



Following Evolution of 3D Reciprocal Space Maps of Insulating and Metallic Phases of VO₂ During Phase Transition

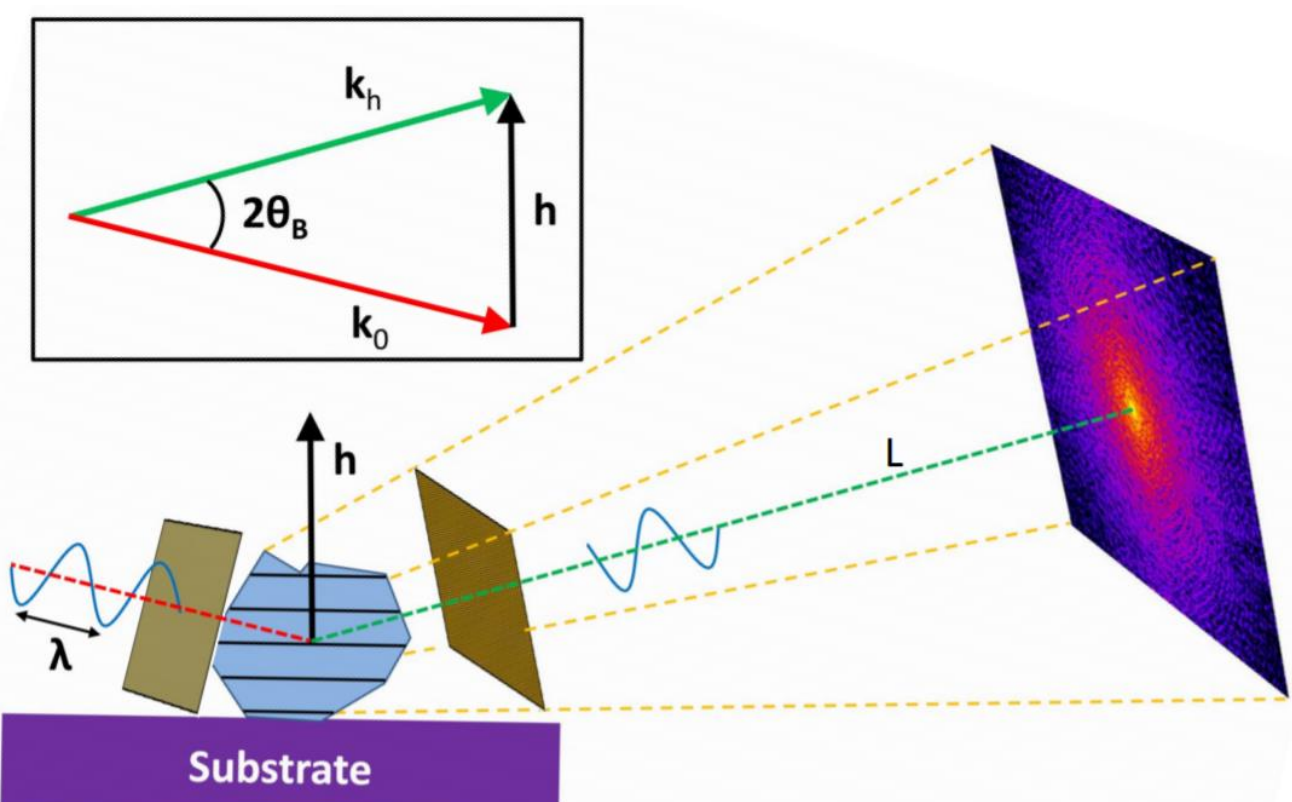
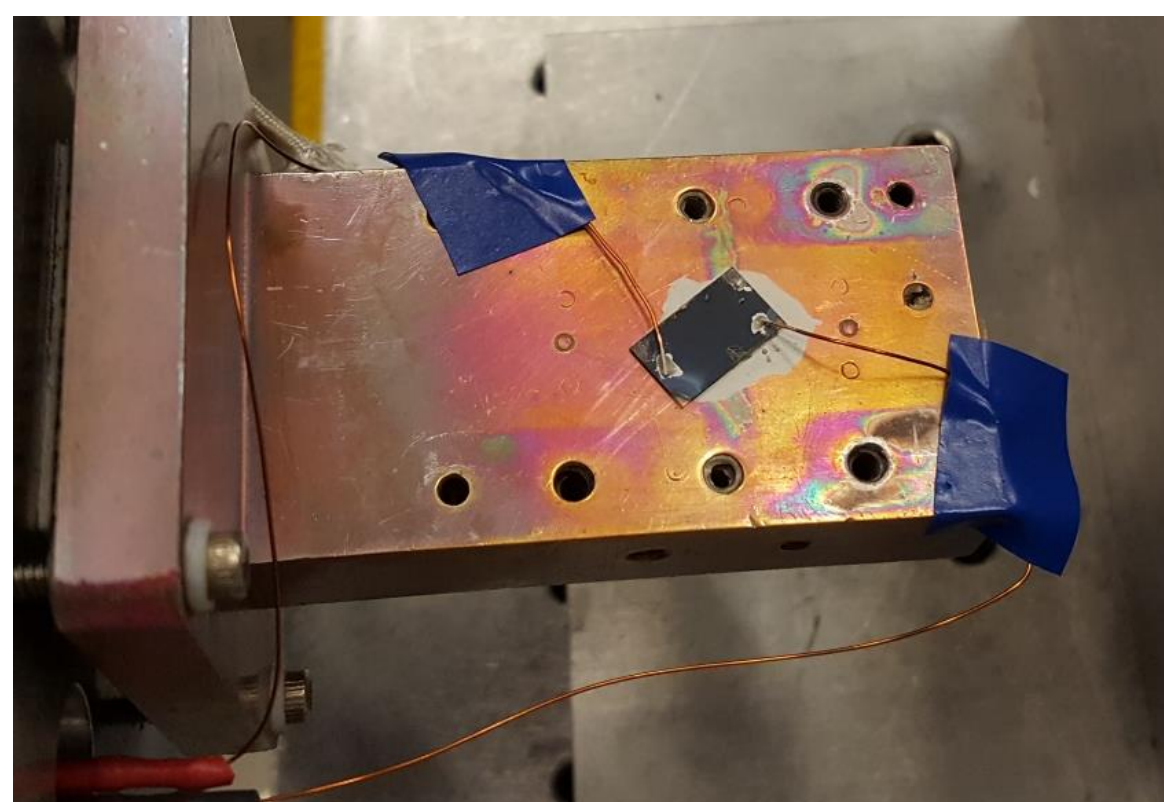
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Abstract

