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Isomorphic phase transitions in CuCl at high pressures

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The T-P phase diagram of CuCl was investigated in the temperature interval 4.2–800°K and at pressures $\lesssim 35$ kbar. Isomorphic phase transitions are observed with small low thermal ($\Delta Q \leq 0.1$ kcal/mole) and volume ($\Delta V/V_0 \leq 1.5\%$) effects in the dielectric phase. The obtained T-P diagram of CuCl is explained within the framework of the already existing theoretical concepts.

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INTRODUCTION

Copper halides with sphalerite structure have a number of anomalous physical properties when compared with the properties of all other tetrahedral compounds (II–VI or III–V compounds and covalent crystals). For example, the CuCl crystal has an anomalously small bulk modulus.[1,2] The well known Lyddane–Sachs–Teller (LST) relation does not hold for CuCl.[2] The anomalies of the properties of copper halides become most pronounced, however, at high pressures. It is known[3] that in covalent crystals and in III–V compounds with small asymmetry of the pseudopotentials, pressure produces a dielectric–metal phase transition with tetragonal symmetry of the β-Sn type. This transition is attributed to the overlap, under pressure, of the maximum of the valence band at the Brillouin Γ point and the bottom of the conduction band in the vicinity of the X point, i.e., under pressure, the indirect energy gap $E_{\Gamma X} \to 0$. In II–VI compounds and in the most ionic of the III–V compounds, a transition to the NaCl structure takes place under pressure. Nonetheless, instead of the expected[3] phase transition into the NaCl-type structure under pressure, what is produced in CuCl is first a dielectric–metal transition without change of lattice symmetry, followed by a transition into a structure of the β-Sn type, and only at higher pressure ($P > 100$ kbar) does a transition take place into a dielectric phase with a structure of the NaCl type.[2,4,5]

It was shown[2] that all the known anomalies of the properties of CuCl can be explained if one assumes the presence of a small indirect gap between the maximum of the valence band at the point Γ and the minimum of the conduction band at the point X ($E_{\Gamma X} \approx 0.3$ eV). This model of the band structure is confirmed by the experimental dependence of the resistivity on the temperature,[6] by calculations of the band structure, and by measurements of the optical absorption of CuCl crystals.[7] Owing to the large ratio of the effective masses of the holes and electrons, and also because of the hindrance rules for transitions in the vicinity of the X point of the Brillouin zone, the gap $E_{\Gamma X}$ is difficult to observe by standard optical measurements on thin CuCl films.[2] Inasmuch as the experimental binding energies of the direct excitons is $E^d \approx 0.2$ eV[8] such a dielectric state with $E^d \approx E_{\Gamma X} \ll E_D$ (direct gap between the maximum of the valence band and the minimum of the conduction band at the point Γ) can be unstable with respect to electron-hole pairing, i.e., with respect to a phase transition into the state of an excitonic dielectric. The physical properties of the excitonic dielectric CuCl should exhibit many anomalies. Owing to the polarization of the electron-hole pairs, the LST relation is violated.[2] Owing to the interaction of the exciton (two-particle) and phonon branches in the spectrum of the elementary excitations, the speed of sound decreases sharply,[9] and with it the bulk modulus of the CuCl crystal. At an energy $E = 0.358$ eV,[7] an additional absorption band appears and corresponds to single-particle excitations in the exciton system.

Anomalies arise in the short-wave region of the spectrum.[10] With the increasing pressure, the indirect gap $E_{\Gamma X}$ for the nonrestructured electron spectrum decreases. As a result, an insulator-metal transition takes place at $P = 42$ kbar without a change in the lattice symmetry[5] and with a small volume effect. In addition to these experimental observed effects, the theory predicts a number of additional effects, which have not yet been observed in the case of CuCl. Thus, it follows from the theory of Guseĭnov and Keldysh[11] that a phase transition from a dielectric into an excitonic dielectric is of first order with a small volume effect. Consequently, if the CuCl crystal is indeed an excitonic dielectric, then isomorphic phase transitions with very small volume effect $\Delta V/V_0$ should be observed on the T–P phase diagram in the dielectric region. To check on this possibility it is necessary to study in detail the T–P diagrams in a wide range of temperatures and

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pressures; this was the purpose of the present investigation.

