



# **Structural and magnetic properties of cast iron for cyclotrons**

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# Outline

- Introduction
  - Context and purpose
- Iron samples
- Investigations of the microstructure
  - Grain size, Scanning Electron Microscopy (SEM) analysis, Energy Dispersive Spectroscopy (EDS) X-ray Diffraction (XRD) detector analysis
- Magnetic measurements
  - Magnetization curve, hysteresis loop
- Efficiency of heat treatment and internal stress
- Conclusions

# Introduction

## Context and purpose

- IBA CYCLONE230® cyclotron magnet iron is cast, cooled, roughly machined, annealed, machined, transported, precisely machined, transported, assembled etc.
- The suppression of only one operation = cost reduction by few percent
- IBA: one permeameter to routinely measure the magnetization  $BH$  curve
  - Quality control related to different certifications
  - Created library allows different studies and analyses
- Istituto Nazionale di Ricerca Metrologica (INRIM), Torino, Italy
  - Optical microscope, Scanning Electron Microscope SEM, Energy Dispersive Spectrometer X-ray Diffraction detector + software tools + highly skilled people
- Let's analyse the cyclotron magnet iron more extensively...

# Iron samples

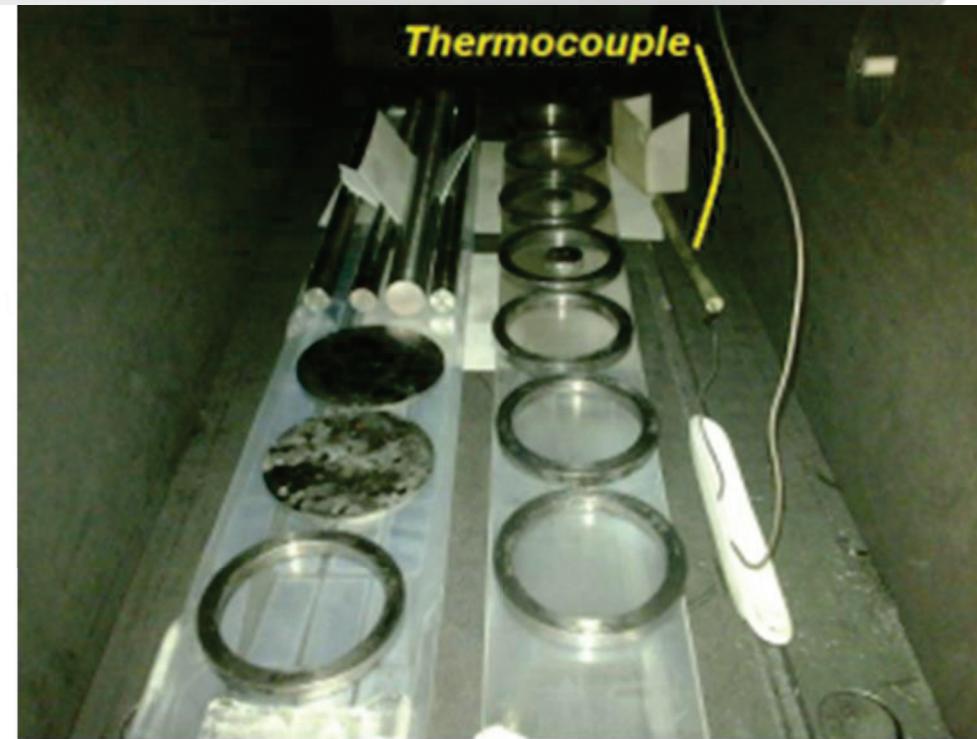
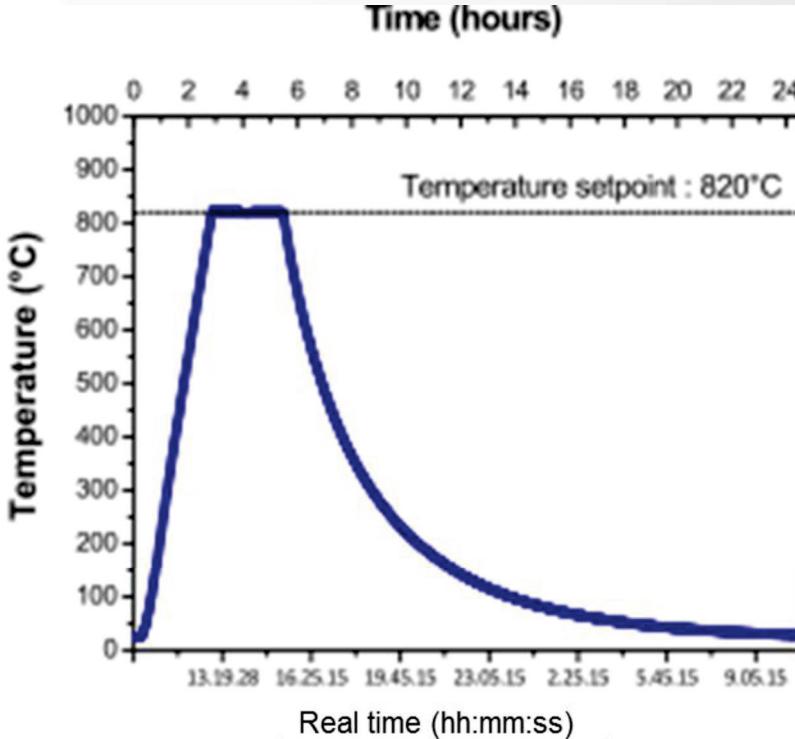
## Variety



