Effect of magnetic fields on the metal-insulator transition in $BaVS_3$

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We report measurements of the high field magnetoresistance of single crystals, and of the low field Hall effect and high field magnetization of polycrystalline samples of $BaVS_3$. Below the temperature $T_{MI} \simeq 70$ K of the metal-insulator (MI) transition, the magnetization is linear for $0 < H < 45$ T. The magnetoresistance varies as $a(T)H^2$ for $0 < H < 18$ T, and $a(T)$ varies proportionally to the logarithmic derivative of the zero field resistivity $(1/\rho)d\rho/dT$. This result allows us to estimate the zero temperature critical field $H_0 \simeq 260$ T $(=175$ K); for this value, the spin gap is substantially smaller than the charge gap given by $\Delta_c \simeq 500$ K. The Hall constant increases to very large positive values for $T < T_{MI}$, and shows considerable precursive activity for $T > T_{MI}$ indicative of fluctuations in the gap above the MI transition. We use these results to discuss several models for the MI transition. [S0163-1829(99)01942-6]

I. INTRODUCTION

The room temperature structure of $BaVS_3$ is hexagonal $(P6_3/mmc)$ wherein the V atoms are surrounded by face-sharing S octahedra, forming chains along the c axis.[1] Since the intrachain V-V spacing (2.8 Å) is much shorter than the interchain spacing (6.7 Å) a common belief is that the material exhibits nearly one-dimensional (1D) behavior.[2] The behavior at room temperature is metallic and the susceptibility exhibits a Curie-Weiss law indicative of nearly local V $(d^1)s = \frac{1}{2}$ paramagnetism.[2] Below $T_s = 240$ K the compound transforms to an orthorhombic $(Cmc2_1)$ structure,[3] but remains a nearly local paramagnetic metal. At $T_{MI} = 70$ K there is a metal-insulator (MI) transition [Fig. 1(a)] where there is a sharp cusp in the susceptibility[4] [Fig. 2(a)]. The entropy change at T_{MI} is close to $Rln2$,[5] which suggests that the degrees of freedom associated with the V(d^1) $s = 1/2$ spins are liberated at the MI transition. Below $T_x = 30$ K, hyperfine fields are observed in NMR (Ref. 6) and Mossbauer[2,4] and anomalously large electric-field gradients at the V site are seen in NQR, suggestive of orbital ordering;[7] however, no anomalies are observed in the thermodynamic or transport behavior. (An upturn in the susceptibility near T_x [Fig. 2(a)] has been attributed to this transition.[6]) The crystal symmetry remains[8] orthorhombic $Cmc2_1$ below T_{MI} and T_x and no magnetic long range order has been observed by neutron scattering in any of the phases.[3]

The nature of the transitions at T_{MI} and T_x remains a mystery and there have been several proposals concerning the metal-insulator transition. Early proposals considered that the MI transition could be due to the onset of antiferromagnetic[4] or Peierls[6] order. However, no such order was observed by neutron diffraction[3] and no static local fields were observed in NMR,[6] NQR,[7] Mossbauer,[2,4] and spin-flip scattering[9] experiments for $T_x < T < T_{MI}$. Because of the expected one dimensionality, one belief is that the transition at T_{MI} is not a true 3D phase transition, but a 1D ''transition'' below which only short range order occurs; T_x might then correspond to the 3D transition.[2] Recent photoemission experiments[10] have been interpreted as indicative of Luttinger liquid behavior in the metallic state, consistent with a high degree of one-dimensionality. Band structure calculations[11] suggest, however, that the actual degree of anisotropy is rather small, essentially due to V $(3d)/S$ hybridization across the chains. The band calculations do support an older model due to Massenet[12] wherein the distortions of the S octahedra split the V t_{2g} states in such a way that a broad (3 eV wide) d_{z^2} band overlaps a narrow (0.7 eV wide) d_{xy} band; the metallic conductivity arises from the former, the nearly local paramagnetism from the latter. Although the band theory demonstrates that the increase in the distortion with decreasing temperature cannot give rise to a simple band gap, it does show that the distortion causes a partial transfer of electrons from the d_{z^2} band to the narrow d_{xy} band. These calculations have led to the suggestion that if the transfer is large enough to put the narrow band at half occupancy, then electron correlations can give rise to a Mott-Hubbard MI transition.[13] The d_{xy} electrons would become nonmagnetic in the insulating phase, explaining the loss of paramagnetism below T_{MI}.

