DIFFUSION OF OXYGEN IN NIOBIUM DURING BAKE-OUT

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Abstract

Bake-outs at temperatures between 100 ºC and 150 ºC for duration up to two days have become customary for optimising the performance of bulk niobium cavities. This treatment results in the diffusion of oxygen, originating from the surface oxide, into the niobium. The theoretical oxygen profile has been simulated using the diffusion equations, and compared with some experimental results.

1 THE DIFFUSION EQUATION

The natural niobium oxide presents a layered structure [1], comprising Nb_2O_5 as the outermost layer and reducing over a few nm to NbO_2 and NbO as the innermost layer. This oxide is a potential source of oxygen during bake-out, although the dissociation of NbO and NbO_2 to release O in solid solution is not energetically favourable, as illustrated in Fig. 1. However, some oxygen is always present in solid solution at the NbO-Nb boundary. The maximum oxygen concentration at equilibrium for any given temperature is called solubility limit (C_0).
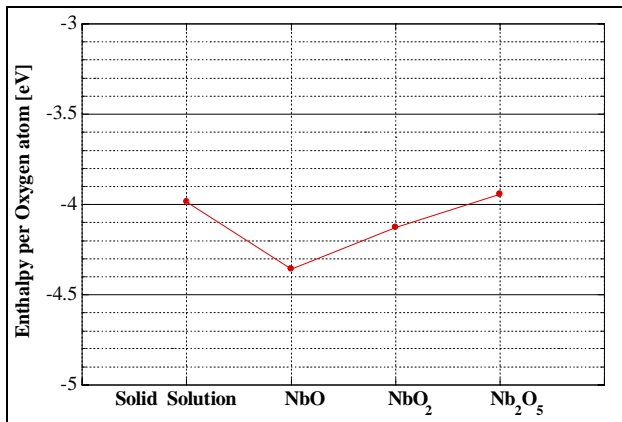

Figure 1: Enthalpies of formation for different Nb-O compounds.

It is assumed that the surface oxide reduces upon heating, becoming an inexhaustible source of oxygen which can diffuse into the bulk, and that the oxygen concentration at the NbO-Nb boundary attains C_0. The oxygen diffusion during bake-out is then described mathematically as the diffusion in a semi-infinite medium ($x>0$), where the boundary is kept at a constant oxygen concentration C_0, the initial concentration being zero throughout the medium. The oxygen concentration as a

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function of the position x and of time t may be obtained form the diffusion (Fick) equation:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2}$$

which satisfies the boundary condition {$C=C_0$, $x=0$, $t=0$} and the initial condition {$C=0$, $x>0$, $t=0$}.

The boundary condition $C=C_0$ corresponds to imposing that the concentration at $x=0$ (the oxide/bulk interface) reaches the solubility limit. This value is known from literature [2] only for $T>600$ ºC, and an exponential extrapolation to the low temperatures of interest here has too large an uncertainty for being applicable. The diffusion equation must thus be solved for the relative quantity C/C_0. The diffusion coefficient D is known even at low temperatures [2], in particular in the range 100ºC÷150ºC it results D=1.38x10^{-2}exp(-111530/RT).

The required solution is:

$$\frac{C}{C_0} = \text{erfc} \frac{x}{2\sqrt{Dt}}$$

2 CALCULATED RESULTS

The diffusion profiles as a function of the depth have been calculated in the relative units C/C_0, for different bake-out duration at 145 ºC. The result is illustrated in Fig. 2. It must be noted that the concentration profiles will not change upon cool down, in spite of the decrease of the solubility limit. The time required for re-diffusion of the oxygen and formation of the oxide phase is in fact much longer that the typical cool down time and thus the oxygen is frozen out of equilibrium in a metastable state.

