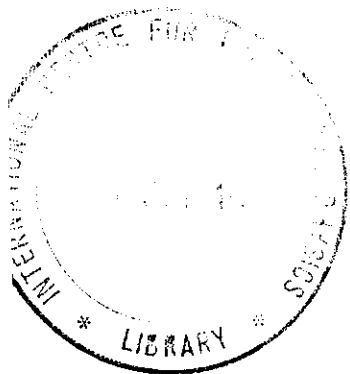


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SPRING COLLEGE IN MATERIALS SCIENCE
ON
"METALLIC MATERIALS"
(11 May - 19 June 1987)

POSITRON STUDIES ON DEFECTS
AND MECHANICAL PROPERTIES OF METALS

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POSITRON STUDIES on DEFECTS and MECHANICAL PROPERTIES of METALS.

I. Introduction to PA

Special advantages:

Sensitive to the presence of defects.

$\sim 10^{-7}$ vacancy concentration

For studies of nondilute alloys

$> 1\% \rightarrow 100\%$

For studies under wide range temperatures

Wide range of fields

metals, semiconductors, ionic solids, polymers....

mechanical engineering, materials science,

solid state physics, theoretical physics...

Not very expensive

$\sim$ US \$ 20,000 - 40,000.

References

1. P. H. Geiger, *Positronium in Solids*,

Springer, Berlin, 1961.

2. M. L. Huggins and A. J. G. Rees,

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Sch. Phys. "Enrico Fermi", North-Holland

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3. P. C. Jain, R. M. Singru and K. P. Gopinathan,

Positron Annihilation, Proc. ISPA-7.

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4. S. W. Ling and N. H. March, *Cryst. Latt.*

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I. 1. Thermalization

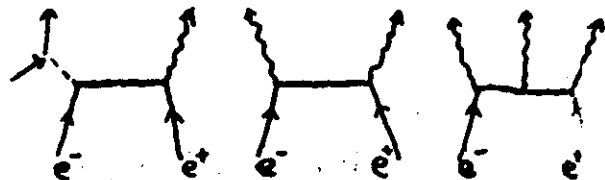
e^+ Source, ^{22}Na , ^{64}Cu , ^{60}Co , ^{68}Ge γ -ray
(0.54 Mev) (0.65) (0.48) (1.4) (1.28 Mev)

time: $\sim 10^{-12}$ sec.

distance: 10 - 1000 μm .

energy: $kT (\sim 30^\circ\text{C}) \sim 0.025$ eV.

2. Annihilation, ($\gamma \ll \alpha = \frac{1}{137}$)

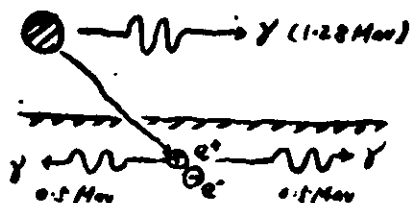


$$\sigma_{(12)} / \sigma_{(12)} \approx \alpha, \quad \sigma_{(12)} / \sigma_{(12)} \approx \alpha^2$$

$$\sigma_{(12)} = \frac{\pi r_0^2 c}{v}$$

$$\Gamma_{(12)} = \sigma_{(12)} v n_0 = \pi r_0^2 c n_0$$

$$2m_0 c^2 = 2 \times 0.511 \times 10^6 \text{ eV}$$



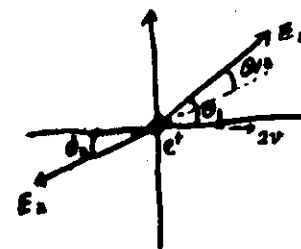
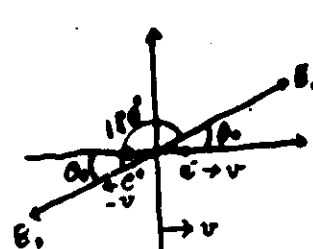
II. Lifetime $\tau = \Gamma^{-1}$

	Solid.	Liquid	Gas.	$\tau_p < \tau_d$
τ (sec)	$\sim 10^{-10}$	$\sim 10^{-9}$	$\sim 10^{-7}$	

III. Angular correlation

e^-, e^+ static, $\vec{p}_1 + \vec{p}_2 = 0, \vec{e}_1 = -\vec{e}_2, 180^\circ$

e^- motion, $\vec{p}_1 + \vec{p}_2 \neq 0, \vec{e}_1 \neq -\vec{e}_2, \theta_{12}$

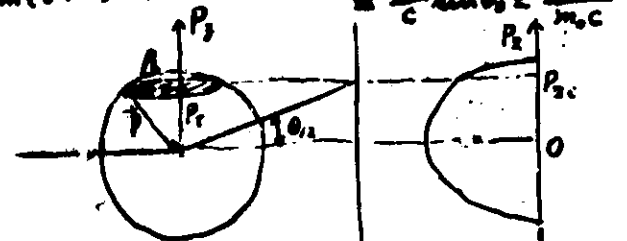


(a) axis system of center of mass

(b) laboratory axis system

$$\sum \vec{P}_i = m(\vec{v}_1 + \vec{v}_2) = 0$$

$$\theta_{12} \approx \theta_1(0, -\theta_1) \approx \frac{2\gamma \sin \theta_1}{1 - v^2/c^2} = \frac{2\gamma \sin \theta_1}{m_0 c}$$

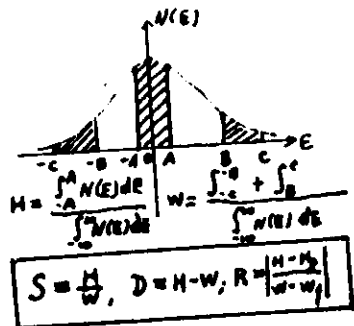


$$N(\theta_2) = c \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} dp_x dp_y \Gamma(p_x, p_y, \theta_2, m, c) = N(p_2)$$

