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Spin correlations in Na_xCoO_2 with $x = 0.8$ and 0.5

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Abstract

We present magnetization, specific heat, and Hall coefficient measurements on high quality Na_xCoO_2 single crystals with $x = 0.8$ and 0.5. Similar to $Na_{0.85}CoO_2$, a metamagnetic transition at about 20 K with $H \parallel c$ was observed in $Na_{0.8}CoO_2$ under a magnetic field of 9 T. This suggests that the common metamagnetic transition around 20 K would occur under a moderate magnetic field of ~9 T in Na_xCoO_2 with high x. For $x = 0.5$, we carried out elaborate specific heat investigations around 85 K under different magnetic fields. The results show an anisotropic nature of the transition at 85 K. This anisotropic property, revealed by both susceptibility and specific heat measurements, indicates that the spins are mainly pointing perpendicular to the c axis for $x = 0.5$ crystal. Moreover, by fitting the specific heat data below 46 K, a gap in low-lying excitation spectra is found and the gap value is estimated to be about 7.6 meV. This opening of the gap would account for the insulating ground state of $Na_{0.5}CoO_2$.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Recently, investigations of cobalt oxides have attracted extensive attention since the discovery of a surprisingly high thermoelectric power [1, 2] and superconductivity [3] in the Na_xCoO_2 system. The Na_xCoO_2 crystal consists of triangular layers of cobalt between close-packed layers of oxygen, separated by Na layers. A phase diagram with changing sodium content x has been proposed by Foo *et al* [4]. They found a crossover from a paramagnetic metal for $x \sim 0.3$ to an unusual Curie–Weiss metal for $x \sim 0.7$. The two metallic regimes are separated by a charge-ordered insulating phase at $x \sim 0.5$. The ground state shows antiferromagnetic ordering at low fields for $x \geqslant 0.75$ [5] and exhibits a metamagnetic transition at high fields in $Na_{0.85}CoO_2$ [6]. Further experiments are necessary to see whether or not such a metamagnetic transition at about 20 K would also occur in other crystals of Na_xCoO_2 with high x.

Superconductivity with $T_c \sim 5$ K is observed when $x \sim 0.3$ and sufficient water is intercalated between the CoO_2 layers. A number of theoretical works [7–10] have proposed that superconductivity in $Na_xCoO_2{\cdot}yH_2O$ can be described in terms of Anderson resonating valence bond (RVB) states in a doped Mott insulator on a triangular lattice, like high-T_c cuprates on a square lattice. Several experiments [11, 12] have also shown that superconductivity in $Na_xCoO_2{\cdot}yH_2O$ is unconventional, probably with spin triplet pairing. While some groups reported spin singlet pairing for this superconductivity system [13–16]. All these results suggested that the spin correlations play a crucial role in the understanding of superconductivity in Na_xCoO_2.

Neutron scattering experiments on $x = 0.75$ crystal [17, 18] and $x = 0.82$ crystal [19] showed that the in-plane spin correlations are ferromagnetic (FM) while the inter-plane spin correlations are antiferromagnetic (AF). The existence of in-plane FM correlations is consistent with the band structure calculation [20] and magnetization measurement [6]. However, the in-plane spin correlation for low Na concentration x, especially for x around 1/3, is still not clear. The ^{59}Co nuclear quadrupolar resonance measurement suggests there exists two-dimensional AF correlation in $Na_{0.35}CoO_2{\cdot}yH_2O$ [21], while the static magnetic susceptibility (χ) measurement shows that χ is weakly temperature-dependent, indicating a Pauli paramagnetic behavior [4, 22].

