CALORIC MATERIALS

FOR COOLING

AND HEATING

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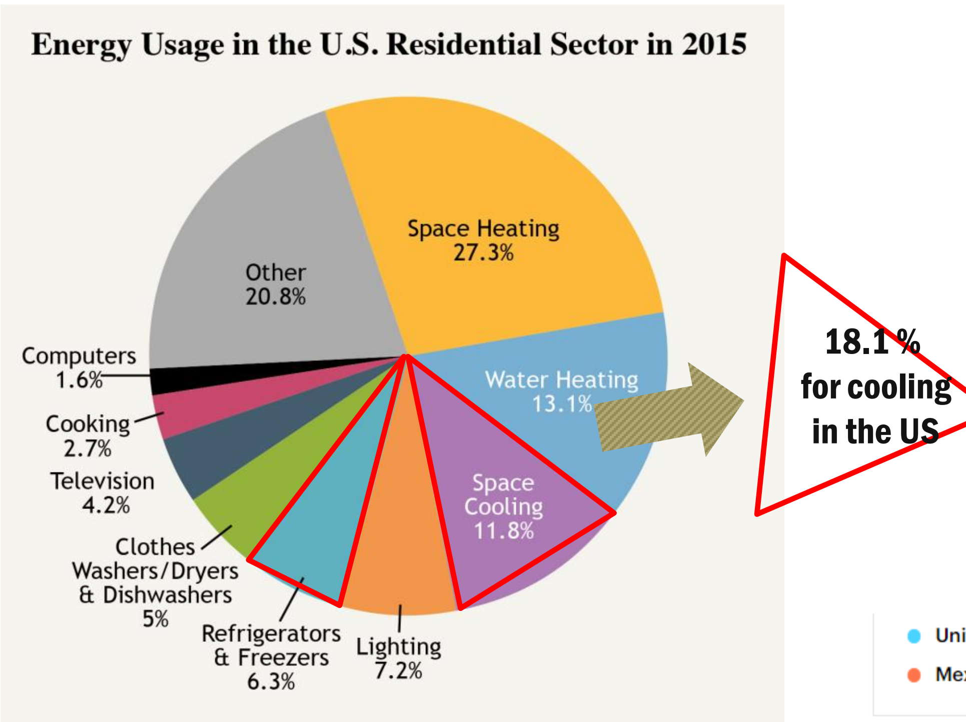
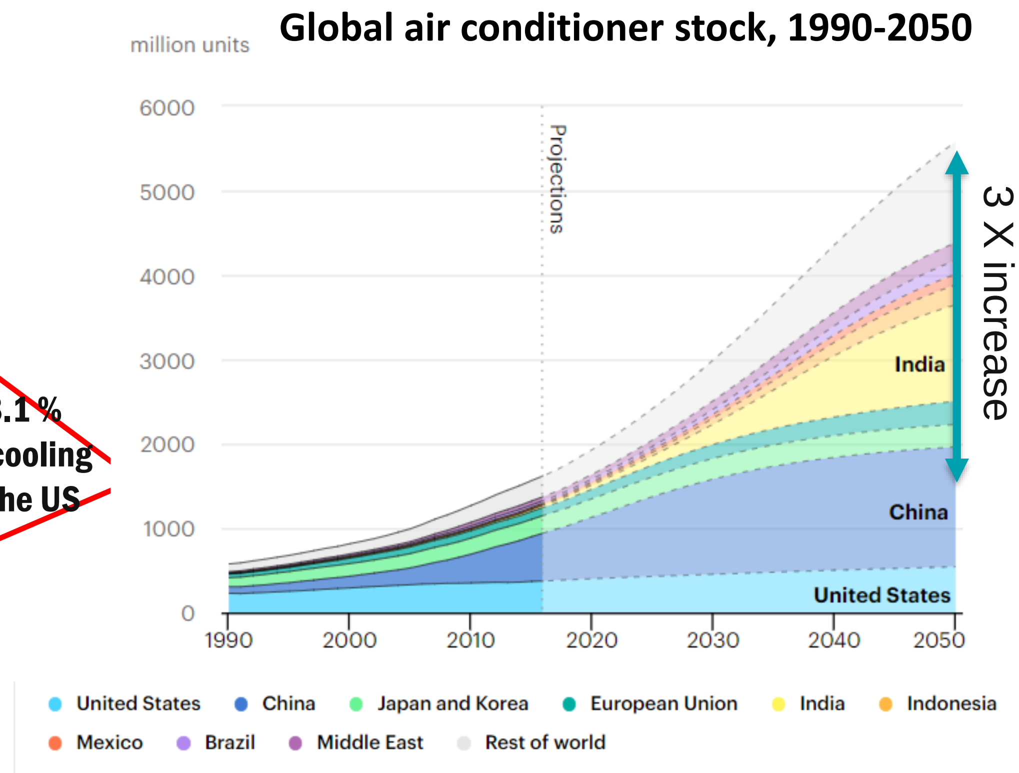
LUXEMBOURG INSTITUTE OF SCIENCE AND TECHNOLOGY LIST

HOW MUCH ENERGY DOES COOLING STAND FOR ?





http://needtoknow.nas.edu/energy/energy-use/home-work/

https://www.iea.org/reports/the-future-of-cooling

World consumption for cooling = 20 % of electricity in 2020
Can reach 50 % in 2050
Global warning does not help

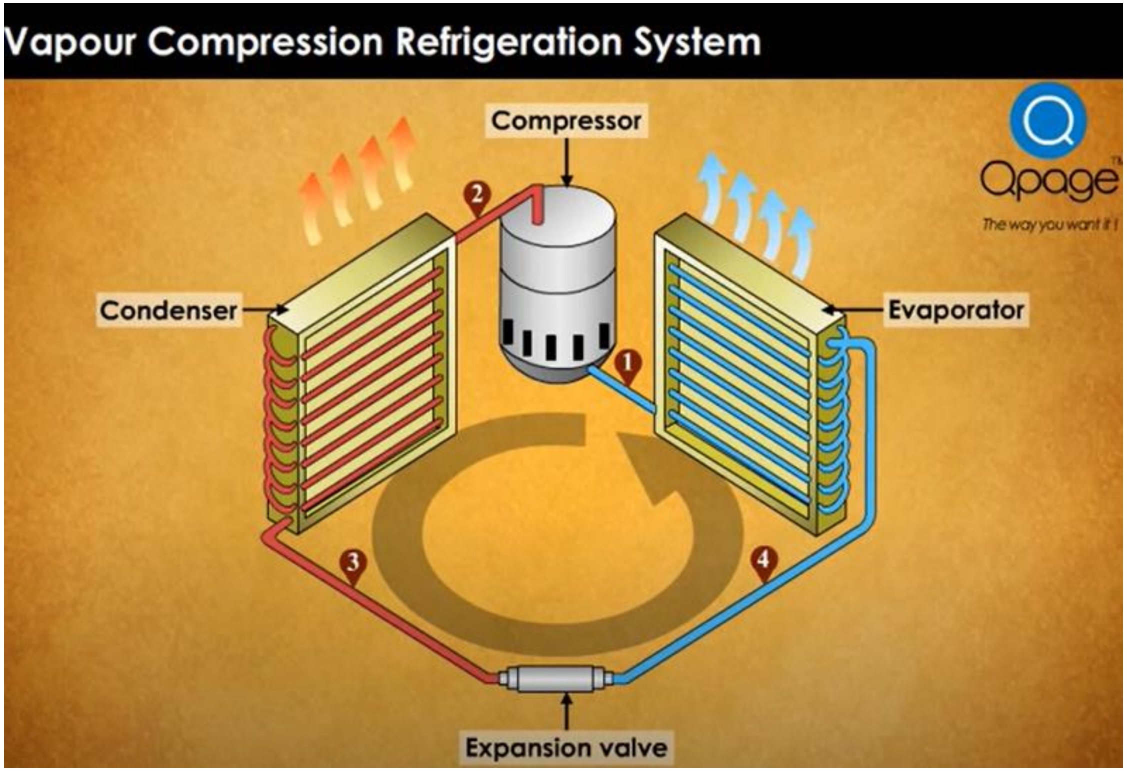
More than 50 % heating + cooling !

CHALLENGES

- Decrease greenhouse gases

- Increase energy efficiency
  - Less energy required for cooling and heating
  - Heat pump programme in EU

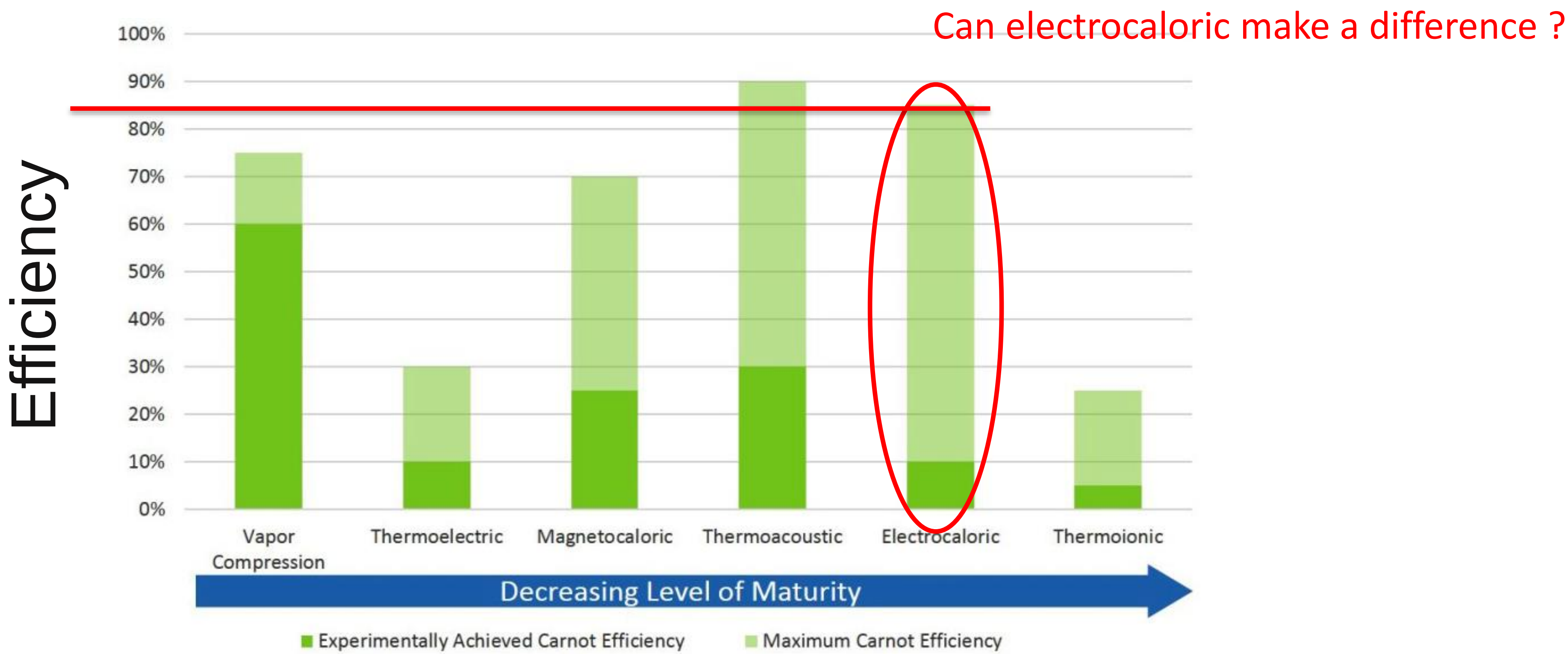
CURRENT TECHNOLOGY



https://www.youtube.com/watch?v=-Wj_MO4BqtA

- - By far the most used system
- - Developed since 1850s
- - Work either with substances dangerous for health or greenhouse gases

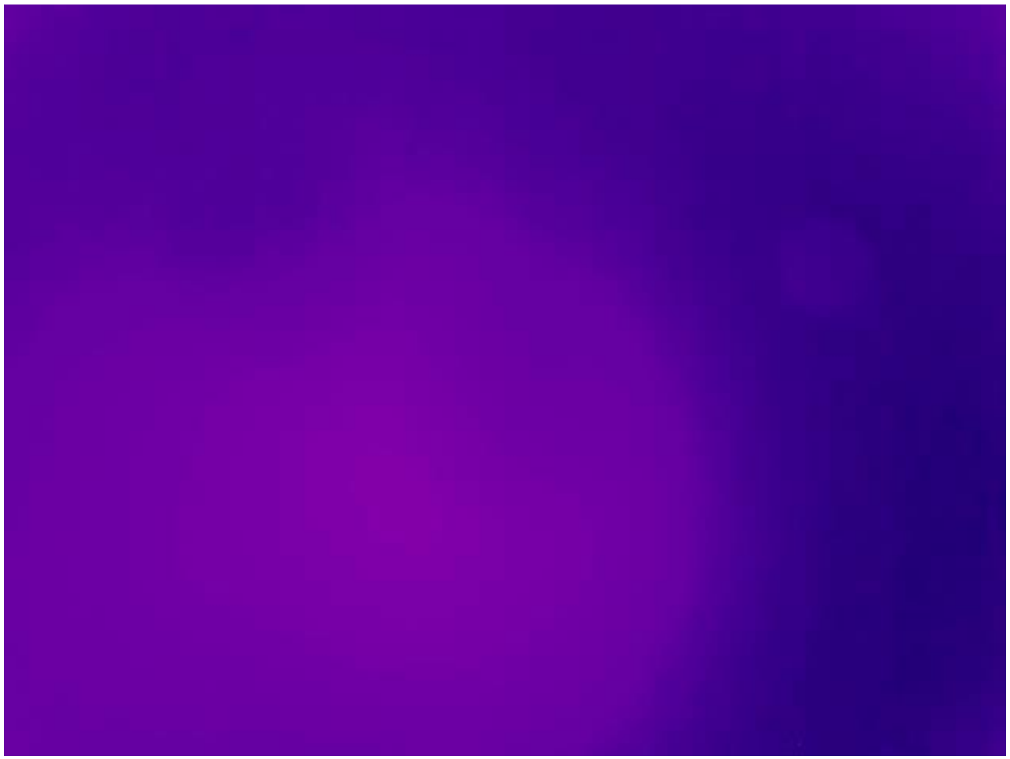
ALTERNATIVE TECHNOLOGIES



CALORIC MATERIALS

Example of elasto-caloric effect in a balloon

Cf Feynman’s lecture on thermodynamics

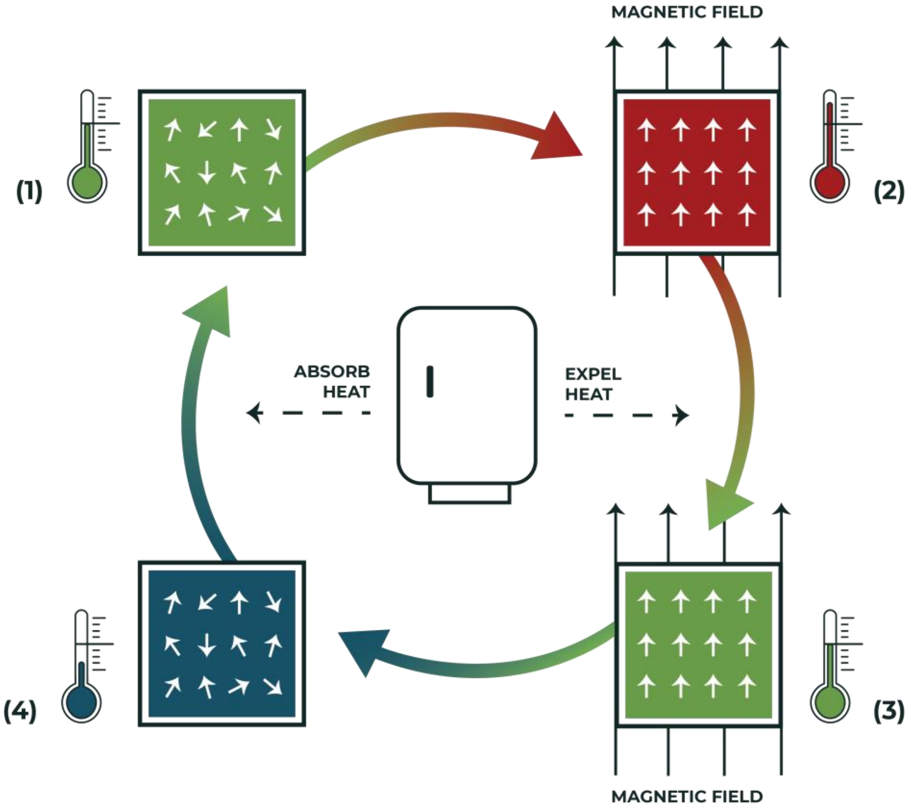


Courtesy X. Moya (Cambridge, UK)

- **Mechanical stress infers molecules rearrangement => entropy change**
- **Thermodynamic effect => positive then negative temperature change**
- **A cycle is required to make a fridge**

CALORIC MATERIALS – FOUR POSSIBLE EFFECTS

- Magnetocaloric
- Elastocaloric
- Barocaloric
- Electrocaloric



MAGNETIC COOLING PRINCIPLE.