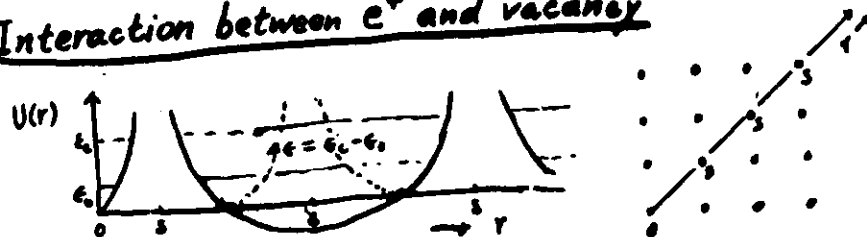


$$\approx m_0 c \pm \frac{CP_L}{2} = E. \pm SE$$

$$P_L = 2m \cdot v \cos \theta$$



V. Interaction between e^+ and vacancy



VI. trapping model

$$\frac{dn_f(t)}{dt} = -\left(\frac{1}{\tau_f} + \sum_{j=1}^m \sigma_j(t) C_j\right) n_f(t) + \sum_{j=1}^m \nu_j n_j(t)$$

$$\frac{d\eta_j(t)}{dt} = -\frac{\eta_j(t)}{\tau_j} + \sigma_j(t) C_j \eta_j(t) - \lambda_j \eta_j(t)$$

$(j = 1, 2, \dots, m)$

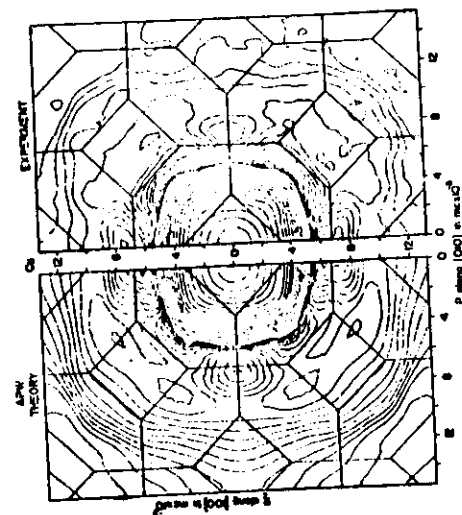


Fig. 9 Contour maps of experimental vs theoretical $N(p, q) - g(p, q)$ along the $[001]$ direction. $N(p, q)$ is a smooth tetrahedrally symmetric surface as discussed in the text, and is used to establish the anisotropies at high momenta. The steps of the contour lines are changed at around $p = 3.5$ mrad as discussed in the text.

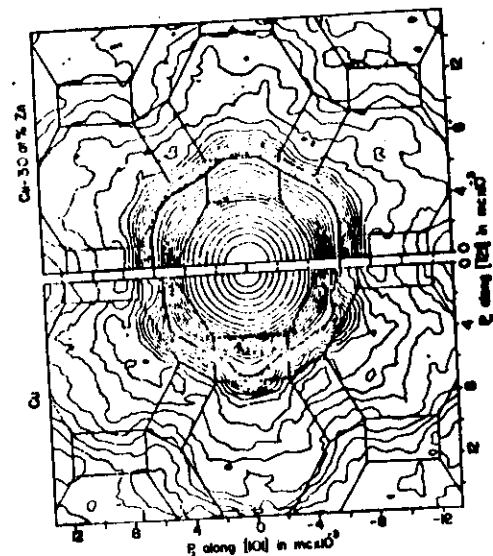


Fig. 11 The contour map of the $H(p_2)$ - (p_1, p_2) surface from Ca to Ca-30 at $z = 2n$, oriented with p_1 along the [111] direction. $H(p_2)$ is a smooth practically symmetric surface as illustrated in the text, used to exhibit also anisotropies at high energies.

[illegible]

Assuming: $\nu_j = 0$ (no detrapping) A. Suger
(J. Phys. F, 3 (1973) 248)
 $\sigma_j(t) = \sigma_j$

The solution is

$$n_f(t) = n_f(0) \exp(-t/\tau_0)$$

$$(\frac{1}{\tau_0} = \frac{1}{\tau_f} + \sum_{j=1}^m \sigma_j c_j)$$

$$n_j(t) = \tau_0 \tau_j \frac{\sigma_j c_j}{\tau_0 - \tau_j} n_f(0) \exp(-t/\tau_0) +$$

$$[n_j(0) - \tau_0 \tau_j \frac{\sigma_j c_j}{\tau_0 - \tau_j} n_f(0)] \exp(-t/\tau_j)$$

$$j = 1, 2, \dots, m.$$

For spherical attractive potential wells, one obtains

$$\sigma_j(t) = \frac{4\pi r_j D}{\Omega} [1 + r_j (\pi D t)^{-1/2}] \quad \text{Waite's theory}$$

$$\text{or } \sigma_j(t) \approx \frac{4\pi r_j D}{\Omega}$$

where Ω is the atomic volume, and D the diffusion coefficient.

$$k = \left\{ \frac{\sigma}{\mu} \right\} c_t \quad \leftarrow \begin{array}{l} \text{trapping rate} \\ \text{specific} \sim \text{concentration} \\ \text{of traps of type } j \end{array}$$

$$\bar{\tau} = \frac{\int_0^\infty [n_f(t) + \sum_{j=1}^m n_j(t)] dt}{n_f(0) + \sum_{j=1}^m n_j(0)}$$

$$\bar{\tau} = \tau_f \cdot \frac{1 + \sigma \tau_0 c_{iv}}{1 + \sigma \tau_j c_{iv}} \quad (\text{for monovacancy})$$

$$\text{or: } \frac{\bar{\tau} - \tau_f}{\tau_0 - \tau_f} = \frac{\sigma c_{iv}}{1 + c_{iv} \sigma}; \quad \boxed{\frac{\bar{\tau} - \tau_f}{\tau_0 - \bar{\tau}} = \tau_j \sigma c_{iv}}$$

$$\sigma = \frac{4\pi D r_{iv}}{\Omega}$$

$$\bar{\tau} = \tau_f \cdot \frac{1 + (4\pi r_{iv} c_{iv} \tau_0 D / \Omega)}{1 + (4\pi r_{iv} c_{iv} \tau_j D / \Omega)}$$

$$c_{iv} = \exp(S_{iv}^F/k) \exp(-H_{iv}^F/kT)$$

$$\boxed{H_{iv}^F = kT \left[\frac{S_{iv}^F}{k} + \ln \left(\frac{4\pi D}{\Omega} r_{iv} \tau_j \right) + \ln \left(\frac{\tau_0 - \bar{\tau}}{\bar{\tau} - \tau_f} \right) \right]} \\ (\text{vacancy formation energy})$$

VII.

$$\Gamma_0 = \pi r_0^2 c n_e$$

$$\Gamma_0(p) = (\pi r_0^2 c) \frac{1}{(2\pi)^3} \sum_i \left| \int d\mathbf{r} \exp(-i\mathbf{p} \cdot \mathbf{r}) \psi_i(\mathbf{r}) \psi_i^*(\mathbf{r}) \right|^2$$

$\psi(\vec{r})$: electron wave function

$\psi_+(\vec{r})$: positron wave function

$r_0 = e^2/mc^2$: the classical electron radius.