Most materials are either conductors, which have low resistivity, or insulators, which have high resistivity; however, Vanadium(IV) Dioxide (VO₂) does not follow these characteristics. Typically, VO₂ is an insulator at lower temperatures and a conductor at higher temperatures, but this material can exhibit both behaviors at the same time during phase transition. One possible reason why this material can change its resistivity may be because of changes in its structure. Therefore, we used x-ray nanodiffraction to look at bragg peaks to determine how structure changed when resistance changed. The angles at which we found the bragg peaks are useful as we can use Bragg's Law to determine the distance between the planes of atoms in the material. By finding the spacing between the different layers of the atoms, we could determine how structure changes when temperature changes, and how that leads to changes in resistivity of VO₂. By looking at the 3D maps of the bragg peaks, we concluded that the metal to insulator transition (MIT) occurred around 64°C. This temperature was when we could see two bragg peaks, representing metallic and insulating phases. A change in the bragg peak meant that the nanostructure of VO₂ changed. In addition, resistance dramatically changed around this temperature, so this is evidence that the resistance of VO₂ may be dependent on the structure of it.

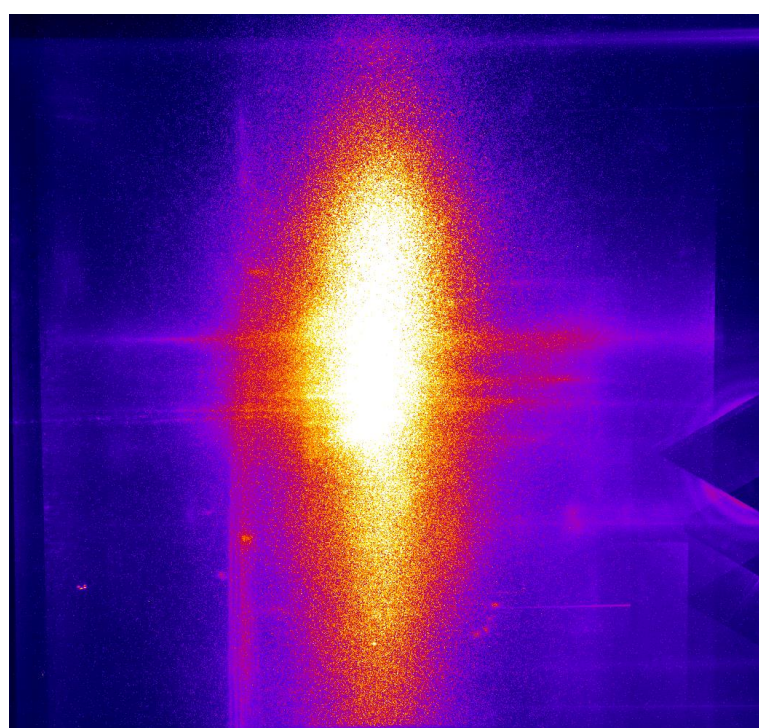
Method



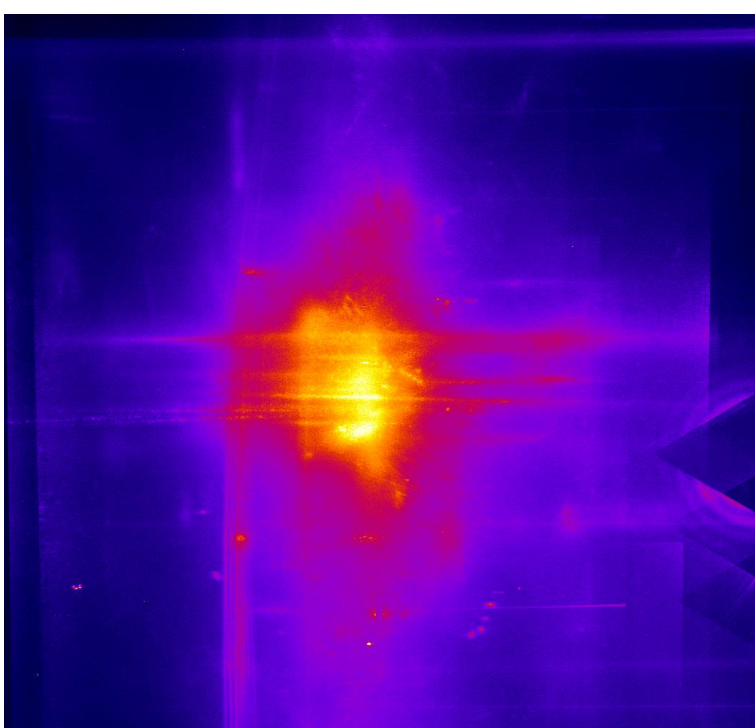
We first grew a 100 nm VO₂ thin film on an r-cut sapphire substrate. In order to image the VO₂ in the reciprocal space, we used a focused x-ray beam that had an energy of 7.35 keV (kiloelectron volts). The x-ray beam hit the sample at a particular angle (θ) and refracted off at another angle (2θ). A CCD detector captured this 2D diffraction pattern. One image represented a slice of the VO₂ in the reciprocal space. Within these images, we saw different slices of the bragg peaks. In order to get a full image of the bragg peak, we needed to take hundreds of images of the peak at different angles so that we could combine them together to create a 3D map of the bragg peaks. Therefore, we changed the angle of the sample, from angle 20.5° to 22° from the horizontal. We kept the point at which the x-ray hits the sample at a fixed location, and then rotated the substrate along this axis. This created a rocking scan. By changing the angle of the platform, we were able to image different parts of the bragg peak so that we could later connect the pieces together.

Results From Diffraction

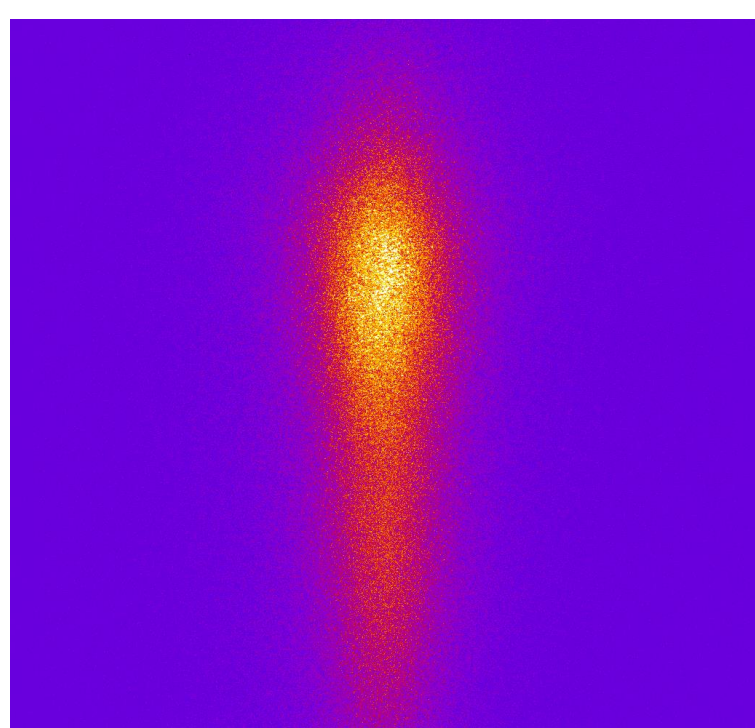
Scan #147 of VO₂ in Reciprocal Space at Temperature 64.6 °C, Cooling



