Institute of Electronic Materials Technology

Joint Laboratory for Characterisation
of Defect Centres in Semi-Insulating Materials

Annealing induced evolution of defect centres in MCz silicon irradiated with a neutron fluence of $1x10^{16}$ cm^{-2}

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Outline

- Samples – annealing conditions
- HRPITS images of spectral fringes for radiation defects in neutron-irradiated MCz silicon – effect of annealing on defect structure of material irradiated with a fluence of 1×10^{16} cm^{-2}
- Photoluminescence results – defect model for the W-line
- Conclusions

Samples

Starting material:

Okmetic MCz <100> silicon wafers, *n*-type, 1 kΩcm, 300 μm thick

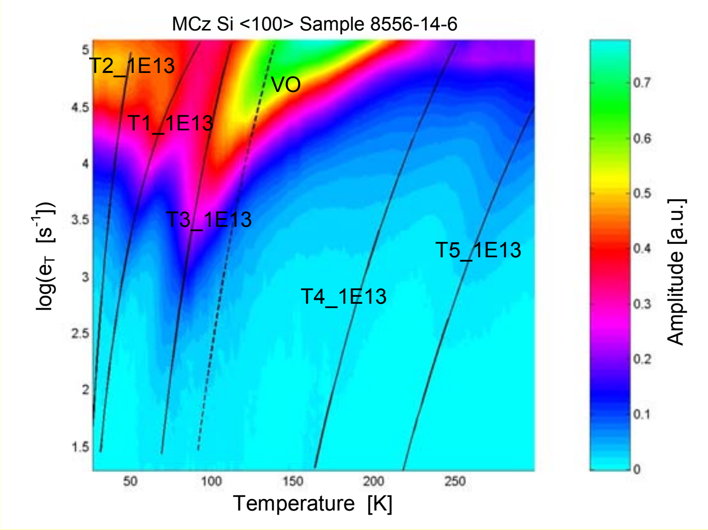
[O] = $5.5\text{x}10^{17}$ cm^{-3}

[C] = $2.5\text{x}10^{15}$ cm^{-3}

Neutron irradiation:

TRIGA reactor in Ljubljana, 1-MeV, fluences: $1\text{x}10^{12}$, $1\text{x}10^{13}$, $1\text{x}10^{14}$, $3\text{x}10^{14}$, $1\text{x}10^{15}$, $3\text{x}10^{15}$, **$1\text{x}10^{16}$**, and $3\text{x}10^{16}$ cm^{-2}

Effect of neutron fluence on defect structure of as-irradiated material

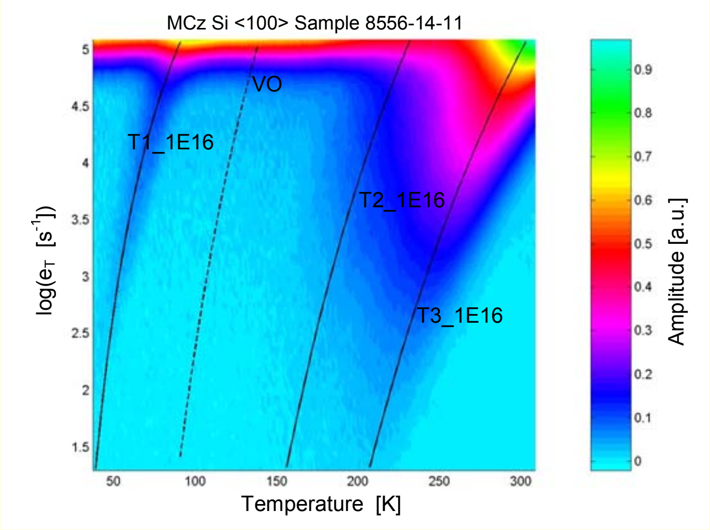


ρ = ~4.0x10^4 Ωcm

1-MeV neutron fluence 1x10^{13} cm^{-2}

Trap label	E_a[meV]	A [$s^{-1}K^{-2}$]	Tentative Identification
T1_1E13	25±2	(2-5)x10^2	shallow donor
T2_1E13	30±2	(3-6)x10^4	shallow donor
T3_1E13	115±5	(8-20)x10^5	$C_iC_s(B)^{-/0}$ or self-interstitials related
T4_1E13	315±10	(2-4)x10^6	$V_2^{2-/-} + C_iO_i^{+/0}$
T5_1E13	470±20	(1-5)x10^7	$V_2^{-/0} + V_2O^{-/0}$