VIII. e^+ and defects pseudopotential and pseudo wave function

1. $\Psi_k(r) = U(r-R) \varphi_k(r)$ (perfect crystal)

$$\left[-\frac{\hbar^2}{2m} \nabla^2 + V_A(r) \right] U(r) = E_w U(r)$$

$$\left. \frac{\partial U}{\partial r} \right|_{r=R_0} = 0 \quad R_0: \text{muffin-tin radius}$$

$$\left[-\frac{\hbar^2}{2m} \nabla^2 + w(r) \right] \varphi_k(r) = E_c(k) \varphi_k(r)$$

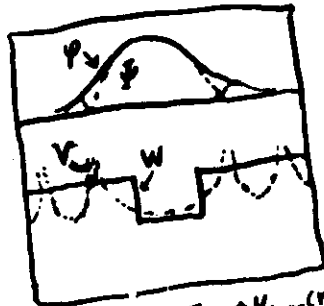
where,

$$w(r) = \begin{cases} E_w + V(r) - V_A(r-R) - \frac{\hbar^2}{m} \frac{\nabla U(r)}{U(r)} \cdot \nabla & (\text{in}) \\ V(r) & (\text{between}) \end{cases} \quad (w \ll v)$$

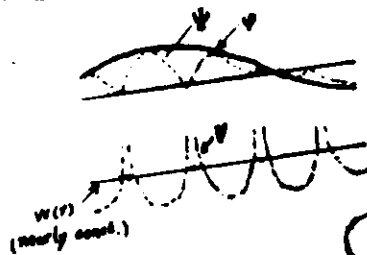
2. defect crystal

$$V(r) = \sum_i V_{ref}(r-R_i) + \Delta V(r) + V_{corr}(r)$$

$$V_{ref}(r) = \begin{cases} V_A(r) & (\text{in}) \\ 0 & (\text{outside}) \end{cases}$$



$$w(r) = \Delta V(r) + E_c + V_{corr}(r) - \frac{\hbar^2}{m} \frac{\nabla U(r)}{U(r)} \cdot \nabla$$



(10)

IX. Annihilation characteristic

1-D angular correlation curve

$$I(p_2) = \int dp_1 \int dp_2 P_0(p_2)$$

isotropic e^+e^- overlapping integral

$$I(p_2) = 2\pi \int_{p_2}^{\infty} dp \cdot \bar{p} P_0(p)$$

homogeneous electron gas: $P_0(p) = \frac{\pi r_F^3 c}{(2\pi)^3}$

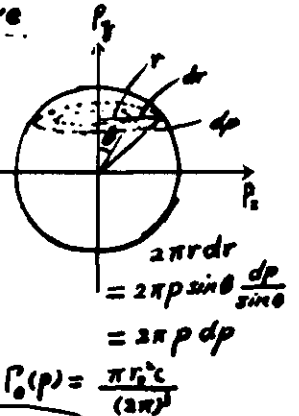
$$I(p_2) = \frac{r_F^3 c}{4\pi} (p_F^2 - p_2^2) \theta(p_F - |p_2|)$$

($|p_2| < p_F$ if, $\theta(p_2) = 1$; $|p_2| > p_F$ if, $\theta(p_2) = 0$)

inhomogeneous electron system:

$$I(p_2) = \frac{\pi^3 c}{4\pi} \int dr |\psi(r)|^2 \lambda[n(r)]$$

$$\text{where, } \lambda[n(r)] = \frac{n(r)}{4\pi} [p_F(r)^2 - p_2^2] \theta(p_F(r) - |p_2|)$$



PA studies on Defects and Mechanical Properties of Metals

Positrons tend to locate in low ion density
or in low electron density regions

in Cryst. Latt. Defects and Amorph. Mater.
Vol. 12 (1981) 31-63

Positron-vacancy interactions

Binding energy $E_b = \Delta E = E_a - E_o$

The Schrödinger equation for the trapped e^+ is

$$H\psi_p(r) + E_b \psi_p(r) = 0$$

E_o : the energy of the drifted positron in a perfect solid

minus the energy of the trapped positron.

$$H = -\frac{\hbar^2}{2m} \nabla^2 + V_c$$

$$V_c = -\frac{1}{r} + V_e(r) + V_{ion}(r)$$

electrostatic ion density

Hodges C.H. (1970),

$$E_b = 3.81 \text{ eV} - 2.54 \text{ eV} (r \text{ in } \text{\AA})$$

Mannien et al. (1975)

Comparison between K. and TF methods

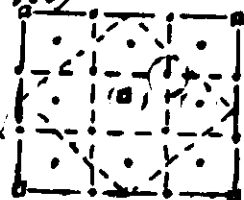
Metal	E_b	
	K.	TF
Li	0.00	0.04
Na	0.02	0.19
K	0.02	0.26
Cs	0.01	0.26
Be	0.80	1.32
Hg	0.59	1.04
Al	1.75	2.61
Tl	1.72	2.50
Sn	2.64	3.59

Gupta and Siegel (1977, 1980)