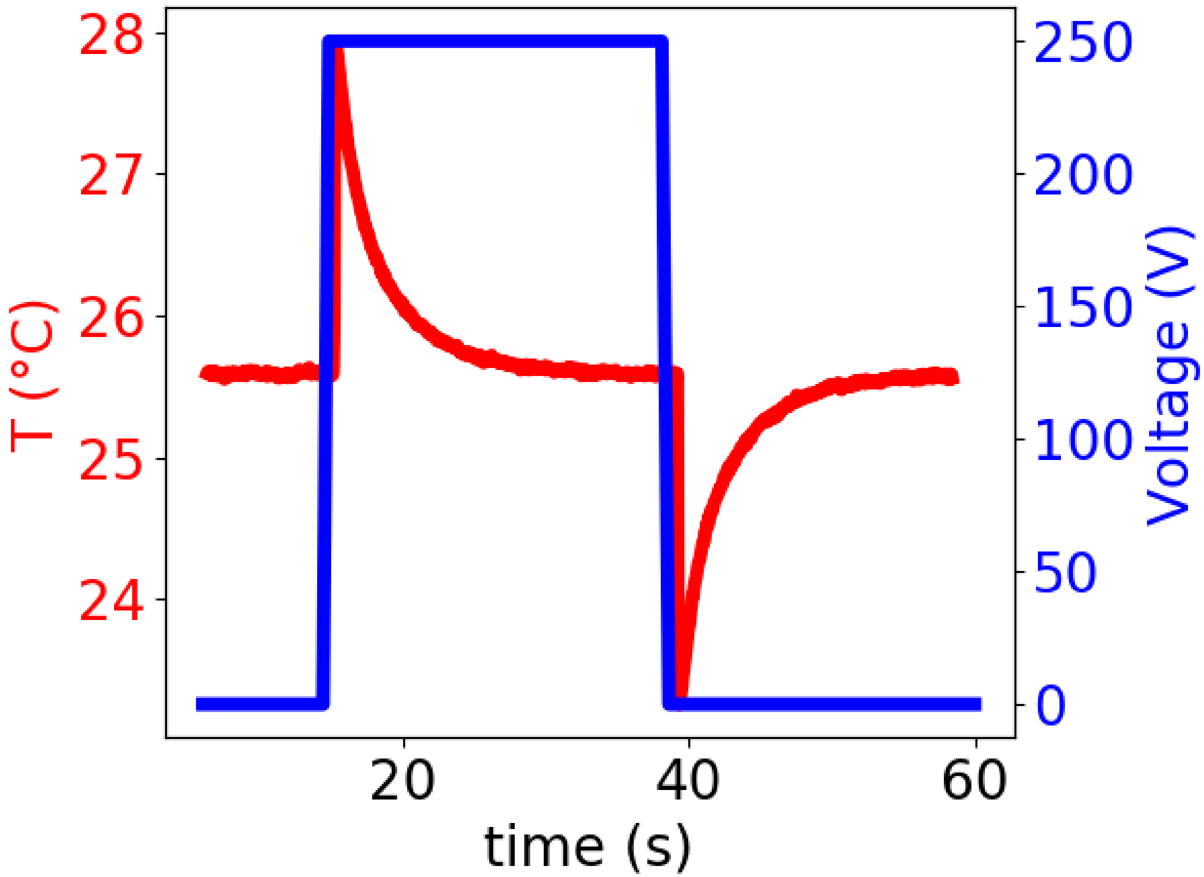
OUTLINE

- The electrocaloric effect
- Free energy description
- Electrocaloric materials
- A key element – multilayer capacitors
- Characterization
- Electrocaloric cooling systems

THE ELECTROCALORIC EFFECT

Ceramic $Pb(Sc,Ta)O_3$

THE ELECTROCALORIC EFFECT



FREE ENERGY SIMPLE ANALYSIS

- Free energy description of a ferroelectric material, close to ferro-para transition

$$G = G_0 + \frac{1}{2}\alpha(T - T_C)P^2 + \frac{1}{4}\beta P^4 - EP$$

Gibbs free energy *G*

Polarisation *P*

Temperature *T*

α, β: parameters of *G*

G at zero polarisation G_0

Electric field *E*

Transition Temperature = T_C

Entropy *S*

$$dG = -SdT - PdE$$

$$S = -\left(\frac{dG}{dT}\right)_P = -\frac{1}{2}\alpha P^2$$

=>

$$\Delta S = -\frac{1}{2}\alpha\left(P_{\max}^2 - P_{min}^2\right)$$

Large variations of S need large *α* and large variations of *P*

FREE ENERGY SIMPLE ANALYSIS

$$G = G_0 + \frac{1}{2}\alpha(T - T_C)P^2 + \frac{1}{4}\beta P^4 - EP$$

$$\left(\frac{dG}{dP}\right)_T \; equilibrium = 0$$

$$\left(\frac{dG}{dP}\right)_T = \alpha(T - T_c)P + \beta P^3 - E = 0$$

$$\left(\frac{d^2G}{dP^2}\right)_T = \alpha(T - T_c) + 3\beta P^2 = \left(\frac{dE}{dP}\right)_T = \frac{1}{\varepsilon}$$

ε – dielectric constant

If P is small $\varepsilon = \frac{1}{\alpha(T-T_c)}$

For large ΔS, at similar ΔP², we need large *α*, meaning small *ε*

ADIABATIC VARIATION OF TEMPERATURE

- Adiabatic conditions => $\Delta S_{total} = \Delta S_{lattice} + \Delta S_{dipoles} = 0$

$$T\Delta S_{lattice} = \rho C_p \Delta T$$

$$\Delta S_{dipoles} = -\frac{1}{2}\alpha\left(P_{\max}^2 - P_{min}^2\right)$$

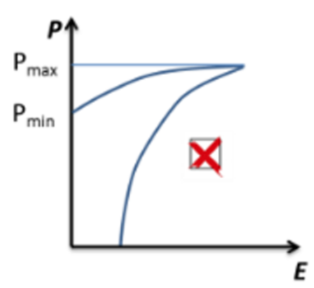
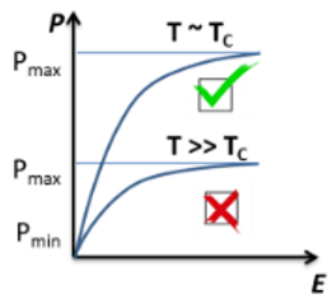
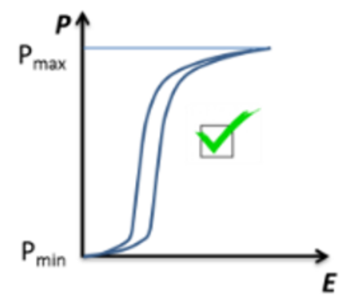
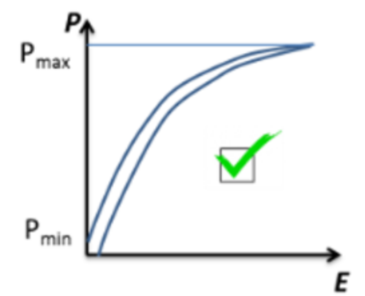
$$\Delta T = \frac{T}{2\rho C_p}\alpha\left(P_{\max}^2 - P_{min}^2\right)$$

- ρ $- density$
- C_p $- heat\ capacity$

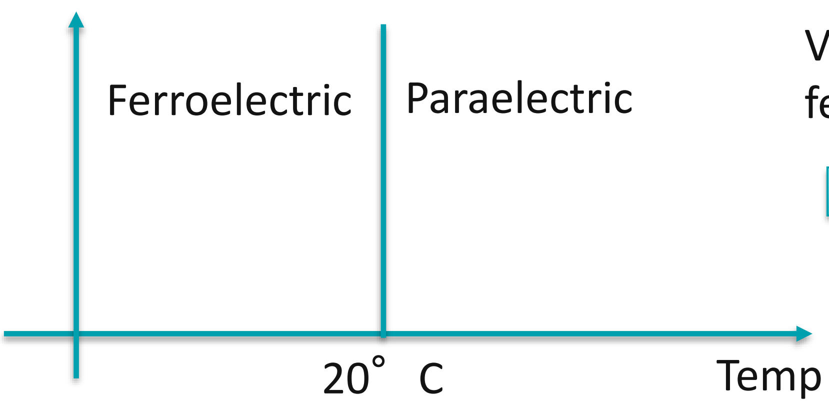
Large variations of T need large *α* and large variations of *P*

FREE ENERGY SIMPLE ANALYSIS

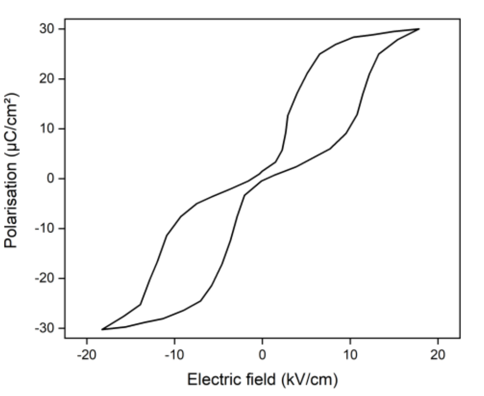
- **Large variations of S (and T) need large variations of *P***

Materials	*Ferroelectric*	*Paraelectric*	*Antiferroelectric*	*Relaxor*
$\Delta S \, \alpha \, (P_{max}^2 - P_{min}^2)$				

- **Interesting case – voltage induced phase transition in PST ceramics**



Voltage forces ferroelectricity

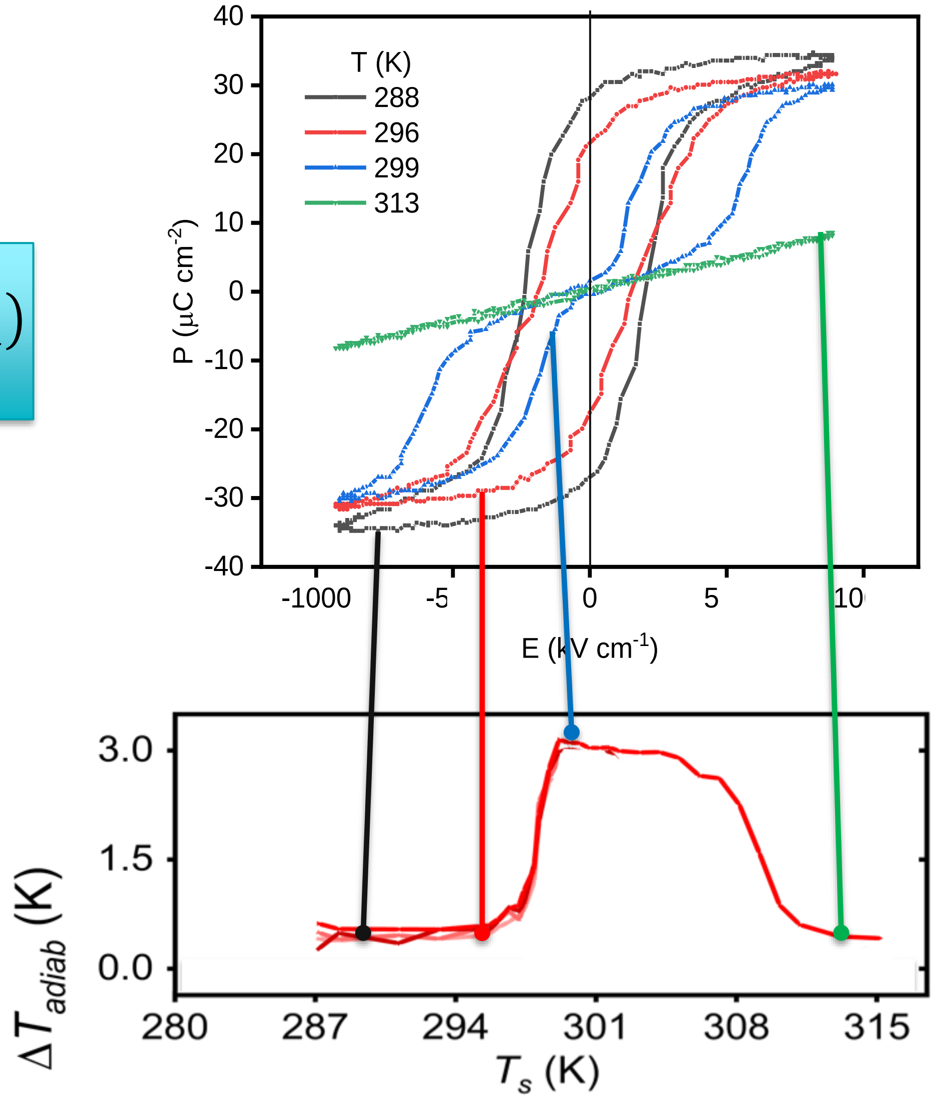


FREE ENERGY SIMPLE ANALYSIS

Interesting case – field induced phase transition in PST ceramics

$$\Delta T = \frac{T}{2\rho C_p}\alpha\left(P_{\max}^2 - P_{min}^2\right)$$

PE loop



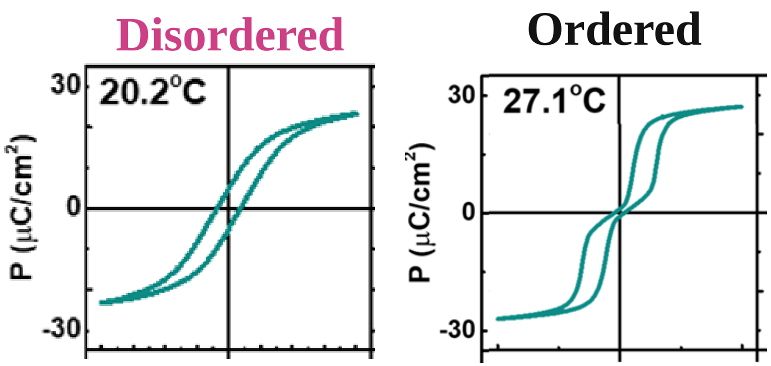
Electrocaloric effect at 10 kV cm^{-1}

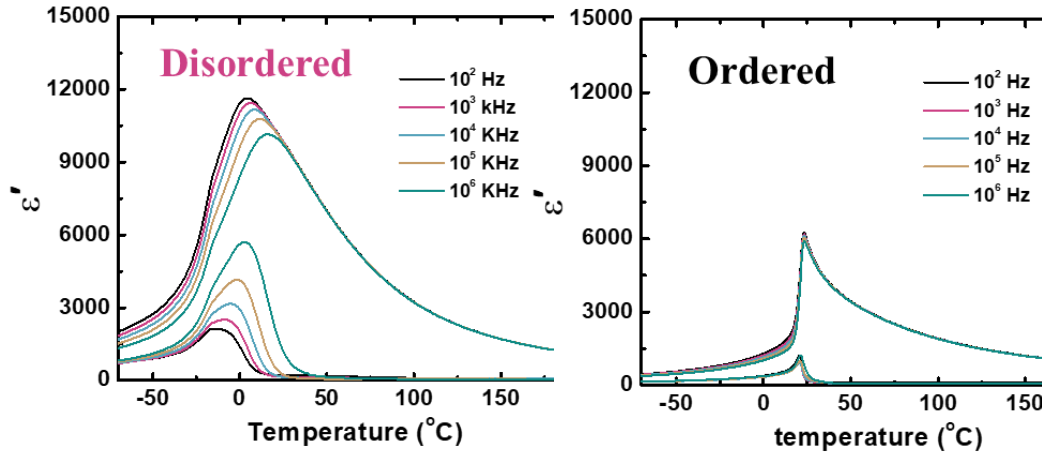
IR camera characterization

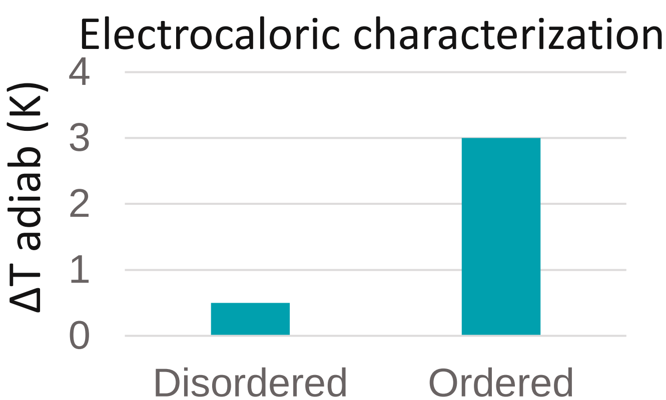
FREE ENERGY SIMPLE ANALYSIS

Example on lead scandium tantalate (PST)