Among the Na_xCoO_2 family, $Na_{0.5}CoO_2$ is also of special interest besides the $x \sim 0.35$ one where the superconductivity occurs; it has distinct properties and is considered to be the parent phase of superconductivity in this system because the Co valence of the superconductor is found to be close to 3.5 [23–25]. So an understanding of the electronic and magnetic states of $Na_{0.5}CoO_2$ is very important, and may shed light on the origin of superconductivity. There are four phase transitions in $Na_{0.5}CoO_2$ that are not observed for other Na concentrations. A sharp metal–insulator transition near 50 K is observed due to the formation of charge ordering, and a variety of methods including electron diffraction [4, 26], infrared (IR) spectroscopy [27–29], angle resolved photoemission spectroscopy (ARPES) [30], Shubnikov–de Haas (SdH) [31], NMR [13, 32] and neutron scattering [33] have been used to investigate this insulating state. A charge density wave gap with $\Delta \sim 125$ cm^{-1} opening at this temperature was observed by infrared spectroscopy measurements [27]. An anisotropic single-particle gap is also detected to be about 6–11 meV by ARPES [30]. At about 120 K, structural distortion due to the Na ordering is observed by electron diffraction and Raman scattering measurements [33, 34]. At about 87 K, another phase transition is observed from susceptibility, specific heat, and NMR measurements [4, 13, 22, 33, 35, 36]. The cause of this transition is still not fully known. Huang *et al* suggested that this transition may be associated with structural distortion [33], while the NMR and neutron scattering studies evidenced an antiferromagnetic ordering below 87 K [13, 36]. Below about 20 K, the slope of temperature-dependent resistivity increases with decreasing the temperature, indicating another phase transition which is not exhibited in the specific heat and magnetic data [33]. The transition may be due to the spin reorientation suggested by μSR measurements [37]. A spin freezing picture below ~20 K is suggested by Imai *et al* [38]. The ARPES study of $Na_{0.5}CoO_2$ indicates that a good part of Fermi surface (FS) is nested around 60 K and most of the FS is gapped near 20 K, consistent with the published resistivity data [30]. Although a lot of studies have been done on $Na_{0.5}CoO_2$, including specific heat studies on the low-temperature transitions under a zero field in $Na_{0.5}CoO_2$ polycrystalline samples, our specific heat study is the first elaborate specific heat investigation on the low-temperature transitions under applied fields in a single crystal of $Na_{0.5}CoO_2$.

In this paper, we present magnetization, specific heat, and Hall coefficient measurements on high quality Na_xCoO_2 single crystals with $x = 0.8$ and 0.5, particularly an elaborate specific heat study for the $x = 0.5$ sample. Our results on $Na_{0.8}CoO_2$ show the existence of a metamagnetic transition, being similar to that of $Na_{0.85}CoO_2$. For the $x = 0.5$ crystal, specific heat investigations around 85 K under different fields show the anisotropic nature of the transition at 85 K. This anisotropic property indicates that the spins are mainly pointing perpendicular to the c axis. Moreover, we found a gap in low-lying excitation spectra below ~46 K and estimated the gap value to be about 7.6 meV by fitting the specific heat data.

2. Experimental details

Single crystals of $Na_{0.8}CoO_2$ were prepared under flowing O_2 in a floating-zone optical image furnace. The detailed preparation procedure is published elsewhere [6]. The Na_xCoO_2 crystals with $x = 0.5$ are obtained by Na de-intercalation of $Na_{0.80}CoO_2$ in a solution of bromine-dissolved acetonitrile. The Na:Co ratio of the crystals is determined by induction-coupled plasma measurements. From the x-ray diffraction measurements, no impure phase is detected within an experimental error of 2%. The c-axis lattice constant c is determined to be 10.82 Å and 11.1 Å for $x = 0.8$ and 0.5, respectively. The c dependence of x is consistent with the data published by other groups [4]. The magnetization, specific heat, and Hall coefficient measurements were performed in Quantum Design PPMS systems. The specific heat measurement was carried out using the relaxation method. The field dependence of the thermometer and addenda was carefully calibrated before the specific heat measurements.

