



Rapid synthesis of MgB₂ bulks by inductive heating

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Outline

- Introduction
 - Fabrication Process
- The rapid synthesis
 - Advantages with respect to a traditional process
- Results
 - Good quality MgB_2 bulk obtained in few minutes
 - B_{C2} enhancement
 - Higher J_C with the rapid synthesis
 - Microstructural Investigations - X-ray diffraction / SEM
- Conclusions & Outlook



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Fabrication technique - IMD

Internal Magnesium Diffusion (IMD)
In-situ synthesis process

50 wt.% Mg excess

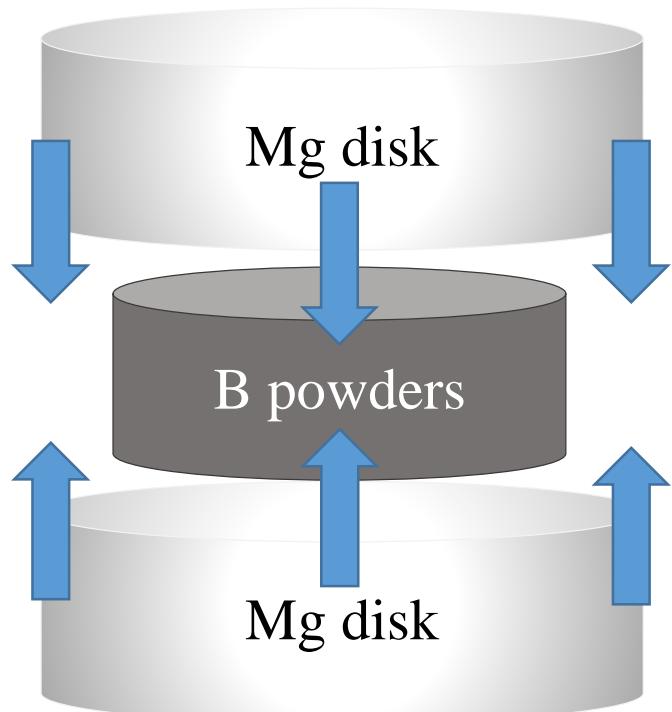
B powders from
Strem Chemicals
99+% $\approx 0.5 \mu\text{m}$

Mg rods
from ChemPur
99.9+%

filled in stainless steel metal sheaths
that act as crucibles

Synthesis using
- Commercial Muffle Furnace
- Induction Furnace

Obtained bulk
 $\varnothing \approx 9.5\text{mm}$
 $H \approx 20 \text{ mm}$



The Induction Furnace

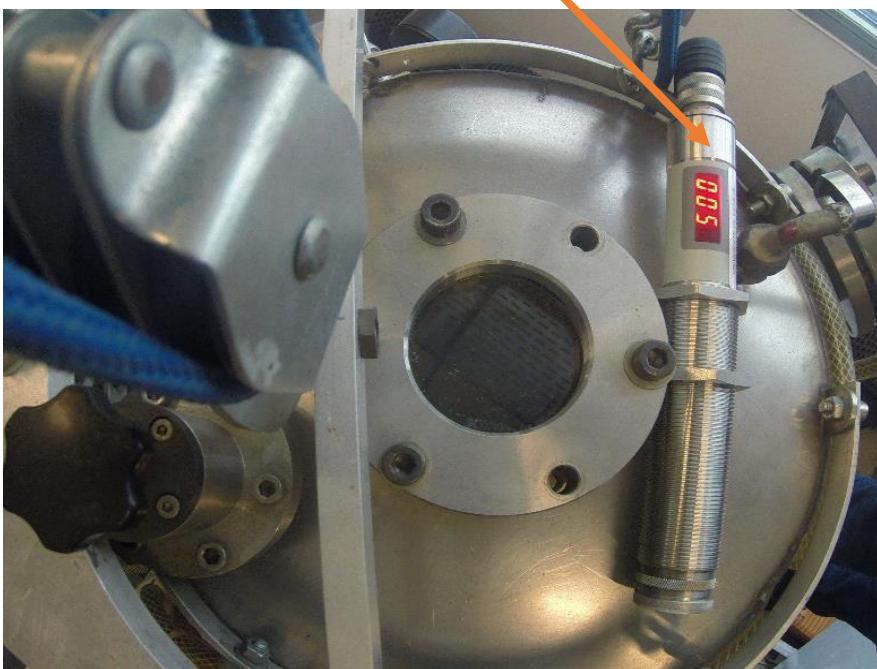
gas injection
Ar-gas
(up to 15 bar)

inductive
copper coil
water cooled

calibrated
pyrometer
to monitor the T



Internal view of the system



Upper cover of the reaction chamber

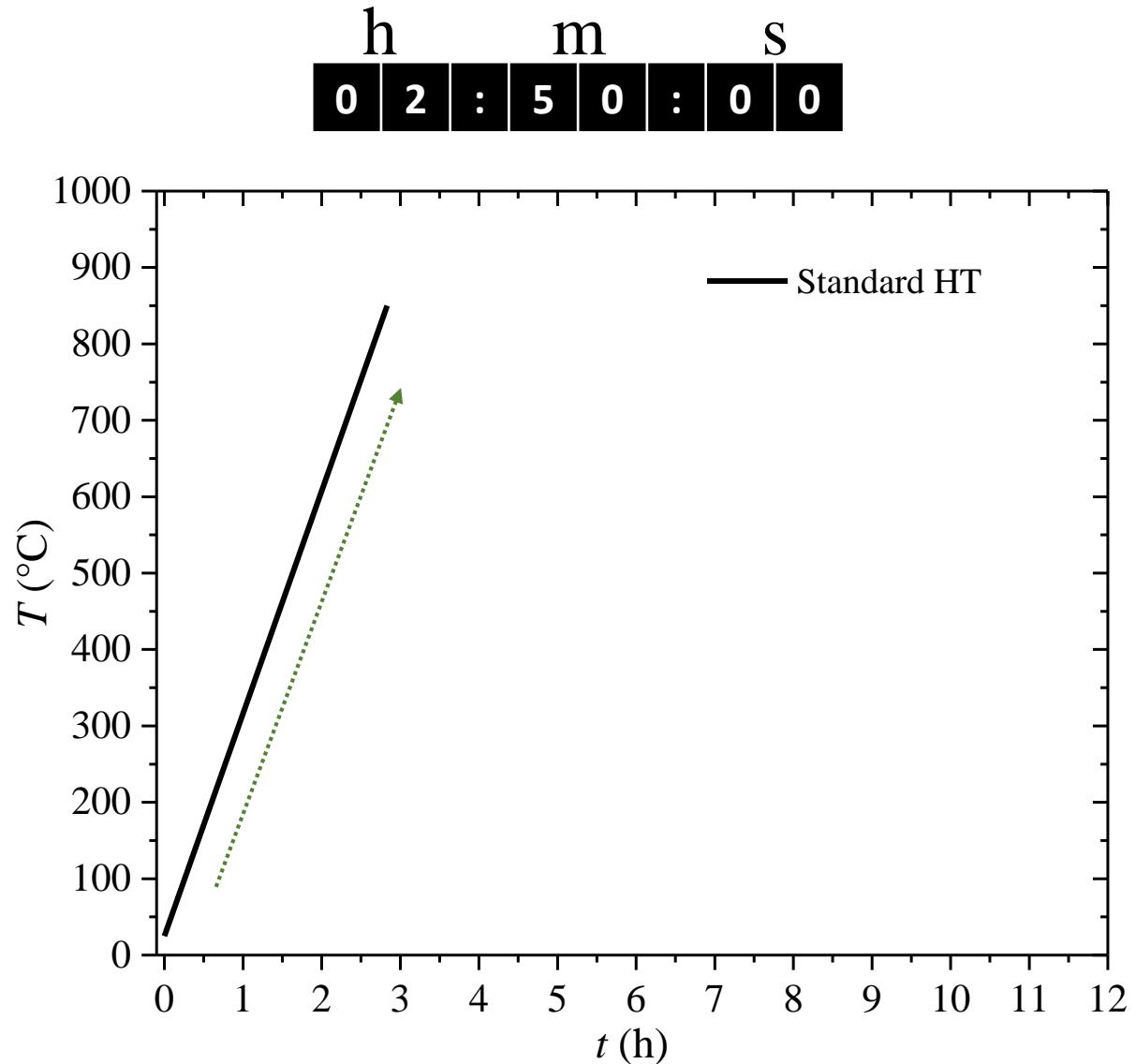


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Resistance Furnace – HT Profile



