Plasma Treatment Strategies for Tailoring the Thickness and Surface Chemistry of VSe_2 and V_2O_5 for Magnetic Material Design

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Introduction

Two-dimensional (2D) van der Waals (vdW) materials have garnered significant attention due to their unique electronic, magnetic, and optical properties, making them promising candidates for applications in next generation nanoelectronics, quantum computing, and energy storage devices [1, 2]. These materials are typically fabricated using methods such as mechanical exfoliation, chemical vapor deposition (CVD), or molecular beam epitaxy (MBE). However, certain post-processing techniques, such as plasma treatment, can be essential for modifying their structural and electronic properties, which are essential for the development of more efficient electronic and optoelectronic devices. Specifically, these post-processing steps could play a key role in advancing quantum devices [1].

Plasma-based treatments can be broadly categorized into plasma etching and plasma cleaning, each serving distinct roles in nanofabrication and surface engineering [2]. Plasma etching is a process that utilizes reactive ions and radicals to selectively modify or remove specific material layers, making it useful for precise patterning and thickness control of 2D materials [1, 2]. Techniques such as reactive ion etching (RIE) or inductively coupled plasma (ICP) etching have been employed for materials like MoS_2, graphene, and VSe_2, allowing selective thinning or structuring while preserving underlying layers [1].

However, the weak interlayer bonding and high surface sensitivity of vdW materials necessitate precise control over plasma parameters to minimize unwanted damage and defect formation [2].

Downstream plasma cleaning is a less aggressive technique and is primarily used to remove contaminants, residues, or organic layers without significantly altering the underlying material [3,4]. This process typically employs O_2, Ar, or N_2 based plasmas to eliminate surface adsorbates, making it valuable for substrate preparation, residue removal post-lithography, and improving interfacial adhesion in [3]. Unlike plasma etching, plasma cleaning does not involve substantial material removal, though minor surface modifications can still occur, particularly in oxygen-sensitive transition metal dichalcogenides (TMDs) [3].

The ability to precisely control plasma parameters—such as gas composition, power, pressure, and exposure time determines whether a process behaves as an etching or cleaning step. This distinction is particularly critical for vanadium-based 2D materials, where selective etching of oxide phases (e.g., V_2O_5) while preserving the parent VSe_2 structure enables the design of materials with enhanced magnetic and electronic properties [1,2].

Procedure:
In this study, VSe_2 flakes were mechanically exfoliated onto a SiO_2/Si substrate, followed by Raman spectroscopy conducted both before and after plasma treatment to assess structural and chemical modifications. The micro-Raman spectrometer in-Via (Renishaw, UK), equipped with a reflected light microscope, allowed for precise analysis of very small areas—down to a few micrometers in size. The system was configured with objective lenses at magnifications of 5x, 20x, and 50x and a motorized sample stage, enable highly accurate positioning of the sample for analysis. The spectrometer uses 488 nm laser excitation, and the Raman shift resolution was 0.5 cm^{-1}, providing detailed spectral data. The primary objective of this experiment was to evaluate the threshold limits of RF plasma exposure in terms of its impact on 2D VSe_2 flakes, specifically investigating the extent to which the process remains non-destructive under these conditions.

The samples were processed using two different ICP plasma designs. The instrument from SPTS is called an Advanced Oxide Etcher (AOE) with high power (ICP coil **and** platen power). The second instrument used was the GVX Downstream Asher, developed by ibssGroup, for eliminating hydrocarbon contamination in electron microscopes including SEM, TEM, and FIB. The system was installed on the SEM Nova Nano230 (ThermoFisher, Figure 1). It generates a downstream plasma, producing reactive oxygen and hydrogen species that chemically react with hydrocarbons, converting them into gaseous byproducts such as CO, CO_2, and H_2O. These byproducts are then efficiently removed from the vacuum chamber, ensuring a cleaner environment for high-resolution imaging. Unlike conventional plasma cleaners that rely on kinetic sputtering, the GVX employs a gentler chemical etching process, protecting delicate samples and instrument components. Please see Table 1 for the detailed instrumental conditions.

Results & Discussion
The instrument from ibssGroup is designed to remove surface carbon. The impact of plasma treatment on the structural integrity of VSe_2 was evaluated for surface damage. As illustrated in Figure 2(a), the VSe_2 flake remains structurally intact following exposure to the downstream plasma.

