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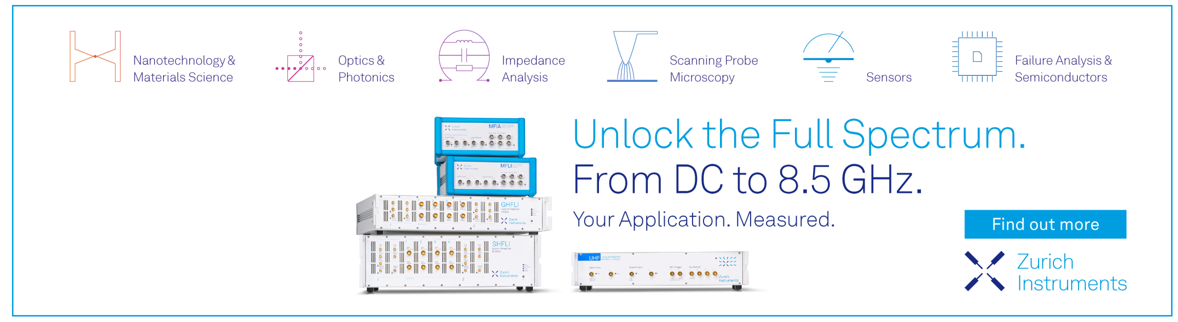

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ABSTRACT

The growth of VO_2 epitaxial films has been researched extensively for obtaining excellent phase-transition performance. However, previous methods typically necessitate high temperatures or post-annealing processes, which elevate both experimental complexity and cost. In this work, we prepared high-quality VO_2 epitaxial films by reactive magnetron sputtering directly under a low growth temperature. Benefiting from the determination of the oxygen pressure ratio from the theoretical analysis of the sputtering process model, single-stoichiometric VO_2 epitaxial films could be prepared under 450 °C with a resistance change of 10^3, and above 500 °C with a resistance change exceeding 10^4. The mechanism of achieving low-temperature growth of VO_2 epitaxial films was analyzed utilizing Thornton's zone model; finally, the epitaxial characteristics of VO_2 on the sapphire substrate were confirmed from in-plane and out-of-plane directions. This work presents a guideline for the low-temperature growth of VO_2 epitaxial films with enhanced phase-transition performance, thereby reducing both the cost and the requirements associated with the epitaxial growth of VO_2 films.

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VO_2 has become one of the most popular strongly correlated materials due to its profound physical mechanisms and various changes in physical properties during the phase transition.[1] Around 340 K, it undergoes the metal–insulator transition (MIT), accompanied by a structural phase transition from the low-temperature monoclinic phase (P21/c) to the high-temperature tetragonal rutile phase (P42/mnm). The physical properties of VO_2 films such as electrical resistance,[2–4] optical transmittance,[5] and macroscopic volume[6] exhibit changes across the MIT, which can be triggered by various stimuli.[7–10] Utilizing these properties, various novel devices such as microelectromechanical devices,[11,12] infrared detector,[13,14] camouflage,[15] and actuator[16–18] have been widely studied. The performance of advanced VO_2 devices critically depends on film quality, and compared with polycrystalline VO_2, the epitaxial VO_2 can further enhance the MIT property. However, the preparation of pure stoichiometric VO_2 epitaxial films has been a significant challenge due to vanadium oxides being sensitive to oxygen, and the growth usually needs high temeprature.[19,20]

Various techniques have been used to grow VO_2 epitaxial films.[21–24] For instance, a VO_2 epitaxial film was prepared using RF-plasma assisted oxide molecular beam epitaxy (MBE) under 550 °C, which had a third-order-of-magnitude resistance change (ΔR) across the MIT.[23] It could also be grown under 400 °C by MBE and post-annealing, but the ΔR was only 25.[24] Pulsed laser deposition is often employed for the preparation of VO_2 epitaxial films as well.[25,26] Under the growth temperature of 600 °C, a VO_2 epitaxial film was grown on sapphire, showing fourth-order-of-magnitude ΔR.[26] In addition, for producing high-quality and large-scale thin films, the sputtering technique has proven to be an efficient method.[27–29] The published works employed ion-beam sputtering,[30,31] DC, or RF-sputtering.[32–34] Among them, an epitaxial VO_2 film was prepared under the growth