VO: E_a= 0.17 eV; A= 9.6x10^6 s^{-1}K^{-2}



1-MeV neutron fluence 1x10^{16} cm^{-2}

Trap label	E_a [meV]	A [$s^{-1}K^{-2}$]	Identification
T1_1E16	41±3	(1-3)x10^3	self-interstitials, mono-interstitials (*I*)
T2_1E16	325±10	(1-5)x10^7	$V_2^{2-/-} + C_iO_i^{+/0}$
T3_1E16	455±15	(1-5)x10^7	$V_2^{-/0} + V_2O^{-/0}$ + X (vacancy aggregates)

ρ = ~5.0x10^5 Ωcm, decrease in charge carriers lifetime at 250 K by two orders of magnitude

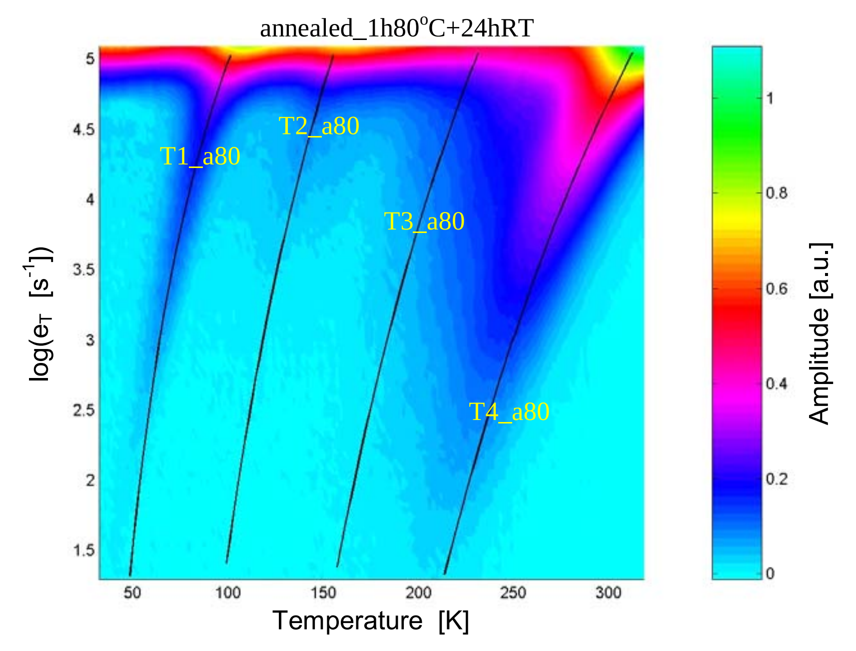
Annealing stages

HT1: 1h, 80 ^{o}C + 24 h, RT

HT2: (1h, 80 ^{o}C + 24 h, RT) + 1h, 160 ^{o}C + 24 h, RT

HT3: (1h, 80 ^{o}C + 24 h, RT + 1h, 160 ^{o}C + 24 h, RT) + 1h, 240 ^{o}C + 24 h, RT

HRPITS image after annealing HT1 (1h, 80 °C + 24 h, RT)

