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Electronic Instabilities of Two-Dimensional Metals

$K_3Cu_8S_6$ and $Rb_3Cu_8S_6$

by

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Electronic Instabilities of Two-Dimensional Metals, $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$

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ABSTRACT.   The two-dimensional metals $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ show anomalies in their temperature dependent electrical conductivities which suggest charge-density waves.   The band structure calculation of the mixed-valence CDW compound $K_3Cu_8S_6$ shows that the Fermi surface of the compound is mainly composed of sulfur p electrons.   The results of the calculations are supported by XPS studies.

Introduction

Attention has been focussed on alkali-metal copper chalcogenides as candidates for new conducting mixed-valence materials.   In 1952, Rudorff et al. [1] first reported these remarkable mixed-valence solids, KCu_4S_3, $K_3Cu_8S_6$, and $Na_3Cu_4S_4$.   The fact that all of these compounds show high electrical conductivities at room temperature ($100\sim1,000$ $\Omega^{-1}cm^{-1}$) and are mixed-valence in character has stimulated a growing interest in these materials.   Brown et al. [2] reported the single crystal X-ray diffraction results and physical properties of KCu_4S_3. KCu_4S_3 has a double layer structure with copper-sulfur (S-Cu-S-Cu-S) linkages separated by potassium ions and exhibits metallic properties in its electrical conductivity.   According to X-ray photoelectron spectroscopy (XPS) experiments [3], KCu_4S_3 has only copper in oxidation state Cu(I) rather than mixed valence Cu(I) and Cu(II).   Thus the valence state of KCu_4S_3 can be described formally as $K^{+1}Cu^{+1}{}_4(S^{2-})_2(S^-)$, a charge distribution which shows the mixed valence of sulfur atoms.   The electrical and magnetic properties of $RbCu_4S_3$ and $CsCu_4S_3$, which are isostructure with KCu_4S_3, have been reported by Ghosh et al. [4]. $RbCu_4S_3$ and $CsCu_4S_3$ also show metallic behavior with room temperature conductivities of 1,400 $\Omega^{-1}cm^{-1}$ and 1,250 $\Omega^{-1}cm^{-1}$, respectively. $Na_3Cu_4S_4$ has a one-dimensional (1-D) crystalline structure [5] which is composed of $[Cu_4S_4]^{3-}$ chains separated by sodium cations.   This compound shows metallic behavior in the temperature dependence of its electrical conductivity ($\sigma_{RT}=300$ $\Omega^{-1}cm^{-1}$) without any phase transitions arising from the Peierls instabilities which

are typically found in 1-D metals [6]. Whangbo et al. [7] reported a band electronic structure calculation on $Na_3Cu_4S_4$, and used the results to examine the absence of abnormal electrical phenomena in this 1-D compound

Recently, it has been reported [8] that the mixed valence metallic compound, $K_3Cu_8S_6$, which has a two-dimensional layered structure, exhibits two phase transitions, one at 153 K and a second one at 55 K. These were suggested to arise from charge-density wave instabilities. On the basis of XPS data for KCu_4S_3, it is expected that $K_3Cu_8S_6$ may have mixed valence sulfur i.e., S^{2-} and S^-. Thus the charge distribution in $K_3Cu_8S_6$ can be written as $K^{+1}_3Cu^{+1}_8(S^{2-})_5(S^-)$. This leads to the conclusion that the conduction states of $K_3Cu_8S_6$ originate from the holes in the valence band, and it will be shown here that this has largely sulfur p orbital character. This is the first example of a material exhibiting the CDW phenomenpn and which has sulfur p character in the Fermi surface. Usually, other 1-D CDW compounds, such as transition-metal dichalcogenides [9], transition-metal trichalcogenides [10], blue bronze [11], and KCP [12], have transition-metal d electrons in the conduction bands. In this report, the mixed valent states of $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ are examined by study of copper 2p core peaks using XPS. The temperature dependence of the electrical properties of $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ are also reinvestigated, and a band structure calculation on $K_3Cu_8S_6$ has been carried out to examine the electronic structure of Fermi surface.