3. Results and discussion

We first give a brief account for the $Na_{0.8}CoO_2$ crystal. Figure 1 shows the magnetic properties of $Na_{0.8}CoO_2$ crystal. Figure 1(a) shows the magnetic susceptibility versus temperature T from 2 to 300 K in a field of 5 T applied both along and perpendicular to the c axis. At high temperatures, χ shows a Curie–Weiss-like behavior. Below about 21 K, χ drops down sharply for $H \parallel c$ but goes up rapidly for $H \perp c$. The sharp drop in χ for $H \parallel c$ reveals a phase transition from a paramagnetic to an AF state. The increase of χ for $H \perp c$ indicates that the staggered moments are only along the c axis. The results are consistent with those observed in $Na_{0.82}CoO_2$ crystal [5] and $Na_{0.85}CoO_2$ crystal [6]. Figure 1(b) shows the magnetization M as a function of field at 2 K for $H \parallel c$ and $H \perp c$, respectively. For $H \parallel c$, a rapid superlinear rise in $M(H)$ is clearly seen at about 9 T. However, for $H \perp c$, the magnetization shows a typical paramagnetic behavior with increasing H up to 10 T. Figures 1(c) and (d) show the low-temperature susceptibility of $Na_{0.8}CoO_2$ measured in applied fields up to 12 T oriented both along and perpendicular to the c axis. For $H \parallel c$, below ~21 K, the susceptibility decreases rapidly at 1 and 5 T, and the Curie tail at 1 T attributed to paramagnetic impurities is suppressed by a field of 5 T. However, the susceptibility at 12 T increases below the transition temperature. The low-temperature magnetization behavior under fields along the c axis implies that a metamagnetic transition from an AF state to a quasi-FM state develops at a field between 5 and 12 T (~9 T seen from figure 1(b)). However, for $H \parallel ab$, the susceptibility at low temperatures decreases with increasing field. Similar to $Na_{0.85}CoO_2$, the anisotropic magnetic response of $Na_{0.8}CoO_2$ reveals a metamagnetic transition at about 20 K with $H \parallel c$ [6]. This suggests that such a metamagnetic transition around 20 K, which can be observed under a moderate magnetic field of ~9 T, should be a common feature in the Na_xCoO_2 system with high x. Furthermore, our results coincide with the A-type AF spin structure of Na_xCoO_2 with high x revealed

