Equivalent Mechanical Properties of X-Ray and E-Beam Crosslinked Vitamin E Blended Polyethylene

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1. Introduction

Oxidation ↔ Wear ↔ Osteolysis

Oxidation – 14 years Shelf-Life

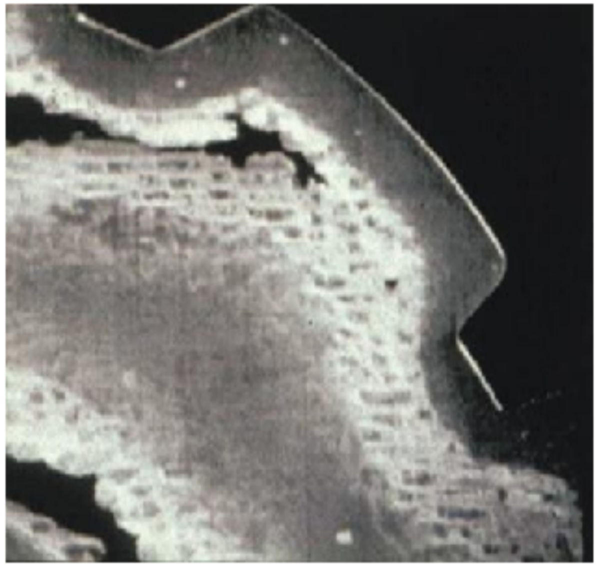


Figure 8 : Polyéthylène âgé de 14 ans stocké sur étagère.

[Maîtrise othopédique, n° 204, Mai 2011]

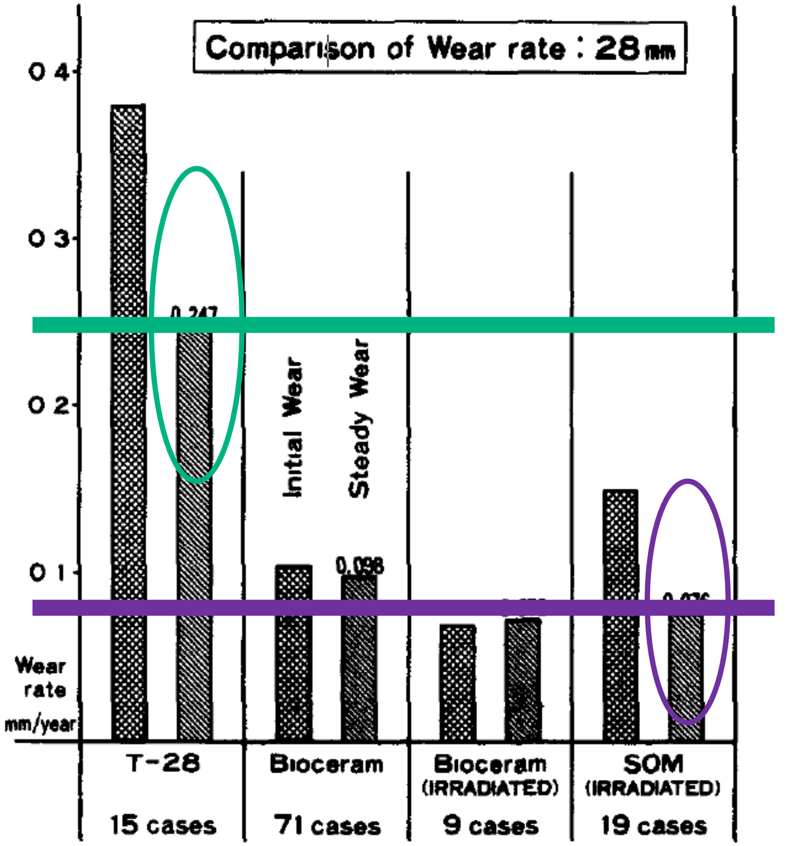
(1)

(2)

[Gómez-Barrena et al. 2009]

[Klutzny, Uni. Magdeburg, 2018]

1. Introduction



uncrosslinked **crosslinked**

Oonishi et al. used the radiation crosslinking at the beginning of the 1970s

XLPE (= highly crosslinked polyethylene) acetabular liners have shown significant improvements in decreasing wear and osteolysis in total hip arthroplasty patients

[Kurtz et al. 2011, Oral and Muratoglu 2011, Bragdon et al. 2011]

