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REVIEWS

Electrical Transport in Vanadium Dioxide Nanostructures – New Physics and Potential Applications

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Abstract | VO_2 is an interesting correlated electron material that exhibits an above room temperature metal-insulator transition that spans more than four orders of magnitude in conductivity. This system has been studied extensively due to the fundamental questions about the mechanism behind the metal-insulator transitions and the possibility of utilizing these nanostructures in a variety of applications such as electrical switches, optical switches and bolometers. The tunability of the transition temperature, hysteresis widths, and magnitude of hysteresis using a slew of experimental techniques make this system an interesting material to study. In this review, we present an experimental overview of the electrical transport properties of vanadium oxide nanostructures and discuss its potential for applications.

1 Insulator-Metal Transition in VO_2 Nanobeams

Correlated electron materials exhibit a variety of unexpected physical phenomena. The repulsive electron-electron interaction plays a pivotal role in determining the electronic, magnetic and optical properties of such systems.[1] Unlike commonly known materials such as silicon that are used in the majority of commercial electronics, correlated electron materials have vastly different phases that can be accessed by a variety of external stimuli giving rise to exotic properties such as magnetoresistance, superconductivity or metal-insulator transitions.[2–4] Albeit the exhaustive list of interesting studies on strongly correlated electron systems, these systems are still being explored in the hopes that our every day lives can utilize the exotic nature of these materials.

Vanadium oxides are a branch of compounds that have always been at the center of correlated material research. Their rich phase diagrams contain conductivity, magnetic and structural phase transitions that span across a wide range of temperatures and pressures.[5] Arguably the most well known compound in the family, vanadium dioxide (VO_2), has been a focus of condensed matter research over the past few decades. At temperature (T) ~ 342 K, VO_2 undergoes a massive first order insulator to metal transition (IMT) leading to many orders of magnitude change in electrical conductivity. Simultaneously it undergoes a structural transition from a monoclinic, insulating phase to a rutile, metallic phase. The fundamental mechanism behind the IMT is still debated as being due to a purely electronic effect (Mott transition) or an electron-lattice interaction effect (Pierels transition). The Pierels model suggests that the 3d electron states in vanadium are separated into degenerate levels from crystal field splitting. Overlap with 2p states of oxygen results in a larger splitting leading to an energy gap.[6] In contrast, the Mott transition model describes electron-electron interactions forming a Mott-Hubbard gap which is consistent with phases that were later discovered through doping or applying strain such as the M2 (another monoclinic) and triclinic phases.[7]

The transition is not limited to being triggered by temperature, but can also be triggered by uniaxial strain, voltage or light potentially giving rise to many applications such as Mott field effect transistors to pressure based gas sensors.[8–11] Due to the timescale at which the transition occurs (sub picoseconds) VO_2 has also been explored as an ultra-fast switch.[12] Additionally, it has been investigated as a potential material for smart

Correlated electron materials: A wide class of materials that exhibit unusual, but useful electronic, structural, optical and magnetic properties due to the interaction of the charge carriers.

Insulator to metal transition: A switching or sudden change in the electrical conductivity. An abrupt switching can be potentially useful in electronic switching applications.

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windows due to the changes in optical properties from a transmitive monoclinic phase to a reflective rutile phase.[13]

The VO_2 nanobeams studied in this work are grown using a stepwise hydrothermal process.[14] These high quality crystals are made from a solution based method and are not chemically attached to any substrates. This helps to ensure no built in strain is involved giving rise to a pure M1 insulating phase without any M2 domains forming.[15,16] Recent works have mapped the strain phase diagrams and showing regions of coexistence of multiple phases.[17] The M1 to rutile transition (Fig. 1) is seen in the Raman spectra acquired for a single crystal nanobeam showing only peaks associated with those phases.[18,19] During evolution to the other insulating phases the largest changes would occur in the 191 cm^{-1}, 222 cm^{-1} and 614 cm^{-1} peaks which would be shifted to a higher wave number.[20,21] Raman spectroscopy is also a useful tool to monitor metallic evolution based on the intensity and rounding of the peaks as a VO_2 sample is doped with various metals. It has recently been used to observe the metallization of VO_2 nanobeams through electrolyte gating.[22] When the transition to a rutile phase occurs, the monoclinic peaks disappear giving a featureless signature indicating that the structural transition is complete. The data was acquired using a 514 nm laser with 2400 lines/mm grating. The large peak at 520 cm^{-1} is from the silicon substrate.

