

# Metal-insulator crossover behavior at the surface of NiS<sub>2</sub>

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We have performed a detailed high-resolution electron spectroscopic investigation of NiS<sub>2</sub> and related Se-substituted compounds, NiS<sub>2-x</sub>Se<sub>x</sub>, which are known to be gapped insulators in the bulk at all temperatures. A large spectral weight at the Fermi energy of the room temperature spectrum, in conjunction with the extreme surface sensitivity of the experimental probe, however, suggests that the surface layer is metallic at about 300 K. Interestingly, the evolution of the spectral function with decreasing temperature is characterized by a continuous depletion of the single-particle spectral weight at the Fermi energy and the development of a gap-like structure below a characteristic temperature, providing evidence for a metal-insulator crossover behavior at the surfaces of NiS<sub>2</sub> and of related compounds. These results provide a consistent description of the unusual transport properties observed in these systems.

## I. INTRODUCTION

The properties of two-dimensional (2D) electron systems, particularly in the presence of strong interaction and disorder, are a matter of considerable interest. While the ground state of a noninteracting disordered 2D electron system is predicted to be an insulator,<sup>1</sup> a recent experiment involving semiconductor inversion-layer devices has suggested a metal-insulator transition (MIT) in such a system.<sup>2</sup> This initiated a number of studies<sup>3</sup> that aim at understanding whether such a quantum phase transition can occur in two dimensions. The question arises if electron interaction can lead to a metallic ground state even in a disordered 2D system. This has actually been suggested assuming that electron interaction opposes the effects of disorder.<sup>4</sup> A recent theoretical study, however, predicts the absence of a true MIT in a 2D system in the simultaneous presence of disorder *and* interaction and proposes instead a crossover behavior.<sup>5</sup> The very nature of the mentioned systems based on Si metal-oxide semiconductor field effect transistors and various semiconducting heterostructures with very low electron densities,<sup>2</sup> however, does not allow one to study their electronic structure by photoemission (PE), a method that has been applied successfully to MITs in 3D systems.<sup>6</sup>

The surface of NiS<sub>2-x</sub>Se<sub>x</sub> ( $x \leq 0.4$ ) represents a well-suited system for addressing several central questions concerning the ground-state electronic structure of 2D systems as well as for studying the evolution of the electronic structure by PE across a metal-insulator-like transition in a strongly correlated 2D system. While for bulk NiS<sub>2-x</sub>Se<sub>x</sub>, with  $x > 0.4$ , an  $x$ - and  $T$ -driven MIT is well known and has

been the subject of several studies,<sup>7-11</sup> we focus here on  $x \leq 0.4$ , where remarkable transport properties have been observed: While optical studies show that the bulk material is a gapped insulator at all  $T$ ,<sup>12</sup> this is not reflected in the dc resistivity data.<sup>13,14</sup> Near room temperature, the resistivity exhibits an activated behavior, with a gap of  $\approx 0.2$  eV, in agreement with optical conductivity/reflectivity measurements.<sup>12</sup> Below 120 K, however, no activated behavior is observed, and the resistivity even decreases with decreasing  $T$ . On the basis of detailed transport and Hall-effect measurements on samples with different surface-to-volume ratios, it was concluded that this resistivity behavior is due to a metallic surface layer with a thickness of a few unit cells.<sup>13</sup> This is remarkable in view of the inevitable presence of disorder in real systems that should ensure a 2D insulating ground state.<sup>1</sup> And in fact, this is suggested by the recent observation of a steep increase in resistivity at low temperatures,<sup>14</sup> rendering the formation of a metallic surface layer at higher temperatures<sup>13</sup> even more intriguing.

Many questions arise from these experimental observations, e.g., how the low- $T$  insulating phase connects to the high- $T$  metallic phase, whether there is a real phase transition or rather an unusual crossover behavior, and whether the surface is metallic at higher temperatures or the transport properties have to be interpreted in a different way. It is known that the bulk (3D) insulating behavior of NiS<sub>2</sub> at all temperatures is due to the formation of a Mott-Hubbard gap in the single-particle excitation spectrum driven by electron-electron interaction, while so far there is no information on the nature of the low- $T$  insulating phase of the 2D surface of this system. One may ask if its insulating state is induced by

electron-electron interaction or by Anderson localization in view of the increased effect of disorder in the lower dimension.