Superlattice lattice model

APV method

$$E_b(\text{Al}) = 3.36 \text{ eV}$$

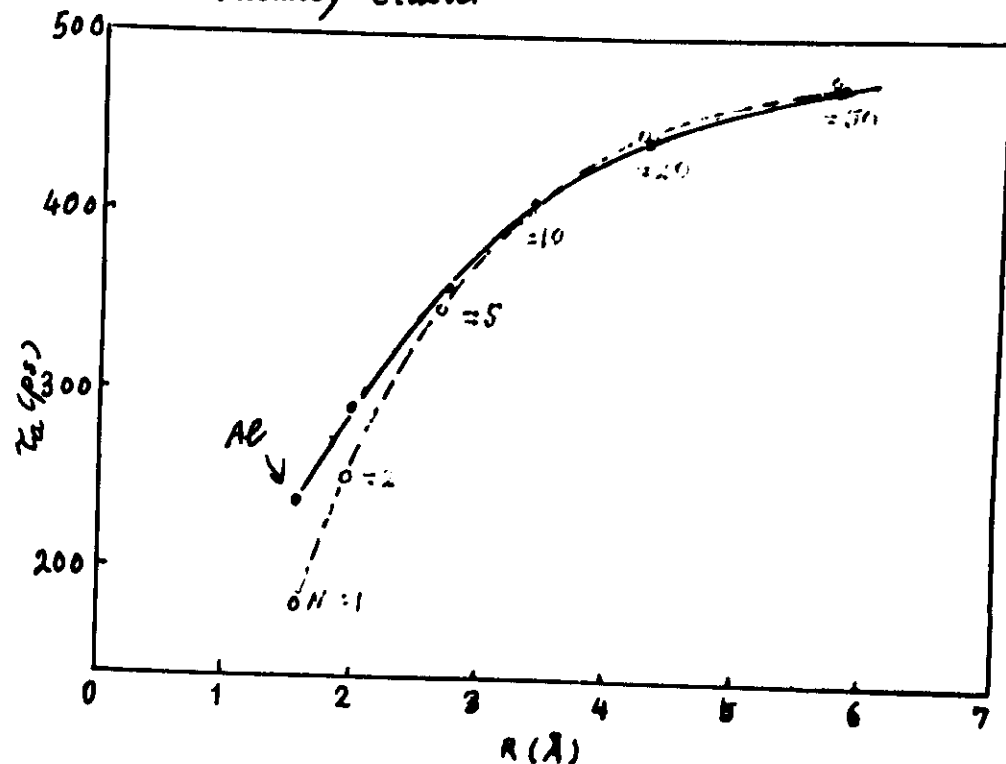


• Al

□ Vacancy

○ muffin tin potential

Vacancy cluster



--- P. Hautajärvi et al. (1977)

••• P. Hautajärvi et al. (1977)

H.E. Hansen et al. J. Appl. Phys. 61 (1987) 1499

Computational analysis of positron experiments

1305

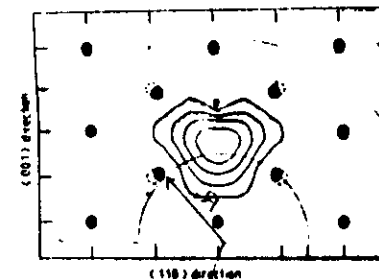


Figure 2. Wavefunction of a positron trapped in an N-decorated Mo vacancy. Full circles indicate Mo ions (note the ~7% relaxation of the nearest neighbours to the vacancy). The N atom is indicated by a small open circle.

between the fact that the calculated binding energy of the clean vacancy is twice that of the N-decorated vacancy and the experimental results that the positron trapping rates into these two defects differ little (Nielsen et al 1983). For trapping mediated by electron-hole pairs the trapping rate scales linearly with the binding energy if the conduction electrons are treated as plane waves in both initial and final states (Niemi and Lachinen 1979). However, one might also argue that introducing the impurity into the vacancy does not necessarily decrease the trapping rate even if it does so for the binding energy; the trapping rate is proportional to the local density of final electron states, which may be enhanced by the impurity.

3.3. Hydrogen-decorated vacancies

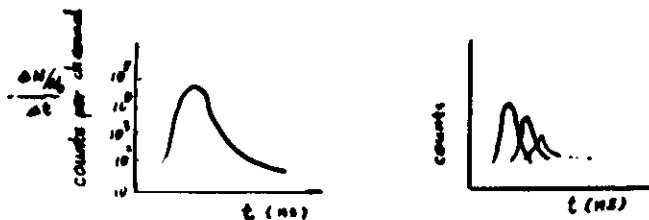
Hydrogen interactions with lattice defects in metals attract widespread attention (Picaux 1981), partly because of their technological significance. Currently, positron lifetime spectrometry is being introduced into this field with the objective of gaining new detailed information at the atomic level. We expect our model calculations to aid the progress of this work.

Unlike its group V neighbours in the Periodic Table, Mo has a large positive enthalpy of solution for H ($\Delta H_{\text{sol}} \approx 0.5 \text{ eV}$, $\Delta H_{\text{ad}} \approx -0.3 \text{ eV}$ (Völkl and Alefeld 1973)) implying a H solubility 14 orders of magnitude smaller than in Mo at room temperature. Therefore even a small concentration of defects capable of trapping H may increase considerably the solubility of H in Mo just as in the case of N (Anzula and Hirvonen 1978, Nielsen et al 1982, Hansen et al 1982). Such defects (dislocations, vacancies, voids) may also decrease the H diffusion and thus be responsible for the rather large differences between the reported migration energies of H in Mo (Völkl and Alefeld 1973).

Recent experiments by Nielsen and co-workers (to be published), have clearly demonstrated the potential of the positron lifetime technique for investigation of H interactions with defects in Mo. In the case of vacancies the response is fairly small: the long lifetime in electron-irradiated Mo decreases from 180 to 165 ps after electrolytic injection of H.

Again we shall ask whether this lifetime should be attributed to vacancies decorated by

Vacancy studies with PA



The resulting experimental time spectrum $S(t)$ can be written as the sum of n components

$$S(t) = \sum_{i=1}^n I_i \Gamma_i e^{-\Gamma_i t}$$

For simplicity, we discuss the two state trapping model.

$$S(t) = I_1 \Gamma_1 e^{-\Gamma_1 t} + I_2 \Gamma_2 e^{-\Gamma_2 t}$$

$$\Gamma_1 = \lambda_f + k = \tau_1^{-1} \quad I_1 = \frac{\lambda_f - \lambda_2}{\Gamma_1 - \lambda_2}$$

$$\Gamma_2 = \lambda_c = \tau_2^{-1} \quad I_2 = \frac{k}{\Gamma_1 - \lambda_2}$$

$$(\lambda_f = \tau_f^{-1}, \quad \lambda_c = \tau_c^{-1}, \quad k = \sigma C_t)$$