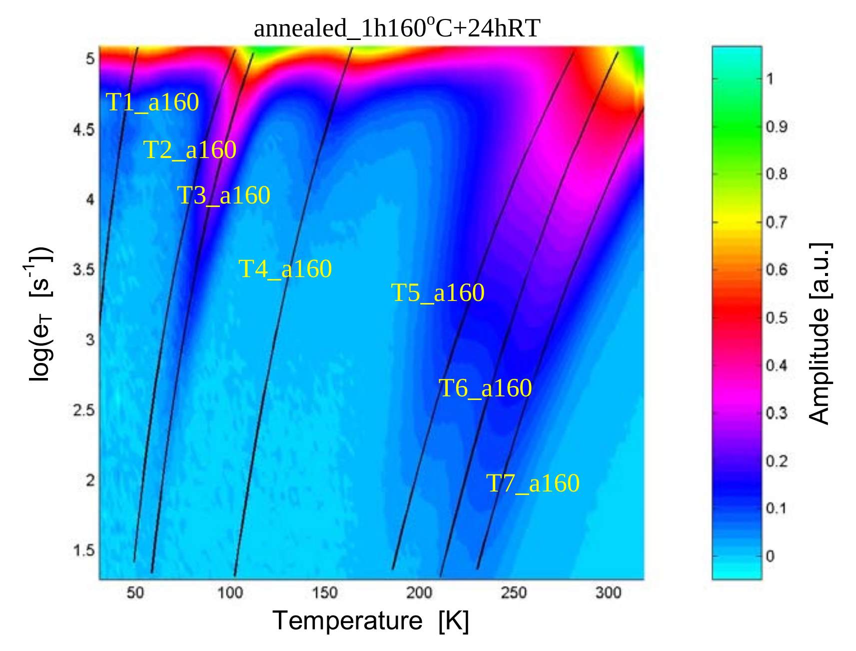


Laser: 650 nm, 5mW;
U_A= 20 V;
Gain: $1x10^7$ V/A;
Line width [samples]: 50000;
Time Resolution [μs]: 5
Period [ms]: 263; Average: 500;
Illumination pulse width: 50 ms

ρ = ~$6.0x10^5$ Ωcm
τ/τ (as-irr.) = 0.66
E_{TDDC} = 428 meV

Trap label	Ea [meV]	A $[s^{-1}K^{-2}]$	e_1 $[s^{-1}]$	Amp_{e1} [a.u]	e_2 $[s^{-1}]$	Amp_{e2} [a.u]	Tentative identification
T1_a80	55±5	$(5-7)x10^3$	$1x10^3$	0.042	$3.2x10^4$	0.188	di-interstitials (I_2)
T2_a80	175±5	$(2-3)x10^6$	$1x10^3$	0.01	$3.2x10^4$	0.1	$VO^{-/0}$+ $V_2O^{2-/-}$
T3_a80	325±10	$(1-5)x10^7$	$1x10^3$	0.03	$3.2x10^4$	0.13	$V_2^{2-/-}$ + $C_iO_i\ ^{+/0}$
T4_a80	455±15	$(1-5)x10^7$	$1x10^3$	0.14	$3.2x10^4$	0.43	$V_2^{-/0}$ + $V_2O^{-/0}$ + X (vacancy aggregates)

HRPITS image after annealing HT2
(1h, 80 °C + 24 h, RT) + (1h, 160 °C + 24 h, RT)

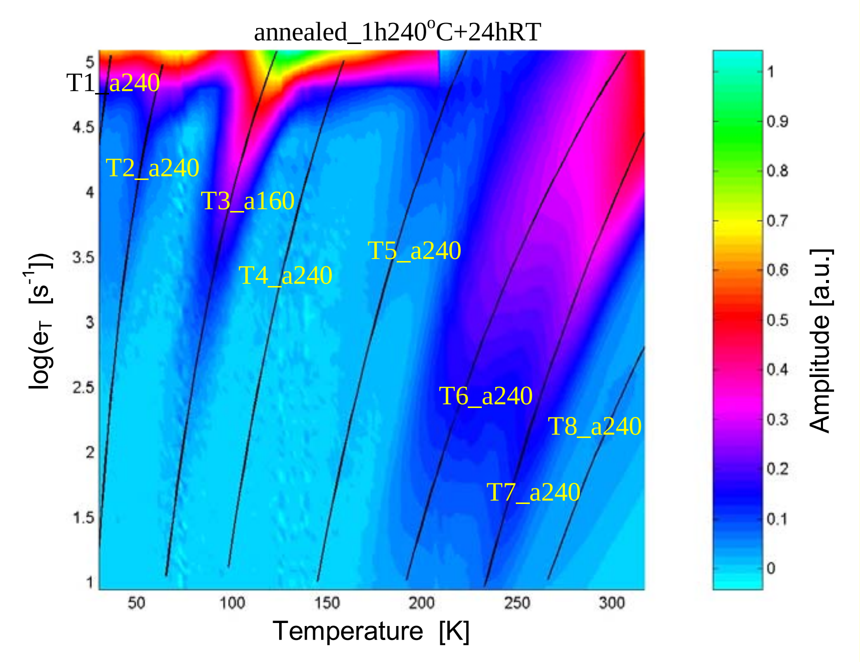


Laser: 650 nm, 5mW;
U_A= 20 V;
Gain: 1x10^7 V/A;
Line width [samples]: 50000;
Time Resolution [µs]: 5
Period [ms]: 263; Average: 500;
Illumination pulse width: 50 ms

