

The interaction of dislocations with impurities redistributes the impurities in the sample. Under thermodynamic equilibrium, impurity "Cottrell" clouds are produced near the dislocations and pin the dislocations.[13] This effect was observed in Li:Sn alloys by electron microscopy. Before the heat treatment, prior to the establishment of thermodynamic equilibrium in the sample, fast motion of the dislocations was observed. Electron-microscopy data on samples rapidly heated to $T = 440$ K attested to pinning of the dislocations.

The interaction of the impurities with dislocations can be of elastic, electrostatic, and chemical origin. It is known (see, e.g., Ref. 14) that the principal interaction in metals is due the difference between the sizes of the impurity and matrix atoms. In this case the dissolved impurities interact with the field of the elastic stresses of the dislocations. This interaction has a long-range character and in the case of low impurity densities the expression for the distribution of impurities around an edge dislocation is of the form 13

$$c=c_0\exp\left(-\frac{\beta}{r}\frac{\sin\theta}{kT}\right)$$
$$\beta=\frac{\mu b}{3\pi}\frac{1+\nu}{1-\nu}(v_s-v_a). \tag{2}$$

Here μ is the shear modulus, b is the dislocation Burgers vector, ν is the Poisson coefficient, v_s and v_a are respectively the atomic volumes of the impurity and of the matrix, and θ and r are the coordinates of the impurity in a cylindrical coordinate frame. Thus, the impurities become nonuniformly distributed in the sample in the presence of dislocations under thermodynamic equilibrium conditions.

If the sample contains a source of impurities (in our case, apparently, the Li_3Sn phase), additional solubility appears, due to the dislocations, and should lead to an additional impurity contribution to the CEPR line width. In this case the rate of the spin scattering of the conduction electrons by the impurities to be determined by the impurity density averaged over the entire volume of the sample, since the spin mean free path of the electrons is $l_s \approx 10^{-3}$ cm ($l_s = (2DT_2)^{-1/2}$, where $D = 14$ cm^2/sec is the coefficient of conduction-electron diffusion in lithium[15)] is much larger than the spatial scale 3×10^{-4} cm of the inhomogeneities (at $\rho_d \approx 10^{11}$ cm^{-2}). The excess impurity density in the sample can be calculated in the form

$$c_d=v_0\rho_d\int_{r_0}^{R} r\,dr\int_{0}^{2\pi}(c-c_0)\,d\theta, \tag{3}$$

where v_0 is the unit volume, $r_0 \sim b$, and $R \sim \rho_d^{-1/2}$.

If it is assumed that the cross section for spin scattering of the electrons by an impurity does not depend on its coordinates relative to the dislocation axis, then the additional sample linewidth contribution existing prior to the melting down can be sufficiently well described (see Fig. 4) by assuming a dislocation density $\rho_d = 1.6\cdot10^{11}$ cm^{-2} and a binding energy $\beta/b = (0.34 \pm 0.04)$ eV. Analysis of the results of the numerical calculation of expression (3) shows that a coordinate dependence of the electron scattering can lead mainly only to a change of ρ_d and hardly manifests itself in the binding energy. It must be noted that the calculation of the density c_d within the framework of linear elasticity theory includes also the region of the nucleus, although Hooke's law does not hold in the dislocation core and the binding energy can differ from $\beta/b = 0.34$ eV. The estimates show nevertheless that the change of the binding energy in the dislocation core does not influence the calculation result noticeably.

A similar additional impurity solubility should appear in the absence of dislocations also in the Li:Al alloy. This, however, has not been observed in experiment, and is probably evidence that the binding energy is low in this case. Recognizing that the line width is measured with a 5% error, it can be shown that the binding energy in Li:Al is $\beta/b \leqslant 0.19$ eV.