For large ΔS, we need large ΔP² **and small *ε***







Large P and small *ε*
favour electrocaloric
effect

As expected by Landau's model

$$\Delta T = \frac{T}{2\rho C_p}\alpha\left(P_{\max}^2 - P_{min}^2\right)$$

ANOTHER COMMENT FROM LANDAU

Legendre transform

$$dU = TdS + EdP \quad \rightarrow \quad G = U - TS - EP$$

U: internal energy

G: Gibbs energy

Maxwell relation

$$dG = -SdT - PdE \quad \rightarrow \quad \left(\frac{\partial P}{\partial T}\right)_E = \left(\frac{\partial S}{\partial E}\right)_T$$

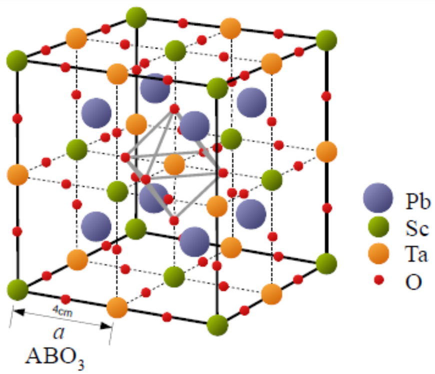
$$\text{isothermal } \Delta S = \int_{E_{min}}^{E_{max}} \left(\frac{\partial P}{\partial T}\right)_E dE$$

$$\text{adiabatic } \Delta T = -\frac{T}{c_E}\int_{E_{min}}^{E_{max}} \left(\frac{\partial P}{\partial T}\right)_E dE$$

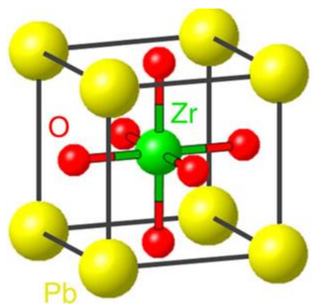
The variation of P with temperature is the engine of electrocalorics

ELECTROCALORIC MATERIALS

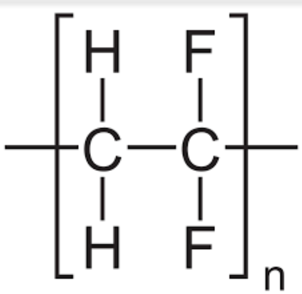
- Lead scandium tantalate
  - Probably the best EC ceramic
  - Phase transition at room T°



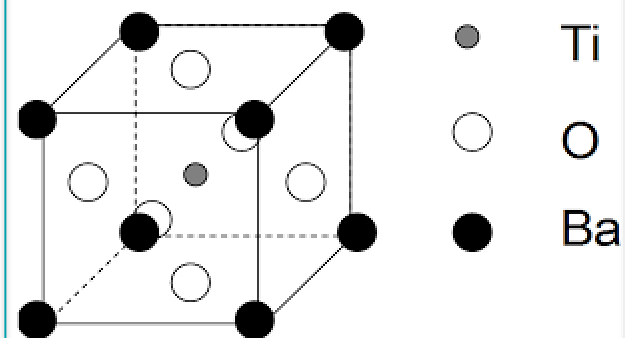
- Lead zirconate
  - Large <0 EC effect



- Polyvinylidene Difluoride PVDF
  - Alternative to ceramics



- Barium titanate
  - The lead free alternative
  - Phase transition at 120° C

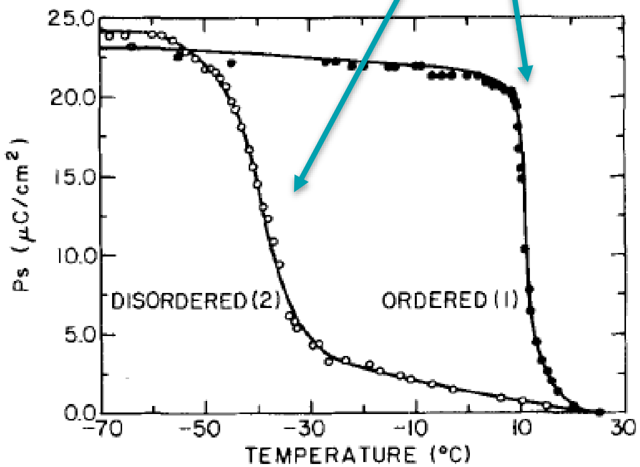


LEAD SCANDIUM TANTALATE - PST

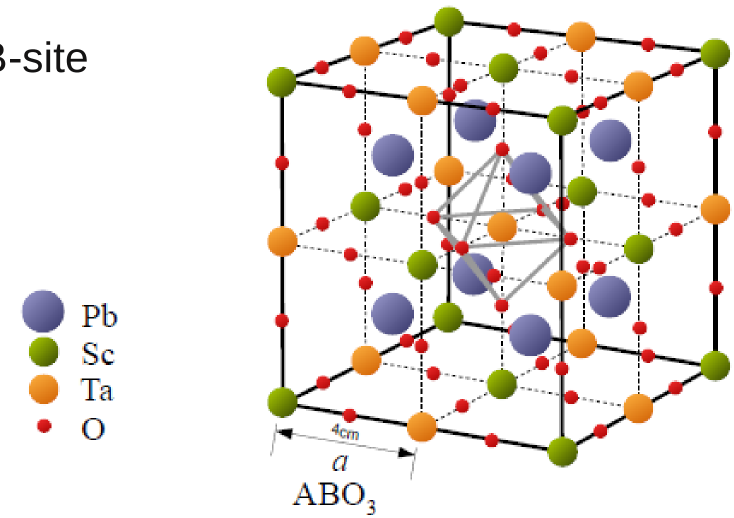
- Perovskite ABO3
- Ordered – regular alternance of Sc and Ta on B-site
- When ordered => 1st order phase transition
- When disordered => ferroelectric relaxor

$$\Delta S = \int_{E_{min}}^{E_{max}} \left(\frac{\partial D}{\partial T}\right)_E dE$$

Steep => large ΔS



N. Setter, JAP 1980



RESEARCH ARTICLE | JULY 09 2008

The role of B-site cation disorder in diffuse phase transition behavior of perovskite ferroelectrics

N. Setter; L. E. Cross

Check for updates

Journal of Applied Physics 51, 4356–4360 (1980)
https://doi.org/10.1063/1.328296

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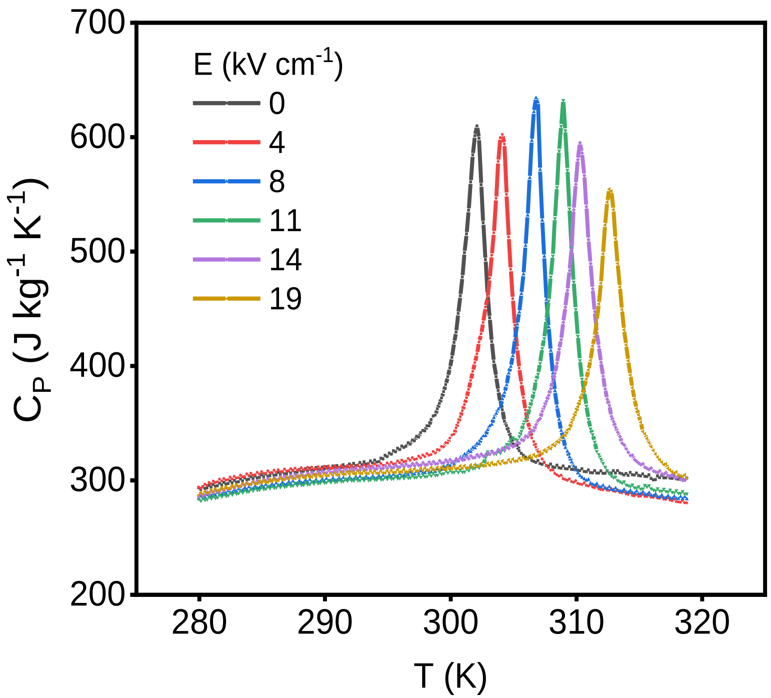
LEAD SCANDIUM TANTALATE - PST

Calorimetry on PST bulk

- First order phase transition => latent heat at the transition
- Transition temperature depends on electric field (isofield DSC)



Max values

$L = 1000\ J\ kg^{-1}$

$\Delta S = 3.4\ J\ kg^{-1}\ K^{-1}$

$\Delta T_{adiab} = 3.7\ K$

nature COMMUNICATIONS

ARTICLE

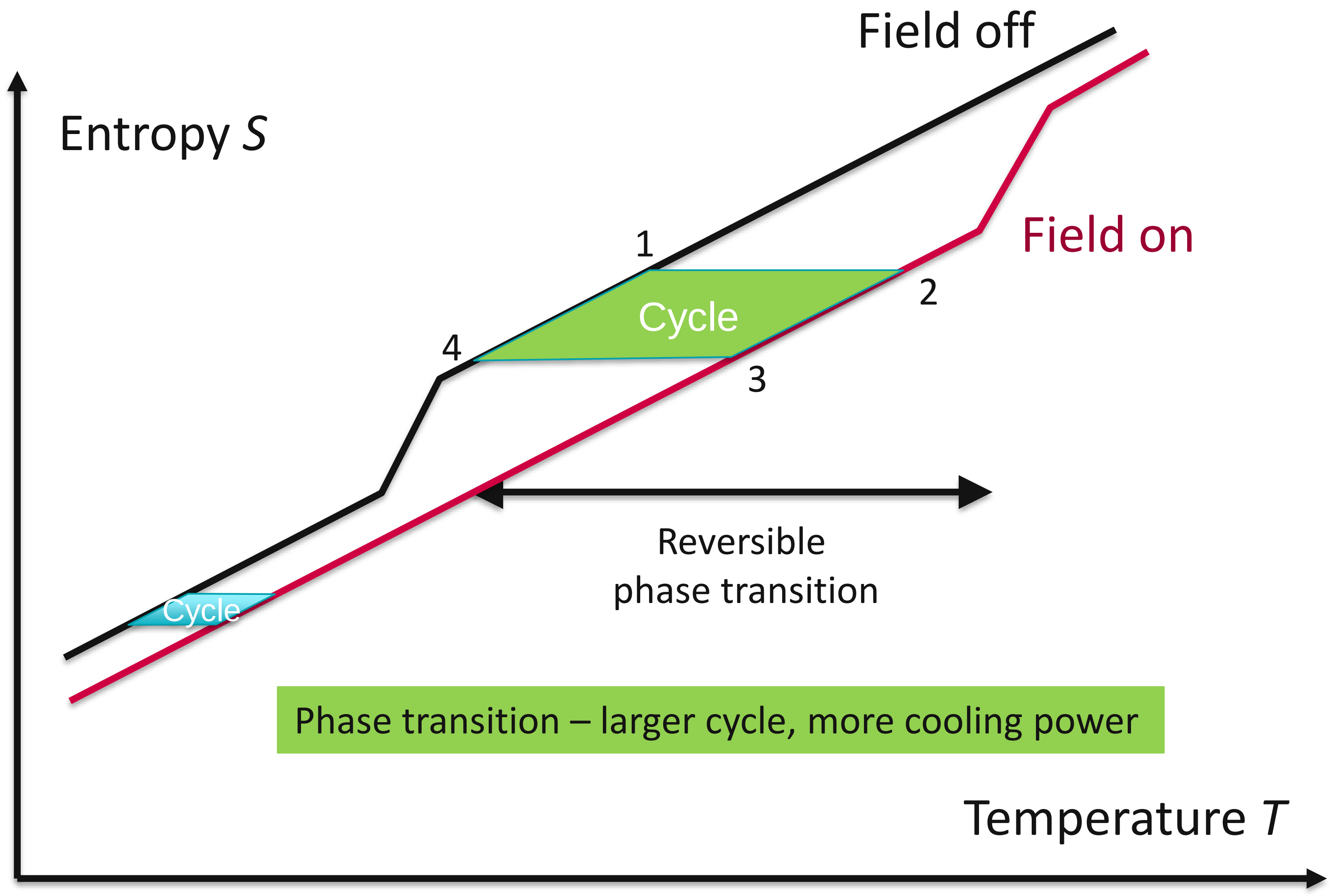
https://doi.org/10.1038/s41467-021-23354-y OPEN

Giant electrocaloric materials energy efficiency in highly ordered lead scandium tantalate

Youri Nouchokgwe[1,2], Pierre Lheritier[1], Chang-Hyo Hong[3], Alvar Torelló[1,2], Romain Faye[1], Wook Jo[3], Christian R. H. Bahl[4] & Emmanuel Defay[1]

