TRAPPING OF STAGE III DEFECTS BY $^{111}$IN IN NIOBIUM$^{*)}$

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The interaction of defects in ultrapure Niobium metal with $^{111}$In probe nuclei was studied in an isochronous annealing program using the $\gamma-\gamma$ TDPAC technique. The formation of two unique probe atom-defect configurations was observed, one of which is tentatively ascribed to the trapping of a single vacancy at the $^{111}$In probe.

The bcc metals are of increasing technical importance specially in environments where a detailed knowledge of property changes due to lattice damage is crucial (e.g. fusion reactor walls etc.). However in contrast to the fcc metals the microscopic structure and thermal behaviour of lattice defects is far less well understood. Specially in Niobium there are open questions concerning the nature of the stage III defect annealing /1/. To a great extent these difficulties are caused by the tendency of the relatively open bcc Nb lattice to dissolve extensively small impurity atoms as H,C,N,O which can lead in resistivity recovery measurements to the suppression of intrinsic recovery stages and the appearance of impurity stages /2/. The TDPAC technique, which was used in the experiments described below, in contrast to the classical methods (e.g. resistivity recovery measurements) gives information about the microscopic structure of defects trapped by the probe atom and can thus lead to a better understanding of the defect nature.

Due to the difficulties mentioned above decarburized and carefully outgassed 30 µm Niobium foils were used in the experiments /3/. The concentration of metallic impurities and dissolved C+N+O was estimated to be less than 3 at ppm respectively, the hydrogen concentration approx. 50 at ppm /1,4/.

The foils were implanted at room temperature with the TDPAC probe nucleus $^{111}$In with an implantation energy of 80 keV at the Bonn isotope separator. The implanted dose was typically $4 \times 10^{13}$ at/cm$^2$ which led to an average concentration in the implanted region of approx. 600 at ppm In.

An isochronal annealing program (At=10 min.) was carried out in an atmosphere of purified argon.

For the angular correlation measurements of the $173$ keV - $247$ keV $^{111}$In $\gamma-\gamma$ cascade a 3 detector apparatus with fast-slow electronics was used. From the measured time spectra $R(t)$ asymmetry ratios equal to $A_{22}G_2(t)$ were formed. $A_{22}$ is the anisotropy of the observed $\gamma-\gamma$ cascade and $G_2(t)$ describes the perturbation of the angular correlation by interactions of the nuclear quadrupole moment of the 247 keV $^{111}$In $\gamma$ level and electric field gradients (EFG) present at the site of the probe nucleus. Since on a substitutional site in an undisturbed cubic lattice no EFG is present any perturbation of the angular correlation must be attributed to a local disturbance of the cubic symmetry, possibly caused by a trapped defect.

In Fig.1 the experimental results of our measurements after several annealing stages are shown. In all spectra strong modulations of the angular correlation can be observed. Qualitatively one can separate the precession pattern into three parts: Two fractions $f_1$ and $f_2$ of $^{111}$In nuclei which underly interac-
Figure 1:

Time differential anisotropy of the 173 keV - 247 keV γ-γ cascade of $^{111}$In in Nb after annealing at different temperatures. The solid line represents a fit to the experimental data as explained in the text.

The fitted expression for the anisotropy $A_{22}(t)$ is given by:

$$A_{22}(t) = f_1 \sum_{n=1}^{3} s_{2n} \cos(n \omega_1 t) \exp(-1/2n \omega_1^2 t) + f_2 \sum_{n=1}^{3} s_{2n} \cos(n \omega_2 t) \exp(-1/2n \omega_2^2 t) + f_3 \sum_{n=1}^{3} s_{2n} \exp(-1/2n \delta_3 t)$$

where $f_1 + f_2 + f_3 = 1$.

This was used to describe the data, where for $\omega_1$ and $\omega_2$ the theoretical function for $I=5/2$ was assumed. The exponential factors allow for different EFG's and a third fraction interacting with a broad distribution of very small EFG's causing the slow exponential drop in anisotropy.