Magnetic fields should have a different effect on the MI transition depending on which of these scenarios is correct. For transitions involving antiferromagnetic interactions (including spin-density wave and Peierls order), a sufficiently large magnetic field should cause an appreciable shift in T_{MI}. For other models, large magnetic fields should have a much smaller effect. For this reason, we have measured the high field magnetoresistance and magnetization in an effort to put further constraints on possible theories. In this paper we report these measurements as well as measurements of the Hall effect.

II. EXPERIMENTAL DETAILS

Polycrystalline samples were grown by direct reaction of BaS, V, and S in sealed quartz tubes at 900 °C.[2] Single crys-

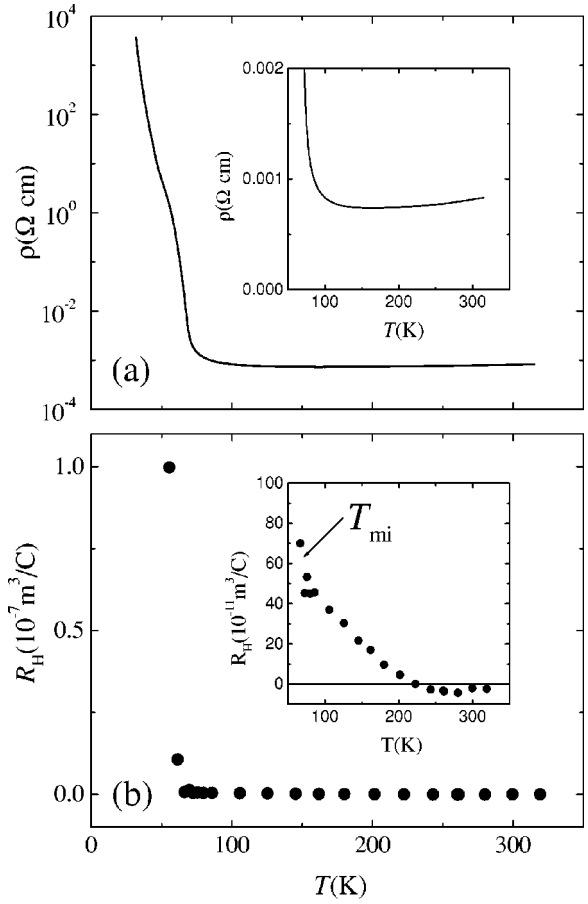

FIG. 1. (a) Resistivity $\rho(T)$ versus temperature for a single crystal of $BaVS_3$, showing the metal insulator transition near $T_{MI} \approx 70$ K. Inset: details of the resistivity, showing the resistance minimum near 150 K. (b) Hall coefficient R_H versus temperature, showing the large increase of R_H at T_{MI}, indicating the loss of carriers. Inset: R_H vs temperature on an expanded scale showing the precursive increase of R_H with decreasing temperature for $T > T_{MI}$.