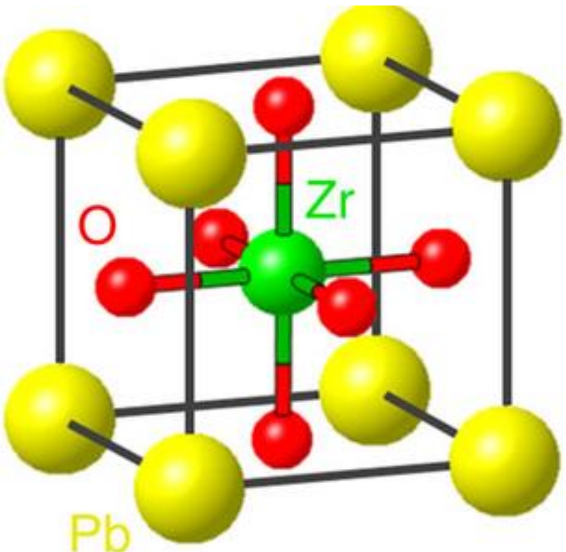
LEAD SCANDIUM TANTALATE - PST

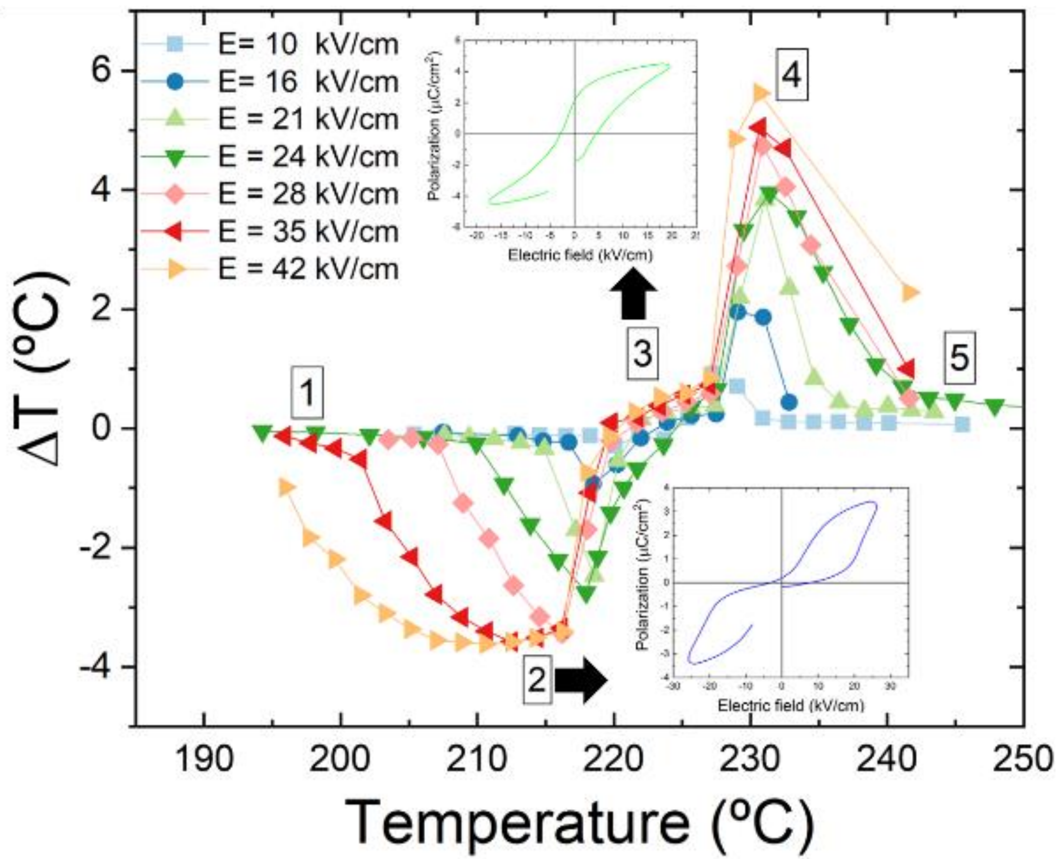
Entropy change with temperature at constant field (isofield)



LEAD ZIRCONATE PZO

- Perovskite ABO3





PHYSICAL REVIEW B **103**, 054112 (2021)

Positive and negative EC effects 20 K apart !

Origin of large negative electrocaloric effect in antiferroelectric $PbZrO_3$

Pablo Vales-Castro,[1,*] Romain Faye,[2] Miquel Vellvehi,[3] Youri Nouchokgwe,[2,4] Xavier Perpiñà,[3] J. M. Caicedo,[1] Xavier Jordà,[3] Krystian Roleder,[5] Dariusz Kajewski,[5] Amador Perez-Tomas,[1] Emmanuel Defay,[2] and Gustau Catalan[1,6,†]

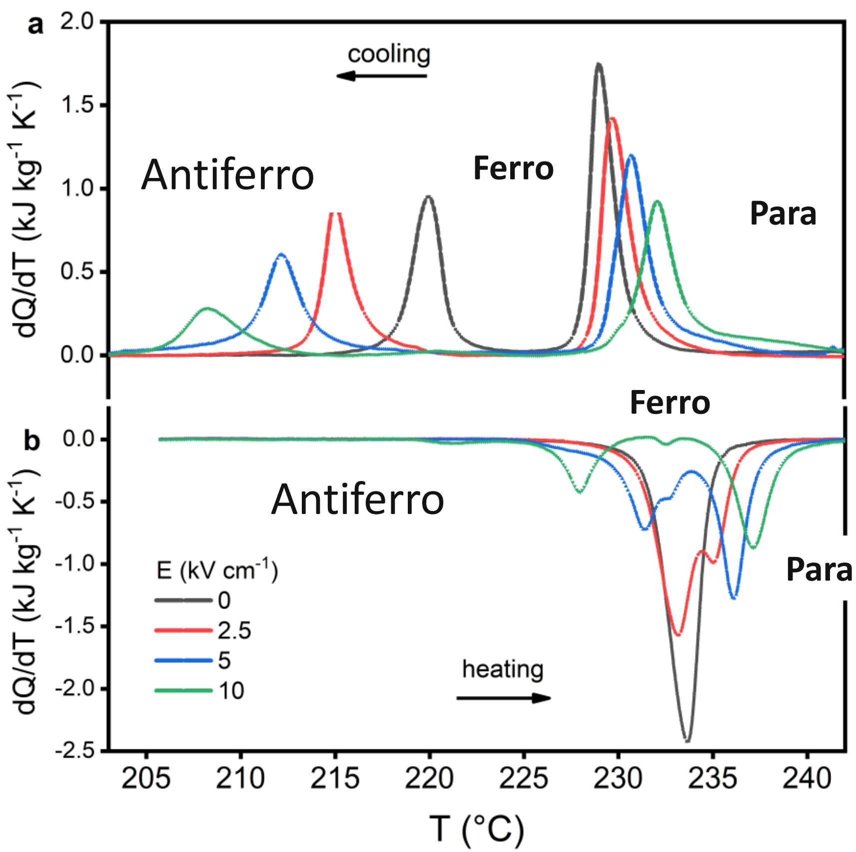
[1]*Catalan Institute of Nanoscience and Nanotechnology (ICN2), Campus Universitat Autonoma de Barcelona, Bellaterra 08193, Spain*

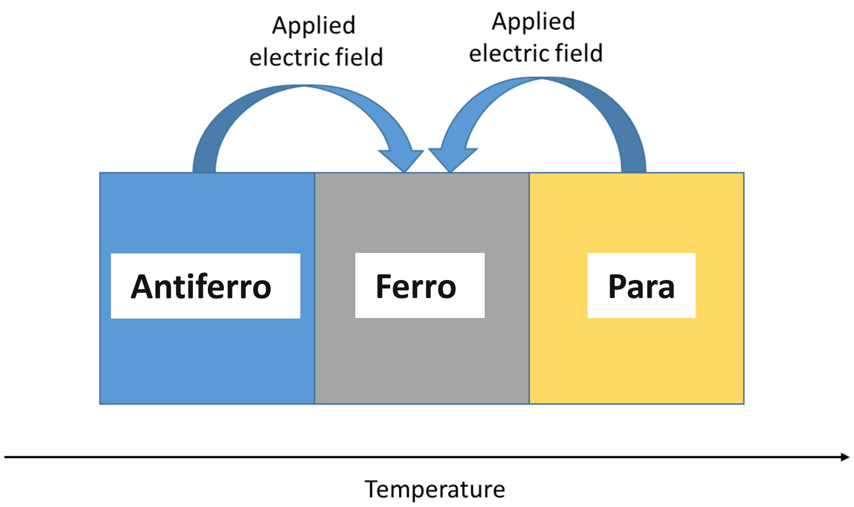
ICN2

LEAD ZIRCONATE PZO

Transition driven by EC field

- Differential Scanning Calorimetry





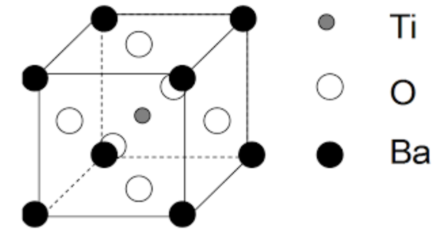
Electric field always brings to ferro !
antiferro to ferro => negative EC effect
Para to ferro => positive EC effect

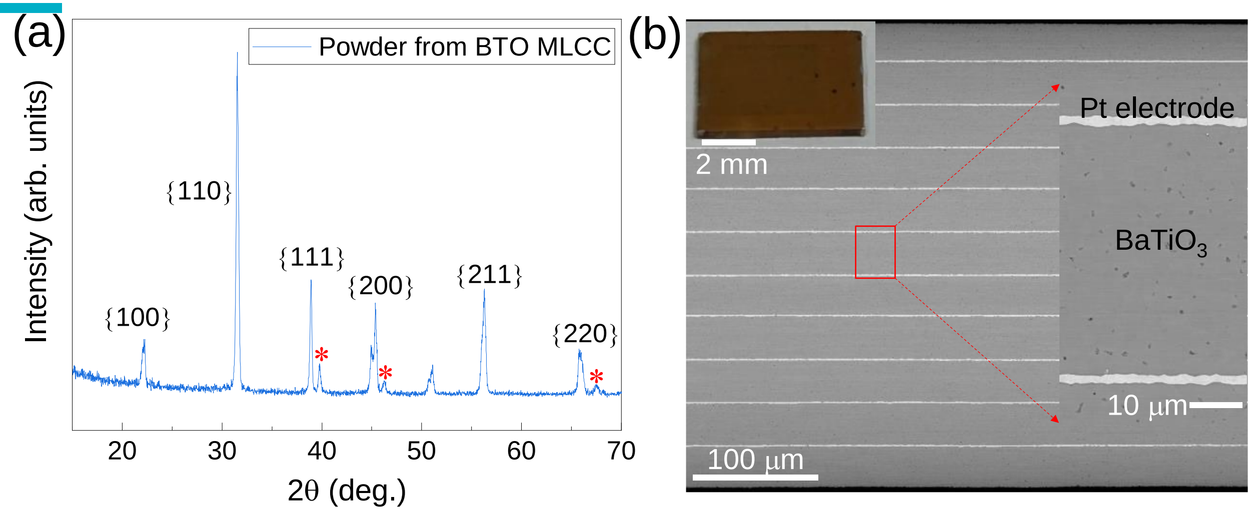
Phase transition is key

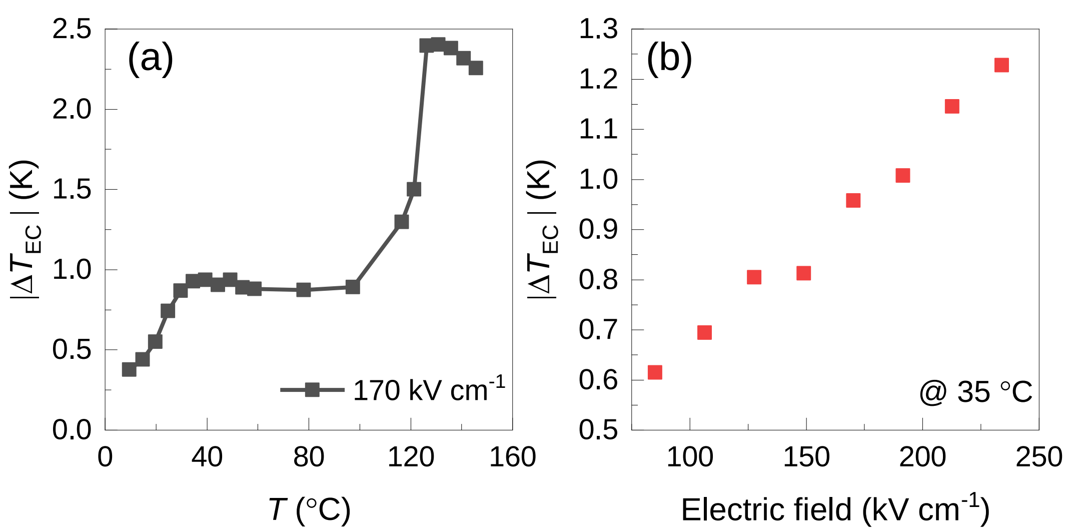
Pablo Vales et al., PRB 2020

BARIUM TITANATE

Recent work on multilayers made of $BaTiO_3$







- ΔT max = 2.4 K @ 120 ° C
- ΔT max = 1.25 K @ 35 ° C
- Two phase transitions

J. Phys. Energy 5 (2023) 024017 https://doi.org/10.1088/2515-7655/acc972

Journal of Physics: Energy

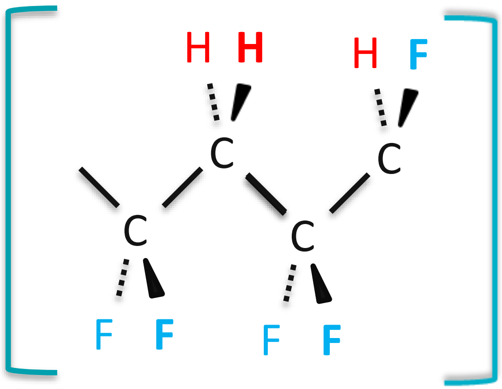
PAPER

Electrocaloric effect in $BaTiO_3$ multilayer capacitors with first-order phase transitions

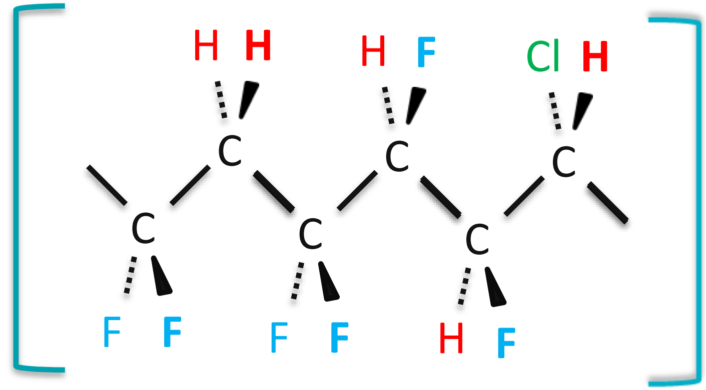
Junning Li[1], Alvar Torelló[1], Youri Nouchokgwe[1], Torsten Granzow[1], Veronika Kovacova[1], Sakyo Hirose[2] and Emmanuel Defay[1,*]

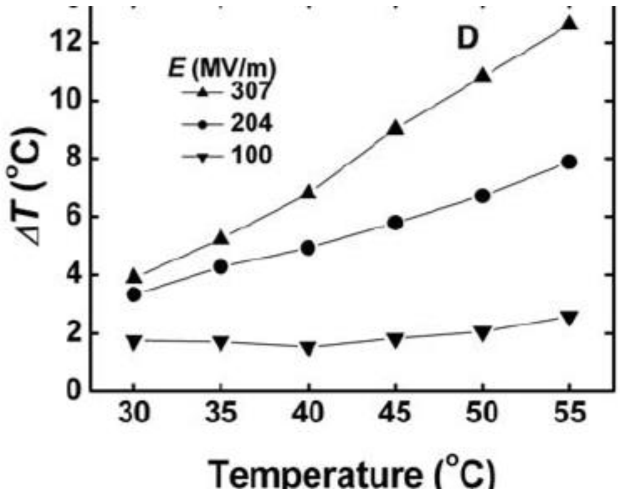
PVDF (Polyvinylidene Difluoride)

P(VDF-TrFE)



P(VDF-TrFe-CFE)





- Strong EC effect in P(VDF-TrFE) and P(VDF-TrFE-CFE)
- Neese et al., *Science,* 2008, Pennsylvania State Univ.
- ΔT_{max} = 12 K
- ΔS_{max} = 65 J/(KgK)
- Electric field$_{max}$=3MV/cn

Science AAAS

Large Electrocaloric Effect in Ferroelectric Polymers Near Room Temperature
Bret Neese, *et al.*
Science **321**, 821 (2008);
DOI: 10.1126/science.1159655