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temperature of 590 °C, demonstrating a high ΔR of 10^4 and infrared modulation of 50%.[35] Some work also attempted to grow VO_2 films below 400 °C, but the polycrystalline nature of VO_2 only allows for a three-order-of-magnitude change in resistance.[36] Additional treatments such as post annealing,[37] substrate bias,[38] and seed layer[39] were often required. Until now, there is still a lot of work dedicated to growing high-quality VO_2 epitaxial films using magnetron sputtering. However, most of them require high growth temperatures even reaching up to 650 °C[40] or post-annealing treatments.[33,41]

In this study, we realize the growth of the VO_2 epitaxial film by reactive magnetron sputtering under growth temperature as low as 450 °C through systematically investigating the growth conditions. With the help of the sputtering process model, the theoretical oxygen pressure ratios required for the formation of VO_2 was analyzed, and different stoichiometric vanadium oxides were prepared in the experiment according to the theoretical condition. Based on the obtained precise oxygen pressure ratio, a single-stoichiometric VO_2 film was grown on a sapphire substrate under different growth temperatures. Subsequently, the nanomembrane quality was characterized by x-ray diffraction (XRD), Raman spectroscopy, and temperature-dependent resistance measurements, confirming the property and epitaxial relation with the sapphire substrate of a VO_2 film.

For preparation of a VO_2 film by magnetron sputtering, a metal vanadium target and a DC power of 200 W were utilized under a background vacuum of E^{-6}Torr. The influence of O_2 pressure ratio was initially investigated using argon (Ar) as the working gas at a fixed pressure of 13 mTorr. To determine the O_2 pressure ratios for VO_2 growth, the sputtering process model is established as shown in Fig. 1(a).[42] There are mainly four steps during the growth: gas injection, Ar ionization, sputtering V atoms, and V reacting with O_2. When a specific pressure of Ar is introduced, determining the amount of O_2 required to obtain VO_2 can be achieved by estimating the quantity of V atoms involved in the reaction through the two intermediate steps. (i.e., Ar ionization and sputtering V atoms). The ionization efficiency is used to describe how much Ar can be ionized into Ar^+ to bombard the V target. According to previous reports, the ionization efficiency of Ar in our experiment is approximately 60%.[43,44] Moreover, when an Ar^+ bombards the V target, the amount of V atoms that can be sputtered out is called the sputtering yield.[45] Based on the applied DC power and gas pressure, the estimated sputtering yield of V atoms is approximately 10%.[46,47] As a result, the number of sputtered V atoms can be calculated according to the Ar pressure introduced. Assuming the sticking coefficient is approximately 1, meaning all sputtered V atoms can react with O_2,[48] the theoretical O_2 ratios required to

