Search for power-efficient wide-range reversible resistance modulation of VO$_2$ single crystals

Bertina Fisher$^{1,3}$, Larisa Patlagan$^1$ and Lior Kornblum$^2$

$^1$ Department of Physics, Technion—Israel Institute of Technology, Haifa 32000-03, Israel
$^2$ Andrew and Erna Viterbi Department of Electrical Engineering, Technion—Israel Institute of Technology, Haifa 32000-03, Israel

E-mail: phr06bf@physics.technion.ac.il

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Abstract

The abrupt metal insulator transition in VO$_2$ is attracting considerable interest from both fundamental and applicative angles. We report on DC $I$–$V$ characteristics measured on VO$_2$ single crystals in the two-probe configuration at several ambient temperatures below the insulator–metal (I–M) transition. The insulator-mixed-metal-insulator transition is induced by Joule heating above ambient temperature in the range of negative differential resistivity (NDR). In this range the stability of $I(V)$ is governed by the load resistance $R_L$. Steady state $I(V)$ is obtained for $R_L > |dV/dI|_{\text{max}}$ in the NDR regime. For $R_L < |dV/dI|_{\text{max}}$ there is switching between initial and final steady states associated with peaks in the Joule power, that are higher the lower $R_L$ is. The peaks caused by steep switching are superfluous and damaging the samples. On the other hand, the large $R_L$ needed for steady state is the main power consumer in the circuit at high currents. The present work is motivated by the need to avoid damaging switching in the NDR regime while reducing the power consumption in the circuit. Large resistance modulation can be obtained under steady state conditions with reduced power consumption by increasing the ambient temperature of the device above room temperature. Under steady state conditions, the transition to the mixed metal-insulator state is smooth and is followed closely by appearance of sliding domains.

Keywords: vanadium dioxide, metal-insulator transitions, negative differential resistance

(Some figures may appear in colour only in the online journal)
serve as guides for the design of thin film devices, as well as for freestanding nanocrystal applications. Under an applied voltage at ambient temperature, the resistance drops due to Joule heating, the voltage reaches a maximum \( V_{\text{max}} \) and a current controlled negative differential resistivity (CC-NDR) regime sets on. The mixed I–M phase appears within the NDR regime. The stability of \( I(V) \) in this regime is governed by the magnitude of the load resistance \( R_L \) in series with the sample; \( I-V \) is stable for \( R_L \geq \frac{dV}{dI}_{\text{max}} \) – the absolute value of the steepest slope of \( V(I) \) in the NDR regime [17]. For lower \( R_L \), portions of the NDR regime are unstable and \( I(V) \) switches from the last steady state towards a steady state with minimal entropy production (minimal Joule heating) [18].

While in films the mixed state consists of metallic filaments embedded in the semiconductor [19], in single crystals it consists of metallic and semiconducting domains with boundaries crossing the widths of the samples in favorable inclinations [20]. It was recently shown that in contrast to thin-film channels in MoS2-FETs, single-crystalline nanowires have superior sensitivity for transport modulation, resulting in ten-fold higher resistance range; this is attributed to the coexisting I–M states in the crystalline channel [21]. VO2 single crystals or free-standing, crystalline nano-wires have an additional, unique property: in the mixed M–I state: narrow metallic filaments embedded in the semiconductor [19], in single crystals or free-standing, crystalline nano-wires have an additional, unique property: in the mixed M–I state: narrow metallic filaments crossing the widths of the samples in favorable inclinations [21]. VO2 single crystals have superior sensitivity for transport modulation, resulting in steeper, damaging switching is prevented in VO2 single crystals connected to large enough load resistances.

The threshold voltage \( V_{\text{max}} \) at the onset of NDR regime may be estimated by equating the Joule power with Newton’s law of cooling i.e. \( P = V^2/R = \alpha \Delta T \), where \( \Delta T \) is the excess above ambient temperature \( T-T_0 \) and \( \alpha \) is an effective coefficient. \( R \) is approximated by the activated resistance as \( R = R_0 \cdot \exp(-\Delta T/T_1) \) where \( R_0 \) is the resistance at ambient temperature, and \( T_1 \) a fitting parameter [27].

\[
R(T) = R(T_0 + \Delta T) = R_0 \exp \left( \frac{E_a}{kT} - \frac{E_a}{kT_0} \right) \approx R_0 \exp \left( \frac{T}{T_1} \right)
\]

where \( \Delta T = T - T_0 \) and the parameter \( T_1 = kT_0/\langle E_a \rangle \).