Electrocalorics comparison

Material		PZT 95/5	P(VDF-TrFE)
Reference		*Mischenko, Science 2006*	*Neese et al., Science, 2008*
$\lvert\Delta S_m\rvert$	[J K^{-1} kg^{-1}]	8	65
$\lvert\Delta S_v\rvert$	[kJ K^{-1} m^{-3}]	62	97
$\lvert\Delta T\rvert$	[K]	12	12
$\lvert\Delta E\rvert$	[kV cm^{-1}]	480	3000
$\varepsilon/\varepsilon_0$ max	[-]	750	70
T_C	[°C]	222	80

10 x more entropy change in polymers for the same mass

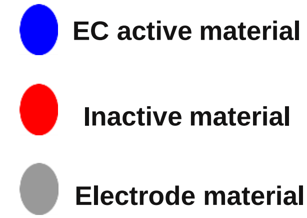
Science AAAS

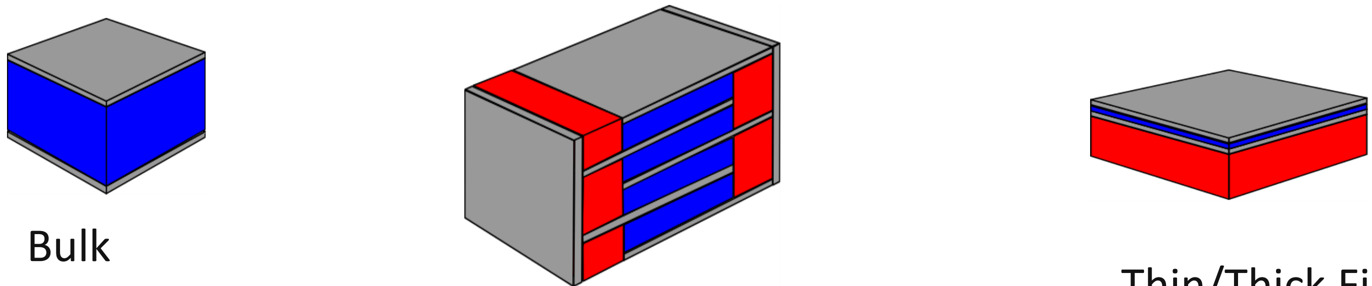
Giant Electrocaloric Effect in Thin-Film PbZr0.95Ti 0.05O3
A. S. Mischenko, *et al.*
Science **311**, 1270 (2006);
DOI: 10.1126/science.1123811

Science AAAS

Large Electrocaloric Effect in Ferroelectric Polymers Near Room Temperature
Bret Neese, *et al.*
Science **321**, 821 (2008);
DOI: 10.1126/science.1159655

A KEY ELEMENT – MULTILAYER CAPACITORS





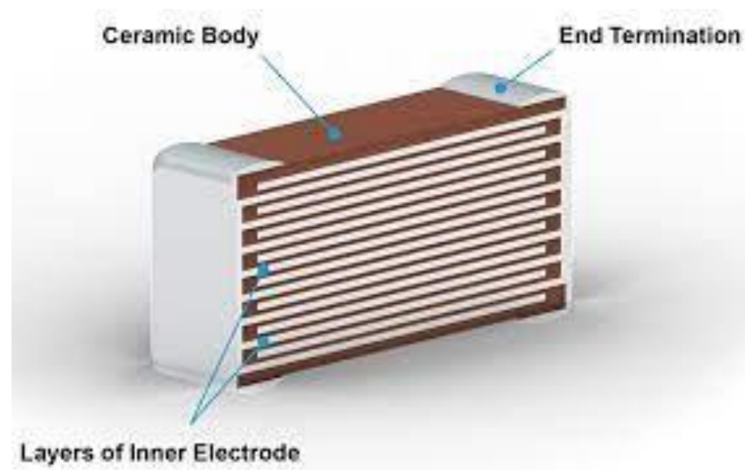
Bulk

Thin/Thick Film

Multi-Layer Capacitor (MLC)

MLCs combine bulk and films properties

- Macroscopic object
- Large field can be applied (supercritical regime)
- Inner metal electrodes increase effective thermal conductivity

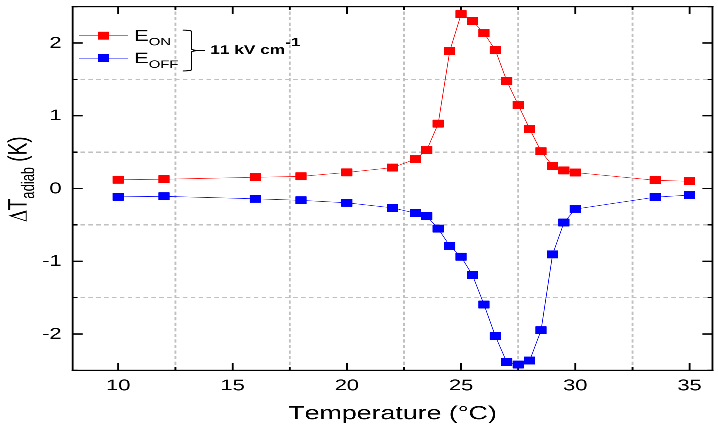


Best samples for electrocaloric prototypes

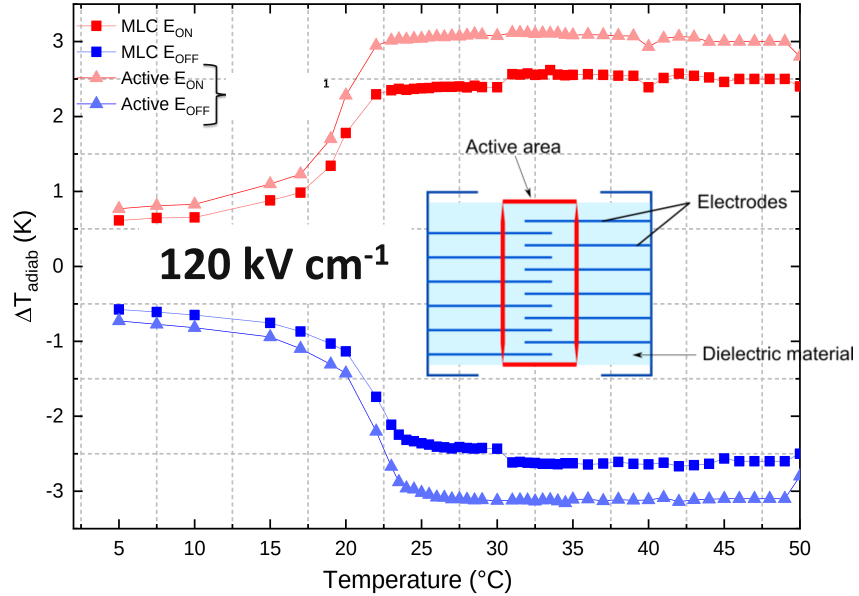
MLC BEHAVIOUR COMPARED TO BULK

BULK PST

11 kV cm^{-1}



MLC PST



- Much larger field applied in MLC
- Much larger active temperature range in MLC
- Similar heat generated

nature COMMUNICATIONS

ARTICLE

https://doi.org/10.1038/s41467-021-23354-y OPEN

Giant electrocaloric materials energy efficiency in highly ordered lead scandium tantalate

Youri Nouchokgwe[1,2], Pierre Lheritier[1], Chang-Hyo Hong[3], Alvar Torelló[1,2], Romain Faye[1], Wook Jo[3], Christian R. H. Bahl[4] & Emmanuel Defay[1]

Scripta Materialia 219 (2022) 114873

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journal homepage: www.journals.elsevier.com/scripta-materialia

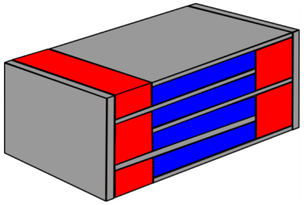
ELSEVIER

Materials efficiency of electrocaloric lead scandium tantalate multilayer capacitors

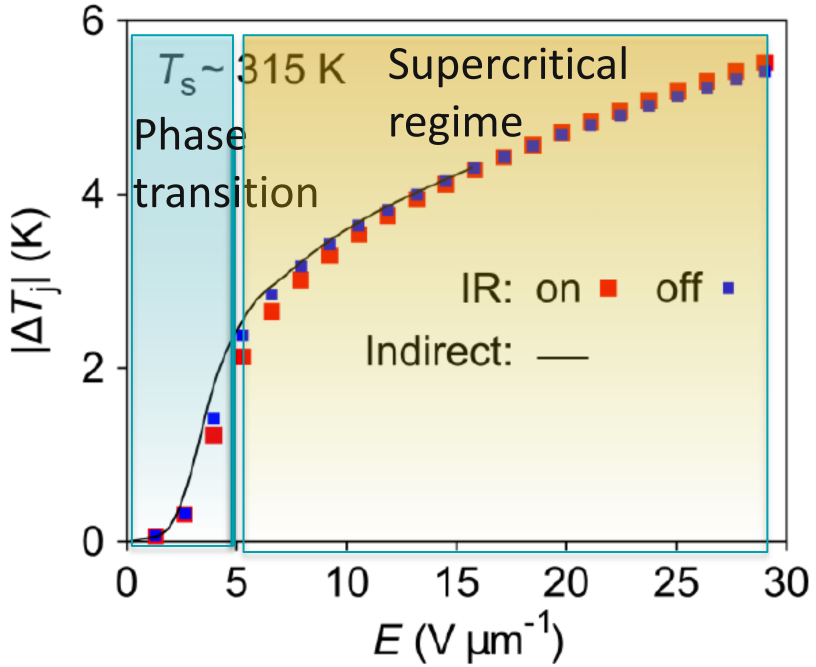
Youri Nouchokgwe[a,b,*], Pierre Lheritier[a], Tomoyasu Usui[c], Alvar Torello[a,b], Asmaa El Moul[a], Veronika Kovacova[a], Torsten Granzow[a], Sakyo Hirose[c], Emmanuel Defay[a,*]

ROLE OF PHASE TRANSITIONS ON EC EFFECT

Example on PST multilayer capacitors (MLCs)



- Two regimes in ΔT = f(electric field) at constant temperature



Article

Large electrocaloric effects in oxide multilayer capacitors over a wide temperature range

nature

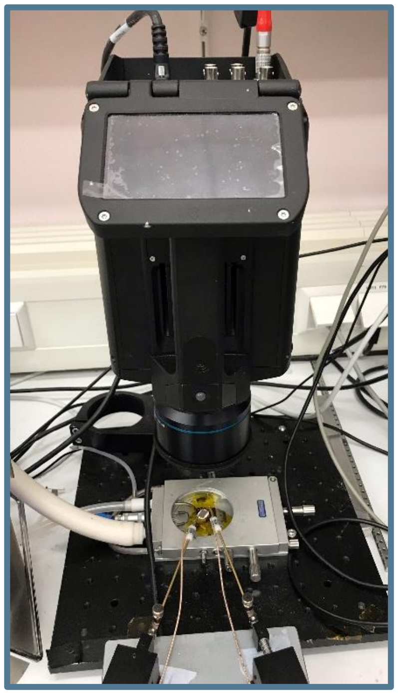
https://doi.org/10.1038/s41586-019-1634-0
Received: 27 September 2018

B. Nair[1], T. Usui[2], S. Crossley[1], S. Kurdi[1], G. G. Guzmán-Verri[1,3,4], X. Moya[1]*, S. Hirose[2]* & N. D. Mathur[1]*

MLC PST
ΔT max = 5.5 K

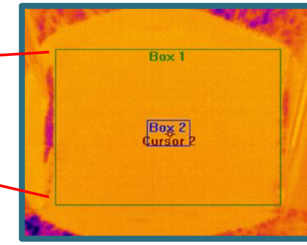
ELECTROCALORIC CHARACTERIZATION

Direct method (IR imaging)

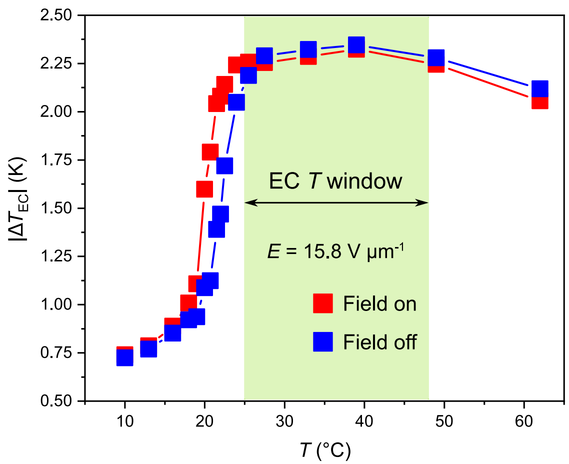


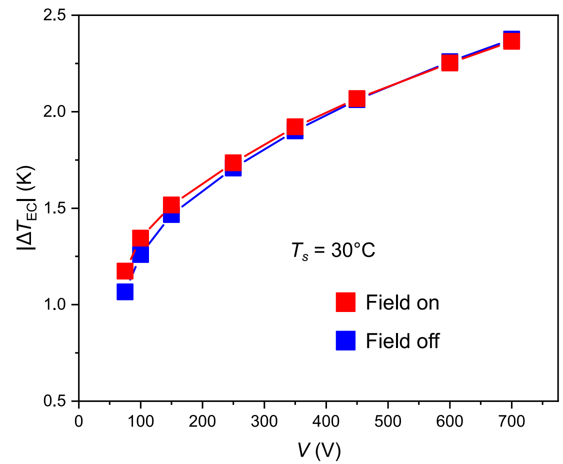
Direct method (IR imaging)

PST MLC (emissivity = 1)



Entire sample area





ΔT_{EC} = **2.2 K** from 25 to 50 °C at 600 V

Other means

- Thermocouple directly on devices (big samples)
- DSC (big samples)
- Indirect methods, from Maxwell (ergodic materials, thin films)

Cooling Systems

What do we need to build a cooler?

1 Cooling mechanism

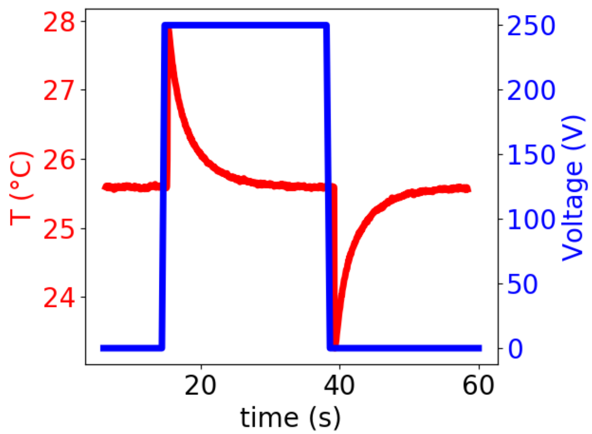
- Vapour compression / expansion
- Electrocaloric Effect

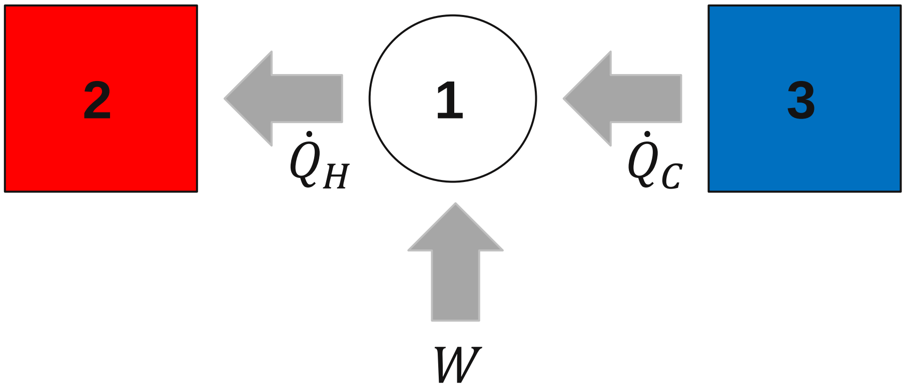
2 Hot side

- To release the heat generated by the active material to the surroundings.