Data and Dark



Dark

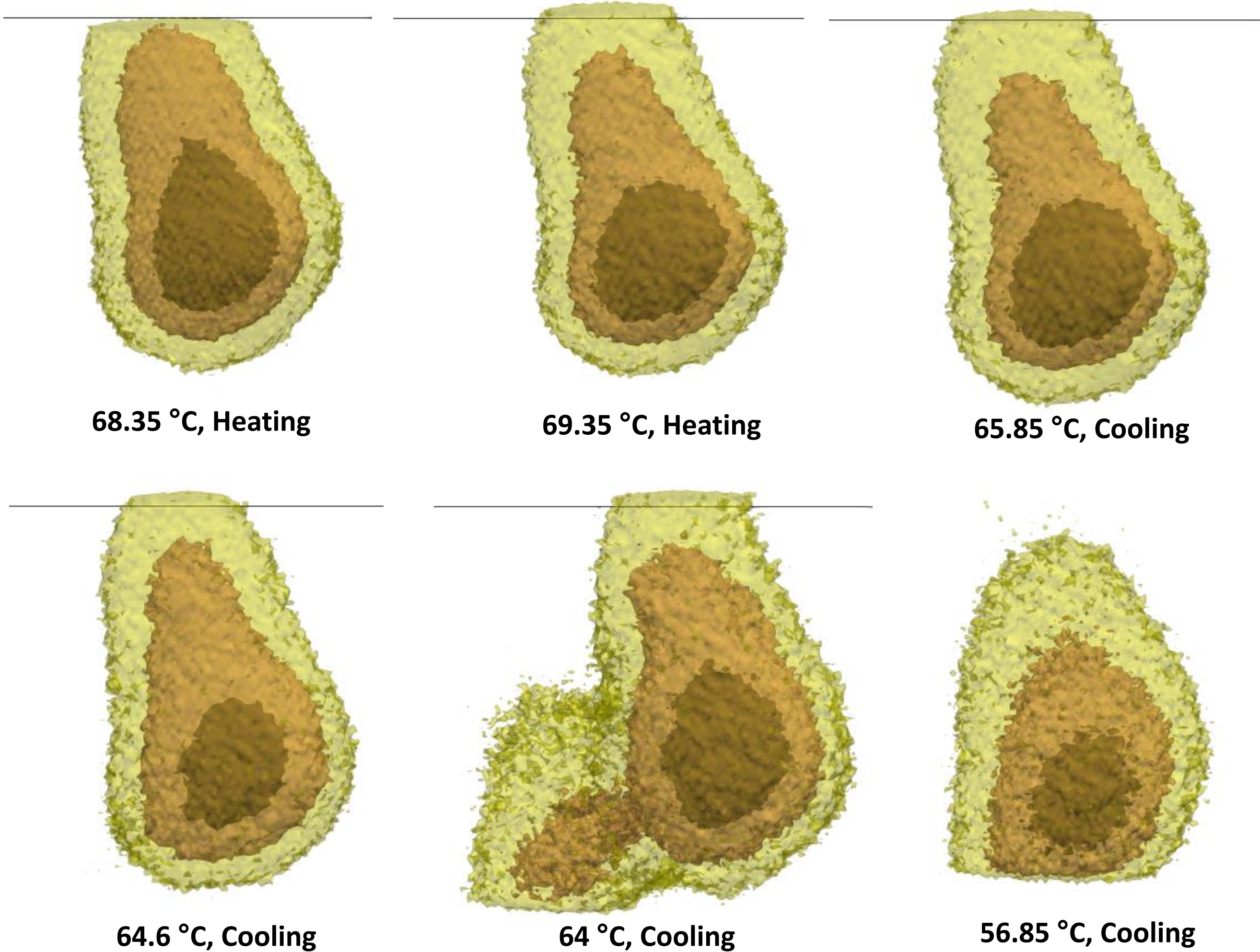


Data

With the data collected by the CCD detector, we had to separate any unnecessary background to get the raw data. The CCD detector imaged the diffraction pattern and the background behind it. To separate the background from every picture, we took a single "dark" picture, representing just the background that the CCD detector captured when no x-rays were being emitted. Then, using the program ImageJ, we removed the darks from the original images to get a clear version of just the data. The data above represented a slice of VO₂ in the reciprocal space at 64.6°C while we were cooling the material. The bright red smear represented a bragg peak. We took data from five other scans, representing five other temperatures. Specifically, we looked at the data at 56.85 °C, 64 °C, 64.6 °C, and data at 65.85 °C when cooling VO₂ and 68.35 °C and 69.35 °C when heating VO₂. We were most interested in the data from this temperature range because this was the range in which MIT occurred.

Mapping 3D Image of Bragg Peak

Our next step was to take the 2D images captured by the CCD detector and stack them so that we can create a 3D map of VO₂ in the reciprocal space at different temperatures. However, we could not simply stack these images on top of each other because the sample was set on a rocking curve. If we were to stack these images immediately, the 2D images would not align perfectly and the 3D image of the bragg peak would either look stretched or compressed. Therefore, we created a script to correct and realign the 2D images. After correcting the misalignment, we used the program ParaView to create an isosurface around the 2D images. Here, we can observe the changes of the bragg peak when temperature changes.



