Quenching and partitioning heat treatment on a ductile cast iron

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Introduction

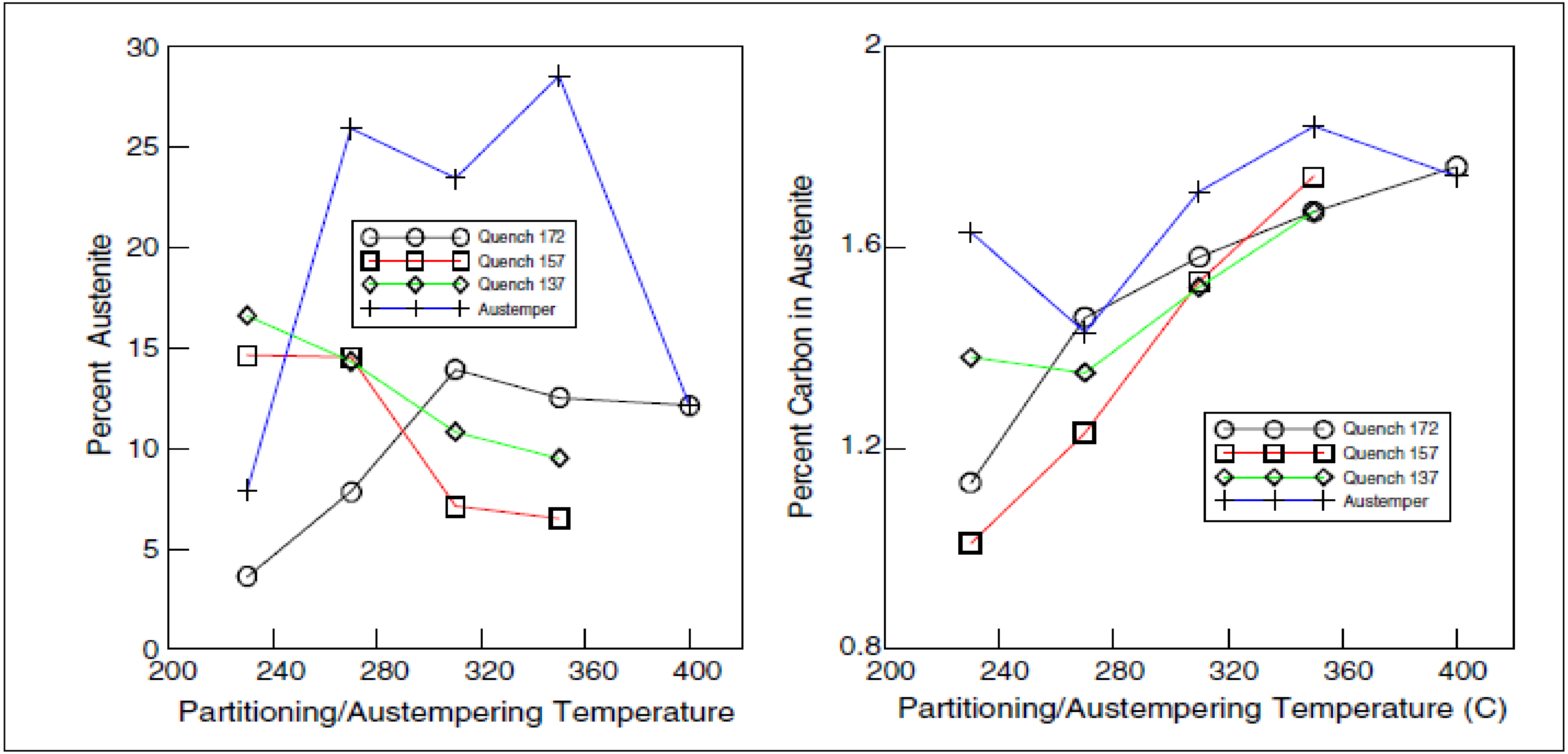
- In this work, the kinetics and microstructural evolution during Q&P of an spheroidal (ductile) graphite cast iron is studied.
- Q&P (Speer et al, 2003) is a new route to obtain steels with multiphase microstructures composed of a martensite matrix and substantial amounts of carbon-stabilized retained austenite. Martensite confers high strength, while stabilized austenite favor good ductility due to occurrence of transformation-induced plasticity (TRIP) effect and/or enhanced strain hardening .
- The choice of the material is justified by the extensive body of knowledge, both scientific and technological existing on austempered ductile iron (ADI), a material with a similar mixture of carbon stabilized austenite and acicular ferrite, obtained through a processing route involving the interrupted bainite transformation (bainite stasis) before carbide precipitation ADI.
- ADI, while being a established route for obtaining cast iron parts with an matrix of acicular bainitic ferrite containing stabilized austenite films (ausferrite), with excellent mechanical properties, have being loosing competition against conventional quenched & tempered forged steel parts due to the need for costly and environmentally harmful isothermal salt baths treatments.

Introduction

- If an ausferrite-like structure can be obtained by Q&P, without the need for costly salt bath treatments, and its properties proved to be equal or superior to ADI, then a new class of cast iron materials will be available for engineering purposes.
- This idea was proposed by Speer and initially tested by a group of his students: ”Unger S, Grahmann J, Shutts A, Wolf C, Gibbs J. Quenching and partitioning of ductile cast iron, Colorado School of Mines Internal Report, 2004” (Apud Speers et al, 2003)
- This work is part of a larger project with the objective of demonstrating the feasibility of a new processing route for ductile cast iron , Q&P “quenching and partitioning”, understanding the phase transformations involved and measuring the mechanical properties of the products.
- **Questions of ALEMI interest are whether the athermal martensite is really immobile during the partition, the eventual occurrence of isothermal martensite above Ms (at *swing back* range) and the precipitation of nanobainite.**

Quenching & Partitioning in Nodular Cast Irons

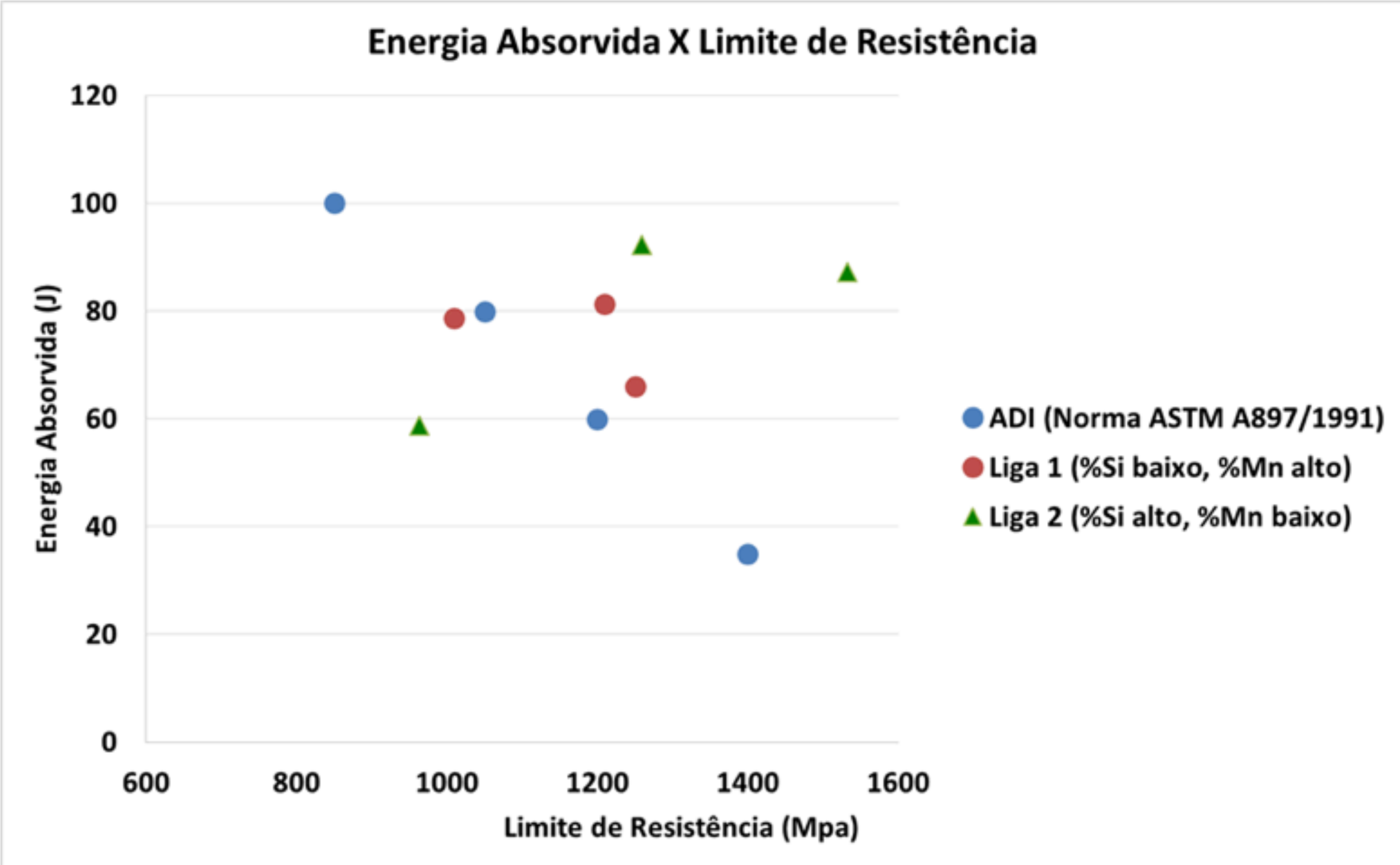
Spheroidal graphite cast iron with 2.5%Si, 0.34%Mn and 0.17%Cu
Austenitization 850°C/2h, quenching at 137,157 e 172°C.
Partition at 230 and 390°C/30 minutes



Source: Unger S, Grahmann J, Shutts A, Wolf C, Gibbs J. Quenching and partitioning of ductile cast iron. Colorado School of Mines Internal Report, 2004 (Apud Speers et al)

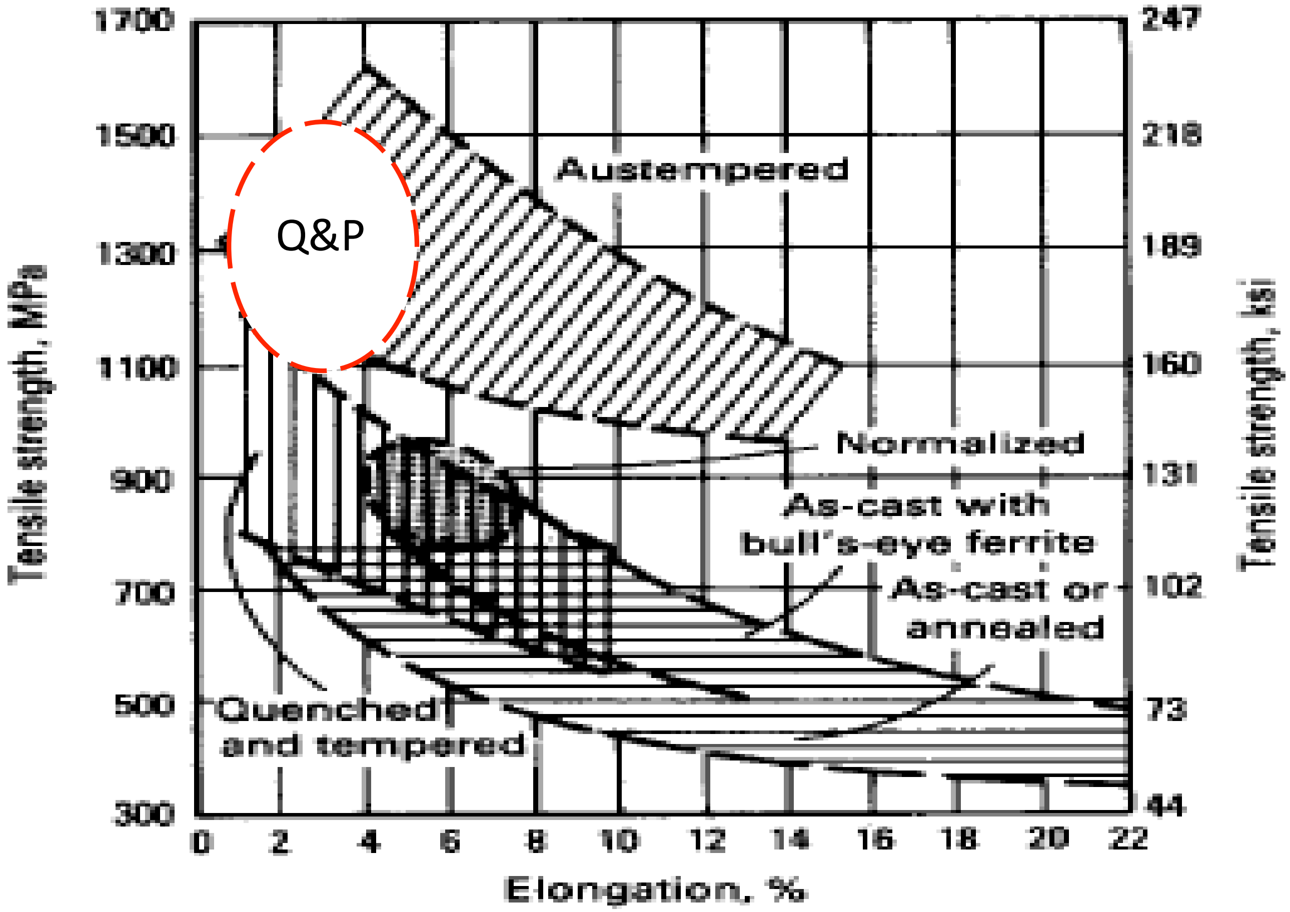
Anderson Tomas MsC thesis: mechanical properties of ADI vs Q&T cast iron

- “banana plot” comparison between the mechanical properties of ADI cast iron according to ASTM A897/1990 and Q&T: quite similar properties despite the cast iron being strongly segregated, with high Mn intercellular regions and high Si near nodules.