Raman spectroscopy: A technique based on the scattering of light, used to observe different structural phases of materials.

After ensuring the nanobeams are of the highest quality, they are dispersed in an isopropanol solution and sonicated before being sprayed onto Si/SiO_2 substrates with an oxide thickness of 300 nm. Photolithography is used to pattern electrical contacts composed of Cr/Au layers which are deposited using electron beam evaporation. After wire bonding the samples are mounted on the sample stage of a homemade cryostat for variable temperature electrical transport measurements.

Photolithography: A microfabrication process used to pattern thin films on substrates. The method uses UV light on a light-sensitive chemical.

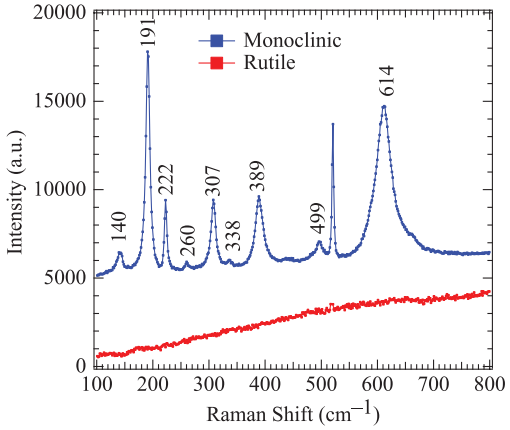

Figure 1: Raman spectra of the M1 monoclinic insulating phase and the rutile metallic phase. Loss of the monoclinic peaks in the rutile phase point to the structural transition.

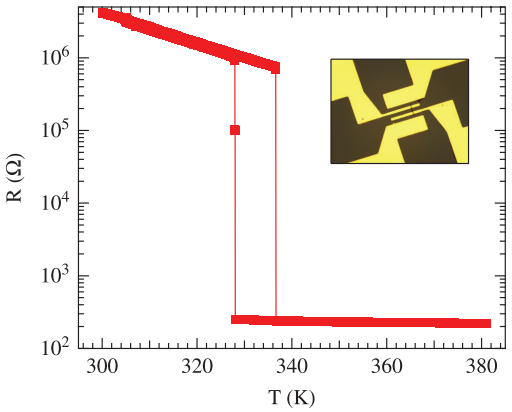

Figure 2: Resistance vs. *T* plot of a single nanobeam device showing a sharp IMT at $T_c = 338$ K in the heating cycle. The inset shows a standard two terminal device.

To initially characterize a device, the resistance of the single nanobeam device is measured as a function of temperature (*T*) (Fig. 2). Here we have shown a device with a large, four orders of magnitude transition to the metallic phase at T_c = 338 K. When the temperature is lowered from the metallic phase, the device returns to the highly resistive, insulating phase at 328 K thereby forming a wide, sharp hysteresis in the process.

The transition temperature in VO_2 being above room temperature makes this system more ideal than some other oxide materials having transitions at lower temperatures such as V_2O_3 (160 K)[23] and Fe_3O_4 (120 K).[24] The ability to tune the hysteresis is desired for many applications. The shape and width have been controlled through electrochemical gating and/or metallic doping.[25–27] Recently tungsten doped VO_2 has been used to achieve these features making an improved microbolometer.[28]

