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CENTRE D'ÉTUDE DE L'ÉNERGIE NUCLÉAIRE

IRRADIATION BEHAVIOUR OF THREE CANDIDATE STRUCTURAL MATERIALS FOR ADS SYSYEMS: EM10, T91 and HT9 (F/M STEELS)

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Introduction: why F/M steels?

- Typical requirements for structural materials in ADS systems
  - Reproducible fabrication, workability and weldability
  - Heat resistance (limited decrease of strength and toughness)
  - Dimensional stability (limited irradiation swelling/creep)
  - Mechanical resistance (ductility and toughness) under irradiation in liquid metal
  - Corrosion resistance in Pb-Bi
  - Compatibility with Pb-Bi (resistance to LME – Liquid Metal Embrittlement)

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Window-design ADS Systems: foreseen service conditions

- Assuming that:
  - Maximum proton current density = 70 μA/cm²
  - One full calendar year of operation
- The following estimates were obtained from numerical computations:
  - Atomic displacements of the order of 100 dpa (window) and 50 dpa (container structure of the target)
  - Production of H (≈ 90000 appm), He (≈ 5000 appm) and other spallation elements (Ca, Ti, V, P, S)
  - Consequences on the mechanical properties of steels after irradiation:
    - Hardening (increase of tensile strength)
    - Embrittlement (degradation of toughness)

Considerations on the available materials

- Al alloys ➡ poor heat resistance and severe embrittlement
- Ni-based alloys ➡ high affinity to dissolve in Pb-Bi and microstructural instability under irradiation
- Zr alloys ➡ drastic loss of strength and ductility under high T irradiation, especially in the presence of H
- Austenitic steels ➡ very susceptible to irradiation-induced swelling and creep + poor corrosion resistance in liquid Pb
- Ferritic/Martensitic steels ➡ most promising candidates both for fuel cladding and structural applications; presently considered also for Fast and Fusion reactors

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The SPIRE Project
(5th European Framework Programme)

- Coordinated by CEA (F); running from 2001 to 2004
- Aimed at investigating mechanical and microstructural properties for F/M steels that are candidates for the spallation target window
- SCK•CEN contribution to Work Package 4 (*Neutron Irradiation and Post-Irradiation Examination*):
  - Irradiation at 200 °C up to 2 different doses of tensile, Charpy and fracture toughness specimens of conventional 9Cr and 12Cr steels in non-doped condition, in flowing water and no spectrum tailoring
  - Subsequent PIE

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The materials selected

- Materials: EM10, T91, HT9 (undoped)

	C	**Ni**	**Cr**	**Mo**	**Cu**	**Si**	**S**	**Al**	**Nb**	**Co**	**V**
EM10	0.099	0.07	8.97	1.06	0.05	0.46	<0.003	<0.016	<0.002	0.03	0.013
T91	0.099	0.24	8.8	0.96	0.05	0.32	0.004	<0.01	0.06	0.03	0.24
HT9	0.204	0.66	11.68	1.06		0.45	<0.003		0.03		0.29
	Ti	**N**	**P**	**Mn**	**O**	**B**	**W**	**Sn**	**As**	**Sb**	**Fe**
EM10	0.01	0.014	0.013	0.49	0.001	<0.001	<0.002	<0.005	0.003	0.01	bal.
T91	<0.005	0.03	0.02	0.43		<0.0005	<0.01	0.006	0.011	0.012	bal.
HT9			0.020	0.63			0.47				bal.

EM10 supplied by CEA
- Normalised at 990°C/50′
- Tempered at 750°C/60′

T91 provided by UGINE (heat 36224)
-Normalised at 1040°C/60′

-- Tempered at 760°C/60′

HT9 provided by Aubert&Duval (orig. denomination: 56 B.I.)
- Normalised at 1050°C/30′
- Tempered at 700°C/120′

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Irradiation in BR2 (MISTRAL rig)

Irradiation temperature:
T = 200 ± 5 °C
(32 thermocouples used)

1st batch: 6 cycles
(July 02 → July 03)
2nd batch: 8 cycles
(July 02 → Dec 03)
Fast flux: 3.5×10^{14} n/cm²·s

Test matrix

Material	**Test type**	**Specimen type**	**Or.**	**Condition**	**# of tests performed**
EM10	Tensile	Cylindrical D = 2.4 mm	T	Unirradiated	2
				Irradiated	1 (batch 1) 1 (batch 2)
	Charpy	KLST	T-L	Unirradiated	12
				Irradiated	12 (batch 1) 11 (batch 2)
	Toughness	PCCv	T-L	Unirradiated	10
		Precracked KLST		Irradiated	12 (batch 1) 12 (batch 2)
T91	Tensile	Cylindrical D = 2.4 mm	T	Irradiated	1 (batch 1) 1 (batch 2)
	Charpy	KLST	T-L	Irradiated	12 (batch 1) 12 (batch 2)
	Toughness	PCCv	T-L	Unirradiated	10
		Precracked KLST		Irradiated	12 (batch 1) 12 (batch 2)
HT9	Tensile	Cylindrical D = 2.4 mm	L	Irradiated	1 (batch 1) 1 (batch 2)
	Charpy	KLST	L-T	Irradiated	12 (batch 1) 12 (batch 2)
	Toughness	Precracked KLST	L-T	Irradiated	12 (batch 1) 12 (batch 2)

Additional tests, not included in the original test programme

Dosimetry

Material	Batch	Test type	Fluence (E > 1 MeV, n/cm^2)	Dose (dpa)
EM10	1	Tensile	1.96×10^{21}	2.93
		KLST	1.69×10^{21}	2.53
		PKLST	1.94×10^{21}	2.91
		All	**1.83×10^{21}**	**2.74**
	2	Tensile	2.91×10^{21}	4.36
		KLST	2.51×10^{21}	3.76
		PKLST	2.86×10^{21}	4.29
		All	**2.71×10^{21}**	**4.06**
T91	1	Tensile	1.94×10^{21}	2.91
		KLST	1.62×10^{21}	2.43
		PKLST	1.67×10^{21}	2.51
		All	**1.69×10^{21}**	**2.53**
	2	Tensile	2.91×10^{21}	4.36
		KLST	2.39×10^{21}	3.58
		PKLST	2.49×10^{21}	3.74
		All	**2.71×10^{21}**	**3.74**
HT9	1	Tensile	1.78×10^{21}	2.67
		KLST	1.65×10^{21}	2.47
		PKLST	1.67×10^{21}	2.51
		All	**1.67×10^{21}**	**2.51**
	2	Tensile	2.73×10^{21}	4.10
		KLST	2.47×10^{21}	3.70
		PKLST	2.48×10^{21}	3.71
		All	**2.50×10^{21}**	**3.75**

Batch 1 (overall)

Fluence = 1.73×10^{21} n/cm^2

Dose = 2.60 dpa

σ = 13%

Batch 2 (overall)

