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DEPOSITION AND CHARACTERISATION OF V_3Si FILMS FOR SRF APPLICATIONS*

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Abstract

A15 superconducting materials, like V_3Si and Nb_3Sn, are potential alternatives to Nb for next generation thin film SRF cavities when operated at 4 K. Their relatively high T*c* and superconducting properties could allow for higher accelerating gradients and elevated operating temperatures. We present work on the deposition of V_3Si thin films on planar Cu substrates and an open structure 6 GHz cavity, using physical vapour deposition (PVD) and a V_3Si single target. The surface structure, composition and DC superconducting properties of two planar samples were characterised via secondary electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX) and in a magnetic field penetration facility. Furthermore, the first deposition using PVD of a V_3Si film on a 6 GHz split cavity and the RF performance is presented.

INTRODUCTION

As bulk and thin film niobium (Nb) superconducting RF (SRF) cavities approach their theoretical limit, the requirement for alternative superconducting materials is increased. Thin film A15 superconductors 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_{c2}) of 28 and 24.5 T respectively [1]. These properties may allow for operation of SRF cavities at higher temperatures (≥4 K instead of ≈2 K), potentially reducing cost of cryogenics and may allow increased accelerating gradients, simplifying infrastructure of the particle accelerator [2].

Previous production of thin V_3Si films have implemented a variety of methods: thermal diffusion, co-sputtering and single alloy target magnetron sputtering, with varying levels of success. In the thermal diffusion method, Si is either deposited on a vanadium substrate or introduced via SiH_4 gas. The substrate is then annealed (850 °C) forming the correct A15 phase. This method resulted in a successful superconducting cavity, however it suffers from surface Si and performance lower than a typical Nb cavity [3]. Co-sputtered magnetron methods have produced successful V_3Si thin films on Cu, they also observed that the high deposition and annealing temperatures produces Cu-Si phases and Cu inclusions [4]. Further magnetron sputtered films on a stripline resonator formed on a thin sapphire substrates show an RF response inline with weak coupling BCS theory predictions with surface resistances significantly lower than Nb [5].

This paper focuses on the current progress of V_3Si thin films using pulsed DC magnetron sputtering deposition using a single target. The surface structure, composition and DC superconducting properties of two planar samples were characterised via secondary electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDX) and in a magnetic field penetration facility [6]. The latter section of this report presents the first deposition of a a V_3Si film on a split 6 GHz cavity with surface resistance measured in the RF characterisation facility [7, 8].

THIN FILM V_3Si ON PLANAR Cu SUBSTRATES

Sample Preparation and Deposition

Two V_3Si thin films were deposited on polycrystalline oxygen-free, high conductivity (OFHC) Cu substrates (Labelled S1 and S2). Both substrates underwent *ex situ* chemical treatment with BPS-172 solution, etching any surface oxide formation and atmospheric contamination. Once loaded into the vacuum system they were both heated to 500 °C for 24 hours to remove any residual contamination from the loading process.

V_3Si deposition was performed in a stainless steel vacuum chamber, with a base pressure 5×10^{-9} mbar, equipped with a single planar magnetron source. The source was equipped with a commercially bought V_3Si target. The sample stage is positioned 100 mm away from the magnetron source, with sample heating up to 800 °C. Deposition was conducted using a Pulsed DC magnetron power supply using the deposition parameters shown in Table 1. During deposition Kr is admitted into chamber as the process gas to a pressure of 5×10^{-3} mbar. Substrate temperature between S1 and S2 were 670 °C and 710 °C respectively.

