

mentum of the atom due to the zero-point energy associated with the transverse localization has a distribution which is very similar to a particle in a 2D ideal gas at a temperature of a few kelvins. Thus, in these

models, the critical cone is broadened by the ground-state momentum of the localized adatoms, but the resulting desorption distribution is similar to the results of Fig. 3.

## Defect Structures below the Surface in Metals Investigated by Monoenergetic Positrons

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A new compact system has been developed to produce monoenergetic positrons of variable energy. The energy range available at present is between 150 eV and 28 keV, so that depth profiles can be measured. This positron beam is being used to investigate defects and defect structures close to the surface in metals and alloys. The technique of Doppler broadening of the annihilation radiation is applied for these measurements.

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The positron annihilation technique has been used for many years for the investigation of various defects and defect configurations in metals and alloys.<sup>1</sup> The positron is a very sensitive probe in particular for vacancylike defects and vacancy agglomerates. Since changes in the positron annihilation characteristics can be observed already for vacancy concentration as low as  $10^{-7}$  this method is especially useful for low defect concentrations. Because of this high sensitivity, positrons can be used to study the vacancy agglomeration to voids in metals, which is also of great technological interest with respect to materials used in high-flux fission and fusion reactors. Positrons emitted from a radioactive source have an energy distribution with maximum energies between 0.5 and 1.5 MeV for various isotopes. Thus the penetration depth of the positrons into the sample to be investigated is of the order of 100  $\mu\text{m}$ . Therefore in order to obtain results which are characteristic for the status of the sample, the concentration and the distribution of the defects has to be homogeneous over the total range of the positrons. For monoenergetic positrons of variable energy the damaged region can be relatively thin and close to the surface, since the penetration of the positrons can be adjusted according to their energy. By varying the energy of the positrons, different damage profiles in various depths from the surface can be sampled. This opens the possibility of investigating materials irradiated by low-energy light ions (H, He) and heavy ions (self-ions) by positron annihilation techniques. The first measurements on neutron- and helium-irradiated

metals are reported here.

Positrons from a 25-mCi  $^{22}\text{Na}$  source are thermalized in a 10- $\mu\text{m}$  gold foil covered with MgO (positron converter).<sup>2</sup> A fraction of about  $10^{-4}$  of the positrons are remitted from the converter foil with energies of approximately 1-2 eV. By electrical and magnetic lenses, these positrons are focused to form a positron beam of about 6 mm diameter and are then deflected  $180^\circ$  in a magnetic field. At present any energy between 150 eV and 28 keV can be obtained. Therefore the penetration profile of the positrons can be varied from a few times  $10^{-10}$  to  $10^{-6}$  m. The vacuum in the target chamber is  $3 \times 10^{-10}$  Torr and a lock system is installed to facilitate changing specimens while maintaining ultrahigh-vacuum conditions. The intensity of the positrons at the target is  $10^5$  per second. A tungsten collimator is placed in such a way that the high-purity Ge detector used for the Doppler-broadened 511-keV  $\gamma$  rays only measures annihilations emanating from the target itself.

A positron-annihilation Doppler-broadening spectrum provides momentum-distribution information about the electrons with which the positron annihilates. In well-annealed metal samples the positron annihilates while in a delocalized state in the essentially defect-free lattice. In irradiated samples positrons can be trapped at various defects and annihilate then from a localized state, and the annihilation characteristics give information about the nature of the defects. Results on neutron- and helium-irradiated copper specimens are shown in Fig. 1. All positron annihilation measurements were performed at

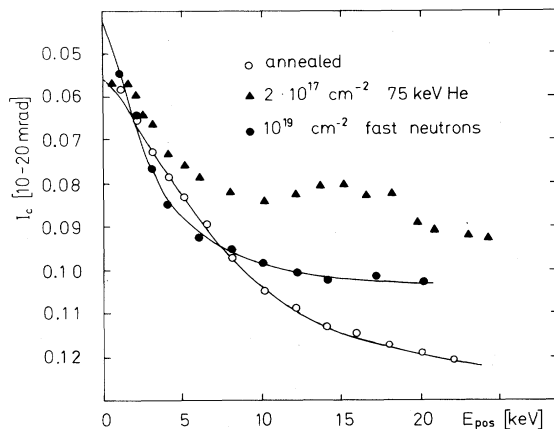


FIG. 1. The Doppler-broadening line-shape parameter  $I_c$  as a function of the positron energy for annealed, helium-implanted, and neutron-irradiated copper. The solid lines through the data points for the annealed and the neutron-irradiated copper are fits according to Eq. (1).