EXPERIMENTAL PART

We investigated the $T-P$ diagram of CuCl in the temperature interval 4.2–800 K and up to 35 kbar. The measurements were made on transparent colorless crystals with sphalerite structure, as verified by x-ray diffraction and by observation in polarized light. The method used to obtain the crystals was analogous to that employed in[6]. In the temperature interval 300–800 °K, the measurements were performed in a hydrostatic chamber up to pressures on the order of 24 kbar, using the techniques of differential thermal analysis (DTA) and dilatometry. The presure-transmitting medium was organosilicon oil, which was subjected to special heat treatment to prevent its decomposition at high temperature. The pressure was measured with a manganin resistance manometer calibrated against a piston manometer at the All-Union Research Institute for Physicotechnical and Radiotechnical Measurements. The pressure-measurement error did not exceed ± 150 bar. In the DTA method we used cylindrical samples of 3–4 mm diameter and 10–12 mm height. The standards were NaCl crystals with the same dimensions. To measure the temperature we used chromel-alumel thermocouples. The error in the determination of the transition temperature depended on the rate of heating (in the DTA case) and amounted to ± 2–3° at a heating rate 10–20 deg/min and ± 5–6° at a heating rate 70–80 deg/min. The rates of heating and cooling used by us (in the DTA and in the dilatometry) depended on the transition temperatures and amounted to 10–30 deg/min when transitions at 290–570 °K were determined, and 30–70 deg/min at transition temperatures 570–800 °K. The limits of the transitions with large thermal effects CuCl (I $\rightleftarrows$ II), CuCl (I $\rightleftarrows$ III) and the melting curve (Fig. 1) were measured with a smaller error, ± 3°, then the limits of the transitions with small thermal effect CuCl II (1 $\rightleftarrows$ 2), CuCl II (2 $\rightleftarrows$ 3), CuCl II (1 $\rightleftarrows$ 3), since the transition temperature could be determined by using the readings of either an absolute or a differential thermocouple. The DTA procedure used by us made it possible to record thermal effects in which the sample temperature changed by 0.05° and more. To record changes in the linear dimensions of the sample (with changing temperature and pressure) we used a dilatometer with an inductive pickup, which made it possible to register transitions with a volume effect $\Delta V/V_0 \geq 0.05\%$. The dimensions and shapes of the samples used for the dilatometry were the same as in DTA. The error in the determination of the transition temperatures did not exceed ± 3°.

Investigations at $T = 4.2$–300 K were carried out up to $P = 35$ kbar in a chamber of the piston-cylinder type,[14] using graphite as the pressure-transmitting medium. The apparatus made it possible to carry out dilatometric measurements with variation of the pressure applied to the sample. In addition, a system of measuring coil, connected in a differential-transformer circuit, made it possible to register the magnetic-permeability changes occurring in superconducting or magnetic transitions.[14] The pressure in the chamber was calibrated against the phase transitions in Bi, RbI, and KCl. The error in the measurement of the relative elongation of the sample $\Delta l/l_0$ with our apparatus did not exceed 0.1%.

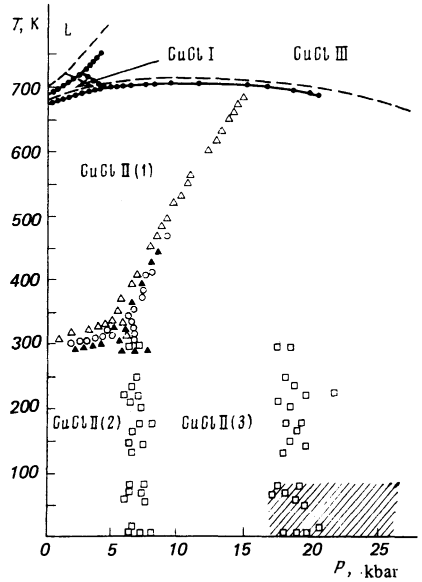

FIG. 1. Phase diagram of CuCl. CuCl I—wurtzite, CuCl II—sphalerite, CuCl III—phase with structure of AgI type, L—liquid phase, CuCl II (1, 2, 3)—phases with sphalerite structure. ●, △—DTA (at constant pressure) under hydrostatic conditions; ○—dilatometry (at constant pressure) under hydrostatic conditions; ▲—dilatometry (at constant temperature) under hydrostatic conditions; □—dilatometry (at constant temperature) under quasi-hydrostatic conditions; dashed curve—data of[18]. The section in which the transition is accompanied by a volume effect $\Delta V/V_0 \leq 1\%$ and by a jump of the magnetic permeability is shown shaded.