Here we present the results of a high-resolution PE study of the surface electronic structure of  $\text{NiS}_2$ , giving evidence for a metallic state at high temperatures as suggested by the resistivity data. In addition, we find that the unusual transport properties of this compound and of the related  $\text{NiS}_{2-x}\text{Se}_x$  system, with  $x \leq 0.4$ , are reflected in  $T$ -dependent changes of the surface electronic structure. With electron interaction and disorder driving the system towards localization instead of opposing each other, the ground state of this 2D system is proposed to be an Anderson-localized insulator despite the presence of strong electron interaction.

## II. EXPERIMENT

The experiments were performed on both single crystals and sintered polycrystalline samples; the details of sample preparation were described earlier in Ref. 9. All samples were single phase as checked by x-ray diffraction, and the stoichiometries were confirmed by an energy-dispersive x-ray analysis in a scanning electron microscope. Photoelectron spectroscopic experiments were carried out in spectrometers that are equipped with Scienta analyzers, Gammadata vacuum ultraviolet (VUV) lamps and monochromators to suppress the satellite radiations. We have used the high-intensity He I VUV radiation ( $h\nu = 21.2$  eV) for these experiments. The total-system energy resolutions were set at 8 meV full width at half maximum (FWHM) for most of the measurements. The samples were cooled by continuous-flow He cryostats and the temperature was controlled within  $\pm 0.5$  K at any given temperature. The Fermi energy ( $E_F$ ) was determined at each temperature from spectra of polycrystalline Ag in electrical contact with the sample. Single-crystalline samples were primarily cleaved and used for angle-resolved band mapping at a few selected temperatures, while scraped polycrystalline samples were used to study in more detail the variation of the spectral weight at  $E_F$  as well as to check reproducibility in case of thermal cycling.

## III. RESULTS AND DISCUSSION

In order to address subtle temperature-dependent changes in the electronic structure of any material with respect to the transport properties, it is most relevant to monitor the spectral changes at and near the Fermi energy,  $E_F$ . However, this requires a reliable normalization procedure before different spectra can be compared. In general, two distinct approaches have been adopted in the literature for this purpose. In one approach, different spectra are scaled to match at a given binding energy (BE); the specific BE (typically 0.5–0.6 eV) is normally chosen to be sufficiently removed from  $E_F$ , such that subtle changes in the electronic structure of the system that can be induced by temperature, are not expected to have any effect in the vicinity of the chosen BE. In the other method, the total integrated area under the spectrum over a certain energy window is normalized, referring to the conservation of the number of electrons in the system. In the

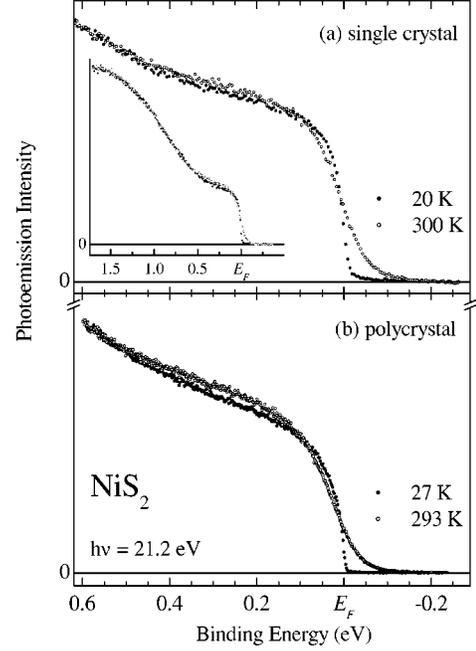


FIG. 1. PE spectra of  $\text{NiS}_2$  from (a) single-crystalline and (b) polycrystalline samples at the given temperature. The inset illustrates the normalization procedure over a larger range of binding energies.

present system, we find that both of these approaches converge and lead to the same result, as illustrated in the inset to Fig. 1(a) for the spectra of  $\text{NiS}_2$  recorded at two extreme temperatures of 20 and 300 K. The spectra are normalized at a 0.6-eV BE; interestingly, this normalization leads to the matching of the two spectra down to 1.6 eV starting from the 0.5-eV BE. This extensive matching over the entire high-BE window ensures that the two spectra shown in the inset of Fig. 1(a) have almost the same total integrated areas. In fact, a normalization based on total integrated areas leads to spectra essentially indistinguishable from those given in the inset. It is also evident that there are no gross changes in the spectra as a function of  $T$ ,<sup>15</sup> which could influence the normalization procedure.