room temperature. The behavior of the line-shape parameter  $I_c$  (corresponding mainly to annihilations with core electrons; equivalent to an angular region of 10–20 mrad in angular-correlation measurements) is given as a function of the positron energy. Because of the low background in positron beam experiments, due to the absence of high-energy  $\gamma$  rays,  $I_c$  gives better statistical accuracy than  $I_v$ . The total number of counts over the annihilation peak is typically  $10^5$  to  $3 \times 10^5$ . Unless indicated the standard deviations of the data points are equal to or less than the size of the drawing symbols used.  $I_c$  for the neutron-irradiated (homogeneously damaged) sample containing dislocation loops is markedly different compared to the helium-irradiated sample containing helium bubbles and dislocations. The mean range of the implanted helium atoms at 75 keV is 2500 Å according to the calculations of Brice.<sup>3</sup> The solid lines through the data points for the annealed and the neutron-irradiated sample ( $10^{19}$  n/cm<sup>2</sup> at 60 °C) are obtained by a fit of the following relation which is valid for a homogeneous sample:

$$I_c(E) = I_c^\infty + \frac{I_c^0 - I_c^\infty}{1 + (E/E_0)^\kappa} \quad (1)$$

$I_c^0$  and  $I_c^\infty$  are the line-shape parameters for zero and very large ( $E \rightarrow \infty$ ) positron energy, respectively.  $E$  is the positron energy and the energy  $E_0$  is a fitting parameter. The exponent  $\kappa$  is chosen as 1.7 according to the results for the

TABLE I. The fitted energy  $E_0$  and the derived diffusion length  $l_d$ .

	$E_0$ (keV)	$l_d$ ( $10^{-10}$ m)
Cu, annealed	$7.29 \pm 0.28$	$703 \pm 46$
Cu, neutron irradiated (dislocation loops)	$2.87 \pm 0.12$	$145 \pm 10$
Ni, annealed	$8.28 \pm 0.58$	$873 \pm 104$
Ni <sub>76</sub> Si <sub>12</sub> B <sub>12</sub> , amorphous	$3.68 \pm 0.29$	$220 \pm 20$
Al, neutron irradiated (dislocation loops and voids)	$2.36 \pm 0.15$	$343 \pm 37$

mean penetration length of electrons<sup>4</sup> and positrons.<sup>5</sup> Equation (1) results from a one-dimensional diffusion model<sup>6</sup> of the positron in the sample. In Table I the diffusion lengths  $l_d$  are given according to the equation<sup>4</sup>

$$l_d = (215/\rho)(E_0)^\kappa \quad (2)$$

$\rho$  is the density of the material and  $E_0$  and  $\kappa$  are the same parameters as in Eq. (1). The values of  $l_d$  are in units of  $10^{-10}$  m when the units for  $E_0$  and  $\rho$  are keV and g/cm<sup>3</sup>, respectively. By simply measuring the positronium fraction at the surface, the positron diffusion length in a material can also be extracted under certain assumptions<sup>7</sup>; however, no information about the type of the defects can be obtained from these measurements in contrast to the Doppler-broadening results. A pronounced peak<sup>8</sup> of the  $I_c$  parameter is observed at positron energies of about 15 keV for the helium-irradiated copper. At this energy the mean penetration length of the positrons corresponds to the maximum of the implanted helium atoms.<sup>3</sup> The positron annihilation characteristics for positrons trapped at helium bubbles and at dislocation loops are quite different. For low positron energies (about 500 eV) almost all positrons annihilate from surface states which result in a characteristic  $I_c$  parameter, independent of the irradiation, and which is larger than the value obtained for positrons trapped at defects. There is strong evidence that the surface is a very effective “trap” for the positrons. At increasing positron energies there are mainly two competing processes for the diffusing positrons: “trapping” at the surface and trapping at defects in the bulk. Because of the relatively high defect concentration in the neutron-irradiated copper the typical bulk value is obtained at a much lower positron energy than the corresponding one for well-annealed copper.

The results for nickel irradiated with helium at various energies and fluences are shown in Fig. 2. From transmission electron microscopy<sup>9</sup> it is already known that under these conditions helium bubbles and dislocations are formed. At low positron energies the  $I_c$  parameter is found to be also independent of the status of the specimens. Lattice distortions, impurity layers, and the formation of parapositronium at the surface could account for this behavior; however, at present it is not possible to clearly separate these various possibilities.

For the annealed specimen the variation of the  $I_c$  parameter as a function of the positron energy can be well described by Eq. (1). For the specimen with the highest helium dose ( $1.2 \times 10^{18}$  He/cm<sup>2</sup> at 50 keV) the maximum of the implanted helium concentration is at about 1250 Å which corresponds to a mean range of 10.2-keV positrons. For the specimen irradiated with 75-keV helium ( $4 \times 10^{16}$  He/cm<sup>2</sup>) the behavior of  $I_c$  is quite similar whereas for the sample with  $2 \times 10^{17}$  He/cm<sup>2</sup> the distorted region is extended to higher positron energies. The defect-specific  $R$  parameter<sup>10</sup> was determined for various depths (i.e., positron energies) inside the specimen.