RESULTS AND THEIR DISCUSSION

Results of measurements in hydrostatic chamber. The phase diagram of CuCl is shown in Fig. 1. The phase-equilibrium lines, which were determined earlier,[18] are shown dashed. As seen from Fig. 1, our measurements, carried out up to 24 kbar in a hydrostatic chamber, coincide in the main with the data of[18], where the measurements were performed under quasi-hydrostatic conditions. The CuCl (II $\rightleftarrows$ I), CuCl (II $\rightleftarrows$ III) phase equilibrium lines coincide within the limits of experimental error with the data of[18]. The maximum of the CuCl (II $\rightleftarrows$ III) phase equilibrium line is located at a pressure 9.4 kbar and amounts to 708 °K, in good agreement with the data of[18], where this point has the coordinates 9.2 kbar and 712 °K. We have determined also the initial section of the melting curve up to 4 kbar, the slope of which agrees with the data of[18]. The only discrepancy is a certain non-coincidence of the triple points of the intersection of the CuCl (I $\rightleftarrows$ II), CuCl (II $\rightleftarrows$ III), CuCl (I $\rightleftarrows$ III) phase equilibrium lines on the two diagrams. This can be attributed to difficulties in the measurement of pressure up to 4 kbar in[18]. To determine the CuCl (II $\rightleftarrows$ III), CuCl (I $\rightleftarrows$ III), CuCl (I $\rightleftarrows$ II) phase equilibrium lines and the melting curve we used only the DTA method. The limits of all these transitions were determined from the midpoints between the heating and cooling. The hysteresis amounted to 1–2%, which agrees with the data of[18]. All the foregoing transitions were accompanied by distinct DTA signals and by breaks on the heating and cooling curves.

Transitions with small thermal and volume effects. The presence of a maximum on the CuCl (II $\rightleftarrows$ III) phase equilibrium line is evidence of the proximity of one more phase transition, which was determined by us (Fig. 1, the line CuClII (1 $\rightleftarrows$ 3)).

Figure 1 shows the phase-equilibrium lines, which we have designated CuClII (1), CuClII (2), CuClII (3), and which will henceforth be written in the form CuCl (1), CuCl (2), CuCl (3) for brevity. All these phases have the cubic sphalerite structure.[5] The boundaries between these phases were determined both with a dilatometer and by DTA. Some discrepancy between the lines determined by the different techniques is due to the different methods used in DTA and dilatometry to fix the start of the transition. Typical dilatometer patterns and DTA peaks for the phase transitions CuCl (1 $\rightleftarrows$ 2), CuCl (2 $\rightleftarrows$ 3), CuCl (1 $\rightleftarrows$ 3) are shown in Fig. 2. The phase boundaries were drawn through the midpoints determined from the heating and cooling curves. When the DTA method was used we were able to determine phase transitions only in the course of heating and cooling (at constant pressure). The CuCl (2 $\rightleftarrows$ 3) phase equilibrium line was determined mainly from dilatometry data, since the DTA signal produced when this line is crossed is very weak and we were able to measure only two points by this method (Fig. 1). The remaining equilibrium lines were determined by both procedures, with dilatometry used both at variable temperature and constant pressure and at variable pressure and constant temperature. The CuCl (2 → 3), CuCl (1 → 3) phase transitions are accompanied by absorption of heat (Fig. 2). The CuCl (2 $\rightleftarrows$ 1) transition is accompanied by two transformations, the first of which (marked *a* and *b* in Fig. 2) is accompanied by absorption (release) of heat when the sample is heated (cooled), and the second (marked *b* and *c* in Fig. 2) is much weaker and is accompanied by release of heat when the sample is heated. In view of the small temperature interval between them (2–10°) it is frequently difficult to distinguish between these transformations, and we consequently did not attempt to separate an individual line on the T–P diagram, and used as the basis for the determination of the CuCl (2 $\rightleftarrows$ 1) phase equilibrium line only the transformation in which heat was absorbed during the heating. Dilatometry fixes only one transition in this temperature and pressure interval (Fig. 2A).