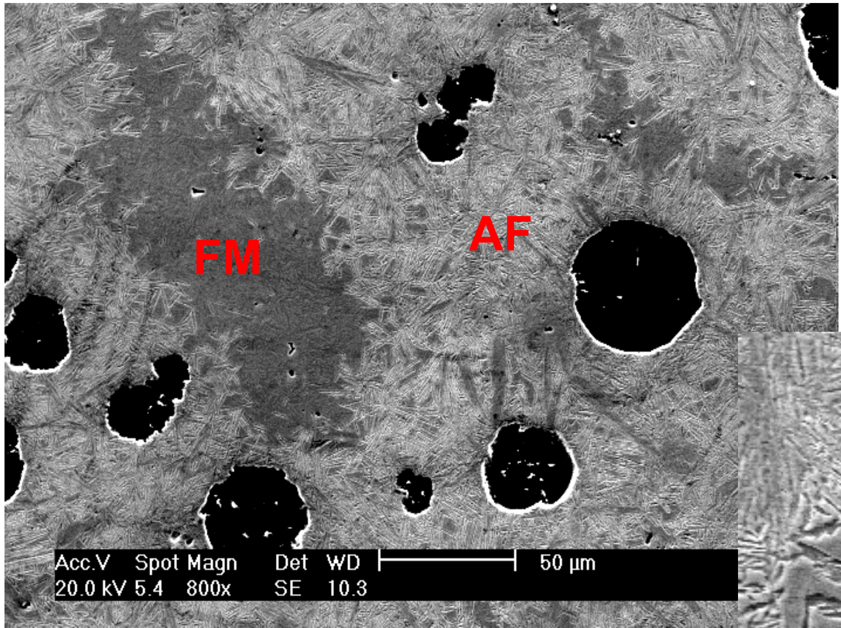


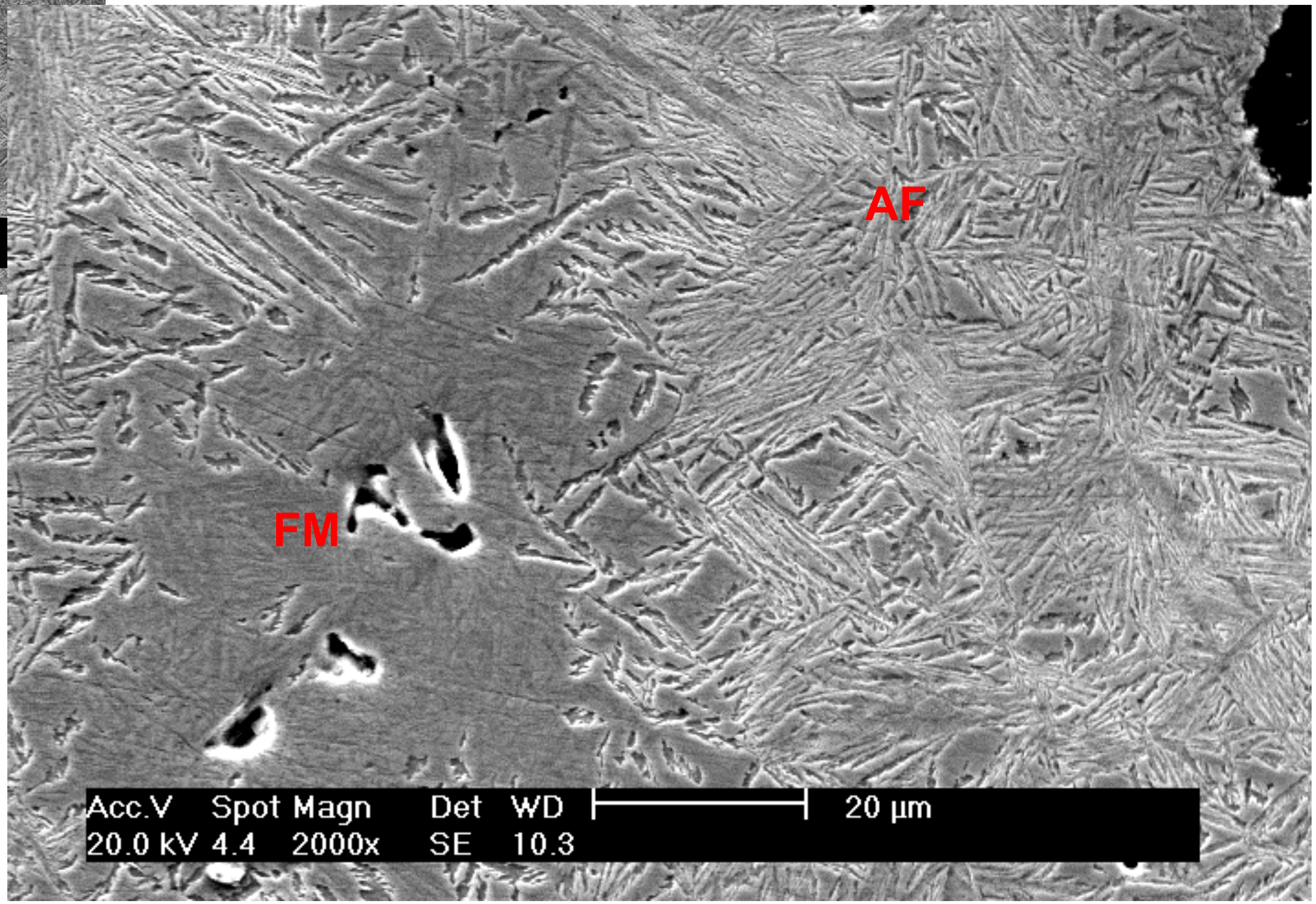
Mechanical properties of ADI vs Q&T cast iron

- Tensile Strenght X Elongation diagram situate Q&P SG cast iron inside the field of properties displayed by ADI



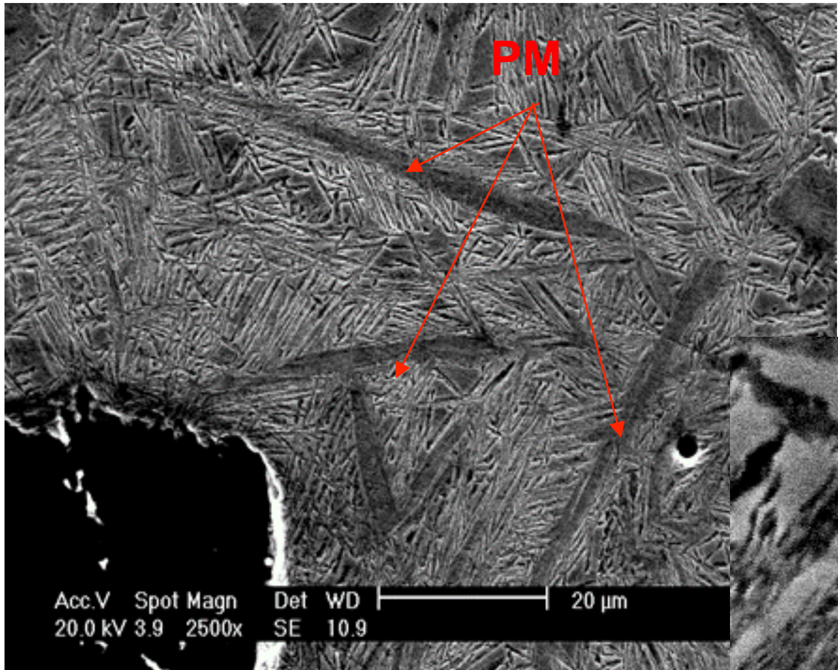
Segregation of Mn into the intercelular region was the problem found by early work (Anderson Tomaz)

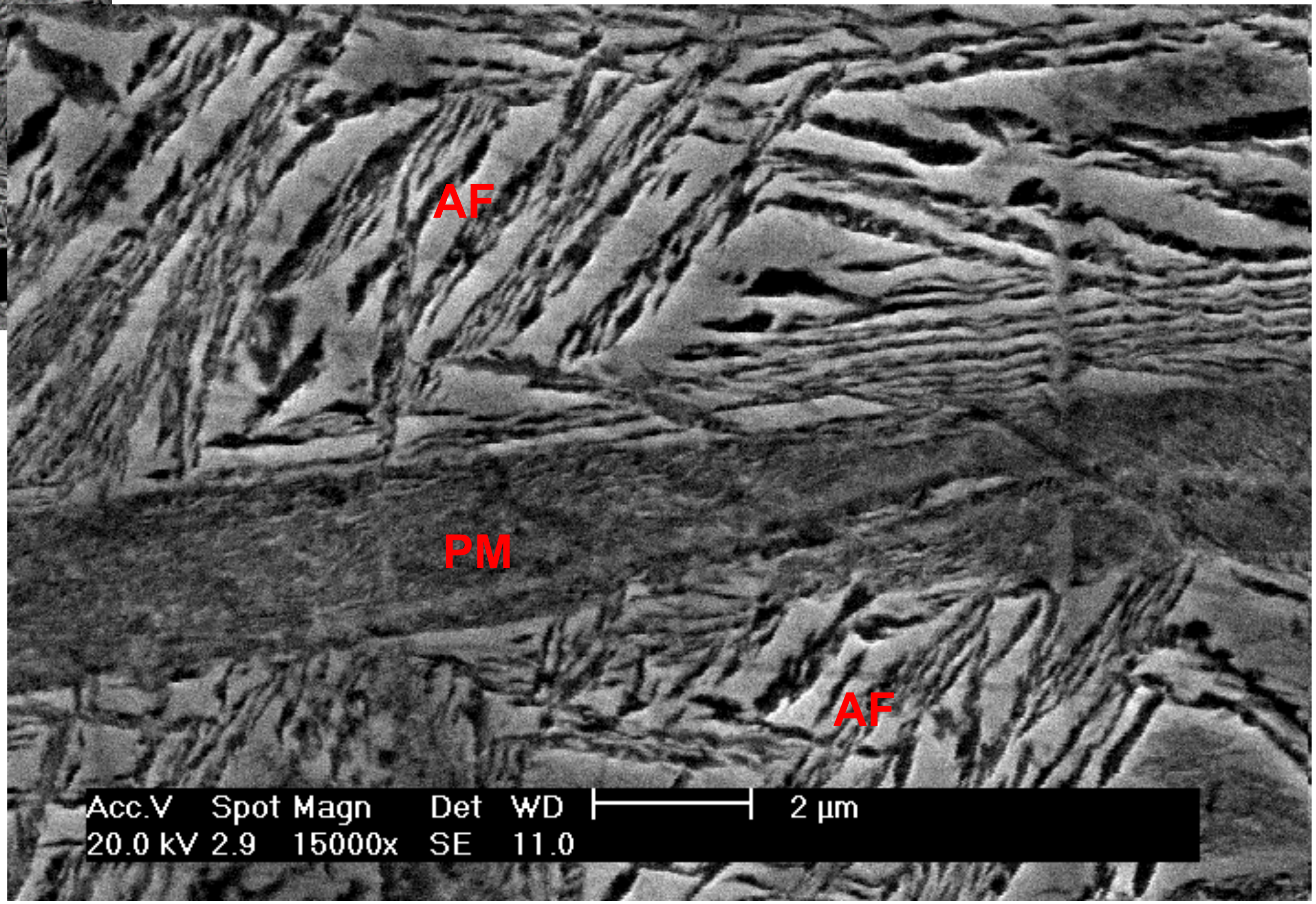




FM = Fresh Martensite (high %C)

AF = Ausferrite



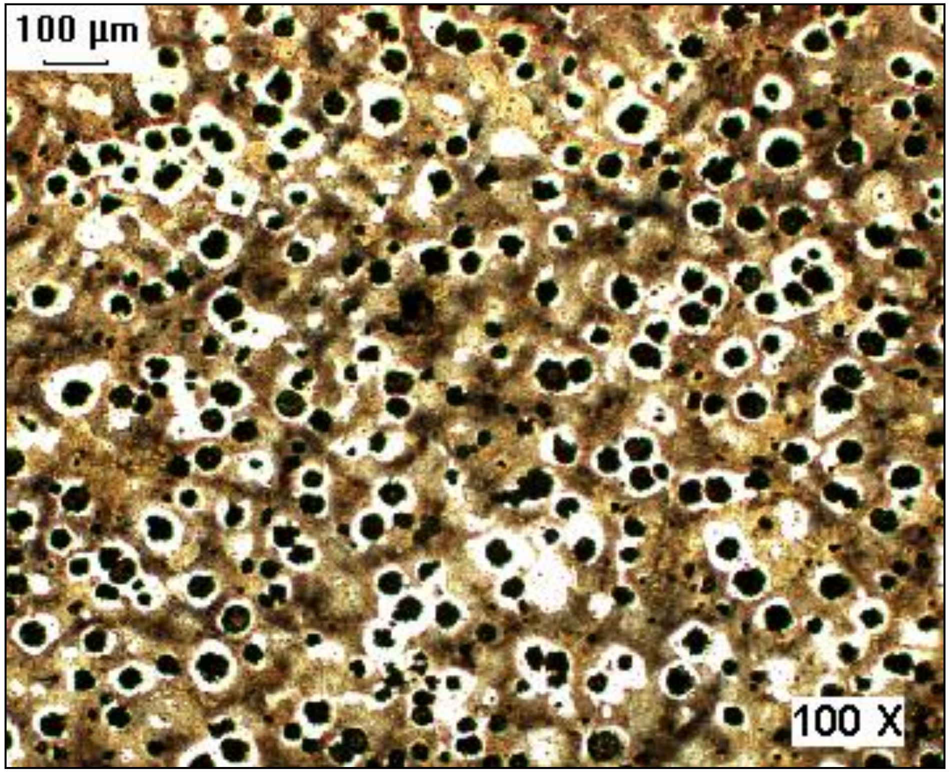


PM = Partitioned Martensite (low %C)