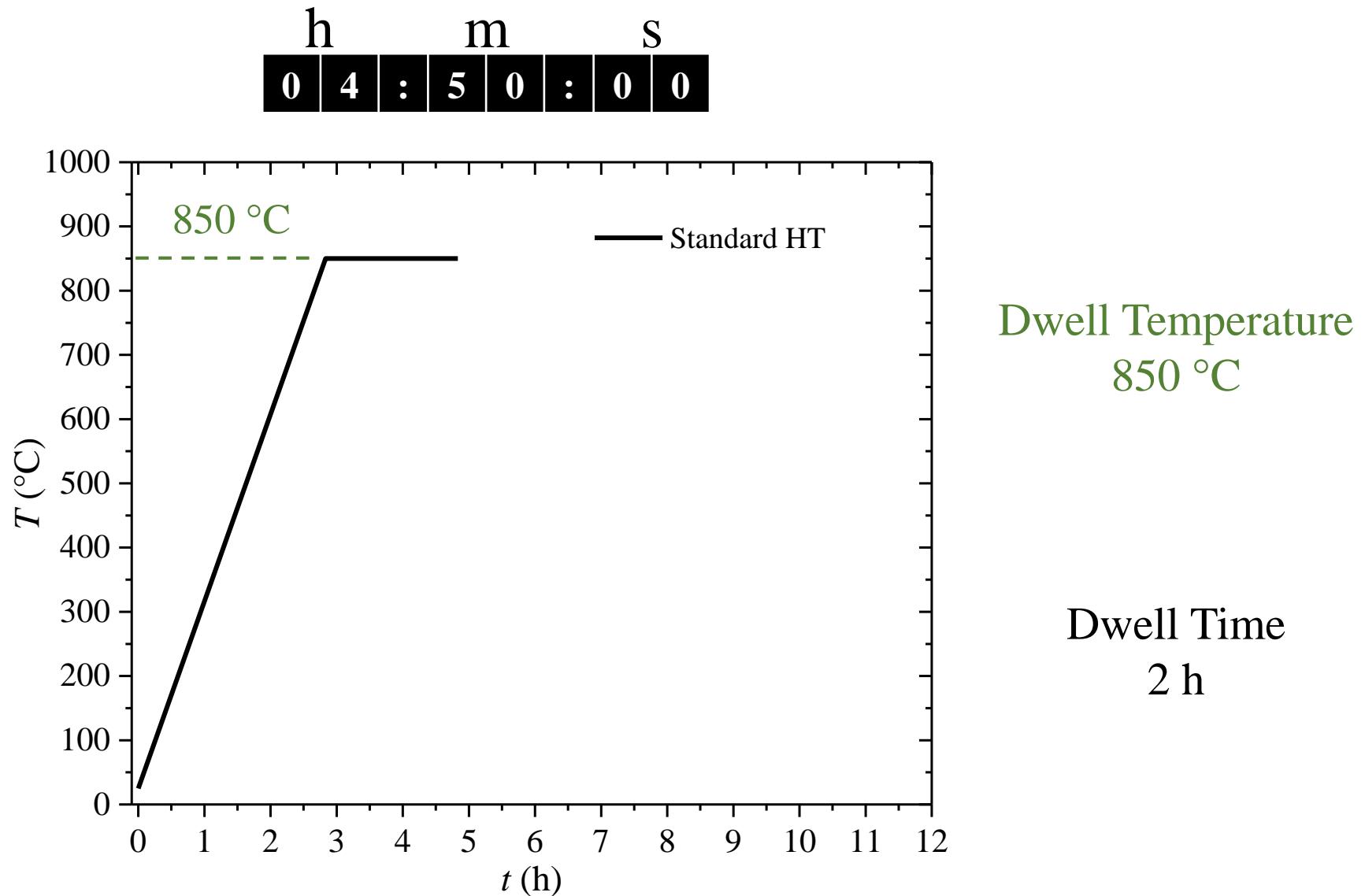
Heating ramp rate
 $\sim 5 \text{ } ^\circ\text{C/min}$