- Iron castings from the preferred IBA supplier
- Samples additionally treated:
  - thermal treatments:
    - unannealed
    - annealed
  - different shapes
  - surface preparations:
    - as it is = lathe machined
    - ground, polished and cleaned
    - etched or unetched

# Thermal treatment

## Annealing



- Annealing temperature profile and samples in the oven chamber

# Investigations of the microstructure

## Grain size and measurement problems



- Observations using an optical microscope and an SEM
- Irregularly shaped large grains and inclusions of impurities produce:
  - multiple intersections with an arbitrarily selected test lines
  - underestimate of average intercept length

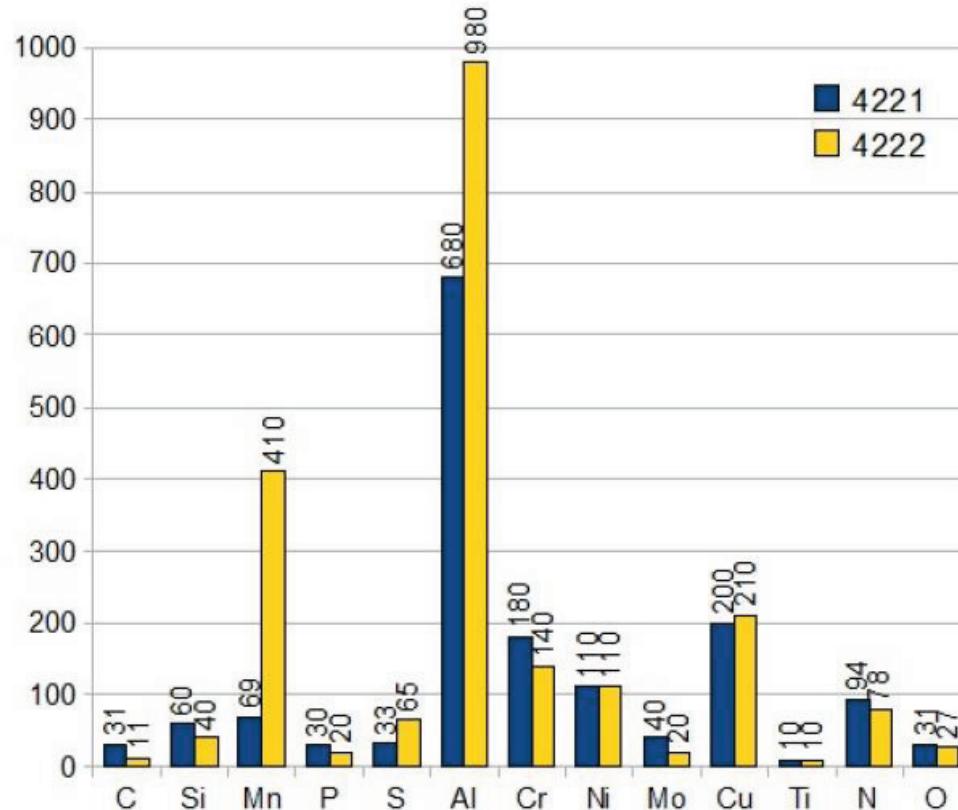
# Investigations of the microstructure

## Grain size

- The average intercept length  $\bar{x}$  is related to the grain size
- The relation  $\sigma > \approx \bar{x}$  indicates the large variation in the grain size