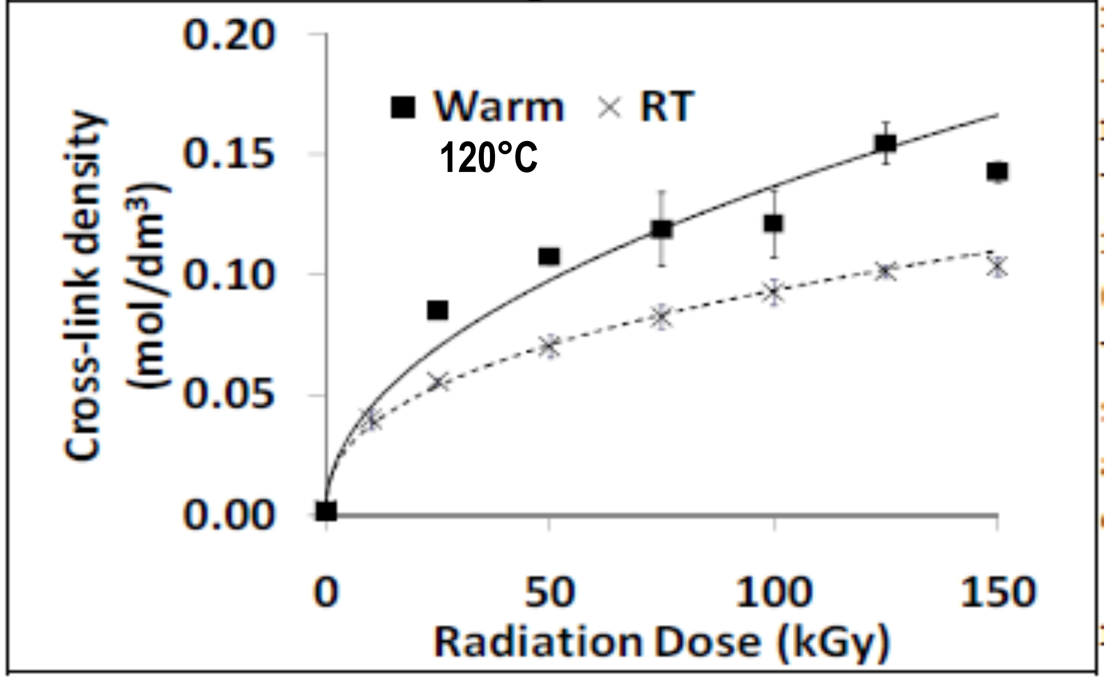
→ Crosslinking : Reduce wear

→ Vitamin E : Oxidation Stabilisation

1. Introduction: Why X-ray?

Advantages of warm irradiation

Figure 3. Cross-link density of vitamin E-blended, irradiated UHMWPE as a function of radiation dose and temperature.



"warm irradiation allowed for increased preservation of the antioxidant, increased grafting, increased cross-linking and decreased wear."

[Oral et al, 2011, 2013]

⇒ **Temperature control during processing necessary**

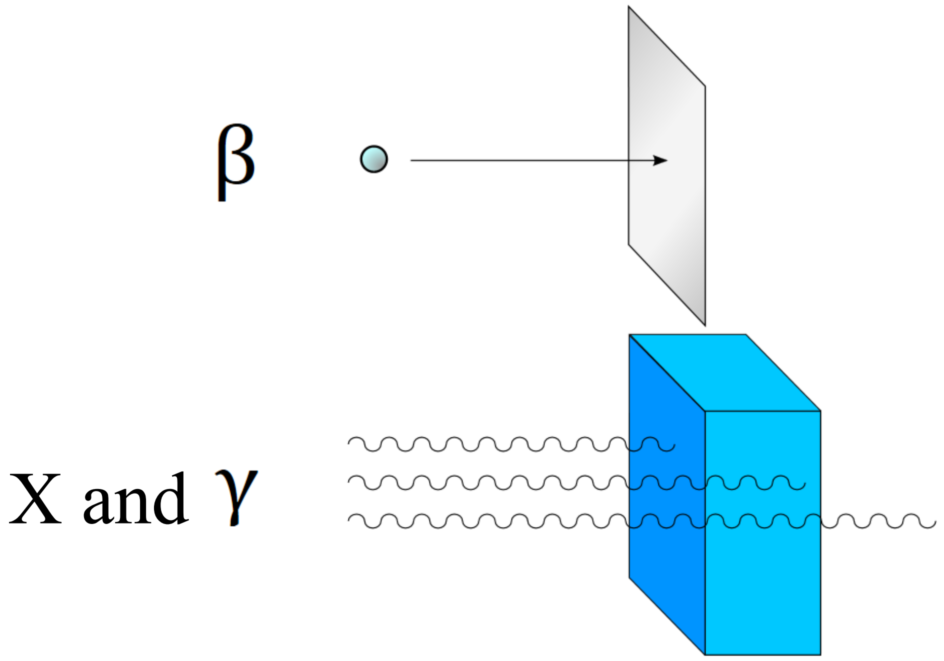
1. Introduction: Why X-ray?