In fact, temperature-dependent changes occur only close to  $E_F$  over a narrow energy range, as illustrated in the main panels of Figs. 1(a) and 1(b) for single-crystalline and polycrystalline samples, respectively. Interestingly, the data from single- and polycrystalline samples are almost identical. In order to understand this observation, we have carried out a detailed angle-resolved PE study of single-crystalline samples (not shown here). While we found extensive dispersions of the main intense valence band spectral region appearing at higher binding energies, our angle-resolved measurements established a relative insensitivity of the spectral features close to  $E_F$  with respect to the angle of detection. This is consistent with the striking similarity of the spectral features from the single-crystalline and polycrystalline samples.

In order to discuss the changes close to  $E_F$  in more detail, in Fig. 2 we show (main panel) a set of representative spectra covering a narrow energy scale at various temperatures. Far

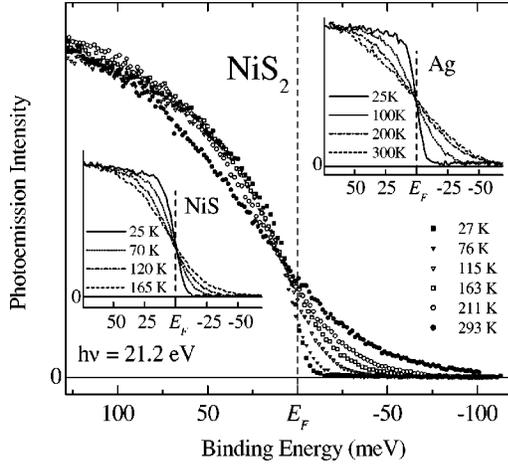


FIG. 2. PE spectra of polycrystalline  $\text{NiS}_2$ , recorded at various temperatures. Insets: corresponding spectra for polycrystalline Ag and polycrystalline NiS.

below  $E_F$  ( $\text{BE} > 0.5$  eV), the spectra are essentially identical, as already illustrated in Fig. 1 for the two extreme temperatures. In the BE region between 100 and 500 meV, however, there is a systematic and continuous, though small, depletion of spectral weight with decreasing temperature, suggesting some changes in the spectral weight over this energy window as a function of  $T$ . However, these high energy states are not directly related to the transport properties. Therefore, we focus here on the electronic states close to  $E_F$ , responsible for the transport properties, which exhibit remarkable changes with temperature. It is well known that in the case of a metal the spectral weight at and near  $E_F$  changes significantly with temperature, which is easily understood in terms of the Fermi-Dirac statistics. The changes observed here, however, are distinctly different from the Fermi-Dirac type, as we shall show below by a more detailed analysis.

Already without recourse to a detailed analysis, several important features can be recognized, which are particularly interesting in connection with the transport data reported for  $\text{NiS}_2$ . On the basis of the shown PE spectra, there is compelling evidence for the surface of  $\text{NiS}_2$  to be metallic at room temperature. These proofs are as follows. (i) There is a large spectral weight at  $E_F$  in the room-temperature spectrum. (ii) There is a continuous and substantial spectral weight up to about 100 meV above  $E_F$  in the same spectrum. (iii) The spectral weight *above*  $E_F$  shows a characteristic Fermi-Dirac type depletion of spectral intensity with decreasing temperature, spread over the expected energy scale related to the thermal energy. In view of the total experimental energy resolution of the present PE experiments of 8 meV (FWHM), none of these observations can be explained by broadening due to finite resolution. The observations are clearly incompatible with the optical data that show  $\text{NiS}_2$  to be a wide-gap insulator. On the other hand, the results are consistent with the Hall-effect data<sup>13</sup> and suggest that the surface of  $\text{NiS}_2$  is metallic, particularly when considering the high surface sensitivity of PE with a mean sampling depth of  $\approx 7$  Å. Thus, the surface layer of  $\text{NiS}_2$ , only a few

unit cells thick, has metallic character near room temperature, while the bulk of the sample, inaccessible to PE, remains an insulator.

