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## Defect recovery in aluminum irradiated with protons at 20 K

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Aluminum single crystals have been irradiated with 7.0-MeV protons at 20 K. The irradiation damage and its recovery are studied with positron-lifetime spectroscopy between 20 and 500 K. Stage-I recovery is observed at 40 K. At 240 K, loss of freely migrating vacancies is observed. Hydrogen in vacancies is found to stabilize the vacancies and prolong stage III to above 280 K, where the hydrogen bound to vacancies is released. Single and multiple occupancy of hydrogen atoms at monovacancies is put forward as the reason for the two recovery stages between 280 and 400 K. A binding energy of  $0.53 \pm 0.03$  eV is found for a hydrogen atom trapped at a monovacancy. The results are in excellent agreement with recent ion-beam-analysis results and also with theoretical estimates.

### I. INTRODUCTION

In recent years the behavior of hydrogen in metals has received much attention,<sup>1</sup> mainly due to its technological significance. The interaction between hydrogen and defects has important implications on the mechanical behavior of metals; hydrogen concentrations of a few ppm may cause severe embrittlement and change of ductility of metals. Change of the transport properties of hydrogen by immobilizing it at defects instead of accumulation of hydrogen at potential fracture sites could probably reduce these technological problems.<sup>2</sup>

Many experimental techniques have been developed<sup>1,3</sup> to give a better understanding of hydrogen-defect interactions in metals. Gas release and permeation studies, ion beam analysis, and resistivity measurements are widely employed techniques for these investigations. An interplay with theoretical approaches<sup>2</sup> can provide a deeper insight into the microscopic nature of the trapping mechanism of hydrogen at defects.

It is particularly desirable to develop a method that is sensitive to hydrogen interactions on the atomic scale.<sup>4</sup> The positron-annihilation technique (PAT) has been developed into a defect spectroscopy method that is selectively sensitive to lattice defects such as vacancies, dislocations, and voids.<sup>5,6</sup> Positrons are especially sensitive to vacancy-type defects and essentially insensitive to interstitials. In the presence of vacancies the positron gets trapped<sup>7</sup> and the positron lifetime increases by about 50% compared to the perfect bulk state. The lifetime in a vacancy in Al is about 240 ps and about 160 ps in the perfect crystal. Vacancy clusters are recognized by positron lifetimes up to about 500 ps.<sup>7</sup> Decoration of vacancies<sup>8,9</sup> and voids<sup>10,11</sup> by hydrogen changes the electronic structure in the defect and thereby the positron lifetime or trapping rate<sup>8</sup> characteristic for the specific defect type.

Calculations by Hansen *et al.*<sup>8</sup> have shown that the positron lifetime in an Al vacancy decorated with one hydrogen atom is lowered by about 7 ps while vacancy decoration by six hydrogen atoms reduces the lifetime by about 60 ps. The ability of positrons to sense different defect sites and their decoration by hydrogen makes PAT a powerful method to study hydrogen-defect interactions. Recently such PAT studies have successfully demonstrated this ability.<sup>10-14</sup>

We report here on an approach to study hydrogen in aluminum utilizing the positron-annihilation technique. Hydrogen has been implanted into Al at 20 K followed by isochronal annealings up to 500 K. The evolution of the defects induced by the 7.0-MeV protons has been studied by positron lifetime spectroscopy.<sup>5</sup>

The migration of interstitial atoms at 40 K (stage I) is observed. The migration of monovacancies occurs around 240 K. Vacancies decorated by hydrogen are found to be stabilized up to around 295 K, where the hydrogen is released. All defects are annealed out at 500 K.