Fluence = 2.57×10^{21} n/cm^2

Dose = 3.86 dpa

σ = 12%

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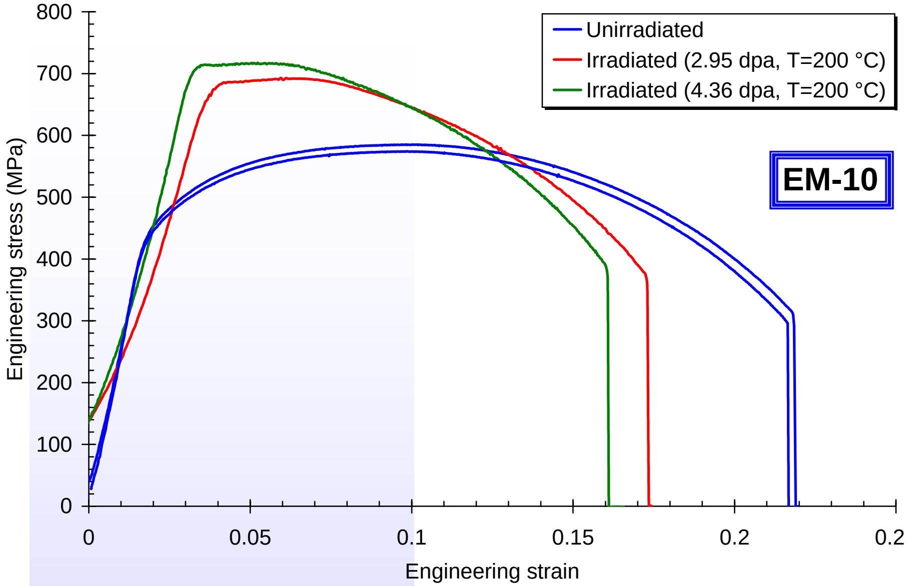
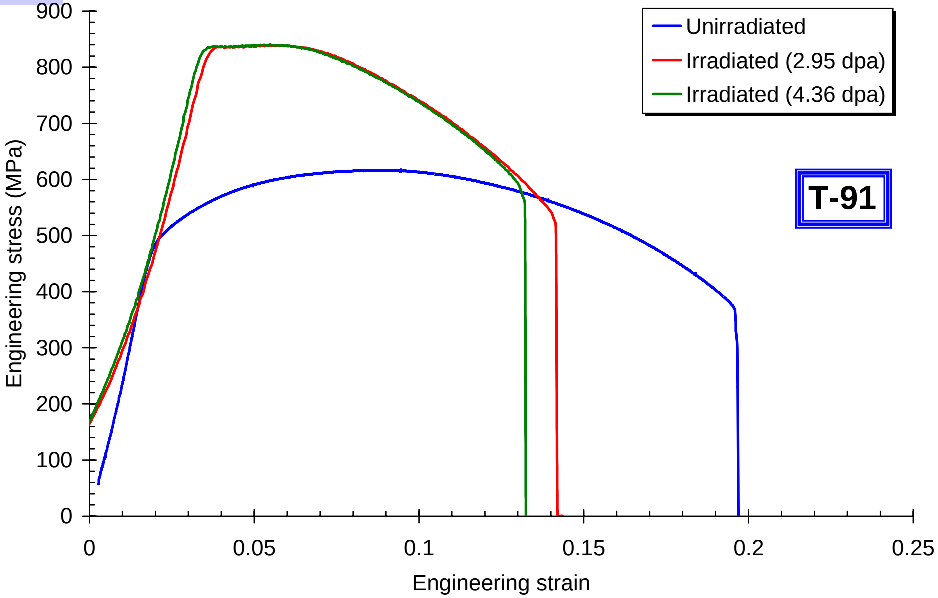
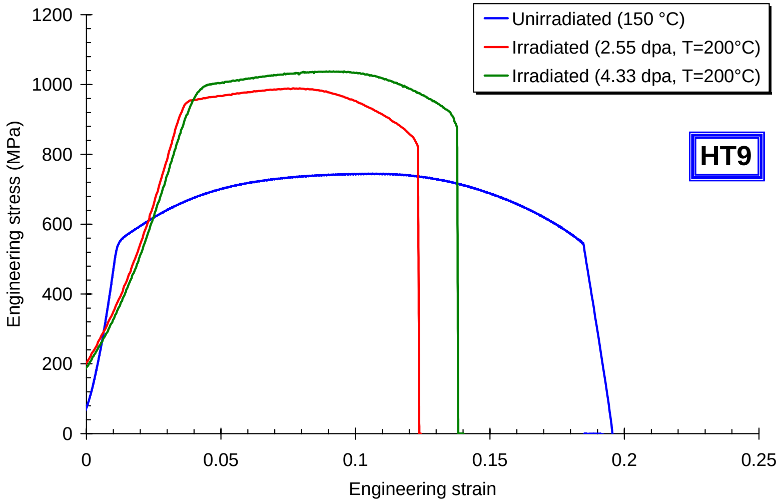
Tensile tests: comparison among stress-strain curves







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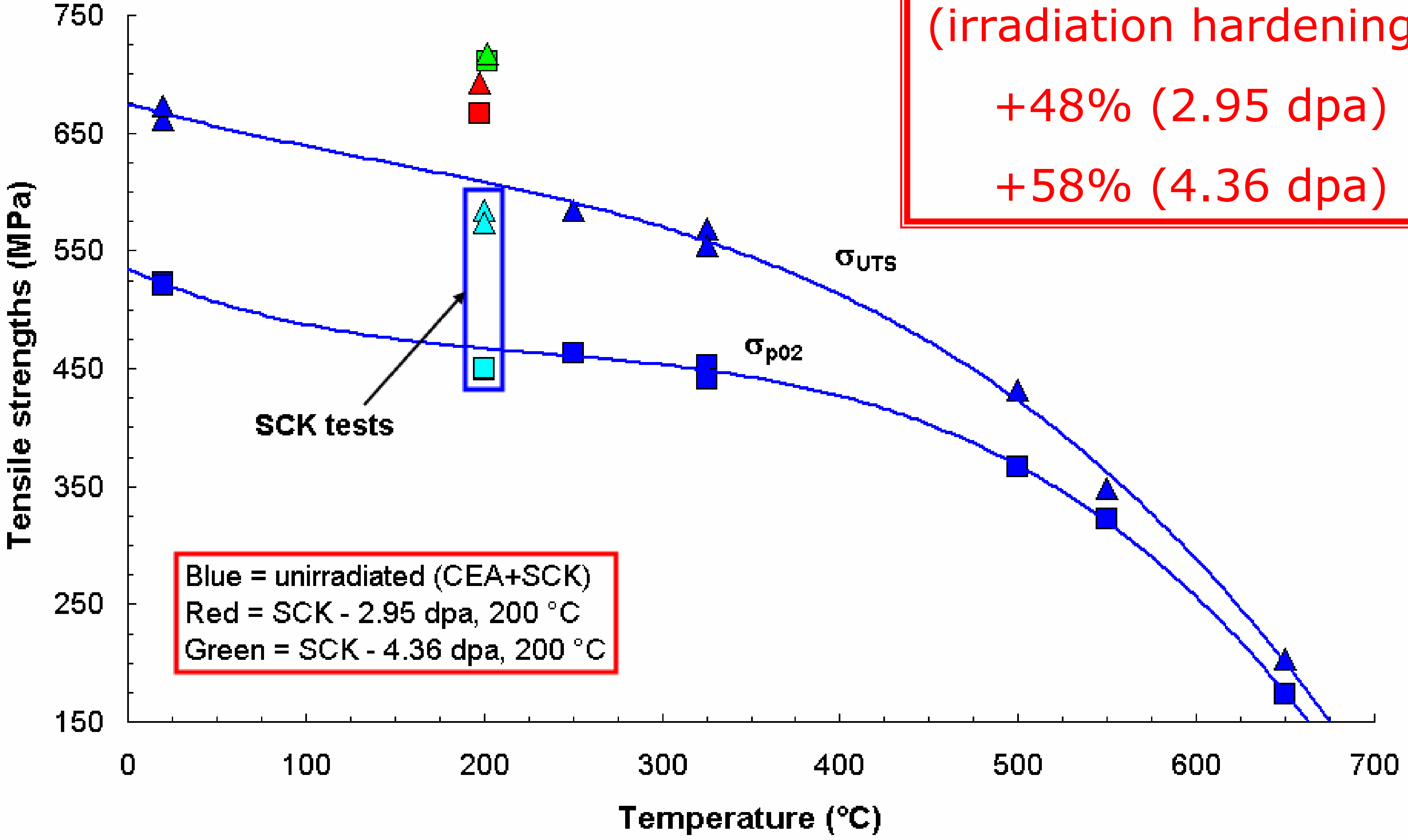
Tensile test results – EM10 (or.T)

Yield strength increase (irradiation hardening):

+48% (2.95 dpa)

+58% (4.36 dpa)



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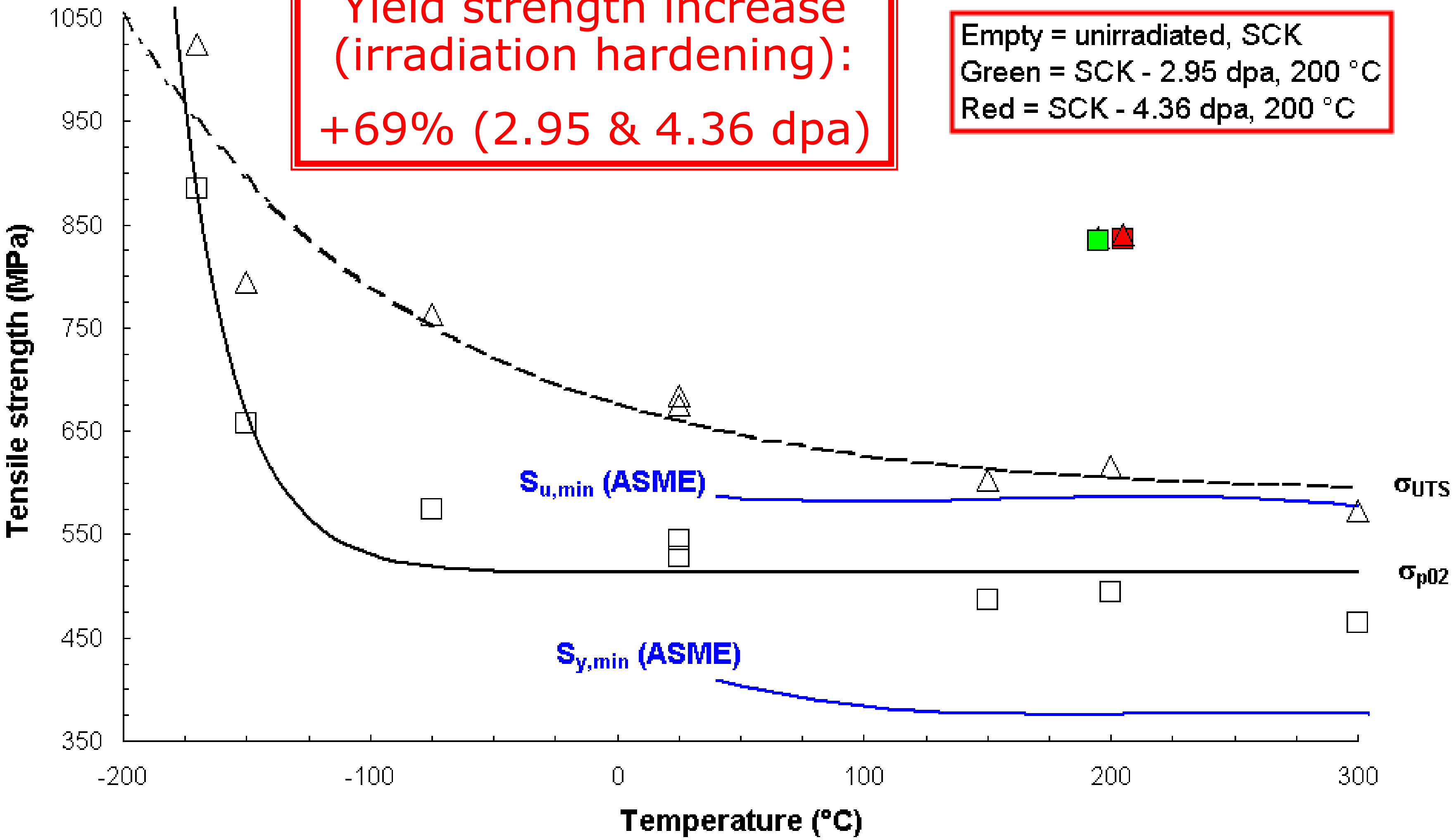
Tensile test results – T91 (or.T)