2 Electrically Driven MIT

Besides the thermally driven transition shown above, the IMT can also be triggered by applying a high voltage across the device in the insulating phase. Whether the voltage driven transition is an electric field effect or a Joule heating effect is still under debate.[29,30] Fig. 3 shows sweeps from 0 to 2 V while in series with a 670 Ω resistor at different, increasing temperatures. Current increases non-linearly but smoothly until it hits the threshold voltage for the transition. As the voltage is ramped back, many small jumps occur

before another sharp jump returns the device to its original insulating state. This yields qualitatively distinct transitions with hysteresis widths that shrink and become rounded with increasing temperature. This basic resistive switching is a hallmark property for materials finding a role in the technological world.[31] The timescales probed imply the possibility that the transition is the result of purely an electric field effect,[32] this has also been implied for other materials such as magnetite (Fe_3O_4)[33–36] and V_2O_3.[37] However, our recent work has shown that Joule heating to be the major factor in raising the temperature of the sample, however inhomogeneous conduction occurs and likely drive the transition.[38]

In Fig. 4 the values of threshold voltage for upward and downward sweeps can be extracted from the IV plot. Both cases yield a different behavior that can be fitted. When going from an insulator to a metal, $V_{th\uparrow} \propto \exp[-T/T_0]$ whereas going from a metal to insulator, $V_{th\downarrow} \propto \sqrt{T_c - T}$. Similar behavior has also been seen in W-doped VO_2.[39]

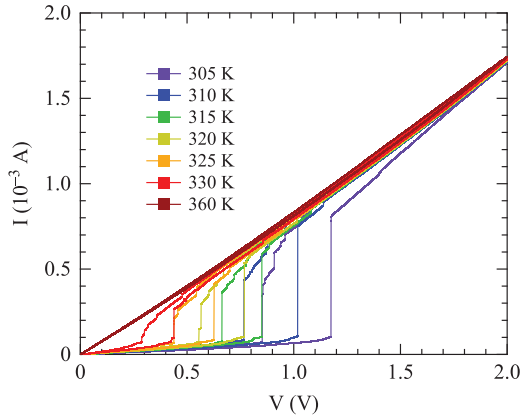

Figure 3: Current versus source-drain voltage (IV) plot of a VO_2 nanobeam at different temperatures below and above the transition. A hysteresis is seen for all temperatures below T_c yielding values for threshold voltage both up ($V_{th\uparrow}$) and down ($V_{th\downarrow}$).

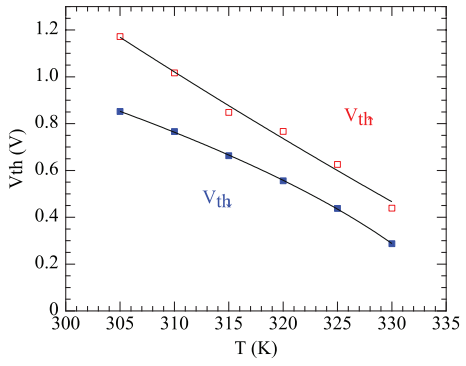

Figure 4: Threshold voltage vs. *T* for increasing and decreasing voltage sweeps fitted to $V_{th\uparrow} \propto \exp[-T/T_0]$ and $V_{th\downarrow} \propto \sqrt{T_c - T}$ respectively.

3 Electrolyte Gating and Suppression of IMT

Liquid electrolyte gating of oxide materials have proven to be an interesting way of tuning phase transitions in correlated materials. The observation of a complete suppression of the metal-insulator transition[40–43] and the emergence of superconductivity[44,45] are good examples of such novel behavior. The question of whether the electrolyte gating is purely electrostatic in nature or electrochemical reactions play a role makes these studies interesting and provide an alternate defect engineering route to tune phase transitions.

Ionic Liquids/Liquid Electrolytes: They are salts in the liquid state at room temperature. The application of a voltage can move the ions and hence be useful in modulating electrical properties on the nanoscale.

