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V_3Si: AN ALTERNATIVE THIN FILM MATERIAL FOR SUPERCONDUCTING RF CAVITIES*

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Abstract

Superconducting materials such as: V_3Si, NbN, NbTiN and Nb_3Sn, are potential alternatives to Nb for next generation thin film SRF cavities. In comparison to the Nb, their relatively high critical temperature (T_c) could allow for operation at higher temperatures (≥4 K) and the higher critical field could lead to higher accelerating gradients. The deposition of thin film V_3Si using single target high-power impulse magnetron sputtering is investigated on sapphire and niobium substrates. We report on the T_c and relate it to thin film characterisation using X-ray photoelectron spectroscopy, scanning electron microscopy and grazing incidence X-ray diffraction.

INTRODUCTION

Current generation bulk Nb superconducting RF (SRF) cavities are approaching their theoretical limit for performance and next generation accelerators are demanding higher accelerating gradients at higher operational temperatures. Alternative superconducting materials, such as, V_3Si and Nb_3Sn are promising candidates due to their relatively high critical temperatures of T_c = 18 and 17 K, and upper critical fields (H_c) of 720 and 540 mT respectively [1]. These properties may allow for production of thin film SRF cavities that can operate at higher temperatures (≥4 K) and higher accelerating gradients. This would reduce the complexity of the cryogenic systems and the infrastructure of the particle accelerator [2].

V_3Si is a cubic A15 phase superconductor. In bulk form, Hardy *et. al.* measured a critical temperature (T_c) of 17.1 K whilst also reporting the effect of impurities of less than 0.1 % can drastically reduce T_c [3]. V_3Si thin films have been manufactured following a few different methods with varying levels of success: Zhang *et. al.* deposited a vanadium (V) thin film on a silicon (Si) layer and annealed the sample in high vacuum at temperatures ranging from 650 °C to 900 °C, forming V_3Si by interdiffusion of the V into the Si, resulting in a $T_c \geq 13$ K [4]. Deambrosis *et. al.* deposited using dc magnetron co-sputtering and reactive sputtering techniques introducing silane to the process gas to improve Si stoichiometry. On sapphire substrates they reported a T_c of 16.8 K, while on Nb substrates no superconducting phase was observed, due to the large diffusion rate between V and Nb [5].

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A series of V_3Si thin films were deposited on both Nb and sapphire substrates and at varying temperatures using high impulse power magnetron sputtering (HiPIMS). The films are characterised using surface characterisation techniques: Scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS) and grazing incidence X-ray diffraction (GI-XRD). Superconducting performance was characterised by four-point probe (FPP) and SQUID vibrating-sample magnetometer (SQUID-VSM).

EXPERIMENTAL DETAILS

Sample Preparation

Nb foil and sapphire substrates were cleaned in isopropanol and acetone ultrasonic baths removing any adventitious carbon and other contaminates that may be present. The samples were loaded into an ultrahigh vacuum (UHV) load lock and heating to deposition temperature, ≥700 °C, and left to thermally stabilise and for the sample stage to outgas over 12 hrs. Sample is then transferred into the deposition chamber at a pressure of $<1\times10^{-8}$ mbar, whilst substrate is heated. Depositions were conducted using a HiPIMS power supply using the deposition parameters shown in Table 1. Kr was used as the process gas for all depositions at a partial pressure of 3×10^{-3} mbar.

Sample Characterisation

Structural characterisation was performed via GI-XRD using a Rigaku SmartLab with a 9 kW rotating anode Cu source. Thin film composition was obtained by XPS. Spectra were acquired using a non-monochromated Al Kα x-ray source and a Thermo Alpha 110 hemispherical analyser. Survey and core region spectra were acquired with a pass energy of 50 and 20 eV respectively. Surface morphology was imaged by Hitachi SU500 SEM. T_c characterisation was conducted using a cryogenic four point probe for sapphire [6] and Quantum Design MPMS 3 SQUID-VSM.