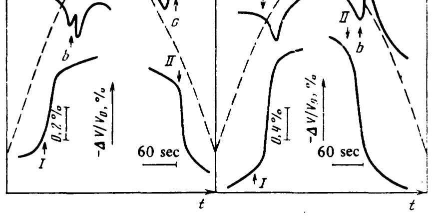

FIG. 2. Typical DTA signals and dilatometer patterns for transitions with small thermal and volume effects. T—temperature, $\Delta V/V_0\%$—volume effect in percent, t—time, ΔT—readings of differential thermocouple. A. CuClII (2 $\rightleftarrows$ 1); $P = 4.7$ kbar; a, c—transition with absorption (release) of heat in the course of heating (cooling): a—heating ($T = 339\,^\circ$K), c—cooling ($T = 330\,^\circ$K); b, d—transition with release (absorption) of heat in the course of heating (cooling): b—heating ($T = 344\,^\circ$K), d—cooling ($T = 339\,^\circ$K). I, II—sections of dilatometer patterns (heating and cooling) in the region of the phase transition CuClII (2 $\rightleftarrows$ 1); 1—heating ($T = 327\,^\circ$K); II—cooling ($T = 325\,^\circ$K). B. CuClII (3 $\rightleftarrows$ 1): a, b—DTA signals in heating ($T = 350\,^\circ$K: $P = 10.8$ kbar) and cooling ($T = 362\,^\circ$K, $P = 10.8$ kbar). I, II—dilatometer patterns in the region of the transition CuClIII (3 $\rightleftarrows$ 1): 1—heating ($T = 359\,^\circ$K), II—cooling ($T = 359\,^\circ$K), $P = 6.8$ kbar.

TABLE I. Thermal and volume effects of certain transitions (from results of measurements under hydrostatic conditions).

Transition	ΔQ, kcal/mole	$(\Delta V/V_0)$, %
CuClII (2 $\rightleftarrows$ 1)	0.07±0.005	0.6±0.05
CuClII (2 $\rightleftarrows$ 3)	0.02±0.005	0.6±0.05
CuClII (3 $\rightleftarrows$ 1)	0.10±0.005	1.5±0.05

Using the procedure described in[16], we estimated the values of the thermal effects of the phase transitions, which are listed in Table I, and obtained the following ratios of the absolute values of the thermal effects:

$$\frac{\Delta Q(\mathrm{II}\rightleftarrows\mathrm{I})}{\Delta Q(1\rightleftarrows 2)}=3\cdot 10, \qquad \frac{\Delta Q(L\rightleftarrows\mathrm{I})}{\Delta Q(1\rightleftarrows 2)}=4\cdot 10$$

$$\frac{\Delta Q(L\rightleftarrows\mathrm{I})}{\Delta Q(2\rightleftarrows 3)}=1\cdot 10^2 \qquad \frac{\Delta Q(L\rightleftarrows\mathrm{I})}{\Delta Q(3\rightleftarrows 1)}=2\cdot 10$$

$$\frac{\Delta Q(\mathrm{II}\rightleftarrows 1)}{\Delta Q(3\rightleftarrows 2)}=1\cdot 10^2 \qquad \frac{\Delta Q(\mathrm{II}\rightleftarrows\mathrm{I})}{\Delta Q(3\rightleftarrows 1)}=1\cdot 10.$$

Our calculations yield only the orders of magnitude of the quantities, since they are based on the thermal effect at atmospheric pressure for the melting of the wurtzite phase,[16] which is equal to 2.4 kcal/mole. The volume effects, in percent (see the table) were calculated from the dilatometry data. When determining the thermal effects we used where possible the areas under the DTA peaks obtained at identical pressures, since an increase in pressure increases the thermal conductivity of the liquid and influences the DTA signal. To estimate the thermal effects in the transitions CuCl (3 $\rightleftarrows$ 1) and CuCl (2 $\rightleftarrows$ 3), however, we were forced to use the corresponding ratios of the areas of the DTA peaks, taken at different pressures, since we had no initial data.