This is allowed by the narrow temperature range between room temperature and \( T_{\text{IMT}} \) (e.g. figure 1). For single crystals with activation energies of \( 0.4 \leq E_a \leq 0.5 \text{eV} \), this parameter is: \( 22 \geq T_1 \geq 17 \text{K} \). These approximations lead to \( IV = T_1 \ln \left( \frac{dV}{dT} \right) \) from which the differential resistance \( dV/dI \) is derived:

\[
\frac{dV}{dI} = \frac{V(\alpha T_1 - IV)}{\alpha T_1 + IV} = \frac{V}{I} \left( \frac{T}{T_1} - 1 \right)
\]

The onset of CC-NDR occurs when \( dV/dI = 0 \). At this point on the \( I-V \) characteristic \( T_1 = \Delta T \) (independent of \( \alpha \),[27]). This yields:

\[
R = \frac{V}{I} = R_0 e^{-1}
\]

\[
V_{\text{max}} = \sqrt{R_0 e^{-1} T_1}.
\]

The high current limit of the NDR regime \( \frac{dV}{dI} = -\frac{V}{I} \) cannot be reached in the insulating state since the onset of the mixed state sets on at finite \( \Delta T = T_{\text{IMT}} - T_0 \).

\[
\frac{dV}{dI}_{\text{max}} \text{ in the NDR regime (that determines the minimal } R_0 \text{ required for stability) is found by setting: }
\]

\[
\frac{d^2V}{dT^2} = \frac{2\sqrt{3}}{T} \left( \frac{dV}{dI} \left( \frac{T}{T_1} \right) \right) = 0
\]

which leads to:

\[
\frac{IV}{\alpha T_1} = \sqrt{3}
\]

and

\[
\left[ \frac{dV}{dI} \right]_{\text{max}} = e^{-\sqrt{3}} \left[ 1 - \frac{\sqrt{3}}{1 + \sqrt{3}} \right] R_0 = 0.04741 R_0.
\]

Equations (4) and (5) show that upon decreasing \( R_0 \) the maximal voltage on the sample (at the onset of NDR) is reduced and even more important, the load resistance (the main consumer of the applied voltage at high currents) can be reduced. The solution suggested in [20] for reducing \( R_0 \) while preserving the quality of VO2 and its IMT would be to raise the ambient temperature around the functioning VO2 device.

Figure 1. Two-contact resistance: \( R \) versus 1000/T for crystals D5(3), D5(4), D5(6), D3(11), D3(16), D3(17) and D1(11).
There may be another reason for increasing the ambient temperature of the VO2 device close to T_{IMT}: when current is applied on the device at high temperature the I–M transition occurs at a low voltage that has no direct effect on the crystal except of heating. The high voltage needed to induce the transition in the more resistive device at low temperature may affect the lattice causing permanent alterations. The consequences of high voltages applied at low temperatures was shown to have dramatic effects in thin films [14].

Finding optimal conditions for obtaining maximal resistance modulation under steady state conditions and minimal external voltage applied on single crystals is the main objective of this work. In addition, this work provides a test-bed of the simple model of ‘Joule heating in VO2 single crystals’ derived above.

2. Experimental

Single crystals of VO2 have been grown by self-flux-evaporation [22] from 99.99% V2O5 powder (Sigma Aldrich). Freestanding needle-like shaped crystals have been used in this work (table 1). R(T) of the VO2 crystals was measured in the two-probe configuration (with connected adjacent voltage and current probes) using indium-amalgam dots for contacts. These form Ohmic contacts of low resistance for insulating (semiconducting) VO2. With these contacts, the samples are free to move, being held only by surface tension. I–V measurements were carried out at ambient temperature in the two-probe configuration by varying the applied voltage and the load resistance. The I–V loops were recorded on a YEY type 3036 X-Y recorder while the samples were viewed under the microscope. The video clip shown in the supplementary material (stacks.iop.org/JPhysD/52/385302/mmedia) was taken with a smart phone camera mounted on a Zeiss stereo microscope.

