An investigation of the isotope effect in $SnMo_6S_8$

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The dependence of T_c for the compound $SnMo_6S_8$ on the isotopic mass of Sn and Mo has been investigated. It was found that T_c decreases with increasing M_{Mo} (the normal isotope effect) but increases with increasing M_{Sn} (the anomalous isotope effect). The values of T_c correlate with the values of the derivative of the resistivity $\partial\rho/\partial T$ at high temperatures. Application of a hydrostatic pressure of ~ 7 kbar leads to a decrease in the "anomaly" of the isotope effect with respect to tin. The results are ascribed to the peculiarities of the $SnMo_6S_8$ phonon spectrum associated with the cluster nature of the compound.

It is well known that the discovery of the isotope effect—the dependence of the superconducting transition temperature T_c on isotopic mass M—provided the key to an understanding of the mechanism of superconductivity. According to the BCS theory, the exponent β in the expression $T_c = \text{const}\cdot M^{\beta}$ is determined by the reduction in the average phonon frequency $\langle\omega\rangle$ with an increase in isotopic mass M, and since $\langle\omega\rangle \sim 1/M^{1/2}$ for ordinary phonon spectra, then $\beta = -0.5$ (Ref. 1). This conclusion agreed well with experimental results on a number of simple metals (Cd, Tl, Hg, Sn, Pb). However, a weaker reduction in T_c with increasing M was observed for most of the transition metals, and $\beta \approx 0$ for some (Ru, Zr). The small value of β for transition metals was explained by an increase in the role of Coulomb interaction between electrons, due to the existence of a narrow d band.[2] More realistic calculations of the isotope effect according to the McMillan formula[3] lead to a similar conclusion.

The anomalous sign of the isotope effect, i.e., the increase in T_c with increasing isotopic mass, was first observed in α-U. It was reported in the only work published[4] that $T_c(\alpha\text{-U}) \sim M^{2}$. It should, however, be noted that α-U only becomes superconducting at pressures $P > 2$–3 kbar, so that this result was obtained at $P \approx 11$ kbar. Besides, the masses of the isotopes studied, ^{235}U and ^{238}U, differ by only 1.3%.

More reliable results on the anomalous isotope effect were obtained on palladium hydrides. According to Stritzker and Buckel[5] and Skoskiewicz *et al.*,[6] $T_c(\text{PdH}) \approx 9$ K and $T_c(\text{PdD}) \approx 11$ K. To explain this effect, suggestions were advanced about changes in either the electronic[7] or phonon[8] spectra, and also suggestions of a simultaneous change in the electronic and phonon characteristics of the palladium hydrides.

We reported early on the finding of an anomalous sign for the isotope effect with respect to tin in the compound $SnMo_6S_8$, while the effect with respect to molybdenum in the same compound had the normal sign.[9] It was suggested there that the main reason leading to the reverse sign of the isotope effect with respect to tin was the large anharmonicity in the vibrations of the tin atoms, which was confirmed by preliminary experiments on the effect of pressure on the isotope effect in $SnMo_6S_8$.

In the present work we give the results of further studies of the anomaly which was found, including the effect on it of hydrostatic pressure. In addition, the effect of the mass of the tin atoms on the temperature dependence of the electrical resistivity $SnMo_6S_8$, over a wide temperature range, was determined. The results can be explained by the features of the phonon spectrum of $SnMo_6S_8$, related to the cluster character of the compound studied. Since studies of the effect of pressure on the normal isotope effect in simple metals had not been carried out earlier, we made such measurements on the ^{112}Sn and ^{124}Sn isotopes of white tin. The results of these measurements are compared with those obtained for $SnMo_6S_8$.

THE EXPERIMENTS

The reproducibility of results when studying the isotope effect in compounds is mainly determined by the technology of preparation of the specimens. Ternary molybdenum chalcogenides have a fairly broad homogeneous region, and their critical superconducting transition temperatures depend both on the composition of the charge and on the conditions of synthesis and subsequent annealing.