### II. EXPERIMENTAL TECHNIQUE

The 7.0-MeV proton irradiations of well-annealed and carefully electropolished 6N purity Al single crystals were performed with the MC-20 cyclotron at the University of Jyväskylä. The samples were mounted in a sample holder attached to the cold head of a two-stage closed-cycle helium refrigerator. The cryostat is constructed for irradiations at 15 K with subsequent positron lifetime measurements. Figure 1 shows the setup with the specimens mounted in the sample-source-sample geometry. The samples could not be manipulated after cooling and, hence, the samples were proton irradiated from their backsides, as shown in Fig. 2. The 7.0-MeV protons reached a mean depth of 334  $\mu\text{m}$  with a longitudinal

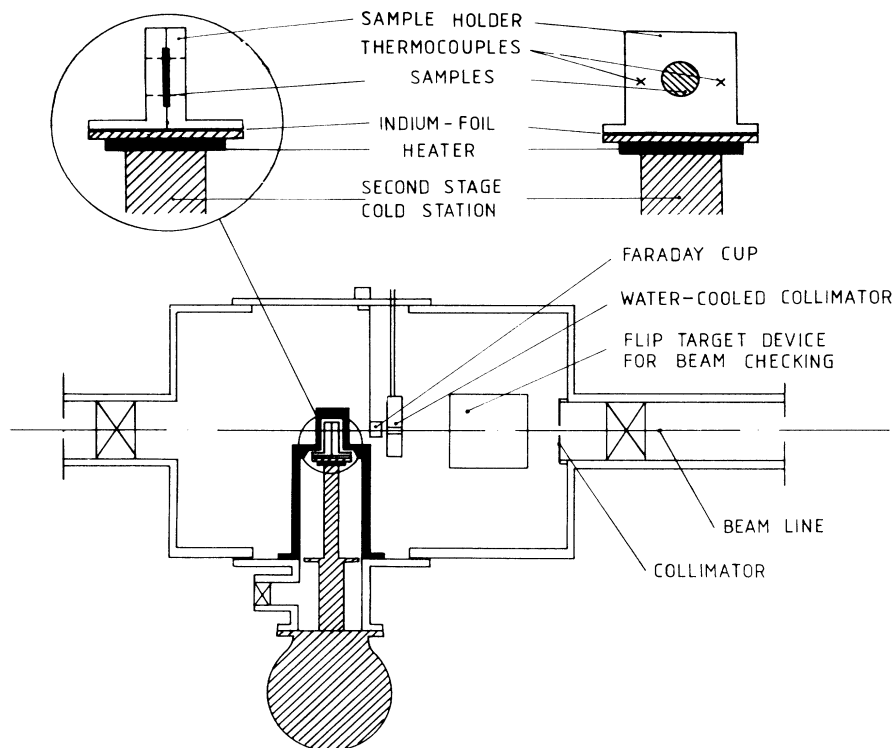


FIG. 1. The setup for the proton irradiation facility at the University of Jyväskylä. The sample chamber is separated from the beam line with a window of  $1.7\text{-mg/cm}^2$  Havar foil.

straggling of  $5.4\ \mu\text{m}$ .<sup>15</sup> Al degraders with thicknesses of  $10\ \mu\text{m}$  were utilized to achieve proton implantation depths in the range  $290\text{--}340\ \mu\text{m}$ . The mass absorption coefficient of the almost exponential implantation profile is  $45\ \text{cm}^2/\text{g}$  in Al (Ref. 16) for positrons originating from  $^{22}\text{Na}$ . The thickness of  $350\ \mu\text{m}$  of the Al specimens is sufficient to stop 99% of the positrons inside the samples. The energy of the injected protons and the thickness of the specimens were chosen to maximize the overlap of the proton and the positron implantation profiles, as sketched in Fig. 2. Approximately 60% of the positrons annihilated in the region with hydrogen, 20% in the region without hydrogen but with irradiation damage, and

20% in the defect-free region. A beam current of  $100\ \text{nA}$  ( $6.22 \times 10^{11}\ \text{H}^+/\text{s}$ ) provided a mean hydrogen density of  $0.0822\ \text{ppm/s}$  with the specimens continuously kept below  $20\ \text{K}$  during the irradiations. The total dose was  $100\ \text{ppm}$  in the hydrogen-doped region.