The different colors shown on the images represent different isosurface levels, or levels of intensity; the highest intensity of the bragg peaks is in the center of the images and is represented by the color brown, the middle intensity is represented by the color orange, and the lowest intensity is represented by the color yellow. All of the different temperatures show a bragg peak, representing either a metallic or an insulating phase. However, the image at 64°C shows two bragg peaks: one that is a high phase peak and another that is a low phase peak. Thus, at this temperature, some parts of the VO₂ film behave like a conductor and other parts behave like an insulator.

Discussion and Conclusions

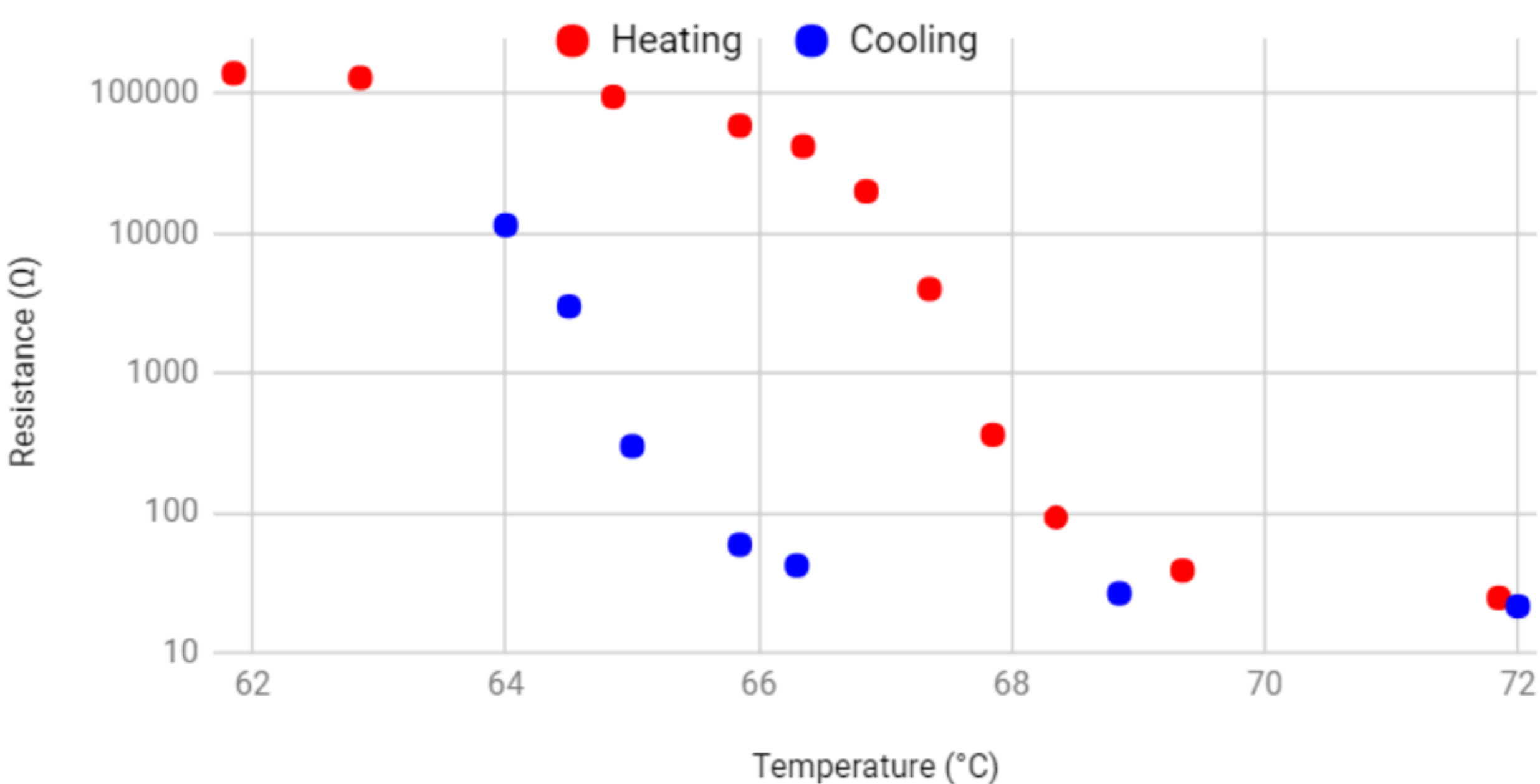
X-ray nanodiffraction can have several applications. For example, when we study batteries through x-ray nanodiffraction, we can look at its bragg peaks and understand how the structure of the battery can lead to different traits. With this data, engineers can fine-tune characteristics that are not as effective to create a more useful battery. From a biological standpoint, x-ray nanodiffraction can be used to determine structures on a cellular level. By understanding the structure of nanomaterials, we can understand how and why they work. In fact, x-rays were used to determine that the structure of DNA was a double helix; this discovery led to a wider understanding of how DNA works.