The calculated diffusion profiles have been verified experimentally by making use of the data illustrated in Fig. 3. In this experiment, the BCS surface resistance of a cavity has been measured before and after a 145 ºC / 45
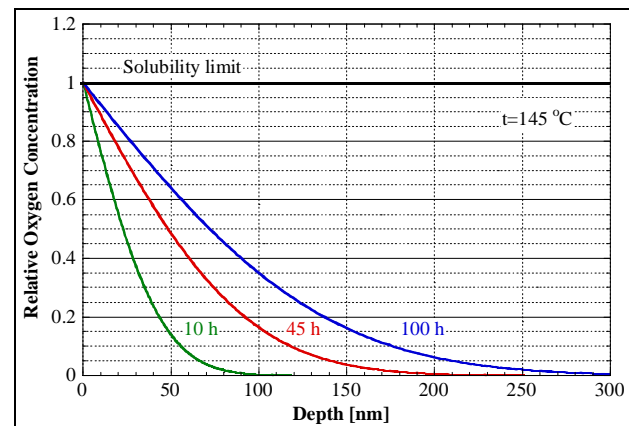

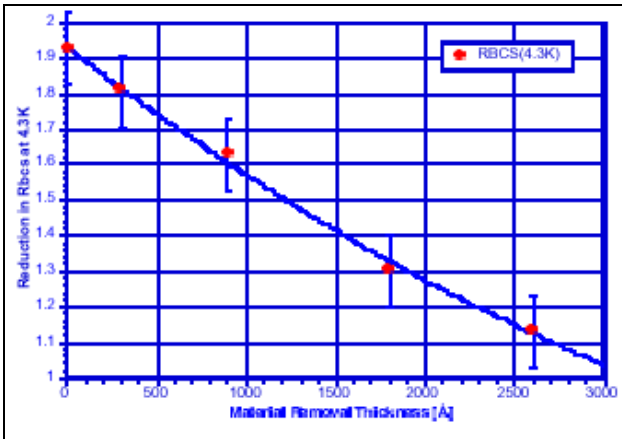Figure 2: Oxygen diffusion profiles after heating at 145 ºC for different periods of time.

Figure 3: Reduction in R_{BCS} after bake-out (from a pre-bake-out value of 970 nΩ), and after progressive surface removals [3].
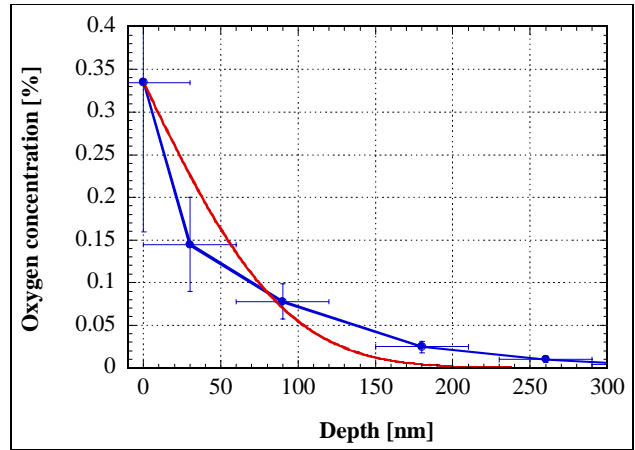

Figure 4: Comparison of the diffusion profile extrapolated from the data of Fig. 3 (dots) with the calculated one, under the assumption that C_0 at 145 ºC is 0.33%. The horizontal error bars are equivalent to one RF penetration depth, to account for the variation of the concentration over this scale.

hours bake-out, and then after progressively removing small surface layers until recovering the original R_{BCS} value [3].

From these data, the oxygen content that would give rise to the values of R_{BCS} measured at the different depths has been extrapolated, albeit without correcting for the change in concentration within the penetration of the RF field. The result of the extrapolation is reported in Fig. 4, together with the concentration profile calculated with the diffusion equation. There is a good agreement between the two profiles, when the solubility limit C_0 at the chosen temperature is made to correspond to the experimental oxygen concentration of about 0.33% for x=0.

3 CONCLUSIONS

The diffusion equation for the problem of oxygen diffusion at low temperature inside niobium has been solved. A good agreement with the available diffusion profile, obtained by measuring the BCS surface resistance at various depths, has been reached. The solubility limit C_0 at 145 ºC has been extrapolated, and is equal to 0.33%.

REFERENCES

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