Advantages of X-ray crosslinking

- High penetration depth
- Moderate dose rate

	Dose rate [kGy/s]	Penetration depth
Gamma rays	0.001	+++
E-Beam	100	- - -
X rays	1-10	+++

[Makuuchi et al, 2012]



[Stannered, Wikipedia]

2. Materials and Methods

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Materials

	Raw Material	Dose [kGy]	Temperature
E-Beam (Vitelene®)	Chirulen®1020E	80	Warm
X-Ray		80	Room (RT)
			Warm
		100	Room (RT)
			Warm

Methods

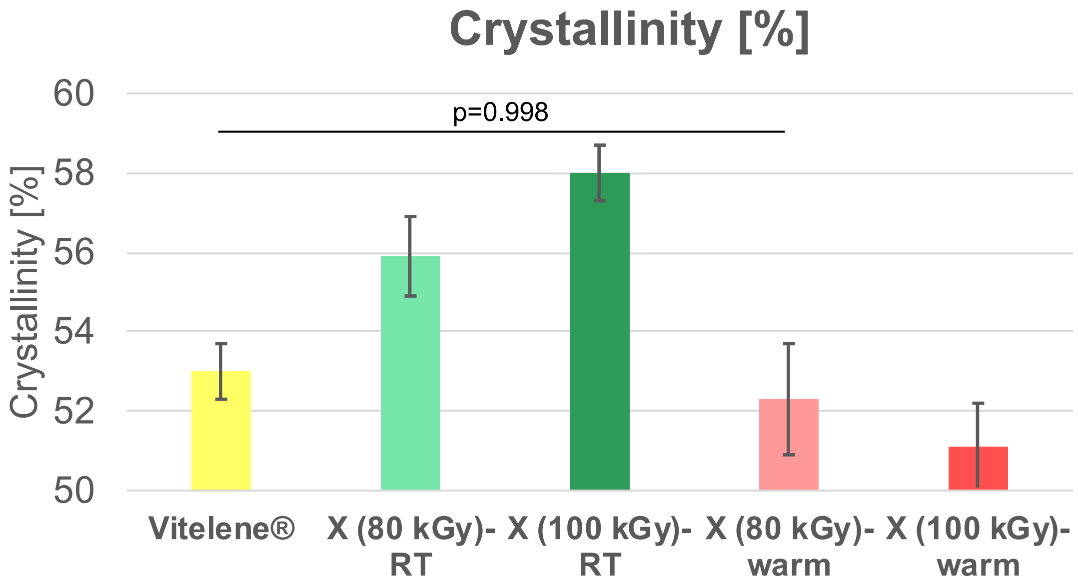
- Differential scanning calorimetry acc. ASTM F2625
- Uniaxial tensile strength acc. ASTM D638
- Biaxial tensile strength, Small Punch Testing (SPT) acc. ASTM F2183
- Izod impact strength acc. ASTM D256