AF = Ausferrite

Experimental procedure

- Alloy cast as “Y” blocks at Tupy Fundições S.A.
- Start with a spheroidal graphite cast iron with minimum segregation: low Mn and extreme inoculation
- 3.5%C, 2,5%Si, 0.2%Mn, 0.04%P, 0.006%S, 0.03%Cr, 0,4% Cu 500+ nodules per mm^2, mostly pearlitic structure

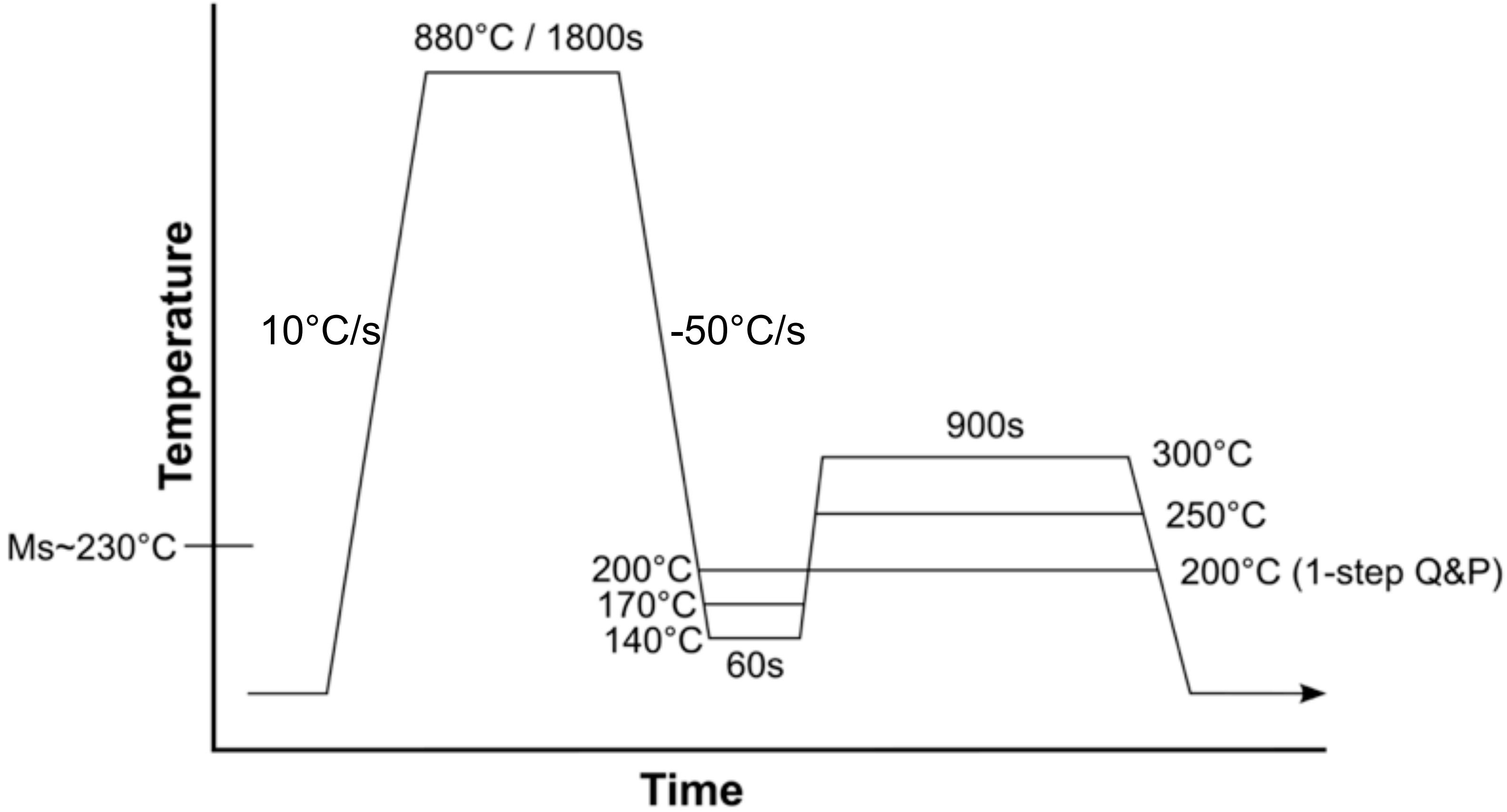


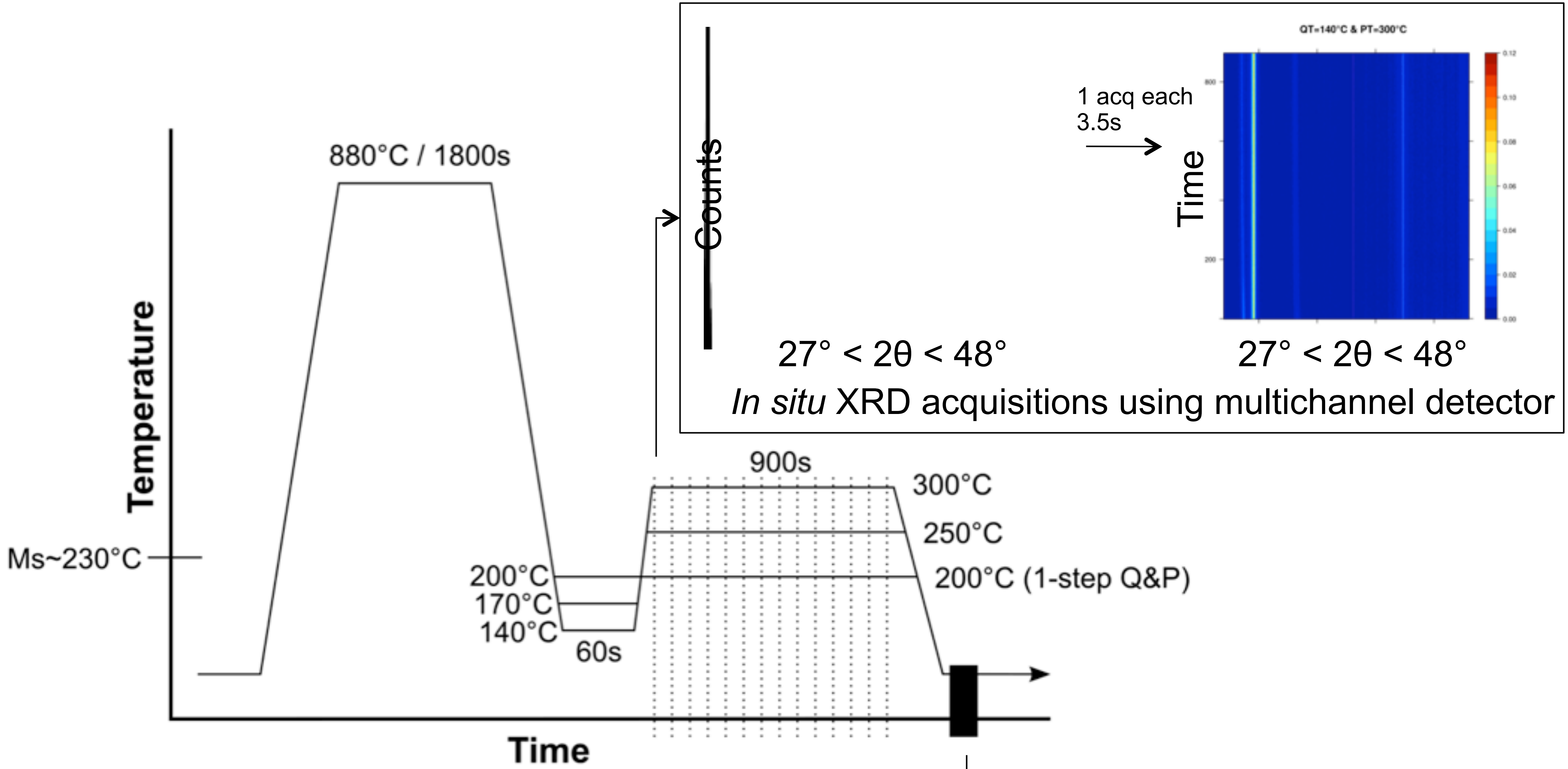
Experimental procedure

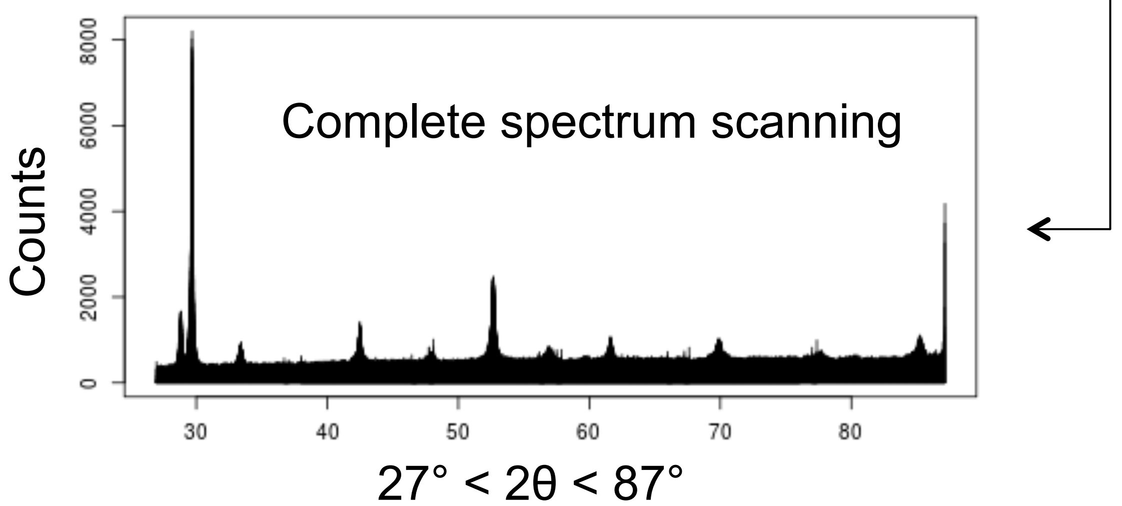
- Heat treatments were conducted on a Bähr 805A dilatometer and at the XTMS experimental station facilities at Brazilian Synchrotron Laboratory (LNLS) on a customized Gleeble thermo-mechanical simulation machine
- The evolution of phases was monitored by means of *in situ* X-ray diffraction and by dilatometry.
- Real time information about the kinetics of carbon-enrichment of austenite and competitive reactions were obtained based on those results

Experimental procedure

Austenitizing temperature AT, was 880ºC
Quenching temperatures QT were 200ºC, 170ºC and 140ºC (Ms temperature ~230ºC)
Partitioning temperatures (PT) were 200ºC (1 step Q&P), 250ºC and 300ºC for 90s.