To avoid
overheating

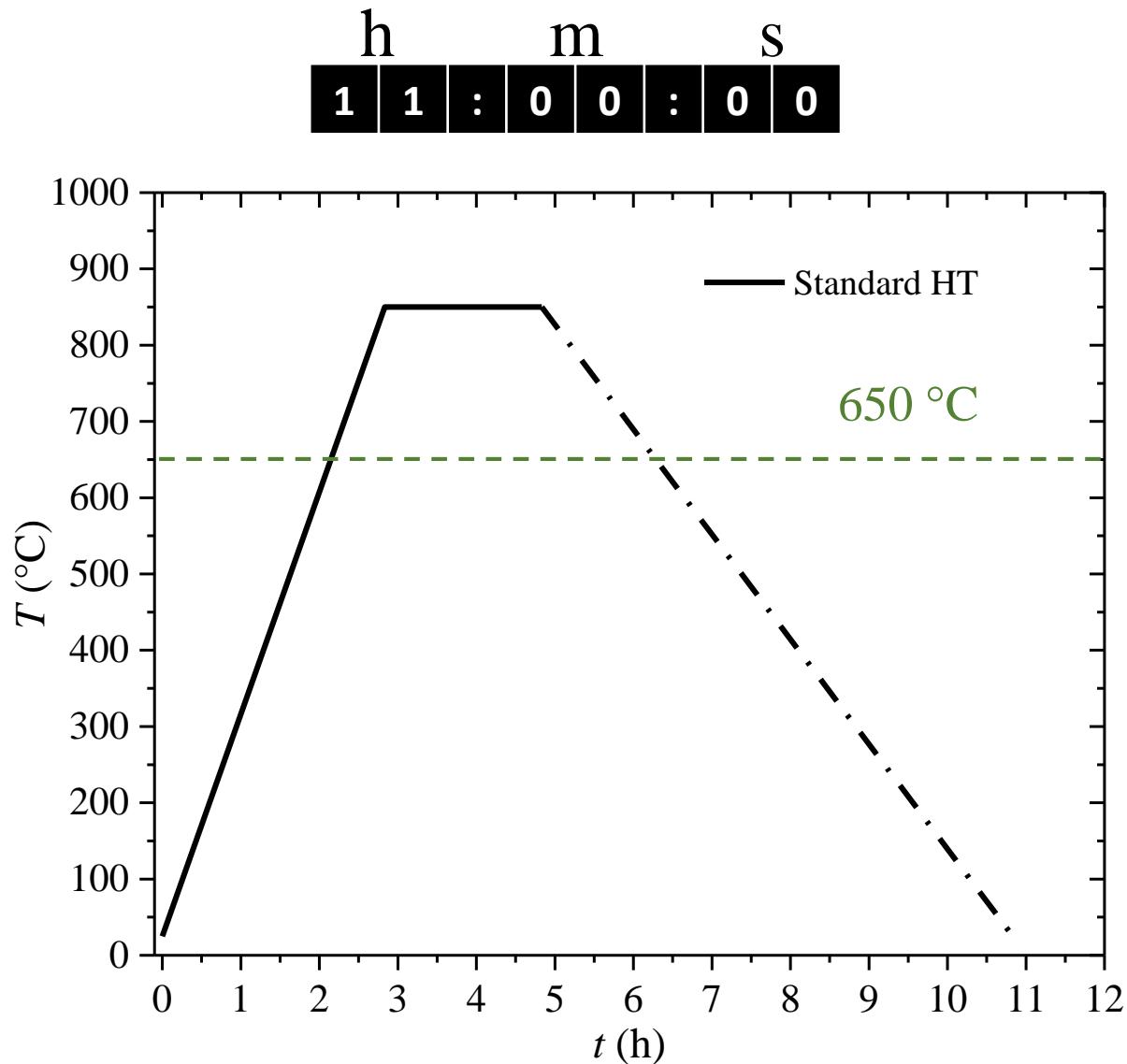
From RT
Up to
850 °C
in ~ 3 h



Resistance Furnace – HT Profile



Resistance Furnace – HT Profile

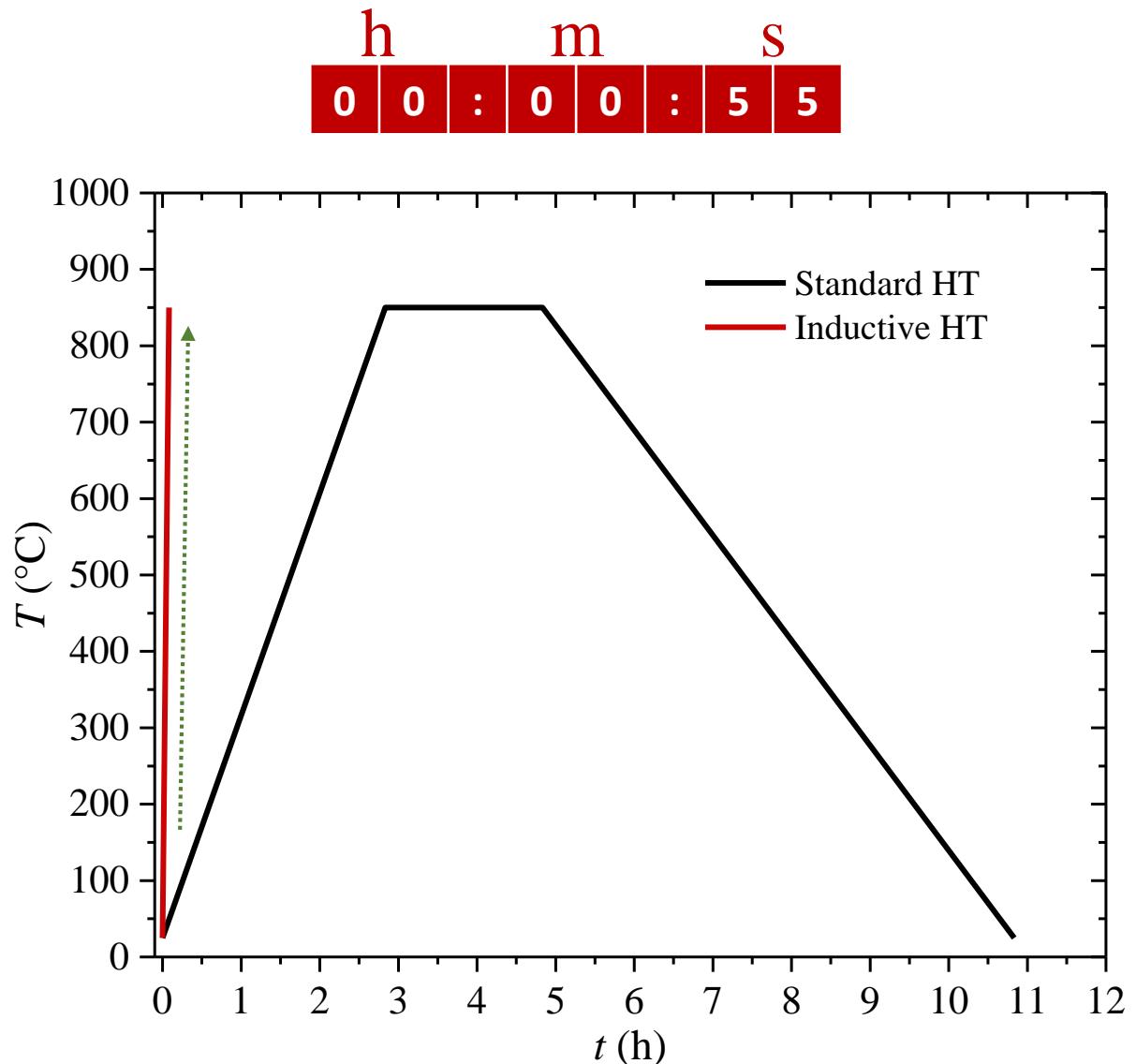


Half a day to synthesize
a sample !!!

The reaction starts
at T higher
than ~ 650 °C
at ambient pressure

The bulk in the case of
“Standard HT”
has been reacted
for almost 6h

Induction Furnace – HT Profile

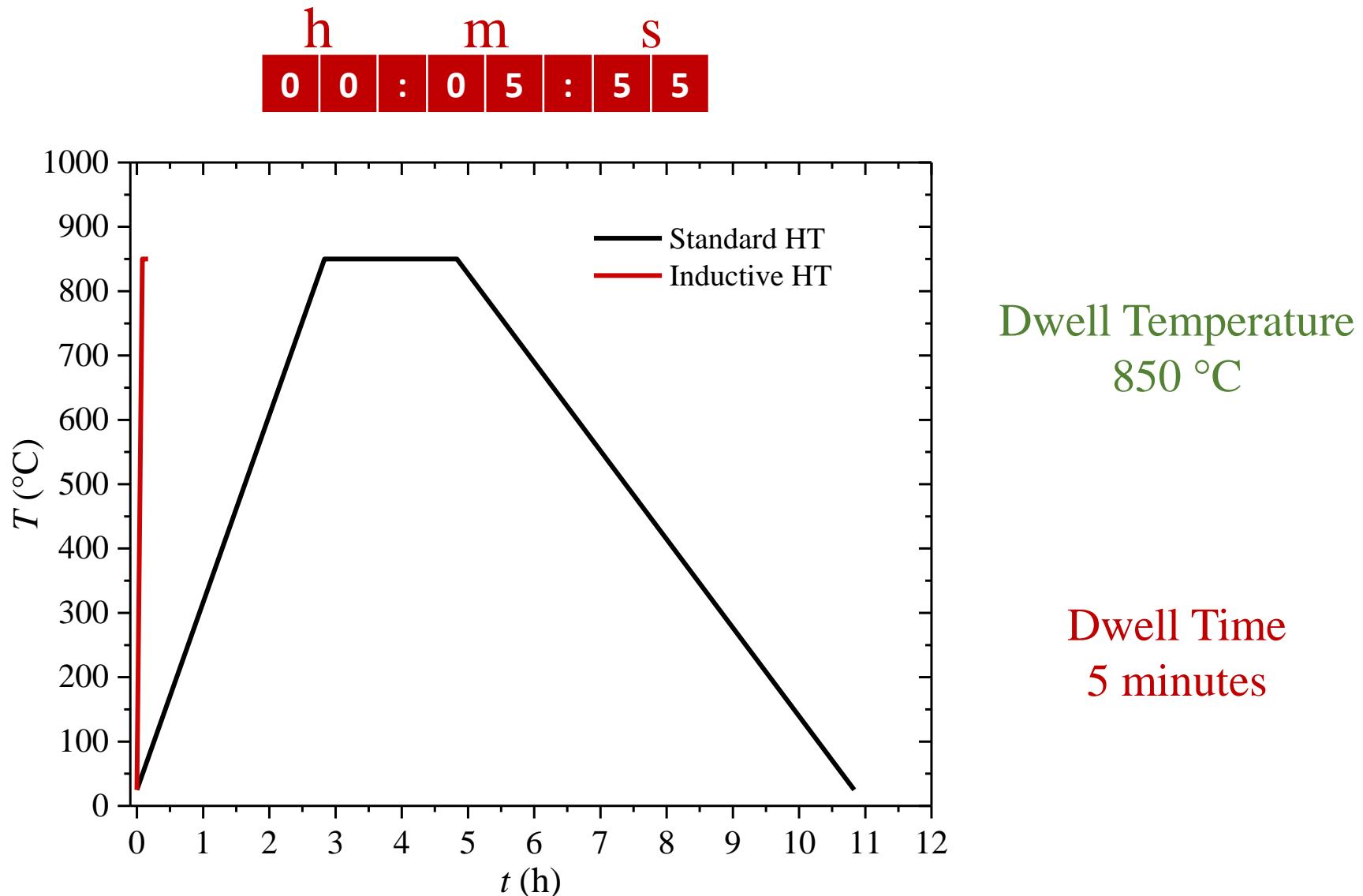


Heating ramp rate
 ~ 900 °C/min

overheating
can be kept under
control

From RT
Up to
850 °C
in ~ 50 s

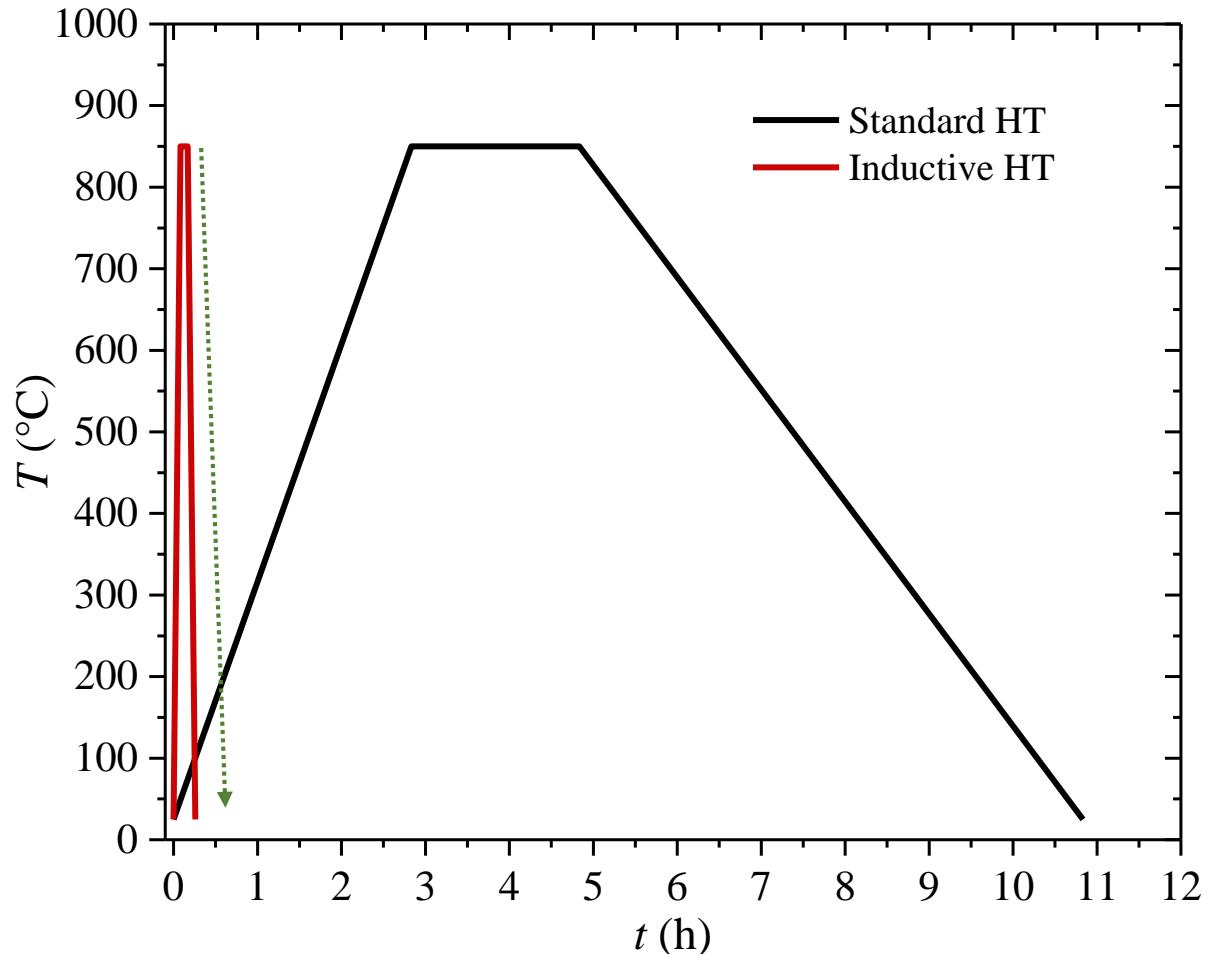
Induction Furnace – HT Profile



Induction Furnace – HT Profile

h m s
 0 | 0 | : | 0 | 6 | : | 2 | 5

The sample is ready !!!



