Vacancy-Hydrogen Dynamics in Samples during Low Temperature Baking

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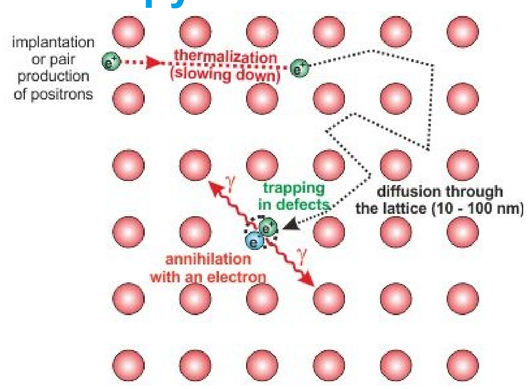
Baking and SRF Performance

- The influence of hydrogen on rf losses ('hydrogen Q-disease') of cavities and the need of outgassing cavities is known for quite some time.
- The operating temperature of superconducting accelerating cavities is 2-4 K, and while crossing the temperature range of 200-50 K during cool down, different phases of niobium hydride on the rf surface are forming, causing the increased losses.
- To prevent this, cavities are baked at 700 − 900°C at pressures below 10^{-6} mbar to purify the material
- A 120°C bake for 48h after the final electropolishing has shown to reduce the losses and cure the high field Q-slope, while both effects are not fully understood yet.

Working Hypothesis

- Lattice deformations, interstitials and vacancies are known to have high trapping potential for interstitials, especially hydrogen.
- Formation of so-called "nanohydrides" which are only weakly superconducting by proximity effect up to a certain threshold of applied field is assumed to cause losses above the threshold causing the high field Q-slope[1] .
- The assumption is that the modified low T baking procedure [2] might influence the vacancy-density and their interaction with hydrogen in the relevant rf penetrated layer in a beneficial way to prevent formation of lossy nanohydrides.
- The new bake includes a 75°C step before the 120°C. At this temperature a NbH β→α' phase formation takes place [3,4] which potentially influences Nb-H dynamics during cooldown.
- So called vacancy-hydrogen (v+nH) complexes have been studied and found to play a role already in the standard 120°C bake [5].

Positron Annihilation (Lifetime) Spectroscopy



- Positrons are easily trapped in vacancies and are very sensitive to their chemical environment
  - When the energy of the annihilation photons is obtained, an energy shift ΔE can be observed (CDB shift). This energy shift depends on the chemical surroundings of the annihilation site and also on the density and types of defects.

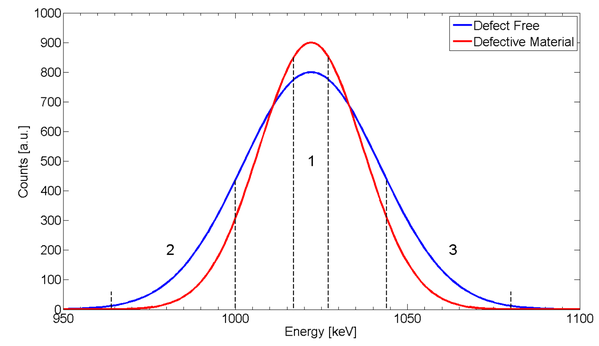


Figure 1: Annihilation spectrum of positrons in metallic material. The central area A_1 is used to quantify the S-parameter, while the areas A_2 and A_3 are used for the W-parameter.

- At pulsed sources, the lifetime of positrons in the material can be measured and the density and types of defects will impact the result.

Sample Preparation

- The sample preparation followed closely the standard cavity preparation.
- Sample chemistry and high pressure rinsing holders have been developed to use the standard infrastructure for cavity treatment at DESY.
- The final preparation step, the low temperature bake, was then studied with in-situ and ex-situ approaches using different positron spectroscopy set-ups. An overview of the samples and the measurements is given in table 1.

Table : List of samples used, including material and temperatures applied.

Sample	Material	Method	Facility	Treatment
6	Ningxia	PALS, DB-PAS	Prague	70^o C for 4h, 120^o C for 4 h, 120^o C for 40h in p $\leq 10^{-3}$ mbar
73	Tokyo Denkai	PALS, DB-PAS	Prague	70^o C for 4h, 120^o C for 4 h, 120^o C for 40h in p $\leq 10^{-3}$ mbar
14	Ningxia	DB-PAS	AIDA	DESY sample furnace at 70^o C for 4 h in p $\approx 10^{-6}$ mbar
17	Ningxia	DB-PAS	AIDA	Steps from 70^o C to 350^o C for 4 h each in p $\approx 10^{-10}$ mbar
64	Tokyo Denkai	DB-PAS	AIDA	Steps from 70^o C to 350^o C for 4 h each in p $\approx 10^{-10}$ mbar
78	Tokyo Denkai	PALS	MePS	Steps from 70^o C to 250^o C for 4 h each in p $\leq 10^{-7}$ mbar

Vacancy Evolution as a Function of the Temperature

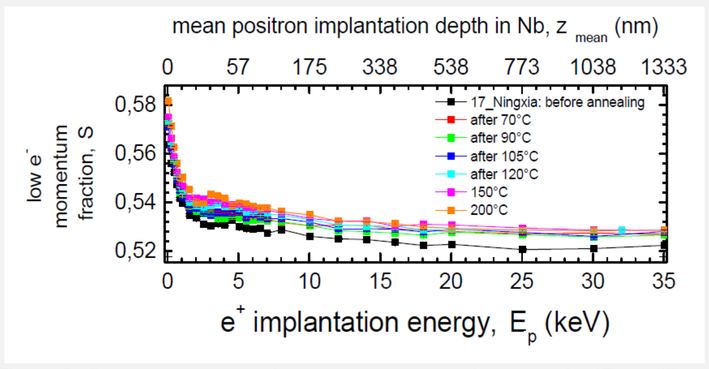


Figure 2: S-parameter vs. positron energy measured on sample 17 at different temperatures for in-situ annealing. The vacancy density increases with temperature while baking for 4h up to 200°C.