After the irradiations isochronal annealings were carried out with an annealing rate of  $25\ \text{K/h}$ . Up to an annealing temperature of  $320\ \text{K}$ , positron time spectra were acquired at  $15\ \text{K}$ . A standard fast-fast coincidence timing spectrometer<sup>17</sup> with a time resolution of  $325\ \text{ps}$  full width at half maximum (FWHM) was used for these measurements. A  $150\ \mu\text{Ci}\ ^{22}\text{NaCl}$  source deposited on a  $1.7\text{-mg/cm}^2$  Havar foil provided a count rate of  $30\ \text{cps}$ . Each spectrum contained about  $6 \times 10^5$  counts before a 15% source correction. The RESOLUTION and POSITRONFIT programs<sup>18</sup> were utilized for the analyses.

After annealing at  $320\ \text{K}$  the samples were transferred to a liquid-nitrogen cryostat. The sample set was packed at  $77\ \text{K}$  in the sample-source-sample geometry and transferred while continuously kept below  $80\ \text{K}$ . Isochronal annealings were continued up to  $500\ \text{K}$  with the measurements carried out at  $80\ \text{K}$ . A  $100\text{-}\mu\text{Ci}\ ^{22}\text{NaCl}$  source, evaporated onto a  $0.45\text{-mg/cm}$  Ni foil, yielded about  $60\ \text{cps}$  with the standard fast-slow time spectrometer<sup>5</sup> employed for these measurements. Two displaced Gaussians, as found by the RESOLUTION program,<sup>18</sup> were used to describe the time resolution function (FWHM =  $250\ \text{ps}$ ). Utilizing the results of Linderoth *et al.*<sup>16</sup> the source corrections were found. The time spectra, that contained about  $6 \times 10^5$  counts before about 10% source

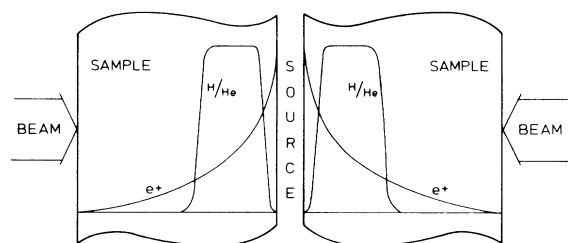


FIG. 2. Schematic drawing of the positron and proton implantation profiles in the Al samples. The positron source is situated between two identical specimens which are irradiated with protons from their backsides.

correction, were analyzed with the computer program POSITRONFIT.<sup>18</sup>

The time spectra were decomposed into two lifetime components. The longer positron lifetime was  $240 \pm 15$  ps in the whole annealing temperature range from 20 to 500 K. The inhomogeneous damage production caused the positron time spectrum to be sufficiently complex to make the statistical scattering, in the two-component fitting, unsatisfactorily large. This led us to employ the mean positron lifetime given by

$$\bar{\tau} = \sum_i I_i \tau_i, \quad (1)$$

where  $\tau_i$  and  $I_i$  are the lifetime and intensity, respectively, of the  $i$ th component. This procedure gave only small statistical uncertainties on  $\bar{\tau}$ .

### III. RESULTS AND DISCUSSION

#### A. Preirradiated and as-irradiated states

Prior to the proton irradiations, positron lifetime spectra were acquired at different temperatures of the Al specimens. No significant change was observed in the mean positron lifetime when lowering the temperature from 300 to 15 K, which indicates that the dislocation density was negligible in the well-annealed samples.<sup>19</sup>

Proton irradiations were performed to give a mean concentration of 100 ppm of hydrogen in the region sketched in Figure 2. Measurements were performed after accumulated hydrogen concentrations of 1 ppm and 10 ppm in order to follow the effect of the irradiation on the mean positron lifetime. A mean lifetime of 205 ps was obtained after an accumulated irradiation dose of 100 ppm in the hydrogen-containing region. In a two-component fitting the longer positron lifetime was found to be about 240 ps, which is similar to the lifetime in a monovacancy. This indicates that only Frenkel pairs were created during the irradiation.