The first stage in preparing the specimens studied in the present work was direct synthesis from the elements in a quartz ampule filled with pure helium gas. The characteristics of the monoisotopic components used for preparing the specimens are shown in Table I. After synthesis, the specimens were ground in an agate mortar and then pressed under a load of 15–30 ton on 2 mm diameter cylinders, 5–7 mm long. Subsequent annealing was carried out in doubly sealed quartz ampules, filled with helium gas at a pressure of 200

TABLE I. Certified data on Sn and Mo isotopes used for preparing $SnMo_6S_8$ specimens.

Isotope	Actual atomic weight of the isotope	Fe content, wt.%
^{112}Sn	113,2	<0,007
^{124}Sn	123,9	<0,008
^{92}Mo	92,3	<0,019
^{100}Mo	99,9	<0,005

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mm Hg (at room temperature). The annealing temperature was 1000 °C, the usual annealing time was 24 h, the longest 100 h. Five specimens of ordinary non-monoisotopic components were prepared and the normal single anneal was carried out to determine the effect of the preparation conditions on T_c for $SnMo_6S_8$. The width of the measured superconducting transitions was 0.2–0.3 K, and the maximum departure of T_c from the mean value was not more than 0.07 K. Unfortunately, when measuring several series of specimens with monoisotopic components, the scatter of the experimental values of T_c was noticeably larger. This scatter could be produced, for example, by the less definite composition of the charge of the monoisotopic specimens due to the necessity of using small amounts of the starting components. We used the results of Sergent *et al.*[10] to overcome the effect of indeterminate composition on T_c; they found that a high-temperature long-duration anneal of compounds of composition $M_x PbMo_6S_8$ led to approximately one and the same value of T_c, almost independent of the initial composition of the specimens. The experiments which we carried out showed that the scattering in T_c could also be reduced, for monoisotopic specimens with composition $SnMo_6S_8$, by carrying out an anneal of long duration or several repeated ordinary anneals, and a unique dependence of T_c on isotopic mass could be obtained. In all, 9 series of single-crystal specimens were prepared and studied (the total number of specimens was 24). In general, the specimens of each series first underwent an ordinary anneal and then either several ordinary and one long-duration anneal, or only one long-duration anneal. Measurements of T_c were carried out after each thermal treatment. After the end of the measurements, x-ray pictures were taken of 4 monoisotopic specimens, and the following lattice parameters were obtained: $a = 9.186 \pm 0.003$ Å, $c = 11.38 \pm 0.01$ Å.

The critical temperatures T_c of the specimens prepared were determined by measuring the resistance, using the usual four-probe method. The pressed current and potential leads were made of bronze wire. The specimens were stuck to a copper thermal block through tin cigarette paper or mica, its temperature being measured by a TGS-2 germanium thermometer, calibrated to an accuracy of 0.05 K. Two specimens were measured simultaneously. The voltages taken from the potential contacts in the specimens were fed to photoelectric amplifiers and then to digital voltmeters. The thermometer voltage was fed directly to a digital voltmeter. The output signals from the voltmeters were punched on a tape and analyzed with a computer. The superconducting transition curves were recorded on a two-coordinate, two-pen chart recorder to check on the measurements. The critical temperature was determined as the center of the superconducting transition curve.

Measurements of the effect of hydrostatic pressure on the isotope effect in $SnMo_6S_8$ were caried out in a high-pressure bomb with an 8 mm diameter inner channel, made of beryllium bronze. T_c for two specimens $^{112}SnMo_6S_8$ and $^{124}SnMo_6S_8$ were measured simultaneously. Electrical contact of the current and potential leads to the specimens was achieved with the help of an In–Ga eutectic; the contacts were covered with a nitrocellulose adhesive for mechanical binding. The pressure was measured with a manganin gauge and the temperature from readings of a Cu–Cu + 0.01% Fe thermocouple, inserted into the bomb.