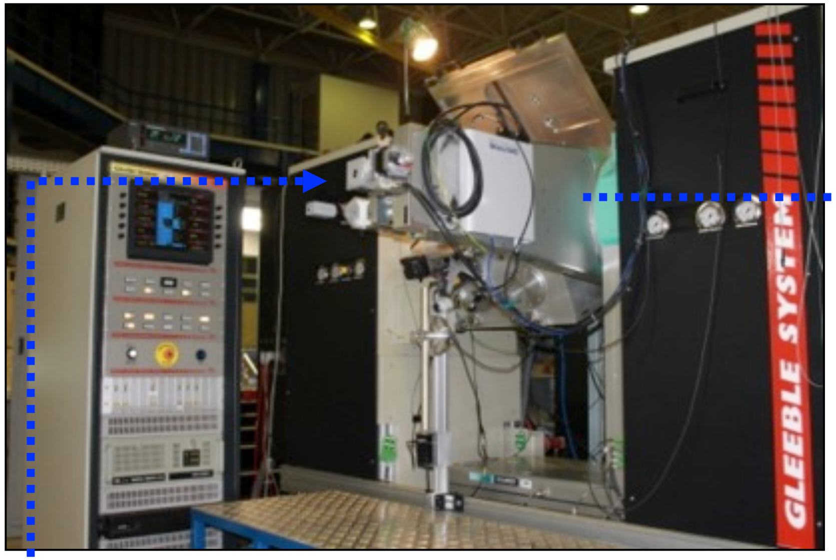


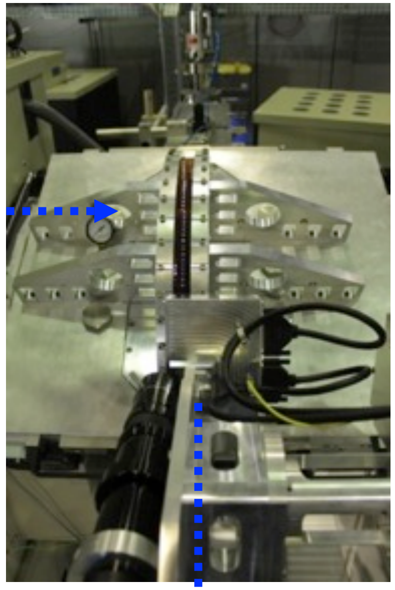


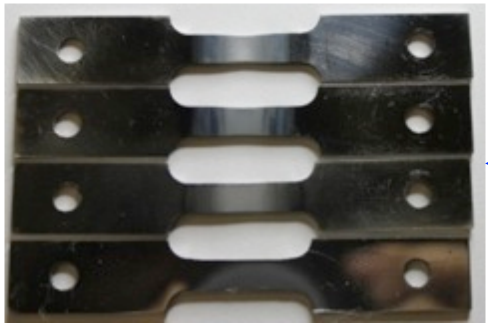


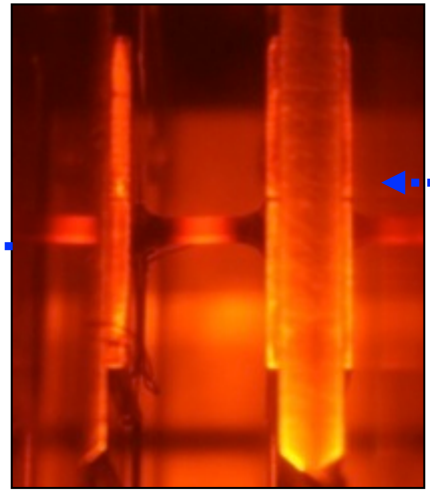
-Beam energy: 12keV → $\lambda = 1.033$Å

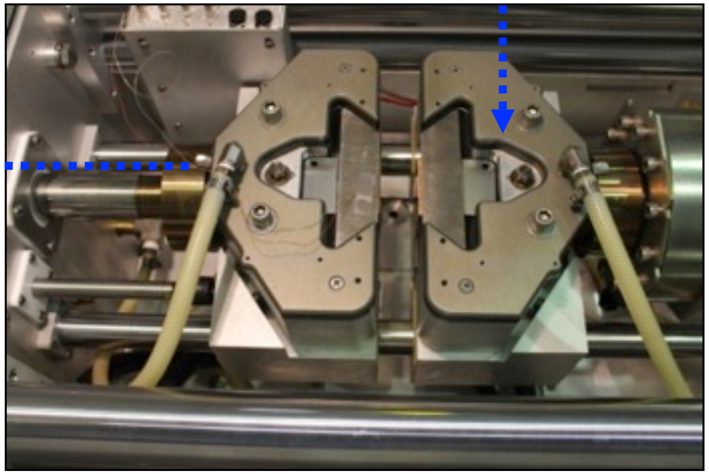
XTMS experimental station facilities – *Brazilian Synchrotron Laboratory (Campinas – Brazil)*











Photos courtesy of Anderson Ariza

Raw data

Peak fitting using Gaussian function (i)

$$I = I_0 \exp\left[-\left(\frac{2\theta - 2\theta_0}{w}\right)^2\right]$$

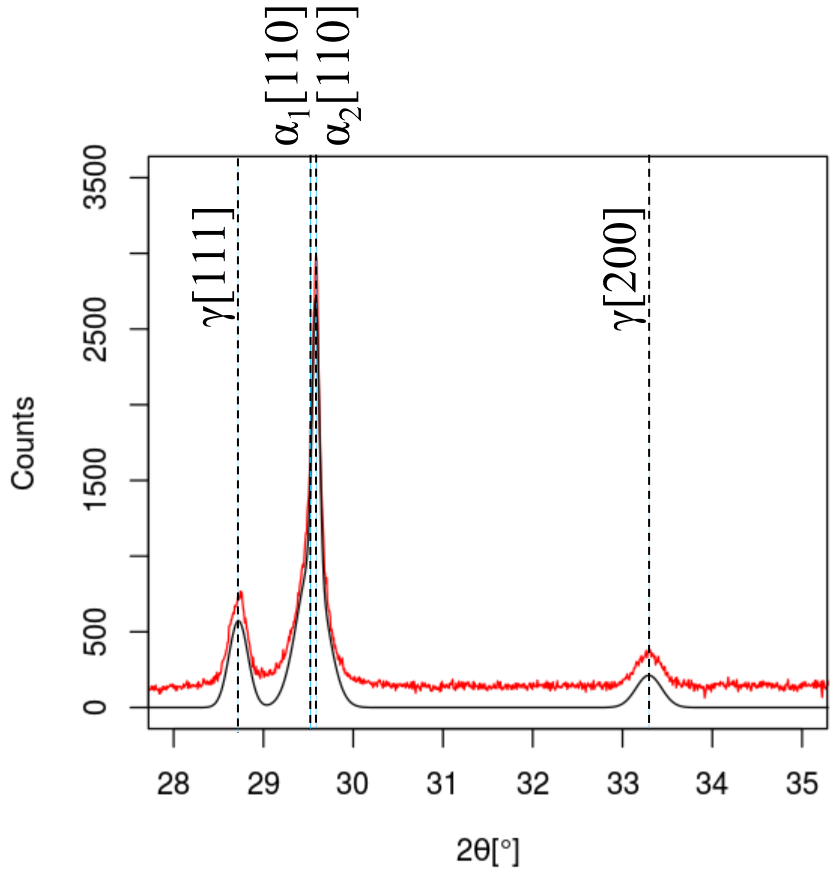
Calculation of α and γ volumetric fractions

Peak integration:

$$A = \int_{-\infty}^{\infty} I_0 e^{-\left(\frac{2\theta-2\theta_0}{w}\right)^2} d\theta = $$
$$= I_0 |w| \sqrt{\pi}$$

Comparison of integrated intensities with calculated peak intensities

$$f_\gamma = \frac{A_\gamma/R_\gamma}{A_\alpha/R_\alpha + A_\gamma/R_\gamma}$$



Calculation of γ carbon content

Bragg's law → convert $2\theta_0$ to lattice parameter of austenite (a_0).

In situ experiments were made at PT → transform a_0^{PT} to a_0^{300K} using lattice expansion equation. (ii)

$$\frac{\Delta L^\gamma}{L_0^\gamma} = B_\gamma T + B_\gamma \Theta_D^\gamma \left[\exp\left(\frac{-T}{\Theta_D^\gamma}\right) - 1\right]$$

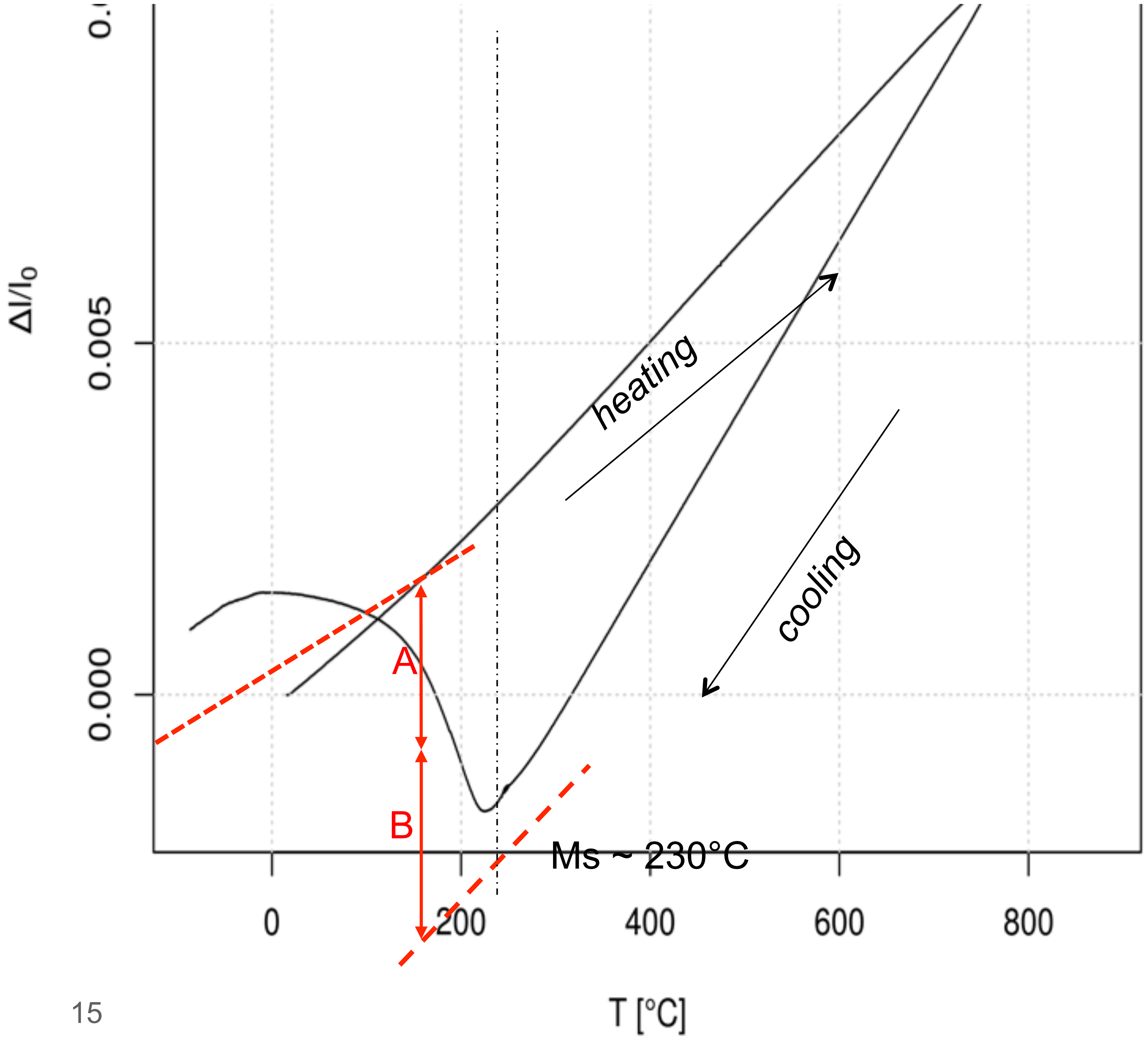
where $B_\gamma = 24.8\times10^{-6}$ K^{-1} and $\Theta_D^\gamma = 280$ K

Compute carbon content using *lattice parameter--carbon content relationship* (iii,iv)

$$a_\gamma = 3.5780 + 3.3\times10^{-2}\%wt_c + $$
$$+9.5\,10^{-4}\%wt_{Mn} + 6\times10^{-4}\,\%wt_{Cr}$$

(i) Babu SS, Specht ED, David SA, Karapetrova E, Zschack P, Peet M, Bhadeshia HKDH. Metall Mater Trans A 2005;36:3281.
(ii) van Bohemen SMC. Scr Mater 2013;69:315.
(iii) Bhadeshia HKDH, David SA, Vitek JM, Reed RW. Mater Sci Technol 1991;7:686.
(iv) Dyson D, Holmes B. J Iron Steel Inst 1970;208:469.