Sister samples were processed thru the (Advanced Oxide Etcher); results are presented in Figure 3 which illustrates the effects of AOE plasma treatment on the same VSe_2 crystals. The optical image clearly reveals evidence of surface etching (1/3 of the flake gone). Notably, the region highlighted within the black circle has undergone complete etching, exposing the underlying SiO_2 substrate (SiO2 thickness fringes). The Raman spectrum (black) shows the strong 300cm^{-1} peak of SiO_2, thereby demonstrating the aggressive nature of this plasma.

Conclusion:
Raman spectroscopy of the downstream samples confirmed that the characteristic VSe_2 peak at 207 cm^{-1} exhibited no spectral shift, despite increasing the "effective plasma dose" from 5 to 20 minutes reinforcing the conclusion that the downstream plasma treatment did not induce detectable structural or chemical modifications.

These findings demonstrate that the downstream ICP remote plasma does not adversely affect 2D van der Waals materials, distinguishing it from other plasma processing methods, which typically involve higher power levels and reactive-ion bombardment which may lead to etching or structural damage. [Ref 3]. This suggests that downstream ICP Plasmas are a viable option for surface cleaning and processing without inducing significant material degradation, making it particularly suitable for applications requiring minimal structural or chemical modification of 2D materials.

Table 1 detailed parameters of the two ICP instruments used in this research

Vendor	Etcher Name/Model	Equipment Design Goal	Plasma Source	Recipe Gasses	Active Specie	ICP	Platen	Time
IBSS	Mobile Cubic Asher	Surface Carbon removal	downstream ICP	Room Air+H_2O vapor	Oxygen radicals (2X) Hydrogen radicals	50W	N/A	10mins
SPTS Tech	Advanced Oxide Etcher	Silicon and Dielectrics (SiO2, SiN, SiON)	ICP	Argon and Nitrogen	Nitrogen	700W	100W	10mins