Measurements of the temperature dependence of the electrical resistance from 5 to 300 K were carried out in a metal cryostat with a temperature regulator. Temperature control was achieved with a Cu–Au + 0.07% Fe thermocouple. The results of the measurements were punched on tape. The current through the specimens was reversed automatically to get rid of spurious thermal emf's. When analyzing the experimental results with a computer, the measured values of the resistance of the specimens was averaged over a 1 K temperature interval. For each temperature interval there were, on average, 5 experimental points.

Measurements of T_c for two white tin isotopes ^{112}Sn and ^{124}Sn were carried out in the high-pressure bomb by the inductance method. For this purpose the specimens were given the shape of 0.8 mm diameter cylinders, 8 mm in length, which were housed inside two pickup coils, placed side to side, consisting of 780 turns of PÉL-0.5 wire. The pickup coils were connected to the input of an amplifier with a phase-sensitive detector. A small coil wound on the bomb case provided the 0.3 Oe amplitude modulating magnetic field. Temperature was controlled by a Cu–Cu + 0.01% Fe thermocouple, let into the bomb, and by ^4He vapor pressure. Measurements of the signal from the pickup coils were made while slowly lowering the temperature of the helium bath. While the temperature was above T_c for both specimens the signal from the pickup coils was then zero; the transition of the specimen with the larger T_c into the superconducting state led to the appearance of an out-of-balance signal, which disappeared on the transition of the second specimen into the superconducting state. An example of such a trace is given in Fig. 1.

RESULTS

The results obtained on the effect of the isotopic mass of tin and molybdenum on T_c in the compound $SnMo_6S_8$ are shown in Table II. Although the results for different series of $^{112}SnMo_6S_8$ and $^{124}SnMo_6S_8$ specimens differ somewhat among themselves, the results for all the series of specimens

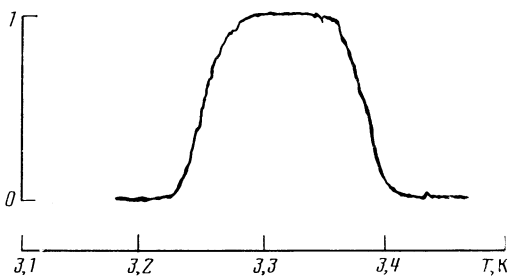

FIG. 1. Trace of the superconducting transition of isotopic specimens of metallic tin ^{112}Sn and ^{124}Sn at $P = 8.56$ kbar.

TABLE II. Critical temperatures of $SnMo_6S_8$ specimens with monoisotopic components.

Anneal	Series 1		2		3		4	
	^{112}Sn	^{124}Sn	^{112}Sn	^{124}Sn	^{112}Sn	^{124}Sn	^{112}Sn	^{124}Sn
1	11,91	12,13	12,48	12,44	12,08	12,54	12,10	11,89
2			11,60	11,92			12,71	12,14
3							12,67	12,45
4							13,41 *	13,80 *
ΔT_c	+0,22		+0,32		+0,46		+0,39	

Anneal	5		6		7		8		9	
	^{112}Sn	^{124}Sn	^{112}Sn	^{124}Sn	^{112}Sn	^{124}Sn	^{92}Mo	^{100}Mo	^{92}Mo	^{100}Mo
1	12,24	12,16	11,43	11,62	11,36	11,40	12,55	12,28	12,43	12,16
2	12,19	12,13	11,56 *	11,68 *	11,52 *	11,67 *				
3	13,01 *	13,32 *								
ΔT_c	+0,31		+0,12		+0,15		−0,27		−0,27	

Note: *After long-duration anneal.

which have undergone a long-duration anneal enable one to state that T_c ($^{124}SnMo_6S_8$) is higher than T_c ($^{112}SnMo_6S_8$). The average value $\langle\beta\rangle_{Sn} = +0.23 \pm 0.04$, i.e., an anomalous isotope effect with respect to tin is observed in the compound $SnMo_6S_8$. On the other hand, results obtained for the specimens $Sn^{92}Mo_6S_8$ and $Sn^{100}Mo_6S_8$ show that the isotope effect with respect to molybdenum in this compound has the normal sign and $\langle\beta\rangle_{Mo} = -0.5 \pm 0.1$.