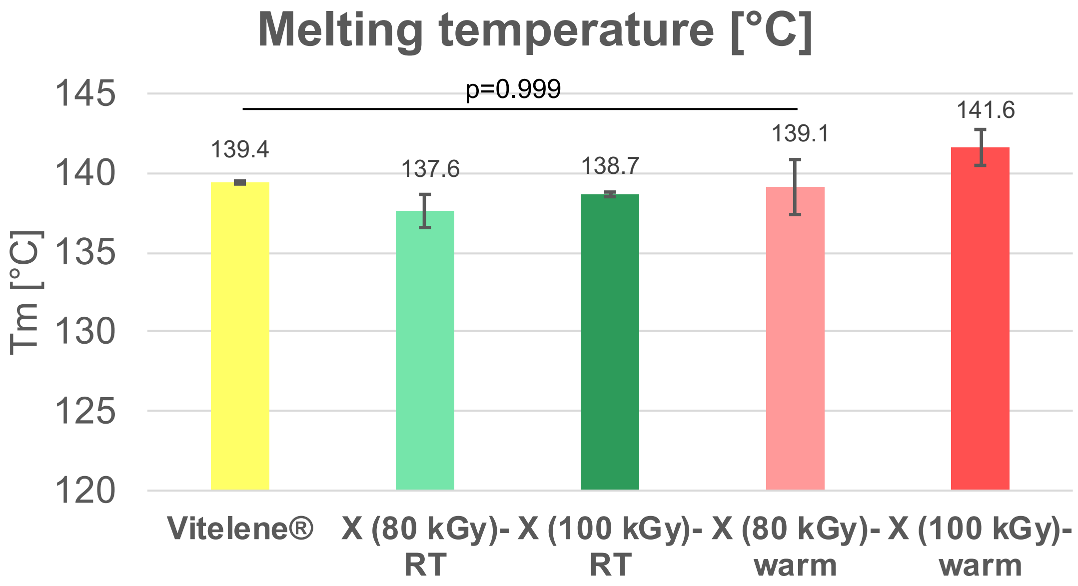
UHMWPE

3. Results: thermal properties

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm

Differential scanning calorimetry ASTM F2625





T↗ ⇨ ↘ Crystallinity

T↗ ⇨ ↗ Melting point

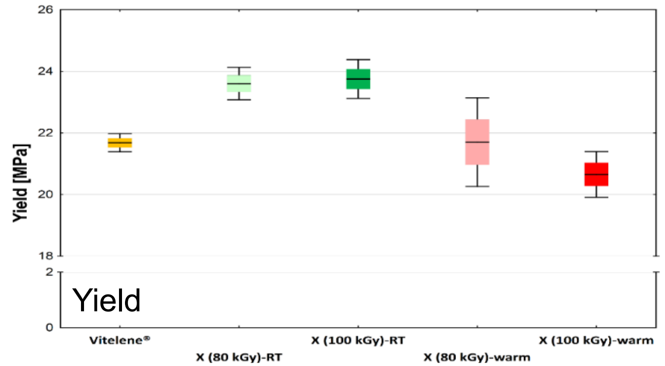
- Crosslinks inhibit the recrystallization [Slouf et al, 2008]
- Crosslinks disturb the melting of the crystals [Premnath et al, 1999]

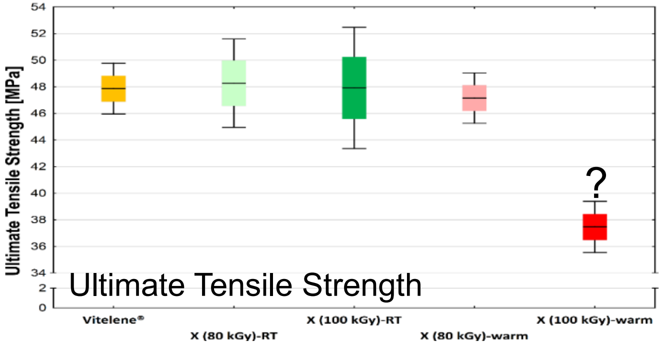
No significant difference between E-Beam and X-Ray

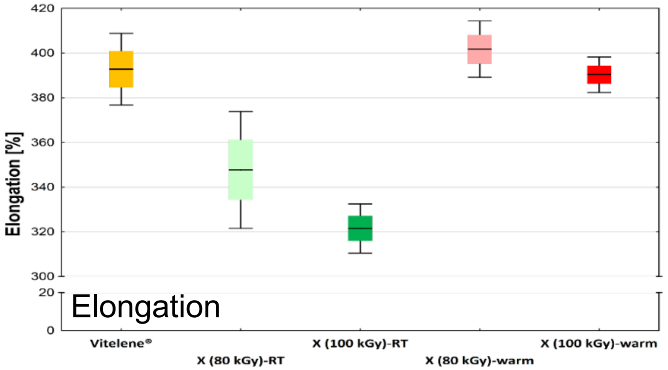
4. Results: mechanical properties

Tensile properties ASTM D638

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm







Influence of **temperature**:

T ↗ ⇨ ↘ Yield strength

- ➢ Loss of crystallinity

[Bracco et al, 2017]