Sample-quench
30 seconds to reach RT

The bulk in the case of
“Inductive HT”
has been synthesized
in ~ 6 minutes !!!



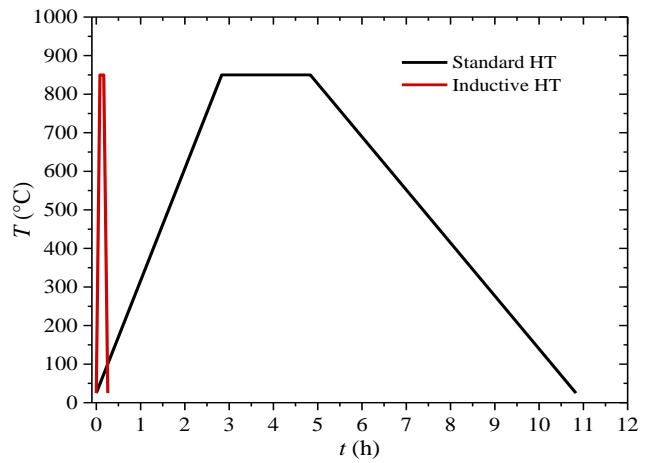
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Standard HT vs. Inductive HT

h	m	s
1	1	0
0	0	0
0	0	0

“Standard HT” ■
 850°C 2 hours
 no Quench



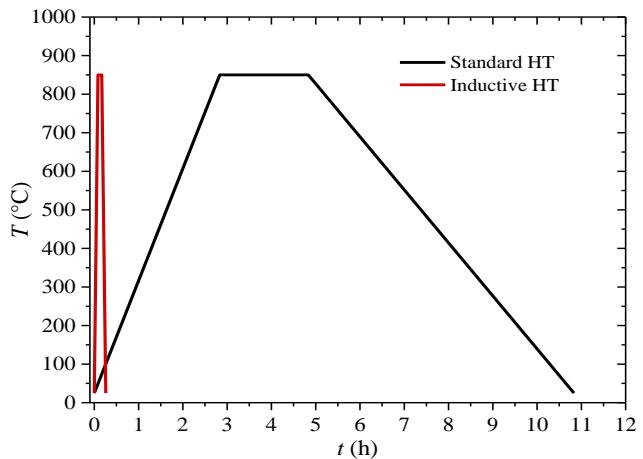
0	0	0
0	6	2
5		

“Inductive HT” ●
 850°C 5 minutes
 + Quench

Standard HT vs. Inductive HT

h m s
 1 | 1 | : | 0 | 0 | : | 0 | 0

“Standard HT” ■
 850°C 2 hours
 no Quench



h m s
 0 | 0 | : | 0 | 6 | : | 2 | 5

“Inductive HT” ●
 850°C 5 minutes
 + Quench

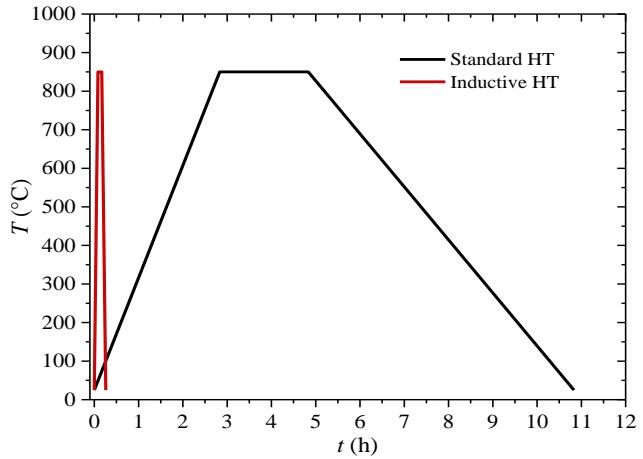
38.7	T_C (K)	38.8
0.1	ΔT_C (K)	0.2
1.44	ρ_{40K} ($\mu\Omega\text{cm}$)	1.96
2.38	D (g/cm^3)	2.13
64.3	K (%)	30.4

T_C and ΔT_C are
 the same

Standard HT vs. Inductive HT

h m s
 1 | 1 | : | 0 | 0 | : | 0 | 0

“Standard HT” ■
 850°C 2 hours
 no Quench



h m s
 0 | 0 | : | 0 | 6 | : | 2 | 5

“Inductive HT” ●
 850°C 5 minutes
 + Quench

38.7

T_C (K)

38.8

0.1

ΔT_C (K)

0.2

1.44

ρ_{40K} ($\mu\Omega\text{cm}$)

1.96

2.38

D (g/cm³)

2.13

64.3

K (%)

30.4

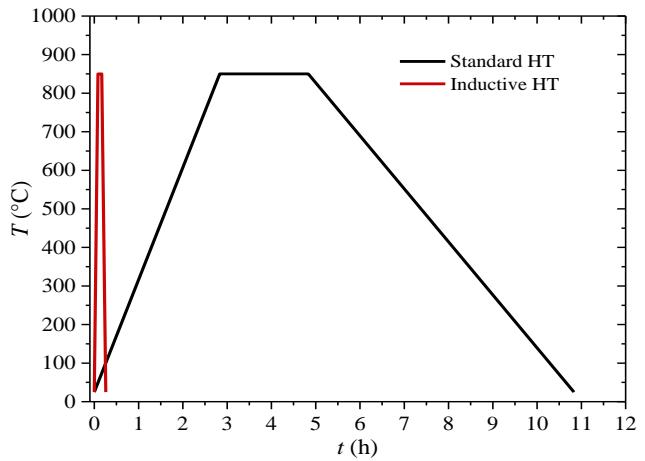
T_C and ΔT_C are the same

ρ (40K) slightly increases

Standard HT vs. Inductive HT

h
1 | 1 | : | 0 | 0 | : | 0 | 0

“Standard HT” ■
 850°C 2 hours
 no Quench



h
0 | 0 | : | 0 | 6 | : | 2 | 5

“Inductive HT” ●
 850°C 5 minutes
 + Quench

38.7

T_C (K)

38.8

0.1

ΔT_C (K)

0.2

1.44

ρ_{40K} ($\mu\Omega\text{cm}$)

1.96

2.38

D (g/cm³)

2.13

64.3

K (%)