Characterisation and Performance

Figure 1 displays two SEM images of the surface topography of V_3Si thin films on (a): S1 and (b): S2 samples. The surface topography of S1 is a dense structure of small grains that are a few hundred nanometers in size. S2 shows a similar structure, however, the grain size is much larger approaching micrometer in size. Further analysis using EDX showed a difference in composition between the two samples:

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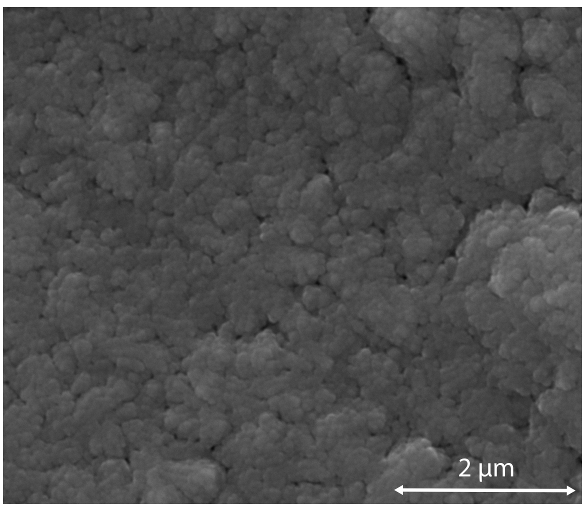
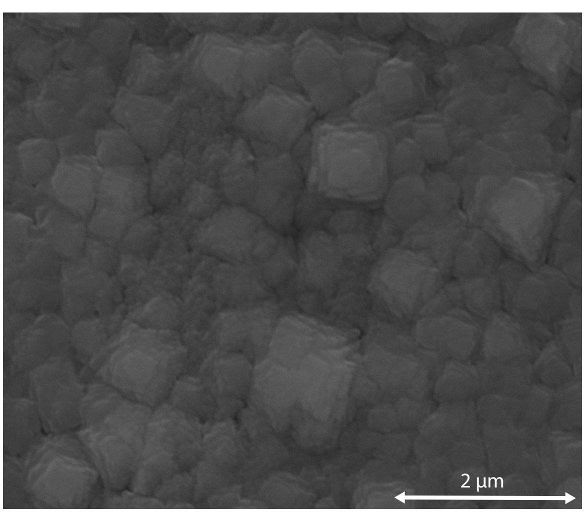
Table 1: DC Pulsed Magnetron Sputtering Deposition Parameters

Deposition Parameters	Units	
Voltage	[V]	566
Current	[A]	0.53
Power	[W]	300
Frequency	[kHz]	350
Pulse Length	[µs]	1.1
Deposition Length	[min]	180
S1 Substrate Temperature	[°C]	670
S2 Substrate Temperature	[°C]	710



(a) Surface topography of S1



(b) Surface topography of S2

Figure 1: Secondary electron microscopy images investigating the surface topography of the V_3Si thin films on planar copper substrates.

S1 had a measured V:Si percentage of 71 : 29%, whilst S2 a V:Si percentage of 76 : 24% was measured. The samples were deposited using the exact same pulsed DC magnetron deposition parameters and the same sputtering target therefore the difference must be associated with the increase in substrate temperature during deposition. Both samples are within the tolerance limit of the required stoichiometry of the A15 phase, although S1 is slightly Si rich [9].

Once grown the superconducting properties of the films were investigated using the magnetic field penetration (MFP) facility at Daresbury Laboratory. The DC magnetic penetration was measured at a series of fixed temperatures (4.2 - 16 K), where the full experimental procedure is described in detail by Turner *et al* [6]. Figure 2a shows the ratio (R) of the measured magnetic field as a function of magnetic field strength at 4.2 K for both samples. Three values are extracted as the first penetration (B_{fp}): the first derivatives, 99% and 98%. At 4.22 K, a significant difference is observed between S1 and S2, where samples sustained a Meissner state with a B_{fp}(98%) measured to be 258 mT and 189 mT for S1 and S2 respectively.

Figures 2b and 2c shows the magnetic field at B_{fp} as a function of temperature. This highlights the performance difference between the two depositions as S1 sustains a Meissner state at significantly higher applied fields, at comparable temperatures. However, in both cases the Meissner state is not observed and a B_{fp} could not be extracted when the sample temperature exceeded the 10 K suggesting similar T_c.

Discussion

Overall, a significant performance difference is observed between the two depositions with temperature being the only variable. SEM clearly shows a difference in grain sizes and uniformity between the two samples. Whilst EDX results suggests S1 to be Si rich (29%) and S2 to be about the correct composition with a Si % of 24%. At first glance this would suggest S2 will have better superconducting properties, however, in the magnetic field measurements we observe the exact opposite.