Yield strength increase (irradiation hardening):

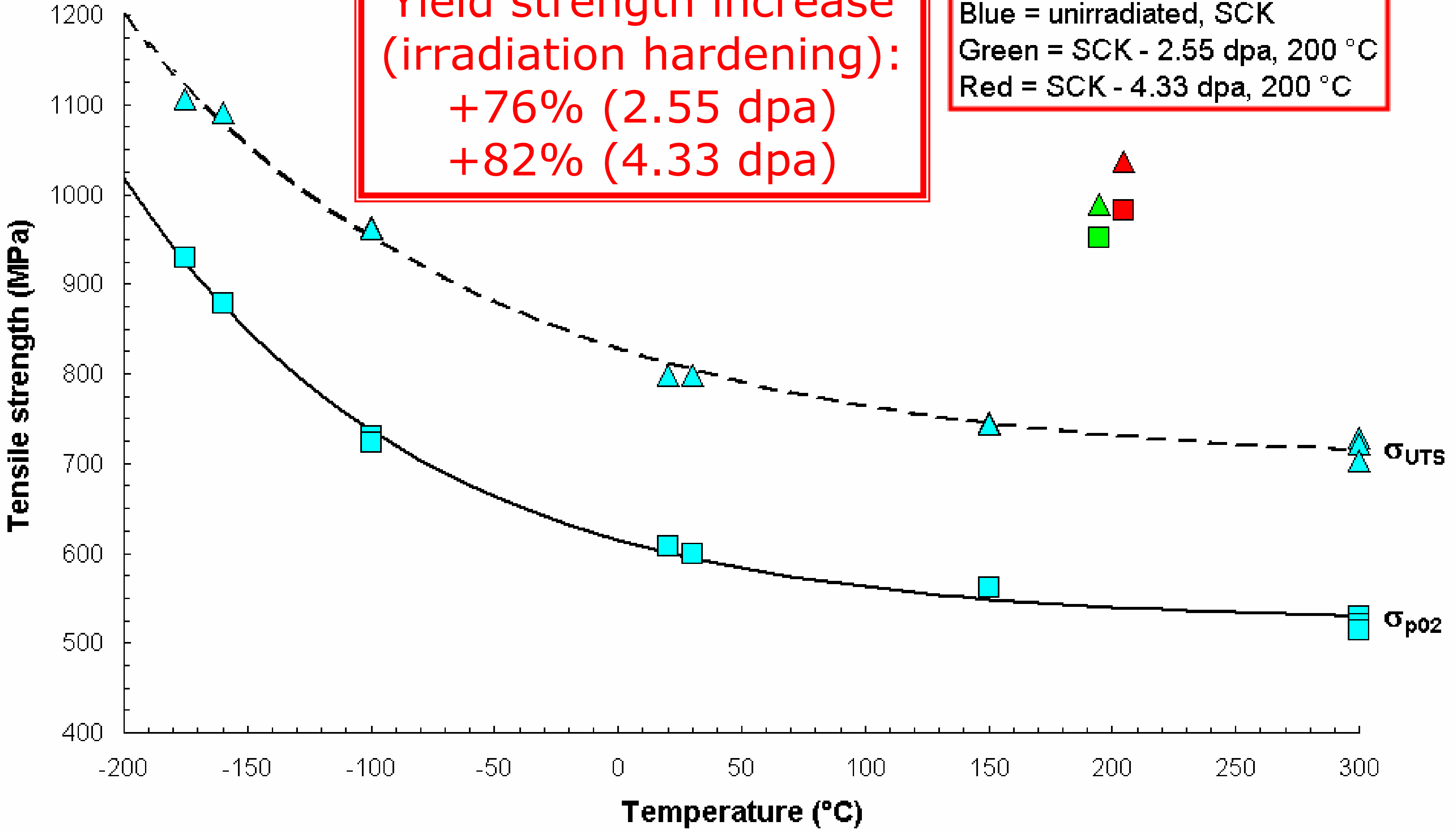
+69% (2.95 & 4.36 dpa)

Empty = unirradiated, SCK
Green = SCK - 2.95 dpa, 200 °C
Red = SCK - 4.36 dpa, 200 °C

Tensile test results – HT9 (or.L)

Yield strength increase (irradiation hardening):
+76% (2.55 dpa)
+82% (4.33 dpa)

Blue = unirradiated, SCK
Green = SCK - 2.55 dpa, 200 °C
Red = SCK - 4.33 dpa, 200 °C



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Tensile test results – Summary

12% steel more irradiation-sensitive than 9% (formation of α' phase)

In T91, presence of V and N promotes formation of small carbides which facilitate hardening

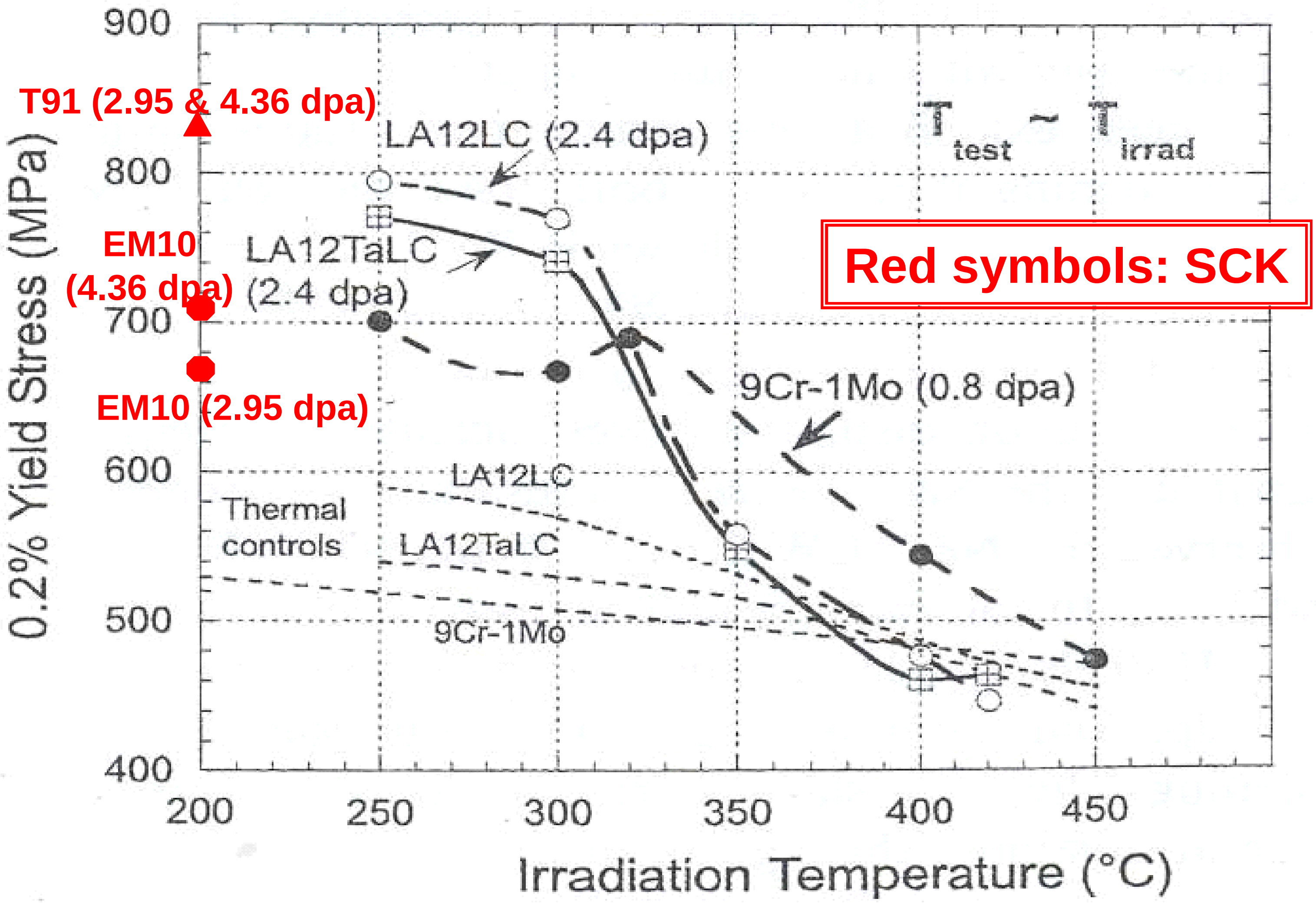
Material	Dose: 2.67-2.93 dpa					Dose: 4.10-4.36 dpa				
	$\Delta\sigma_y$ (%)	$\Delta\sigma_m$ (%)	$\Delta\varepsilon_u$ (%)	$\Delta\varepsilon_t$ (%)	ΔZ (%)	$\Delta\sigma_y$ (%)	$\Delta\sigma_m$ (%)	$\Delta\varepsilon_u$ (%)	$\Delta\varepsilon_t$ (%)	ΔZ (%)
EM10	+48	+19	-5.5	-5.5	-20.5	+58	+24	-5.5	-6.5	-2.5
T91	+69	+36	-4	-7	+7	+69	+36	-4	-8	-7
HT9	+76	+35	-6	-9	-27	+82	+42	-5	-8	-26

CEA data – 40 dpa, $T_{irr} = T_{test} = 325$ °C

Material	$\Delta\sigma_y$ (%)	$\Delta\sigma_m$ (%)
EM10	+130	+96
T91	+164	+119

Where is the saturation?