$$\sigma C_t = I_2 \left(\frac{1}{\tau_1} - \frac{1}{\tau_2} \right)$$

$$C_t = \exp(S_f/k_B) \exp(-H_v^F/k_B T)$$

$$\sigma C_t = I_2 (\tau_1^{-1} - \tau_2^{-1})$$

$$C_t \equiv C_{iv} = \exp(S_f/k_B) \exp(-H_v^F/k_B T)$$

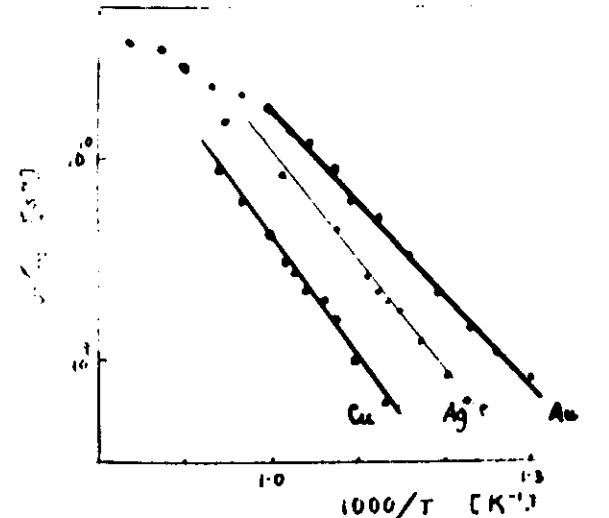


Fig. Arrhenius plots of the trapping rates

σC_t with T^{-1}

Proc. ICPA-7 (1982) 448

Stack and Schaefer

Table: Vacancy formation enthalpies H_v^F by PA

Metal	H_v^F (ev)	Metal	H_v^F (ev)
Mg	0.85 ± 0.10	Ta	2.8 ± 0.60
Al	0.68 ± 0.04	Cr	2.00 ± 0.20
In	0.55 ± 0.03	Mo	3.0 ± 0.20
Cu	1.29 ± 0.07	W	4.0 ± 0.30
Ag	1.12 ± 0.07	d-Fe	1.6 ± 0.15
Au	0.89 ± 0.04	Co	1.34 ± 0.07
Zn	0.52 ± 0.07	Ni	1.78 ± 0.10
Cd	0.52 ± 0.02	Pd	1.25 ± 0.25
Pb	0.57 ± 0.06	Pt	1.32 ± 0.04
V	2.1 ± 0.20	Sn	0.50 ± 0.01
Nb	2.65 ± 0.40	U	≈ 1.0

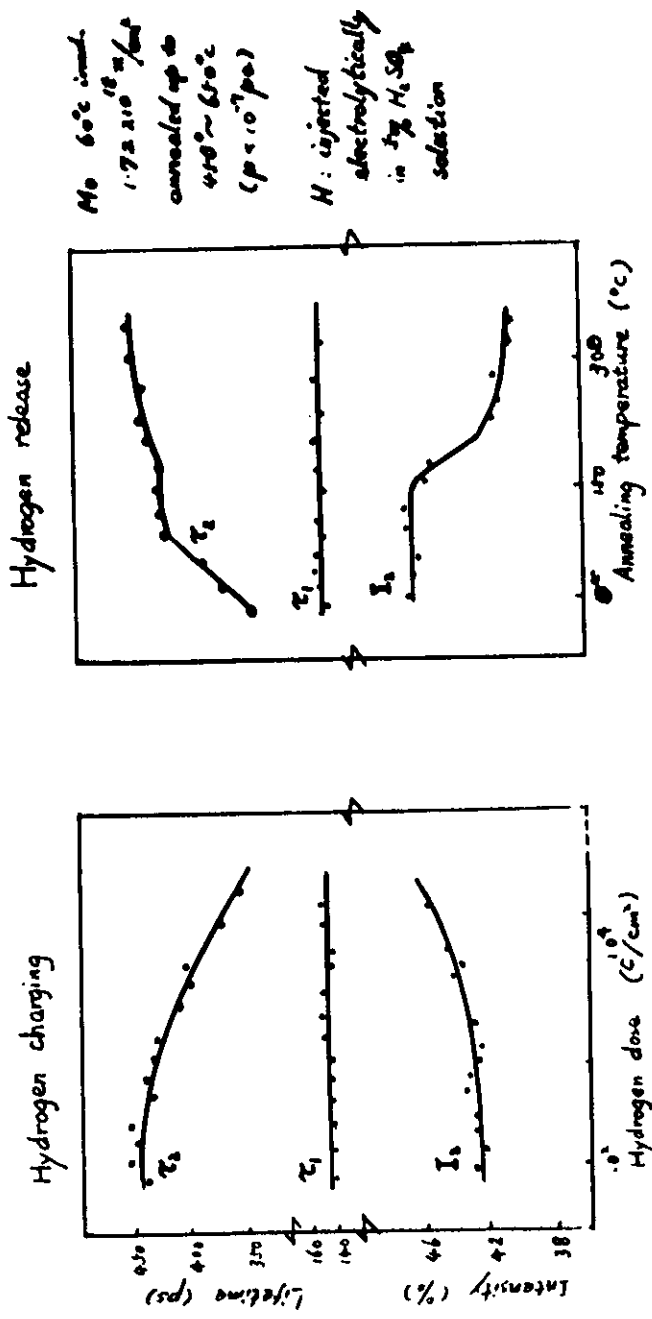
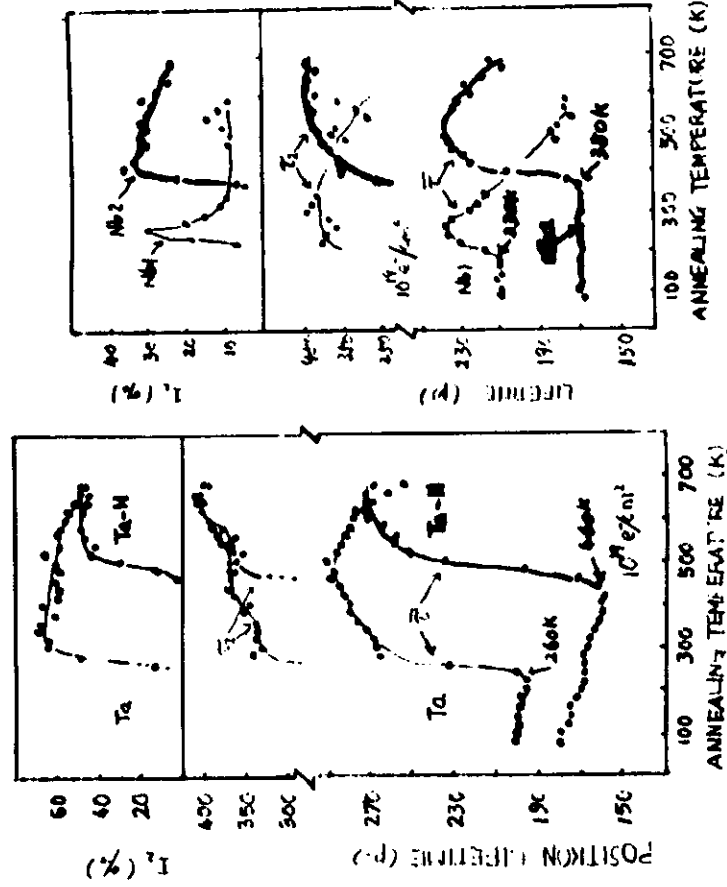


Fig. Positron lifetime parameters in Mo containing voids
 ($T_{\text{prep}} = 450^{\circ}\text{C}$) during electrolytical H injection
 (uncal. scale) and thermal H release.