T ↗ ⇨ ↘ Ultimate strength

- ➢ Loss of crystallinity

T ↗ ⇨ ↗ Elongation at break

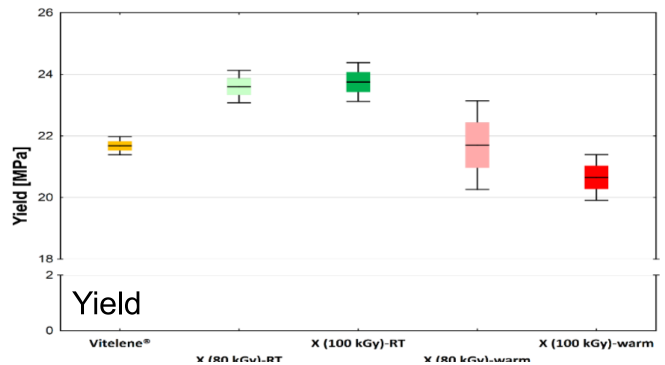
- ➢ **RT**: Higher crystallinity, higher brittleness, reduced creeping
- ➢ **Warm**: Lower crystallinity, higher ductility

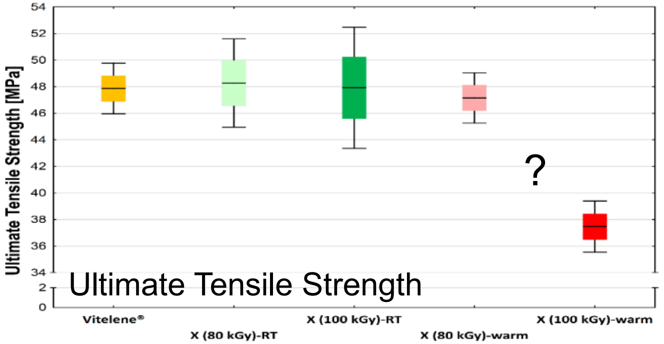
[George et al, 2014]

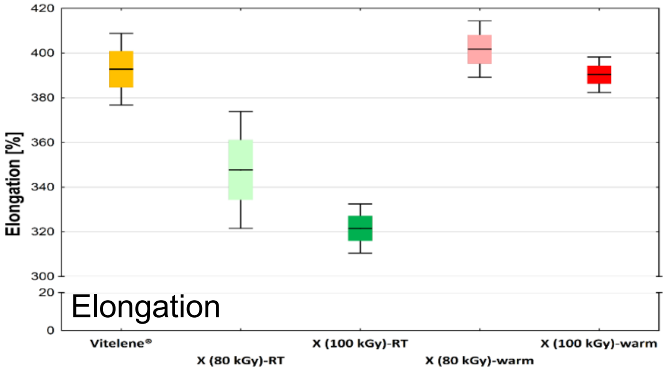
4. Results: mechanical properties

Tensile properties ASTM D638

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm







Influence of **dose** ↗ :

Yield:

⇨ Little effect

Ultimate Tensile Strength (UTS):

Room Temperature: no significant difference

Warm: UTS 100 kGy << UTS 80 kGy !!!

➢ Loss of crystallinity

No significant difference between E-Beam and X-Ray

Elongation At Break (EAB):

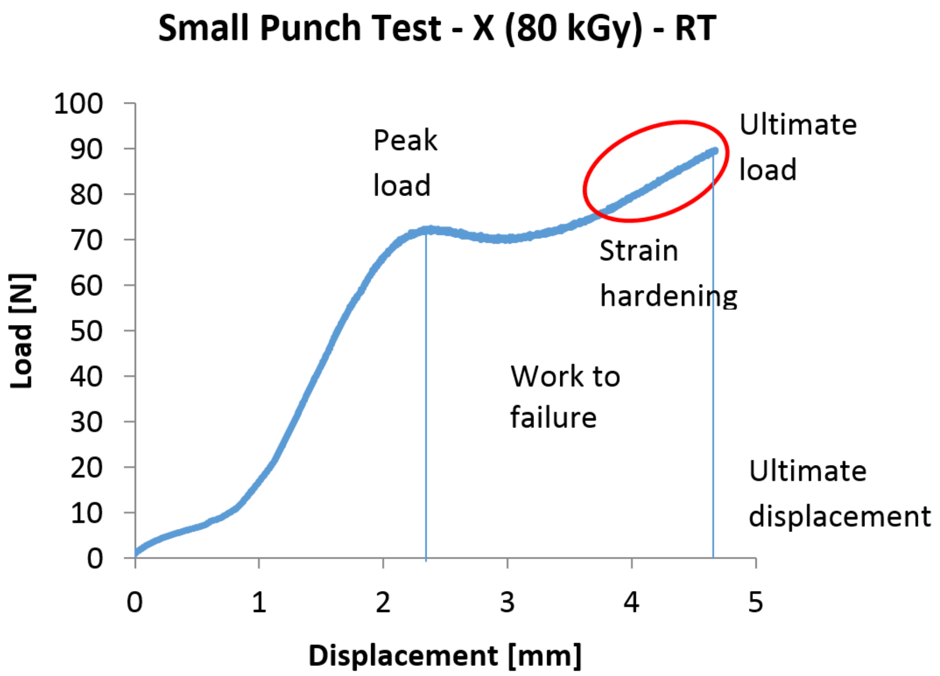
Room Temperature and **Warm :** EAB 100 kGy < EAB 80 kGy