- $0.77 \text{ mm} < \bar{x} < 1.52 \text{ mm}$ ,  $x_{\max} = 8 \text{ mm}$ ;  $0.84 \text{ mm} < \sigma < 1.64 \text{ mm}$
- No changes observed between the as-cast state and the fully annealed state
- Conclusion: The heat treatment does NOT produce:
  - recrystallization in samples
  - grain growth in samples

# Investigations of the microstructure

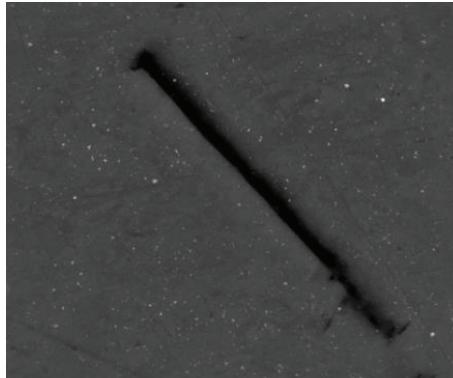
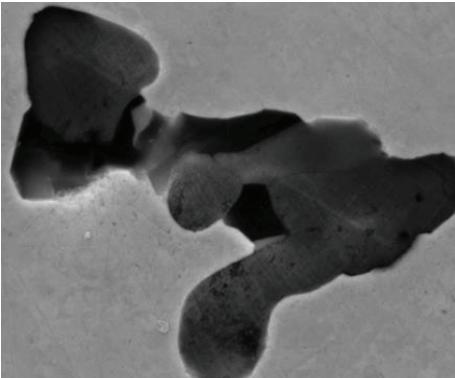
## Chemical composition of impurities



- The chemical composition of the cast iron provided by the supplier
- The impurity content independently determined using an EDS X-Ray Diffraction detector
- EDS XRD analysis regularly detected chemical compounds:
  - AlN – aluminum nitride
  - MnS – manganese sulfide
  - $\text{Al}_2\text{O}_3$  – aluminum oxide
- Agglomerates of any 2 or all 3 compounds created most of inclusions

# Investigations of the microstructure

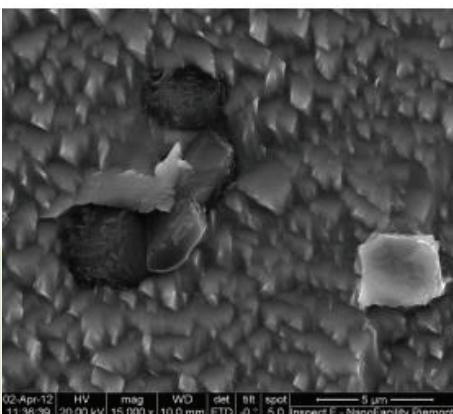
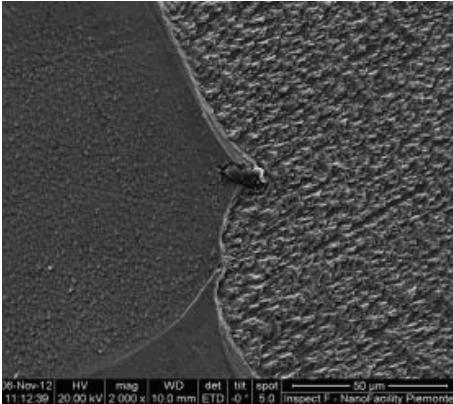
Examples of inclusions in unetched samples