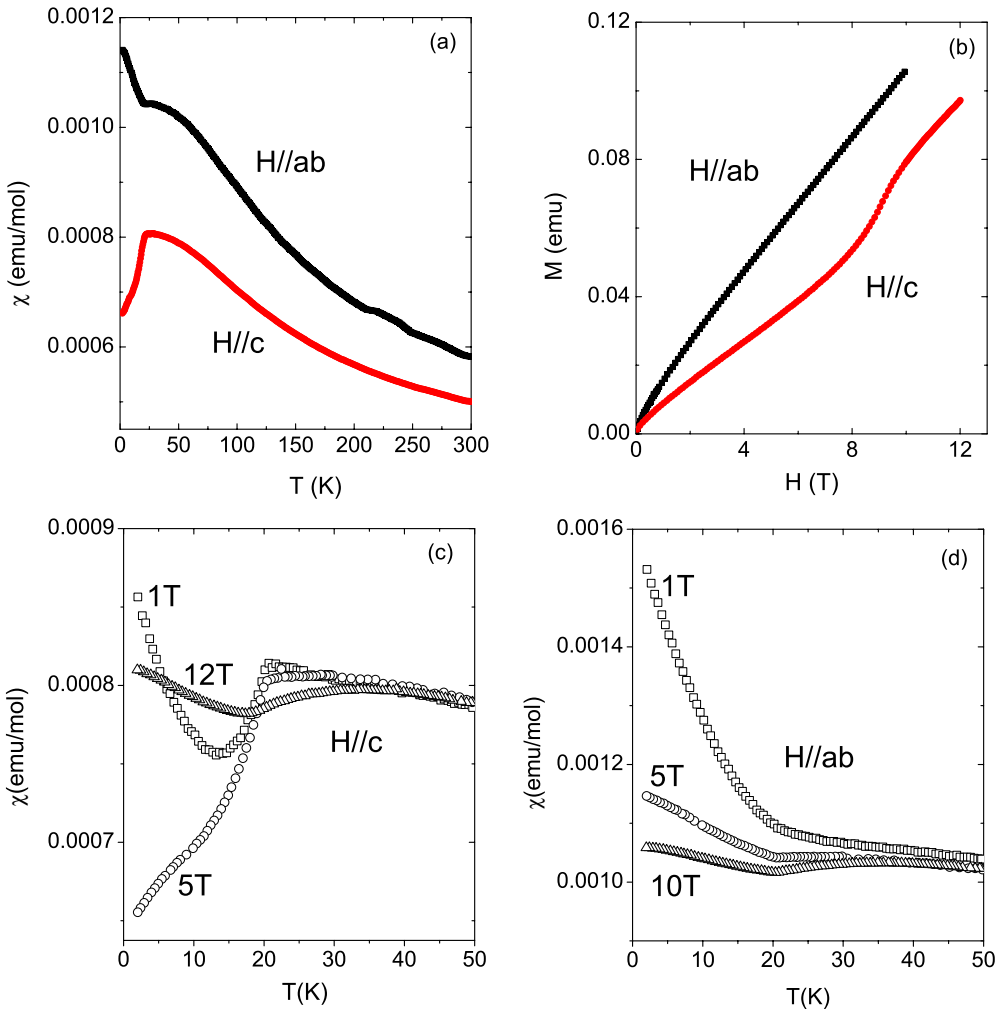

Figure 1. (a) The magnetic susceptibility versus temperature T for $Na_{0.8}CoO_2$ in a magnetic field of 5 T applied along and perpendicular to the c axis. (b) The magnetization M versus field H of $Na_{0.8}CoO_2$ at 2 K for $H \parallel c$ and $H \perp c$, respectively. (c), (d) Low-temperature susceptibility in different fields applied along and perpendicular to the c axis, respectively.

by neutron scattering experiments [17–19]. It is also found that a magnetic field of ∼9 T applied along the c axis is not sufficient to destroy the strong inter-plane AF coupling. To change into an energetically favored state, the spins suddenly lay down within the CoO_2 plane from the direction of the c axis at the critical field via a spin-flop transition. Thus, the metamagnetic transition in Na_xCoO_2 with high x originates from the rearrangement of antiferromagnetically ordered moments, corresponding to a spin-flop transition from an AF to a FM polarized state along the c axis. The weak FM behavior along the c axis in high fields implies that there is a slight canting angle with respect to the CoO_2 plane for the spins. Different from the spin structure of Na_xCoO_2 with high x, in the CoO_2 plane of $Na_{0.55}CoO_2$ there exist an antiferromagnetic ordering of the Co sites with large magnetic moments and a ferromagnetic ordering of the Co sites with small magnetic moments, and their spin directions are within the CoO_2 plane [39]. These results suggest that the spin structure of Na_xCoO_2 is sensitive to the Na content.

We now focus on spin correlations in the peculiar $x = 0.5$ compound. Figure 2(a) shows the magnetic susceptibility χ of $Na_{0.50}CoO_2$ from 2 to 350 K for $H \parallel c$ and $H \perp c$, respectively. Several features are observed from figure 2(a). Firstly, the values of χ show anisotropic behaviors between $H \parallel c$ and $H \perp c$. The difference in χ between $H \parallel c$ and $H \perp c$ is temperature-independent above 85 K and becomes much smaller at low temperatures. Secondly, χ decreases with decreasing temperature above 25 K for both $H \parallel c$ and $H \perp c$, while in $Na_{0.80}CoO_2$, χ increases with decreasing temperature. A Curie tail is observed below 25 K that prevents the further decrease of χ on lowering the temperature. Thirdly, two transitions with a sudden sharp decrease of χ at ∼85 and ∼46 K are observed for $H \perp c$, but only one transition at ∼46 K is observed for $H \parallel c$. Both transition temperatures are slightly lower than those published by Foo *et al* [4], which may be due to the slight difference in Na concentration. No anomaly at ∼20 K is observed for either $H \perp c$ or $H \parallel c$. Finally, χ tends to saturate at high temperatures for both $H \perp c$ and $H \parallel c$. A similar anisotropic magnetic response for $H \perp c$ and $H \parallel c$ has been observed by Chou *et al* [22].