➢ Dose ↗ ⇨ crosslinking ↗ ⇨ stiffness ↗ ⇨ ↘ ductility

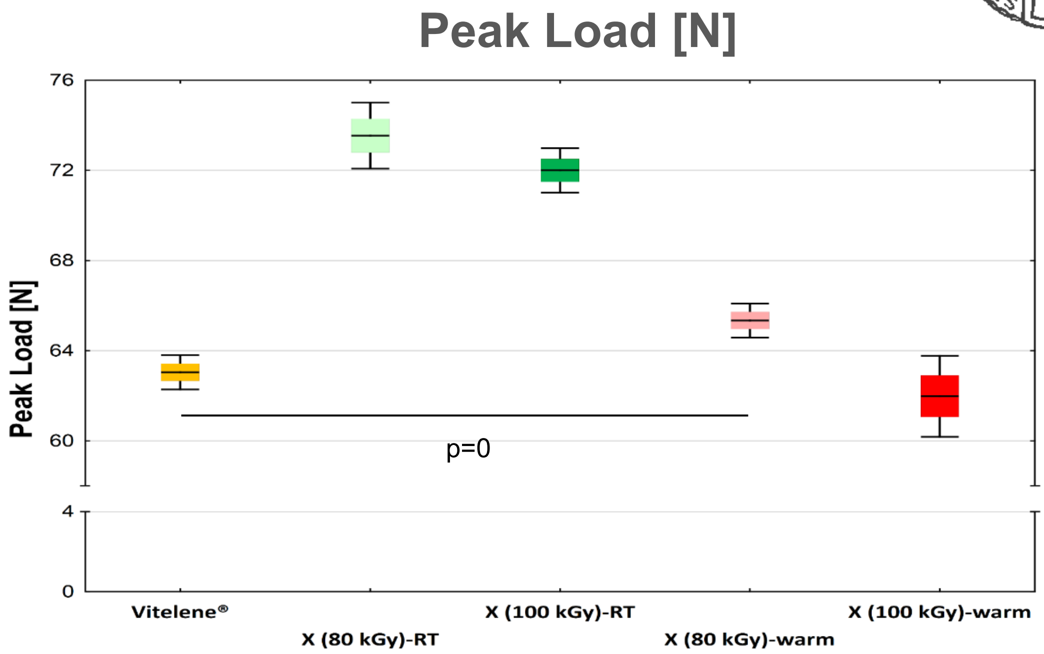
4. Results: mechanical properties

Small Punch Testing ASTM F2183

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm



Typical “crosslink” sloop



↗ Temperature (80 and 100 kGy) ⇨ ↘ Peak Load

- Loss of crystallinity