Tensile test results
Comparison with other F/M steels

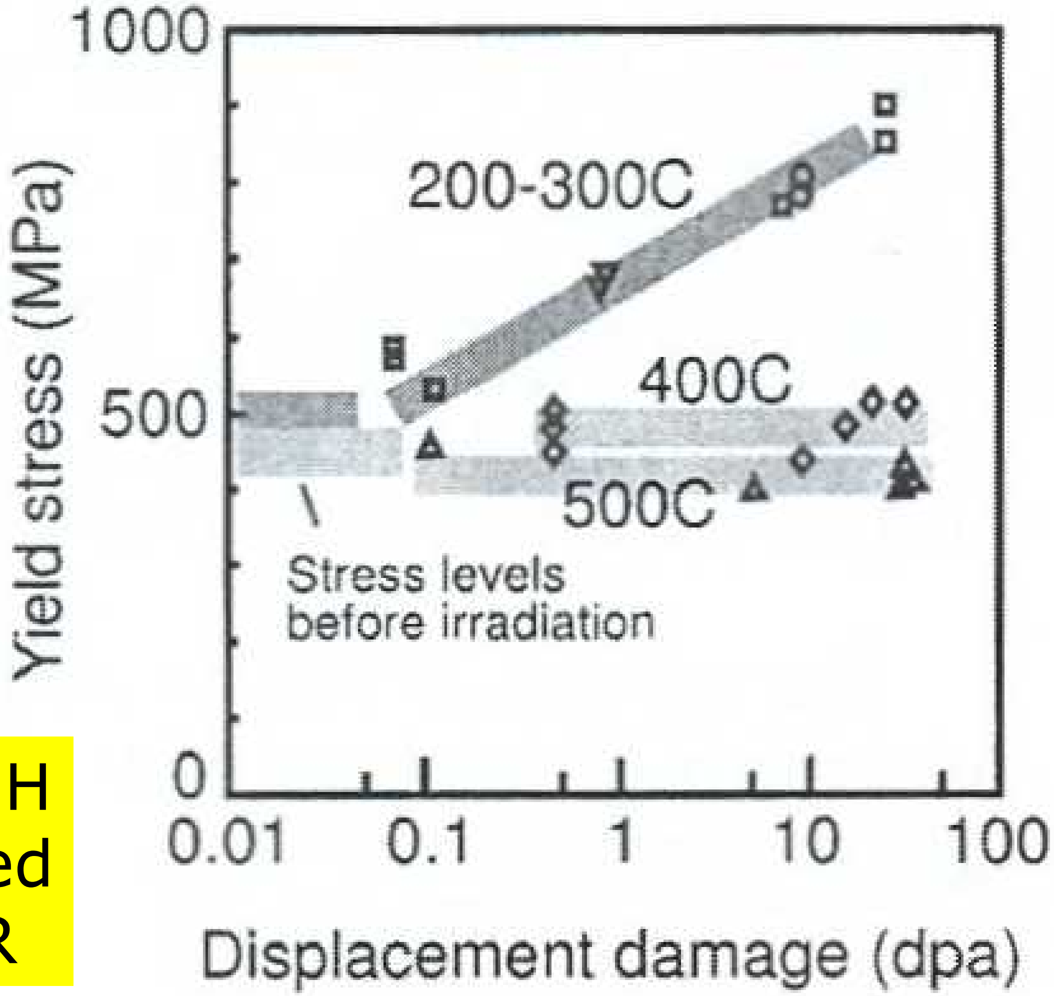


Red symbols: SCK

Literature data: irradiation effects tend to vanish above 300-400 °C

IEA Workshop on Reduced Activation Ferritic/Martensitic Steels (JAERI-Conf 2001-007), Tokyo 2-3 Nov 2000

IEA-F82H irradiated in HFIR



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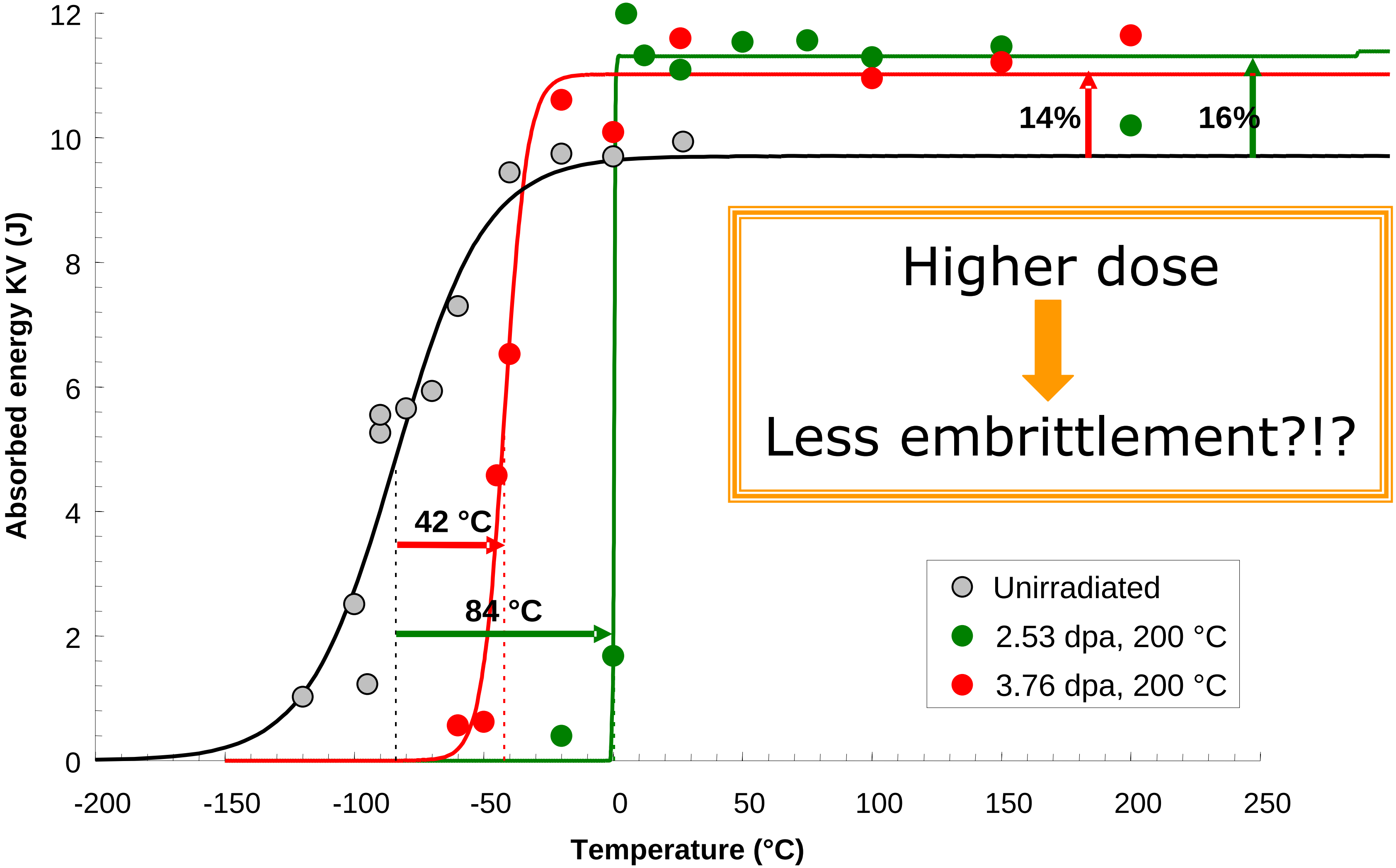
Impact test results – EM10 (or.T-L)



Higher dose

↓

Less embrittlement?!?