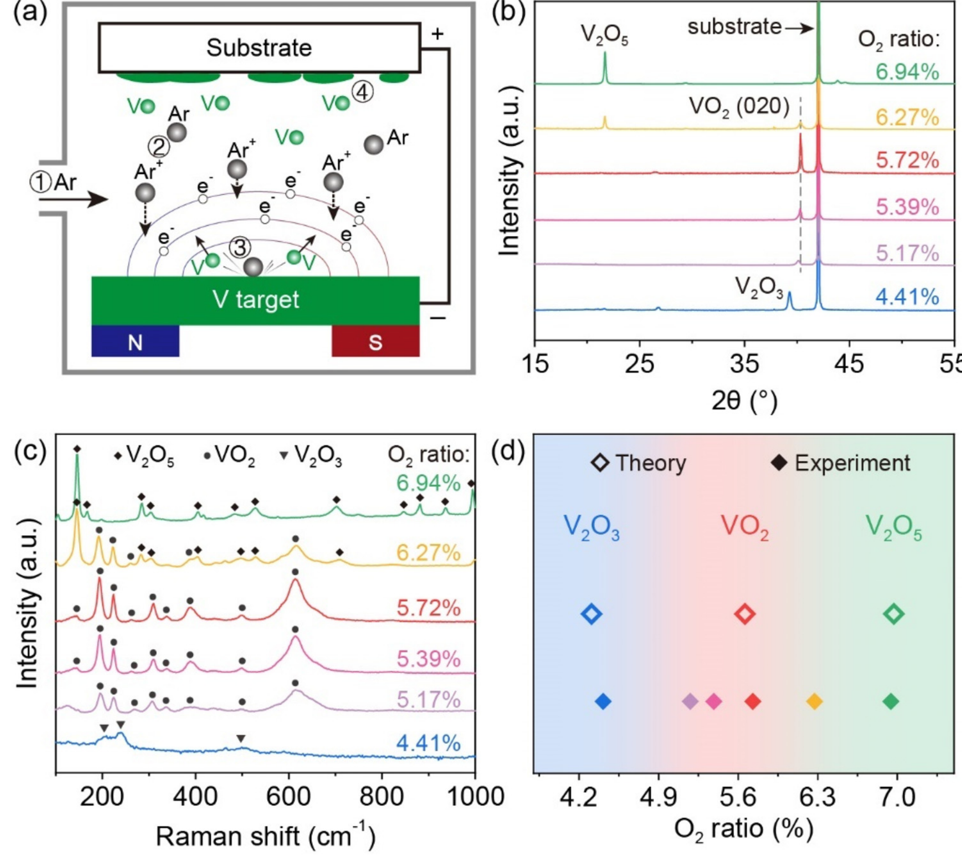

FIG. 1. The effect of O_2 pressure ratio on the formation of vanadium oxides. (a) Schematic diagram of the sputtering growth process of the vanadium oxide film. (b) XRD 2θ-ω curves of vanadium oxide films under different O_2 pressure ratios. (c) Raman spectroscopies of vanadium oxide films under different O_2 pressure ratios. (d) Major phases of sputtered vanadium oxides as a function of O_2 pressure ratios.

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produce V_2O_3, VO_2, and V_2O_5 were calculated to be 4.31%, 5.66%, and 6.97%, respectively.

According to the calculation, we varied the O_2 pressure to 0.6, 0.71, 0.74, 0.79, 0.87, and 0.97 mTorr, and the proportion of O_2 under the total pressure was 4.41%, 5.17%, 5.39%, 5.72%, 6.27%, and 6.94%, respectively. The XRD 2θ curves shown in Fig. 1(b) are to detect structures of vanadium oxide films obtained at different O_2 ratios. It can be observed that when the O_2 ratio is 4.41%, the vanadium oxide film mainly exhibits diffraction peaks of V_2O_3, indicating insufficient O_2 content and the formation of low-valence state vanadium oxides. With the O_2 ratio increasing to 5.17%, the weak VO_2 (020) diffraction peak indicates the formation of the VO_2 phase. Subsequently, the VO_2 diffraction peak intensity gets stronger as the O_2 ratio increases, reaching its maximum when the O_2 ratio reaches 5.72%. However, further increasing O_2 ratio to 6.27% results in the coexistence of VO_2 and V_2O_5 phases, indicating that excessive O_2 oxidizes part of the VO_2 to the higher valence state V_2O_5, and the O_2 ratio of 6.94% leads to the formation of V_2O_5 completely. Raman spectroscopy is also utilized to confirm the phases of vanadium oxides shown in Fig. 1(c), and the results show that the phases corresponding to the Raman vibrational peaks are consistent with the XRD results. By comparing the results in the experiment and in theory in Fig. 1(d), it can be concluded that the experimental results are basically consistent with the theoretical predictions.

Upon determining the O_2 pressure ratio required for VO_2 growth, it is advantageous for investigating the growth temperature's influence because we only need to consider the growth temperature's effect on the crystalline quality of VO_2 film, without having to account for compositional deviations. Thus, VO_2 films were grown at a series of temperatures, including 400, 450, 500, 550, and 600 °C under optimized gas environment conditions. The structure and phase of VO_2 films obtained under various growth temperatures are checked using XRD and Raman spectroscopy. As shown in Fig. 2(a), XRD 2θ curves illustrate that no diffraction peaks are detected at 400 °C, indicating that 400 °C is too low to be favorable for the crystallization of a VO_2 film. Subsequently, as the growth temperature increases to 450 °C, the VO_2 (020) plane diffraction peak appears, which means the realization of the epitaxial growth. Furthermore, the intensity of diffraction peak becomes stronger, indicating that higher temperature promotes the crystallization of VO_2 films, resulting in a significant improvement in film quality. Although all the Raman peaks in the Raman spectra in Fig. 2(b) belong to the monoclinic phase of VO_2, the peak intensity of VO_2 film grown at 400 °C is weaker than that of VO_2 films grown above 450 °C, showing the same trend as the XRD results.