- Unetched samples
- Agglomerates of irregular crystals (different compounds)
- Sharp edge crystal agglomerates
- Bar monocrystal or multicomponent agglomerates
- Cross-shaped crystals

# Investigations of the microstructure

Examples of inclusions in etched samples

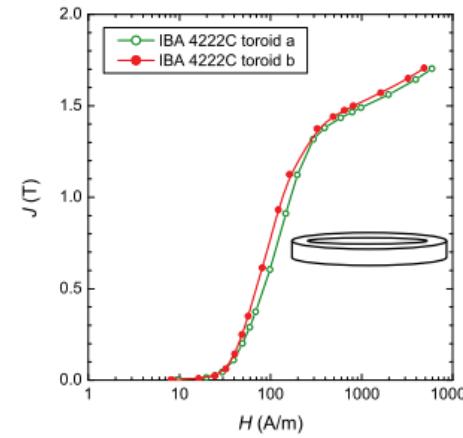
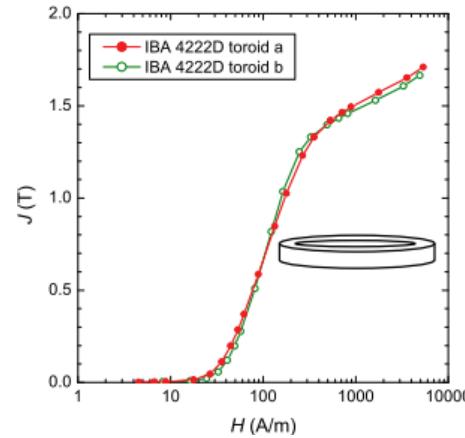


- Inclusions at the grain boundary or within the grain are better revealed by etching
- Surface differences and the strong hardening are observed due to machining in the layer up to 100μm
- Conclusion: After removal of a work-hardened layer there is not any difference between samples as-cast and samples after thermal annealing

# Magnetic measurements

## Ring samples

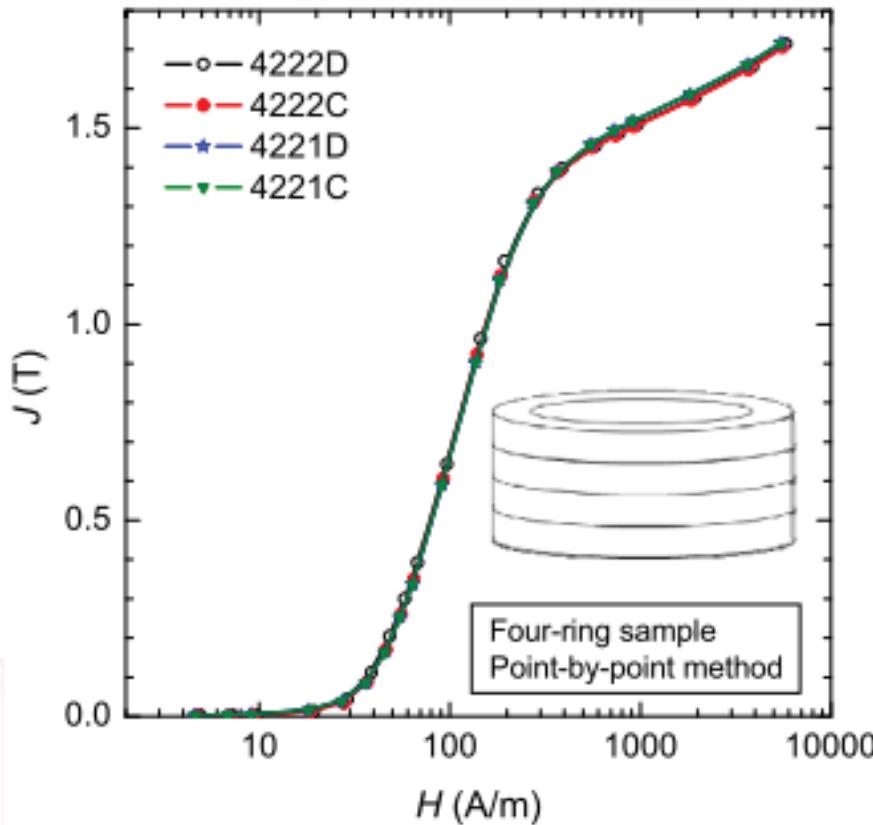
- The point-by-point « ballistic » method and the continuous hysteresisgraph measurements used