A possible explanation in the variation between depositions could be temperature dependent introduction of disorder in the structure in the film. Previous work using Co-sputtered magnetron methods observed that the high deposition and annealing temperatures produces Cu-Si phases and Cu inclusions [4], which would negatively impact performance. Further analysis is also required using X-ray diffraction allowing for in-depth analysis of the material phase to determine if non-superconducting phases are present in S1 or S2, impacting performance.

Previous V_3Si films measured in the MFP facility measured B_{fp}(98%) of 106 mT [10], showing a significant in deposition and film quality has been achieved. Also, Nb thin films measured at the facility recorded B_{fp} values up to 157 mT [6], suggesting the potential of V_3Si for SRF applications. However the samples produced were incompatible with RF measurement facilities.

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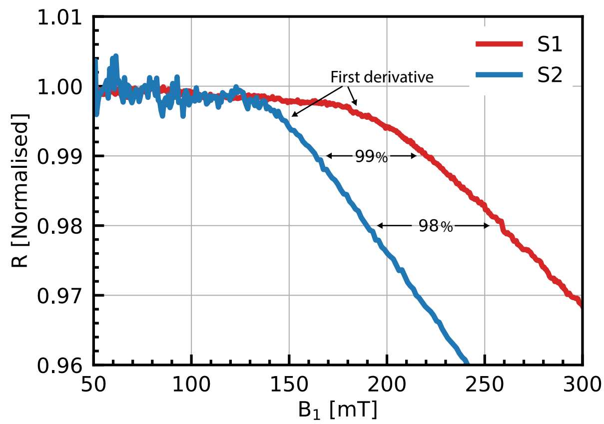
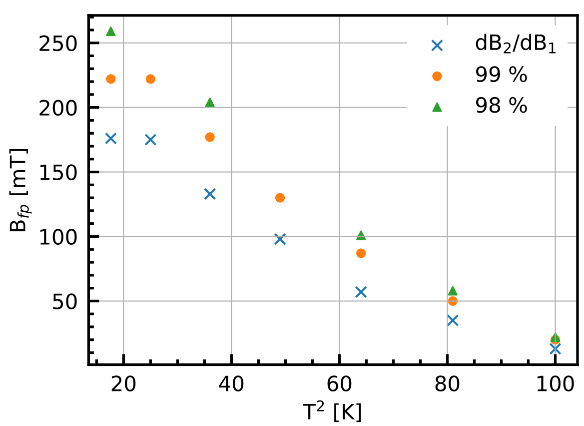
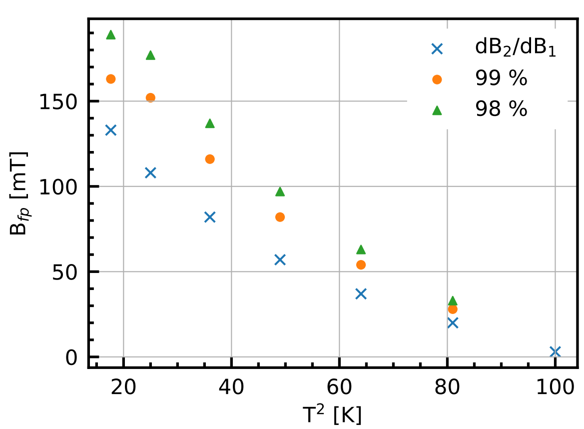


(a) $R(B_1)$ measurements with the MFP facility at 4.2 K. for S1 and S2. Labels indicate the extracted B_{fp} at first derivative, 99% and 99%.



(b) B_{fp} values for S1 at a series of fixed sample temperatures.



(c) B_{fp} values for S2 at a series of fixed sample temperatures.

Figure 2: Magnetic field penetration measurements for S1 and S2.

V_3Si SPLIT 6 GHz CAVITY

Deposition

V_3Si was deposited on a split 6 GHz cavity manufactured from OHFC at Daresbury Laboratory. The design allows for thin film deposition in an open geometry, coating the cavity in two halves using a planar magnetron. The full design and manufacturing process is described in references: [7, 8]. Initially the cavity was coated with a Nb thin film using pulsed DC power supply with a 300 W pulse power of frequency of 350 kHz and width of 1.1 µs. The cavity was mounted onto a infrared (IR) heating stage and heated to 400 °C.