Table 1: HiPIMS Deposition Parameters

Deposition Parameters		Units
Average Power	300	[W]
Frequency	1000	[Hz]
Pulse Length	10	[µs]
Duty Cycle	10	[%]
Deposition Length	3	[hr]

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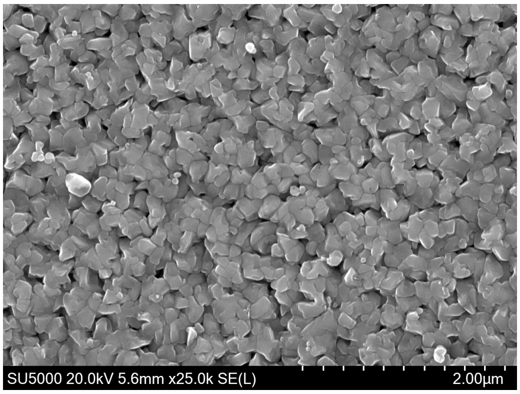
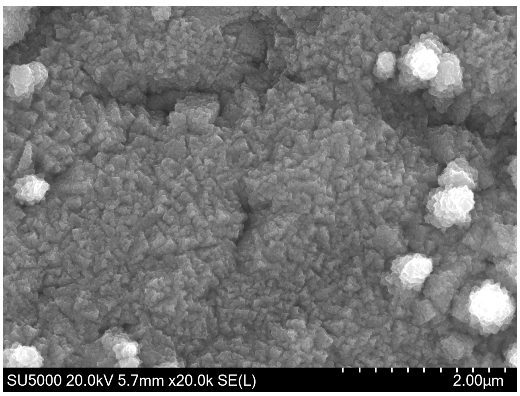


(a) V_3Si thin film on sapphire



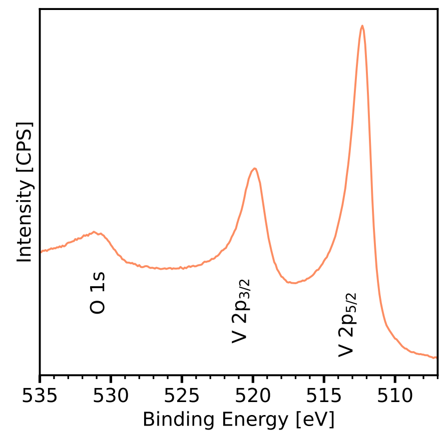
(b) V_3Si thin film on Nb

Figure 1: SEM images of V_3Si thin films on a) sapphire and b) Nb substrates.

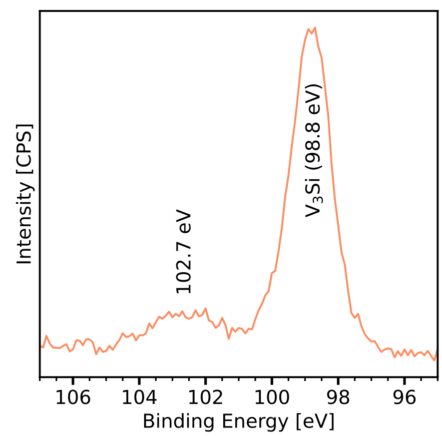
EXPERIMENTAL RESULTS AND DISCUSSION

Figure 1 displays the SEM images of the deposited V_3Si thin films on a) sapphire and b) Nb foil. On the sapphire substrate a V_3Si crystallite structure is observed with small voids existing between grains. This porosity suggests that there is a preferential nucleation on preexisting V_3Si sites instead of the sapphire. The overall topography is related solely to the growth of the film as the underlying sapphire wafer has a roughness of R_a <1 nm. In contrast, the V_3Si crystallites are significantly smaller on the Nb foil which could be related to no preferential nucleation sites on the substrate. The larger structural features are due to the roughness of the Nb substrate used.

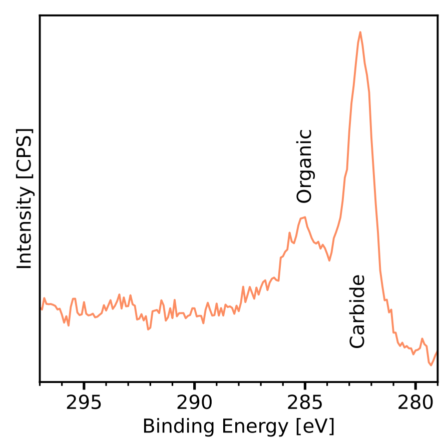
XPS was utilised to determine bulk elemental composition. Figure 2 shows the core region spectra: a) V 2p and O 1s b) Si 2p and c) C 1s present after 4 hours of Kr^+ ion beam sputtering. XPS spectra for the V $2p_{3/2}$ is at a binding energy (BE) of 512.3 eV which corresponds to reference data of metallic vanadium and the Si 2p is shifted by 0.7 eV to 98.7 eV from reference BE for silicon wafer. Contaminates are also present, two C 1s peaks are situated at 282.5 eV and 285.0 eV which aligns with carbide and C-C bonding. A broad O 1s peak is also present at 531.0 eV which couldn't be defined [7]. The oxygen and C-C contamination is likely from the sample being exposed to atmosphere before being loaded into the XPS analysis system. Unfortunately, carbide is introduced during the deposition process and could be from the sputtering target, sample or system out gassing. Zhang *et. al.* [4], measured BEs of 513.0 eV and 99.0 eV for V $2p_{3/2}$ and Si 2p respectively for multiple V-Si phases: V_5Si_3, VSi_2 and V_3Si.