ρ = ~2.5x10^5 Ωcm
τ/τ (as-irr.) = 2.41
E_{TDDC} = 400 meV

Trap label	Ea [meV]	A [$s^{-1}K^{-2}$]	e_1 [s^{-1}]	Amp_{e1} [a.u]	e_2 [s^{-1}]	Amp_{e2} [a.u]	Tentative identification
T1_a160	25±2	(1-2)x10^4	3.2x10^3	0.014	3.2x10^4	0.09	shallow donor
T2_a160	55±5	(5-7)x10^3	1x10^3	0.027	3.2x10^4	0.16	di-interstitials (I_2)
T3_a160	75±5	(2-4)x10^4	1x10^3	0.08	3.2x10^4	0.35	tri-interstitials (I_3)
T4_a160	180±5	(9-20)x10^5	1x10^3	0.01	3.2x10^4	0.11	$VO^{-/0}$ + $V_2O^{2-/-}$
T5_a160	360±10	(3-5)x10^6	1x10^3	0.12	3.2x10^4	0.3	$C_iO_i^{+/0}$
T6_a160	465±15	(2-5)x10^7	1x10^3	0.15	3.2x10^4	0.4	$V_2^{-/0}$ + $V_2O^{-/0}$
T7_a160	500±15	(5-7)x10^7	1x10^3	0.14	3.2x10^4	0.43	EX3, TH6

HRPITS image after annealing HT3
(1h, 80 °C + 24 h, RT) + (1h, 160 °C + 24 h, RT) + (1h, 240 °C + 24 h, RT)