After RF measurements, discussed in more detail in the next section, a V_3Si thin film (around 4 µm) was deposited. The photo shown in Fig. 3 shows the split cavity *in situ* mounted on the IR heated sample stage. The two cavity faces were deposited sequentially using the same vacuum system as described above, using the same parameters stated in Table 1. The sample was only heated to 530 °C due to the limitation of the IR heating stage.

Figure 3: Split 6 GHz cavity during deposition with each half of the cavity deposited sequentially.

Surface Resistance Measurements

The average surface resistance (R_s) measurements were performed in the facility described in references [7]. The surface resistance was calculated from the scattering (S) parameters of the cavity, measured using two antennas connected to a vector network analyser (VNA). The measurement was conducted at VNA low power (10 dBm) and at a temperature range of 4 K to 11 K.

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Figure 4 shows the measured R_s as a function of temperature for both the Nb and V_3Si cavity. At 4.2 K, R_s for Nb was measured to be 134 µΩ, whilst the V_3Si was measured to be around a factor 2 higher at 302 µΩ. At the higher temperatures, a superconducting transition is observed in the Nb coated cavity at approximately 9 K. In comparison, the critical temperature cannot be approximated as a transition is not observed in the V_3Si coated cavity. When the temperature exceeded 10 K, signal to noise hindered acquisition.

Discussion

Work conducted by Deambrosis *et al*, produced a 6 GHz V_3Si cavity following a thermal diffusion method. It was reported that initial RF tests were unsuccessful, however after in vacuo annealing at 850 °C the quality (Q) factor improved up to 1×10^6[11] after 48 hrs. This is inline with the Q factor reported on the split 6 GHz cavity (1.05×10^6 at 4.2 K) with no post processing applied.

The cavity underwent mechanical finishing but did not receive any form of polishing before being coated and tested. As expected the R_s measurements are dominated by the residual resistance of the cavity due to a non-ideal surface quality. However, an increase in the R_s of the V_3Si film relative to the Nb film suggests a non-ideal growth. This can be attributed to the deposition temperature potentially introducing non-superconducting V-Si phases and grain boundaries. Further complications may have arisen from the large diffusion coefficient of V into Nb at high temperatures leading to metallic inclusions further increasing R_s.

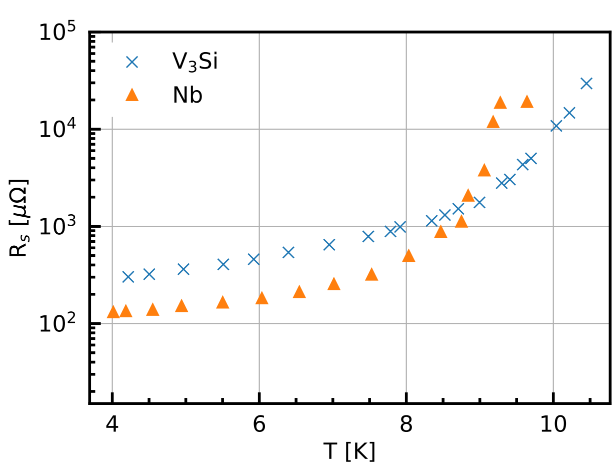

Figure 4: The surface resistance as a function of temperature for the Nb and V_3Si coated split cavity.

CONCLUSION

This report highlights the promising superconducting properties of V_3Si thin films for SRF applications. V_3Si thin films have been successfully grown on planar Cu substrates as well as a split 6 GHz cavity by means of single target DC pulsed magnetron sputtering. SEM shows variation in the surface topography of the planar samples due to different deposition temperatures, which in turn dramatically changed the DC superconducting properties. A first deposition of V_3Si on a 6 GHz cavity at Daresbury Laboratory has been achieved. The R_s was a factor of 2 higher than that of the Nb coated cavity, further improvements can be made through chemical polishing of the cavity and deposition temperature.

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