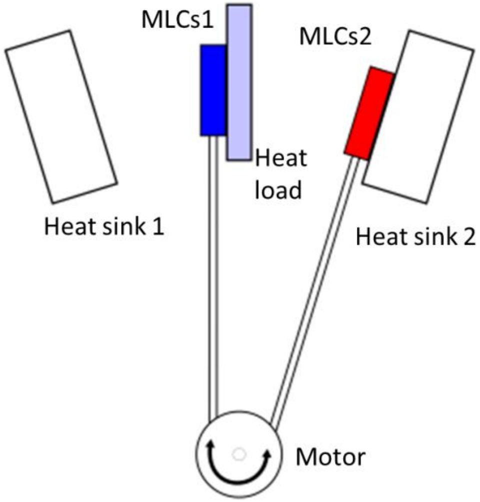
3 Cold side

- The active material is cooled down and absorbs heat from a cooling load.

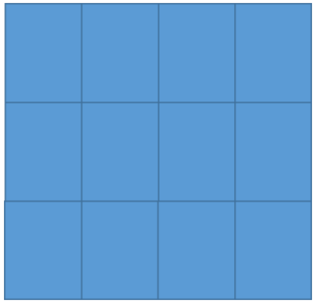


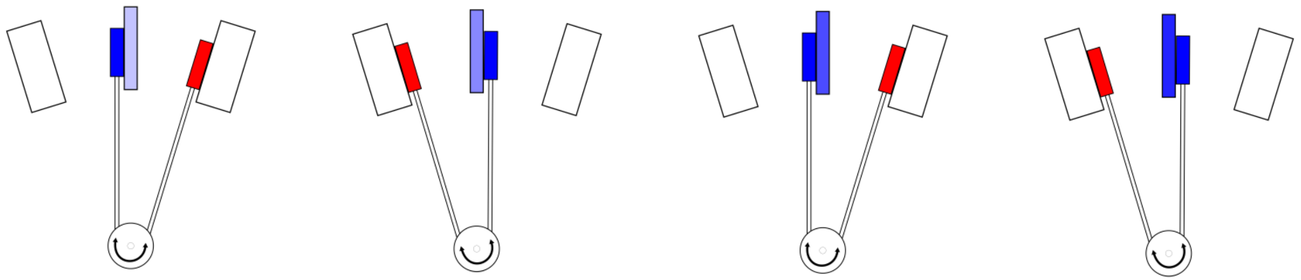


THE SLAPPING MACHINE



2 plates of 12 $BaTiO_3$ MLCs
(3 x 4)





ARTICLE

DOI: 10.1038/s41467-018-04027-9 OPEN

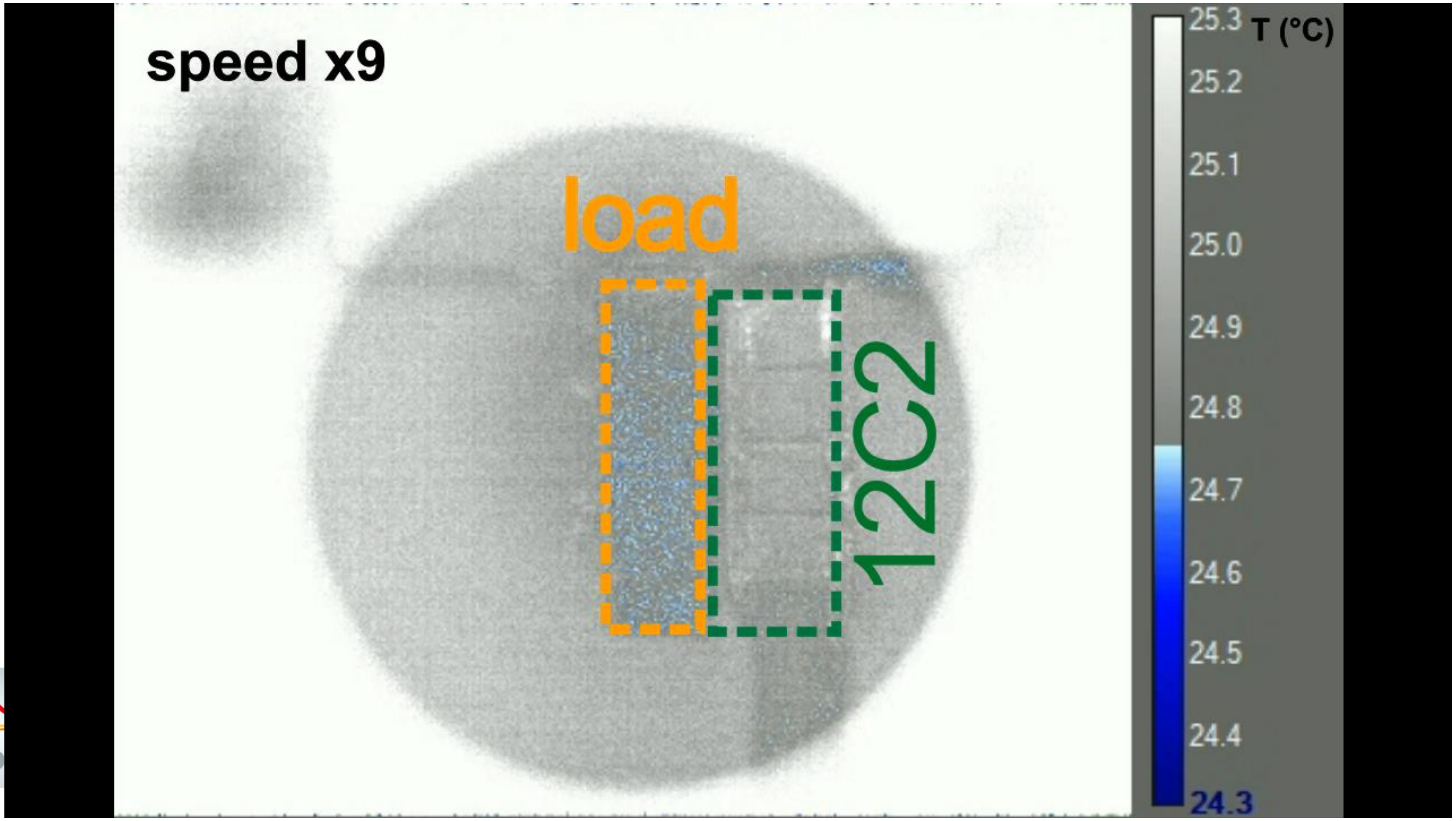
Enhanced electrocaloric efficiency via energy recovery

E. Defay[1,2,3,4], R. Faye[1], G. Despesse[2], H. Strozyk[1], D. Sette[1], S. Crossley[3,5], X. Moya[3] & N.D. Mathur[3]

PROTOTYPE VIDEO

- Final ΔT device = 0.35 K

The slapping machine



nature COMMUNICATIONS

ARTICLE

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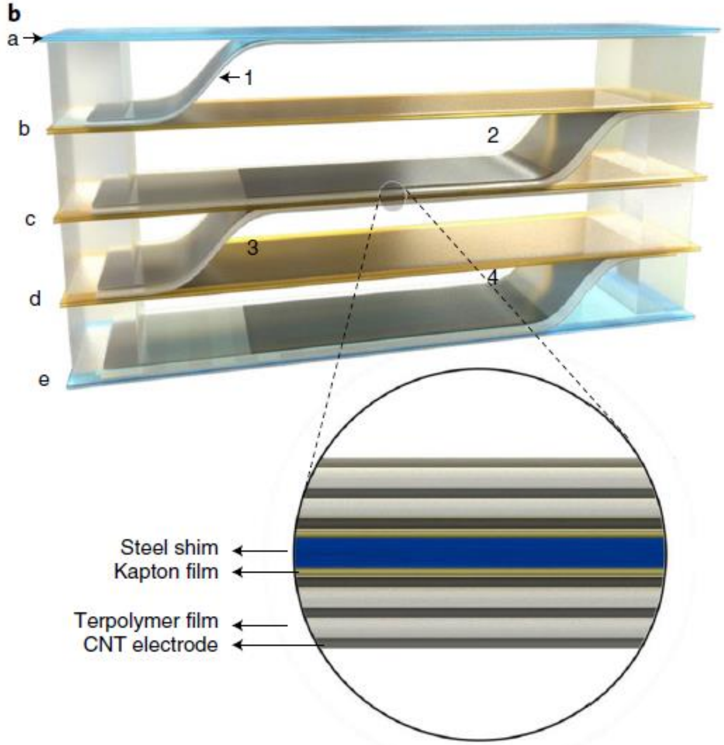
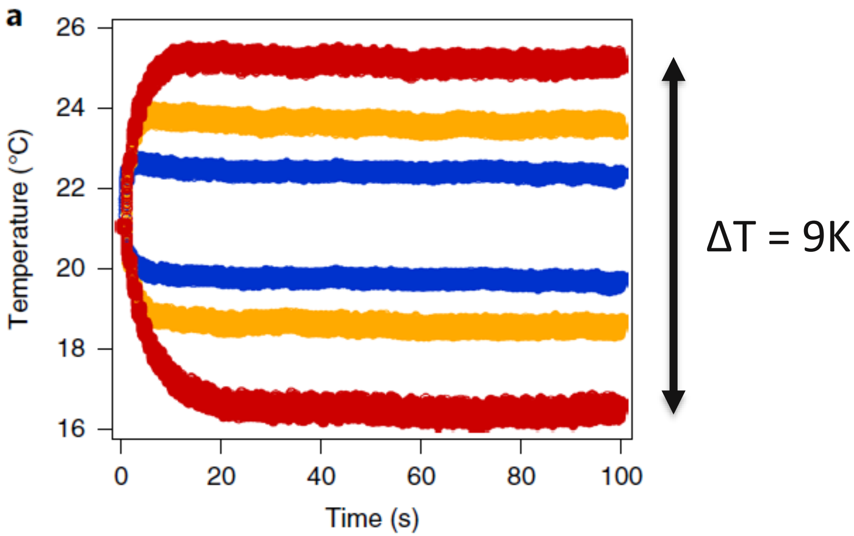
Cascading principle – "pass-the-parcel"





Based on PVDF
Maximum ΔT device = 9 K

nature energy
ARTICLES
https://doi.org/10.1038/s41560-020-00715-3
Check for updates

A cascade electrocaloric cooling device for large temperature lift

Yuan Meng[1], Ziyang Zhang[1], Hanxiang Wu[1], Ruiyi Wu[2], Jianghan Wu[1], Haolun Wang[1] and Qibing Pei[1,3]

nature energy
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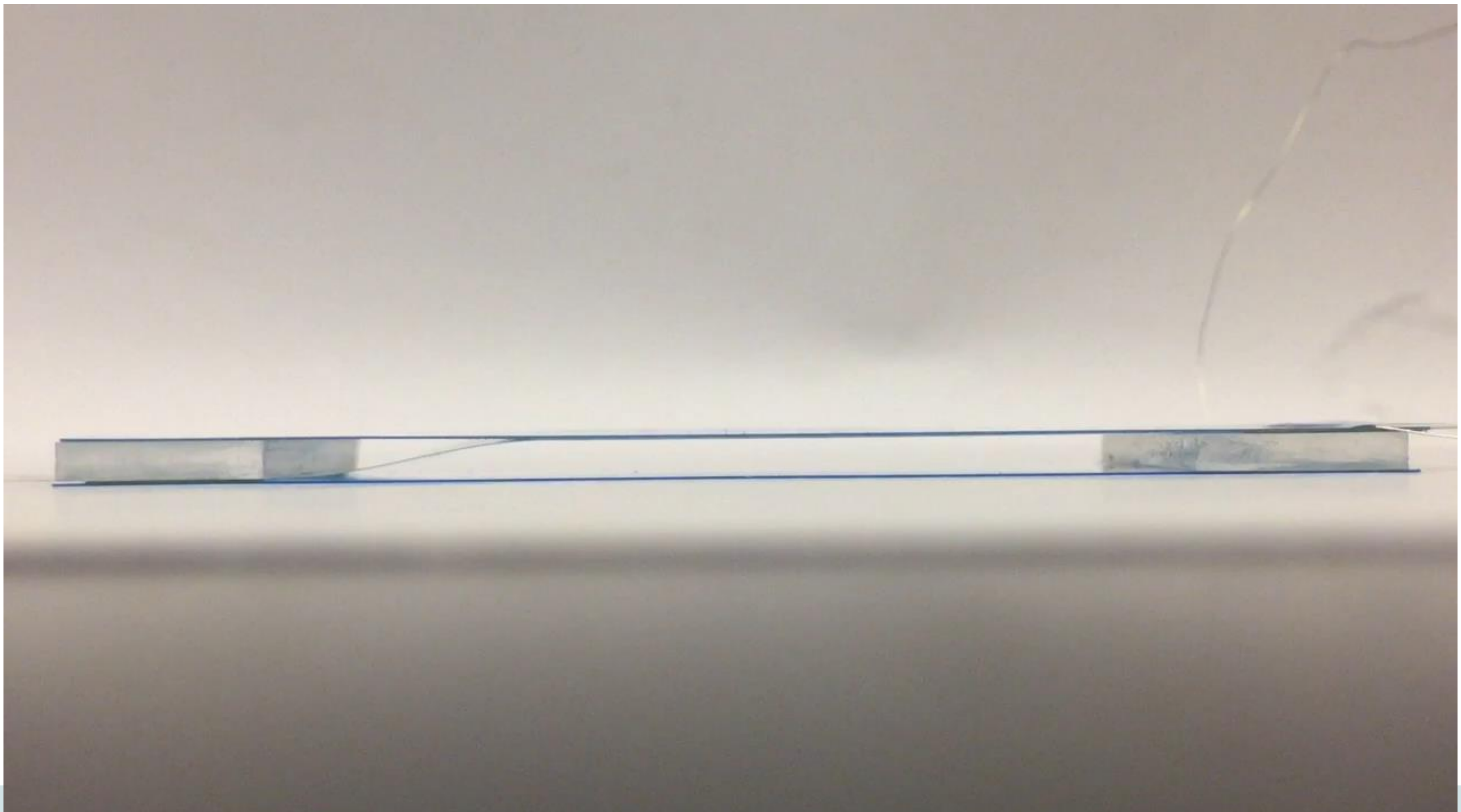
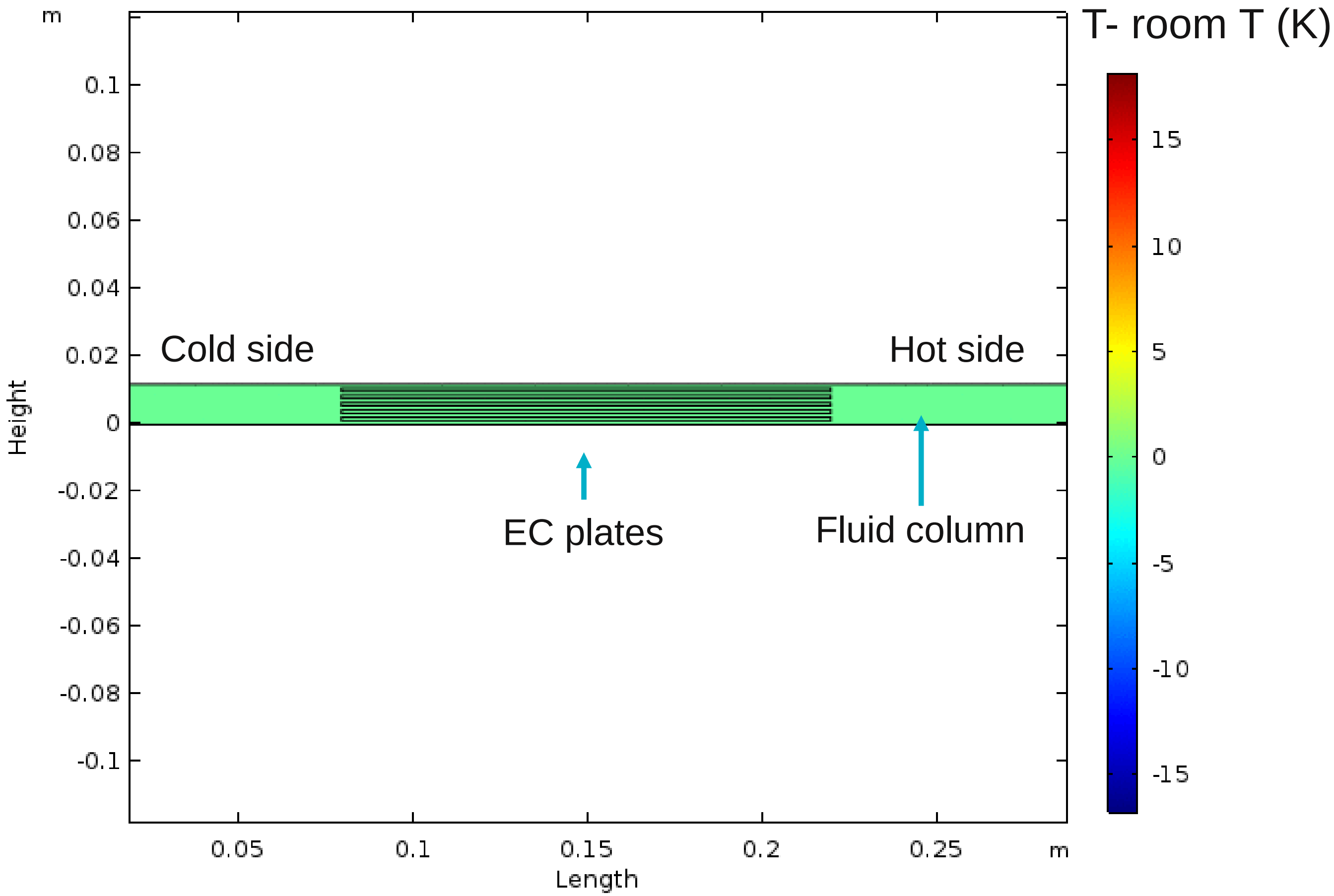


