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Direct Evidence of the Magnetic Properties of V_2O_5 Thin Films Using X-Ray Magnetic Circular Dichroism

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1. INTRODUCTION: Vanadium oxides exhibit a rich spectrum of physical properties that render them highly attractive for applications in non-volatile data storage and optoelectronic devices such as smart windows. Owing to vanadium's ability to adopt multiple oxidation states, a variety of stoichiometric oxides can form, including VO, V_2O_3, VO_2, V_6O_{11}, and V_2O_5[1-3]. Among these, V_2O_5 stands out as one of the thermodynamically most stable phases due to its fully oxidized state. Crystallizing in a distorted orthorhombic structure, V_2O_5 exhibits a layered arrangement that facilitates intercalation processes and anisotropic transport phenomena [1-3]. These structural attributes, combined with its unique electronic, optical, and chemical properties, have positioned V_2O_5 thin films at the forefront of research into functional oxide materials. A variety of deposition techniques have been employed to fabricate V_2O_5 films, including thermal and electron-beam evaporation, atomic layer deposition, RF sputtering, spray pyrolysis, and chemical solution methods such as sol–gel processing [1–2]. Among these, the sol–gel technique offers notable advantages, including compositional control, uniform film morphology, high purity, and scalability, making it especially suitable for large-area device fabrication.

Extensive experimental investigations have been conducted on vanadium pentoxide (V_2O_5), elucidating its structural, optical, and electronic properties. X-ray diffraction analyses consistently confirm that V_2O_5 crystallizes in an orthorhombic lattice, characterized by layered arrangements conducive to intercalation and anisotropic conduction. Optical characterization indicates an indirect band gap of approximately 2.3 eV, with variations attributed to film thickness, grain size, and deposition conditions [1-2]. The optical band gap is typically extracted using Tauc plot analysis, which substantiates the material's semiconducting nature. While bulk V_2O_5 is inherently diamagnetic, emergent magnetic behaviour has been observed in doped or defect-engineered forms, such as sodium-intercalated phases or oxygen-deficient structures. Notably, the magnetic response of pristine V_2O_5 thin films remains largely underexplored, particularly those synthesized via the sol–gel method. Most prior magnetization studies have focused on hybrid systems or composite oxides, leaving a significant gap in understanding the intrinsic magnetic behaviour of sol–gel derived V_2O_5 films.

In this study, we investigate the electronic structure and magnetic response of V_2O_5 thin films through element-specific X-ray absorption spectroscopy (XAS). This technique provides detailed insight into the local electronic states and oxidation environment of vanadium ions. To probe the origin and nature of any emergent magnetism, X-ray magnetic circular dichroism (XMCD) measurements were performed at the vanadium $L_{2,3}$ edges..

2. EXPERIMENT: Pristine V_2O_5 thin films were synthesized via a chemical solution deposition method employing high-purity vanadium pentoxide powder (>99.9%) as the vanadium source. A precursor solution was prepared by dissolving 0.3 g of V_2O_5 in 30 mL of 30% hydrogen peroxide (H_2O_2) under continuous stirring. Upon dissolution, a yellow peroxovanadate complex (HVO_4) solution formed, which was subsequently stirred at 60 °C for 1 h under ambient conditions. This led to the formation of a red-brown viscous gel, indicating gelation of the precursor. The resultant solution was filtered through a 0.45 µm syringe filter to eliminate any undissolved particulates, rendering it suitable for thin film deposition.

The filtered sol was spin-coated onto three types of substrates: bare glass, indium tin oxide (ITO)-coated glass, and Si(111), each of which was pre-cleaned sequentially in acetone, ethanol, and deionized water using ultrasonic agitation. Spin coating was carried out at 3000 rpm for 30 s, followed by thermal drying at 80 °C for 15 min after each deposition cycle. The multilayered films were then annealed in air at 400 °C for 4 h to promote crystallization and remove residual organics.