Experimental Section

The preparation of $K_3Cu_8S_6$ is described in the literature [8]. A mixture of potassium carbonate, copper powder and sulfur powder, taken in a ratio of 6:1:6 was placed into an alumina crucible. The alumina crucible was tightly covered with an alumina cap and was placed inside a tube furnace. The tube furnace was flushed with argon for 1/2 hour before the reaction. A slow flow of argon was continued during the preparation to maintain an inert atmosphere during the reaction process. The reaction temperature of 825°C was reached with heating at the rate of 100°C/hour, and the reactants were soaked for 2 hours at 825°C. To obtain high quality crystals, a slow cooling process was used (50°C/hour). Blue-black shiny needles were separated from the polysulfides by washing with a deoxygenated 50:50 mixture of ethanol and distilled water. The crystals were thoroughly dried under vacuum. $Rb_3Cu_8S_6$ was prepared by using the same reaction condition as $K_3Cu_8S_6$, except that a mixture of rubidium carbonate, copper and sulfur in a ratio of 10:1:6 was used as reactants. The compounds were characterized by powder X-ray diffraction.

Temperature-dependent electrical resistancies were measured by using the standard four-point probe technique. Samples for the conductivity measurements were pellets with a 6.5 mm diameter and approximately 1 mm thickness. Commercial probes (Alessi Industry) with a 1.25 mm probe spacing were employed. Constant current (5 mA) was
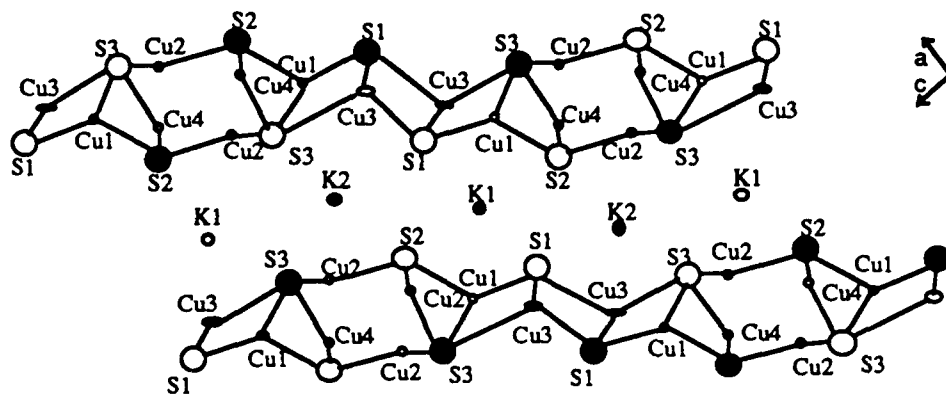
Figure 1. The crystal structure of $K_3Cu_8S_6$ as viewed down to the $[Cu_4S_4]^{n-}$ chain axis.

applied by using a Keithley Model 224 programmable current source, and the voltage drops were measured by Keithley 181 nanovoltmeter. Low temperatures were obtained by use of an Air Products Co. closed-cycle helium refrigerator.

XPS data were acquired by using a PHI 5400 XPS instrument manufactured by Perkin Elmer. Calibration of the instrument was done by using the Pt $4f_{1/2}$ peak at 80.0 eV and the Cu $2p_{3/2}$ peak at 952.6 eV. Excitation of a Mg anode was used for this work. The pass energy for the measurement was 35.8 eV, and the binding energy data were reproducible in the range of +0.1 eV.