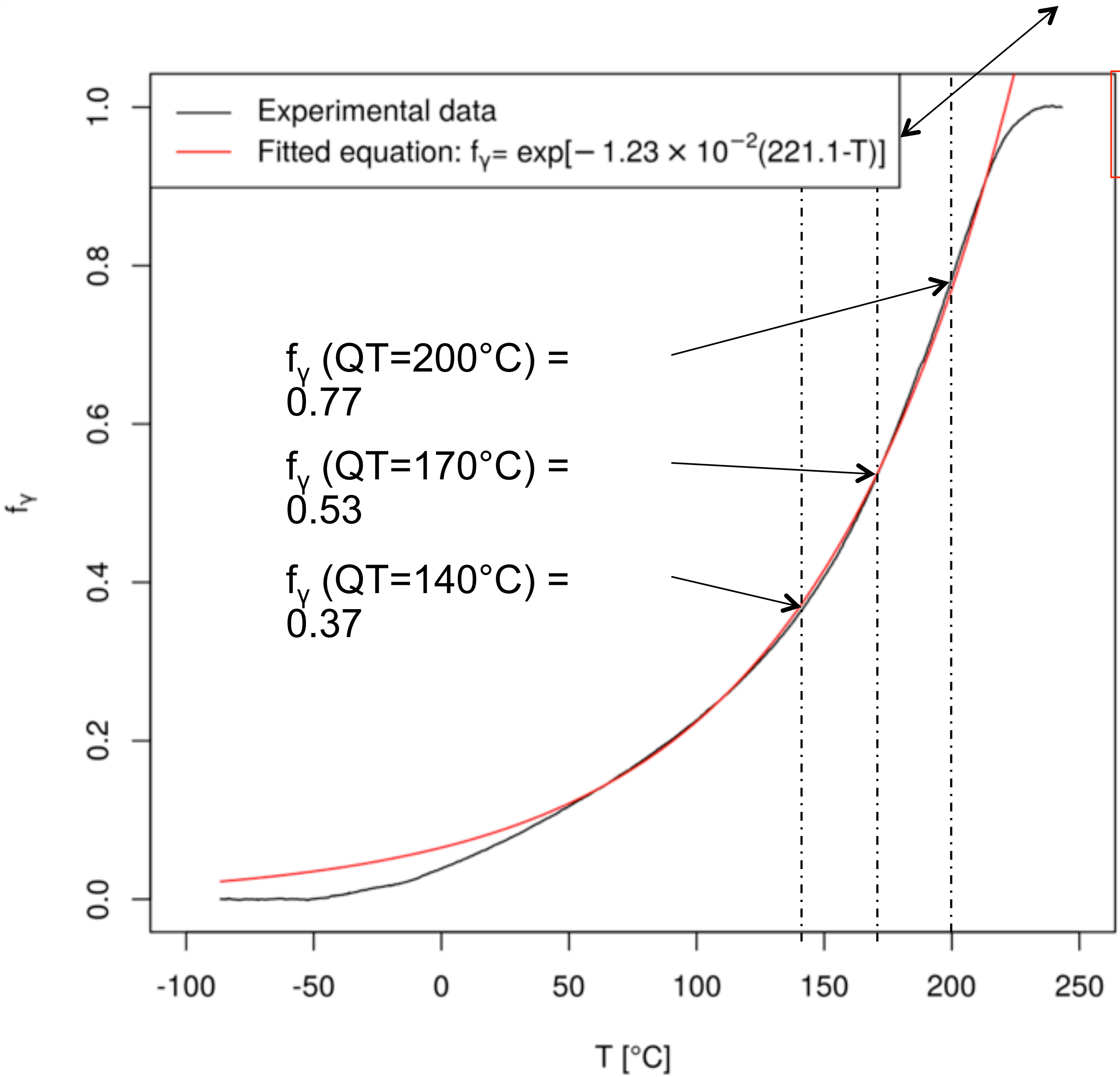
Results: Ms determination



Level rule:

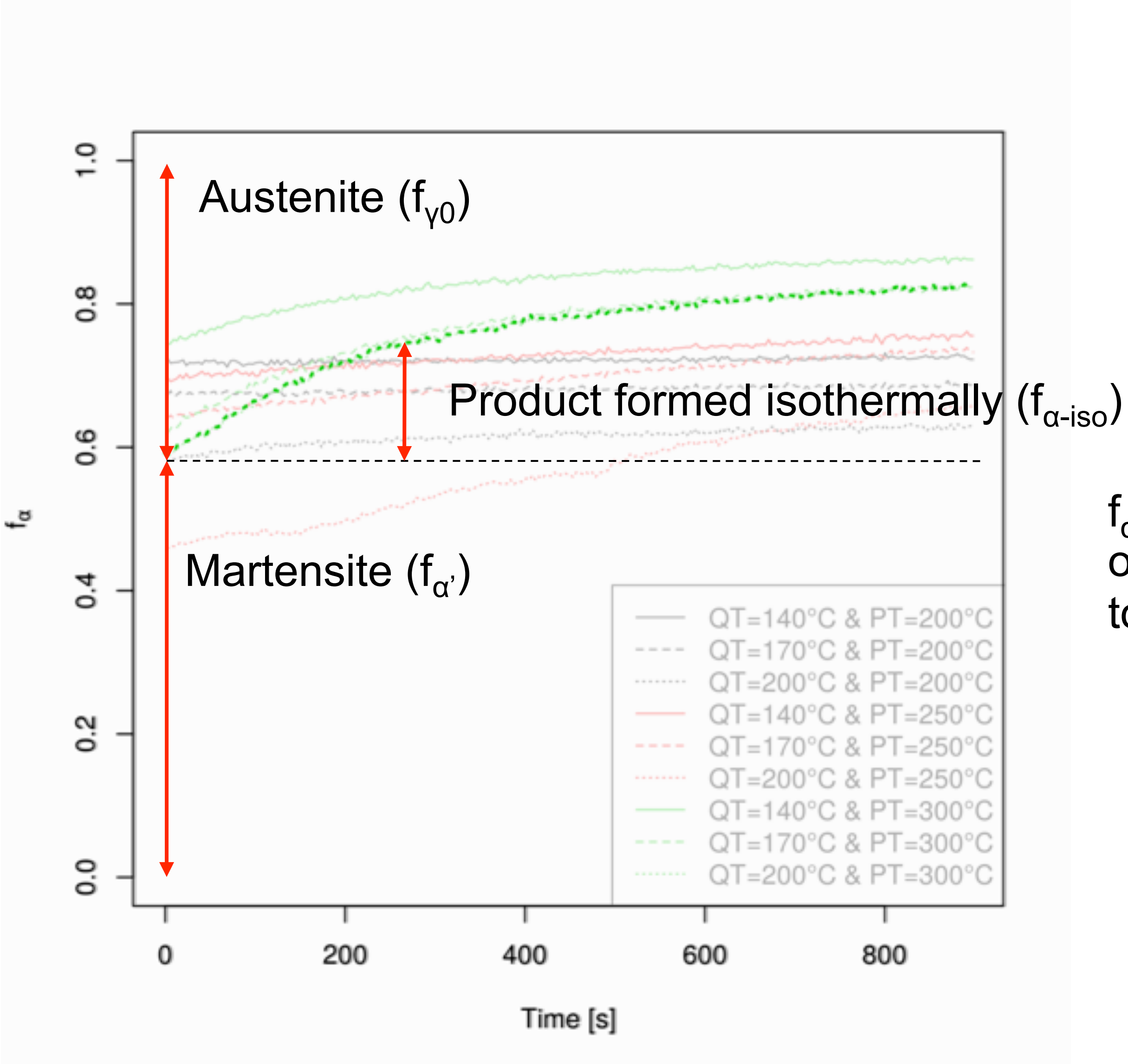
$$f_\gamma = \frac{A}{A+B}$$

Results: Ms determination



Kostinen-Marburger:
$f_\gamma = \exp[-1.1 \times 10^{-2}(Ms\text{-}T)]$

Results: Evolution of bcc phase fraction (f_α) during partitioning step

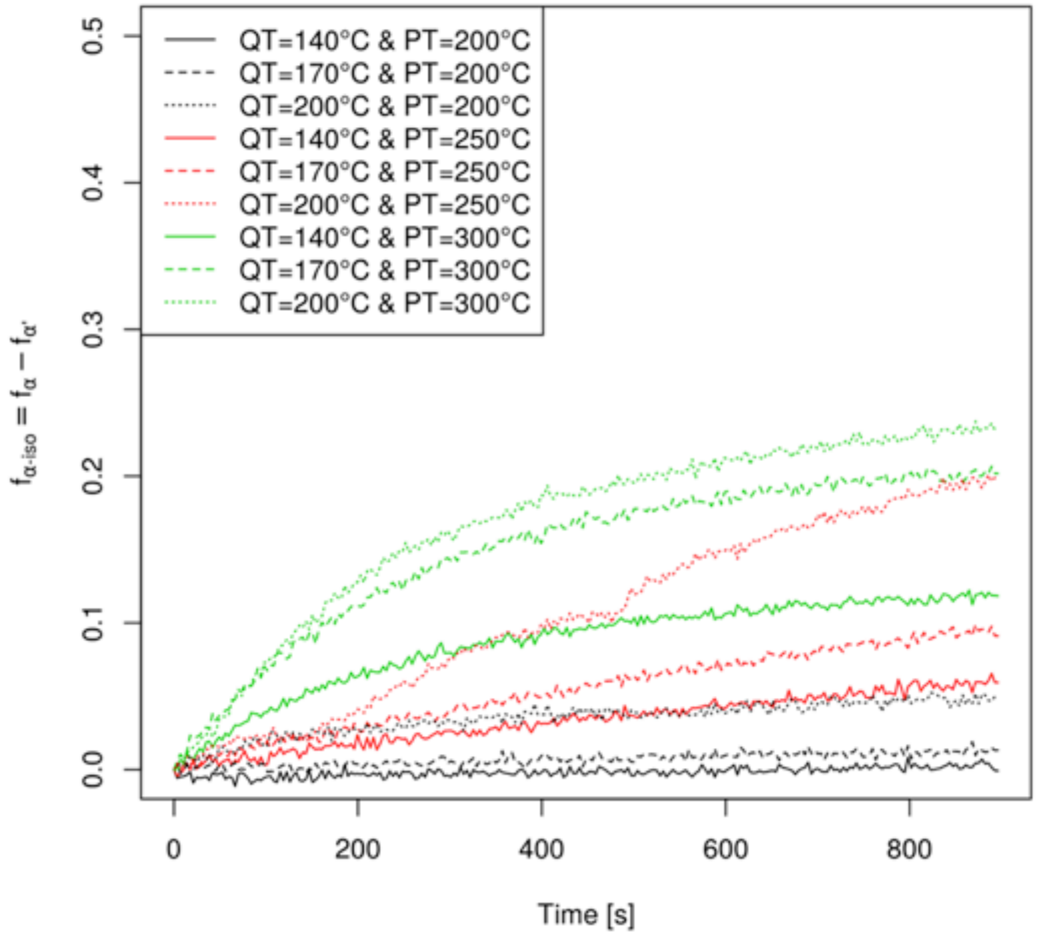


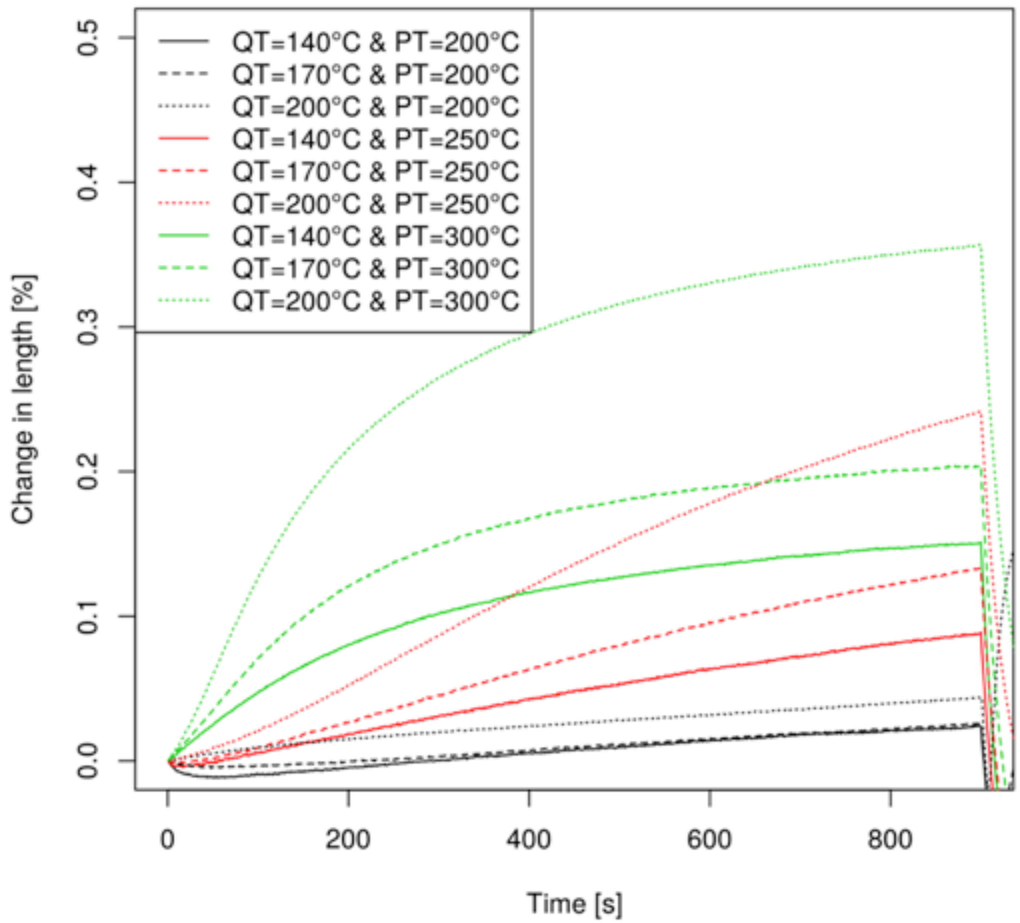
$f_{\alpha'}$ values are higher than values obtained by dilatometry probably due to ferrite formation during quenching

Results: formation of α-iso

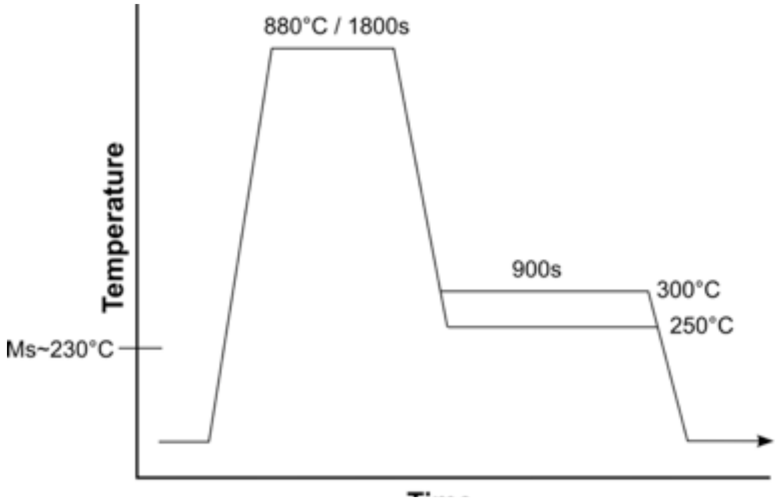
$$f_{\alpha\text{-iso}} = f_\alpha - f_{\alpha'}$$