The temperature dependences of electrical resistance were measured on specimens of compositions $^{112}SnMo_6S_9$ and $^{124}SnMo_6S_8$. Examples of such curves are shown in Fig. 2. A linear increase in resistivity is observed as the temperature is raised immediately above the superconducting transition. A gradual slowing down in the growth in resistivity takes place in the range 90–150 K, and at $T > 200$ K the $\rho(T)$ relation again becomes linear. The values of the derivative $\partial\rho/\partial T$ at high temperatures correlate with the values of T_c: the larger T_c, the larger $\partial\rho/\partial T$.

The results of studying the effect of pressure on the isotope effect in $SnMo_6S_8$ are shown in Table II. The "anomaly" of the isotope effect with respect to tin in this compound decreases with increasing pressure. If the results obtained are extrapolated to the pressure P_0, at which $\langle\beta\rangle_{Sn} = 0$, then it turns out that $P_0 \approx 30$ kbar.

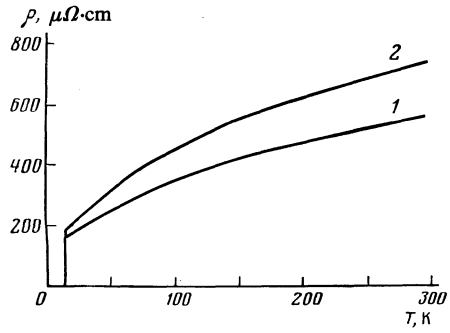

FIG. 2. Temperature dependences of the resistance of specimens of 1) $^{112}SnMo_6S_8$ and 2) $^{124}SnMo_6S_8$.

For comparison, the effect of pressure on the normal isotope effect in white tin was studied. It can be seen from Fig. 3 that in this case there is no influence of pressure on the isotope effect.

DISCUSSION

The main feature determining many physical characteristics of ternary molybdenum chalcogenides is their complicated phonon spectrum. The complexity of the phonon spectrum is associated with properties of the crystal structures of these compounds: Mo_6S_8 groups form compact clusters, between which the atoms of the third component, which are relatively weakly bound to them, are positioned. The phonon spectrum of these compounds can therefore be represented as consisting of three branches: the usual Debye branch, the intracluster vibration branch, and the branch of vibrations of the atoms of the third component. Because of the large difference between the masses of the Mo_6S_8 clusters and of the atoms of the third component, and also the weak bonding between them, the branches of the vibrations of the atoms of the third component degenerate into a local mode of strongly anharmonic and anisotropic optical vibrations. Such ideas about the phonon spectrum are confirmed by

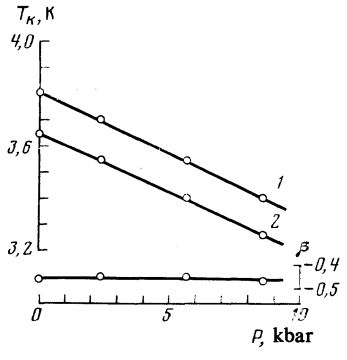

FIG. 3. The effect of pressure on the isotope effect in metallic tin: 1) T_c (^{112}Sn), 2) T_c (^{124}Sn).

neutron scattering results[11] and by results of studying the Mössbauer effect,[12] and explain the measured specific heat[13] and the linear variation of the electrical resistivity at low temperatures.[14]

The experimental results of the present work and calculations made by us show that the features of the isotope effect in $SnMo_6S_8$ are also determined to a significant extent by the nature of the phonon spectrum of this compound.