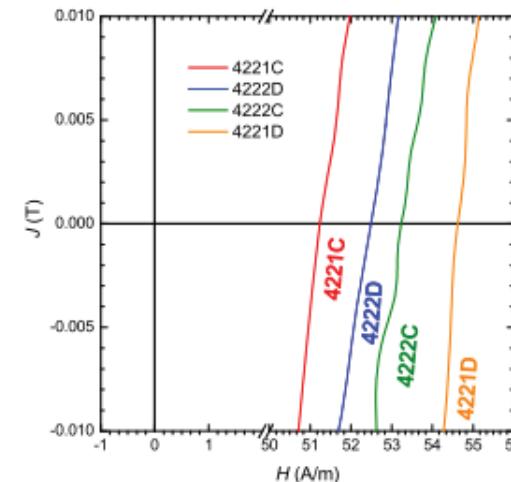
- For one ring sample, a lack of reproducibility of the hysteresis curve is observed due to coarse grain structure

# Magnetic measurements

## Ring samples

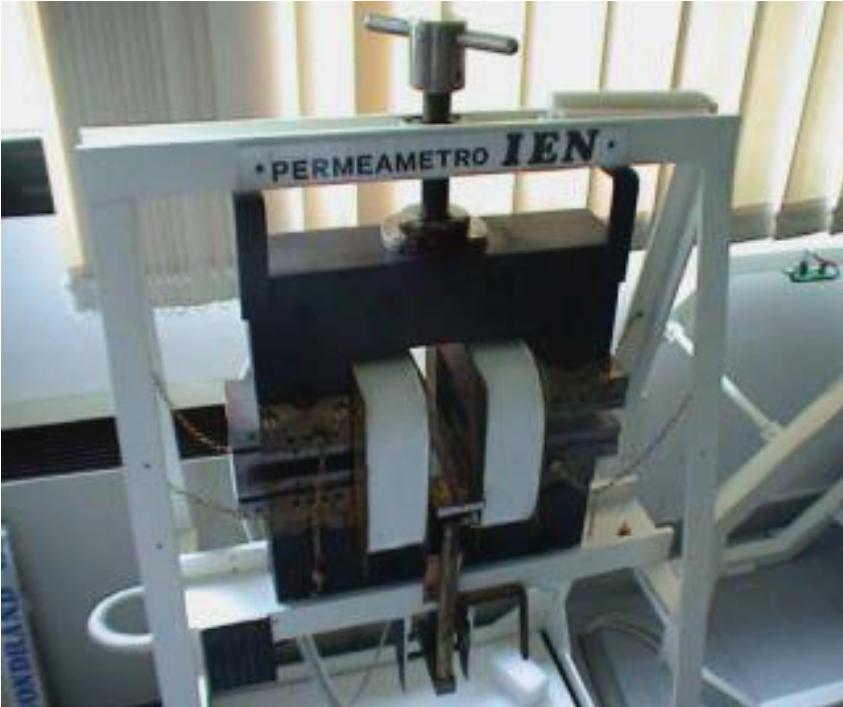


- Grouping (stacking) of the ring samples from the same batch and location produces nearly identical hysteresis curves
- Coercivities between 51-54 A/m observed after grouping