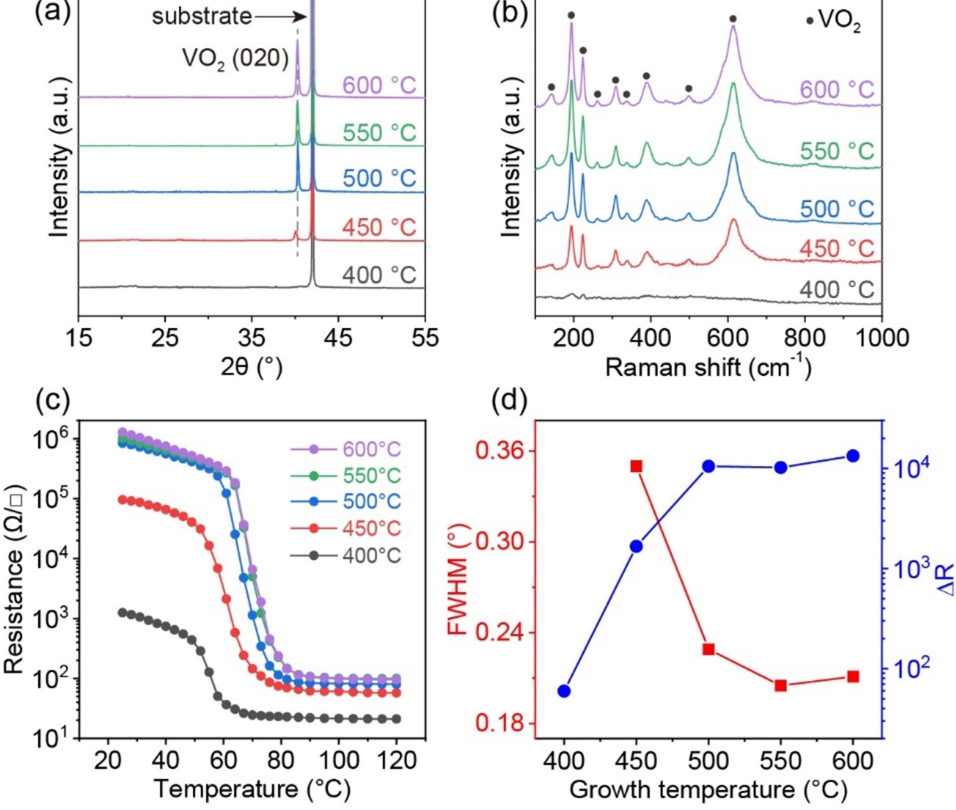

FIG. 2. The effect of growth temperature on VO_2 films. (a) XRD 2θ-ω curves of VO_2 films grown under different growth temperatures. (b) Raman spectroscopies of VO_2 films grown under different growth temperatures. (c) Temperature-dependent electrical resistance of VO_2 films grown under different growth temperatures. (d) FMHW of XRD diffraction peaks and ΔR of VO_2 films as the variation of growth temperatures.

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The electrical MIT properties are also characterized by temperature-dependent resistance curves as shown in Fig. 2(c). When the growth temperature is 400 °C, the film shows ΔR of ∼59 during the heating process, indicating a poor crystallization of the VO_2 film. The temperature-dependent resistance curve changes steeper during the phase transition when the growth temperature of VO_2 film is 450 °C, and the ΔR is $\sim10^3$, which is comparable to the electrical MIT performance of most VO_2 thin films.[20,33,41,49,50] It is already feasible to achieve the fabrication of good VO_2 epitaxial films at 450 °C. Further increasing the growth temperature to 500, 550, and 600 °C, the MIT phenomenon gets further enhanced with the ΔR of $\sim10^4$, which is the benchmark of high quality for a VO_2 epitaxial film.[2,3,51,52] By comparing the FWHM of XRD peaks and the ΔR, as shown in Fig. 2(d), it can be seen that both the crystalline quality and the ΔR during the MIT increase with increasing growth temperature from 400 to 500 °C, and they remain relatively constant above 500 °C.