For the surface region we find an  $R$  parameter which is slightly smaller than the one for a deformed (dislocations) specimen. For the helium-irradiated samples this parameter, determined

at various positron energies, shows a clear dependence on the total fluence of the implanted helium. This indicates that more than one defect type is acting as positron trap. From the specimen with lowest dose ( $4 \times 10^{16}$  He/cm<sup>2</sup>) to the one with the highest dose ( $1.2 \times 10^{18}$  He/cm<sup>2</sup>) this  $R$  parameter increases 28% and is far above the value obtained for positron trapping at dislocations. The annihilation characteristic of positrons trapped at or in helium bubbles differs markedly from that of positrons trapped at the core region of dislocations or dislocation loops.

For the metallic glass Ni<sub>76</sub>Si<sub>12</sub>B<sub>12</sub>, the  $I_c$  parameter as a function of positron energy (Fig. 3) shows a similar behavior to that in the case of the neutron-irradiated copper (Fig. 1). The asymptotic values (high positron energy) of the  $I_c$  and the  $R$  parameter are the same as for deformed pure nickel.<sup>11</sup> This indicates that in both samples trapping sites of similar kind are effective for the positrons. From the saturation behavior of  $I_c$  a positron mean diffusion length can be deduced (Table I) and hence an estimate can be made about the concentration of the acting trapping sites for the positron. A density of about 1 at.% of effective trapping centers is obtained. After the implantation with 8-keV helium a considerable change of the  $I_c$  value is observed in the positron energy range between 2 and 10 keV. The experimental results indicate that the

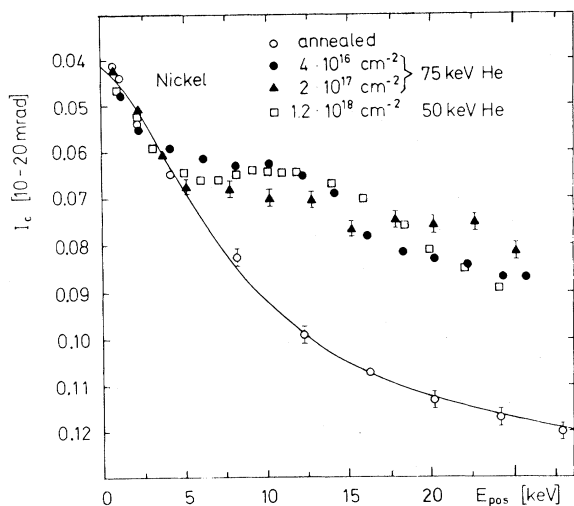


FIG. 2. The Doppler-broadening line-shape parameter  $I_c$  as a function of the positron energy for annealed and helium-implanted nickel. The solid line through the data points for annealed nickel is a fit according to Eq. (1). The 50-keV helium ions were implanted at 45° relative to the surface.

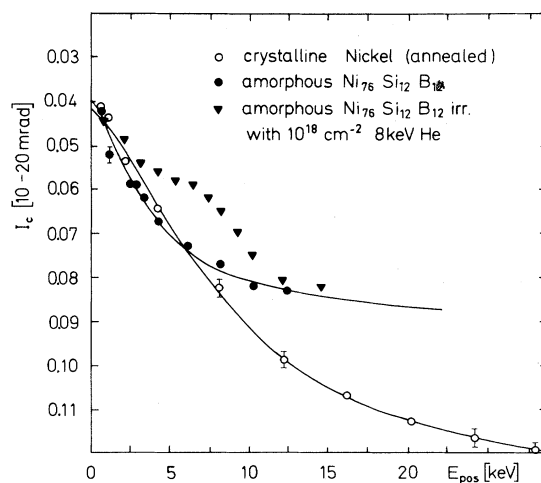


FIG. 3. The Doppler-broadening line-shape parameter  $I_c$  as a function of the positron energy for the nickel-based metallic glass Ni<sub>76</sub>Si<sub>12</sub>B<sub>12</sub> as received and implanted with 8-keV helium. For comparison the values for crystalline nickel (Fig. 2) are given. The solid lines through the data points for annealed nickel and the amorphous glass are fits according to Eq. (1).

penetration depth of the implanted helium atoms has to be extended about twice over the calculated mean range. The trapping of positrons at helium again is quite obvious.

These first measurements on helium-irradiated specimens using monoenergetic positrons of variable energy combined with the Doppler-broadening technique show the potential and the application possibilities of such a device. The measured Doppler-broadening spectra and the hence derived  $I_c$  parameter are mean values of the annihilation characteristics of the local defect configurations folded with the range distribution and the diffusion of the positrons. The local variation of the annihilation parameters due to a specific damage profile is therefore larger. A model is being developed to unfold the observed annihilation parameters at the various positron energies in order to obtain more detailed information about the nature and the range of the damaged regions.

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<sup>8</sup>Since the scale of the  $I_c$  parameter is from top to bottom, a peak in  $I_c$ , as mentioned in the text, is in fact a decrease in the actual value of  $I_c$ .

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