↗ Dose warm and RT ⇨ ↘ Peak Load

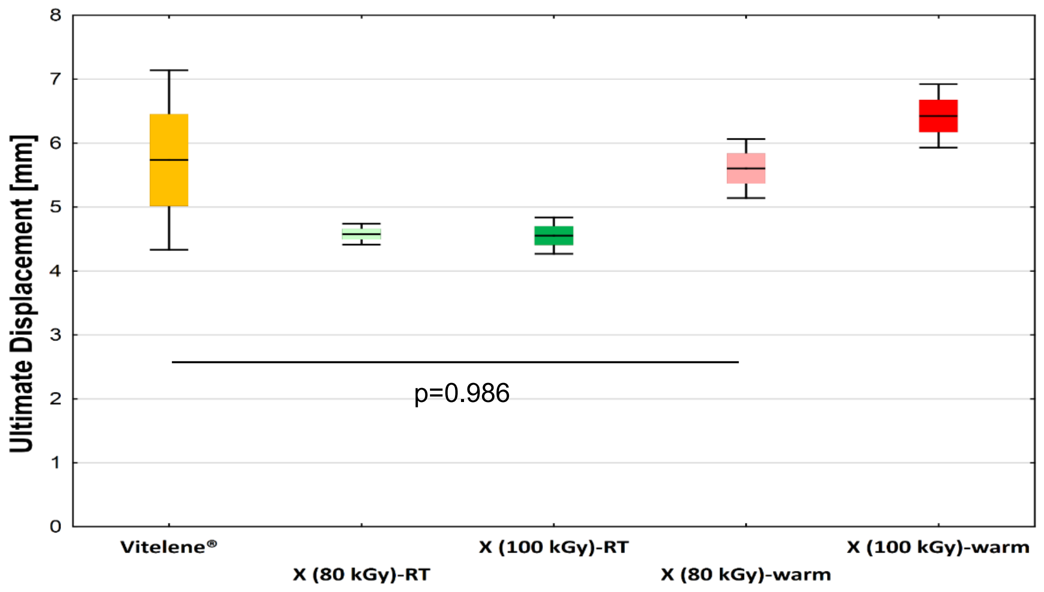
- Scission/crosslinking ↗

4. Results: mechanical properties

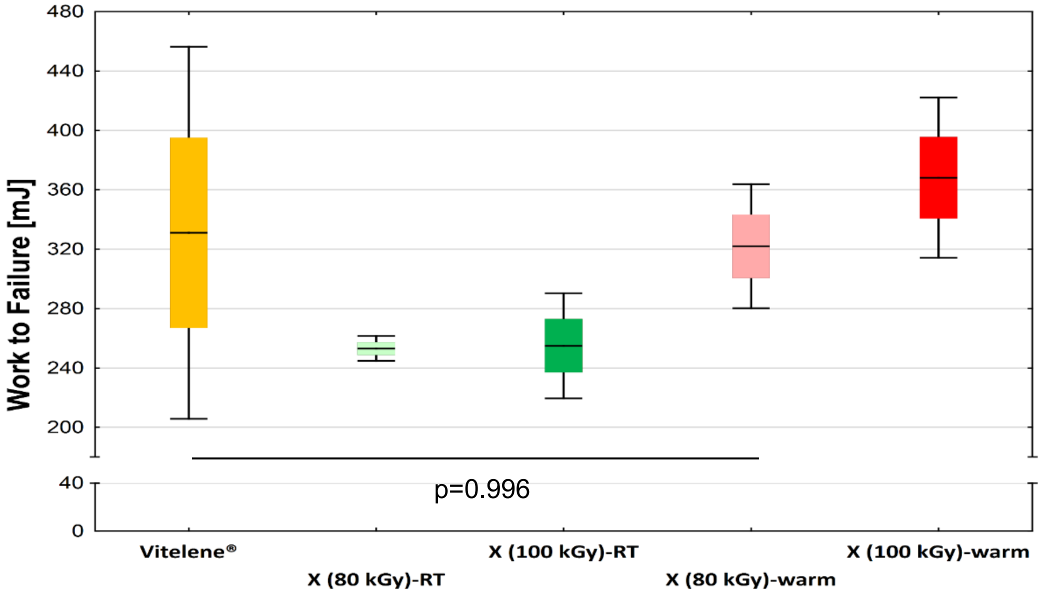
Small Punch Testing ASTM F2183

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm

Ultimate Displacement [mm]



Work to Failure [mJ]