The upturn below 25 K is supposed to be due to a small number of impurities and can be fitted by the Curie–

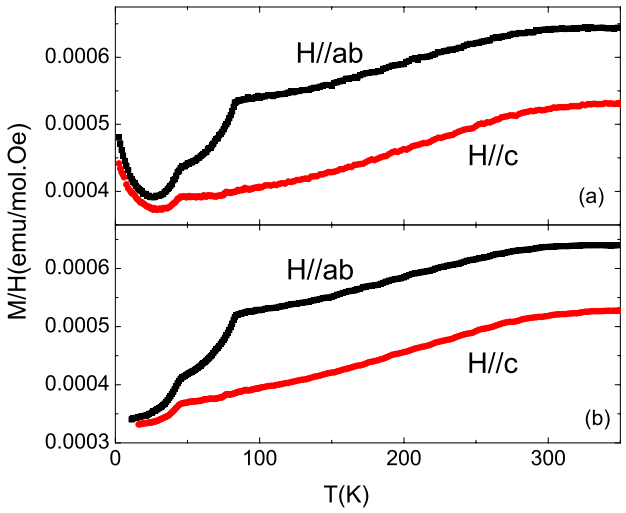

Figure 2. (a) The magnetic susceptibility $\chi = M/H$ versus temperature T for $Na_{0.5}CoO_2$ in a magnetic field of 1 T applied along and perpendicular to the c axis. (b) $\chi(T)$ for $H \parallel c$ and $H \parallel ab$ after subtracting the Curie–Weiss contributions of the impurities.

Weiss law, i.e. $\chi = C/(T + \theta)$. The fitting gives C and θ to be 0.00117 emu K mol^{-1} Oe^{-1}, 3.64 K and 0.0013 emu K mol^{-1} Oe^{-1}, 7.72 K for $H \parallel ab$ and $H \parallel c$, respectively. Figure 2(b) shows behavior of $\chi(T)$ for $H \parallel ab$ and $H \parallel c$ after subtracting Curie–Weiss contributions of impurities. The decreasing χ at 85 K corresponds to an antiferromagnetic ordering transition. The transition at 85 K is only observed for $H \parallel ab$, indicating that the ordering spins point within the CoO_2 plane, consistent with the neutron scattering experiments on $Na_{0.5}CoO_2$ crystal [40]. Such a result is different from the AF ordering at higher Na concentrations, where the spins point along the c axis. With further decreasing temperature, the anomalies of susceptibility are observed at 46 K for both $H \parallel ab$ and $H \parallel c$.

Figure 3(a) shows the temperature dependence of specific heat C divided by temperature T, C/T, ranging from 2 to 160 K for $Na_{0.5}CoO_2$ crystal in zero field. The specific heat shows a sharp peak at around 85 K. It corresponds to the magnetic phase transition observed in the susceptibility measurement. The inset of figure 3(a) shows C/T versus T around 85 K in zero field and a field of 12 T applied along and perpendicular to the c axis, respectively. The shape of the specific heat peak around 85 K shows a typical shape of a second-order phase transition and the peak exhibits anisotropic field dependence. We can see the phase transition temperature as well as the magnitude of the specific heat peak decrease in a field of 12 T applied perpendicular to the c axis. In a magnetic phase transition from a paramagnetic state to an AF state, the applied field suppresses the AF correlation and thus the transition temperature will decrease with increasing applied field. Our field dependence of specific heat shows that the phase transition at 85 K is an AF ordering phase transition. In a field of 12 T applied along the c axis, the specific heat keeps the same position and shape as the zero one. This means that the applied field along the c axis does not affect the AF phase transition at 85 K. These results suggest the ordered spins