Our recent studies show that with the use of ionic liquid gating, oxygen vacancies can be readily created in VO_2 to modulate the conductivity and lead to a complete suppression of the IMT.[46] A droplet of N, N-diethyl-N-methyl (2-methoxyethyl) ammonium bis (trifluoromethylsulfonyl) imide (DEME-TFSI) is used as the electrolyte in our studies. Sweeps of the gate voltage in the monoclinic and in the rutile phase show different response to the gating, some dramatic enough to be irreversible. When a large gate voltage is applied in the rutile phase the IM transition is completely suppressed upon cooling down and the device remains metallic in the temperature of our study (Fig. 5). This stabilization is only achievable by gating in the rutile phase suggesting oxygen vacancy formation is more favorable in this phase. With this large oxygen vacancy concentration, the device can remain metallic even at temperatures far below T_c. A reversal of the gate voltage can be used to return the device to its original state. Density functional theory (DFT) calculations showed that when interference of the dimerization of V-V pairs that need to form when transitioning back to the monoclinic insulating phase occurs, then delocalized electrons from vanadium chains with altered bonds appear disrupting the change. These weakened bonds also result in lower transition temperatures closer to room temperature. This sheds light on the irreversibility of this reaction since any oxygen that leaves vacancies in the nanobeam will be consumed by the TFSI anion. Many unanswered questions still exist regarding the exact structure of this gate induced metallic phase compared to the thermally achieved rutile phase.[47] A recent work[48] has shown that an expansion occurs during gating as opposed to the expected contraction suggesting

Density Functional Theory: A computational modeling method used in material science to study the electronic structure of materials.

that the two phases, although both highly conductive, may not be identical in structure.

Figure 5 shows resistance-temperature plots at a 50 mV bias at different gate voltage values. The gate voltage is always applied in the rutile phase at 360 K. As the gate voltage value increases, the hysteresis begins to grow wider and smoother until at a certain voltage the trace flattens and becomes fully metallic. After stabilizing, the device can be removed and washed with isopropyl alcohol, deionized water and then blow dried to remove any remaining electrolyte on the surface. After this the device still retains its metallic phase for several days. Leaving it exposed in the air allows it to return to its original state. This result points to the possibility of a defect engineering route allowing the phase to be easily tuned much like how metal doping can be used except here the added benefit is instant reversible tuning that can not be achieved in substitutional doping with a metal.

White noise: Random signal with constant spectral density. This noise power does not depend on the frequency.

4 Noise Measurements Across the MIT

Noise spectroscopy is an old yet interesting tool to probe microscopic details of various transport processes in condensed matter. The fluctuations in physical quantities such as resistance or magnetic susceptibility can give insights into the underlying physical phenomena.[49] Noise spectroscopy is proven to be a tool to gain fundamental physical insights about various materials and also as an invaluable tool in device research since various digital systems rely on achieving a certain minimum level of signal to noise ratio.[50] For these reasons noise studies have witnessed a large growth during the recent decades.[51]

To be able to fully utilize noise spectroscopy, one needs to have a basic understanding of the physical processes behind the fluctuations in the system of study. For example, thermal noise (Johnson's noise) is a result of the random motion of electrons at $T > 0$[52] and shot noise is a consequence of the fundamental discreteness of charges at the microscopic level. Both of these types are frequency independent (white noise). An interesting type of noise is the 1/f noise that has a frequency dependence and the physical origins are extremely difficult to determine. This is due to the fact that all solid state systems show 1/f noise behavior in the measured quantities[51,52] and the driving mechanisms behind this noise are subtle.

In the study of VO_2, one long-standing question has been about the physical origins of the thermally driven and the electrically driven transitions. We employ noise measurements as a direct probe of the microscopic processes in the system during the thermally and electrically driven transitions.