The superconducting transition temperature in the case of superconductors with tight binding, to which $SnMo_6S_8$ belongs, can be calculated by the McMillan formula[15]

$$T_c = \frac{\langle\omega\rangle}{1{,}2}\exp\left[-\frac{1{,}04(1+\lambda)}{\lambda-\mu^*(1+0.62\lambda)}\right], \quad (1)$$

where λ is the electron-phonon interaction parameter, determined by the relation

$$\lambda=\eta/M\langle\omega^2\rangle, \quad (2)$$

μ^* is the effective Coulomb interaction between the electrons. The terms $\langle\omega\rangle$ and $\langle\omega^2\rangle$ which occur in Eqs. (1) and (2) are determined by averaging over the phonon spectrum

$$\langle\omega\rangle=\frac{\int\alpha^2(\omega)F(\omega)\,d\omega}{\int\omega^{-1}\alpha^2(\omega)F(\omega)\,d\omega}, \quad \langle\omega^2\rangle=\frac{\int\omega\alpha^2(\omega)F(\omega)\,d\omega}{\int\omega^{-1}\alpha^2(\omega)F(\omega)\,d\omega}. \quad (3)$$

The exponent β in the expression for the isotope effect $T_c = \text{const}\cdot M^{\beta}$ is given by the derivative of Eq. (1) with respect to mass

$$\beta=\frac{\partial\ln\langle\omega\rangle}{\partial\ln M}+\frac{1.04(1+0.38\mu^*)\lambda}{[\lambda-\mu^*(1+0.62\lambda)]^2}\frac{\partial\ln\lambda}{\partial\ln M}. \quad (4)$$

Since for ordinary spectra $\langle\omega^2\rangle\sim 1/M$, the changes in atomic mass M and in $\langle\omega^2\rangle$ cancel one another out in the expression for λ, and $\partial\lambda/\partial M = 0$. The isotope effect is thus determined by only the first term in Eq. (4), giving $\beta = -0.5$. Such a cancellation may not occur for complicated phonon spectra and if the change in $\langle\omega^2\rangle$ exceeds the change in M, then an anomalous sign for the isotope effect should be observed.[1] Calculations show that just such a situation can occur in the compound $SnMo_6S_8$.

It was noted above that the phonon spectrum of $SnMo_6S_8$ can be represented as the sum of three contributions:

$$F(\omega)=k_EF_E(\omega)+k_DF_D(\omega)+k_IF_I(\omega), \quad (5)$$

where $F_E(\omega)=\delta(\omega-\omega_E)$ is the Einstein mode associated with vibrations of the tin atoms,

$$F_D(\omega)=\begin{cases}3\omega^2/\omega_D^3, & 0\leqslant\omega\leqslant\omega_D,\\ 0, & \omega>\omega_D,\end{cases}$$

is the Debye branch, $F_I(\omega)$ is the contribution of intracluster vibrations, k_E, k_D and k_I are weighting factors.

In carrying out the calculations we did not take account of the contribution of intracluster vibrations in $F(\omega)$. Such a neglect can be partly justified by the fact that the characteristic frequencies of intracluster vibrations lie much higher than ω_D and ω_E. The values of the remaining numerical parameters of the phonon spectrum of $SnMo_6S_8$ were given by the following: $\omega_E = 100$ K, $\omega_D = 150$ K, $k_E = 2k_D$. With such a choice of parameters, the mean phonon frequency $\langle\omega\rangle = 100$ K, which agrees well with a calculation of $\langle\omega\rangle$ from the results of measurements of the Mössbauer effect.[16] In addition, we put $\alpha^2(\omega)=\text{const}$, $\mu^*=0.15$, $\omega_E\sim 1/M_{Sn}^{1/2}$, $\omega_D\sim 1/(6M_{Mo}+8M_S)^{1/2}$. The results of the calculations and the experimental values of $\langle\beta\rangle$ are given in Table IV. It can be seen that the calculations of β agree qualitatively with the experimental results. The quantitative agreement could evidently be improved appreciably if account were taken of the change in Coulomb interaction with the isotopic mass.[2]

It follows from Table IV that the anomalous sign of the isotope effect with respect to tin is determined by the depedence of the electron-phonon interaction constant on isotopic mass. This conclusion is confirmed by the results of measurements of the temperature variation of electrical resistance. According to Allen,[17] at high temperatures

$$\partial\rho/\partial T=\frac{\pi k_B}{e^2N(0)\langle v_F^2\rangle}\lambda. \quad (6)$$