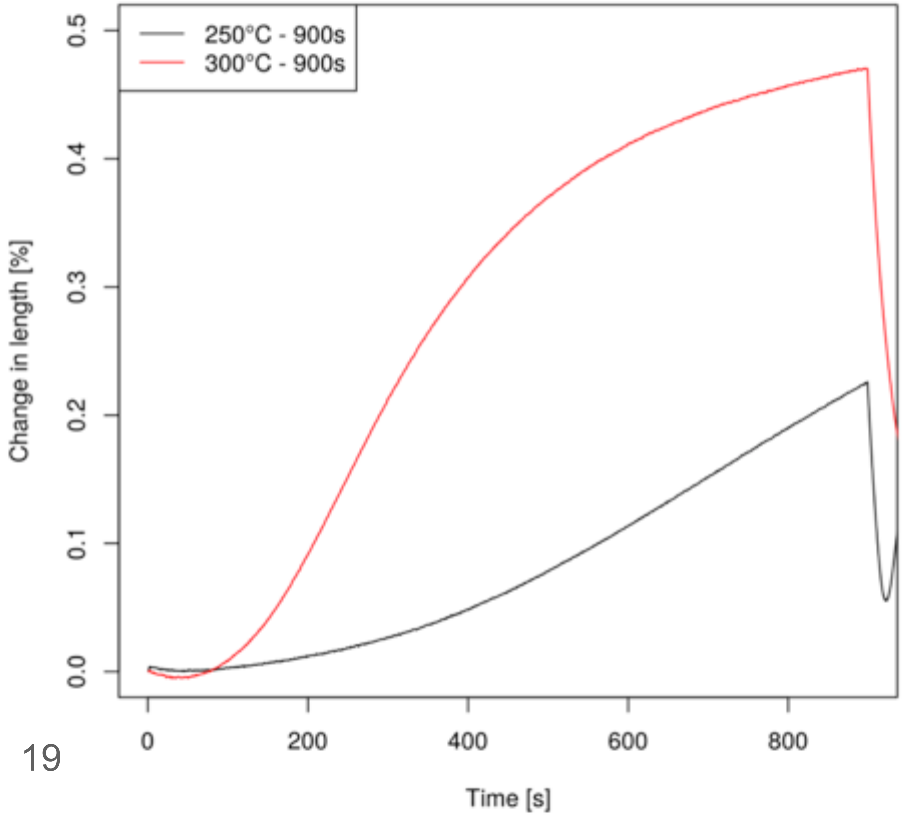
Expansion is also observed accompanying formation of α-iso

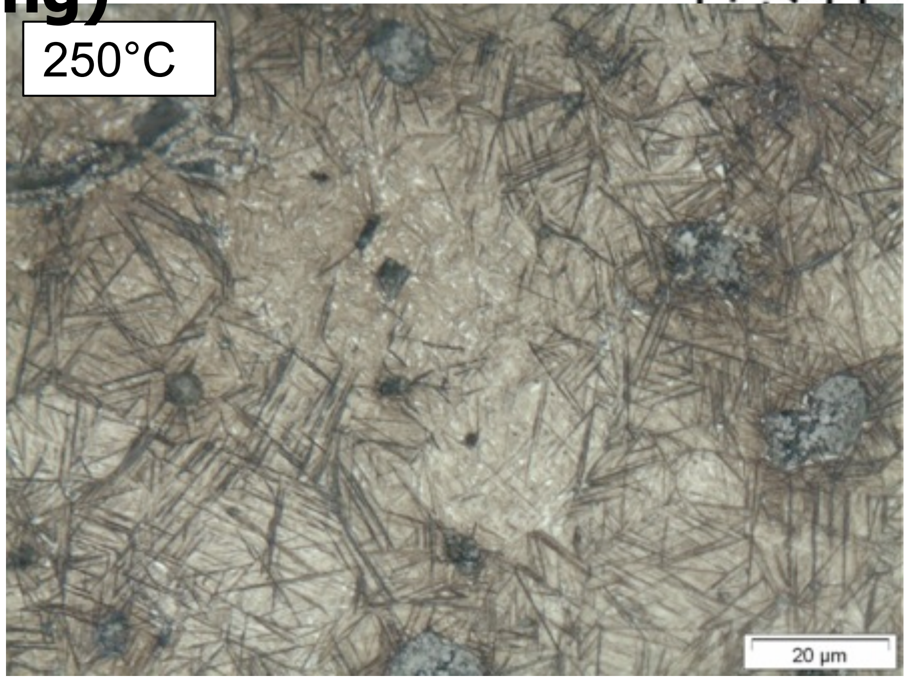


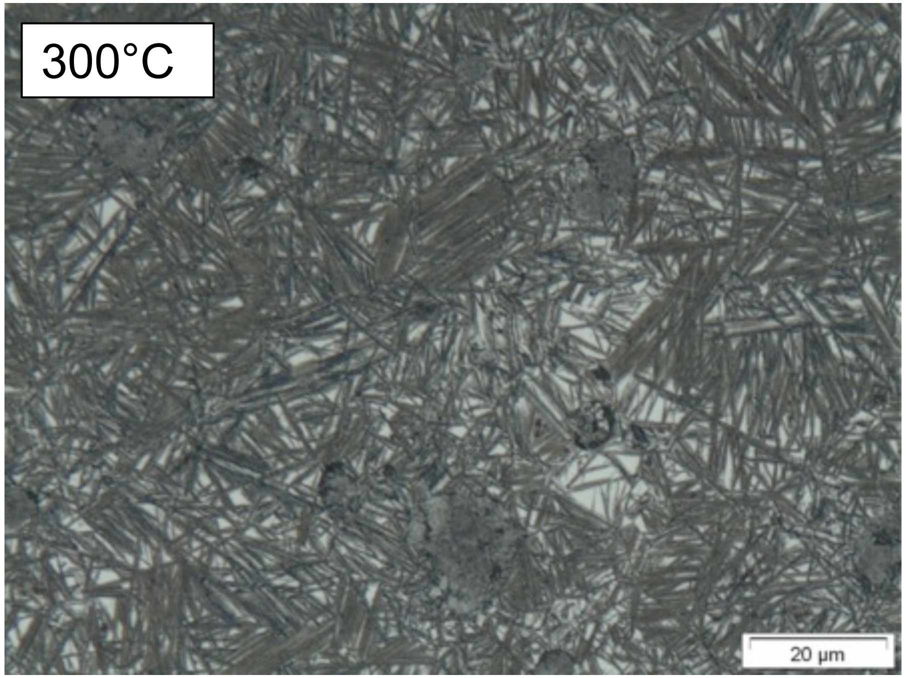


Results: formation of α-iso (conventional austempering)



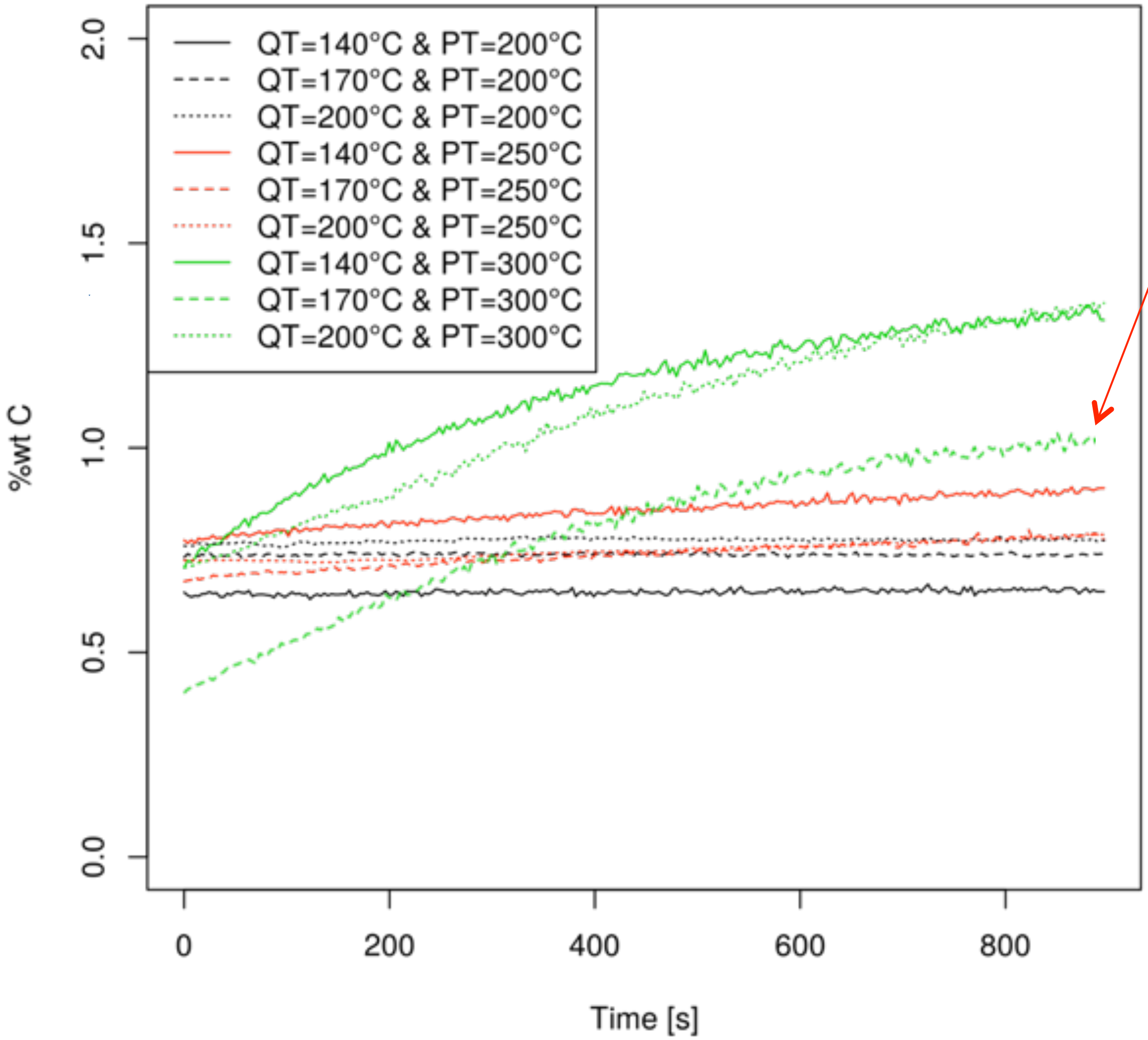






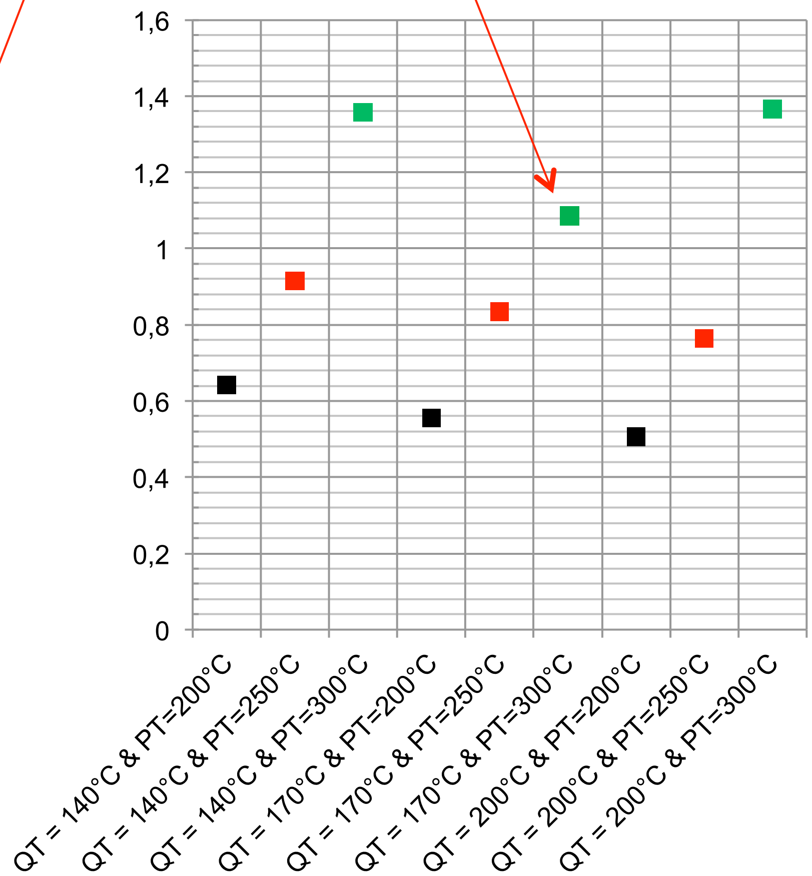
Results: Austenite carbon content

Evolution of austenite carbon content during partitioning step. Calculated using γ[111]