We have measured the resistance fluctuations across several VO_2 nanobeam devices and found that when converted to frequency domain using Fourier transform, the fluctuations result in a pure 1/f dependence as shown in Fig. 6. As mentioned earlier, this is a very common type of noise. In metals 1/f noise is believed to be due to the motion of various types of defects and the scattering of charge carriers off them.[52] For simplicity, we only pick the magnitude of the noise magnitude at certain frequency intervals (centered around 1.0 Hz) and the result is plotted as a function of temperature in Fig. 7. The noise magnitude shows an orders of magnitude drop across the insulator-

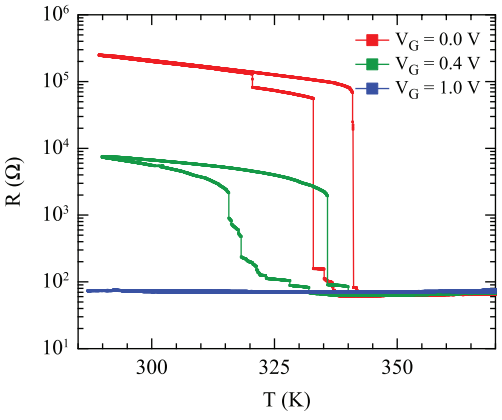

Figure 5: RTs done at different gate voltages applied in the rutile phase (360 K). Resistance decreases and the hysteresis shape widens until a fully metallic state is reached. On the metallic side, resistance shows the opposite trend of slowly increasing with gate voltage showing the difference in electrochemical reaction of the two phases.

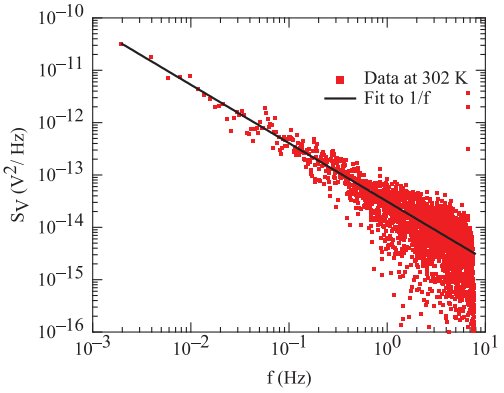

Figure 6: Noise magnitude measured at 302 K from a single nanobeam VO_2 device as a function of frequency. The solid line fit shows the 1/f dependence of the noise magnitude over four orders of magnitude in frequency.

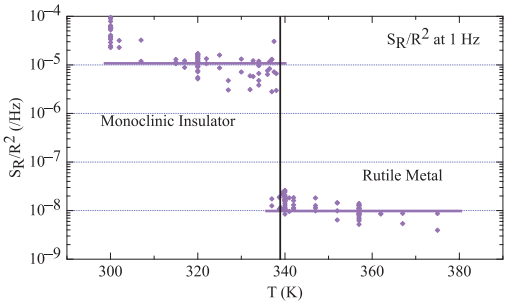

Figure 7: Noise magnitude at 1 Hz is plotted as a function of temperature across the metal-insulator transition.

metal transition. However, the magnitude remains nearly constant within each phase. The near constant temperature dependence of noise in the metallic and insulating phase suggests that noise models describing the noise mainly arising from thermally activated fluctuations can be ruled out in our case.[53] A comparison of the noise magnitudes and the temperature dependence during the thermally and electrically driven transition is currently underway.

5 Conclusion

VO_2 has been an interesting correlated electron system to study over the last few decades due to the fundamental questions about the mechanism behind the metal-insulator transitions and the possibility of utilizing these nanostructures in a variety of applications such as electrical switches, optical switches and bolometers. The above room temperature massive IMT makes this system more accessible than other materials, including other vanadium oxides that do not have an ideal transition temperature. The tunability of the transition temperature, hysteresis widths, magnitude of hysteresis using a slew of experimental techniques mentioned above make this system an interesting material to study.

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Ali Alsaqqa is a PhD student at the University at Buffalo, SUNY. His research interest is in the use of noise spectroscopy to understand the fundamentals of strongly correlated materials to pave the way for future applications. His past and current projects include studying phase transitions in VO_2 nanobeams, $NdNiO_3$ ultrathin films, and in $NbSe_3$ nanoribbons.