Supplementary Material (ESI) for Chemical Communications

Supporting Information

Ultrathin Epitaxial Superconducting Niobium Nitride Films Grown by a Chemical Solution Technique

Guifu Zou*, Menka Jain, Honghui Zhou, Hongmei Luo, Scott A. Baily, Leonardo Civale, Eve Bauer, T Mark McCleskey, Anthony K. Burrell* and *Quanxi Jia*

Materials Physics and Applications Division, Los Alamos National Laboratory, Los Alamos, NM 87545

Experimental

Film preparation: The precursor used for the growth of NbN films was prepared by an aqueous solution of Nb ion bound to polymer. High purity (>99%) $NbCl_5$, NH_4OH, and 20% HF were dissolved in water where the water was purified using the Milli-Q water treatment system. Polyethyleneimine (PEI) was purchased from BASF Corporation of Clifton, NJ without further purification. Ultrafiltration was carried out under 60 psi nitrogen pressure using Amicon stirred cells which has a 3000 molecular weight cut-off. Metal analysis was conducted with a Varian Liberty 220 inductively coupled plasma-atomic emission spectrometer (ICP-AES), following the standard SW846 EPA (Environmental Protection Agency) method 6010 procedure.

In details, 2 g of $NbCl_5$ were converted to $Nb(OH)_5$ by addition of ammonium hydroxide into the solution according to literature procedures.[11] The $Nb(OH)_5$ was then dissolved in 30 mL of deionized water and 7.5 mL of 20% HF. PEI was then

added in 31 g aliquots (total 3.0g) and mixed after each addition. After stirring, the solution was placed in an Amicon filtration unit containing a filter designed to pass materials with molecular weight < 30,000 g/mol. The solution was diluted 3 times to 200 mL and then concentrated to 35 mL in volume. Inductively coupled plasma-atomic emission spectroscopy showed that the final solution was 400 mM Nb. The solution thus prepared was spun coated onto (001) $SrTiO_3$ substrate at 3000 rpm for 20 s. The film was annealed at 550 ºC for 1h in forming gas (94% Ar + 6% H_2) and then 900 ºC for 5 h in ammonium in a single sequence.

Film Characterizations: X-ray diffraction (XRD) was used to characterize the structure of the films. The surface morphology of the film was analyzed by scanning electron microscopy (SEM) and atom force microscopy (AFM). The microstructure of the films was examined by high-resolution transmission electron microscopy (HRTEM). The superconducting properties of the films were measured by superconducting quantum interference device (SQUID) and a physical property measurement system (PPMS).

References

11. D. Bayot, B. Tinant and M. Devillers, *Catal. Today*, 2003, **78**, 439.