3. Results and discussion

Here, we report on the investigation of the effects of the ambient temperatures and of R_L (=101 kΩ, 11 kΩ and 1 kΩ) on the I–V characteristics of two single crystals of VO2 (labelled D5(3), and D3(11), table 1). The results emphasize the striking difference between I–M switching under steady state (R_L > |dV/dI|_{max}) and non-steady state (R_L < |dV/dI|_{max}) conditions. The I–V characteristics of four more samples, D5(4), D3(16) and D3(17) were also measured at different ambient temperatures but only for R_L > |dV/dI|_{max} in the NDR regime. I(V), R(V) = VI/II and P(I) = IV(I) for samples D5(3) and D5(4) connected to different R_L are presented in the main text, whereas those for D3(11), D3(16), D3(17) and D5(6) are shown in the supplementary material. A smooth transition from insulating to mixed metal-insulator state followed closely by appearance of sliding domains is exhibited by a thin VO2 single crystal—D1(11) examined under the microscope during the I–V cycling under steady state conditions. V(I), P(I) and u(J) (u-a sliding velocity, J-current density) for this sample are shown in the main text and two corresponding video clips are shown in the supplementary material. Concluding results derived from all the traces in the main text and in the supplementary material are shown in the main text.

The semilog plots of R(1/T) for the six samples, measured by two contacts—four probes are shown in figure 1. The dimensions of the samples, their specific resistivity \( \rho \) at 300 K, the activation energy in the semiconducting state, the transition temperatures upon heating T_{IMT} and upon cooling T_{MT} obtained from the traces in figure 1 are summarized in table 1. The resistance jumps are limited by the finite contact resistance in the metallic state (between ~7 kΩ and ~10 kΩ) curves. For all three samples the transition is very steep and the hysteresis ranges between 1 K for D5(3) to 4 K for D5(4)—an indication of their high crystalline quality. The I–V characteristics of D5(3) at 295 K, 310 K and 320 K, R(V) = VI/II and P(I) = IV calculated from the I(V) data are shown in the first column of frames of figure 2 for R_L = 101 kΩ, in the second row for R_L = 11 kΩ and in the third for R_L = 1 kΩ (1 kΩ is the resistor used for the current measurements). In the first row, for the largest R_L, the I–V characteristics show the onset of CC-NDR at V_{max} that decreases as the temperature is increased. Small voltage drops at a current which is independent of T_0 towards a reversible I(V) range mark the onset of the mixed state with the appearance of the first metallic domain (e.g. at ~20 V for 295 K, figure 2(a)). The range of currents in (a) is limited by the large R_L that carries most of the finite source voltage of 200 V. The hysteresis is narrow and the backward transitions occur at slightly lower currents. R(V) calculated from I(V) and |dV/dI|_{max} from fitted cubic or higher order polynomials to V(I) in the NDR regime are shown in figure 2(b); the latter are shown below the R(V) curves. For all three