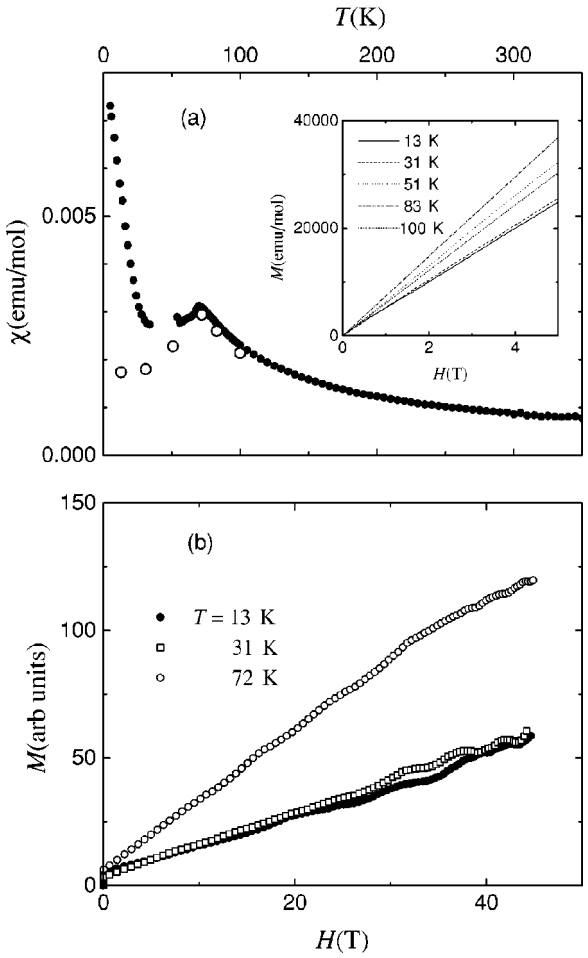

FIG. 2. (a) The susceptibility of a polycrystalline sample of $BaVS_3$ obtained using a magnetic field $H = 0.1$ T (closed circles). A cusp occurs at T_{MI}, and a Curie ''tail'' occurs below 30 K. Inset: The magnetization versus field for $0 < H < 5$ T, obtained using a SQUID magnetometer. The slopes of these lines are plotted as open circles in (a), which show that the Curie ''tail'' is saturated in a small magnetic field. (b) Magnetization versus field for a polycrystalline sample of $BaVS_3$, obtained in a 50 T pulsed field magnet. The units of magnetization are arbitrary. The magnetization is essentially linear up to very high fields.

tals ($0.1 \times 0.1 \times 1 - 2$ mm) were grown from the resulting powder by precipitation from both $BaCl_2$ (Ref. 1) and Te (Ref. 14) fluxes following procedures in the literature. (The starting masses used for the Te flux growth were 1 g $BaVS_3$, 20 mg S, and 10 g Te.) Sulfur-deficient samples are known to exhibit ferromagnetism below 15 K; in order to ensure complete sulfur uptake, samples were annealed in the presence of sulfur vapor, as discussed in earlier reports.[2] There are basically two measures of sample quality: the Curie ''tail'' seen below 30 K should be small, and the resistivity above 150 K should be monotonically increasing (metallic). We found that as long as both conditions are satisfied, the maximum value of the susceptibility $\chi(T_{MI})$ is approximately equal to 3×10^{-3} emu/mol, as stated in most past reports; for polycrystalline samples that do not show a resistance minimum near 150 K but which do show a small Curie tail, the value of $\chi(T_{MI})$ is typically much smaller. All results reported here are for samples which meet these two conditions. We found that crystals grown in either $BaCl_2$ or Te flux gave essentially identical results for the resistivity, susceptibility and magnetoresistance.

The low field ($H = 0.1$ T) susceptibility and low field ($H < 5$ T) magnetization reported here were measured in a SQUID magnetometer. The zero-field resistivity and the Hall effect were measured in the range $15 - 300$ K using a helium-flow cryostat; an LR400 resistance bridge with operating frequency 16 Hz was utilized for both measurements. For the Hall measurement, small misalignment voltages were compensated electronically and the magnetoresistance was cancelled by reversing the polarity of the field (± 1 T). The high field magnetization was measured at the National High Magnetic Field Laboratory's Pulsed Field Facility at Los Alamos (NHMFL/LANL). The magnetoresistance was measured using four-probe geometry and an ac resistance bridge in fields $0 < H < 18$ T generated in a superconducting magnet. The primary error in the magnetoresistance arises from temperature drift during field sweeps, including the drift due to the small but nonzero magnetoresistance of the temperature control sensor. To measure the temperature, we used a Cernox 1050 thermometer for which the field sensitivity is of order $1 - 2 \times 10^{-4}$ K/T^2. We estimate the error in Fig. 3 due

