

Determination of absolute defect concentrations for saturated positron trapping - polycrystalline Ni as a case study

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Introduction

- In case of saturated positron trapping: conventional lifetime / Doppler does not give any information on defect densities any more.
- However, backdiffusion experiments using monoenergetic positron beams do not have the limitation of saturated positron trapping.
- Positrons diffusing back to the surface can detect very high defect densities.

Requirements and Limitations

- The defect should be homogenously distributed over the scan volume of the positron beam.
- There should not be any internal electric fields.

Possible Fields of Application

- Metallic alloys and powders
- Age-hardenable Al alloys etc.

Basics of the Measurement

- The total trapping rate can be determined from the positron diffusion length L_+ (see Ref. [1])

$$\kappa_{total} = \frac{1}{\tau_b} \left(\left(\frac{L_{ref}}{L} \right)^2 - 1 \right) \quad (1)$$

- In case the trapping is caused by several types of defects, positron lifetime can be used to get the fraction of trapping for each individual defect. The following example is valid for saturated trapping and the presence of voids and dislocations:

$$\begin{aligned} \tau_{ave} &= I_1 \tau_1 + I_2 \tau_2 \\ \kappa_{total} &= \kappa_{disl} + \kappa_{voids} \\ \kappa_{disl} &= I_1 \kappa_{total}; \quad \kappa_{voids} = I_2 \kappa_{total} \\ C_{disl} &= \frac{\kappa_{disl}}{\mu_{disl}}; \quad C_{voids} = \frac{\kappa_{voids}}{\mu_{voids}} \end{aligned} \quad (2)$$

Combining (1) and (2):

$$C_{disl} = \frac{I_1}{\mu_{disl} \tau_b} \left(\left(\frac{L_{ref}}{L} \right)^2 - 1 \right)$$

Sample Conditions

- Polycrystalline nickel samples of 4 different material classes were prepared (method of production denoted by the first number in sample number).
- The samples were exposed to cyclic loading at room temperature (second number) with constant plastic strain amplitudes ϵ_{pa}

Production method	Abbreviation	Description
1	BMCMF	multi-forging compaction procedure at room temperature
2	RTECAP	equal channel angular pressing
3	ETECAP	equal channel angular pressing
4	R	material 2 + heat treatment at 1230 K for 2 min (recrystallization)

- The dislocation density was obtained by Bragg diffraction with synchrotron radiation at ESRF Grenoble (for further details see [2]).

Sample	Production method	Deformation	Mean grain size d (nm)	Dislocation density (10^{10} cm^{-2})
Ni1-0	BMCMF	Initial	50	9.3 ± 0.8
Ni2-0	RTECAP	Initial	500	10.0 ± 0.9
Ni2-1	RTECAP	$\epsilon_{pa} = 2.5 \times 10^{-4}$	600	3.2 ± 0.3
Ni2-2	RTECAP	$\epsilon_{pa} = 2.0 \times 10^{-3}$	700	2.4 ± 0.2
Ni3-0	ETECAP	Initial	800	6.2 ± 0.5
Ni3-1	ETECAP	$\epsilon_{pa} = 2.5 \times 10^{-4}$	2000	2.7 ± 0.2
Ni3-2	ETECAP	$\epsilon_{pa} = 2.0 \times 10^{-3}$	2000	1.6 ± 0.1
Ni4-0	R	Initial	2500	0.1 ± 0.1
Ni4-1	R	$\epsilon_{pa} = 2.5 \times 10^{-4}$	2500	0.6 ± 0.1
Ni4-2	R	$\epsilon_{pa} = 2.0 \times 10^{-3}$	2500	0.8 ± 0.1

Positron Experiments

- Positron lifetime spectra were measured at room temperature. Almost all samples showed a two-component spectrum with both lifetimes clearly larger than bulk (for detailed results see the tables).
- A lifetime shorter than bulk could not be found. Thus positron trapping is almost complete.
- Small voids and dislocations are the dominating positron traps.
- The positron diffusion length L_+ was obtained by positron back-diffusion measurements using a continuous monoenergetic positron beam (Fig. 1).
- The $S(E)$ and $W(E)$ profiles were analyzed by VEPFIT separately, and the two values obtained L_w and L_s were averaged.
- From the diffusion length L_+ the total trapping rate K_{total} and the fraction of trapped positrons η was derived using Eq. 1.
- Using the lifetime decomposition, the trapping rate of the dislocations was calculated according to Eq. 2 (third line).
- Finally, the positron trapping rate of dislocations was plotted versus the dislocation density obtained by X-ray analysis (Fig. 2).