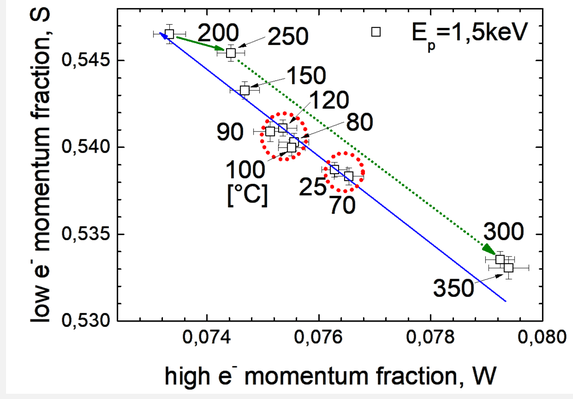


Figure 3: S-W plane at a fixed energy of 1.5 keV of sample 64. All data points up to 200°C are on the same line, hence the defect type does not change, only the density increases. At 250°C the defect type starts to change and the defect density decreases.

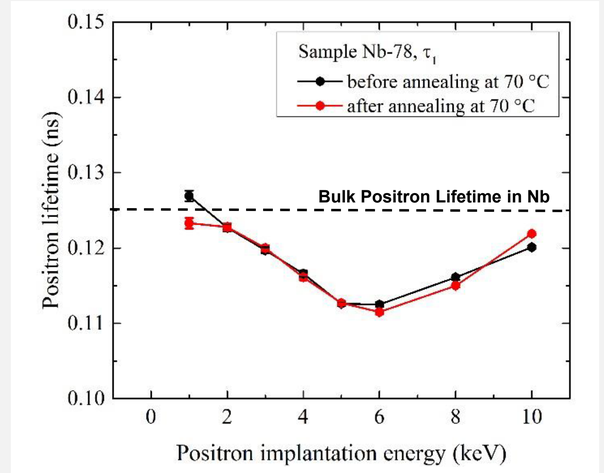


Figure 4: τ_1 lifetime component vs. positron energy measured in-situ for the sample 78 before and after annealing at 70°C. Large v+nH cluster with large n contribute to the near surface decay reducing the lifetime.

Vacancy Concentration and Formation of v+nH complexes during baking

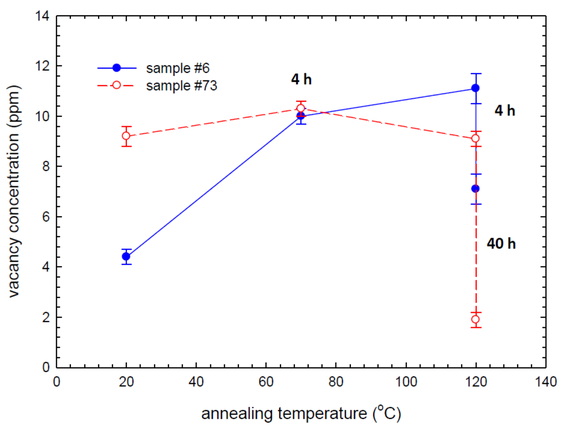


Figure 4: Vacancy Concentration calculated from PALS data using two state positron trapping model vs. annealing temperature at a fixed energy for samples 6 and 73. The annealing was done ex-situ.

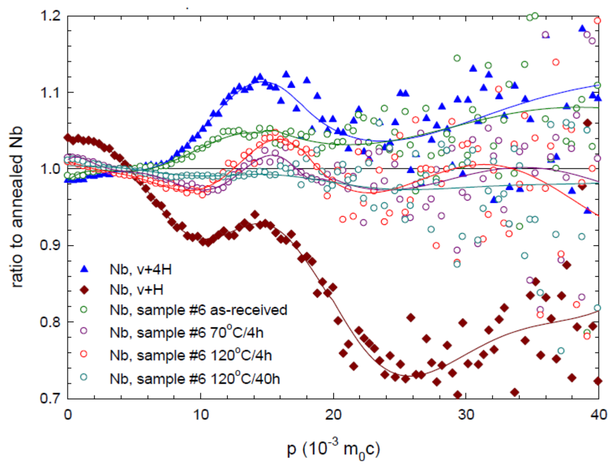


Figure 5: CDB ratio related to pure Nb (annealed at 1000°C) vs. momentum of the annihilating pair for sample 6 (in bulk). Two reference curves for Nb with v+H and v+4H complexes are shown for comparison. While for as-received, some hydrogen is associated to vacancies. v+nH complexes form at 70°C and are stable up to 120°C with short annealing time. After full annealing at 120°C no v+nH complexes are observed in the bulk.

Conclusions

- Virgin samples contain interstitial hydrogen and v+nH complexes and locally exceeding maximum solubility in bcc lattice, hydride precipitates are formed.
- Annealing at 70-80°C, nanohydrides are decomposed and. hydrogen atoms are gradually released and diffuse into the lattice and form v+nH complexes.
- Annealing at 120°C results in gradual disappearance of v+nH complexes in the bulk. H atoms are gradually released diffusing towards grain boundaries, the surface and vacancies start to form clusters.
- Near-surface lifetime measurements show strong contribution with large vacancy clusters paired with hydrogen even after annealing.
- At 250°C the oxide layers Nb_2O_5 and NbO_2 are dissolved into the bulk [6] and start to change the vacancy types.

References

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