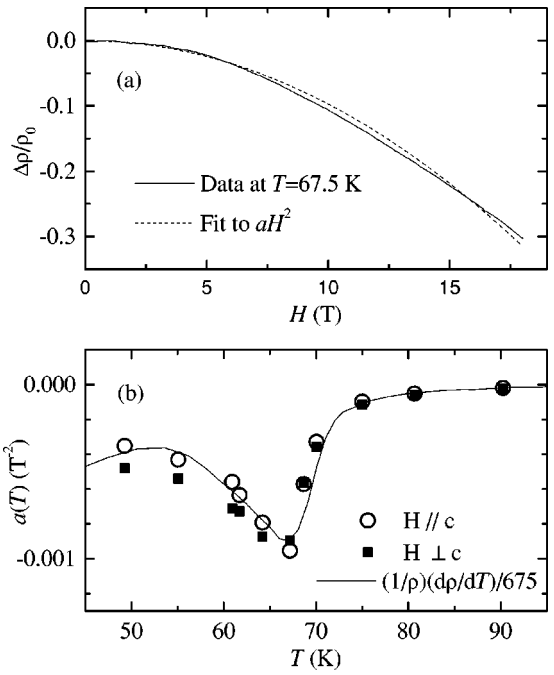

FIG. 3. (a) The magnetoresistance $\Delta\rho/\rho$ of a single crystal of $BaVS_3$ versus magnetic field, at 67 K ($\approx T_{MI}$ for this sample). The current I and the magnetic field are both parallel to the c axis. The magnetoresistance varies as $a(T)H^2$ for $0<H<18$ T. (b) A plot of the coefficient $a(T)$, derived from data similar to that of (a), versus temperature in the vicinity of the metal insulator transition. Open circles have the applied field perpendicular to the c axis, and solid squares denote the field perpendicular to c. I runs along c in both cases. The solid line is a plot of the logarithmic derivative of resistivity $(1/\rho)d\rho/dT$ scaled by the factor (1/675) $[K/T^2]$.

to temperature drift (including the drift due to the control sensor magnetoresistance) to be 10–15 %. Magnetization measurements were performed in a 50 T pulsed magnet; details are given in an earlier publication.[15] The signal is proportional to the magnetization, but is not calibrated in absolute units.

III. RESULTS AND ANALYSIS

The zero-field resistivity of a single crystal for current along the c axis is shown in Fig. 1(a). The results are in good accord with earlier work, showing the steep increase in resistivity at 70 K and exhibiting a resistance minimum near 150 K [Fig. 1(a), inset] that attests to the sample quality. The susceptibility [Fig. 2(a)] is also in good accord with earlier results. The magnetization versus magnetic field for $0<H<5$ T measured at several temperatures for a polycrystalline sample is shown in the inset to Fig. 2(a); the values of susceptibility derived from the magnetization data are shown as open circles in Fig. 2(a). The Curie tail seen in the low field (0.1 T) susceptibility data is clearly suppressed by a very small field, which strongly supports the contention that it is an extrinsic effect. For larger fields [Fig. 2(b)] the magnetization is linear within experimental error up to fields as high as 45 T [Fig. 2(b)].

The Hall effect [Fig. 1(b)], measured for a polycrystal sample, is negative above 240 K and positive below, i.e., it changes sign at the transition to the orthorhombic structure. In the metallic state it increases with decreasing temperature to a value $\approx 6\times10^{-10}$ m^3/C at $T\approx T_{MI}$, then increases by at least two orders of magnitude in the insulating state.

