Development of niobium electroplating for a future radioactive $^{93}\text{Nb}^{m}$ target

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In preparation for a future experiment using a $^{93}\text{Nb}^{m}$ target ($t_{1/2}$ = 16.12 y), the heavy elements group has conducted experiments using stable Nb. The molecular plating technique was chosen for niobium electrodeposition. It is well known that organic solvents have larger electrochemical window (over which the electrolyte is stable) than aqueous solutions and isopropanol is the most popular organic medium for target preparation. In our experiments, an ICP-MS standard niobium solution (1 mg/mL) was used as a stock solution. A 1.3 mL aliquot of the stock solution was evaporated down to ~50 μL, diluted in 10 mL of isopropanol and transferred to the target cell. A high voltage supply capable of providing up to 1250 V and 20 mA was connected to the target cell and operated in constant voltage mode. The high voltage unit was upgraded to be controlled via PC. A GPIB interface was installed and LabVIEW software was created to monitor and record voltage/current data. Also, the new software allows for a constant current mode, although this was not utilized. In order to avoid solution overheating, a sine pulse mode was utilized. Niobium electrodeposition on aluminum foil (cathode) was carried out and the results obtained are shown in Fig. 1. The maximum current was set to 14 mA. Periodically (every hour) 0.3 – 0.5 mL of isopropanol was added to the cell to compensate for alcohol evaporation and to keep the solution volume at the same level. Approximately 5 min after that, a 100 μL aliquot was taken from the target cell solution and 100 μL of pure isopropanol was added to the cell. The niobium concentration in the isopropanol solution was determined by neutron activation analysis. The results obtained indicate that

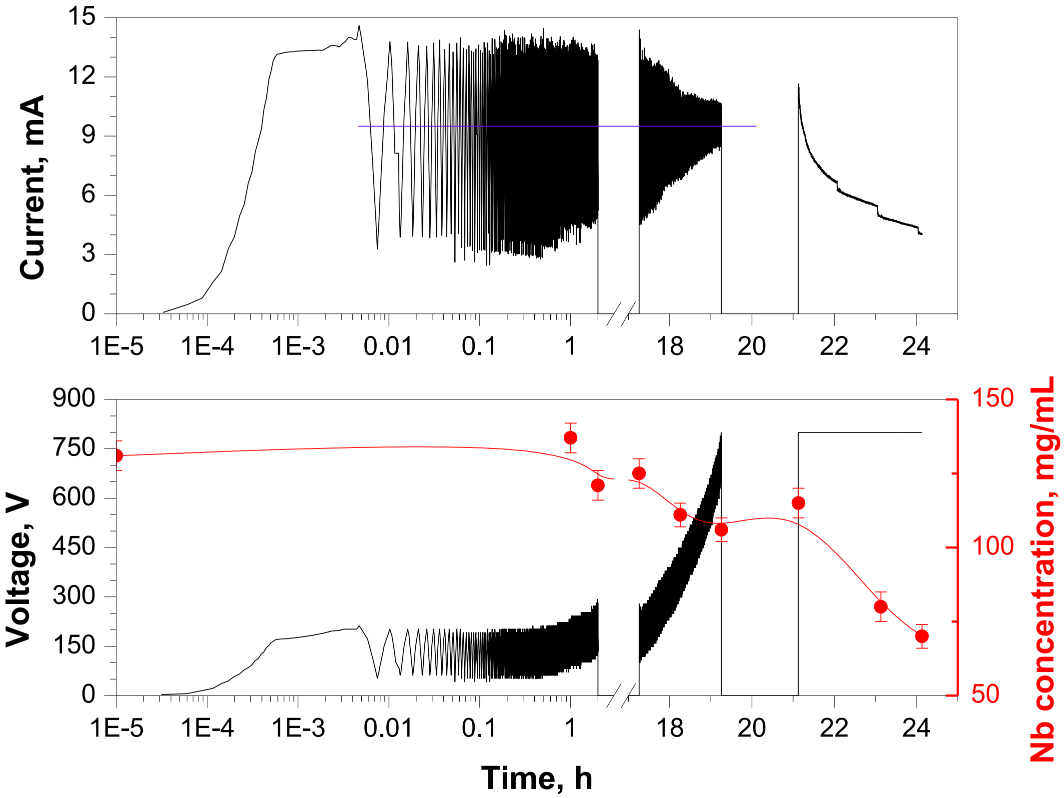

FIG. 1. Niobium electroplating from isopropanol solution.

there is no Nb deposition within the first hour of electroplating. During the second hour, the voltage was automatically increased a little bit due to the metal deposition. After 2 h, the high voltage was turned off and the isopropanol solution with niobium was left in the target cell overnight. The next day, a new aliquot was taken from the target cell before the high voltage was applied, to see if there was any dissolution of deposited material. As can be seen in Fig. 1, there is no significant loss of Nb. After that, the voltage was increased up to 800 V within the next 2 hours and the Nb concentration in the isopropanol decreased by 25%. A new 2 h pause was taken, and then the deposition mode was changed from sine pulse to constant voltage, because the system would have reached 1000 V within several minutes and we decided not to exceed this limit. Three more hours of electroplating resulted in almost 50% overall niobium deposition. Despite the presence of trace amounts of hydrofluoric acid in the ICP-MS standard (which leads to formation of a very stable oxyfluoro $NbOF_3$ complex and might be an obstacle for niobium electroplating), EDS and XPS analyses of the deposited layer (performed at the TAMU Materials Characterization Facility) indicate that niobium was electrodeposited mostly in the form of niobium oxide Nb_2O_3. The next step of this procedure development will be optimization of electrodeposition conditions.