#### B. Annealing below 200 K

The annealing curve is shown in Fig. 3. Around 40 K a rapid decrease of the mean positron lifetime is observed. Recovery in electron-, neutron-, and alpha-particle-irradiated aluminum, investigated by means of electrical resistivity measurements,<sup>20</sup> has shown annealing stages at 18 K (stage  $I_B$ ), around 35 K (stages  $I_C$  and  $I_D$ ) and at about 46 K (stage  $I_E$ ). These recovery stages have been attributed to close Frenkel pair recombinations (stages  $I_B$  and  $I_C$ ) and recovery associated with freely migrating interstitials (stages  $I_D$  and  $I_E$ ). In accordance with this the decrease of the mean positron lifetime  $\bar{\tau}$  around 40 K is due to annihilation of vacancies by migrating interstitials. Stage-I recovery has earlier been observed by the positron lifetime technique in Fe,<sup>21</sup> Mo,<sup>14</sup> and Ni.<sup>9</sup>

Annealing stage II in aluminum comprises the recovery in the temperature range from the termination of stage-I recovery at about 50 K to the beginning of stage III around 190 K. Relatively few investigations have been performed on stage II recovery, and the processes responsible for the rather complex behavior is not yet well under-

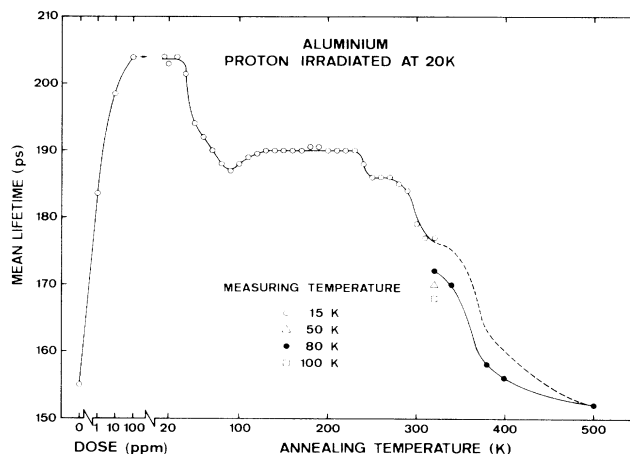


FIG. 3. The mean positron lifetime in aluminum as a function of proton irradiation dose and annealing temperature.

stood.<sup>20,22</sup> Two basic processes are suggested to be important for stage-II recovery: rearrangement of interstitial clusters and detrapping of interstitials from impurities. In neutron-irradiated Al stage-II recovery has been found at around 70, 83, 110, and 140 K.<sup>20</sup> The decrease of  $\bar{\tau}$  between 50 and 90 K, subsequently followed by a 3 ps increase between 90 and 130 K, may thus be attributed to such stage-II processes. Possible processes might also involve hydrogen trapping at vacancies. The migration energy of interstitial hydrogen in Al has been found to be 0.42 eV,<sup>23</sup> which corresponds to a migration temperature of about 100 K, and hydrogen may thus populate further the vacancies around this temperature. Breakup of hydrogen-interstitial complexes in this temperature range is also possible, which would give rise to decoration and annihilation of vacancies, and thereby changes of the mean positron lifetime.

#### C. Annealing above 200 K

The mean positron lifetime stays a constant around 190 ps between 130 and 230 K. At 240 K a decrease of 4 ps is observed, which is due to the loss of vacancies. Electrical resistivity measurements on neutron-irradiated Al have revealed two stage-III processes between 190 and 250 K.<sup>20</sup> A recovery stage at 190 K is attributed to recombination of correlated Frenkel pairs, whereas a stage around 240 K is due to uncorrelated recombinations of Frenkel defects at sinks such as interstitial clusters and surfaces. The number of vacancy migration steps for uncorrelated recombination decreases with increasing irradiation dose, which may shift the high-temperature stage to lower temperatures.<sup>20</sup> The observed decrease of  $\bar{\tau}$  around 240 K is thus attributed to loss of freely migrating vacancies. The intensity of the about 240-ps lifetime component dropped in stage III from about 60% to 50%.