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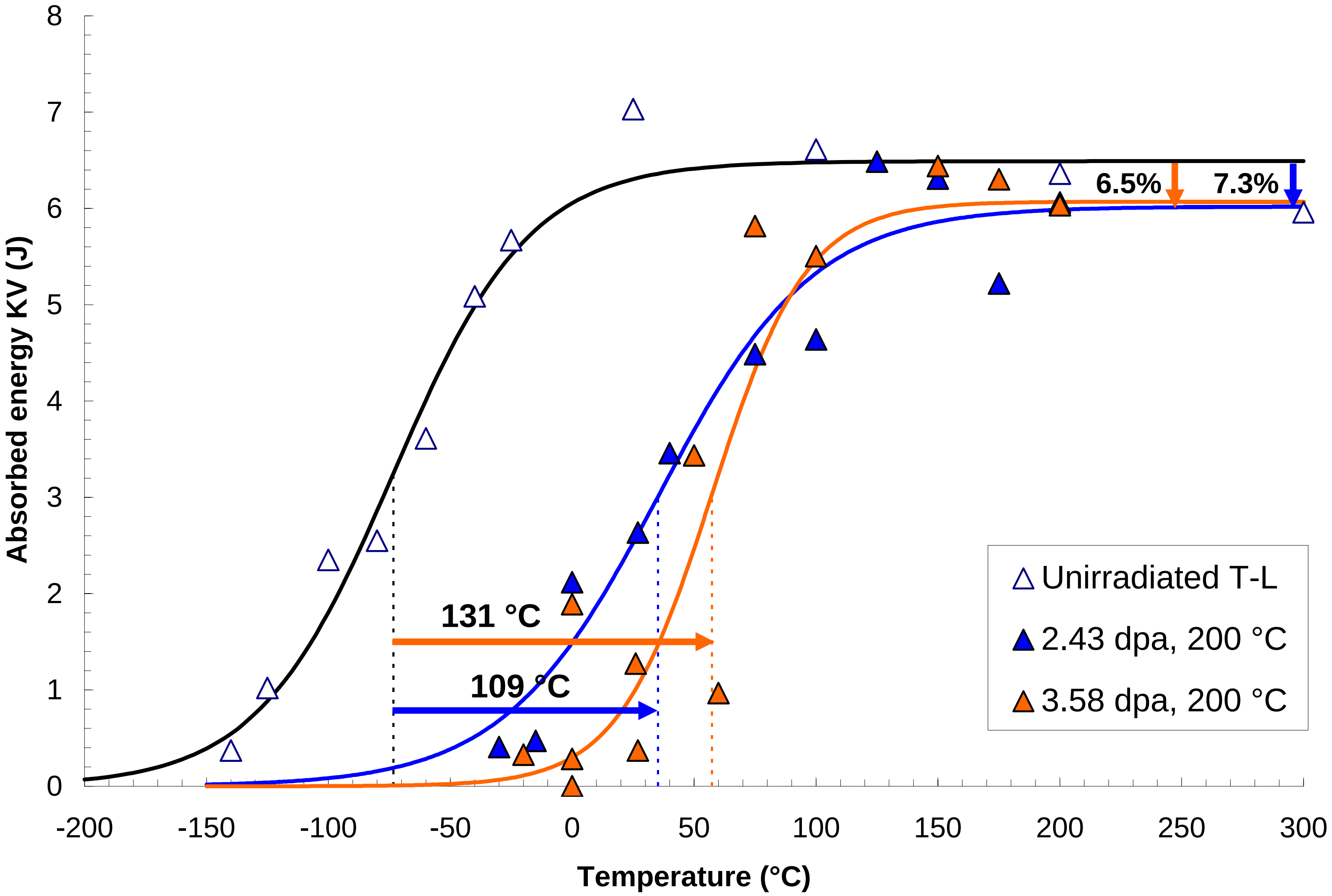
Impact test results – T91 (or.T-L)



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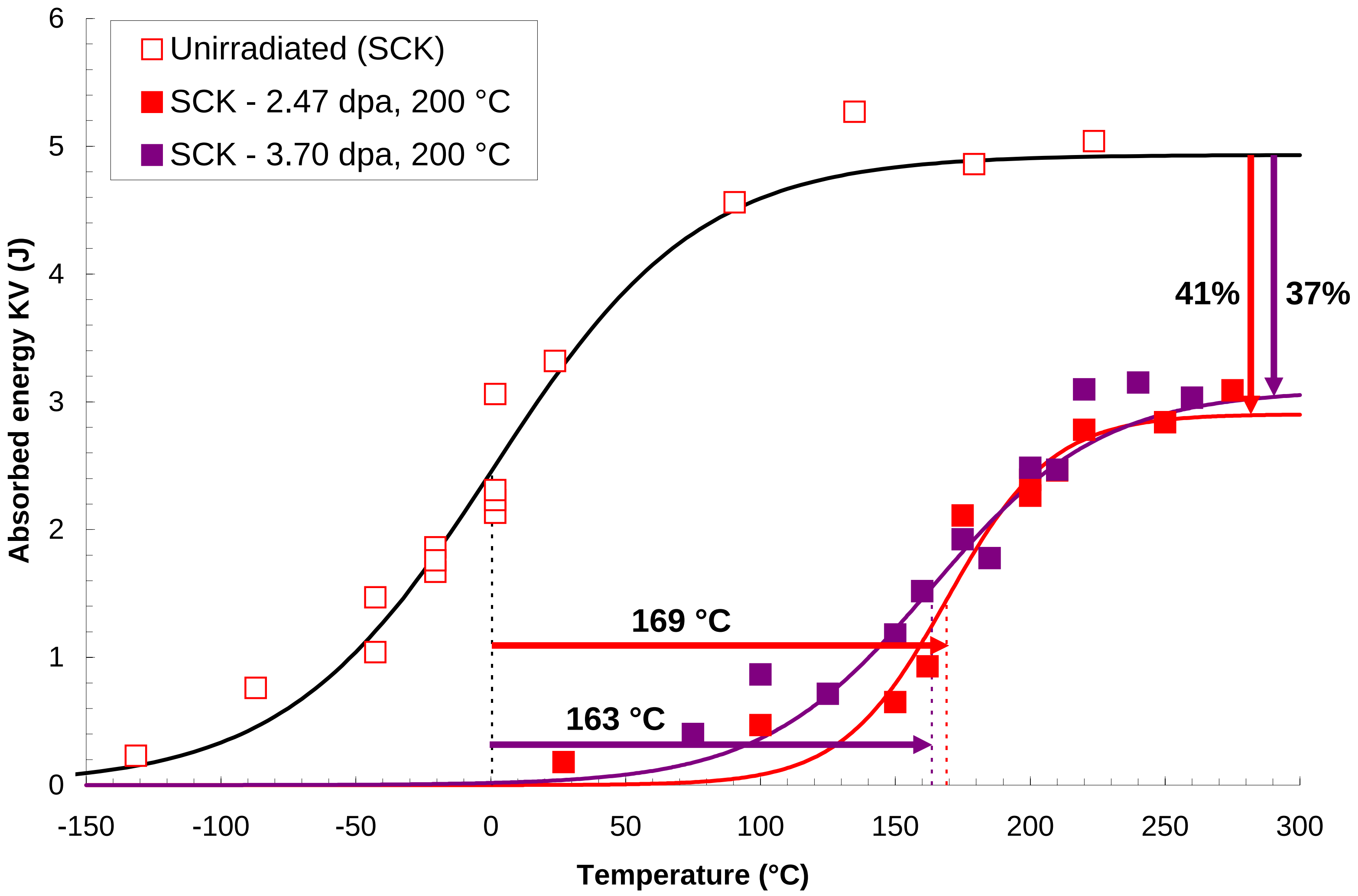
Impact test results – HT9 (or.L-T)