To get a deeper understanding of why epitaxial growth of VO_2 films can be realized under lower growth temperatures, we analyze the grain morphology with the help of Thornton's zone model. The scanning electron microscope (SEM) images of VO_2 films grown under different growth temperatures are shown in Figs. 3(a)–3(e). At 400 °C, it can be observed that the size of the grains is small, and their arrangement is extremely disordered. As the temperature increases to 450 °C, the size of grains becomes more uniform. Then, with further increase in the growth temperature, the size of VO_2 grains also increases. A statistical analysis of the grain size of VO_2 films at each temperature [Fig. 3(f)] reveals that the size of VO_2 grains increases with the rise of growth temperature. The basic sputtering process has been known and can be described as some steps: transportation of the species from the target to the substrate; adsorption and diffusion of the species on the substrate; removal of extra species from the substrate; and moving of coating species to the final position to form the film. The diffusion step is controlled mainly by the substrate temperature and may significantly affect the quality of the sputtered film. According to the substrate temperature (T) and coating material melting point (T_m), the coating microstructure can be divided into several zones (Thornton's zone model).[53–55] In Zone 1 (or T), where $T/T_m < 0.3$, since the melting point of VO_2 is approximately 1525 °C,[33] growth temperature of 400 °C falls into this region, under which the atom migration is extremely insufficient due to the low temperature, resulting in smaller grain sizes and poor quality of VO_2 films. For a growth temperature of 450 °C, the T/T_m is ∼0.295, which is close to Zone 2, where $0.3 < T/T_m < 0.5$, and growth temperatures of 500, 550, and 600 °C are all within this range. Compared with Zone 1, Zone 2 atoms gain sufficient energy for migration on the substrate surface, and the grains gradually merge and grow larger, forming regularly arranged columnar crystals and a smoother surface. Thus, the VO_2 films grown in Zone 2 have better MIT performance. This is why VO_2 film could achieve good epitaxial growth under growth temperatures as low as 450 °C. Zone 3, where $0.5 < T/T_m$, implies that the growth temperature of the VO_2 film needs to be greater than 760 °C. At this point, the grains further grow toward equiaxed grains. However, since current equipment cannot reach such high temperatures, further observation is not feasible.

To obtain a high-quality VO_2 epitaxial film based on optimized growth conditions, the epitaxial relationship between the VO_2 and sapphire substrate is also analyzed. XRD φ-scan results for the VO_2 film (011) plane and the sapphire substrate $(01\bar{1}2)$ plane are shown in Fig. 4(a). The sapphire substrate exhibits three diffraction peaks spaced at 120° apart, indicating the tri-symmetry of the sapphire $(01\bar{1}2)$ plane about the c-axis. In contrast, the (011) plane of VO_2 exhibits six diffraction peaks spaced at 60° intervals. In Fig. 4(b), further characterization through pole figure reveals that only six VO_2 diffraction spots are observed within the 0°–90° in-plane range, indicating that from the out-of-plane direction, there is a unique epitaxial orientation of the VO_2 films, while from the in-plane direction, it is neither a single orientation nor polycrystalline. The most plausible explanation, as illustrated in Fig. 4(c), is that the VO_2 crystals (blue) match the sapphire substrate (green) through oxygen atoms, forming a distribution of 120° rotation symmetry in-plane, i.e., three equivalent crystallographic directions with triple rotational symmetry. Consequently, in the XRD φ-scan, the monoclinic VO_2 phase crystal plane, which inherently possesses a double symmetry about the VO_2 [020] crystallographic direction, combined with the in-plane triple rotational distribution, results in six diffraction peaks, which is consistent with observations reported in the literature.[25,56] The results confirm the good epitaxial growth of the VO_2 film on the c-plane sapphire substrate.

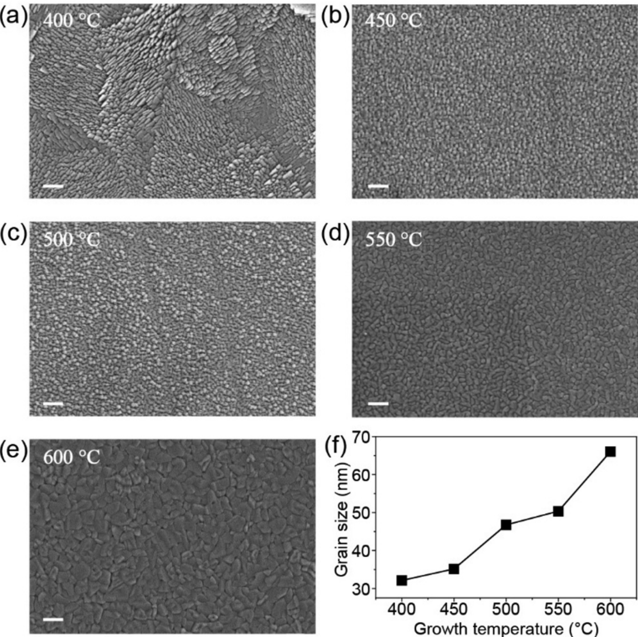

FIG. 3. Morphology of VO_2 films grown under different temperatures. (a)–(e) SEM images of grains of VO_2 films grown under 400, 450, 500, 550, and 600 °C, respectively. Scale bars: 200 nm. (f) Grain sizes of VO_2 films with the variation of growth temperature.