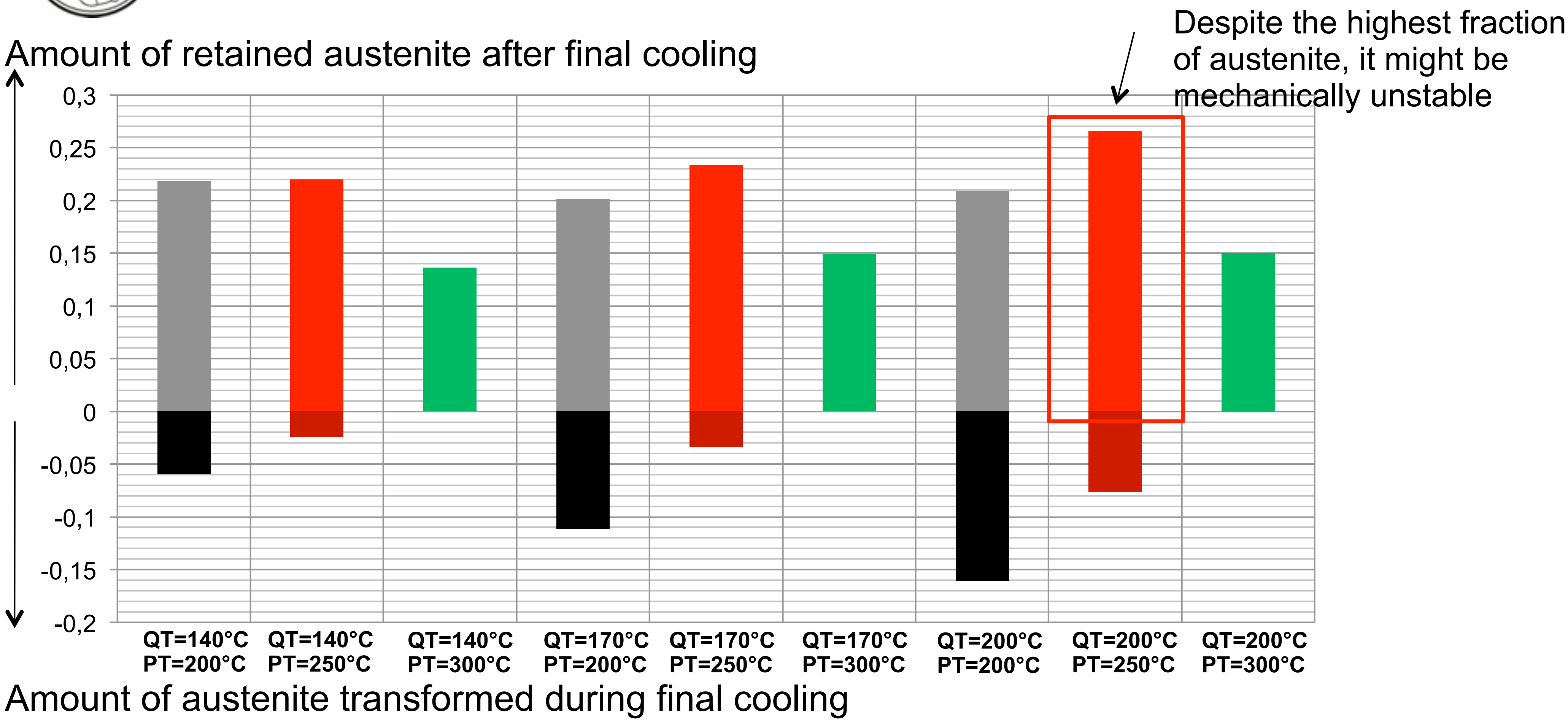


Problem during beam alignment step!

Final austenite carbon content

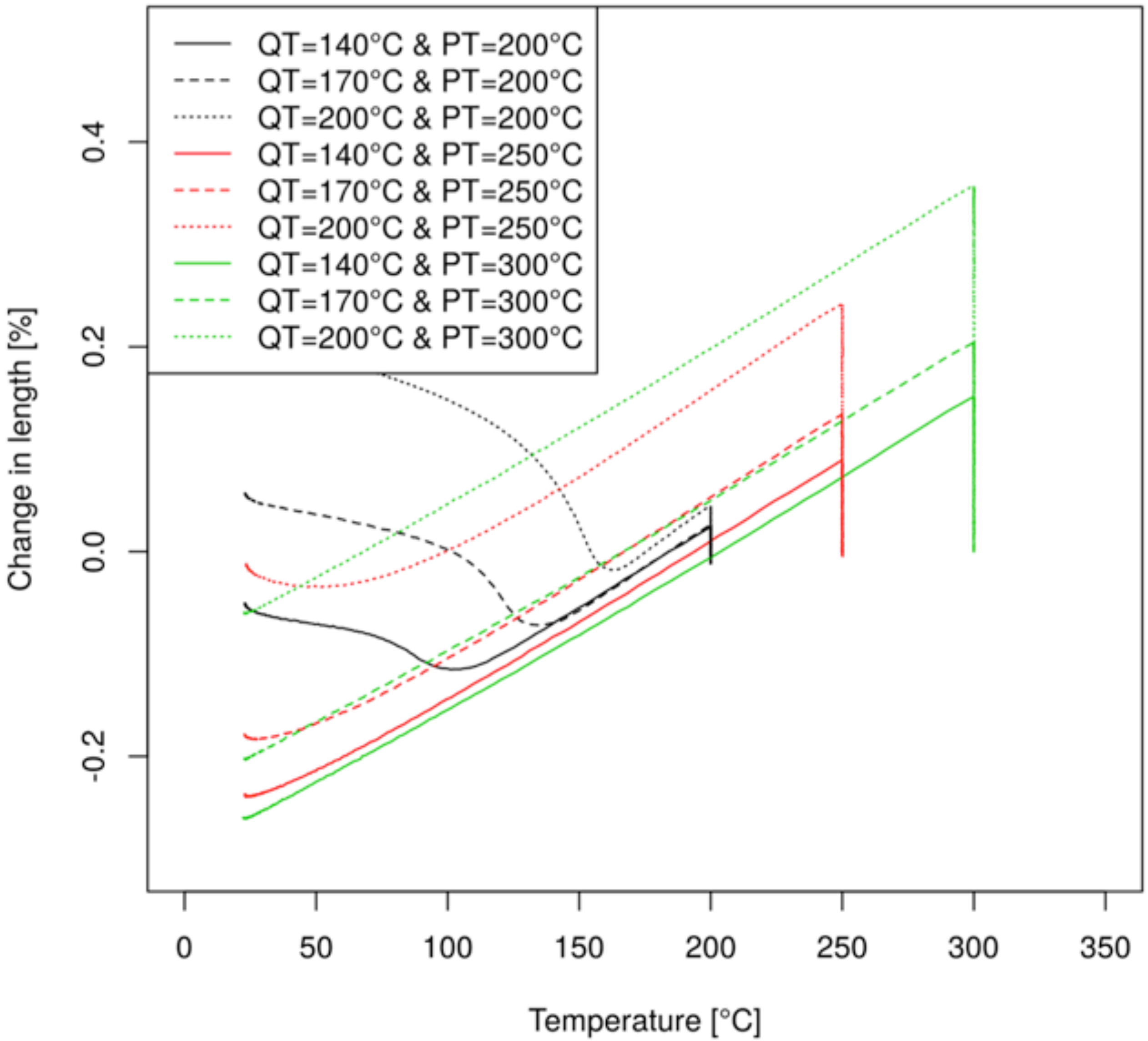


Results: Austenite chemical stability – XRD



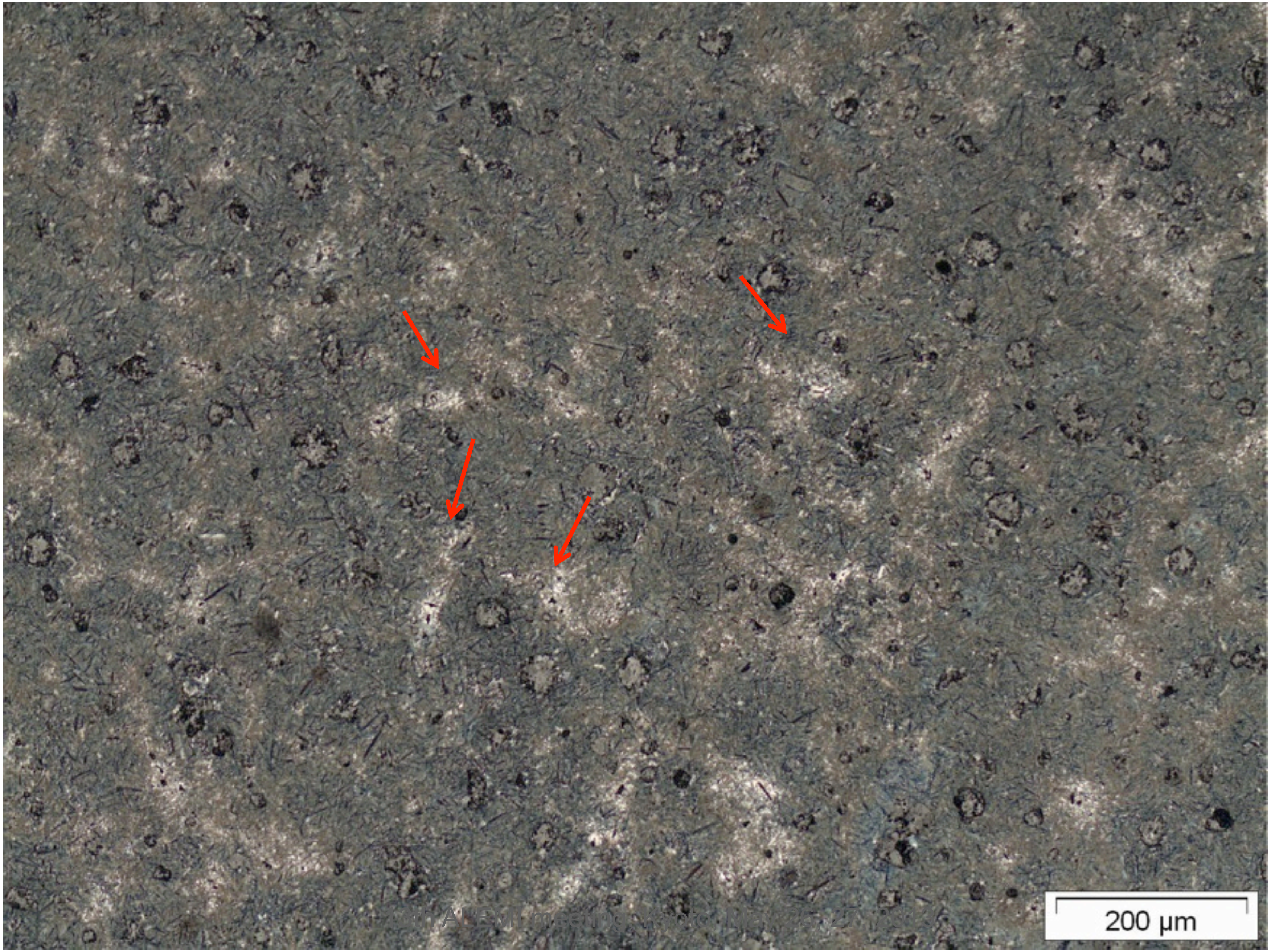
Samples partitioned at 300°C all converge to approximately the same amount of austenite. The amount of isothermal ferrite and athermal martensite formed during the initial quench is different though.

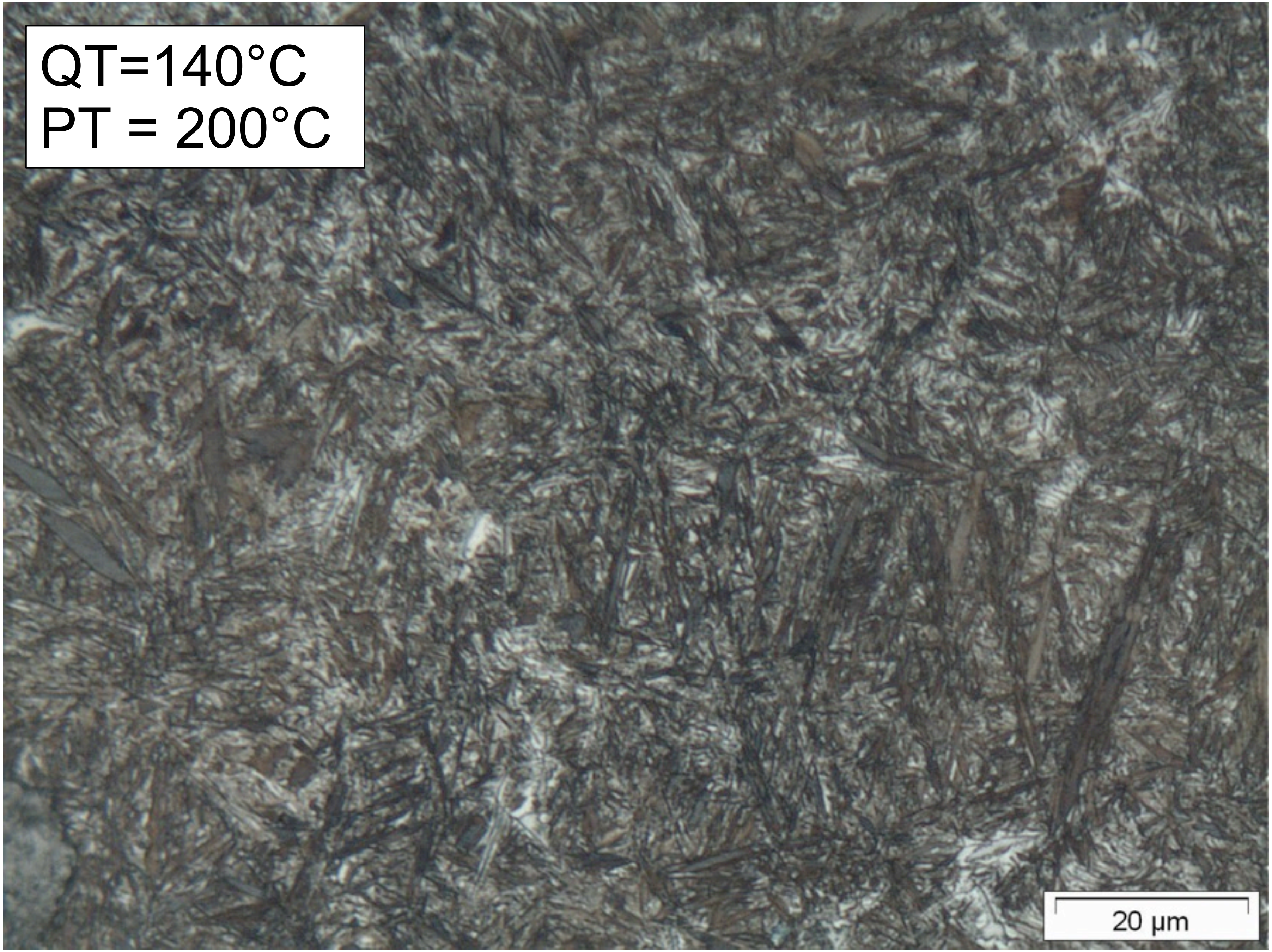
Results: Austenite chemical stability – dilatometry