Structural characterization was performed using grazing incidence X-ray diffraction (GIXRD, Bruker D8) with Cu Kα radiation (λ = 1.5406 Å), using an incident angle of 0.2° and an angular step size of 0.0025°. Detailed specifications of the characterization tools are provided elsewhere [1-2].

3.RESULTS AND DISCUSSIONS: X-ray absorption spectroscopy (XAS) and X-ray magnetic circular dichroism (XMCD) measurements were carried out at the variable-polarization soft X-ray beamline BL-16A at the Photon Factory (KEK, Japan). The experimental geometry for both XAS and XMCD is shown in Fig. 1. The samples were positioned in a vacuum chamber with a pressure of 10^{-9} Torr and subjected to an external magnetic field using a 5 T superconducting magnet. XAS and XMCD spectra were recorded at magnetic field strengths of ±0.1 T, ±1 T, and ±2 T, with only the data collected at ±1 T presented here. Right and left circularly polarized (RCP and LCP) X-rays were used, with an energy resolution of 0.1 eV, employing the surface-sensitive total electron yield (TEY) method at the vanadium $L_{2,3}$ edges and oxygen K-edge.

Previous studies by Gupta *et al.* and Ritika *et al.* have demonstrated that oxygen vacancies in V_2O_5 lead to ferromagnetic phases at room temperature [1-2]. This highlights the importance of conducting element-specific spectroscopic studies to elucidate the underlying mechanisms behind the observed magnetic behavior. The XAS spectra collected at magnetic fields of ±2.0 T are denoted by μ^+ and μ^-, representing right and left circularly polarized light, respectively, as shown in **Fig. 1**. XMCD spectra were obtained by subtracting the XAS data recorded with positive and negative helicities of the circularly polarized light.

The XAS spectrum reveals vanadium absorption peaks at the L_3 edge (518.5 eV) and the L_2 edge (524.7 eV). Additionally, multiplet peaks with lower intensities were observed in the pre-edge (515.4 eV) and post-edge (517.3 eV) regions of the L_3 edge, which is consistent with vanadium in the 5+ oxidation state. This behavior aligns with the localized nature of the V 2p → 3d transitions, which are influenced by the surrounding distorted atomic environment. The XMCD signal recorded at a magnetic field of B = ±2.0 T, well beyond the saturation point, shows a clear ferromagnetic response at the vanadium edges.

The enhanced ferromagnetic behaviour observed in the sample is attributed to a combination of increased crystallinity and a higher concentration of oxygen vacancies, which are known to introduce localized magnetic moments. These vacancies can lead to the partial reduction of vanadium from V^{5+} to V^{4+}, facilitating exchange interactions that stabilize the ferromagnetic alignment. This observation is further corroborated by first-principles theoretical calculations, which predict that oxygen-deficient V_2O_5 systems exhibit a more stable ferromagnetic ground state compared to nonmagnetic or antiferromagnetic states, driven by the formation of unpaired d-electrons localized at vacancy sites. The presence of oxygen vacancies is further validated by the O *K*-edge XAS spectrum, shown in Fig. 1. Using sum-rule [4-8], anyone calculate spin and orbital magnetic moments.

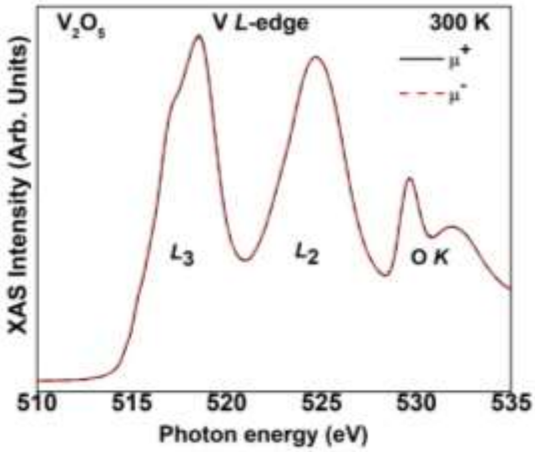

Figure 1. V *L*-edge and O *K*-edge of V_2O_5 thin films.

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