In conclusion, we realize good epitaxial growth of VO_2 films using reactive magnetron sputtering under a growth temperature as low as 450 °C. Based on the sputtering process model, the O_2 pressure ratio needed for VO_2 growth was calculated precisely. Different stoichiometric vanadium oxides could be prepared according to the theoretical O_2 pressure ratios. Benefiting from the optimized gas condition, the ΔR of VO_2 epitaxial film grown at 450 °C exhibits 10^3, while VO_2 epitaxial film grown at 500 °C and above shows a ΔR of 10^4, which indicates a high phase transition performance. By analyzing the morphology of VO_2 grains along with Thornton's zone model, the reason

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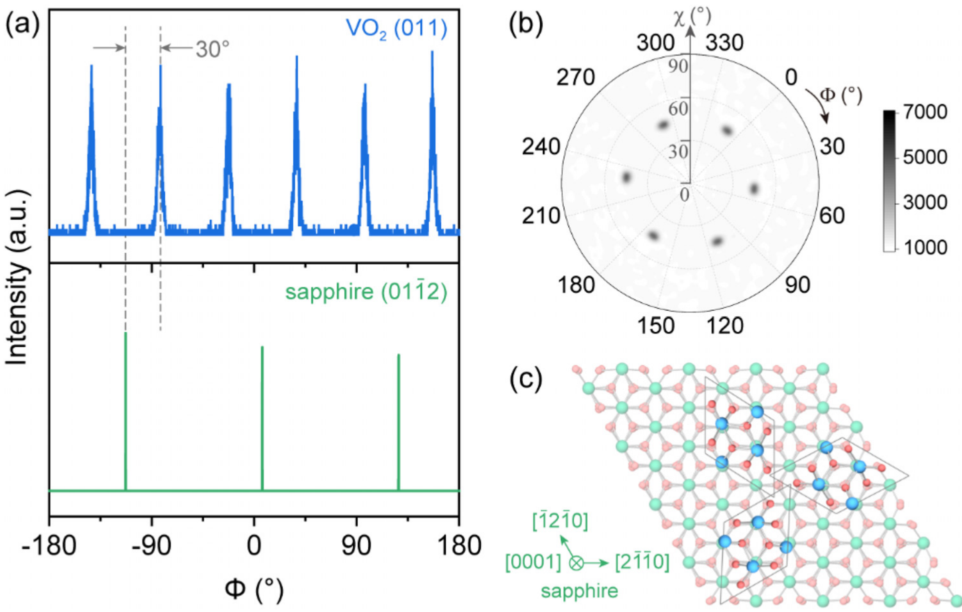

FIG. 4. High-resolution XRD analysis between epitaxial the VO_2 film and sapphire substrate. (a) XRD φ-scan of VO_2 (011) plane and sapphire (01$\bar{1}$2) plane. (b) Pole figure of VO_2 (011) plane. (c) Schematic diagram of epitaxial relation between the VO_2 film and sapphire substrate from out-of-plane direction (blue atoms: V, green atoms: Al, red atoms: O).

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why epitaxial growth of VO_2 films can be realized under low growth temperatures was further elucidated. Finally, the epitaxial relationship between the VO_2 film and the c-plane sapphire substrate was determined, confirming the high epitaxial quality. This work provides a more convenient and achievable method for realizing low-cost and large-area growth of VO_2 epitaxial films.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Chang Liu: Conceptualization (equal); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Validation (equal); Visualization (lead); Writing – original draft (lead); Writing – review & editing (equal). **Zhi Zheng:** Formal analysis (supporting); Writing – review & editing (equal). **Xing Li:** Formal analysis (supporting); Writing – review & editing (equal). **Yang Wang:** Formal analysis (supporting); Writing – review & editing (equal). **Xiang Dong:** Methodology (supporting). **Gaoshan Huang:** Funding acquisition (equal). **Yongfeng Mei:** Conceptualization (equal); Funding acquisition (equal); Validation (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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