| Sample | \( L \) (cm) | \( a \) (cm) | \( b \) (cm) | \( \rho \) (300 K) (Ω cm) | \( E_a \) (eV) | \( T_{IMT} \) (K) | \( T_{MT} \) (K) | Surface area (cm²) | \( V_{max}/R_{L}^{1/2} \) (W/Ω) | \( R^2 \) |
|--------|-------------|-------------|-------------|-----------------|-------------|-------------|-------------|----------------|-----------------|--------|
| D5(3)  | 0.15        | 0.00736     | 0.00485     | 85              | 0.412       | 340.5       | 339.5       | 0.00366        | 0.0573          | 0.963  |
| D3(11) | 0.15        | 0.0210      | 0.0160      | 199             | 0.409       | 339         | 337         | 0.01111        | 0.0928          | 0.924  |
| D5(4)  | 0.15        | 0.0144      | 0.0049      | 240             | 0.414       | 341         | 338         | 0.0058         | 0.0562          | 0.905  |
| D3(16) | 0.42        | 0.0260      | 0.0246      | 194             | 0.476       | 341         | 339.5       | 0.0425         | 0.1202          | 0.922  |
| D3(17) | 0.43        | 0.0088      | 0.0044      | 242             | 0.429       | 343         | 341         | 0.01136        | 0.0926          | 0.803  |
| D5(6)  | 0.15        | 0.00777     | 0.0060      | 177             | 0.400       | 342         | 338         | 0.00411        | 0.0553          | 0.990  |
| D1(11) | 0.13        | 0.0042      | 0.0020      | 128             | 0.467       | 341.5       | 340         | 0.00161        | 0.0442          | —      |
ambient temperatures, $|dV/dI|_{\text{max}} < 101 \, \text{k}\Omega$ but $> 11 \, \text{k}\Omega$. Thus, the data in the NDR regime with $R_L = 101 \, \text{k}\Omega$ were obtained under stable conditions while all others, under unstable conditions. Figure 2(c) shows the increase of $P = IV$ with increasing current (increasing temperature) followed by a small but steep decrease as the first metallic domain appears in the sample, at a 1.3–1.5 mA range for all three temperatures. This is consistent with the decrease in infrared emission of metallic VO$_2$ relative to semiconducting VO$_2$ [28, 29].

The range of currents in figure 2(d) is one order of magnitude larger for $R_L = 11 \, \text{k}\Omega$ compared to 101 k\Omega. Switching occurs along a broken line that is steeper close to $V_{\text{max}}$ and almost flat before the onset of the reversible (stable) regime. A straight line of slope $1/R_L$ connects the point at $V_{\text{max}}$ with that at the onset of the reversible regime. $I(V), R(V)$ and $P(V)$ exhibit large hystereses between forward and backward switching. The lowest resistance reached upon increasing current (figure 2(e)) is still at a safe distance above that of the contact resistance ($<0.1 \, \text{k}\Omega$, figure 1). $P_{\text{max}}$ for $R_L = 11 \, \text{k}\Omega$ (figure 2(f)) is more than twice larger than for $R_L = 101 \, \text{k}\Omega$ at 295 K; interestingly, $P_{\text{min}}$ is close to that for the large $R_L$. The drop of $P_{\text{max}}$ with increasing temperature is dramatic. In the reversible (stable) regime, $P$ increases linearly with $I$. This was quite surprising at the time when it was naively assumed that for fixed temperature ($T_{\text{IMT}}$) $P$ should either be constant or decrease with current due to the increasing fraction of the metallic phase in the mixed state [28]. It was previously shown that the increasing $P(I)$ is correlated to the dynamic phenomena of creation and annihilation of moving M–I domain boundaries. The slope $dP/dj = L^{-1}dP/dI \approx 8 \, \text{V cm}^{-1}$ for this sample ($p$-power density, $j$-current density and $L$-sample’s length) falls within the range of such phenomena observed in [20]. The range of currents for $R_L = 1 \, \text{k}\Omega$ was intentionally limited to only a factor of $\sim 3$ relative to the previous case, so that the lowest resistance would not reach the contact resistance. Switching in figure 2(g) is along a straight line of slope $1/R_L$ from the onset of NDR to the reversible (stable) range of currents. The hystereses in all graphs of the three frames are huge. At 295 K, $P_{\text{max}}$ for 1 k\Omega is larger than $P_{\text{max}}$ for 101 k\Omega by more than an order of magnitude (figure 2(i)).
The effect of $R_L$ on $P_{\text{max}}$ is revealed in figure 3(a) for sample D5(3) and in 3(b) for D3(11) (figure S3, supplementary material). Semilog $P_{\text{max}}(T_0)$ representation was chosen in order to provide equal weights to the data that are spread over almost two orders of magnitude.