30.4

T_C and ΔT_C are the same

ρ (40K) slightly increases

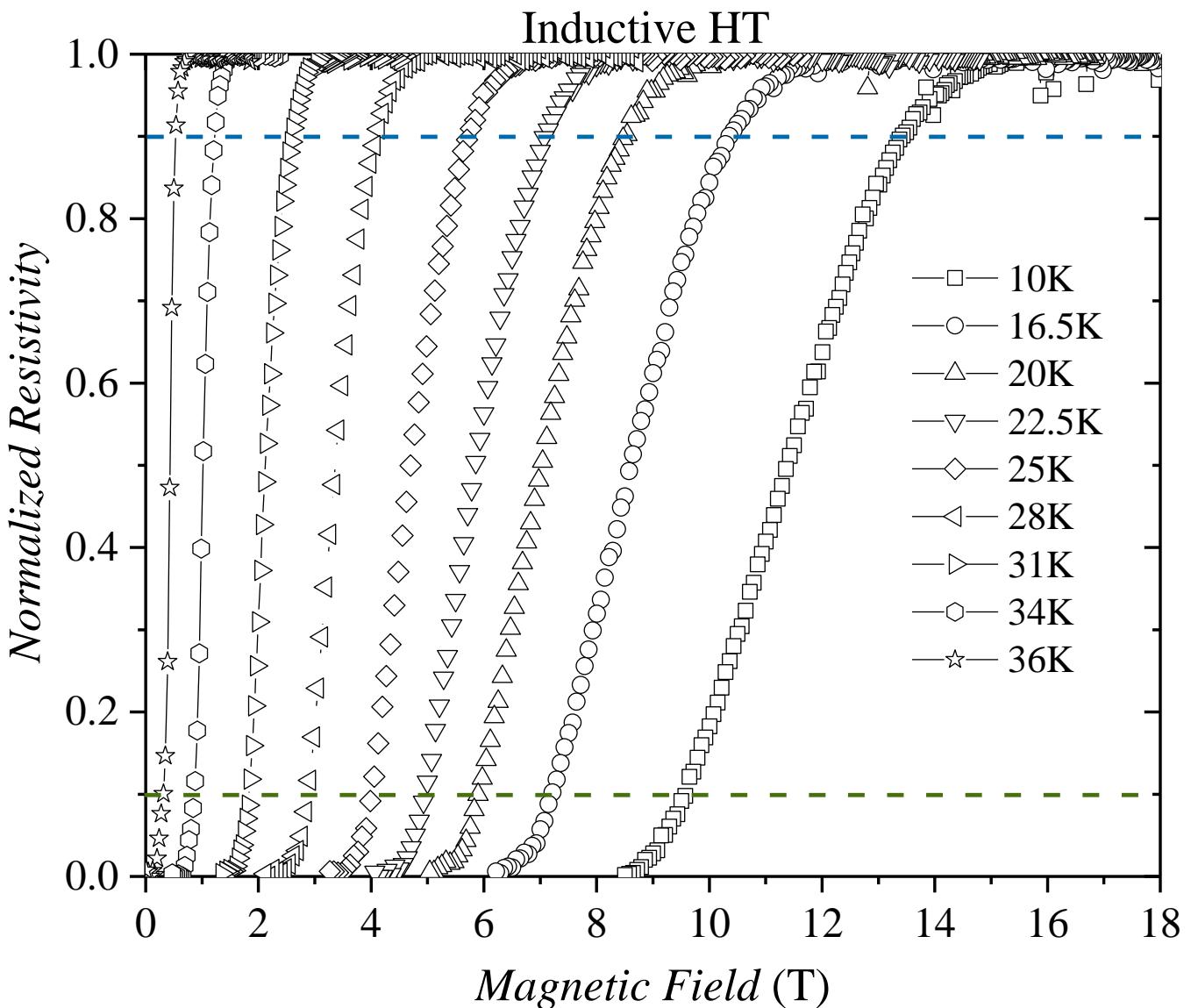
D and K decrease



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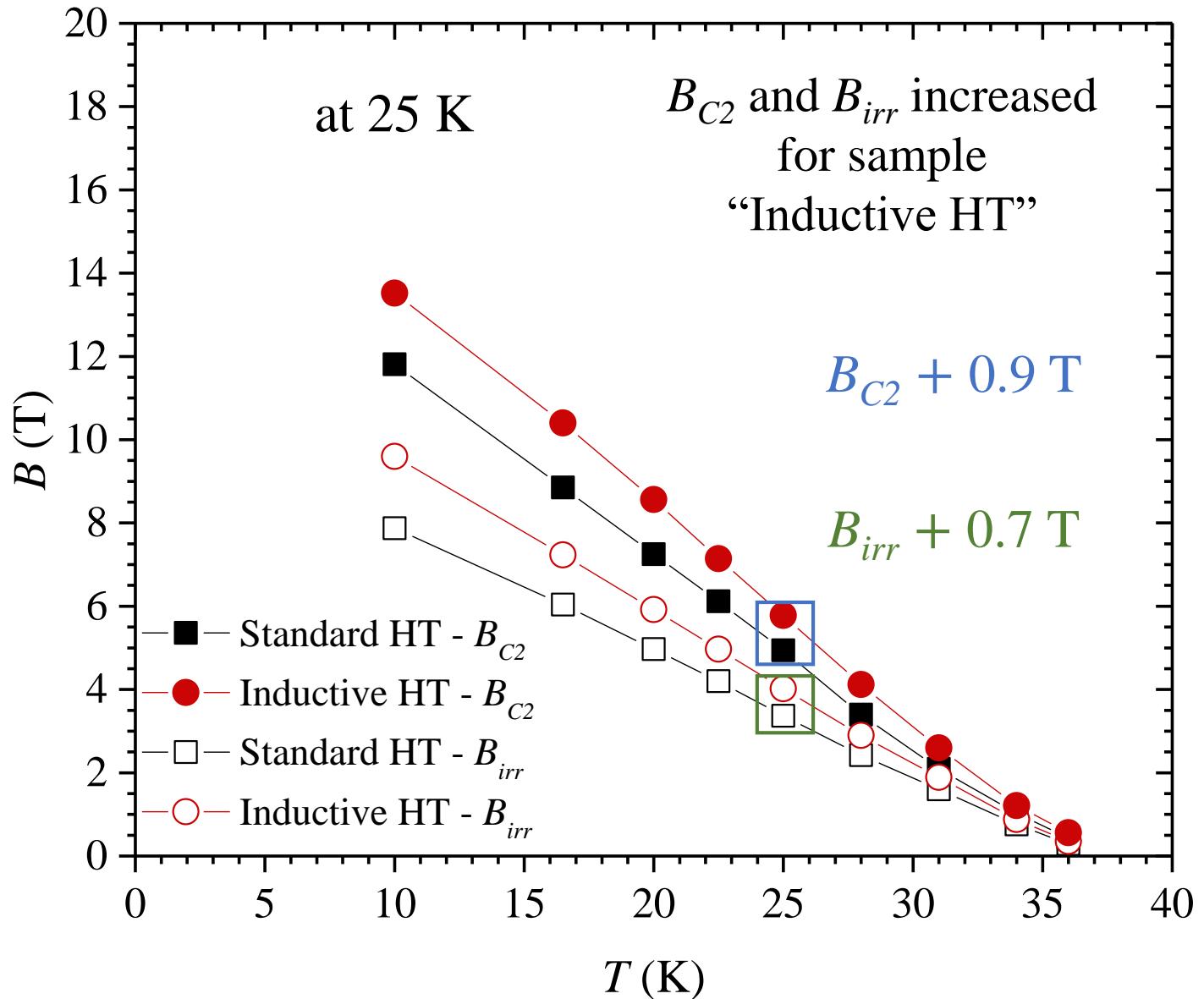
$\rho(T, B) - B_{irr}$ & B_{C2} criteria



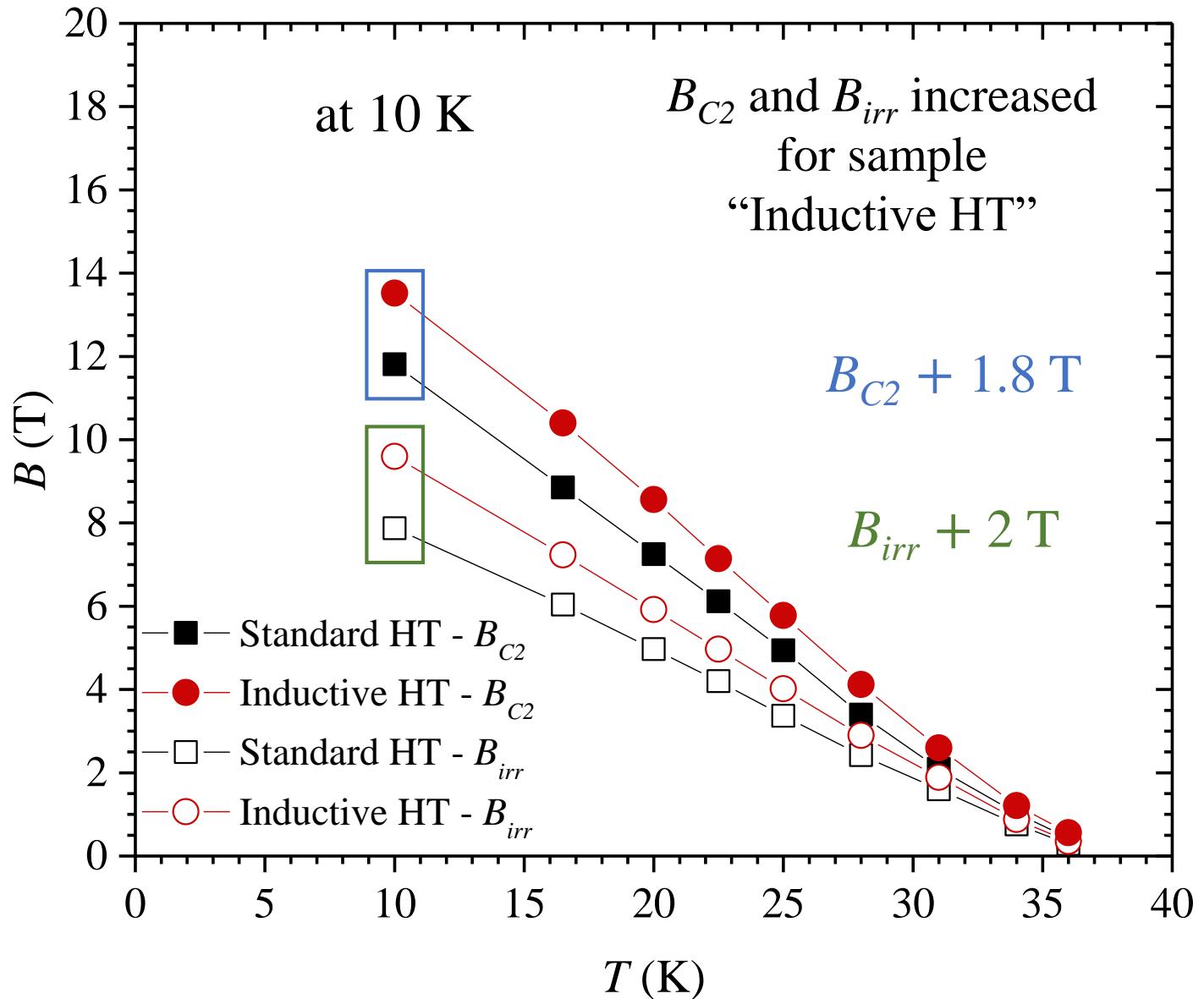
B_{C2} defined as the value corresponding to the 90 % of the transition