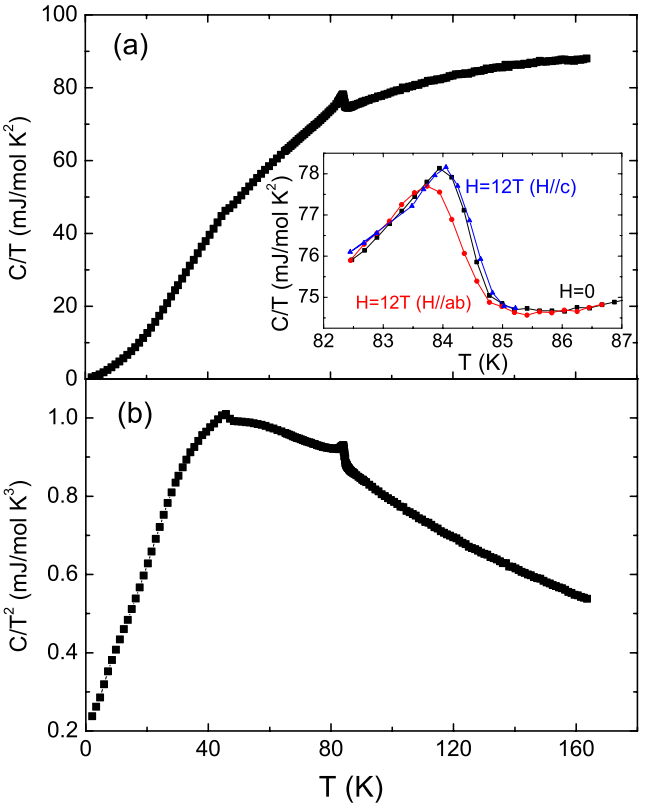

Figure 3. (a) C/T versus T from 2 to 160 K for $Na_{0.5}CoO_2$ crystal in zero field. Inset: C/T versus T around 85 K in zero field and a field of 12 T applied along and perpendicular to the c axis, respectively. (b) Temperature dependence of C/T^2 from 2 to 160 K in zero field.

point within the Co–O plane, in agreement with the anisotropic results in the susceptibility measurement.

Figure 3(b) shows the temperature dependence of C/T^2 from 2 to 160 K in zero field. The transition at 46 K is also clearly observed in specific heat measurement besides that at 85 K, and the specific heat anomaly at 46 K is broader than that at 85 K. However, the phase transition at 20 K and structural distortion above 120 K have not been observed in the specific heat measurement (see also figure 3(a)).

In figure 4, we rescale the specific heat data of $Na_{0.5}CoO_2$ crystal in the temperature interval below about 46 K. As is well known that an insulating state abruptly appears in $Na_{0.5}CoO_2$ at low temperatures, so electronic specific heat can be neglected below 46 K. As shown in the inset of figure 4, C/T decreases with decreasing temperature and a deviation from linearity for the C/T versus T^2 curve below 46 K can be observed. So other contributions should be considered to the specific heat, besides the contribution of phonons. As exhibited in figure 4, the specific heat data below 46 K can be well fitted with

$$C = \alpha T^2 + \beta T^3 + A \exp(-\Delta/T). \quad (1)$$

The presence of the T^2 term may be due to 2D AF ordering of spins, which gives a linear energy dependence of density of states at the Fermi level. The presence of an exponential term in the low-temperature specific heat indicates there is a gap in low-lying electronic excitation spectra. From the

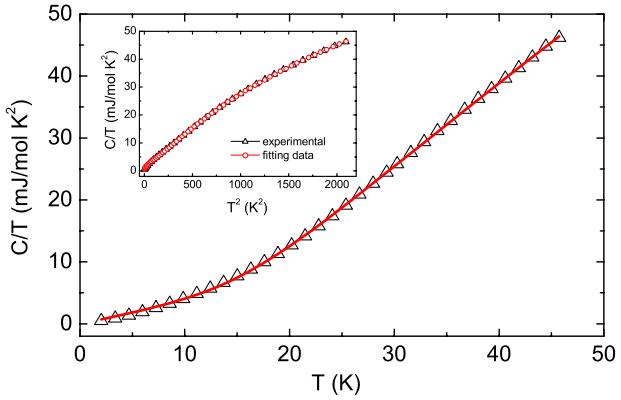

Figure 4. C/T versus T for $Na_{0.5}CoO_2$ crystal in zero field below about 46 K. The solid line is the fitting to the experimental data. Inset: comparison of the experimental data with the fitting plotted in the C/T versus T^2 curve.