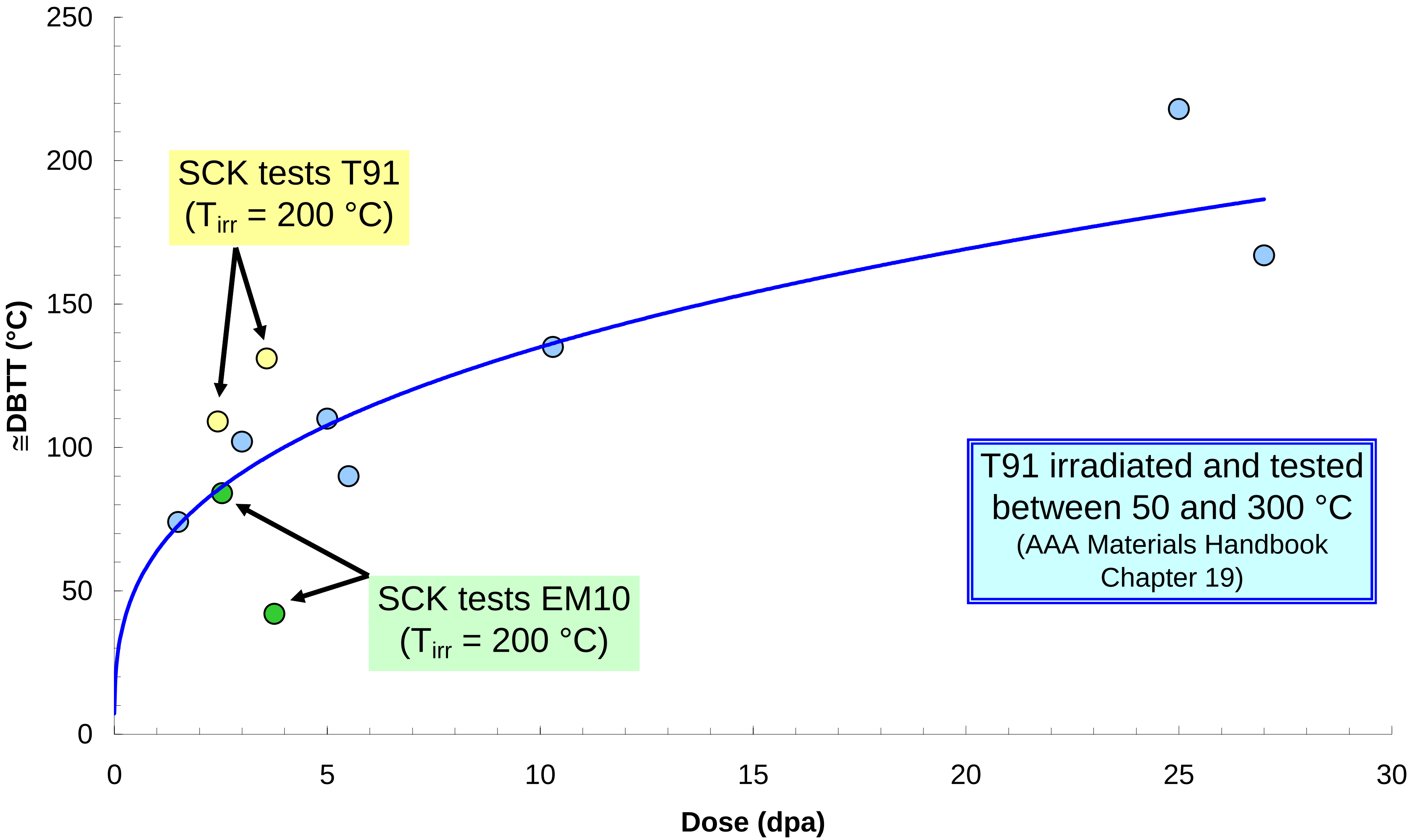


Impact test results – Summary

?

Material	**Dose: 2.43-2.53 dpa**			**Dose: 3.58-3.76 dpa**		
	ΔDBTT (°C)	ΔUSE (J)	ΔFATT (°C)	ΔDBTT (°C)	ΔUSE (J)	ΔFATT (°C)
EM10	84	+16%	69	42	+14%	31
T91	109	-7.3%	120	131	-6.5%	125
HT9	169	-41%	191	163	-37%	181

Impact test results
Comparison with the literature

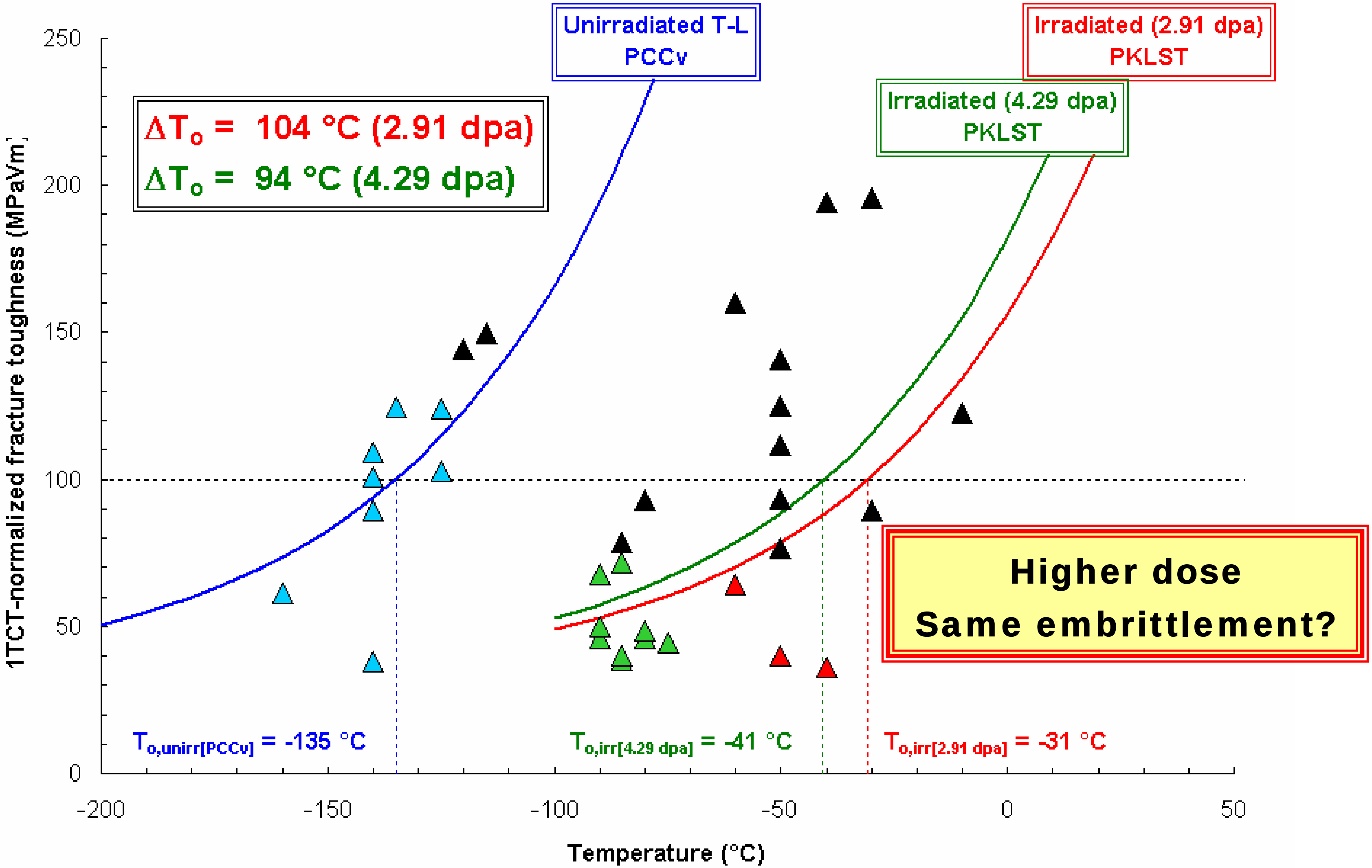


SCK tests T91
(T_{irr} = 200 °C)

SCK tests EM10
(T_{irr} = 200 °C)

T91 irradiated and tested between 50 and 300 °C
(AAA Materials Handbook Chapter 19)

Fracture toughness test results EM10 (or.T-L)



ΔT_o = 104 °C (2.91 dpa)

ΔT_o = 94 °C (4.29 dpa)

Higher dose

Same embrittlement?

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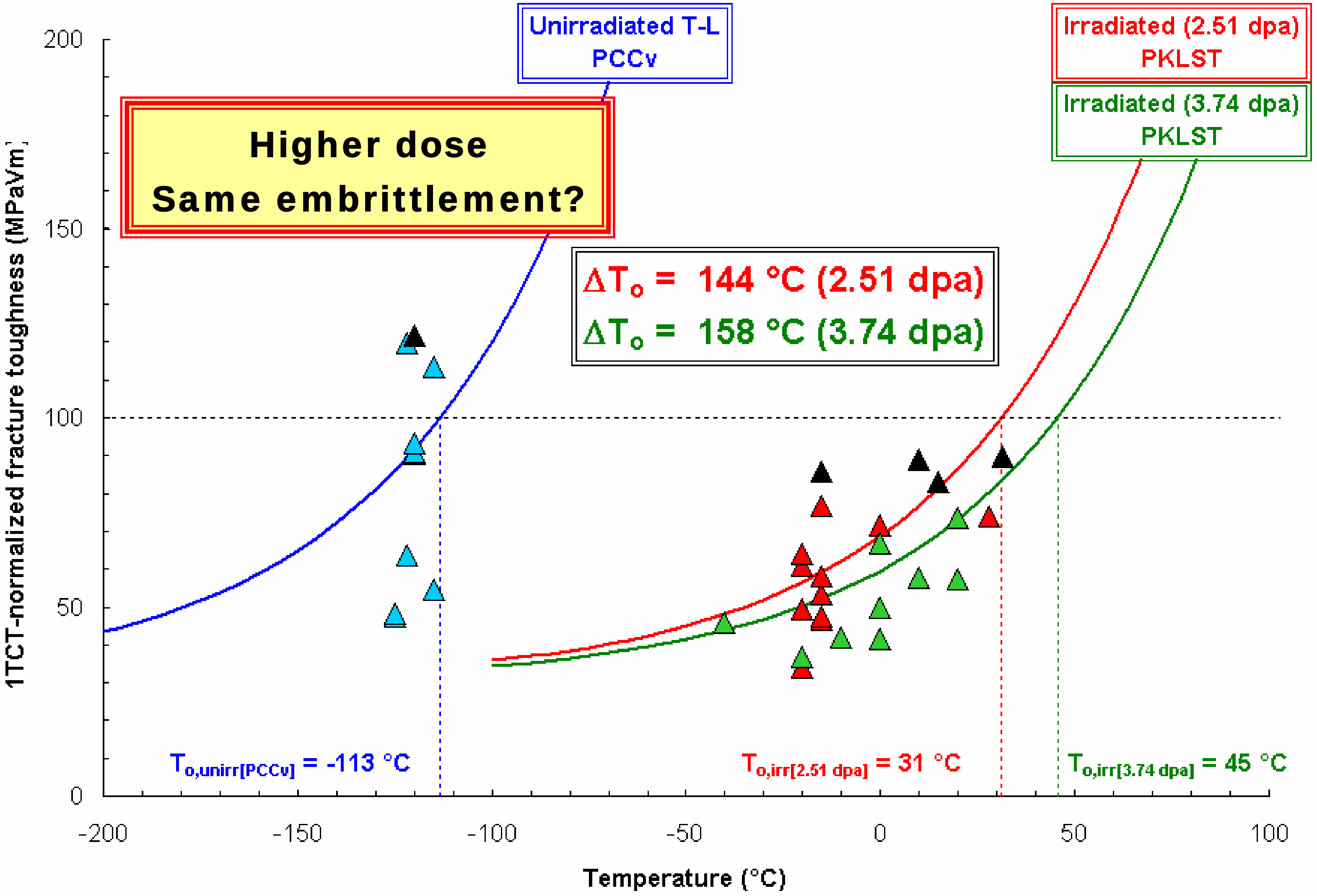
Fracture toughness test results T91 (or.T-L)



Higher dose

Same embrittlement?