Proc. ICPA-7 (1985) 497
 B. Nielsen et al.

- 17 -



Ta: UNV degraded foil;
 in H gas at 500K
 slowly cooling to room
 temp. at 5°C/s

Proc. ICPA-7 (1985) 519

P. Manteghini et al.

- 18 -

Nb annealed at 910°C in HV for 2 hrs before exp.

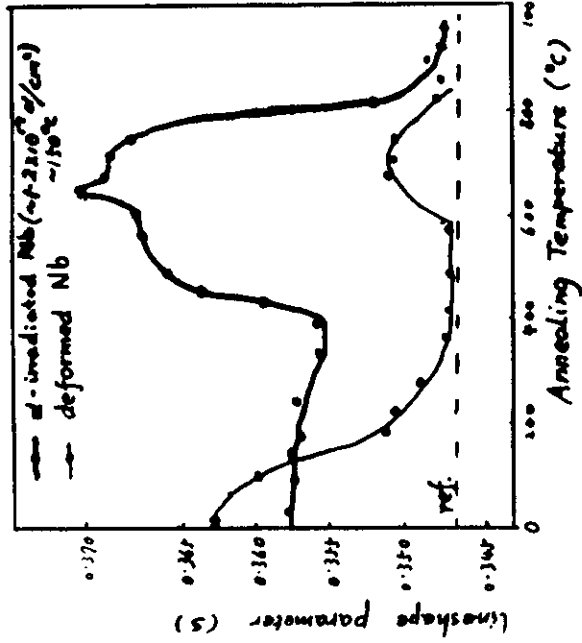


Fig. 5-T curve in d-irradiated and deformed Nb ($2.2 \times 10^{19} \text{ cm}^{-3}$)

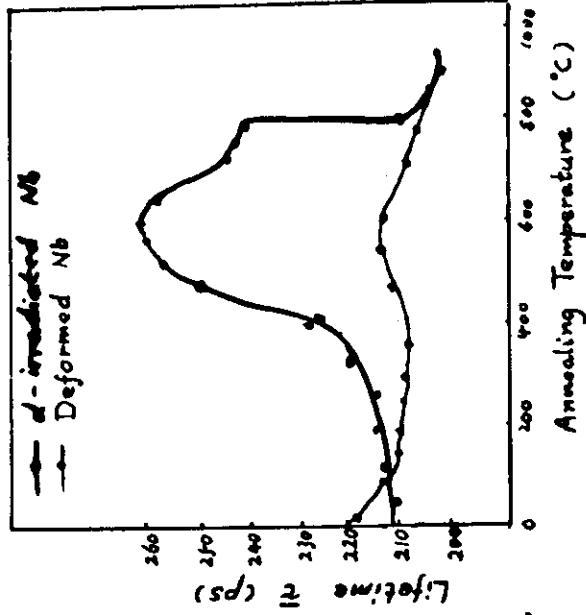
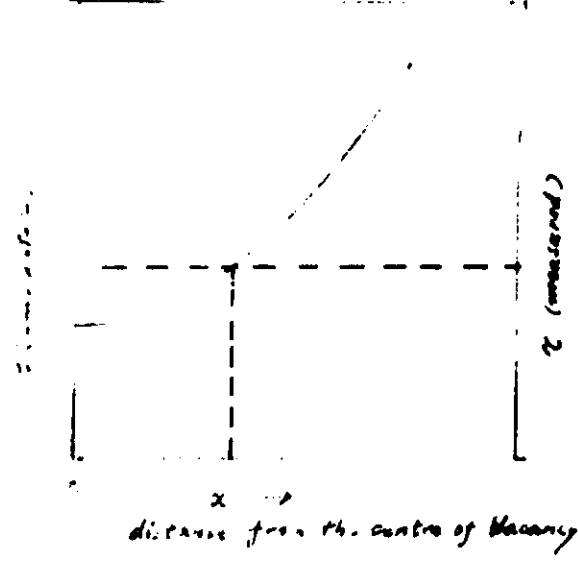


Fig. 7-T in d-irradiated and deformed Nb

Proc. ICPA-7, (1981), vol.

S.V. Merits et al.

-19-



The position of the atom of the impurity atom-decorated vacancy can be determined by combination of theoretical calculation and PA lifetime measurements.

DISLOCATION TRAPS

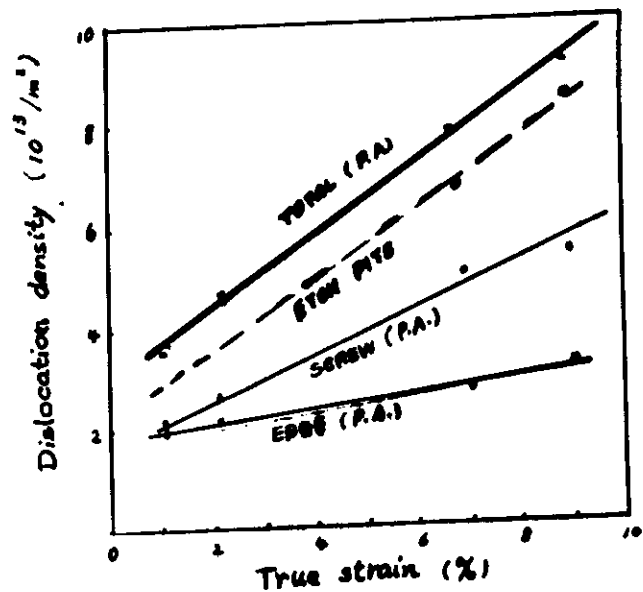


Fig. Dislocation densities measured by PA and etch pits in iron single crystals in tension at 200K.

$$\tau_1 = 148 \pm 3 \text{ ps.}$$

$$\tau_2 = 168 \pm 3 \text{ ps.}$$

$$\epsilon < 10\%$$

$$\tau_p = 114 \text{ ps.}$$

$$\text{jog separation: } 2000b \quad (\text{by TEM})$$

$$\text{no. of jog traps / total traps } \approx 10^{-3}$$

The unresolved (small) jogs can not be ruled out as being the sites for PA.

ICPA-7 (1985) 187

Y.N. Park et al.

