Niobium Nitride Film Growth by Plasma CVD

Alexei Yu. Ganine and Grigori V. Vajenine
Max-Planck-Institut für Festkörperforschung
Heisenbergstr. 1, D-70569 Stuttgart, Germany

Niobium nitride films are of great interest not only due to their superconducting properties (1), but also owing to their extraordinary mechanical and chemical stability (2). NbN films feature relatively high critical temperature (T_c up to 17K), critical fields, and critical current densities (3).

The NbN films have been mostly grown employing physical vapor deposition (PVD) techniques, including pulsed laser deposition (4), molecular beam epitaxy (5), and reactive sputtering (6,7). On the other hand, one should pay more attention to NbN thin film growth by chemical vapor deposition (CVD), a method distinguished from the PVD by rather simple apparatus, high flexibility of the deposition process, better step coverage, higher growth rate etc. NbN films tend to grow at high temperatures (800-1000°C), which appears to be a disadvantage of the thermally activated, conventional CVD (8). Nevertheless, creating a plasma discharge in the reactor can significantly ease the deposition (9). Due to the fact that nitrogen plasma contains partially ionized and dissociated molecules the processing temperature, in many cases, can be kept lower. The choice of precursor is also important: while organometallic precursors such as Nb dialkylamides and alkylimides have been shown to be suitable for deposition of NbN by thermal and plasma CVD at low temperatures, contamination by carbon and especially hydrogen appear to be unavoidable (9, 10). In this work radio frequency (RF) nitrogen plasma induced CVD was applied to grow superconducting NbN thin films using $NbCl_5$ as a precursor.

NbN was deposited onto stainless steel substrates using a gas mixture of N_2 and $NbCl_5$. Syntheses occurred in a quartz vessel between two parallel plates, coupled with an RF-generator. The upper of the electrodes was connected to the 13.56 MHz RF-source through a matchbox, while the lower electrode was grounded. The deposition of NbN took place on the lower electrode at ca. 400^0C, while the pressure was held at 5 Torr, the upper electrode was water-cooled. Nitrogen carrier gas was mixed with $NbCl_5$ evaporated at 65^0C and delivered into the gap between the two electrodes. The deposition time varied depending on the evaporation rate of the $NbCl_5$ precursor, this could be also followed by the color change of the plasma from light green to yellow-orange as the precursor was exhausted. Quantitative analysis of the coatings was performed using an SEM equipped with an EDX setup. The crystal structure of the coating was investigated using X-ray diffraction. The magnetic properties of the deposited material were measured on a SQUID magnetometer.

Niobium nitride coatings were mirror-like with a typical bronze color. The chemical composition of the samples agreed with the formula NbN. Increasing of the $NbCl_5$ precursor content in the feed gas did not appear to influence the nitrogen contents in the films. Contamination by oxygen and carbon was not observed, with carbon and hydrogen impurities being excluded by the choice of the reaction system. The collected XRD patterns indicated a cubic NaCl-type crystal structure of NbN. The samples were measured to be superconducting with transition temperatures up to 12 K. Thus it was shown that the application of the RF nitrogen plasma allows for deposition of superconduncting films of cubic NbN free from H-, C-, and O-impurities from $NbCl_5$ as a precursor.

This work was supported by the Sofja Kovalevskaja Research Award of the Alexander von Humboldt Foundation, the Federal Ministry of Education and Research, and Programme for Investment in the Future (ZIP) of the German Government. We also thank Prof. Arndt Simon for support, Viola Duppel for EDX measurements, and Eva Brücher for magnetic properties measurements.

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