Crystal Structure

The crystal structure of $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$, first solved by Burschka et al. [13], is shown in Fig. 1. These compounds are isostructural and have monoclinic symmetry with the space group C2/m. The basic structural unit of $K_3Cu_8S_6$ is a infinite $[Cu_4S_4]^{n-}$ chain with a slightly distorted trigonal planar Cu-S coordination. The $[Cu_4S_4]^{n-}$ chains follow the b axis in the unit cell. The $[Cu_4S_4]^{n-}$ chains which are bridged by edge-sharing tetrahedra results in the pleated layers which are separated by potassium atoms. There are two types of Cu-S coordination in the structure, threefold coordination within the $[Cu_4S_4]^{n-}$ chains and fourfold coordination within the bridging Cu-S bonds. The elliptical shapes at the fourfold Cu sites in Fig. 1 indicate that these sites are either disordered or that they have large thermal parameters. From a structural point of view, it is clear that there is an in-plane anisotropy in this layered structure. In the following section, it will be shown that how the low-dimensional structure is related to the anomalous electrical properties. It is interesting that the $[Cu_4S_4]^{n-}$ column structure is a fundamental unit in the structure of other alkali-metal copper (or silver) chalcogenides, like $Na_3Cu_4S_4$, [5] KCu_3S_2, [14] and $K_2Ag_4S_3$ [15].

RESISTANCE (OHMS)

5.89

5.75

5.61

5.47

5.33

(a)

60.0   120.   180.   240.   300.

TEMPERATURE (K)   xE0

xE-2

RESISTANCE (OHMS)

1.58

1.44

1.30

1.16

1.02

(b)

60.0   120.   180.   240.   300.