In Fig. 3(a) we show the magnetoresistance $\Delta\rho(H)/\rho(0)$ for $H\|c$ at $T=67$ K for a single crystal sample; the magnetoresistance varies as $a(T)H^2$. We plot the coefficient $a(T)$ for $H\|I\|c$ and $H\perp I\|c$ in Fig. 3(b). We note first of all that the data for $H\|I$ and $H\perp I$ differ by no more than 10 -20 %. This is also the degree to which the magnetoresistance reproduces for measurements in different samples. Hence, within experimental error, the magnetoresistance is independent of the orientation of the field for $I\|c$. Second, the coefficient $a(T)$ varies proportionally with the logarithmic derivative of the resistivity $(1/\rho)d\rho/dT$; from Fig. 3(b) it can be seen that $a(T)\approx\alpha(1/\rho)d\rho/dT$ where $\alpha=(1/675)$ $[K/T^2]$. This proportionality can be understood by noting that the measurement of $\Delta\rho/\rho\propto H^2$ implies that (to lowest order) $T_{MI}(H)\approx T_{MI}(0)-(\alpha/2)H^2$, and that the major effect on the resistivity is to shift the curve down in temperature, i.e., $\rho(T;H)=\rho[T-T_{MI}(H)]$, where the functional form of $\rho[T-T_{MI}(H)]$ is independent of $T_{MI}(H)$. If we further assume an elliptic form for the phase diagram in magnetic field, i.e.,

$$\left[\frac{T_{MI}(H)}{T_{MI}(0)}\right]^2+\left[\frac{H}{H_0}\right]^2=1,$$

then a power series expansion of $\rho(T;H)$ in powers of H leads to the conclusion that for small H

$$\frac{\Delta\rho}{\rho}=\frac{3}{2}\frac{T_{MI}(0)}{H_0^2}\left(\frac{1}{\rho}\frac{d\rho}{dT}\right)H^2.$$

From these equations we then obtain an estimate of the zero-temperature critical field: $(3/2)T_{MI}(0)/H_0^2=(1/675)$ $[K/T^2]$. For our single-crystal sample, $T_{MI}(0)=67$ K, so $H_0\simeq 260$ T. For $g=2$ and $s=\frac{1}{2}$, appropriate for the $3d^1$ states of $BaVS_3$, this is equivalent to an effective temperature $T_{eff}=g\mu_B sH_0/k_B=175$ K. This value of critical field is comparable to the spin gap estimated from recent NMR Knight shift measurements[7] where it was found that $\chi_{spin}\propto T^{1/2}\exp(-\Delta_s/T)$ where $\Delta_s=240$ K.[16]

IV. DISCUSSION

These results allow us to place several constraints on the theory of the metal-insulator transition in $BaVS_3$. First, our magnetization results show that the Curie tail seen in the low temperature susceptibility saturates in a rather small magnetic field, which implies (along with the lack of reproducibility of the tail from sample to sample) that it is an extrinsic effect. This rules out an earlier hypothesis[6] concerning the origin of the hyperfine fields seen below T_x; this hypothesis was based on the assumption that the Curie tail arises from 19% V ions within $BaVS_3$ which remain magnetic and then order below T_x.

Our magnetoresistance data have allowed us to estimate the critical field for the MI transition, $H_0\simeq 260$ T. This is a very large critical field; it means that magnetic fields have only a very weak effect on the MI transition. The fact that the magnetization is linear up to large (45 T) magnetic fields

is consistent with this observation; if the critical field were sufficiently small, then a 45 T field would drive the material back into the metallic, paramagnetic state, at least for T close to T_{MI}. Such a large critical field gives further support to the evidence mentioned above (that no static local fields are observed in NMR, Mossbauer and inelastic spin-flip scattering, and that no magnetic reflections are observed in diffraction experiments) that models of the MI transition based on the onset of dominating antiferromagnetic interactions are incorrect. For example, a value $\gamma=0.46$ in the formula

$$1-\frac{T_c(H)}{T_c(0)}=\gamma\left(\frac{g\mu_B sH}{k_B T_c(0)}\right)^2$$

has been interpreted as important evidence that the 14 K transition in $CuGeO_3$ is a spin-Peierls transition;[17] for $BaVS_3$, due to the large critical field, the value of γ (0.07) is much smaller. Magnetic fields also have a very dramatic effect on spin-density wave (SDW) transitions, including field induced SDW phases.[18]