ΔT_o = 144 °C (2.51 dpa)

ΔT_o = 158 °C (3.74 dpa)

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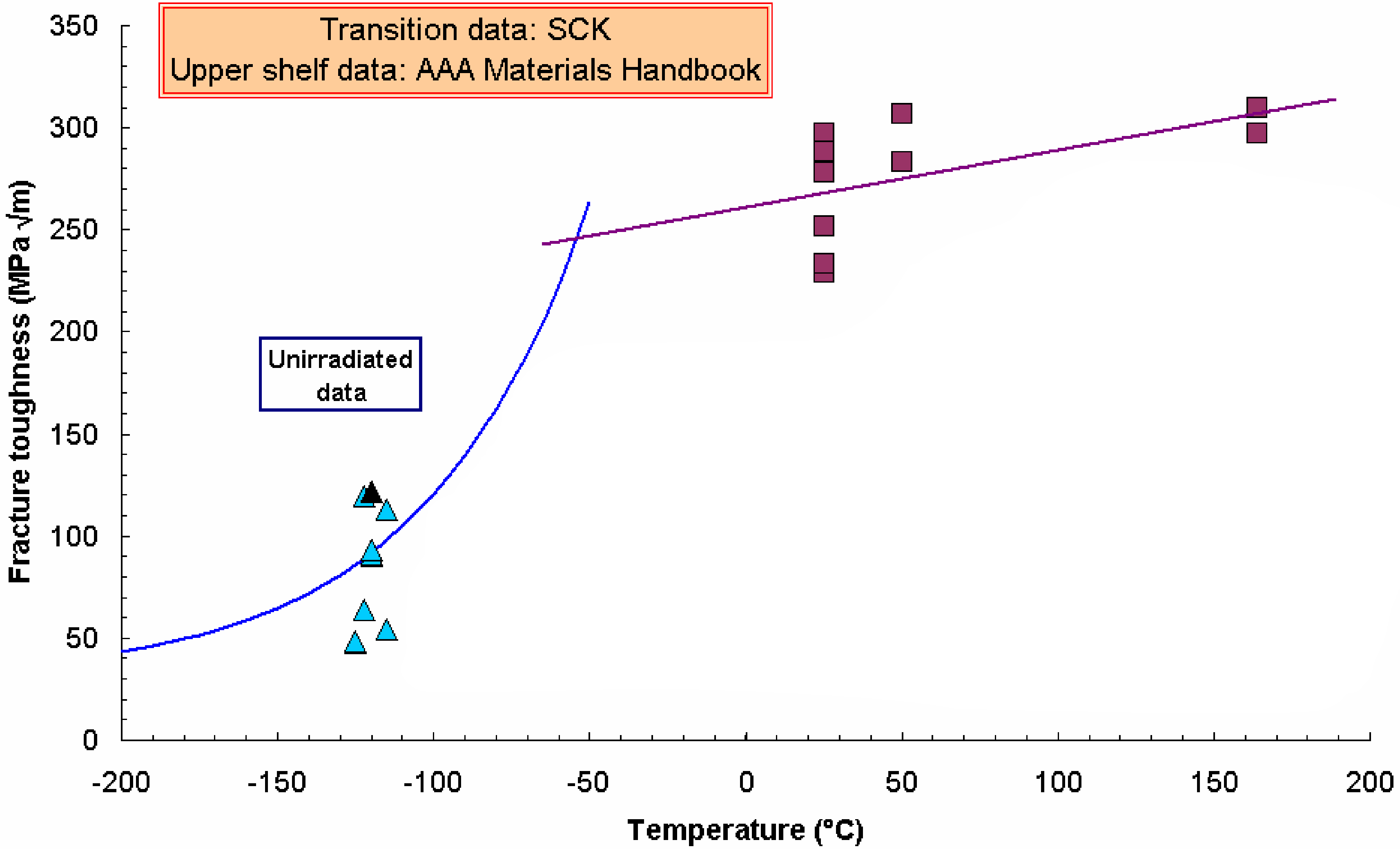
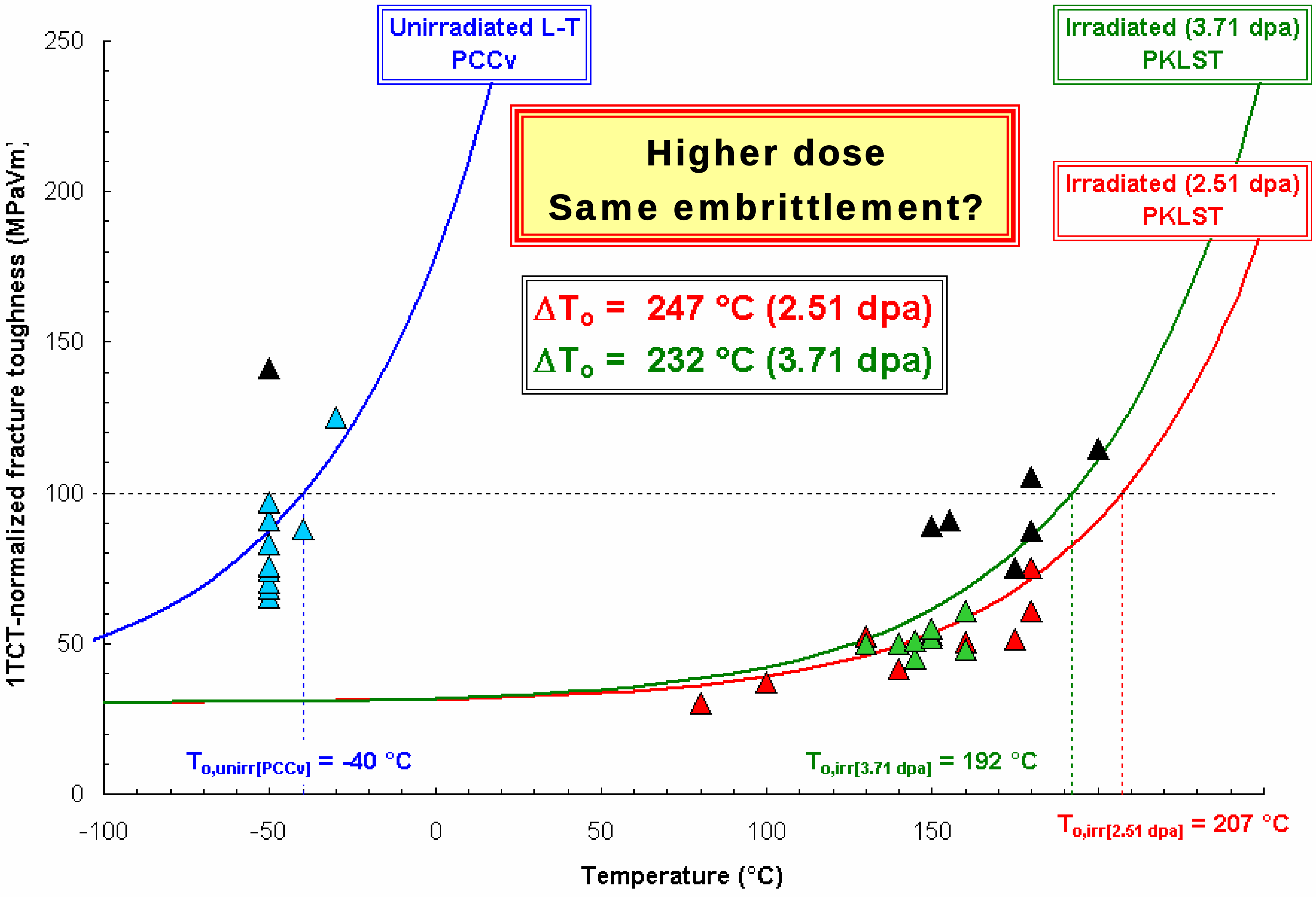
Fracture toughness degradation T91 – SCK data + literature



Fracture toughness test results
HT9 (or.L-T)



Higher dose

Same embrittlement?