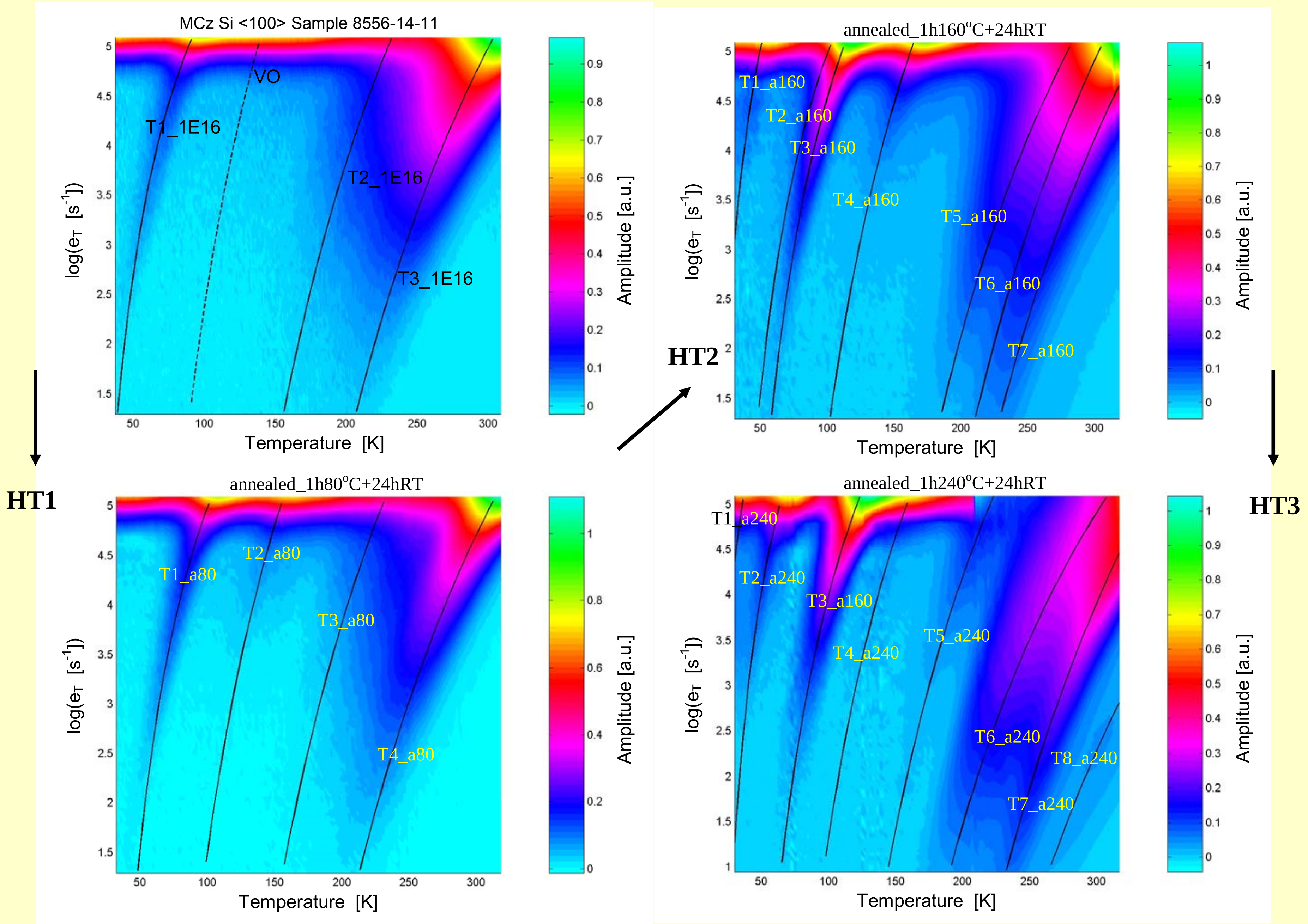


Laser: 650 nm, 5mW;
U_A= 20 V;
Gain: 1x10^7 V/A (20-210 K) and
1x10^6 V/A (210-320 K) ;
Line width [samples]: 50000;
Time Resolution [μs]: 10
Period [ms]: 526; Average: 500;
Illumination pulse width: 50 ms

ρ = ~5.0x10^4 Ωcm
τ/τ (as-irr.) = 7.94
E_{TDDC} = 394 meV

Trap label	Ea [meV]	A [$s^{-1}K^{-2}$]	e_1 [s^{-1}]	Amp_{e1} [a.u]	e_2 [s^{-1}]	Amp_{e2} [a.u]	Tentative identification
T1_a240	18±2	(2-4)x10^4	-	-	3.2x10^4	0.06	shallow donor
T2_a240	35±5	(1-2)x10^4	1x10^3	0.018	3.2x10^4	0.15	shallow donor
T3_a240	95±5	(5-8)x10^4	1x10^3	0.09	3.2x10^4	0.42	I_2+ I_3 + tetra-interstitials (I_4)
T4_a240	177±5	(1-3)x10^6	1x10^3	0.01	3.2x10^4	0.047	$VO^{-/0}$
T5_a240	300±10	(1-3)x10^7	1x10^3	0.027	3.2x10^4	0.11	C_i, C_i-I?
T6_a240	370±15	(1-3)x10^6	1x10^3	0.21	3.2x10^4	0.31	$C_iO_i^{0/+}$
T7_a240	540±15	(2-3)x10^6	1x10^3	0.25	3.2x10^4	0.49	I level, E_c – 0.545 eV, V_3
T8_a240	560±15	(2-3)x10^8	1x10^2	0.036	2.5x10^2	0.042	E_v+0.58 eV (?)