Our experimental results permit also an estimate of the binding energy of the Sn atoms with the dislocations in Li, by analyzing the time dependence of the CEPR line width. It must be borne in mind here that the irreversible temperature variation of ΔH (Fig. 4) in the samples prior to the melting-down cannot be due to an equilibrium process, for in accord with (3) the impurity density near the dislocations should decrease with decreasing temperature, as should also the bulk density c_0. The cause of the irreversibility of the temperature dependence of ΔH can be that with decreasing temperature the characteristic time of the precipitation of the impurities far from the dislocations into a tin-rich phase is much shorter than the corresponding time for impurities near dislocation lines. This conclusion agrees with data on the time dependence of ΔH (Fig. 2) for samples with high and low dislocation densities. It is natural to assume that the temperature dependence of the time needed for an impurity to leave the region of strong interaction with the dislocation can be described by an expression of the type

$$\tau=\tau_0 \exp\,(F/kT), \tag{4}$$

where the energy parameter $F \sim \beta/b$. To calculate τ_0, on the other hand, one must assume a concrete model of the transition of the impurity from the region near the dislocation into the phase. To describe this process, wherein the impurity passes many times through the region of strong interaction with the dislocations, we must know the sizes of the phase inclusions, the distances between them, the impurity diffusion coefficient, and others. If the temperature dependence of τ_0 is assumed weak, it can be obtained from the experimental data (Fig. 2) that at $T = 300$ K the impurity relaxation time is $\tau = 73$ hr (measurements of the residual resistivity yielded $\tau = 60$ hr), and at $T = 400$ K we have $\tau = 1.5$ hr. According to Eq. (4) we obtain $F = 0.4$ eV, in good agreement with the binding energy obtained from the temperature dependence of the CEPR line width.

We discuss now the obtained binding energies. We can estimate them roughly, knowing the difference between the atomic volumes in the model of hard spheres and voids.[14] It was impossible to determine reliably by x-ray methods the change of the volume of the alloy per dissolved atom. On the other hand, using the tabulated atomic radii and disregarding the fact that the elastic constants of the impurity and of the matrix are different, we can obtain for the binding energy the values listed in the table. From a comparison of these estimates with experiment it can be seen that the agreement can be better for Li:Al than for Li:Sn. This is not surprising, since it is known that the model employed is suitable only when the alloy components have close valences as, e.g., in Li:Zn and Li:Cd, for which the estimated binding energies are 0.07 and 0.0003 eV, respectively. When estimating the binding energy in the opposite case, however, it is apparently necessary to take into account the strong local perturbation of the electronic states near the impurity.

§3. Spin scattering of conduction electrons by dislocations

The width $\Delta H = 2.5$ G observed in the "pure" lithium samples investigated by us may, generally speaking, be due to conduction-electron scattering by dislocations in by uncontrollable impurities. The dislocation contribution, however which should decrease abruptly after remelting the sample in the measuring ampoule and returning to room temperature, could not be observed. This seems to indicate that rolling of pure lithium samples at room temperature cannot produce a dislocation density high enough to be observable in CEPR, in view of the high mobility and rapid annihilation of the dislocations. A similar situation was observed in Li:Zn and Li:Cd alloys.

The only indication that conduction-electron scattering by dislocations in lithium can make a perceptible contribution to the line width is provided, in our opinion, by the results of CEPR measurements in Li:Sn (2×10^{-4} at. %). In this sample, after melting down and returning to room temperature, the line width decreases to 1.2 G. The dislocation contribution can apparently be separated in this alloy because the dislocation density attainable in alloys containing an impurity component with a high binding energy is higher than in the pure metal and in the other investigated compounds deformed in like manner.

Direct methods (with the aid of transmission electron microscopy) could not determine reliably the dislocation density ρ_d in our samples. If, however, we use the value $\rho_d = 1.6\cdot10^{11}$ cm^{-3} obtained by us in the discussion of the temperature dependence of ΔH in Li:Sn (1.6×10^{-2} at. %), a rough estimate of the dislocation contribution to the line width per unit dislocation length yields a value $\sim 0.8\times10^{-11}$ G/cm. As for the alloys Li:Bi, Li:Al, and Li:Sn ($>10^{-3}$ at. %), it was impossible to separate in them the dislocation contribution because of the large CEPR line widths, which furthermore depend on the time.

The authors are grateful to the late E. G. Kharakhash'yan and to G. G. Khalilullin for a helpful discussion of the results.

[1)] Preliminary results were published in Ref. 5.

[2)] The CEPR measurements were made 24 hours after the sample preparation; the second measurements after melting down were made after one hour.

[3)] It can be seen from binary phase diagrams[9] that Cd has high solubility in solid lithium, and Zn and Al have a perceptible one (1–3 at. % at the eutectic temperature); for the remaining elements binary diagrams with lithium are either nonexistent or cannot be used to assess the solubility.

[4)] We mention for comparison only liquid bismuth is soluble in Na (Ref. 11).

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