↗ Temperature and ↗ Dose ⇨ ↗ Ultimate displacement, ↗ Work to failure

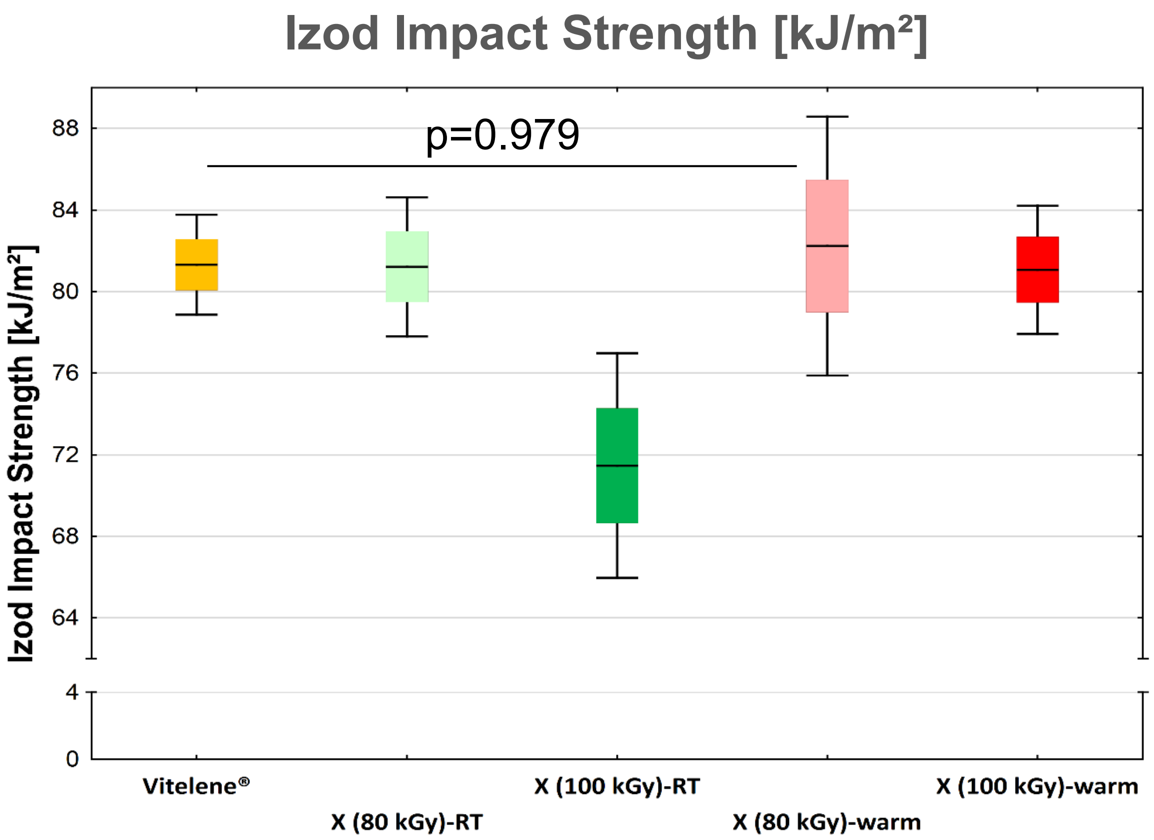
- **RT**: higher crystallinity, ↘ strain hardening, ↘ toughness
- **Warm**: lower crystallinity, ↗ strain hardening, ↗ ductility, ↗ toughness

No significant difference between E-Beam and X-Ray

4. Results: mechanical properties

Izod Impact Strength ASTM D256

β-80 kGy-warm
X-80 kGy-RT
X-100 kGy-RT
X-80 kGy-warm
X-100 kGy-warm



Room Temperature:

↗ Dose ⇨ ↘ Impact Strength

➤ ↗ Crosslinking, ↘ Plasticity

Warm:

No dose influence

Heat overtook the dose effect

No significant difference between E-Beam and X-Ray

5. Conclusion

1. Increasing processing temperature
   - ↘ Crystallinity
   - ↘ tensile strength, peak load
   - ↗ elongation at break, ultimate displacement and work to failure

2. Bigger impact of temperature (100°C vs RT)
   than of dose (80 kGy vs 100 kGy)

3. Equivalent material properties regardless of radiation source
   e-beam or x-ray (80 kGy, 100°C)

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THANK YOU
FOR YOUR TIME

6. Literature

<ins>Tensile properties</ins>

"Warm irradiation and melting of UHMWPE results in higher ductility and lower strength in comparison with cold irradiation".
[Muratoglu et al, 2001]

"The higher level of cross-linking causes a decrease in elongation".
[Kurtz et al, 2002-2003]

Oonishi H. et al, Improvement of polyethylene by irradiation in artificial joints. Radiation Physics and chemistry, 39, 495-504 (1992)

Oral et al. Increasing irradiation temperature maximizes vitamin E grafting and wear resistance of ultrahigh molecular weight polyethylene. J Biomed Mater res Part B 2013:101B:436-440

Makuuchi et al, Radiation processing of polymer materials and its industrial applications,WILEY, 2012, ISBN: 978-0-470-58769-0

Kurtz et al, Comparison of the properties of annealed crosslinked (Crossfire) and conventional polyethylene as hip bearing materials. Bull. Hosp. Joint Dis. 61 (2002-2003) 17-26

Joon Park, R.S. Lakes
Biomaterials: An Introduction
Third edition – Springer 2007
ISBN 978-0-387-37879-4

Malito et al, Material properties of ultra-high molecular weight polyethylene: Comparison of tension, compression, nanomechanics and microstructure across clinical formulations

Asano et al, Dose effects of cross-linking polyethylene for total knee arthroplasty on wear performance and mechanical properties, J. Biomed. Mater. Res. B 83 (2007) 615-622