Electrical resistivity<sup>20</sup> and positron annihilation experiments<sup>24</sup> have shown no further stage-III processes in pure neutron-irradiated Al. Annealing out of three-di-

mensional vacancy clusters and vacancy-interstitial loops is observed between 400 and 530 K with PAT (Ref. 24) and small-angle x-ray scattering.<sup>25</sup> The stages observed between 280 and 400 K in the annealing curve in Fig. 3 can then not be attributed to pure stage-III processes, but are rather processes influenced by the presence of hydrogen in the defected regions. Interstitial hydrogen migrates at approximately 100 K whereafter the hydrogen retained in the samples must be trapped at imperfections. The possibility of hydrogen trapping at Al vacancies was first suggested by Popovic and Stott<sup>26</sup> on theoretical grounds. Experimentally Bugeat *et al.*<sup>27</sup> and Myers *et al.*<sup>28</sup> have found, employing nuclear reaction analysis methods, that deuterium implanted into Al at low temperatures becomes released at about 290 K. This has been interpreted as release of deuterium trapped at vacancies. In the same temperature region we observe recovery of  $\bar{\tau}$ . The intensity of the  $\tau_2$  component decreases from about 50% at 250 K to about 30% at 320 K. The longest positron lifetime resolved is around 240 ps, which means that no vacancy clusters are present in the sample. This also indicates that monovacancies have been stabilized by the hydrogen. These can migrate around 290 K due to release of the trapped hydrogen. A similar elongation of stage III, due to the presence of impurities in vacancies, has previously been observed by PAT for H in Cu (Ref. 12), Nb (Ref. 13), Mo (Ref. 9), for C in Fe (Ref. 29) and Mo (Ref. 30), and for N in Mo (Ref. 31).

The highest annealing temperature available in the closed-cycle helium refrigerator was 320 K, and the sample set was transferred at 77 K to another cryostat, where the specimens could be kept below 80 K. The annealings were continued up to 500 K with all measurements acquired at 80 K. The change in measuring temperature caused the shift in  $\bar{\tau}$  observed at 320 K. Measurements were performed at 50 and 100 K before transferring the specimens. The mean positron lifetime is seen in Fig. 3 to increase with decreasing measuring temperature, which is due to a strongly increased effective trapping of positrons to jogs at dislocation lines at low temperatures. Such traps did not exist in an appreciable amount prior to the irradiations, but have been created during stage I and stage II. Dislocation lines in Al anneal out between 400 and 550 K,<sup>24</sup> and complete recovery of  $\bar{\tau}$  is also observed after the annealing at 500 K. If the measurements would have been acquired continuously at 15 K it is conceivable that the  $\bar{\tau}$  results would follow the dashed line shown in Fig. 3.

The decrease of  $\bar{\tau}$  between 280 and 400 K appears to take place in two stages: stage *A* between 280 and 310 K, and stage *B* between 320 and about 380 K. This recovery curve is very similar to the deuterium-retention profile obtained by ion-beam-analysis measurements on deuterium implanted Al.<sup>28</sup> Myers *et al.*<sup>28</sup> were not able to fit well the retention profile assuming only a single trap state for the deuterium, which then also suggests two release stages. In a recent publication Myers *et al.*<sup>32</sup> propose that vacancies occupied by more than one hydrogen atom will be stabilized to higher temperatures because the probability that the vacancy is empty and free to migrate is reduced. In their computer simulations they find that the migration

temperature of vacancies occupied by several hydrogen atoms will be shifted about 60 K compared to vacancies decorated by only a single H atom. It is possible that stage *B* in our experiment arises from such multiple occupancy. Above approximately 250 K all monovacancies retained in the samples must be stabilized by one or more hydrogen atoms, and it is conceivable that several vacancies have multiple occupancy.