Some groups[2,6] have suggested that the MI transition at 70 K is not a true 3D phase transition, but is a 1D antiferromagnetic ''transition'' below which no static long-range order occurs, but only dynamic short-range order. This would explain the absence of static hyperfine fields, and of magnetic reflections in diffraction. The 3D transition then should occur at a lower temperature $T_{3\mathrm{D}}$, where the ratio $T_{3\mathrm{D}}/T_{\mathrm{MI}}$ is set by the ratio of the interchain to the intrachain coupling; and hence the transition at $T_x=30$ K might be identified with the 3D transition. This idea is roughly consistent with the transition temperature as deduced from the spin gap and the weak-coupling model of Lee, Rice, and Anderson.[19] These authors have shown that a 3D transition $T_{3\mathrm{D}}$ should be reduced from the weak-coupling mean-field transition temperature T_{MF} (given by $2\Delta=3.5k_BT_{\mathrm{MF}}$) by about a factor of 4, that is, $T_{3\mathrm{D}}\simeq T_{\mathrm{MF}}/4$. For our estimate ($\Delta_{\mathrm{spin}}=175$ K) of the spin gap in $BaVS_3$, we have $T_{\mathrm{MF}}=100$ K and $T_{3\mathrm{D}}=25$ K, which is consistent with the estimates of $T_{\mathrm{MI}}\simeq 70$ K and of $T_x=30$ K, respectively. However, at a 1D ''transition'' the thermodynamic quantities should show broad rounded features,[20] and sharp features should be observed at the 3D transition. In $BaVS_3$, the opposite occurs: the susceptibility,[4,21] thermal expansion,[21] and specific heat[5] show very sharp features at T_{MI}, whose widths are much smaller than T_{MI} and no anomalies whatsoever are seen in the thermodynamic and transport measurements at T_x. Hence, in agreement with Nakamura *et al.*,[7] we think this scenario is unlikely.

Even if the MI transition does not coincide with the dominance of antiferromagnetic (SDW, Peierls) interactions, it does involve the V (d^1) $s=1/2$ spin degrees of freedom in an essential way. The sharp cusp in the susceptibility and the fact that the entropy change is nearly equal to $R\ln 2$ at the transition gives evidence for this spin involvement. The fact that our estimate of the critical field ($H_0\simeq 260$ T equivalent to 175 K) is comparable in magnitude to the spin gap ($\Delta_s=240$ K) measured in NMR gives further evidence, because it suggests that the critical field is the field at which the spin gap is closed. We note that the charge gap in these compounds is substantially larger than the spin gap. Assuming that the gap is symmetric around the Fermi energy, the charge gap given by the activation energy for the conductivity is estimated as $\Delta_c\approx 500$ K,[21] consistent with the photoemission threshold [62 meV (Ref. 10) or 720 K]. For a simple band gap the magnetic field should close the gap when $g\mu_B sH=\Delta_c$; for $\Delta_c=500$ K, this would imply a critical field of 750 T. The fact that the spin gap is substantially smaller than the charge gap means that the insulating phase does not arise from a simple band gap and that Coulomb correlations (of the Mott-Hubbard kind) may be important in $BaVS_3$.