Investigations in a quasi-hydrostatic chamber. Figure 3 shows typical dilatometer patterns of crystals, up to pressures $P = 35$ kbar at $T = 300$, $T = 77.4$ and $T = 4.2\,^\circ$K. Inasmuch as under the first loading the initial section of the dilatometer pattern can be distorted by the effect of sample compression, we show the dilatometer patterns of the repeated loading. In addition, the figure shows the dependence of the magnetic permeability of the sample on the pressure at $T = 77.4\,^\circ$K (in relative units). At a pressure $P \lesssim 6$ kbar, the permeability is nearly constant, in the interval 7–18 kbar it depends insignificantly on the pressure, but at $P \approx 18$ kbar a radical change of permeability of opposite sign takes

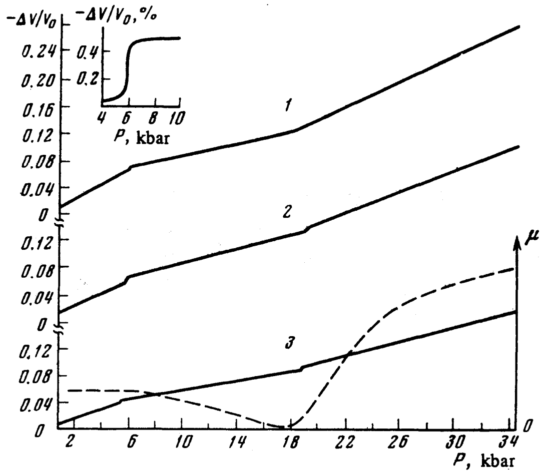

FIG. 3. Typical dilatometer patterns of CuCl crystals, obtained at the temperatures: 1—T=300; 2—T=77.4; 3—T=4.2 K. $\Delta V/V_0$—volume effect, μ—magnetic permeability in relative units. The dashed curve shows the change of the magnetic permeability with pressure at T=77.4 °K. The insert shows the volume effect in percent ($\Delta V/V_0$%) at $P\sim 6$ kbar, measured under hydrostatic conditions.

place. This change of the magnetic permeability at $P\approx 18$ kbar was observed only in the temperature region $T\leqslant 90$ K.

It is seen on the dilatometer patterns that with increasing pressure, at $P\approx 6$ kbar, a noticeable decrease takes place in the compressibility of the sample. Thus, at T=4.2 °K and $P\approx 6$ kbar the compressibility decreases jumpwise by a factor 1.7. With increasing pressure, at $P\approx 18$ kbar, the compressibility again increases strongly by an amount

$$\varkappa(P>18\text{ kbar})/\varkappa(P<18\text{ kbar})=1.8.$$

At a pressure $P\approx 6$ kbar, there is also observed a small volume effect $\Delta V/V_0=0.6\%$. The effect is reproduced on the dilatometer patterns measured at different temperatures from 4.2 to 300 °K, but not for all samples. The insert of Fig. 3 shows the volume effect at the same pressure, $P\approx 6$ kbar, but measured in the hydrostatic-pressure chamber at T=290 °K. The transition is clearly pronounced and takes place in a narrow pressure interval. Since the transition at $P\approx 6$ kbar was also recorded by the DTA method (see Fig. 1), it can be concluded that a phase transition with an abrupt change of the compressibility and with a small volume effect takes place at this pressure in the interval from 4.2 to 350 °K. According to[4,5], the lattice symmetry does not change in this pressure region and no noticeable change occurs in the resistivity. Consequently, we have observed an isomorphic (or a near-isomorphic) phase transition of the insulator–insulator type CuCl ($2\rightleftarrows 3$) (see Fig. 1).

A transition with an abrupt increase of the compressibility and with a jump of the magnetic permeability at P in the interval 18–20 kbar was accompanied by small volume effects $\Delta V/V_0\leqslant 1\%$ only at $T\leqslant 90$ K. Above this temperature, up to T=300 °K, no volume effect was observed, and the jumps of the magnetic permeability also vanish, although the compressibility increased smoothly at P=18–20 kbar. Nor were volume and thermal effects observed in the hydrostatic-pressure chamber up to P=24 kbar. X-ray diffraction and the resistivity[4,5] revealed no singularities of CuCl whatever in this pressure region at T=300 °K. Consequently, the strong increase of the compressibility at P=18–20 kbar takes place in the region of one CuCl phase (3). The change in the compressibility of the CuCl (3) phase is anomalous, inasmuch as usually the compressibility of solids decreases with increasing pressure.

