Superconducting Nanostructures in a Single Step Process

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Abstract. Focussed ion beam-induced chemical vapor decomposition of W(CO)6 allows single-step fabrication of nanoscale superconducting structures without lithography. Nanobridges have room temperature resistivity of around 200 μΩ cm and a Tc above 5 K. We present a study of superconductivity of this amorphous material depending on the deposition conditions and dimensions down to 50 nm across, in correlation with its normal-state properties. We also demonstrate more sophisticated structures, such as SQUIDs. We have fabricated superconducting devices operating at liquid helium temperatures on substrates and surfaces unaccessible to conventional technologies involving photo- or electron beam resists, for example around the tip of an AFM cantilever with potential applications in high-resolution magnetometry.

1. Introduction
It has recently been discovered that ion beam induced chemical vapour deposition (IBICVD) of tungsten hexacarbonyl W(CO)6 using Ga+ ions produces a material superconducting below about 5 K [1]. While bulk tungsten has one of the lowest Tc’s among pure metals ~10 mK and thin film tungsten becomes superconducting at 100-150 mK, many tungsten alloys have a Tc of order of 5 K. It was suggested that superconductivity in W(CO)6 is promoted by disorder in the material near metal-insulator transition [2]. Comparison of ion- and electron-beam deposited (EBICVD) W(CO)6 [3] revealed similar degree of structural disorder, but the latter shows an increase of resistivity at low temperatures, consistent with variable range hopping. Chemical composition of IBICVD material was reported to be typically 40, 40 and 20 at.% for W, C and Ga, respectively, Ga being incorporated from the incident ion beam. In contrast, the electron beam deposited wires had a typical composition of 16
at.%, 63 at.% and 21 at.% W, C and O, respectively. Obviously, gallium plays crucial role in superconducting properties.

2. Fabrication
We use FEI Nova Nanolab 600 dual-beam focused ion beam system for fabrication of superconducting structures. W(CO)$_6$ precursor is heated to 52 °C in vacuum. Standard FEI GIS needle is inserted to less than 100 μm from the sample surface. The flow of the precursor gas is controlled by pressure change in the chamber by about one order of magnitude on opening the needle, i.e. from $3 \times 10^{-6}$ to $3 \times 10^{-5}$ mbar. Four ion beam currents have been studied covering one order of magnitude, from 50 pA to 500 pA. This range of beam currents can be used to produce the finest structures of ~50 nm up to deposits of several μm in width and length in a matter of minutes. It has been found that the shortest beam dwell times available (200 ns), beam overlaps of about 70% and a small defocusing of the beam result in deposition rates that are highly controllable, and thicknesses of deposit can be controlled very accurately by changing the number of passes of the beam over a given area defined by the pattern. In short ‘the trick’ is to limit the current density by defocusing the beam spot during deposition, but overlap the effective spot size to give complete coverage, then use higher beam currents when larger deposits are required. Parameters used are shown in table 1, and results from the deposition tests are shown in figure 1.

| Beam Current | Beam Dwell | Defocus | Pitch | Beam overlap | Deposition rate |
|--------------|------------|---------|-------|--------------|----------------|
| 50 pA        | 200 ns     | 122.39 μm | 63.07 nm | 70%          | 0.4 pm per pass |
| 100 pA       | 200 ns     | 86.58 μm  | 63.34 nm | 70%          | 0.8 pm per pass |
| 300 pA       | 200 ns     | 50 μm     | 64.23 nm | 70%          | 1.9 pm per pass |
| 500 pA       | 200 ns     | 38.72 μm  | 64.91 nm | 70%          | 2.4 pm per pass |

Figure 1. Deposit height as a function of the number of beam passes under the conditions summarised in table 1.
3. Results
Best results were obtained 300 pA of beam current. Structures were deposited in four-terminal configuration onto a silicon substrate pre-patterned with palladium electrodes and contact pads.

Figure 2. Dependence of the superconducting transition temperature on the film thickness.

Figure 3. Critical current of a microbridge as a function of temperature.
We have produced microbridges in a variety of dimensions and studied the dependence of the superconducting transition temperature (Tc) on the geometry. Summary of results is presented in figure 2. Tc saturates at about 5.3 K when the film gets thicker than about 600 nm. It drops by about a Kelvin for 100 nm thick films. This behaviour is consistent with the model in which the Coulomb interaction between the electrons is enhanced by weak localization effects resulting in depression of Tc [4].

The width of superconducting transition is at best about 0.3 K reflecting some degree of non-uniformity of the film. Significant deviation from the optimum deposition parameters leads to even wider or multi-step transitions.

Critical current of the superconducting microbridges increases almost linearly below Tc – the behaviour typical of narrow bridges [5].

4. Discussion
The ability to direct-write superconducting structures on arbitrary substrates in 3D opens new opportunities for superconducting technology. For example, nano-SQUIDs [6] fabricated near the end of a cantilever will combine the spatial resolution of AFM and magnetic sensitivity of a SQUID. A prototype device in figure 4 shows such functionalized nanoprobe.

Figure 4. A nano-SQUID fabricated at the edge of an AFM cantilever with a TEOS tip in the middle of the loop.

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