fitting, we obtained $\Delta \sim 88.8$ K (∼7.6 meV). The gap (∼7.6 meV) estimated from our specific heat measurement coincides with an optical gap ($2\Delta \sim 125$ cm^{-1}) observed by infrared spectroscopy measurement and a single-particle gap ($\Delta \sim$ 6–11 meV) detected by angle resolved photoemission spectroscopy measurement [27, 30].

The above specific heat and magnetic susceptibility measurements show evidence for both the AF ordering transition at 85 K and the charge ordering transition at 46 K. In $Na_{0.5}CoO_2$, the average valence of Co ion is +3.5. Huang *et al* carried out neutron and electron diffraction experiments and found that there are two kinds of cobalt ions, Co1 and Co2, in different oxidation states [33]. They suggested that Co1 is in a lower oxidation state than Co2. The valence of Co1 and Co2 is not very different from the averaged value of 3.5 [35]. Two types of Co signals have also been observed by NMR measurements on $Na_{0.5}CoO_2$ crystal, where the large moments of Co1 align antiferromagnetically with spin direction in the *ab* plane and the small moments of Co2 are parallel to the *c* axis with antiferromagnetic inter-plane correlations [13]. Moreover, Wang *et al*, by means of AMR measurements [41], have suggested that the in-plane magnetic coupling of small moments of Co2 is antiferromagnetic. We consider the transition at 85 K corresponds to the AF ordering of Co1, while the anomaly of susceptibility at 46 K is due to the quantitative change of AF exchange parameters accompanying the metal–insulator transition. Our data can be well understood based on the model shown in figure 5. Above 85 K, the spins of Co1 and Co2 may align with short-range AF correlations because the susceptibility decreases with decreasing temperature below 300 K. Between 85 and 46 K, the spins of Co1 atoms align with a long-range AF chain, whereas the spins of Co2 are still in disorder. Neutron scattering measurements have indicated that below 88 K the spins form a novel antiferromagnetic pattern within the CoO_2 planes, consisting of alternating rows of ordered and 'non-ordered' Co ions [40], where the rows of 'non-ordered' Co ions may correspond to Co ions with fluctuating moments, which do not contribute to the long-range antiferromagnetism. In our experiments, the sudden decrease of χ and the specific heat peak at 85 K are the indications of such an AF transition, and the anisotropic property at 85 K can be well explained by the above spin correlations. The Na NMR study has observed that the 87 K transition does not result in the opening of a gap in the spectrum of electronic excitations, consistent with the previously reported resistivity behavior [4, 36]. An ARPES study on $Na_{0.5}CoO_2$ has shown that below ∼80 K the energy gap increases as the temperature is lowered [30]. At 46 K, the AF exchange parameters change quantitatively accompanying the metal–insulator transition, leading to an anomaly in susceptibility. A neutron scattering study on $Na_{0.5}CoO_2$ suggests the drop in susceptibility near 51 K is not caused by the AF ordered spins below 88 K, but correlates to a gap developing in the excitation spectrum of the 'non-ordered' Co ions [40]. By means of specific heat measurements, we found a gap in low-lying excitation spectra, and this opening of the gap would readily explain the onset of the insulating transition at 46 K.