Summary of HRPITS images after each step of annealing



MCz Si <100> Sample 8556-14-11

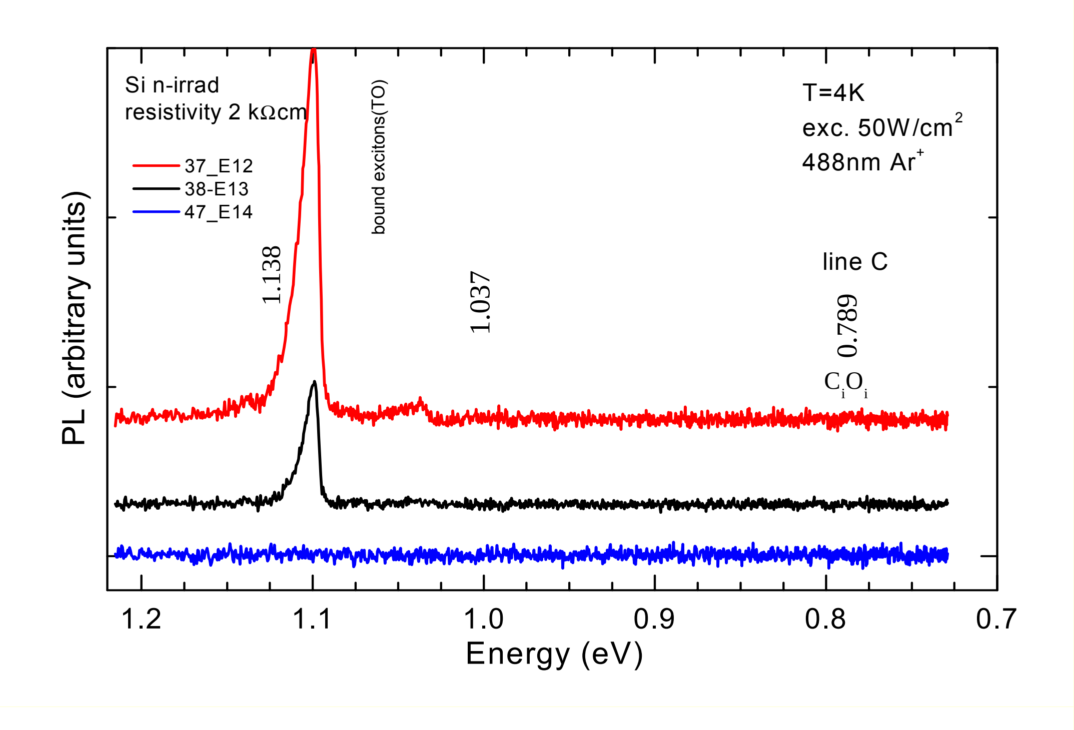
annealed_1h80°C+24hRT

annealed_1h160°C+24hRT

annealed_1h240°C+24hRT

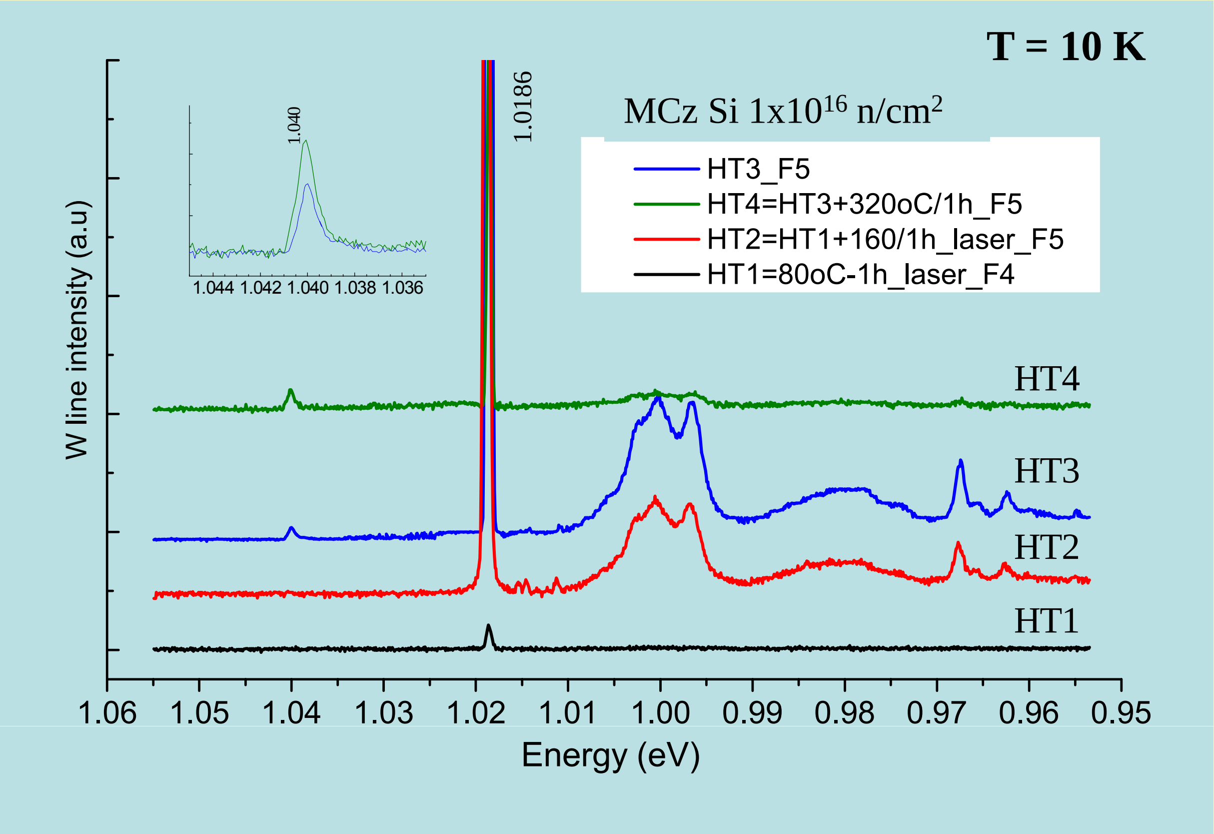
Photoluminescence results

The PL measurements were done using Ar+ laser operated at 488 nm focused to a spot of about 400 micrometers. The luminescence at 4.0-4.3 K was detected using lock-in technique and Hamamatsu