It is interesting to note that the variation of the spectral function with temperature, shown on the main panel of Fig. 2, is very different from that of a normal metal. This can be inferred from the inset that displays corresponding spectra of polycrystalline Ag metal taken with the same setup. Here, the observed  $T$  changes in the spectral function of Ag are readily understandable in terms of Fermi-Dirac (FD) statistics alone. With an essentially constant density of states in the vicinity of  $E_F$  ( $\mathcal{D}(E_F)$ ), this causes the spectrum to recover the weight lost *above*  $E_F$  with decreasing temperature almost in a symmetrical manner and immediately *below*  $E_F$ , thus preserving the total spectral weight. Another consequence of  $\mathcal{D}$  being unchanged with  $T$  is that all spectra of Ag go through a common point at  $E_F$  in spite of changing  $T$ , since the Fermi-Dirac statistics does not affect the spectral weight *at* the Fermi energy. An almost identical behavior of the spectral function is also observed for NiS (see second inset in Fig. 2). In contrast, the behavior of the PE spectra of  $\text{NiS}_2$  is qualitatively different. In particular, the redistribution of the spectral function *below*  $E_F$  takes place over a much wider energy range than one would expect explicitly on a thermal scale, while *above*  $E_F$  it appears to be controlled by  $T$ . Hence, the changes in the spectral function of  $\text{NiS}_2$  cannot be understood in terms of a *fixed*  $\mathcal{D}$  around  $E_F$  and FD statistics, in contrast to normal metals. Instead, we are forced to conclude from the raw data that the single-particle excitation spectrum of the surface of  $\text{NiS}_2$  is characterized by a temperature dependence of  $\mathcal{D}$  itself.

Due to the presence of thermal broadening and resolution broadening, a quantitative estimate of  $\mathcal{D}(E_F)$  at various temperatures cannot be obtained without recourse to simulations of the spectra in terms of a model  $\mathcal{D}(E)$ . With the high energy resolution achieved in the present PE experiments, thermal broadening is by far the dominant effect. This contribution, however, can be readily removed from the spectra by assuming a symmetric  $\mathcal{D}(E)$ ,<sup>16</sup> or by dividing the raw spectra by the FD distribution function.<sup>17</sup> We illustrate the results of the latter analysis by the inset in Fig. 3, demonstrating a remarkable depletion of spectral weight with decreasing temperature over a wide energy range, with the strongest effects at  $E_F$ . As pointed out further above, the corresponding amount of spectral weight is recovered almost uniformly distributed over the BE range from 0.15 to 0.45 eV, with the effects of FD distribution being virtually absent at such a high BE.

Besides these two methods, the  $T$  dependence of  $\mathcal{D}(E_F)$  was also obtained by direct fitting of the spectra of Fig. 2 with a  $\mathcal{D}(E)$  described by a polynomial function, multiplied by the FD distribution at a given  $T$  and convoluted by a Gaussian for the known resolution. All these different analyses result in similar values of  $\mathcal{D}(E_F)$ , demonstrating an insensitivity on details of the model. The resulting  $\mathcal{D}(E_F)$  are plotted in the main part of the upper panel of Fig. 3 as a function of  $T$ , with error bars that contain the variations of  $\mathcal{D}(E_F)$  from the different analyses.  $\mathcal{D}(E_F)$  increases slightly with decreasing temperature from 297 to 260 K, and then