B_{irr} defined as the value corresponding to the 10 % of the transition

B_{irr} & B_{C2} Vs. T



B_{irr} & B_{C2} Vs. T

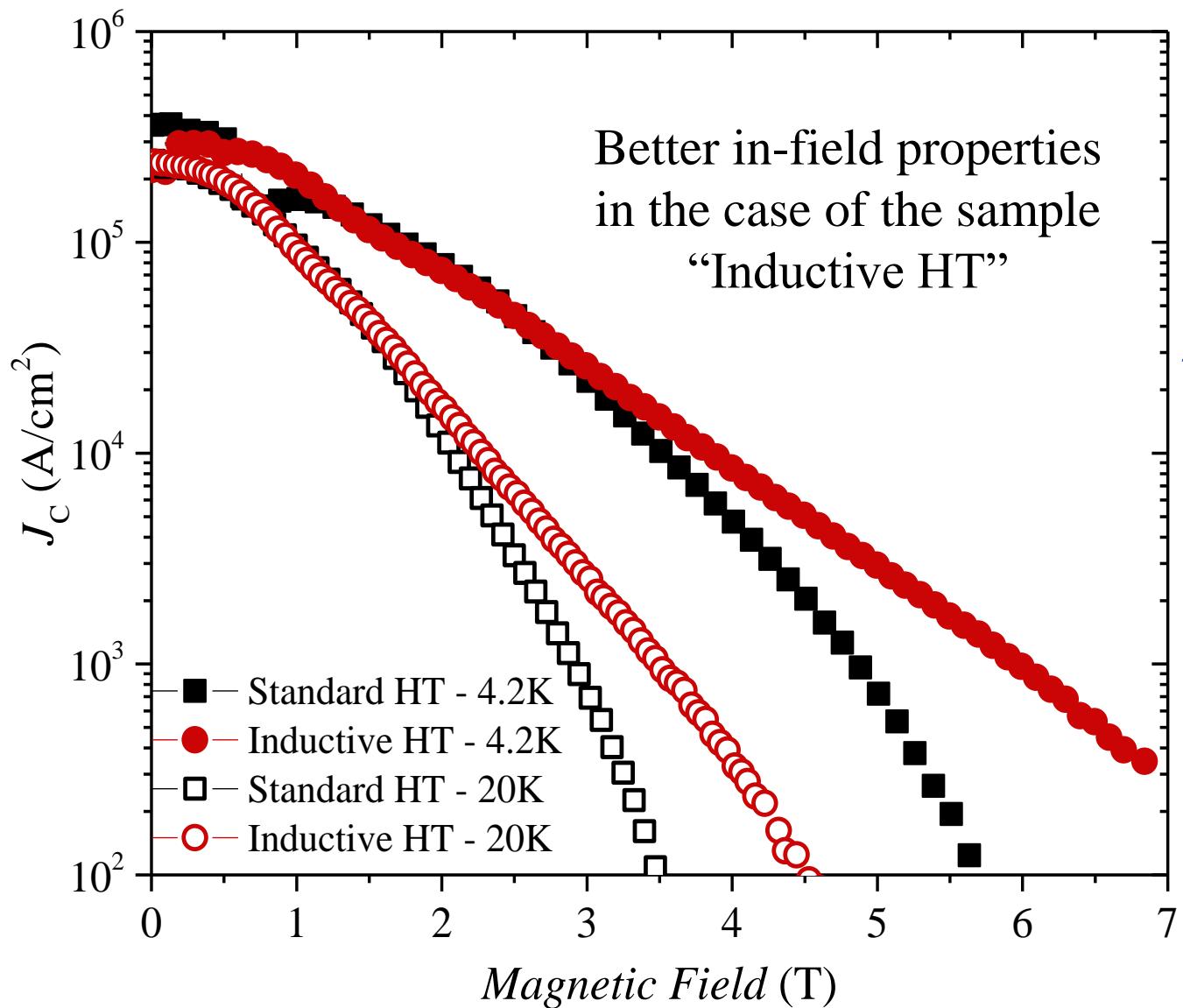




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$J_C(B)$ at 4.2 K and at 20 K



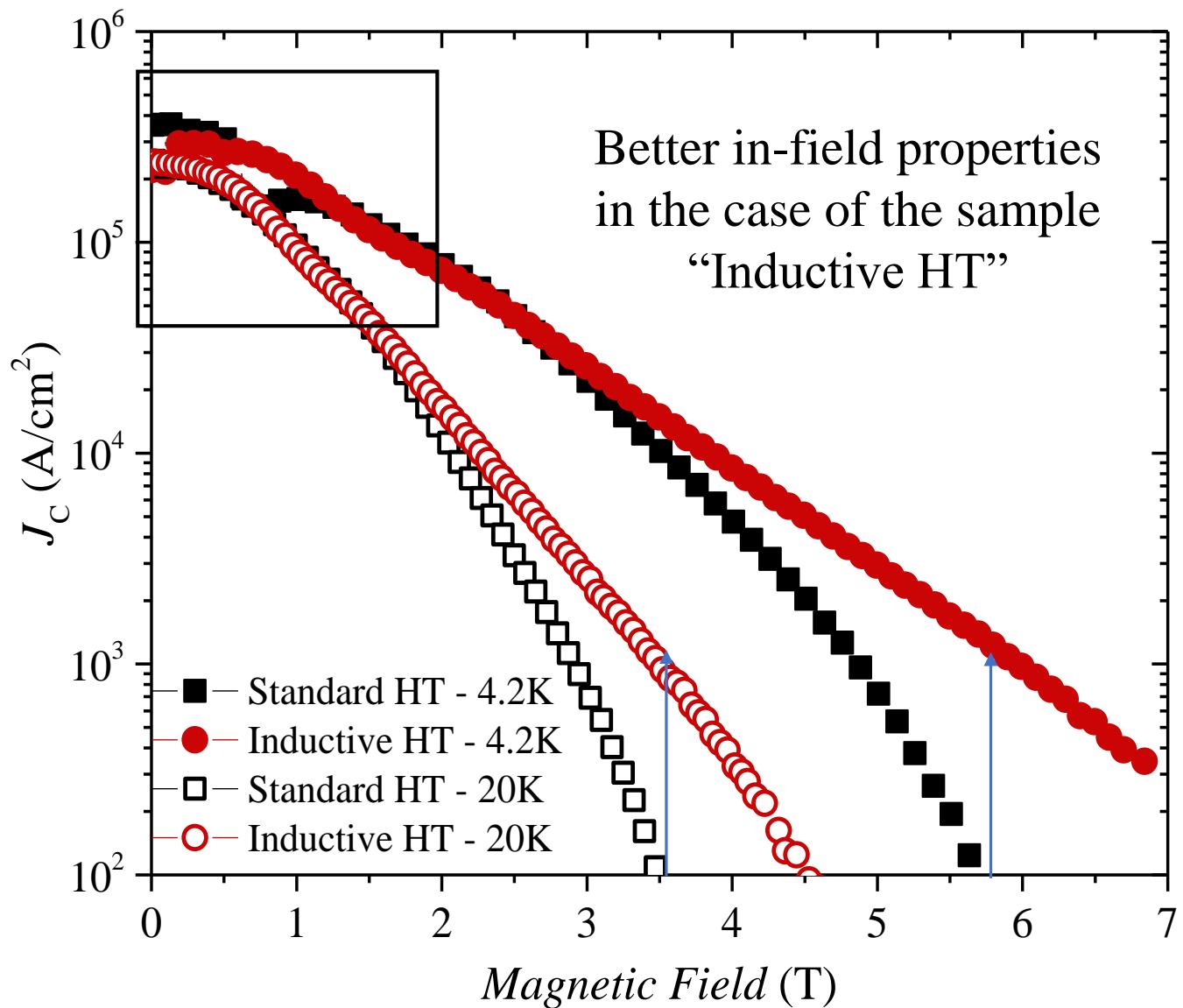
Bean critical state
model
slab in // field