In figure 3(a), the fitted lines for $R_L = 1 \, \text{k}\Omega$ and $R_L = 11 \, \text{k}\Omega$ have relatively higher slopes and below them lies the line for $R_L = 101 \, \text{k}\Omega$ with a much lower slope. In figure 3(b), the line at the top (for $R_L = 1 \, \text{k}\Omega$) has a high slope while the two at the bottom (for $R_L = 11 \, \text{k}\Omega$ and $R_L = 101 \, \text{k}\Omega$) have relatively lower slopes. The data on the lines with high slopes were obtained under non-steady state conditions (i.e. $R_L < |dV/dI| \max$ for the respective characteristic). With one exception, the data on the lines with low slopes were obtained under steady state conditions. The exception is the datum point for $R_L = 101 \, \text{k}\Omega$ obtained under non-steady state conditions (i.e. $R_L = T_0$). For all but sample D3(11) at 289 K (see figure S3 in the supplementary material), it represents the $I$–$V$ characteristic that deviates from the ‘well behaved’ form in figure S3(f) and shows that this characteristic is for a non-steady state. In fact, a line for $R_L = 11 \, \text{k}\Omega$ fitted to the three data points for the higher temperatures would be parallel to the line for $R_L = 101 \, \text{k}\Omega$. According to our simple model $P_{\text{max}} \propto (T_{\text{IMT}} - T_0)$; for narrow ranges the exponential function is close to linear. Therefore, the overall behavior in figure 3 agrees with our proposed model where $P \propto \Delta T$ in steady state.

$I$–$V$ measurements on sample D5(4) (figure 4) were devoted to finding the optimal, steady state conditions for insulator—mixed metal-insulator transition induced by current, that is, maximal resistance modulation for minimal applied voltage. Steady state implies removal of the damaging bell shaped $P(I)$ in the NDR regime (e.g. figure 2(f) and (i)).

The $I$–$V$ characteristics, $R(V)$ and $P(I)$ for $R_L = 101 \, \text{k}\Omega$ are shown in figures 4(a)–(c) for sample D5(4). The traces of $ldV/dlI(V)$ for this large $R_L$ are shown below the $R(V)$ traces in figure 4(b). They show that $ldV/dlI_{\max} = 26.1 \, \text{k}\Omega$, $25.0 \, \text{k}\Omega$ and $24.6 \, \text{k}\Omega$ for $T_0 = 295 \, \text{K}$, $307 \, \text{K}$ and $320 \, \text{K}$, respectively. These traces for $R_L = 34 \, \text{k}\Omega$ (out of caution, not the lowest possible $R_L$) are shown in figures 4(d)–(f). The range of currents was increased by a factor of three and that of the resistance modulation by one order of magnitude. The lowest resistance in 4(e) is more than one order of magnitude higher than the contact resistance. The results in figure 4(f) are in strong contrast with those shown in figures 2(f) and (i) for sample D5(3) (and those of figure S3(i) for sample D3(11)). For all three temperatures $P(I)$ reaches shallow maxima followed by shallow dips associated with the appearance of metallic domains. After a range where $P(I)$ shows a rather noisy behavior, reversible ranges are reached. The widest range of currents over which $P(I)$ is reversible is obtained for $T_0 = 320 \, \text{K}$. The slope of this trace in the reversible regime is $dp/dI \approx 1.5 \, \text{V}$ (comparable to that in figure 2(f)) and corresponds to $dp/dI = 10 \, \text{V cm}^{-1}$. The low $V_{\max}$ and the wide reversible regime emphasize the advantage of working at $T_0 = 320 \, \text{K}$. In other words, at low resistance it is distinctly adventitious to perform current-induced switching of VO$_2$ intuitively be expressed as switching in the conditions where crystals are close to their IMT temperature. This results in less driving force, which promotes a ‘clean’ transition that yields reversible, damage-free behavior.

In figure 5, we compare results obtained for the six samples by which the validity of the simple model of ‘Joule heating’ is examined.