# Magnetic measurements

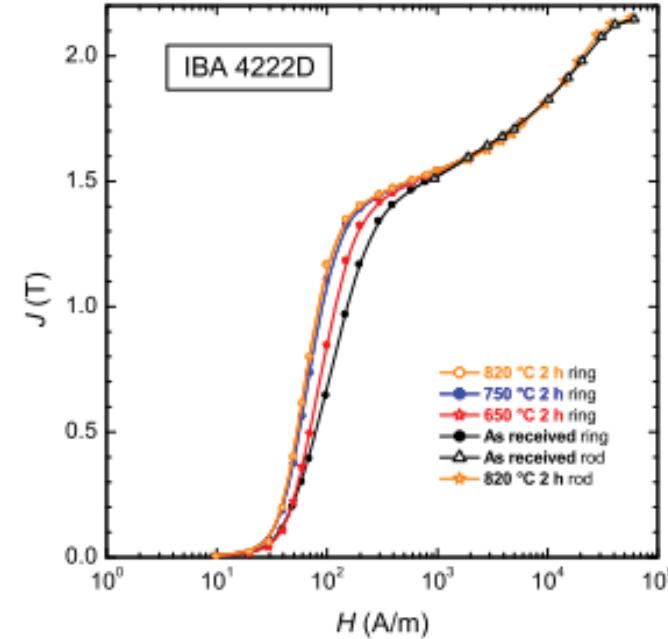
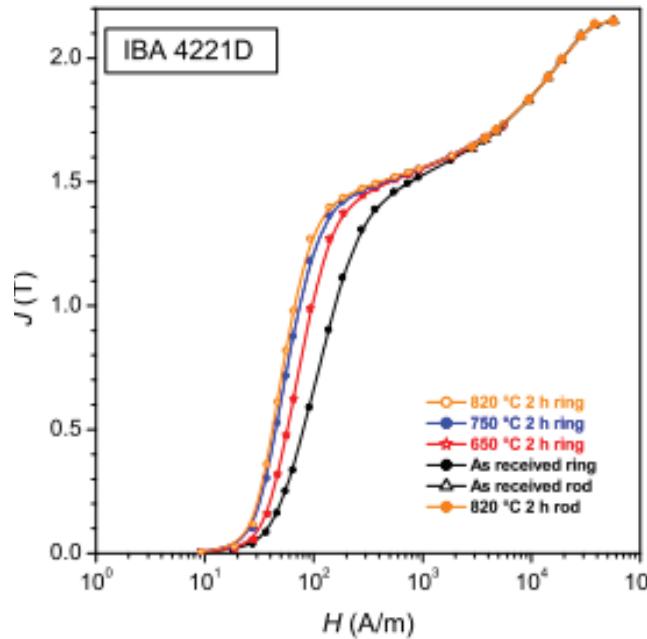
## Rod samples – experimental set-up



- Permeameter set-up
- The coarse grain structure produced important differences in the hysteresis curve at different points along the rod
- Averaging with respect to rod segment length was necessary
  - Magnetic flux measured by over the central 30 mm segment of the rod
  - Three Hall probes at different positions over 30 mm segment
- Chemical etching slightly reduces magnetic response at low magnetic field intensity  $H$