$$J_C(T, B) = \frac{2\Delta m(T, B)}{Vd}$$

$\Delta m(T, B)$
separation between
the 2 branches of the
 $m(B)$ loop

V the volume
 d the thickness
of the sample

$J_C(B)$ at 4.2 K and at 20 K



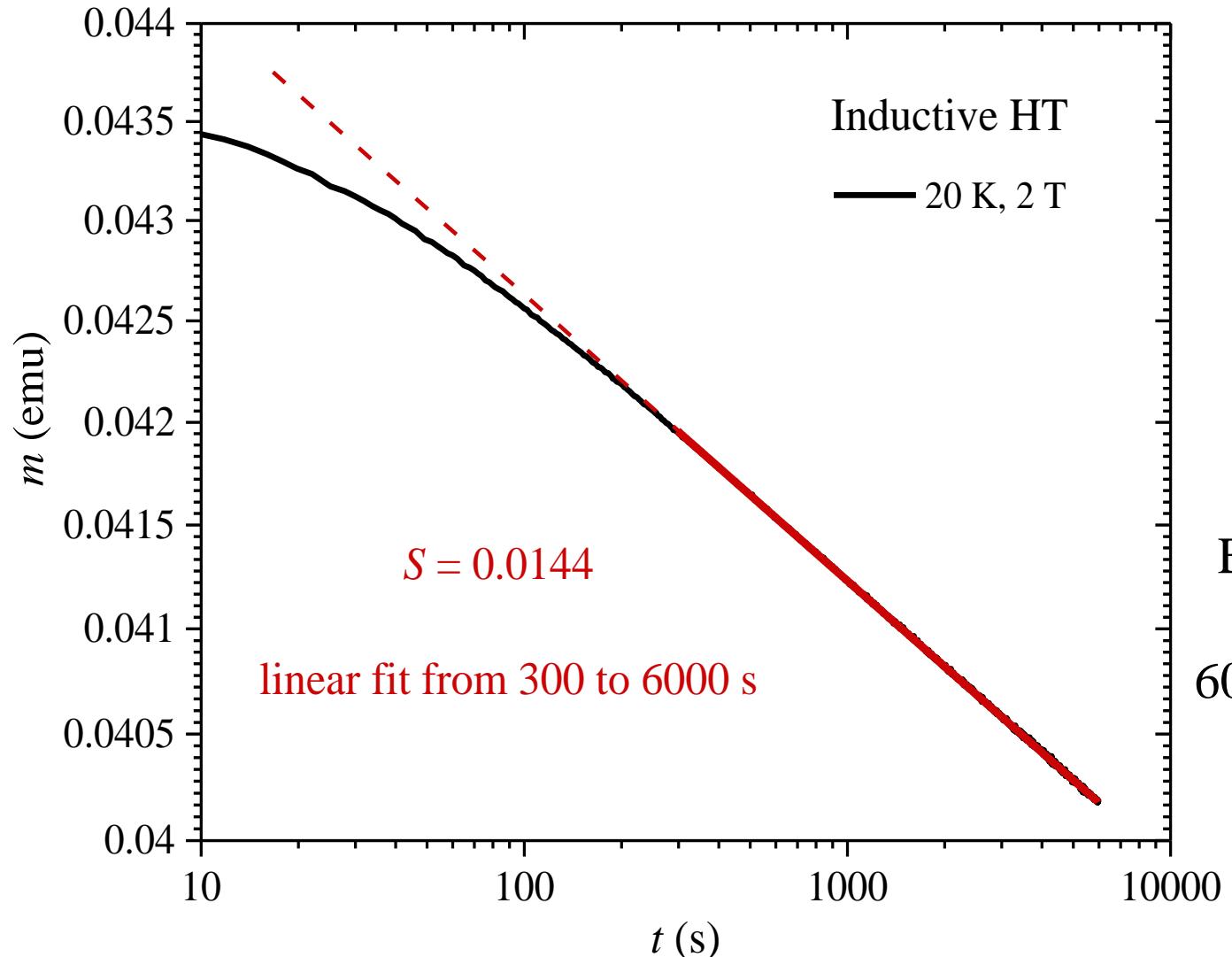
No evident differences in the low field region

$$J_C(20 \text{ K}, 0 \text{ T}) \approx 2 \cdot 10^5 \text{ A/cm}^2$$

Different behavior at high field

J_C up to 1 order of magnitude higher for the Inductive HT

Magnetic Relaxation

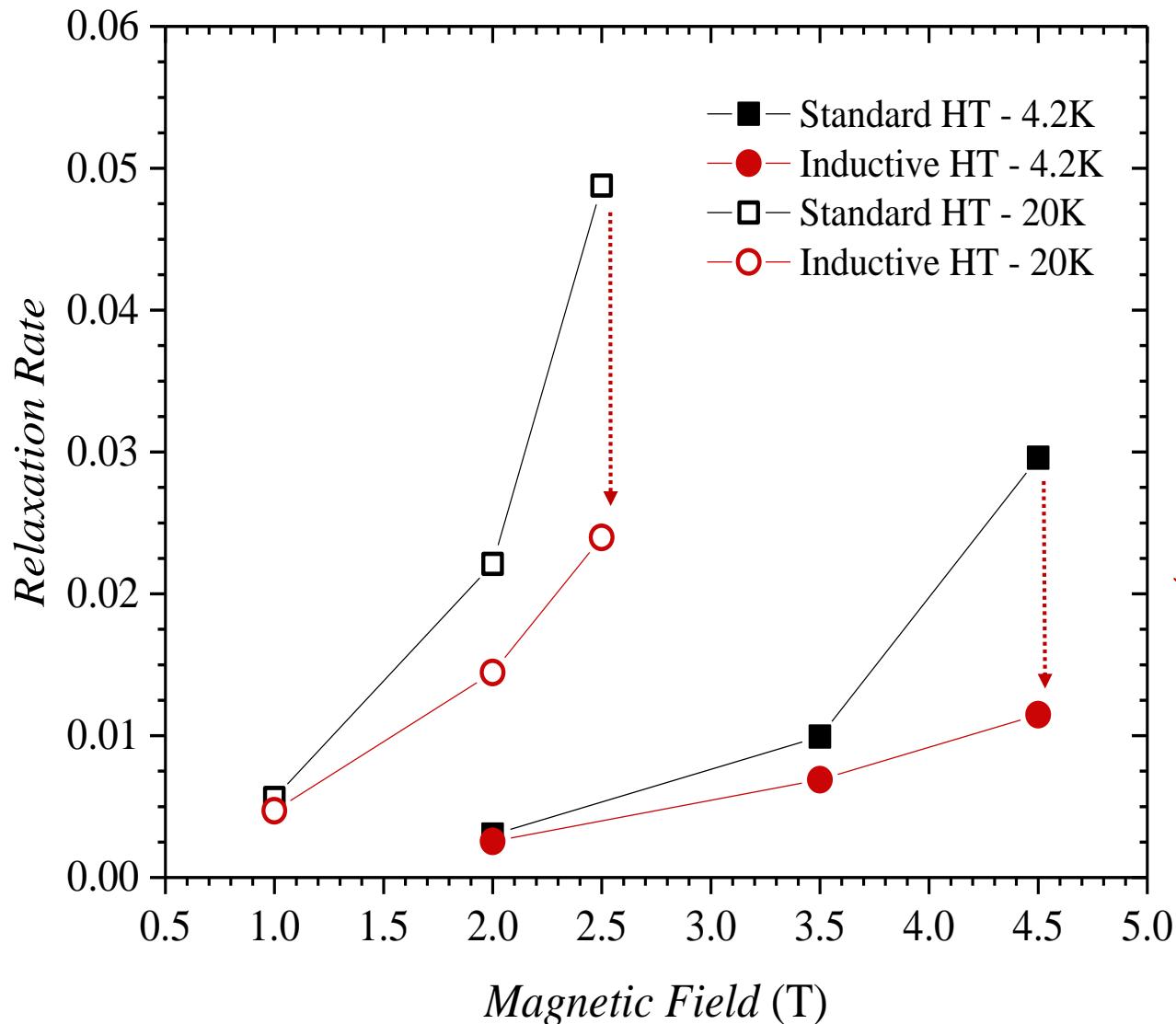


*Magnetic
Relaxation Rate*

$$S = -\frac{d \log m}{d \log t}$$

Evaluated from $m(t)$
curves acquired for
6000 s at various fields
at 4.2 K and 20 K