Figure 6 displays the Hall coefficient R_H as a function of temperature for $Na_{0.5}CoO_2$ crystal. Similar to the result reported by Foo *et al* [4], R_H with a holelike character keeps T-independent above 200 K. Below 200 K, R_H slightly increases with decreasing temperature, and then at around 85 K, R_H plunges to large negative values. Below 46 K, R_H increases sharply in magnitude to a peak at about 20 K. Such a $R_H(T)$

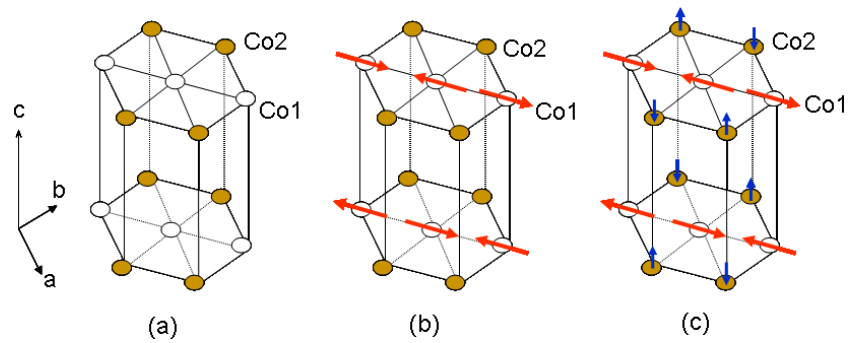

Figure 5. The evolution of spin correlations for $Na_{0.5}CoO_2$ with temperature, where two CoO_2 layers are depicted, and only Co ions are shown for clarity. The circles with arrows indicate magnetically ordered Co ions, where the arrows denote the directions of the magnetic moments, while the circles without arrows indicate Co ions without magnetic order. The open and filled circles represent the Co1 and Co2 ions, respectively. (a) Spin arrangement for $Na_{0.5}CoO_2$ above 85 K. (b) Spin arrangement for $Na_{0.5}CoO_2$ between 85 and 46 K. (c) Spin arrangement for $Na_{0.5}CoO_2$ below 46 K.

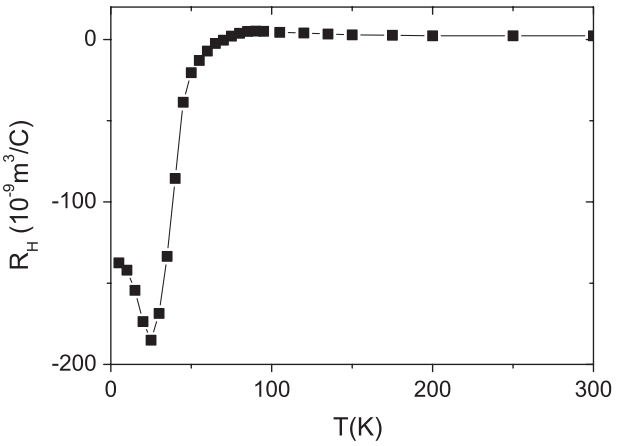

Figure 6. Hall coefficient R_H as a function of temperature for $Na_{0.5}CoO_2$ crystal.

behavior could be understood by the picture described by Imai *et al*, where the insulating behavior of charge degrees of freedom below about 51 K is linked primarily with the Co2 sites [38]. As the temperature decreases from 85 to 46 K, the transverse conductivity becomes smaller and smaller because of the spin and charge ordering of Co1. Therefore, R_H starts to drop to negative values at 85 K. However, the charge carriers of Co2 can still move, so that the longitudinal conductivity is not affected much, which could be seen in the resistivity measurements. Below 46 K, since the ordering of charge carriers of Co1 and Co2, both the R_H and resistivity experience a sudden increase in magnitude.

4. Conclusions

In summary, we present magnetization, specific heat, and Hall coefficient measurements on Na_xCoO_2 single crystals with $x = 0.8$ and 0.5. A metamagnetic transition at about 20 K was observed in $Na_{0.8}CoO_2$, similar to that in $Na_{0.85}CoO_2$. This suggests that the metamagnetic transition around 20 K is inclined to occur in Na_xCoO_2 with high x under a moderate magnetic field. For $Na_{0.5}CoO_2$, our specific heat and susceptibility measurements exhibit an anisotropic type transition at 85 K, where the spins are mainly pointing perpendicular to the c axis. Moreover, we found a gap in low-lying excitation spectra, and by fitting the specific heat data below 46 K estimated the gap value to be about 7.6 meV. This opening of the gap may account for the insulating ground state of $Na_{0.5}CoO_2$.

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