# Magnetic measurements

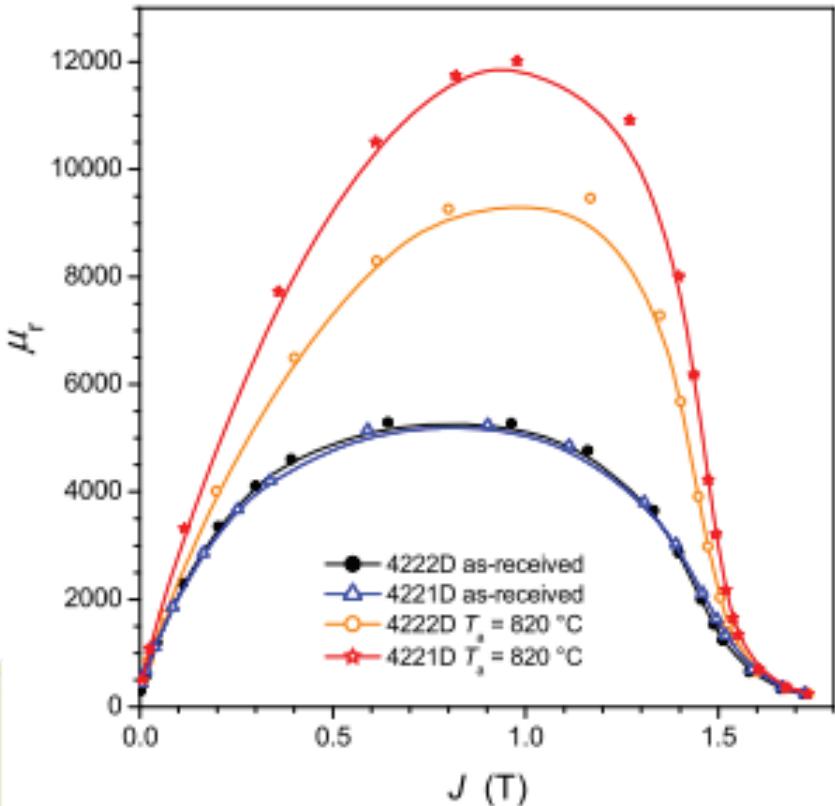
Magnetization and annealing temperature



- Magnetization curves for different annealing temperatures, different sample types and locations

# Magnetic measurements

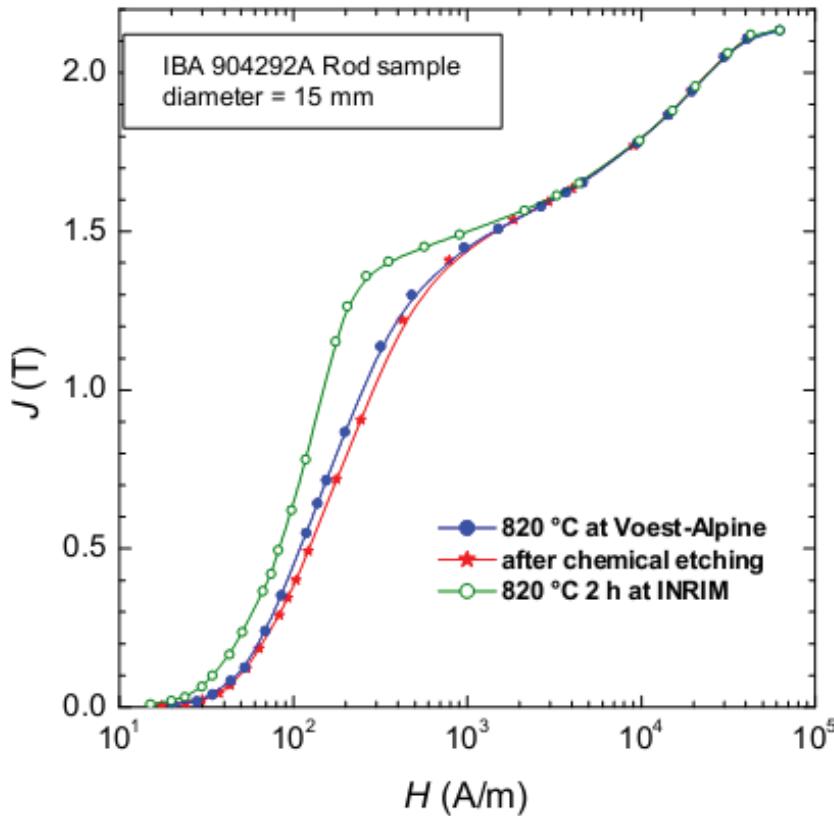
## Magnetization and annealing temperature