(a) V 2p



(b) Si 2p



(c) C 1s

Figure 2: XPS core region spectra of: V 2p, Si 2p, O 1s and C 1s of a V_3Si film on Nb substrate deposited using HiPIMS.

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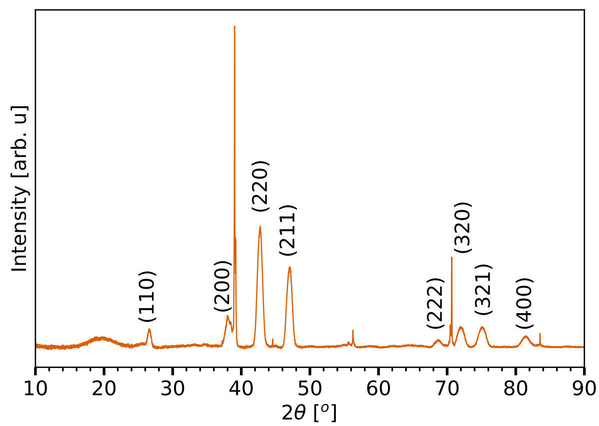

Figure 3: Grazing incidence X-ray diffraction of a V_3Si thin film on Nb (background subtracted). The labelled Miller indices correspond to V_3Si and the unlabelled peaks correspond to the Nb substrate.

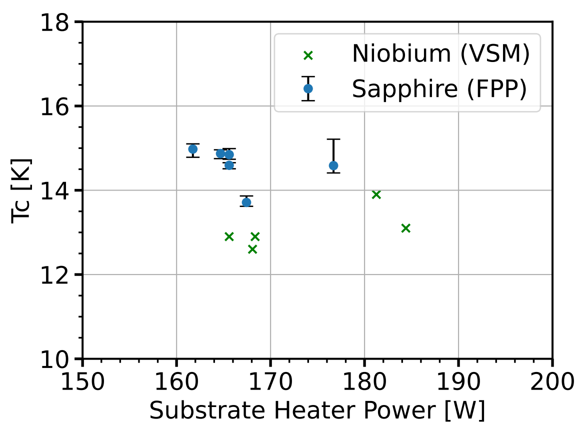

Figure 4: Measured T_c of a series of V_3Si thin films deposited on Nb and sapphire using HiPIMS, at range of substrate heater power.

Additionally, GI-XRD was conducted on the V_3Si film on Nb (Fig. 3) and confirms the formation of the superconducting A15 phase of V_3Si. The peaks associated with the V_3Si are broad suggesting small crystallites of random orientation, which is in agreement with the SEM images previously discussed. The XRD shows no presence of other V-Si phases and the unlabelled sharp peaks are associated with the Nb substrate.

Figure 4 shows the T_c of all the V_3Si depositions on both Nb and sapphire. T_c was obtained using a four point probe for sapphire and vibrating sample magnetometer for Nb substrates. Samples were deposited at varying substrate temperatures, monitored by the heater power. Due to the high temperature required for the formation of superconducting A15 V_3Si phase, the heater power was adjusted close to the systems maximum capability. The highest T_c measured was 14.85 K and 13 K for sapphire and Nb respectively. The lower T_c on the Nb substrates is likely related to the smaller crystallite structure. The oxygen and carbon contamination present in the film explains the T_c being lower than the theoretical maximum on both substrates.

CONCLUSION

Experiments conducted show promise for V_3Si thin films deposited using HiPIMS. Superconducting V_3Si thin films have been successfully grown on sapphire inline with previous research and on Nb substrates. SEM shows a small crystallite structure which is in agreement in GI-XRD. The T_cs measured are still below bulk value which could be as a result of the crystal growth or the contamination observed in the XPS. Additionally, the variation between sapphire and Nb substrates is related to the formation of preferential nucleation sites.

FUTURE WORK

The deposition parameters can be explored further by: adjusting of the duty cycle, introducing Bipolar HiPIMS and increasing substrate temperature. This may show improvement of the V_3Si film by increasing crystallite size. UHV annealing techniques and post processing such as flash [8] or laser annealing is also desirable as it has the potential to improve film crystallisation and reduce defect states.

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