In figure 5(a), we plotted $V_{\max}$ versus $R_0^{1/2}$ according to the model’s predictions (equation (4)). The heights of the symbols in figure 5 exceed the experimental uncertainties in $V_{\max}$ and in $ldV/dlI_{\max}$ respectively. The uncertainties along the horizontal axis are attributed to deviations in $R_0$ caused by small deviations in $T_0$ (≈0.5 K). In principle, for a given sample $V_{\max}$ should depend only on temperature and not on the load resistance. This holds for all but sample D3(11) at 295 K. The slopes of the lines and the respective quality of fit are summarized in the last two columns of table 1. The values of $R^2$ (>0.9) for all but that for sample D3(17) (≈0.8) indicate fairly good agreement between the experimental results and predictions. The fitted lines for D3(11) and D3(17) almost coincide and so do the three fitted lines for D5(3), D5(4) and D5(6). The surface areas of the first pair of samples is incidentally identical (table 1), those for the other three are close but a small uncertainty in their small dimensions could account for the difference between their areas. The correlation between $V_{\max}^2/R_0$ and surface areas found for these five samples stresses the fact that the heat dissipation is almost exclusively from the surface of the samples, and therefore heat conduction through the contacts is insignificant.

The $ldV/dlI_{\max}$ data versus $R_0$ obtained from the $V(I)$ traces for the six samples in the stable states of NDR are plotted in figure 5(b). The data points are best fit by the line
$|dV/dI|_{\text{max}} = 0.0476 \, R_0 \, (R^2 = 0.883)$. The model predicts $|dV/dI|_{\text{max}} = 0.0474 \, R_0$ (equation (5)) for NDR in the insulating state. The onset of the mixed M–I state changes the slope of $dV/dI$ from that derived for the insulating state. For $T_0 = 320 \, K$ (with $T_1$ around 20 K) the onset of NDR (at $T_0 + T_1$) and of the mixed state at $T_{\text{IMT}}$ are very close. Therefore, to warrant steady state $|dV/dI|_{\text{max}}$ should be determined experimentally for each device at each ambient temperature. In a more general case, for applied AC, the load resistance $R_L$ should be replaced by the load impedance, $Z_L$.

$V(I)$ and $P(I)$ for the very thin sample D1(11) examined under the microscope during $I$–$V$ cycling are shown in figures 6(a) and (b) under steady state conditions ($R_L = 500 \, k\Omega$). The temperature measured by a thermocouple close to the sample was 300 K (slightly larger than RT due to the constant illumination of the sample). The most impressive result here is the smooth insulator to mixed-metal-insulator transition exhibited by the bending of the $P(I)$ curve. The bending is followed closely by the appearance of sliding semiconducting domains within the metallic background in the sense of the electric current. In the mixed state $dP/dI = 28 \, V$ and corresponds to $dp/dj = 215 \, V \, cm^{-1}$, far larger than the corresponding values obtained in the thicker samples. The symbols on $I(V)$ and $P(I)$ mark the points where the sliding domains were recorded on video. The full circles mark two video clips recorded at different currents attached to the supplementary material. The sliding velocity ($u$) as function of current density ($J$) are shown in figure 6(c). The fitted $u/J = 0.0005 \, 57 \, cm^3$
As $1/R^2 = 0.89$ is about $1/2$ the maximal value obtained in VO$_2$ single crystals and is comparable to the value obtained in a sample labeled D2(9) (figure 7 in [16]). As in the case of D2(9), the low $u/J$ value obtained for D1(11) may be attributed to its poor morphology.

4. Conclusion

In conclusion, our $I–V$ measurements show the advantage of increasing the ambient temperature around single-crystalline devices avoiding steep switching by maintaining steady state in the NDR regime while reducing power consumption. Since the main problem in electronics is cooling the devices, the increase of ambient temperature relative to room temperature means less cooling, an additional contribution to reduction in power consumption.

The results indicate that the simple ‘Joule heating model’, though not rigorous, is very useful for orientation within the various regimes of the $I–V$ characteristics. Large deviations of the measured $dV/dI_{\text{max}}$ calculated from the model (derived for the insulating regime) are caused by the appearance of the mixed I–M state within the NDR regime.

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Supplementary material

See supplementary material for two graphs supporting the simple model of ‘Joule heating in VO$_2$ single crystals’, graphs of the full electrical analysis of samples D5(6), D3(11), D3(16) and D3(17) and for sliding domains recorded on videos.

ORCID iDs

Lior Kornblum https://orcid.org/0000-0001-6305-7619

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