photomultiplier type R5509-72 with InGaAsP cathode. The spatial resolution was 1.5 nm for 1 µm. The samples were placed on a cool finger and cooled down by using the closed-cycle cooling system.



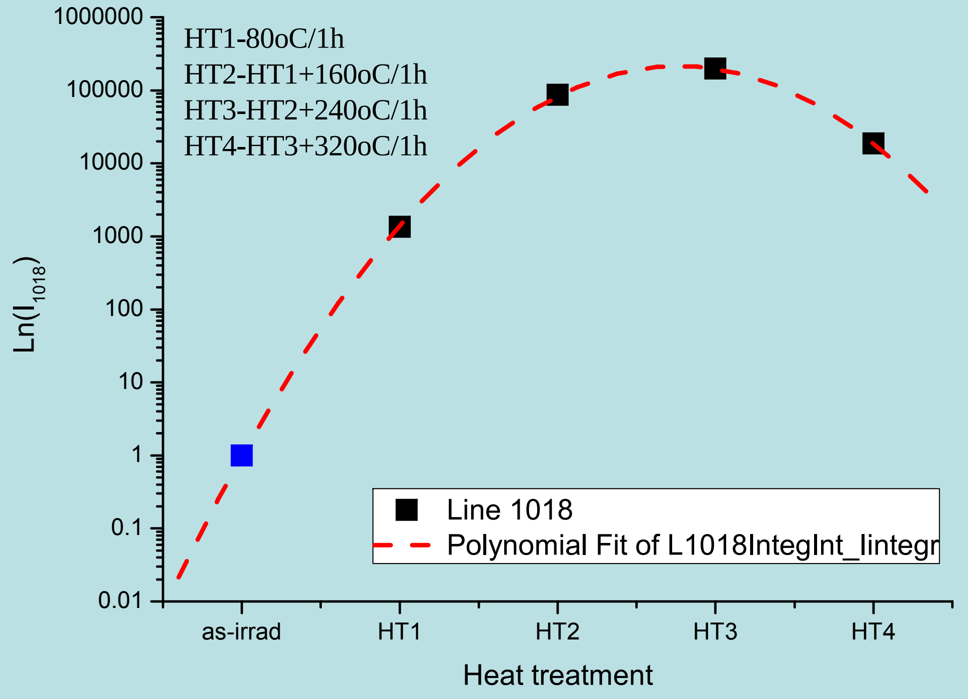
Comparison of photoluminescence spectra for MCz Si samples irradiated with neutron fluences of 1x10^{12}, 1x10^{13} and 1x10^{14} cm^{-2}. The decrease in the radiative recombination efficiency with increasing the fluence is seen.