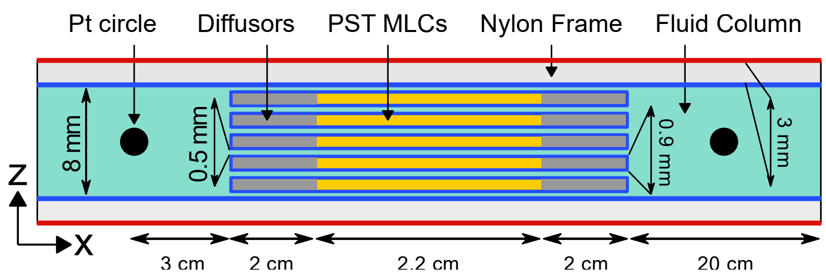
Electrocaloric Cooling

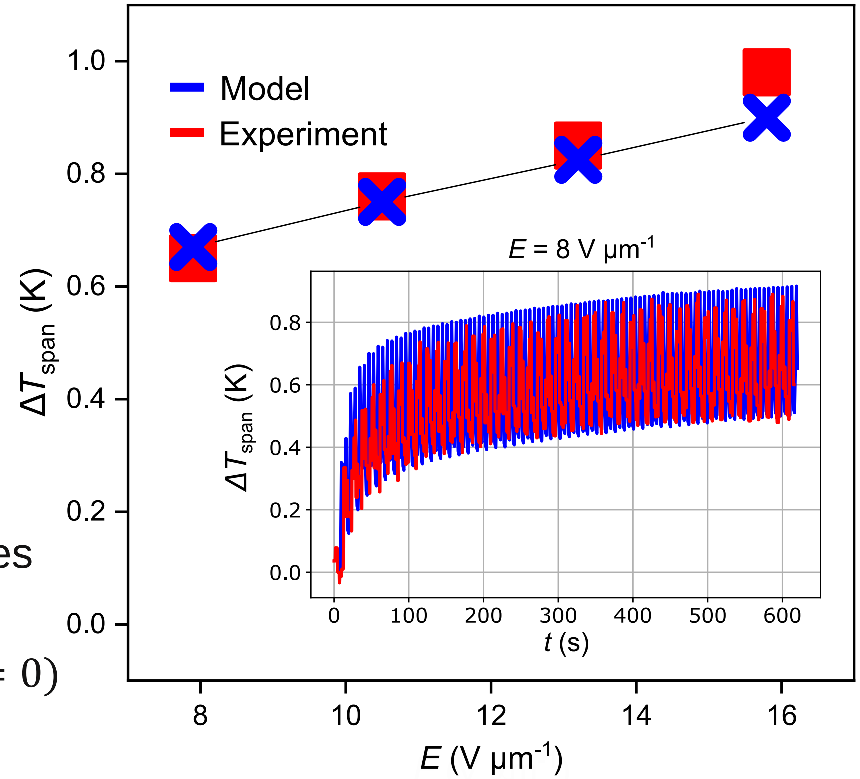
Fluid-based regenerators



ELECTROCALORIC REGENERATOR – MODELLING

2D representation

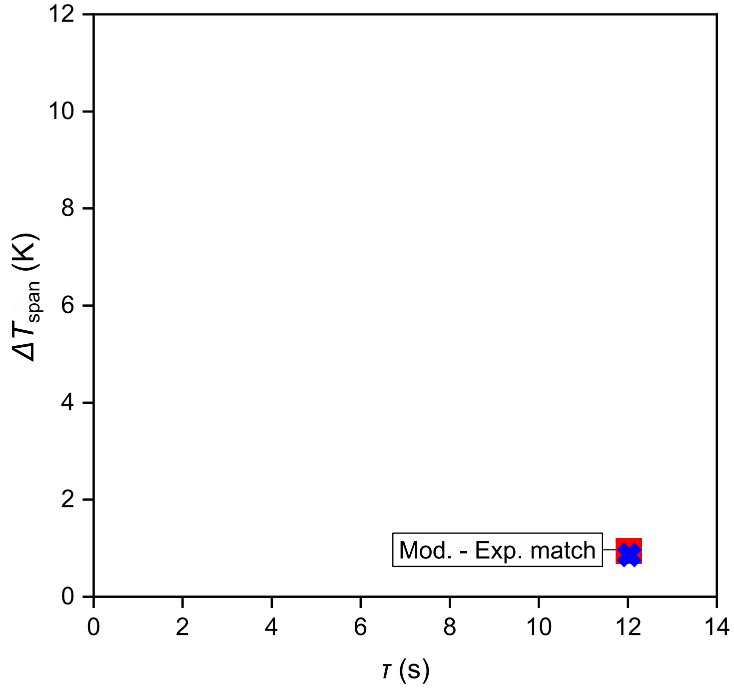




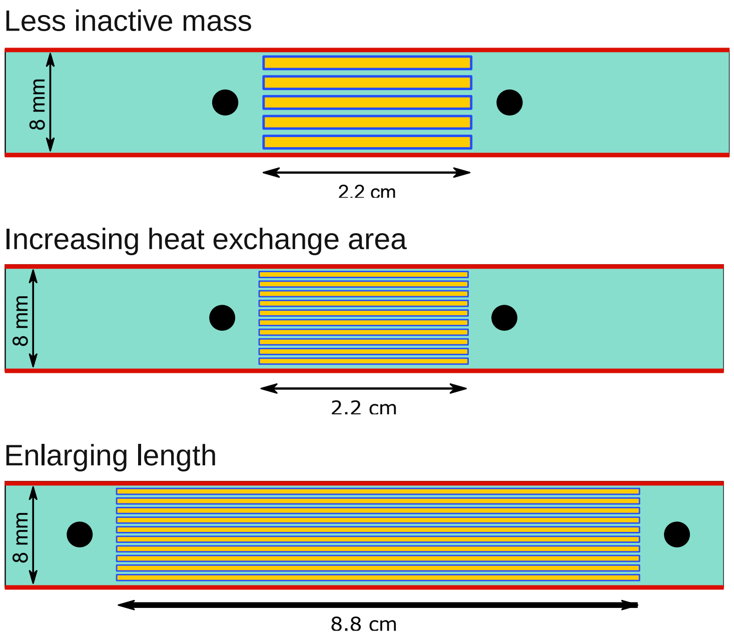
- Coupling **Heat transfer** + **Fluid dynamics** modules
- **Adiabatic conditions** in the **exterior walls** ($\frac{dq_{\vec{n}}}{dt} = 0$)
- **No – Slip boundary** in the **fluid wall** ($u_{\vec{n}} = 0$).
- **Average Temperature** of **Platinum Circle**

ELECTROCALORIC REGENERATOR – MODELLING

Road map

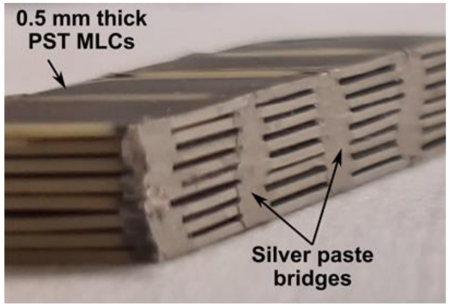


Model configurations



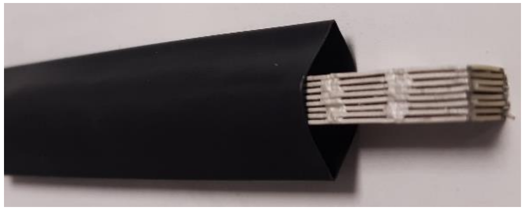
ELECTROCALORIC REGENERATOR – FABRICATION

Self standing Parallel-Plate



- 128 0.5 mm thick PST MLCs (16 col x 8 row)
- Double-sided tape spacers
- Silver paste electrodes

Shrinking Polymer tube to seal structure

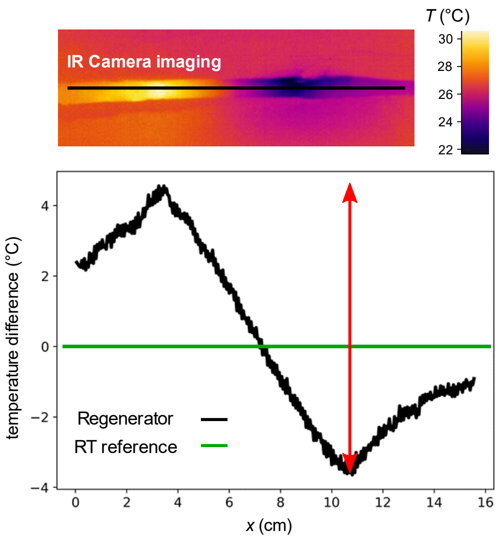


Gluing Polymer tube ends to fluid tubing

Convenient solution

1. Negligible thermal mass
2. Minimum dead volume
3. Low cost, flexible structure

ELECTROCALORIC REGENERATOR – RESULTS

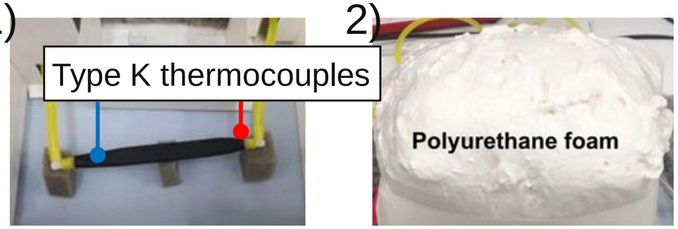


spot maximum temperature difference

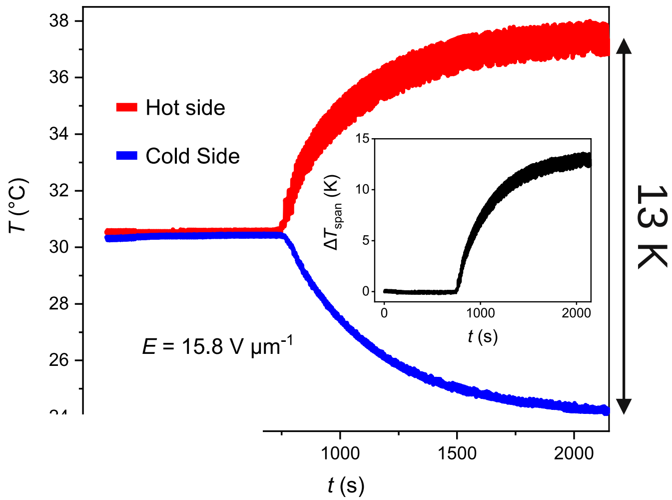
Final fabrication steps

1)

2)



3) In a box to ensure T_i = 30 ° C



temperature span larger than **10 K**

What about cooling power?

Science

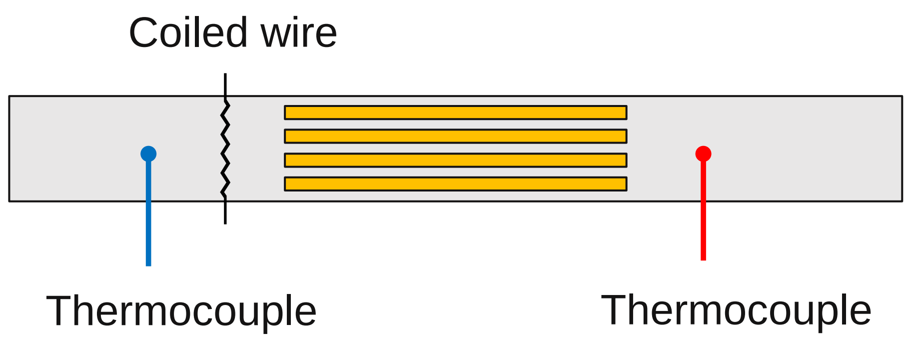
Giant temperature span in electrocaloric regenerator

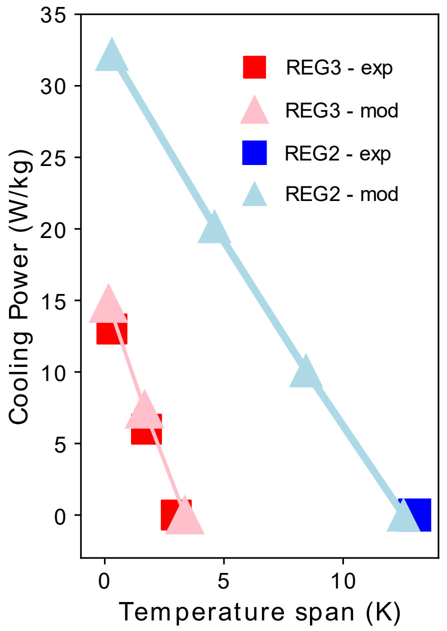
A. Torelló, P. Lheritier, T. Usui, Y. Nouchokgwe, M. Gérard, O. Bouton, S. Hirose and E. Defay