By using the literature values for $N(0)$, $\langle v_F^2\rangle$ and λ (Refs. 13, 18), we obtain $\partial\rho/\partial T\approx 1.2\ \mu\Omega\cdot\text{cm}\cdot\text{K}^{-1}$, which agrees well with the experiment (Fig. 2). The experimental results on $\partial\rho/\partial T$ obtained for specimens with different isotopic content can, therefore, be used for an additional calculation of $\partial\ln\lambda/\partial\ln M$. The measured values of $\partial\rho/\partial T$ are 1.22 and $1.34\ \mu\Omega\cdot\text{cm}\cdot\text{K}^{-1}$ respectively for the specimens $^{112}SnMo_6S_8$ and $^{124}SnMo_6S_8$, which gives $\partial\ln\lambda/\partial\ln M\approx 0.9$. If this value is used to calculate the isotope effect with respect to tin, we obtain $\beta_{Sn}\approx +0.4$, which is 1.5–2 times greater than is measured. The agreement obtained should be recognized as completely satisfactory, bearing in mind the simplified nature of the calculation and the relatively low accuracy in determining $\partial\rho/\partial T$. Besides, it is known that the electron-phonon intraction constants λ, which enter Eqs. (1) and (6), can in general differ somewhat due to the influence of anisotropy of the Fermi surface and of the phonon spectrum.

The results of measurements of the isotope effect and of

TABLE III. The effect of pressure on the critical temperatures of $^{112}SnMo_6S_8$ and $^{124}SnMo_6S_8$ specimens.

	$P=0$	$P=6{,}9$ kbar	$\partial T_c/\partial P$, kbar^{-1}
$^{112}SnMo_6S_8$	12,50	12,19	$-4{,}5\cdot10^{-5}$
$^{124}SnMo_6S_8$	12,78	12,41	$-5{,}4\cdot10^{-5}$
ΔT_c, K	0,28	0,22	

TABLE IV. Calculation of the isotope effect in $SnMo_6S_8$.

Isotope	$\partial \ln \langle\omega\rangle / \partial \ln M$	$\partial \ln \lambda / \partial \ln M$	β	$\langle\beta\rangle_{exp}$
Sn	−0,333	+0,452	+0,060	+0,23±0,04
Mo	−0,115	−0,312	−0,386	−0,5±0,1

the temperature variation of the resistivity are thus in fair agreement and can be explained by the existence in the phonon spectrum of $SnMo_6S_8$ of a local mode of strongly anharmonic optical vibrations of the tin atoms. A reduction in the distance between the tin atoms and the Mo_6S_8 clusters should smear out this feature in the phonon spectrum of $SnMo_6S_8$ and lead to a reduction in the anomalous character of the isotope effect with respect to tin. This suggestion is confirmed by the results of studying the effect of pressure on the anomalous isotope effect in $SnMo_6S_8$ (Table III). An indirect indication of the validity of such a point of view is the pressure independence of the value of the normal isotope effect in metallic tin (Fig. 3). This last result is in agreement with theoretical calculations,[19] according to which the change of β at $P = 10$ kbar for metallic tin should not exceed 0.0014.

As has already been noted,[9] the studies of the isotope effect with respect to tin in $SnMo_6S_8$ carried out by Culetto[20] and Pobell[21] showed, according to the present authors, that the isotope effect with respect to tin in this compound is equal to zero. This result can, evidently be regarded as the lower limit of the anomalous isotope effect found by us.

We are grateful to Yu. A. Deniskin for help in preparing the specimens and in carrying out the measurements.

[1)]Loss of proportionality of $\langle\omega^2\rangle$ and $1/M$ can be considered as a manifestation of anharmonicity.

[2)]However, the Coulomb interaction by itself cannot lead to the opposite sign of the isotope effect.

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