The vacancy production during the proton irradiation is about ten times higher than the concentration of H atoms in the region containing hydrogen. However, due to spontaneous recombinations and during stage I at 40 K and stage III at 240 K all free vacancies are lost, either at surfaces or at interstitial clusters. The vacancies can annihilate in the irradiated region while the hydrogen must migrate to the surface to escape the sample. During this walk the hydrogen atoms may survey a considerable amount of vacancies already stabilized by H atoms and thus provide multiple occupancy.

When we assume that the recovery of  $\bar{\tau}$  in stage *A* is due to dissociation of vacancies decorated by single H atoms, with subsequent loss of vacancies at sinks, we can deduce the dissociation energy of a hydrogen-vacancy pair. We may neglect repeated hydrogen trapping at the released monovacancy and from simple considerations obtain the dissociation energy from

$$E_D = kT \ln(\nu t / \ln 2), \quad (2)$$

where  $k$  is the Boltzmann constant,  $T$  the temperature of 50%  $\bar{\tau}$  recovery in the stage,  $\nu$  the escape attempt frequency, and  $t$  the annealing time around the release temperature. The Al Debye frequency  $\nu_D = 8.21 \times 10^{12}$  Hz (Ref. 33) is utilized as the attempt frequency. With a dissociation temperature of  $295 \pm 10$  K we obtain  $E_D = 0.95 \pm 0.03$  eV. From Fig. 4 we find that the binding energy of the hydrogen to a vacancy, with respect to the interstitial site, is  $E_B = E_D - E_M = 0.53 \pm 0.03$  eV, where  $E_M = 0.42$  eV (Ref. 23) is employed. This result is in excellent agreement with 0.52 eV recently obtained from ion-beam-analysis experiments.<sup>28</sup> In the newly developed effective-

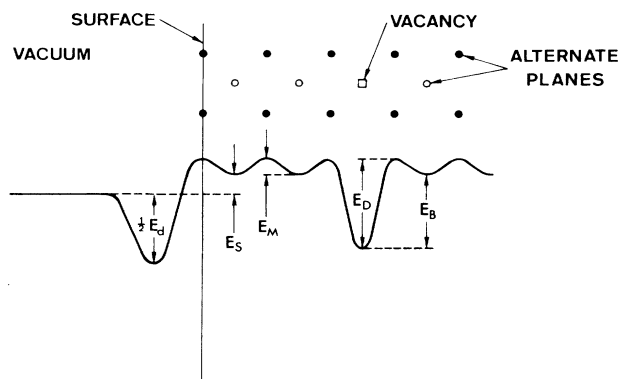


FIG. 4. Schematic drawing showing the definition for the energy of desorption ( $E_d$ ), solution ( $E_s$ ), migration ( $E_m$ ), dissociation ( $E_D$ ), and binding ( $E_B$ ).

medium theory the binding energy to an Al vacancy is calculated to be 0.52 eV,<sup>2,28</sup> also in excellent agreement with both the ion-beam analysis and the present positron-annihilation data.

#### IV. CONCLUSIONS

99.9999%-purity single crystals of Al have been irradiated at 20 K with 7.0-MeV protons. Isochronal annealings between 20 and 500 K have revealed several annealing stages in the positron lifetime results. Annihilation of vacancies by freely migrating interstitials in stage I is observed around 40 K. The changes of the mean positron lifetime between 50 and 130 K are attributed to stage-II recovery or trapping at vacancies of dissociated hydrogen and interstitial atoms. Around 240 K stage-III annealing is observed. Hydrogen in aluminum vacancies is found to immobilize the vacancies and shift the vacancy migration in stage III. Two recovery stages influenced by the pres-

ence of hydrogen are discussed in terms of single and multiple occupancy of hydrogen at monovacancies. From the recovery stage of dissociation of hydrogen-vacancy pairs a hydrogen binding energy to a vacancy is determined to be  $0.53 \pm 0.03$  eV. This result is in excellent agreement with ion-beam-analysis results and theoretical predictions. Complete recovery of the irradiated aluminum samples is found after annealing at 500 K. With the present investigations we wish to emphasize the advantage of the positron-annihilation technique to study hydrogen-defect interactions in metals.

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