Photoluminescence results



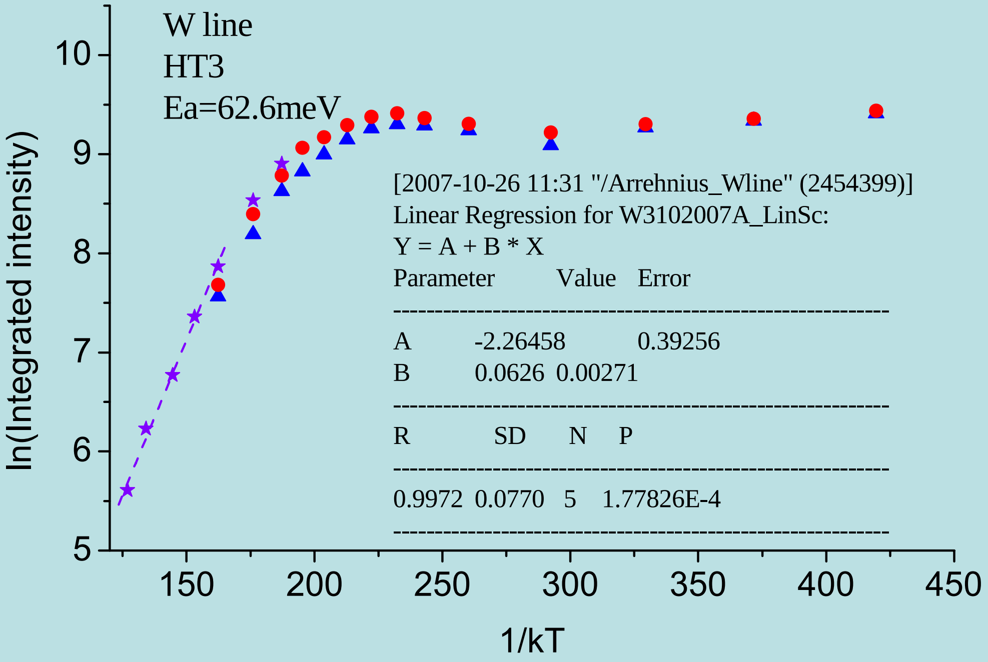
PL spectra taken after each annealing stage for a sample of 1kΩcm MCz-Si exposed to 1-MeV neutron irradiation with a fluence of 1x10^{16} cm^{-2}. A strong increase in the intensity of the *W* line (at 1018 meV) with the rise of the annealing temperature is seen.

Photoluminescence results



Intensity of the luminescence from the *W* line (at 1018 meV), measured at 10 K, as a function annealing temperature. The *W* line is increasing due to restructuring within small clusters.

Photoluminescence results – Defect model for the W line



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[2007-10-26 11:31 "/Arrehnius_Wline" (2454399)]
Linear Regression for W3102007A_LinSc:
Y = A + B * X
Parameter        Value   Error
------------------------------------------------------------
A         -2.26458          0.39256
B         0.0626  0.00271
------------------------------------------------------------
R            SD       N     P
------------------------------------------------------------
0.9972  0.0770   5    1.77826E-4
------------------------------------------------------------
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Main facts

- not affected by the impurities
- occurs after annealing at ~ 100C
- interstitial related
- detailed structure and defect interactions still discussed

Minoru Nakamura and Siro Nagai
PHYSICAL REVIEW B **66**, 155204 (2002)

Gordon Davis *et al.*
PHYSICAL REVIEW B **73**, 165202 (2006)

Temperature dependence of the *W* line (at 1018 meV) intensity. The straight line due to the thermal emission of charge carriers is obtained in the temperature range 50 – 100 K. In the same range the HRPITS fringes assigned to self-interstitials are observed. The activation energy of the *W* center 62.6 meV is close to the value of 55 meV determined for defect centers T1_a80 and T2_a160 attributed to di-interstitials (I_2).

Conclusions

- High-resolution photoinduced transient spectroscopy (HRPITS) with implementation of the imaging procedure and photoluminescence (PL) measurements have been used to studying the annealing-induced evolution of defect centers in 1-kΩcm MCz-Si exposed to irradiation of 1-MeV neutrons with a fluence of $1x10^{16}$ cm^{-2}.

- After the first stage of heat treatment at 80 ^{o}C for 1h, the activation energy of shallow defects increased from 41 to 55 meV. This change has been proposed to be due to the formation of di-interstitials (I_2). The annealing also resulted in narrowing the broad HRPITS band related to vacancy clusters introducing nearly midgap levels and appearing the VO center.

- As a result of the second stage of isochronal annealing at 160 ^{o}C, the 25-meV shallow donor arose and besides the 55-meV level of di-interstitials, the 75-meV level assigned to tri-interstitials (I_3) occurred. On the other hand, the 360-meV level corresponding to C_iO_i complex and the 500-meV level of an unknown defect were well resolved.

- After the third stage of annealing at 240 ^{o}C, the 18-meV and 35-meV shallow donors and the 95-meV level assigned to tetra-interstitials (I_4) were detected. On the other hand, the defect centers related to divacancies disappeared and two midgap centers with the activation energies of 540 meV and 560 meV were revealed.

- The activation energy of the *W* line in the PL spectra was found to be ~ 63 meV and is close to that of 55 meV determined from HRPITS measurements for the defect center attributed to di-interstitials. This result indicates that the same defect is observed by the both methods and supports the defect model assigning the *W* center to di-interstitials.

Acknowledgement

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