We note that the magnetic-permeability jumps observed by us were observed also in[4], where the sample was cooled from room temperature at P=18–20 kbar in a hydrostatic-pressure chamber. At T=85 °K, a large jump of the magnetic permeability of the sample was observed. The effect had a large hysteresis when the pressure was plotted and was observed all the way to 6 kbar. The effect vanished at atmospheric pressure. In our measurements, the effect was preserved until the chamber was fully unloaded. This is apparently due to the residual pressure on the sample (P=1–2 kbar) left after removal of the load. The possibility of a dielectric-superconductor phase transition in CuCl at low temperatures and at P=18–20 kbar was reported in[2]. In view of the limited experimental information, it is difficult at present to offer an unambiguous physical interpretation of the anomalous behavior of the magnetic permeability at $P\approx 18$ kbar and $T\leqslant 90$ K, which is accompanied by small volume effects and by a large increase of the compressibility.

If it is assumed that CuCl is indeed an excitonic dielectric, then the T–P diagram constructed by us (see Fig. 1) can be explained within the framework of already existing theoretical concepts. Heating ($P\lesssim 6$ kbar) is accompanied by an excitonic dielectric–dielectric CuCl ($2\rightarrow 1$) phase transition with small $\Delta V/V_0\leqslant 1\%$. The interaction of the phonon (LA) branch and exciton branch leads to a sharp decrease of the speed of sound V_L, and consequently to an increase of the compressibility of the crystal ($\varkappa=1/B=3/(C_{11}+2C_{12})$, where $C_{11}=\sqrt{V_L}$).[2,11] We use the standard notation: $\varkappa$ is the compressibility, B the bulk modulus, and C_{ij} the elastic constants. An additional absorption peak appears in the spectrum of the single-particle excitations, corresponding to a binding energy E=0.358 eV in the exciton system.[7]

It is known[12,13] that the non-acoustic character of the exciton spectrum (the gap in the spectrum of two-particle excitations), and the small volume effect in the phase transition into the excitonic state are due to the presence in the interaction Hamiltonian of additional terms that describe transitions of two particles from band to band (we shall designate them arbitrarily V_{cv}^{vc}). With decreasing pressure, the indirect gap for the nonrestructed spectrum $E_{\Gamma X}$ decreases, and this leads to an increase of the concentration n of the excitons. When n exceeds a certain critical value, the system becomes unstable to Bose condensation of the interband plasmons, i.e., the terms V_{cv}^{vc} can no longer be regarded as a weak perturbation.[13] Owing to the increase of the gap in the spectrum of the two-particle excitations, the interaction with the phonon branch decreases. The sign of the interaction between an electron and a hole from different bands is reversed in the transition from an excitonic dielectric to a plasmon dielectric.[13] In our case this can take place at $P\approx 6$ kbar, when the size of the gap $E_{\Gamma X}$ for the non-restructured spectrum becomes commensurate with the phonon energy, inasmuch as the optical dielectric constant reverses sign in this frequency region. As a result, with increasing pressure (with increasing n), at $P\approx 6$ kbar, a first-order phase transition of the excitonic dielectric—plasmon dielectric CuCl $(2\rightarrow 3)$ takes place with small $\Delta V/V_0=0.6\%$ and with a strong increase of the compressibility

$$\frac{\varkappa(P>6\ \text{kbar})}{\varkappa(P<6\ \text{kbar})}=1.7.$$

It is known that in a plasmon dielectric, in contrast to an excitonic dielectric, the gap in the spectrum of the single-particle excitations, and consequently the critical temperature T_c, increase exponentially with increasing electron and hole densities. Consequently, the temperature region where the plasmon-dielectric phase is stable should increase on the $T-P$ diagram with increasing pressure. This agrees with the data of Fig. 1, where the stability region T_c increases with increasing pressure. As a result, the transition CuCl $(1\rightleftarrows 3)$ on the $T-P$ diagram is of first order with a small volume effect $\Delta V/V_0=1.5\%$ and with a weak change of the compressibility. We cannot exclude, however, the existence of another explanation of the $T-P$ diagram constructed by us, and also of a number of anomalous properties of CuCl, noted in the introductory part of the article. To verify the hypothesis it would be of interest to attempt to observe directly exciton drops in CuCl under load, as was done recently in[17].

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