There are many macroscopic behaviors such as magnetism, electricity and resistance that intrigue scientists, but the only way to explain these behaviors is by looking at the microscopic traits. X-ray nanodiffraction is often used to understand nano-structured materials that exhibit unique traits, such as the ability for a material that can change from a conductor to an insulator. By creating a 3D image of the bragg peaks at different temperatures, we were able to see that the bragg peaks at the different temperatures were very similar; however, the phase transition was sharp as we could see two bragg peaks at 64 °C. This meant that the structure of the VO₂ film changed at this temperature. Because resistance also dramatically changed at this temperature, we hypothesized that resistance of VO₂ is dependent on its structure. Hypothetically, it is possible that when we changed temperature, the change of the structure allowed more electrons to pass, transforming the material from an insulator to a conductor. Although we do not know this is true, this concept is intriguing as engineers can create materials with a particular structure to achieve a particular resistance. In addition, having a single material that can switch resistances can allow engineers to discard devices that need several different materials that have their own resistances. In a follow-up experiment, we would repeat this method but with temperatures closer to 64 °C so that we can see the full evolution of the MIT.

Analyzing How Temperature Affects Resistance

Determining How Temperature Affects Resistance

Changes of Resistance When Heating and Cooling



To determine when MIT occurred, we measured the resistance of VO₂ at different temperatures. Our group did this by attaching two wires to the film and recording the amount of resistance the material had by looking at an ohmmeter. We changed the temperature through electric heating.

We could tell when VO₂ changed from a conductor to an insulator and vice versa when the resistance rapidly increased or decreased. When plotting a graph of temperature versus resistance, we noticed that when temperature increased, resistance decreased. This meant that VO₂ was becoming more and more metallic as temperature rose. Likewise, as temperature decreased, resistance increased so the VO₂ film was behaving more like an insulator. Our group conducted two experiments: one testing the resistance of the material when heating it (represented by the red points) and the other testing the resistance of VO₂ when cooling it (represented by the blue points). We knew that when we cooled VO₂, it would behave more like an insulator and resistance would increase. However, we noticed that during the cooling process, the resistance did not go back the same way as it did during the heating process.

Temperature (°C)	Resistance When Heating (Ω)
61.85	140000
62.85	130000
64.85	95000
65.85	59000
66.35	42000
66.85	20000
67.35	4000
67.85	362
68.35	92.6
69.35	38.7
71.85	24.6

Temperature (°C)	Resistance When Cooling (Ω)
72	21.46
68.85	26.51
66.3	42
65.85	59.17
65	300
64.5	3000
64	11445

References

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Acknowledgments

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