In quasi-1D systems where $T_{3\mathrm{D}}\ll T_{\mathrm{MF}}\sim\Delta$, 1D fluctuations into the gapped state occur above the 3D transition at $T_{3\mathrm{D}}$. The photoemission experiments[10] in $BaVS_3$ indeed show that a gap begins to develop at temperatures $T>T_{\mathrm{MI}}$. For charge-density wave transitions, such fluctuations give rise to a negative temperature derivative of the resistivity $d\rho/dT<0$ for $T>T_{3\mathrm{D}}$;[22] hence they may be the origin of the negative $d\rho/dT$ observed for $T_{\mathrm{MI}}<T<150$ K in $BaVS_3$ [Fig. 1(a), inset]. Our Hall effect measurements [Fig. 1(b)] also show that R_H exhibits a precursive increase with decreasing temperature at temperatures well above T_{MI}. Hence, while we think it implausible that the MI transition is a 1D ''transition,'' it seems very plausible that 1D fluctuations occur above T_{MI}.

One way this situation could occur would be if the MI transition were due to a CDW instability; this assumes that previous diffraction experiments[3,8] were insufficiently sensitive to resolve the associated superlattice spots. The possibility of a $4k_F$ CDW instability has been suggested by other authors.[10,13] Magnetic field should have only a negligible effect on a CDW transition, consistent with the large observed critical field. This scenario is consistent with the weak-coupling equation mentioned above, except that the appropriate gap is the estimated charge gap: $T_{3\mathrm{D}}=2\Delta_c/(4\times 3.5)=70$ K with $\Delta_c=500$ K. The value of the ratio $2\Delta_c/T_{\mathrm{MI}}\simeq 14$ that we observe in $BaVS_3$ is comparable to the values observed in other quasi-1D CDW materials with MI transitions, such as NbS_3, $NbSe_3$, and others.[22] The problem with this scenario is (as mentioned above) that the band structure calculations[11] indicate that the cross-chain coupling is not particularly small, due to hybridization with the sulfur atoms. Furthermore, for a CDW transition we don't expect in general that $\Delta_s\ll\Delta_c$ as observed for $BaVS_3$; more generally, for a CDW transition we do not expect the V (d^1)$s=1/2$ spin to be involved in such an essential way as is suggested by the data.

Another possibility is that the model proposed by Massenet[12] is correct. In this model, the sharp cusp in the susceptibility, the loss of the V (d^1) $s=1/2$ spin entropy and the lack of static local fields for $T<T_{\mathrm{MI}}$ all reflect the demagnetization which occurs when the gap is established for $T<T_{\mathrm{MI}}$. Since the band theory shows that a simple gap is not expected for the $Cmc2_1$ symmetry, electron correlations would play a crucial role in causing the transition. Such correlations can cause the spin gap to be smaller than the charge gap,[13] as observed. For this model the precursive behavior of the order parameter could arise[21] because the transition is isomorphic (i.e., there is no change in symmetry). This allows for the possibility that the transition is not second order, but is analytic (with no singularity in the free energy), as occurs in a liquid-vapor transition for pressures slightly be-

yond the critical pressure. In this situation, the order parameter can evolve continuously above T_{MI}. We noted in an earlier publication[21] that this situation occurs near the high temperature metal-insulator transition in $V_{2-x}Cr_xO_3$, so it is not without precedent. A large value of energy gap relative T_{MI} is also not without precedent; in V_2O_3, the band gap is estimated as being $0.2-0.6$ eV $(2300-7000$ K), while $T_{MI}\sim 200$ K.[13]

V. CONCLUSION

Our primary result is that magnetic fields as large as 45 T have negligible effect on the metal-insulator transition in $BaVS_3$. Taken together with the fact that no static local fields are observed in NMR, Mossbauer and spin-flip scattering, the large value (260 T) that we estimate for the critical field to destroy the insulating state suggests that it is very unlikely that the MI transition involves a transition into a magnetic state such as a spin-density wave or spin-Peierls state. In addition we have observed considerable precursive activity in the Hall coefficient for temperatures $T>T_{MI}$ which (together with earlier observations of precursive behavior in the photoemission and resistivity) suggests either that the precursive behavior is due to 1D fluctuations or it arises because the transition is isomorphic and analytic. Measurement of the anisotropy of the resistivity is needed to establish the degree of one dimensionality in $BaVS_3$; in addition, future work should include a search for CDW satellites through electron diffraction.

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