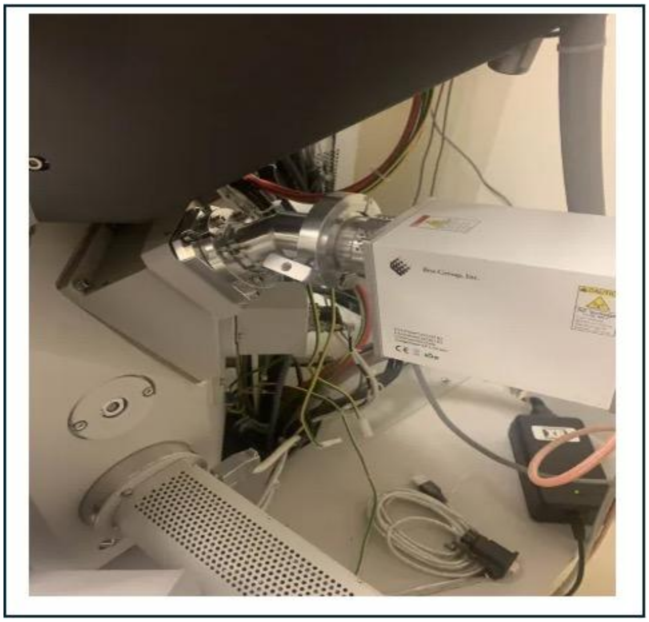

Figure 1 ibssGroup downstream ICP plasma on a NovaNano 230 SEM from ThermoFisher

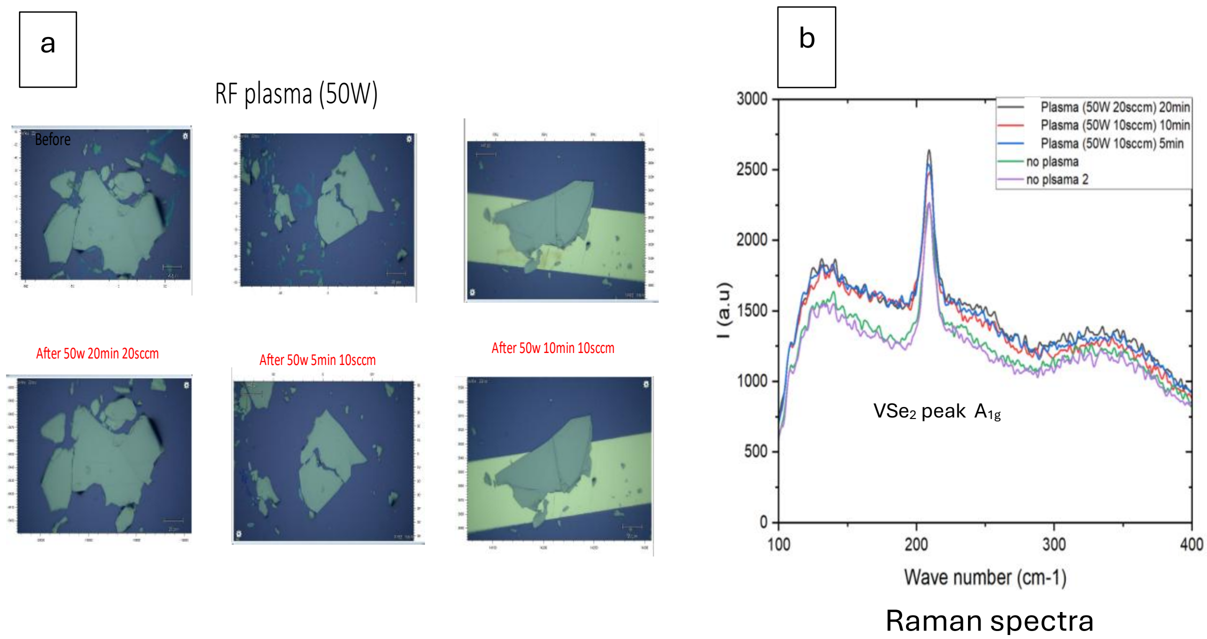

Figure 2 Optical images and Raman spectra of VSe2 flakes before and after downstream ICP plasma

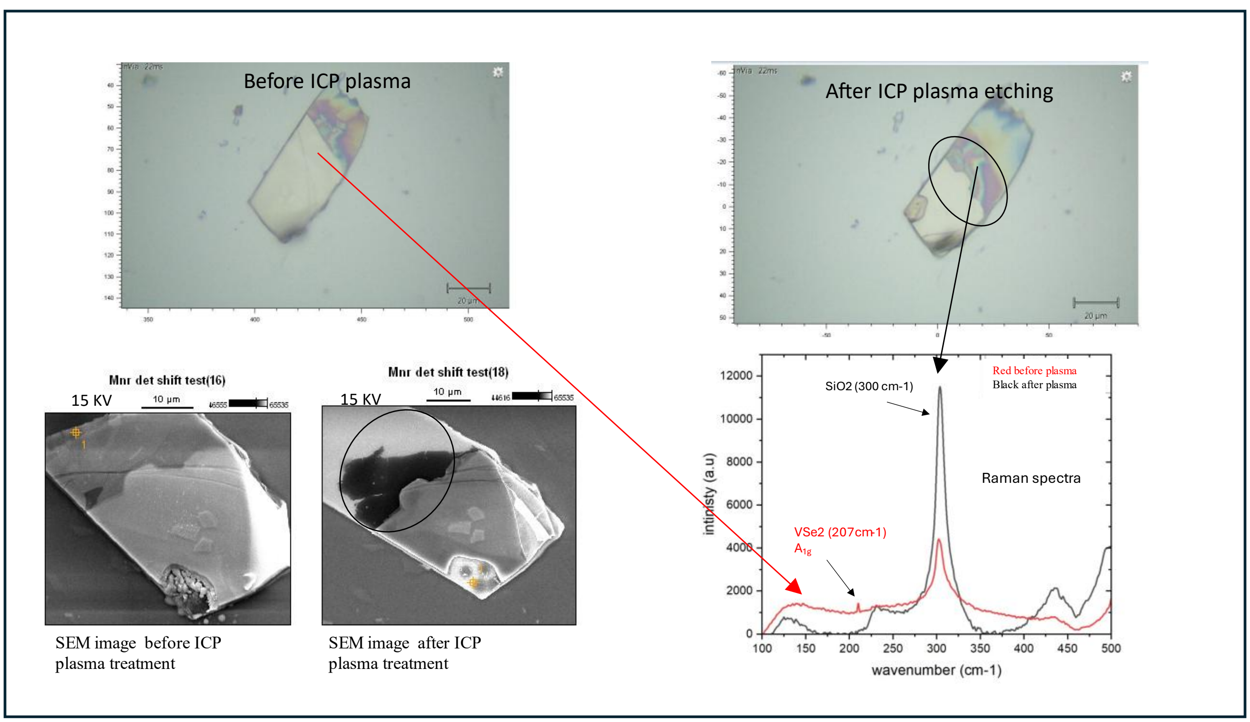

Figure 3 Advanced Oxide Etcher effects on VSe_2 – see the before/after optical images. The effect of this plasma is to eliminate the very small VSe2 peak and remarkable increase the SiO2 signal characteristic of the substrate. The before/after SEM images show considerable surface erosion post Advanced Oxide Etching

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