ΔT_o = 247 °C (2.51 dpa)

ΔT_o = 232 °C (3.71 dpa)

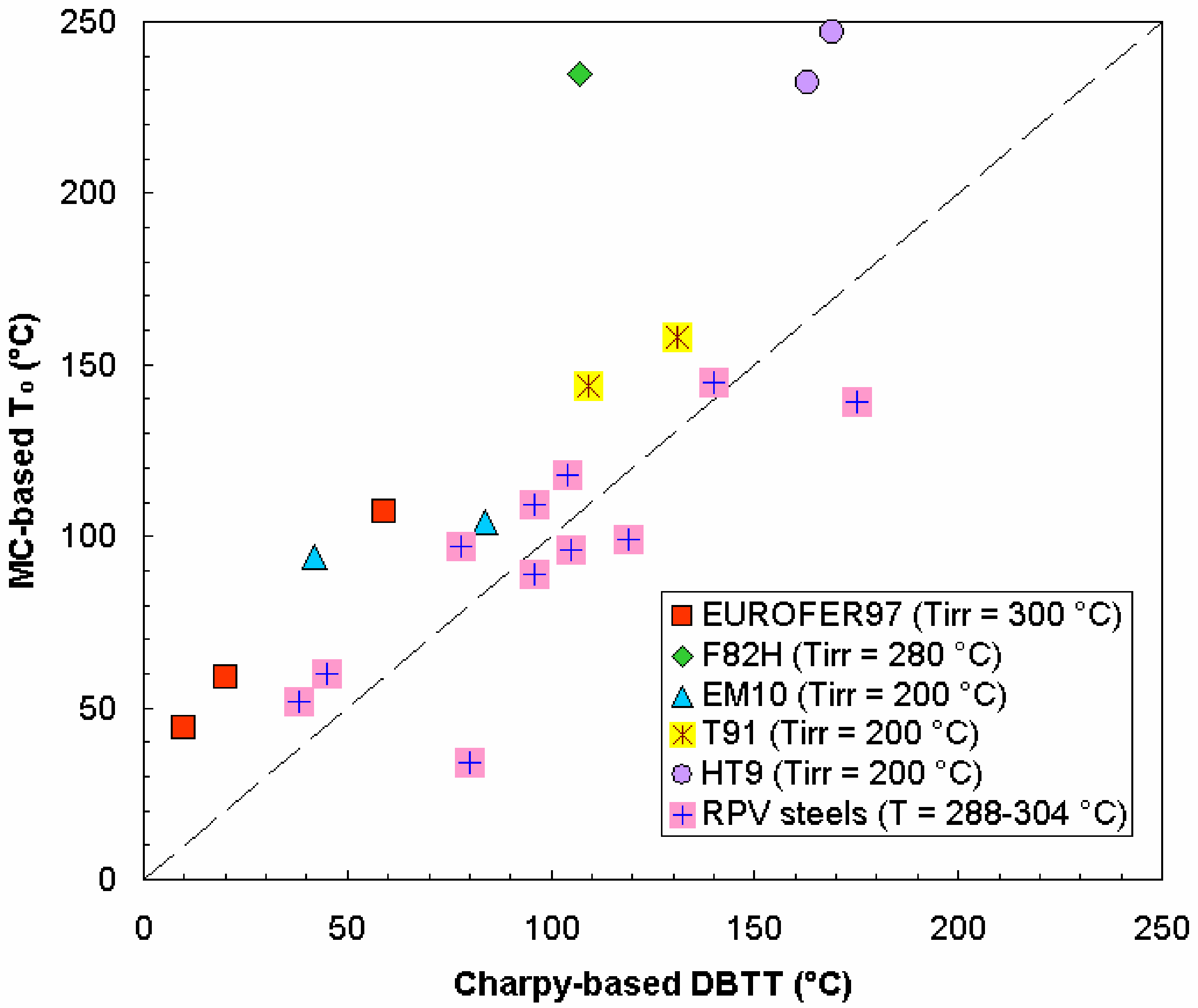
Fracture toughness test results
Summary and comparison with impact data

12% steel much more prone to embrittlement than 9%

Material	**Dose: 2.43-2.91 dpa**			**Dose: 3.58-4.29 dpa**		
	ΔDBTT (°C)	ΔFATT (°C)	ΔT_o (°C)	ΔDBTT (°C)	ΔFATT (°C)	ΔT_o (°C)
EM10	84	69	104	42	31	94
T91	109	120	144	131	125	158
HT9	169	191	247	163	181	232

Fracture toughness shifts are systematically larger than Charpy!

T_o shifts larger than DBTT shift: an issue for F/M (8%-12% steels)?



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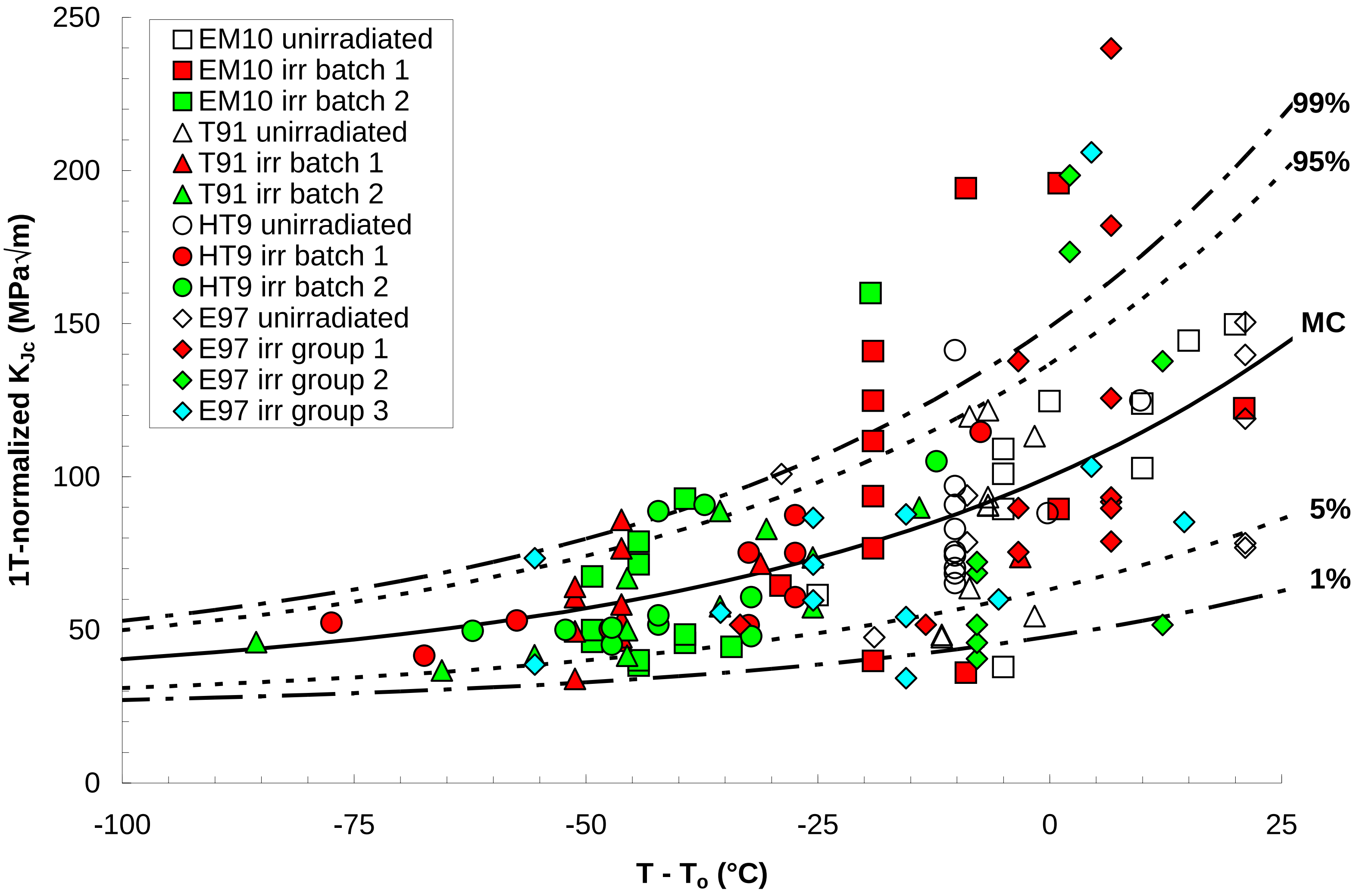
Another potential issue:
applicability of Master Curve to high Cr steels



Conclusions (I)

- Tensile tests
  - EM10 shows the least irradiation hardening (i.e. yield stress increase), HT9 the highest
  - Hardening of T91 is approximately constant between 1st and 2nd batch (but no saturation! ➡ see CEA data at 40 dpa)
  - Hardening data consistent with literature on RAFM steels
- Impact tests
  - EM10: USE increases (???) with irradiation; DBTT shift is moderate but dose effect is reversed (???)
  - T91: irradiation embrittlement (USE and DBTT) is larger than for EM10; limited dose effect
  - EM10 and T91: irradiated data consistent with literature
  - HT9: largest embrittlement

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Conclusions (II)

- Fracture toughness tests
  - Same ranking as for Cv tests
  - T_o shifts systematically larger than DBTT shifts (common feature with RAFM steels – different from RPVS) ➡ implications for safety analyses
  - Potential issue (*further research needed*): applicability of Master Curve to high Cr steels ➡ implications for safety analyses

- T91 has been selected as structural material for MYRRHA, on account of:
  - acceptable mechanical properties before and after irradiation
  - easiness of procurement and reasonable cost
  - insight gained on possible improvements in heat treatment and chemical composition (role of N,V)