Sample	S	S/Sb	W	W/Wb	Ls	Lw	(Ls+Lw)/2	K total	eta
Ni1-0	0.44256	1.12995	0.050209	0.61331	15.93	16.12	16.03	$4.94E+11$	$9.81E-01$
Ni2-0	0.43010	1.09811	0.06227	0.78068	16.8	17.78	17.29	$4.23E+11$	$9.78E-01$
Ni3-0	0.42704	1.09032	0.063269	0.77288	24.17	25.52	24.85	$2.00E+11$	$9.55E-01$
Ni2-1	0.42497	1.08502	0.063993	0.78173	23.94	26.25	25.10	$1.96E+11$	$9.54E-01$
Ni2-2	0.42120	1.09073	0.063091	0.77071	24.69	25.82	25.26	$1.93E+11$	$9.53E-01$
Ni3-2	0.43369	1.10728	0.060526	0.73940	25.55	28.97	27.26	$1.64E+11$	$9.45E-01$
Ni3-1	0.43002	1.09792	0.062054	0.75804	32.39	38.26	35.33	$9.41E+10$	$9.08E-01$
Ni4-2	0.43296	1.10543	0.061184	0.74741	37.83	38.82	38.33	$7.85E+10$	$8.92E-01$
Ni4-1	0.42667	1.08937	0.063913	0.78075	45.81	50.35	48.08	$4.84E+10$	$8.30E-01$
Ni4-0	0.39809	1.01631	0.078521	0.85922	115	117.37	116.19	0.0	0.0
SC	0.39161	1.00000	0.08186	1.00000	110.99	122.04	116.50	0.0	0.0

Sample	L (nm)	eta	K total	Lav	tau1	I1	tau2	K disl
Ni1-0	16.03	0.981	$4.94E+11$	227.3	180.0	67.9	328	$3.32E+13$
Ni2-0	17.29	0.978	$4.23E+11$	160.6	158.0	98.4	350	$4.10E+13$
Ni3-0	24.85	0.955	$2.00E+11$	156.9	153.0	98.5	405	$1.90E+13$
Ni2-1	25.10	0.954	$1.96E+11$	161.8	154.0	98	341	$1.82E+13$
Ni2-2	25.26	0.953	$1.93E+11$	167	155.0	93.2	334	$1.74E+13$
Ni3-2	27.26	0.945	$1.64E+11$	180.1	161.0	89.9	350	$1.42E+13$
Ni3-1	35.33	0.908	$9.41E+10$	162.4	156.0	93.1	255	$8.16E+12$
Ni4-2	38.33	0.892	$7.85E+10$	171.1	156.0	85.2	259	$6.14E+12$
Ni4-1	48.08	0.830	$4.84E+10$	162.1	139.0	86.1	208	$2.59E+12$
Ni4-0	116.19	0.005	$5.17E+07$	109.3	103.0	93.7	209	0.0
SC	116.50							

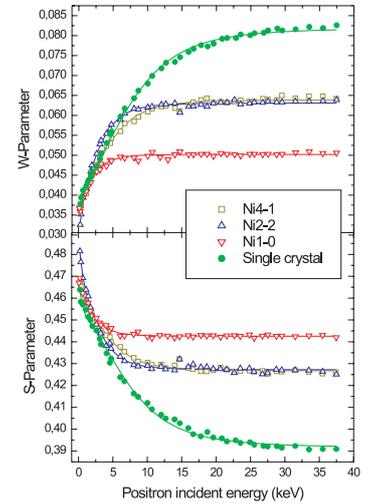


Fig. 1: Some examples of $S(E)$ and $W(E)$ plots obtained by a slow-positron beam system. The data were used to calculate the positron diffusion length L_+ .

Result

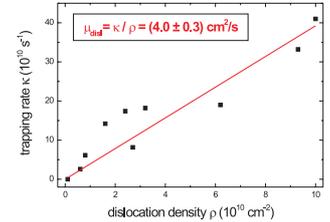


Fig. 2: Plot of the positron trapping rate versus dislocation density. The slope of the curve was obtained by linear regression. It represents the positron trapping coefficient.

As the final result, the positron trapping coefficient for trapping in dislocations in Ni for high dislocation densities is obtained to be $\mu_{disl} = 4.0 \pm 0.3 \text{ cm}^2/\text{s}$.

References

- [1] R. Krause-Rehberg, H.S. Leipner, "Positron Annihilation in Semiconductors", Vol. 127 of Series "Solid-State Sciences", Springer-Verlag, Berlin 1999
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- [3] P. Hautajärvi (Ed.), "Positrons in Solids", Springer-Verlag, Berlin 1979

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