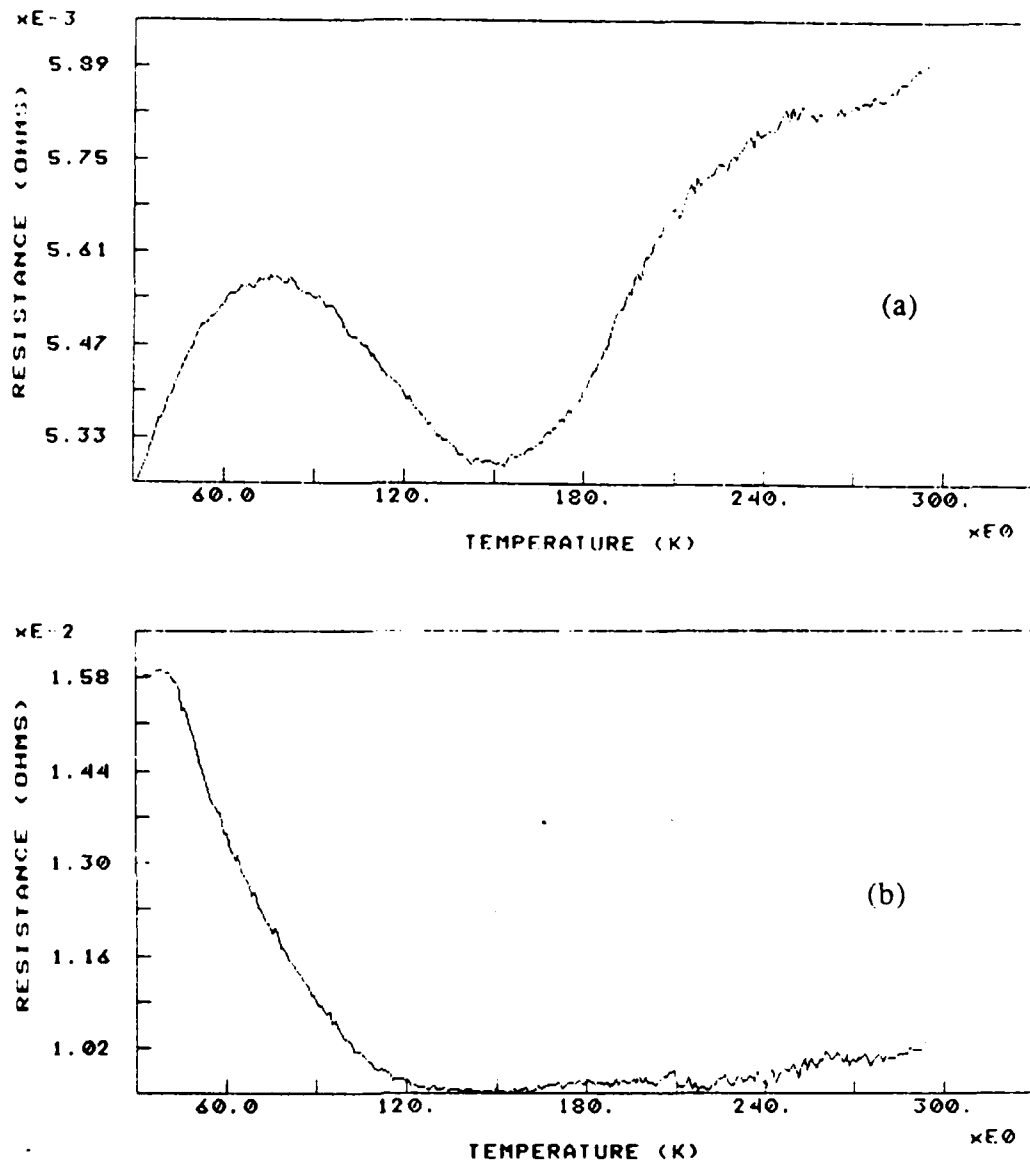TEMPERATURE (K)   xE0

Figure 2. Resistance of a pellet of $K_3Cu_8S_6$ (a) and $Rb_3Cu_8S_6$ (b) as a function of temperature. The room temperature resistivities of $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ correspond to 3.3×10^{-3} Ωcm and 4.1×10^{-3} Ωcm, respectively.

Physical Properties

The temperature dependence of the resistance of a pressed pellet of $K_3Cu_8S_6$ is plotted in Fig. 2(a) for the range 20-300 K. The resistance decreases with decreasing temperature, indicative of metallic behavior, down to about 150 K. There is a second-order phase transition at 150 K as a result of the CDW phenomenon. The resistivity increase of CDW states, which accompany the lattice distortion, are due to the opening of a gap at the Fermi surface. According to the report by ter Haar et al. [8], there are

two phase transitions in a single crystal of $K_3Cu_8S_6$ at 150 K and 53 K, and these are second-order and first-order phase transition, respectively. The absence of a transition at 53 K in our data may be an experimental artifact arising from the fact that the conductivity measurements were carried out by using a pressed pellet rather than a single crystal.

Magnetic susceptibility and specific heat data for $K_3Cu_8S_6$ [8] also show the phase transition at the same temperatures detected in the conductivity curve. Although a CDW-driven phase transition generally results in anomalies in the temperature variation of the resistivity, magnetic susceptibility, and specific heat measurements, observation of a superlattice by electron, X-ray, or neutron diffraction confirms the modulation of the lattice due to the CDW state. A low temperature X-ray scattering study [16] of CDW waves in the $K_3Cu_8S_6$ shows that the second-order transition at 153 K corresponds to the onset of an incommensurate CDW with a wave vector of $q=(0, (1-\delta)/2, 0)$ (where δ measures the incommensurability) and the first-order transition at 55 K produce a new commensurate periodicity with a wave vector of $q=(1/2, 1/2, 0)$. The parameter δ shows a temperature dependence in the second-order transition region ($\delta=0.11$ at 153 K and 0.07 at 53 K).

One of the important properties of CDW is the possibility of a sliding motion of the CDW as first suggested by Fröhlich. Nonlinear conductivity below a threshold electric field, attributed to the sliding CDW, was first reported for $NbSe_3$ by Monceau et al.[17] Since then, a considerable amount of work has been done to explore the dynamics of moving CDW for $NbSe_3$ [18, 19] and other CDW compounds such as TaS_3 [20] and $K_{0.3}MoO_3$ [21, 22]. However, CDW dynamics, such as electric-field-induced motion of the CDW, was not observed in $K_3Cu_8S_6$ [16].