- The relative permeability  $\mu_r$  increases with annealing temperature
- All magnetization curves are unaffected by annealing for the magnetization  $J = B - \mu_0 \cdot H > 1.6$  T
- The increase of the relative permeability is interesting for production of IBA PET cyclotron magnets where acceleration of negative ions requires lower fields
- Unfortunately IBA PET cyclotrons are produced from laminated and/or forged iron slabs

# Efficiency of heat treatment and internal stress

Re-annealing has an effect

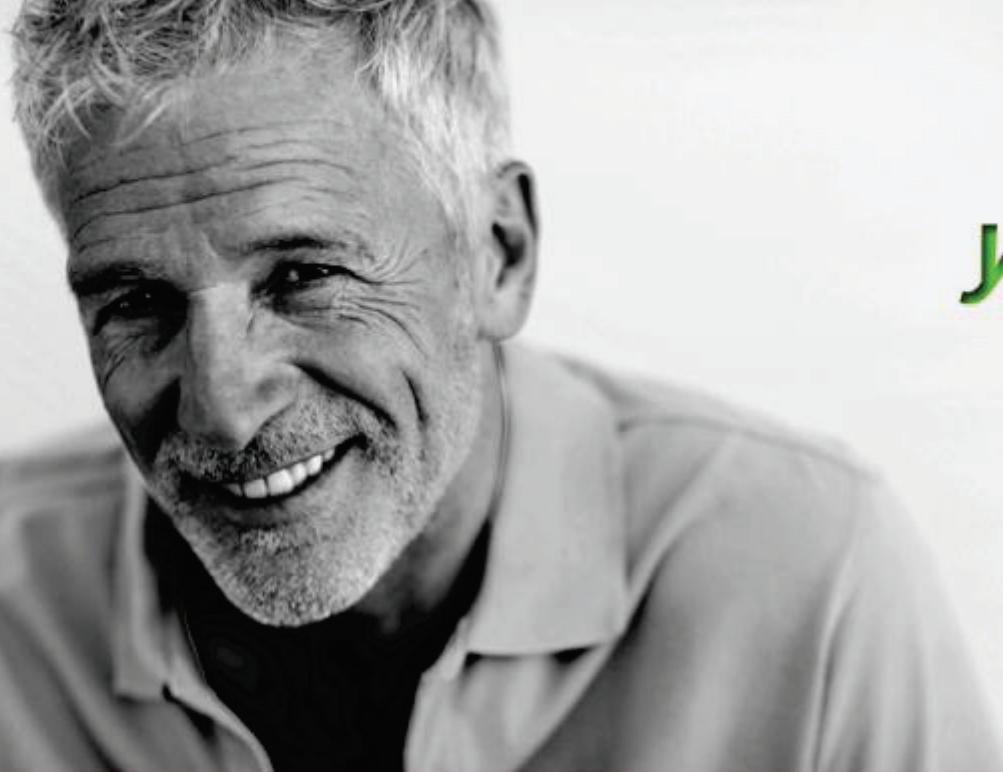


- Annealing at 820C effectively releases the stress probably accumulated during non-uniform cooling of cast iron
- Re-annealing in the lab typically has the same effect as on non-annealed sample
- IBA protontherapy C230 cyclotron has a magnetic field induction  $B > 1.75$  T in the machine center
- Therefore reannealing is practically without significant effects for C230

# Conclusions

## Summary

- Thermal annealing as performed up to date:
  - No effects on the grain size. The grain size already big for « as cast » iron
  - No migration of inclusions, no shape change of inclusions, no changes of directional orientation of inclusions and a good chemical composition confirmed
  - Iron softening observed on magnetization curves but only at magnetic fields induction  $B$  below 1.6 T.
  - Modest effects due to the annealing performed by the iron supplier
- The annealing treatment of the iron cast for CYCLONE230® cyclotrons can be suppressed in the future without significant changes of iron quality
  - Reduced cost of production
  - The know-how gained for other and future IBA cyclotrons...



*what do  
you think?*

**Thank you**

*iBa*