P-N dislocation core model and positron annh. effects 位错中心 P-N 模型和正电子湮没效应

$$u_x = -\frac{b}{2\pi} \tan^{-1}\left(\frac{x}{f}\right)$$

f , semi width of the disl. core $\rightarrow$ simple 2-D model
 是位错半宽度 $\rightarrow$ 二维的简化模型.

$$u_y = -(b/2\pi) \tan^{-1}\left(\frac{y}{f}\right)$$

$$u_z = 0$$

位错中心的体积膨胀 volume expansion in the dislocation core

$$f(r) = \text{div } u$$

$$= -(b/2\pi r) \tan^{-1}\left(\frac{r}{f}\right) - \frac{b}{2\pi f} \frac{1}{1 + (r/f)^2}$$

$$\rho_c(r) = \rho_0 [1 + f(r)] \quad (\text{位错心处正电子密度})$$

ion density in the disl. core

$$= \rho_0 \left[1 - \frac{b}{2\pi r} \tan^{-1}\left(\frac{r}{f}\right) - \frac{b}{2\pi f} \frac{1}{1 + (r/f)^2} \right]$$

$$\Delta\tau = \tau_d - \tau_f = 15 \text{ ps}; \quad E_b = 1.1 \text{ eV}$$

compare with binding energy due to vacancy

$$E_b^v = 2.2 \text{ eV}$$

compare with Martin and Padgett's results of disl. (1972)

$$E_b^d \approx 0.1 \text{ eV.}$$

compare with Harrison et al. (1973)

$$E_b^d = 2.79 \text{ eV.}$$

Shen J.R., Li W.C.W., Wang K.L.

Proc. ICPA-7 (1985)

phys. stat. sol. (b) 134, (1986)

PA parameters in various models

Models	τ_b (ps)	τ_f (ps)	E_b (ev)	$\Delta\tau/E_b$ (ps/ev)
Vacancy	251	168	2.2	37.7
Cylindrical hole	229	168	2.1	29.0
Arponen et al.	229	168	2.8	21.8
P-N Model	183	168	1.1	18.6

The extended character of the defect model increases, $\Delta\tau/E_b$ value drops down.

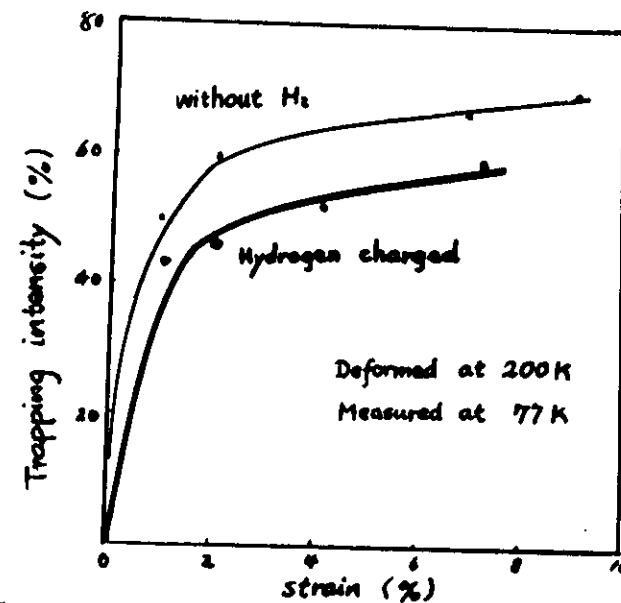
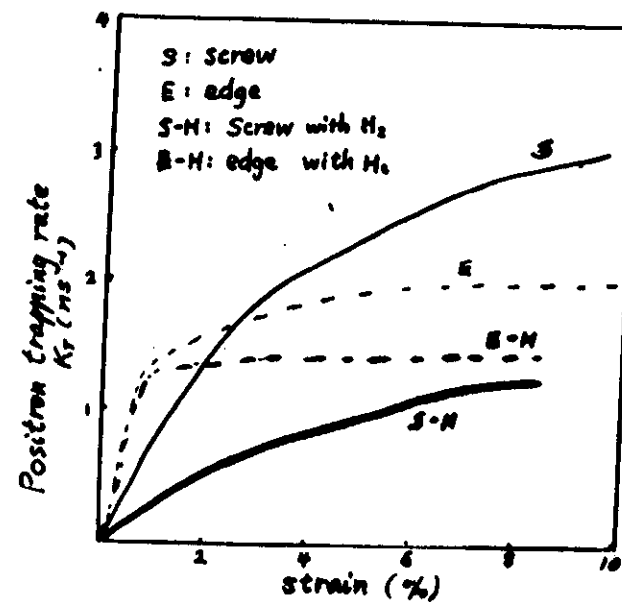


Fig. Fe single crystals deformed in tension at 200K

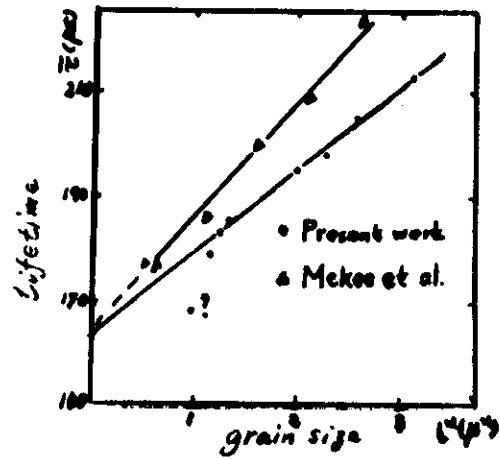
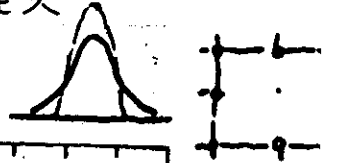


The H effect is larger in a screw dislocation than in an edge dislocation !

Proc. ICPA-7(1985)590

V.K. Park et al.