The temperature dependence of the resistance of the $Rb_3Cu_8S_6$ is shown in Fig. 2(b). $Rb_3Cu_8S_6$ also exhibits a phase transition at 120 K and the resistance turns down around 20 K. It is most likely that this phase transition is due to a CDW which has a similar origin as that of the isotypic compound $K_3Cu_8S_6$. Conductivity measurement provide the simplest method to search for anomalies due to the phase transitions. To ascertain that the phase transitions of $Rb_3Cu_8S_6$ are CDW instability, X-ray or neutron scattering studies are required.

Mixed-Valence States

Among a series of reported potassium-copper-sulfur compounds, KCuS [23], KCu_4S_3, $K_3Cu_8S_6$ and KCu_3S_2, only KCu_4S_3 and $K_3Cu_8S_6$ provide mixed-valence examples. Usually, it is tempting to assign the mixed-valency to the transition metal. However, an early XPS study for KCu_4S_3 [3] shows that KCu_4S_3 contains only Cu(I). Fig. 3(a) shows the core level peaks of Cu $2p_{1/2}$ and $2p_{3/2}$ of $K_3Cu_8S_6$. The binding energy, the shape, and the line width of the signal correspond to those of monovalent copper. There is no evidence of Cu(II), which would show pronounced satellites in the higher binding energy region. Thus, $K_3Cu_8S_6$ can be described as
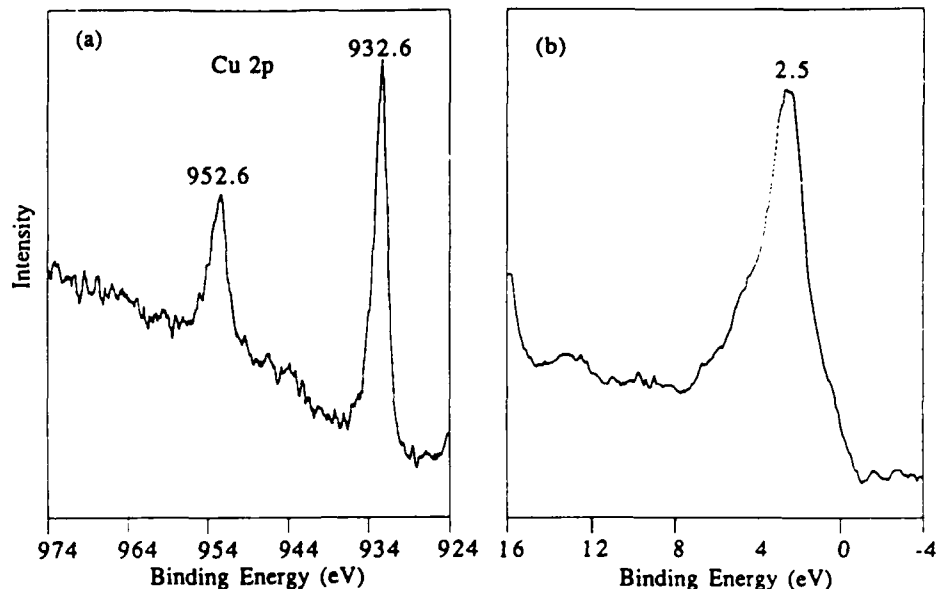
Figure 3. (a) Copper $2p_{1/2}$, $2p_{3/2}$ photoelectron spectrum of $K_3Cu_8S_6$. (b) Photoelectron spectrum of $K_3Cu_8S_6$ in valence band region.

$K^{+1}_3Cu^{+1}_8(S^{2-})_5(S^-)$, a formulation which involves mixed-valence of sulfur.

The valence band region of the spectra of $K_3Cu_8S_6$ is shown in Fig. 3(b). A broad peak about 8 eV wide is assigned to the states which are mainly sulfur 3p character, and a narrow copper 3d peak about 3 eV wide is superimposed on the broder peak.