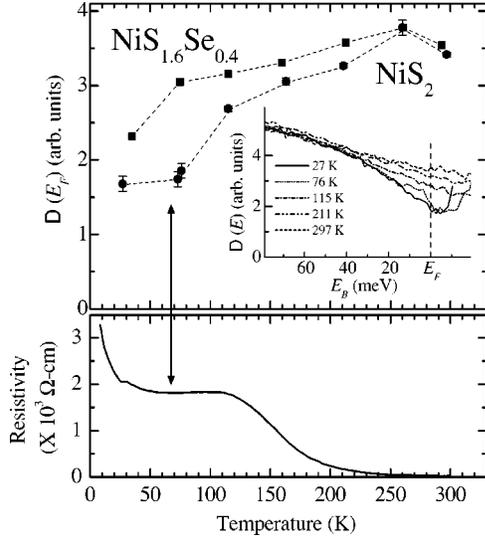


FIG. 3. Upper panel:  $T$  dependence of  $\mathcal{D}(E_F)$  of  $\text{NiS}_2$ , extracted from the spectra of Fig. 2; data for  $\text{NiS}_{1.6}\text{Se}_{0.4}$  are included. Inset:  $\mathcal{D}(E)$  for  $\text{NiS}_2$  at various temperatures, obtained as described in the text. Lower panel: Resistivity of  $\text{NiS}_2$  as a function of  $T$ , adopted from Ref. 14.

decreases progressively down to 115 K. Between 115 and 75 K, there is a more pronounced decrease in  $\mathcal{D}(E_F)$ , suggesting the opening of a gaplike structure in the single-particle excitation spectrum; below 75 K, the gaplike structure is fully developed and  $\mathcal{D}(E_F)$  remains essentially unchanged. The formation of an energy gap in the electronic structure of  $\text{NiS}_2$  thus occurs in the very temperature range, where the resistivity is known to increase.<sup>14</sup> In order to illustrate this, we show in the lower panel of Fig. 3 the resistivity behavior of  $\text{NiS}_2$  as a function of temperature, adopted from Ref. 14. The high temperature resistivity clearly shows the activated behavior of the bulk in the temperature range 150–300 K; the metallic conductivity of the surface region causes a perceptible deviation from the activated behavior below 150 K, and the most pronounced metallic behavior below about 110 K. However, with a further lowering of the temperature, the resistivity of this surface layer begins to increase where the gaplike structure forms in  $\mathcal{D}(E_F)$ , as indicated by the arrow in Fig. 3.

The interesting change in  $\mathcal{D}(E_F)$  with  $T$  is not specific to  $\text{NiS}_2$  alone. As mentioned before, the related compounds  $\text{NiS}_{2-x}\text{Se}_x$  are also bulk insulators, with the resistivity data suggesting a 2D metallic overlayer for  $x \leq 0.4$ .<sup>14</sup> PE spectra of  $\text{NiS}_{2-x}\text{Se}_x$ , with  $x=0.3$  and  $0.4$ , were recorded at various temperatures and reveal a behavior similar to that of  $\text{NiS}_2$ . Data for  $\text{NiS}_{1.6}\text{Se}_{0.4}$  are included in Fig. 3, and they clearly resemble the behavior of  $\text{NiS}_2$ , with a tiny initial increase in  $\mathcal{D}(E_F)$  between 297 and 260 K, followed by a moderate decrease down to 75 K, and then the signature of a gap at  $\approx 35$  K. The PE results for  $\text{NiS}_{1.7}\text{Se}_{0.3}$  (not shown here) are again similar, with the formation of a gaplike structure at  $\approx 55$  K. Interestingly, the gaplike structures in the single-particle excitation spectrum form at progressively lower temperatures with increasing  $x$ , i.e., at about 75, 55, and 35 K for  $x=0, 0.3$ , and  $0.4$ , respectively. This follows the trend of

decreasing  $T$ , at which the upturn in resistivity has been observed for  $\text{NiS}_{2-x}\text{Se}_x$ ,<sup>14</sup> and establishes a close relationship between the transport properties and the observed decrease of  $\mathcal{D}(E_F)$ .

The details of the temperature-dependent surface electronic structure, as derived from the PE spectra of  $\text{NiS}_2$  and related compounds, further suggest several important implications. We first note that  $\mathcal{D}(E_F)$  for any of the three compounds studied, in spite of the pronounced gaplike structure, does not vanish completely, even at the lowest  $T$ . While the gaplike structure in  $\mathcal{D}(E)$  at the low- $T$  limit is presumably driven by strong correlation effects, the persistence of a finite  $\mathcal{D}(E_F)$  suggests that the ground state is not a Mott insulator with a fully developed energy gap; it rather indicates that Anderson localization driven by disorder is the origin of the insulating behavior.

