Electronic Supplementary Information (ESI) for:

The effect of ammonolysis conditions on the structural properties and the oxidation kinetics of cubic niobium oxynitride

Vanessa C. D. Graça[1,2*], Laura I.V. Holz[1,2], Francisco J. A. Loureiro[1,2], Glenn C. Mather[3], Duncan P. Fagg[1,2,**]

[1] TEMA - Centre for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro, 3810-193 Aveiro, Portugal.

[2] LASI - Intelligent Systems Associate Laboratory, Portugal.

[3] CSIC - Instituto de Ceramica y Vidrio (ICV), Madrid, Spain

Determination of N and O content

The N content was otained from an average of three samples produced at each condition. The nitrogen content was calculated by elemental analysis using standard combustion analysis (Truspec 630-200-200) detected by use of a thermal conductivity detector. The oxygen content was determined by thermogravimetry/differential thermal analysis (TGA/DSC; Model STA449/6/G, Netzsch, Jupiter, Germany), by fully oxidizing samples in pure oxygen (O_2) at 1000 °C, to Nb_2O_5, which was confirmed by performing a subsequent XRD analysis on the resultant powder. Figure S1 shows an example of the obtained TGA profile.

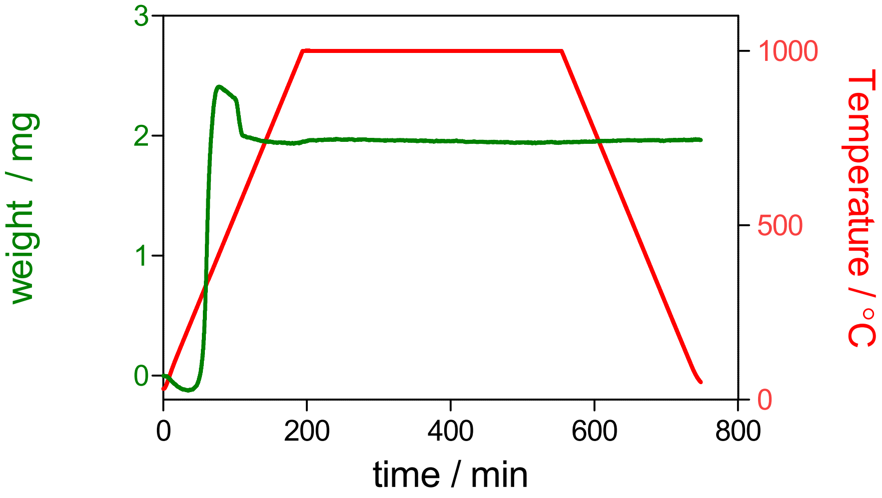

Fig. S1 – TGA profile of the $NbN_{1.02}O_{0.66}$ sample synthetized at 800 °C for 1 h.

$$NbN_xO_y(s) + \left(\frac{2.5-y}{2}\right)O_2(g) \rightarrow \frac{1}{2}Nb_2O_5 + \left(\frac{x}{2}\right)N_2(g) \quad \text{(S1)}$$

The number of mols of NbN_xO_y of the sample measured by elemental analysis ($n_{NbN_xO_y, e.a.}$) was determined by the following expression:

$$n_{NbN_xO_y,\,e.a.} = \left(n_{Nb_2O_5} \times 2\right)\left(\frac{m_{inicial,e.a.}}{m_{inicial,TGA}}\right), \quad \text{where} \quad n_{Nb_2O_5} = \frac{m_{final,TGA}}{M_{Nb_2O_5}} \tag{S2}$$

where $m_{inicial,e.a.}$ is the initial mass of NbN_xOy used in the elemental analysis, $m_{inicial,TGA}$ is the initial mass of NbN_xO_y used in TGA analysis, $m_{final,TGA}$ is the final mass of TGA analysis and M_{Nb2O5} is the molar mass of Nb_2O_5.

The molar mass of NbN_xO_y (M_{NbNxOy}) was therefore calculated according to the subsequent expression:

$$M_{NbNxOy} = \frac{m_{initial,e.a.}}{n_{NbN_xO_y,e.a.}} \tag{S3}$$

The nitrogen and oxygen contents, x and y, respectively, were then calculated as follows:

$$14x = \frac{\left[weight\,\%\,N \times \left(\frac{m_{initial,e.a.}}{n_{NbN_xO_y,e.a.}}\right)\right]}{100} \tag{S4}$$

$$y = \frac{\left(M_{NbNxOy} - M_{Nb} - 14x\right)}{16} \tag{S5}$$

where M_{Nb} is the molar mass of Nb.