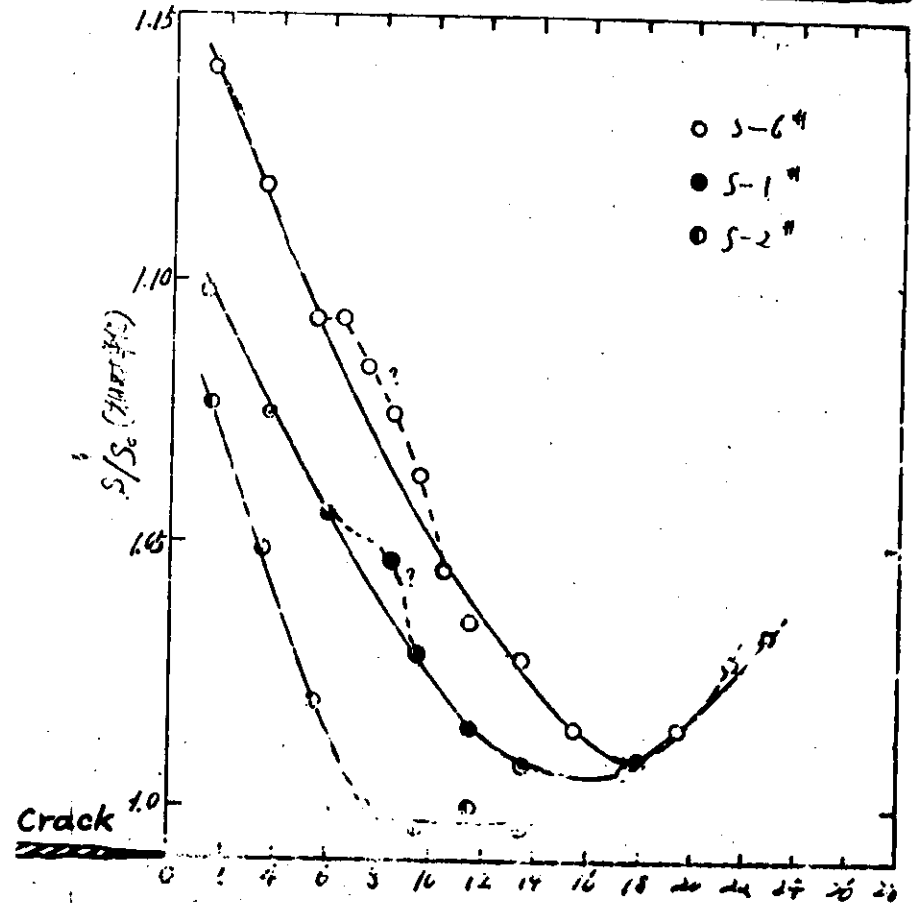
裂缝顶端范性区的正电子湮灭



Heat treatments and measured results

Temp.	Time	quenching	grain size	Lifetime
温度	时间	淬火方式	晶粒尺寸 (μm)	τ (ps)
6 300°C	0.5hr.	油淬	0.65	178
2 350°C	0.5hr.	水淬	0.39	205
250°C		空冷		
1 350°C	0.5hr.	油淬	0.44	198
250°C				
15 280°C	0.5hr.	油冷	0.80	195
3 380°C	0.5hr.	水淬	0.76	188
250°C		水淬		
14 250°C	0.5hr.	空冷	0.86	179
12 280°C	0.5hr.	油淬	1.02	168
16 试样初态			0.80	183

(周先惠, 王淑英, 蒋惠林, 姜健, 龙翔威), 1984.
ZHOU, WANG, JIANG, JIANG, LUNG



Position annihilation in the plastic zone at a crack tip

in «Positron Annihilation», (1982) 469, 1981.6.

North-Holland Publ. (Jiang, Xiong, Lung et al.)

1. Dislocations and vacancies were mixed, can not see DF2
2. can measure the plastic zone size*

* Xiao Jimui et al. Scripta Met. 1982.

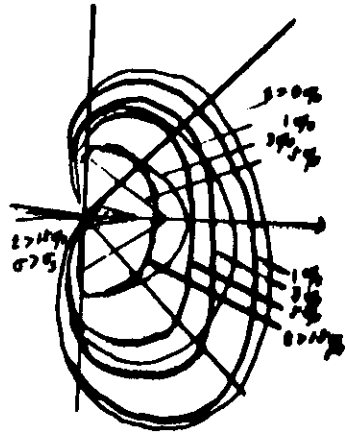


Fig. The H effects on the plastic zone of cracked 40MnNb steel measured by PA.

Tian Z. et al. Scripta Met., 16 (1982), 1383

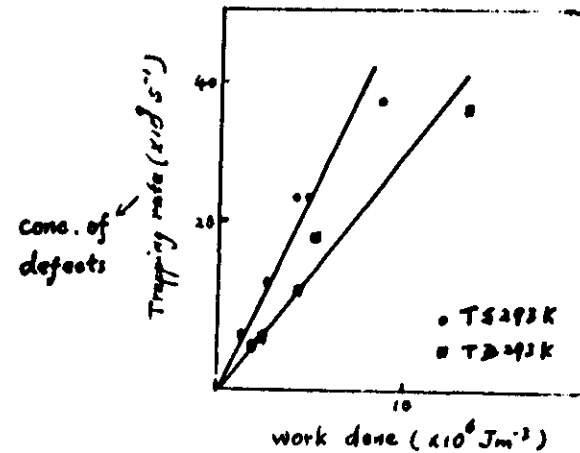
Fatigue:

- L. Diaz, R. Paroja et al. Proc. ICPA-7 (1985) 579.
 L. Diaz, R. Paroja et al. Phil. Mag. 51, 6 (1985) L61.
 S. J. Wang et al. Proc. ICPA-7 (1985) 467.

Wang et al: (Ni polycrystalline)

1. multi-exponential fitting method. (3)
2. τ_i : σ^* free lifetime.
3. τ_{21}, τ_{22} 209-255 $\rightarrow$ 340 ps — $\log N$ ($N: 10 \rightarrow 10^8$)
vacancy clusters
4. τ_{1A}, τ_{1B} saturation value ~ 168 ps
dislocation loop/vacancy
5. $\tau_3 = \tau_{2A}' = \tau_{2B}$ due to specimen surface.

Diaz et al.



Nanocrystalline iron

J.H.E. Schaffer and R. Würschinger, 1986
(Preprint)

Grain crystallite sizes: 5-10 nm (0.005-0.01 μm)

1. volume fraction of interfacial structure

20-50 % (3-10 nm)

2. τ_1 is similar to the lifetime $\tau_{iv} = 175$ ps
observed as at monovacancies in bulk iron.

3. τ_1 persists up to temperature $T = 380^\circ\text{C}$
(not recover below this temperature)

4. τ_2 is attributed to positron trapping at
microvoids at the intersection of several
crystalline interfaces. (τ_2 also persists up
to temperature $T = 320^\circ\text{C}$)

1. What are the limits ?

2. What are the merits and demerits ?

3. Where does PA stand in relation to
other techniques ?

Pls refer:

« Positron Annihilation » Proc. ICPA-7.

1985, p. 437. (SMA/208-2)