The overall dependence of  $\mathcal{D}$  on  $T$  displayed in the upper panel of Fig. 3 shows that the gaplike structure disappears over a relatively narrow temperature range of  $\approx 40$  K rather than by a gradual filling of the gap over a larger  $T$  interval. This cannot be explained by thermal excitations of charge carriers alone implying that the underlying electronic structure itself changes rapidly, accompanying the metal-insulator crossover over a narrow  $T$  range. This behavior might be related to a temperature-dependent screening of correlation effects by itinerant electrons. At high temperatures the screening is highly effective, leading to a less correlated state and consequently to the disappearance of the gaplike structure in the single-particle excitation spectra. At lower temperatures, however, an Anderson localization of the electrons leads to less effective screening, with a gap forming in the excitation spectrum. While this may possibly be a continuous changeover with temperature, as suggested by the slow change of  $\mathcal{D}(E_F)$  at higher  $T$ , it is most remarkable in a narrow temperature interval, where a change in the transport properties was observed. If indeed such a screening mechanism is relevant, it has to have a nontrivial dependence on  $T$  beyond the thermal excitation of mobile charge carriers.

At this point, it is tempting to rationalize the presence of metal-like surface layers in these systems. One obvious possibility is a deviation of the surface stoichiometry from the bulk, leading to a doping of the surface layer with charge carriers. However, the large  $\mathcal{D}(E_F)$  observed near room temperature would correspond to a very high doping level and hence to a significant deviation from stoichiometry. This is incompatible with the intensity ratios of core-level PE spectra from Ni, S, and Se, which were found to follow the nominal bulk compositions in all samples. Moreover, if the doped charge carriers arising from nonstoichiometry were indeed mobile, without altering the underlying electronic structure, there would be no reason for the charge carriers to reside only in the surface layer.

A possible explanation is obtained from the unusual properties of  $\text{NiS}_2$  compared to  $\text{CoS}_2$  and  $\text{CuS}_2$ . While  $\text{NiS}_2$  is a bulk insulator, the latter two are bulk metals.<sup>18</sup> The exceptional behavior of  $\text{NiS}_2$  shows up also in the lattice parameters being *larger* than expected from their systematic variation in the pyrite family,  $M\text{S}_2$ , with  $M=\text{Mn-Zn}$ .<sup>18</sup> The unusually expanded lattice of  $\text{NiS}_2$  is expected to lead to a

reduction of the bandwidth, driving bulk NiS<sub>2</sub> insulating, in contrast to the metallic ground states of CoS<sub>2</sub> and CuS<sub>2</sub>. This point of view is supported by the fact that bulk NiS<sub>2</sub> turns metallic at a pressure of 46 kbar.<sup>19</sup> Given this relation between the lattice parameters and the metallic character of bulk NiS<sub>2</sub>, a reduction of the lattice parameters at the surface could lead to an altered surface electronic structure of NiS<sub>2</sub> with the tendency towards a more metallic character. And in fact, preliminary *ab initio* full-potential calculations of the surface electronic structure of NiS<sub>2</sub> (Ref. 20) indicate a reduction of the lattice parameters and consequently an increase in  $d$  bandwidth at the surface for several crystallographic surfaces.

To summarize, we propose the following scenario: Lattice relaxation near the surface region of NiS<sub>2</sub> and related compounds tends to drive the surface metallic, which, however, is inhibited by the 2D nature of the surface layer; instead Anderson localization leads to an insulating ground state of the surface layer. At higher temperatures, crossover towards a metallic behavior occurs, as evidenced by the transport properties. This crossover is characterized by the disappear-

ance of the gaplike feature in the electronic structure around  $E_F$  and by a rapid increase in  $\mathcal{D}(E_F)$ , driven by a decrease of the effective Coulomb interaction strength as well as of the disorder potential due to screening by a larger number of thermally excited electrons. However, the narrow temperature range, in which this strong change in the electronic structure occurs, is remarkable and requires further theoretical explanation.

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