Science **370** (6512), 125-129.
DOI: 10.1126/science.abb8045

ELECTROCALORIC REGENERATOR – COOLING POWER

Coiled wire to act as a heat source in
a 32 1 mm thick PST-MLC regenerator





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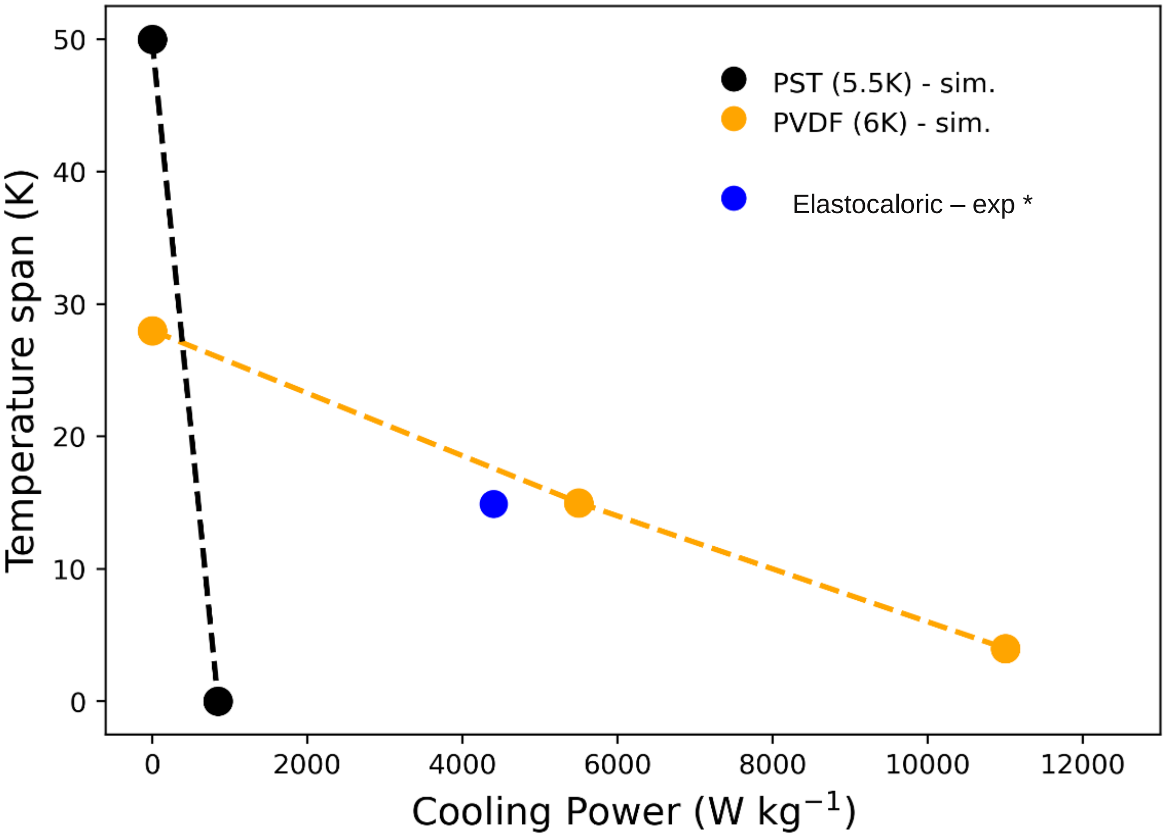
FUTURE PREDICTIONS

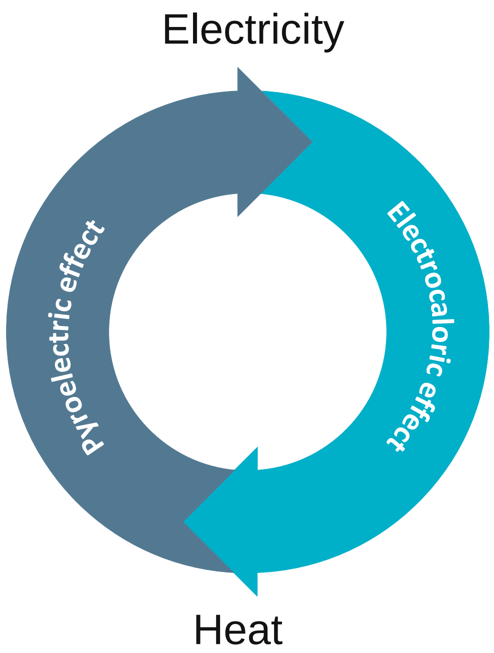


- ΔT = 50 K max with PST
- ΔT = 15 K and cooling power = 5500 W kg^{-1} with PVDF
- Very large values !
- Main issue – heat exchange

- The potential is massive !

Z. Ahčin, .., J. Tusek, Joule **6, 2338-2357 (2022).*

PYROELECTRIC HARVESTING



How good are $Pb(Sc,Ta)O_3$ multilayers at converting heat into electricity?

- Good electrocaloric materials must be good pyroelectrics

Pyroelectric coefficient

$$\Delta T_{EC} = -\frac{T}{c_E}\int_{E_{min}}^{E_{max}} \boxed{\left(\frac{\partial D}{\partial T}\right)_E} dE$$

Can we harvest energy in the Joule range?

- Scaling it up with macroscopic heat harvesting prototypes

THE PRINCIPLE OF PYRO ENERGY HARVESTING IN CYCLES

- 1. charge a capacitance C at temp T_1
- 2. disconnect the capacitance
  - => charge remains the same
- 3. heat it up to T_2
- 4. discharge the capacitance at T_2 (harvest)
- 5. cool it down to T_1
- 6. charge it again in order to cycle

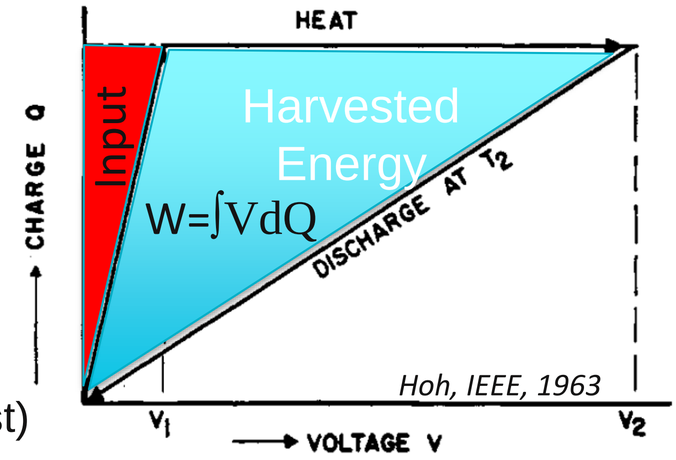


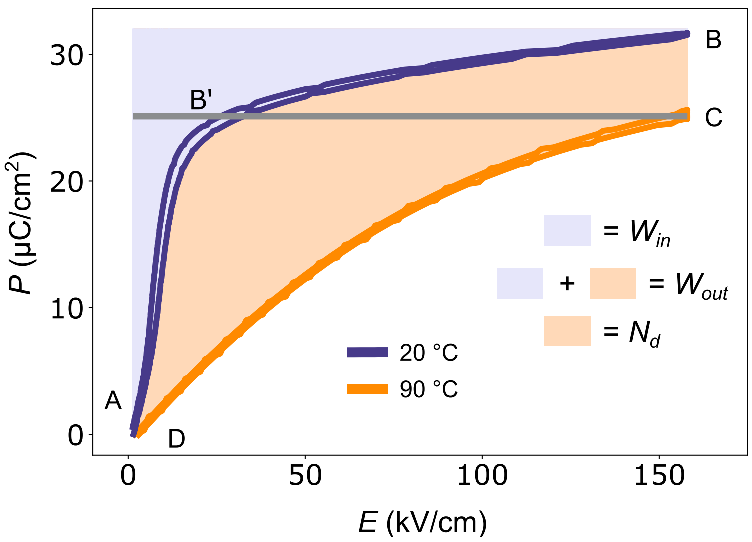
Fig. 7—Operating cycle of converter.

Need for large variation of capacitance
Need for very low leakage

PYROELECTRIC ENERGY HARVESTING IN CHARGE AND VOLTAGE

Temperature oscillations with time

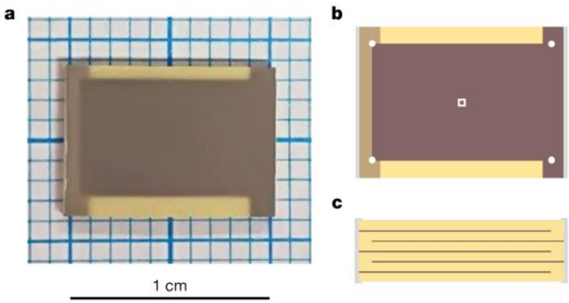
Olsen cycle
AB: isothermal charge
BC: isofield heating
CD: isothermal discharge
DA: isofield cooling



BUILDING A PYROELECTRIC HARVESTER

1. Pyroelectric material (electrocaloric)

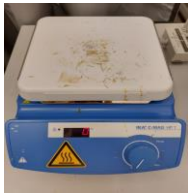
PST-MLCs



B. Nair et al., *Nature* ***575***, 468–472 (2019)

2. Temperature oscillation in time

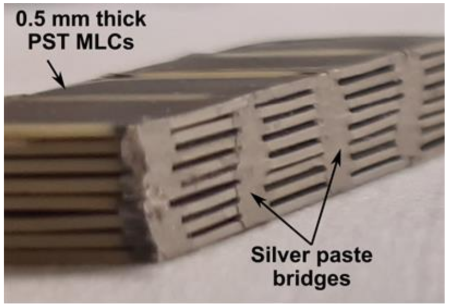
(hot reservoir)



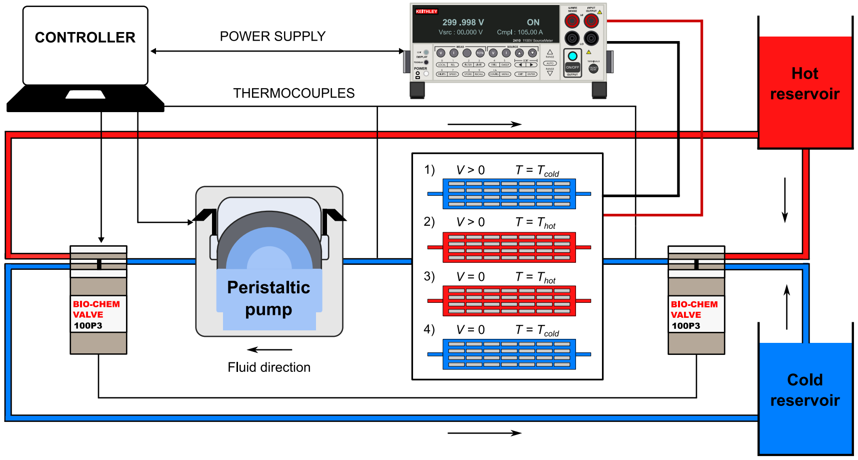
(cold bath)

3. Efficient heat transfer

(dielectric fluid flowing through a parallel plate structure)



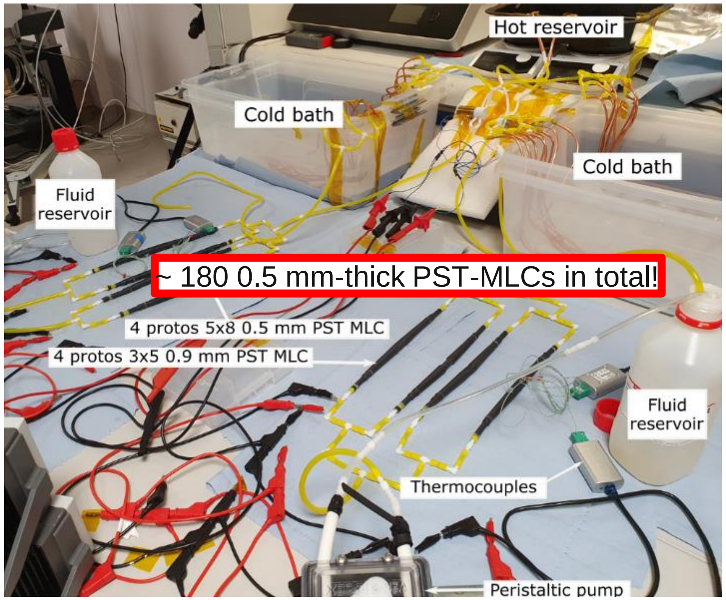
EXPERIMENTAL SET-UP FOR ENERGY HARVESTING



28 1 mm-thick multilayers
Parallel plate matrix: 7 col x 4 rows

One multilayer = 0.3 grams of active material

OUR BEST RESULT



Article

Large harvested energy with non-linear pyroelectric modules

nature

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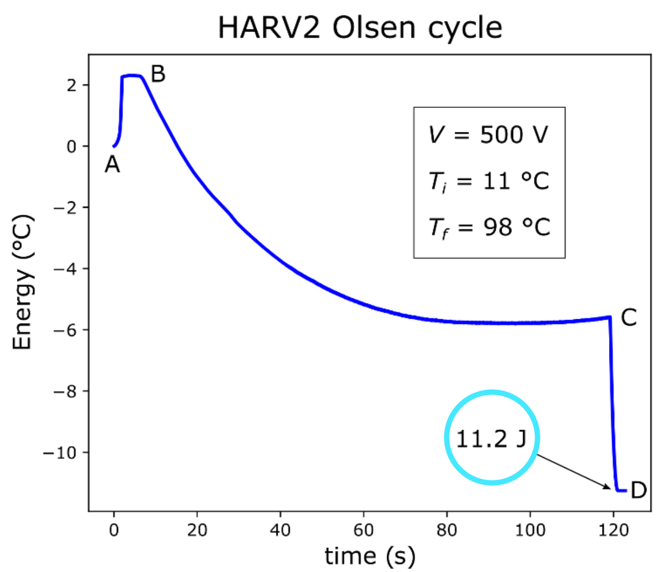
Received: 12 October 2021
Accepted: 4 July 2022

Pierre Lheritier[1,4], Alvar Torelló[1,2,4]✉, Tomoyasu Usui[3], Youri Nouchokgwe[1,2], Ashwath Aravindhan[1,2], Junning Li[1], Uros Prah[1], Veronika Kovacova[1], Olivier Bouton[1], Sakyo Hirose[3] & Emmanuel Defay[1]✉

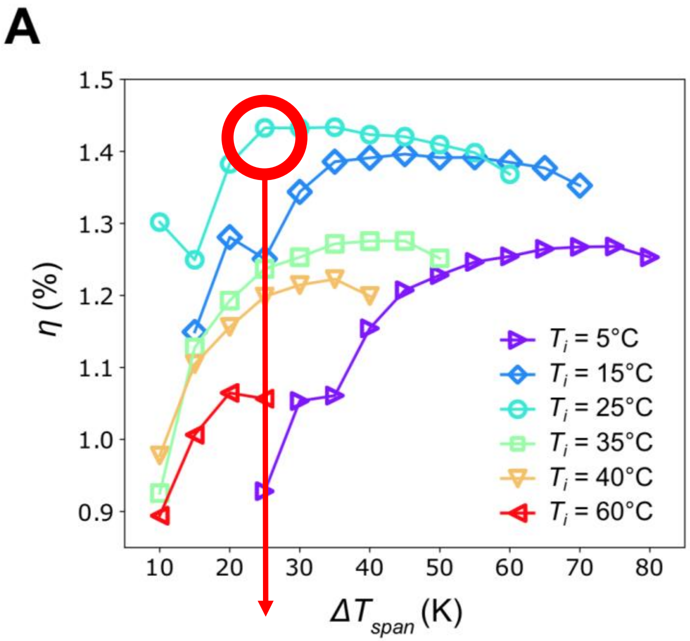


11.2 J with **41.2 g** of active material

Harvesting Joules with grams

Energy efficiency

η_r = efficiency with respect to Carnot



$$\eta = 1.43\% \; (\, solar\ panels \sim 20\%)$$

WHAT CAN WE DO WITH IT ?

Some ideas under investigations

- Autonomous sensors with heat energy harvester (already in the previous study)
- Energy harvesting for large facilities (steel factories)
- Energy harvesting in space (CubeSat project)
- Solar panels

Main challenges

- Materials without lead and with phase transition
- Large heat exchange (water, designs)

CONCLUSION

- Large electrocaloric effect => large variation of polarisation and low *ε*
- Best electrocaloric materials => PST and PVDF
- Field-induced phase transition induces large EC effect
- Multilayer capacitors => excellent structure for prototypes (good material and large field)
- Best prototype : PST MLCs and fluid. ΔT = 13 K @ room T°
- PVDF and PST have a lot of potential. Alternative to PST required.
- Efficiency matters !
- The conjugated effect is also of interest – pyroelectric energy harvesting

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