Band Structure Calculation

The conduction process of $K_3Cu_8S_6$ is different from that of other typical CDW materials. Typically, the metallic conduction of usual CDW materials, for example, transition metal dichalcogenides and transition metal trichalcogenides, originate from transition metal d electrons in the conduction band. However, it can be expected from the mixed valence S^{2-} and S^- in $K_3Cu_8S_6$, that the metallic property of $K_3Cu_8S_6$ is due to the hole which is mainly sulfur p character in the valence band. Thus, the Fermi surface and CDW are originated from sulfur p electrons rather than copper d electrons. But, it is clear that covalence is very important in Cu-S bonds, and it is likely that the wave functions at the Fermi surface of this metallic compounds have considerable copper as well as sulfur character.

Band structural calculation were carried out to get some insight into the origin of the CDW states. Tight-binding band electronic structural calculation on the basis of the extended Hückel method were performed by using a program obtained from Quantum Chemistry Program Exchange (Program No. 571). The atomic parameters employed in this work are
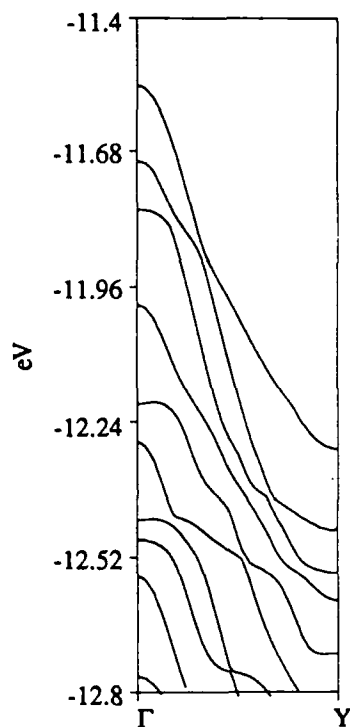
Figure 4. Band electronic structure calculated for the 1-D lattice of $[Cu_8S_6]^{3-}$. The wave vectors Γ and Y are defined as: $\Gamma=(0,0,0)$ and $Y=(0,b^*/2,0)$.

shown in Table 1. The dispersion relations for the $[Cu_8S_6]^{3-}$ layer are shown in Fig. 4. In the dispersion relations of $[Cu_8S_6]^{3-}$ layer, the Fermi level cuts the top two bands, both of which are about 70% of sulfur 3p and 30% copper 3d in character. As expected, covalency of the copper- sulfur bond results in hybridization of the copper (Cu^{2+} and Cu^+) and sulfur (S^{2-} and S^-) electrons in the Fermi surface. However, the XPS result which shows no evidence of the existence of Cu(II), is not consistent with the band calculation. One of the interesting facts is that the top band is mainly composed of copper and sulfur orbitals which make up the tetrahedral bridges between $[Cu_4S_4]^{n-}$ chains. The main part of the second band is due to the copper and sulfur orbitals which compose the $[Cu_4S_4]^{n-}$ chains. As mentioned in the crystal structure section, the disorder of the bridging

Table 1. Atomic parameters used in the calculation

atom	orbital	H_{ii}, eV	ξ_1	ξ_2	c_1	c_2
S	3s	-20	2.12			
	3p	-13.30	1.817			
Cu	4s	-11.4	2.20			
	4p	-6.06	2.20			
	3d	-14.00	5.95	2.30	0.5933	0.5744

copper (Cu3 in Figure 1) may play a main role in the phase transition of $K_3Cu_8S_6$.

The two-dimensional band calculation of $[Cu_8S_6]^{3-}$ shows very weak dispersion along the direction which is perpendicular to the $[Cu_4S_4]^{n-}$ chain direction in the $[Cu_8S_6]^{3-}$ layer. Thus, it can be concluded th $aK_3Cu_8S_6$ is a quasi-one-dimensional material in terms of both the crystal structure and electronic properties.

Conclusion

The mixed-valence inorganic compounds $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ show abnormal electrical properties as a result of the CDW instabilities. $K_3Cu_8S_6$ and $Rb_3Cu_8S_6$ are the first inorganic materials in which the charge carrier and the subsequent CDW have significant p character. The band calculation which we have presented here, reveals that sulfur 3p orbitals contribute significantly to the bands near the Fermi surface in the mixed compound $K_3Cu_8S_6$.

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