S Vs. B at 4.2 K and 20 K



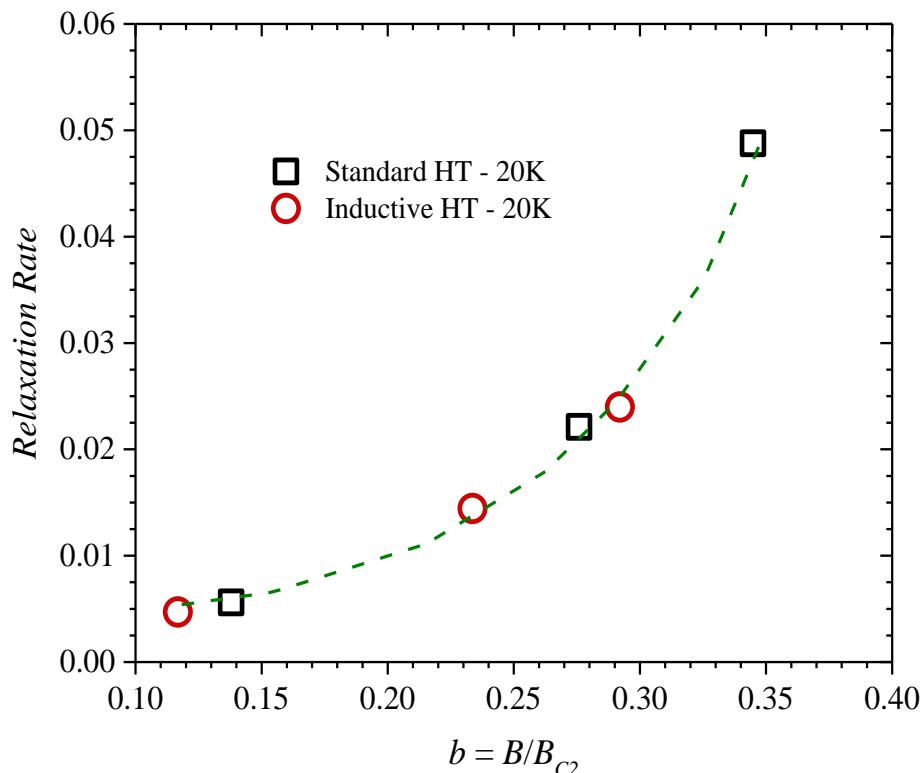
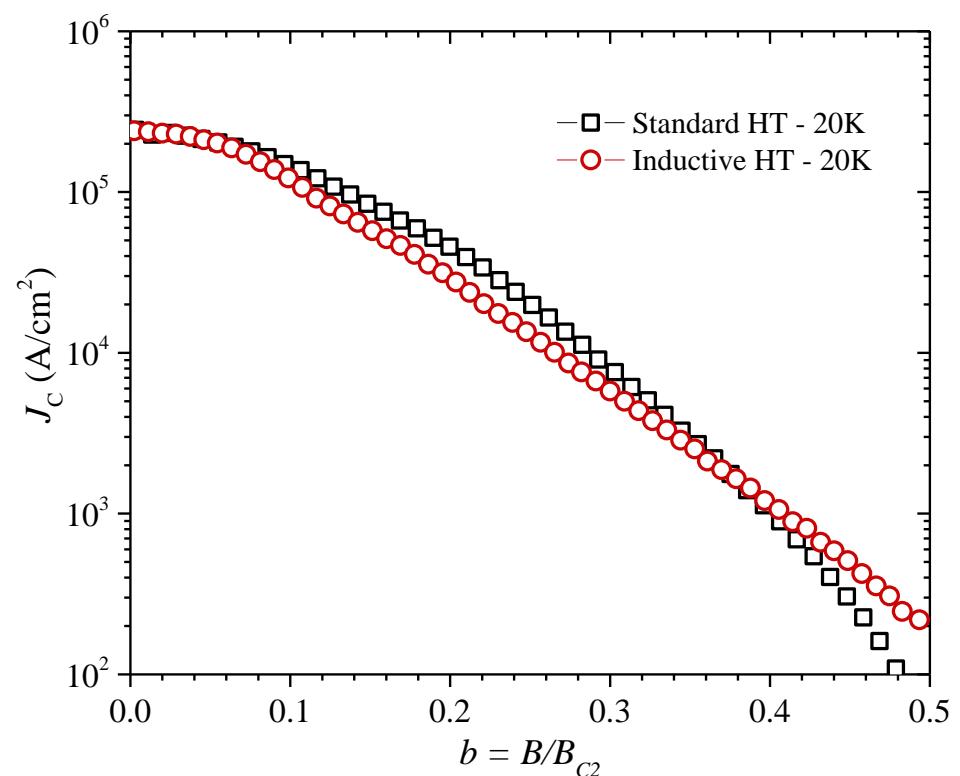
S is related to the pinning potential energy (U_0)

$$S = \frac{K_b T}{U_0}$$

Thus
 $\downarrow S \equiv \uparrow$ pinning efficiency

Inductive HT :
higher pinning
efficiency at high fields

$J_C (B/B_{C2}) \& S (B/B_{C2})$ at 20 K



The enhancement of J_C and the reduction of S at high fields are related to the improvement of B_{C2} in the “**Inductive HT**” sample



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X-ray – Lattice Parameters

Rietveld Refinement

Standard HT

Inductive HT

a (Å)

3.08552 (2)

3.08575 (4)

c (Å)

3.52272 (3)

3.5237 (1)

No relevant variations in the lattice parameters



X-ray – Indexing

Rietveld Refinement

Standard HT

Inductive HT

a (Å)

3.08552 (2)

3.08575 (4)

c (Å)

3.52272 (3)

3.5237 (1)

MgB_2 (wt.%)

79.9 (6)

83.2(4)

Mg (wt.%)

15.7 (2)

10.6 (2)

MgO (wt.%)

4.4 (2)

6.2 (1)

Inductive HT

↑ MgB_2 ($\approx +3\%$) MgO ($\approx +2\%$)

↓ Mg ($\approx -5\%$)



X-ray – Cristallite Size & Strain

Rietveld Refinement

Standard HT

Inductive HT

Thickness - $<001>$ (μm)

0.1315

0.1389

Width - $<100>$ (μm)

0.2161

0.1903

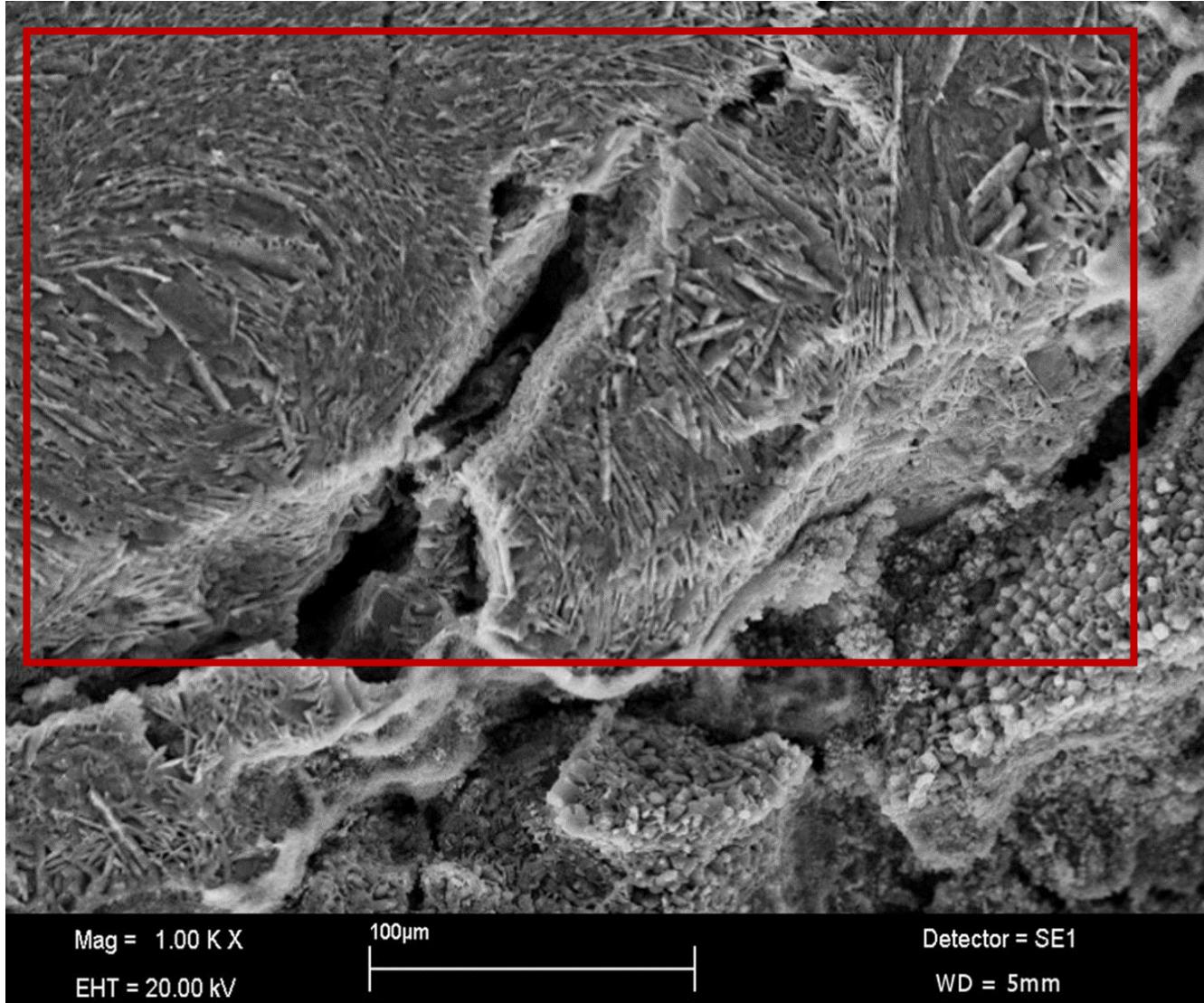
Apparent Strain (%)

0.11

0.11

No significant variations in the apparent crystallite size
&
in the apparent strain

Microstructural Analysis – SEM



SEM analysis

No evident differences in the average grain size

Dendritic regions can be observed in the Inductive HT sample (not in Standard HT)



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Conclusions & Outlook

Alternative route to synthesize good quality MgB_2 samples in few minutes instead of hours

In-field performance improved → increased B_{C2} → higher disorder in the system

Microstructural investigation: in Inductive HT dendritic-like structure, no other clear evidences of enhanced disorder

Other advantages of the proposed route:

- Heat treatment parameters inaccessible with traditional furnaces
- Scalable for powders and wires production

Study the effect on disorder generated by “C doping + rapid synthesis” to evaluate B_{C2} and J_C improvements in MgB_2 bulks

Thank you for your attention !