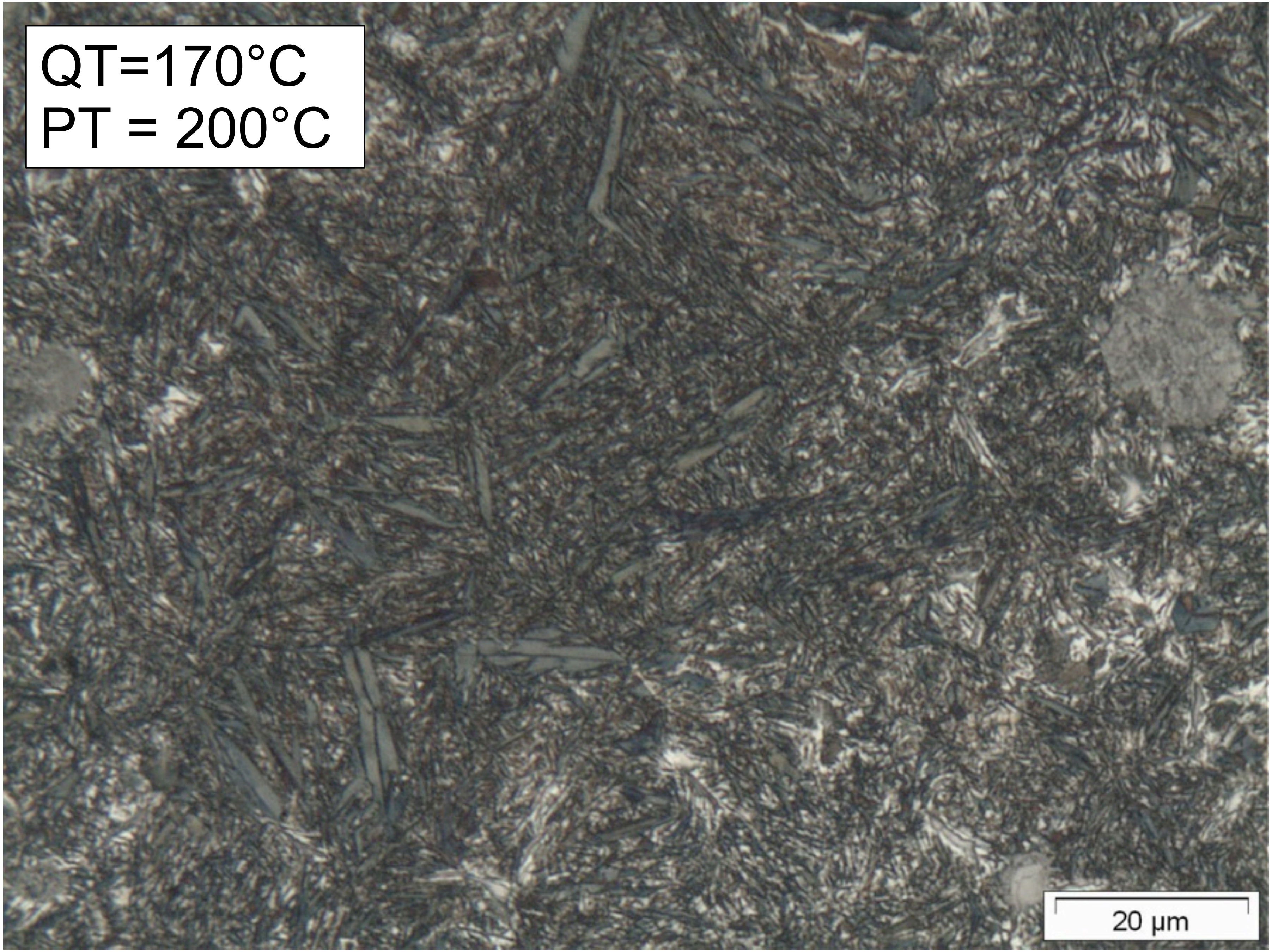


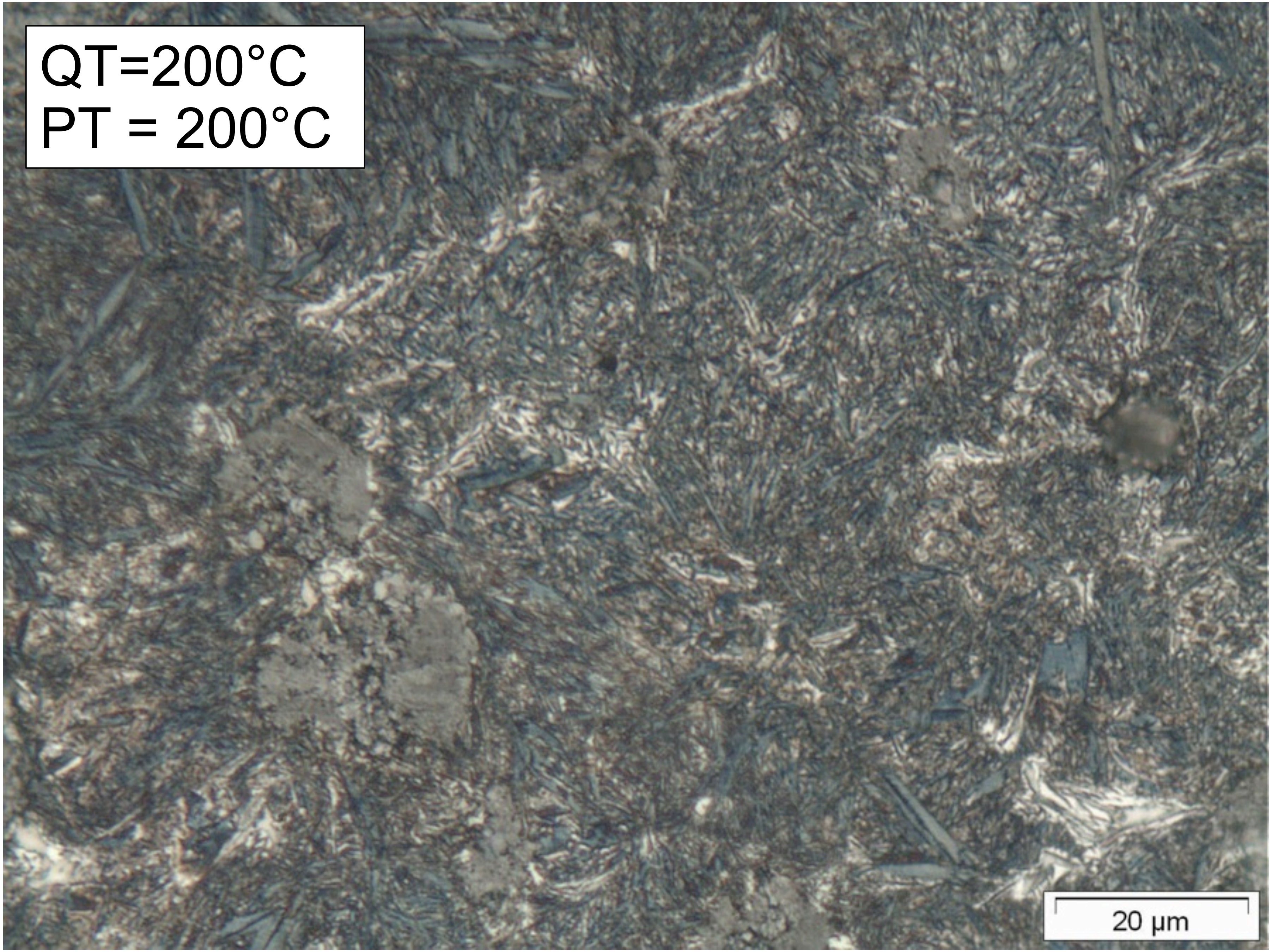
QT [°C]	PT [°C]	Ms [°C]
140	200	119
170	200	148
200	200	168
140	250	35
170	250	77
200	250	109
140	300	below RT
170	300	below RT
200	300	below RT

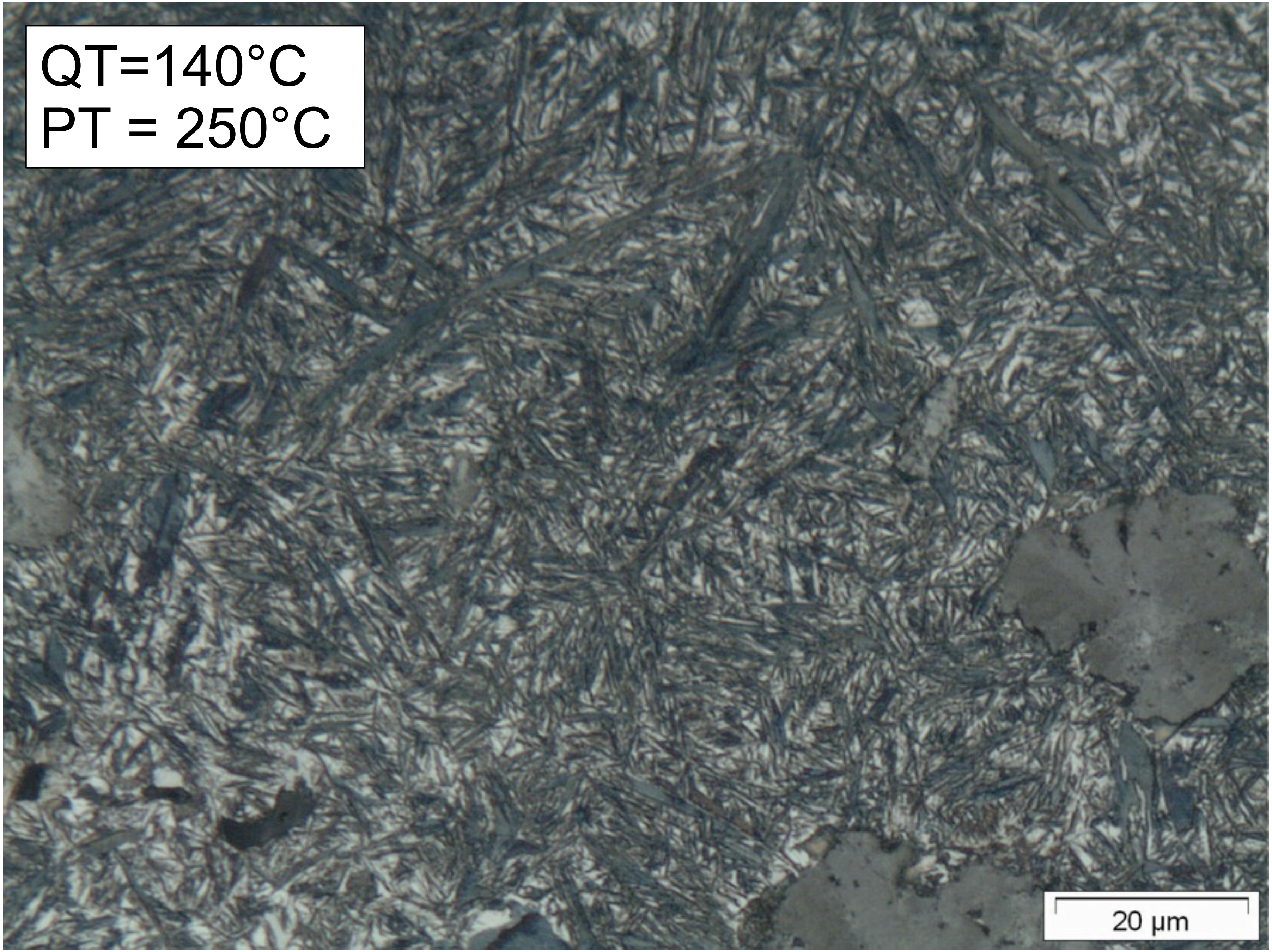
Results: Microstructure

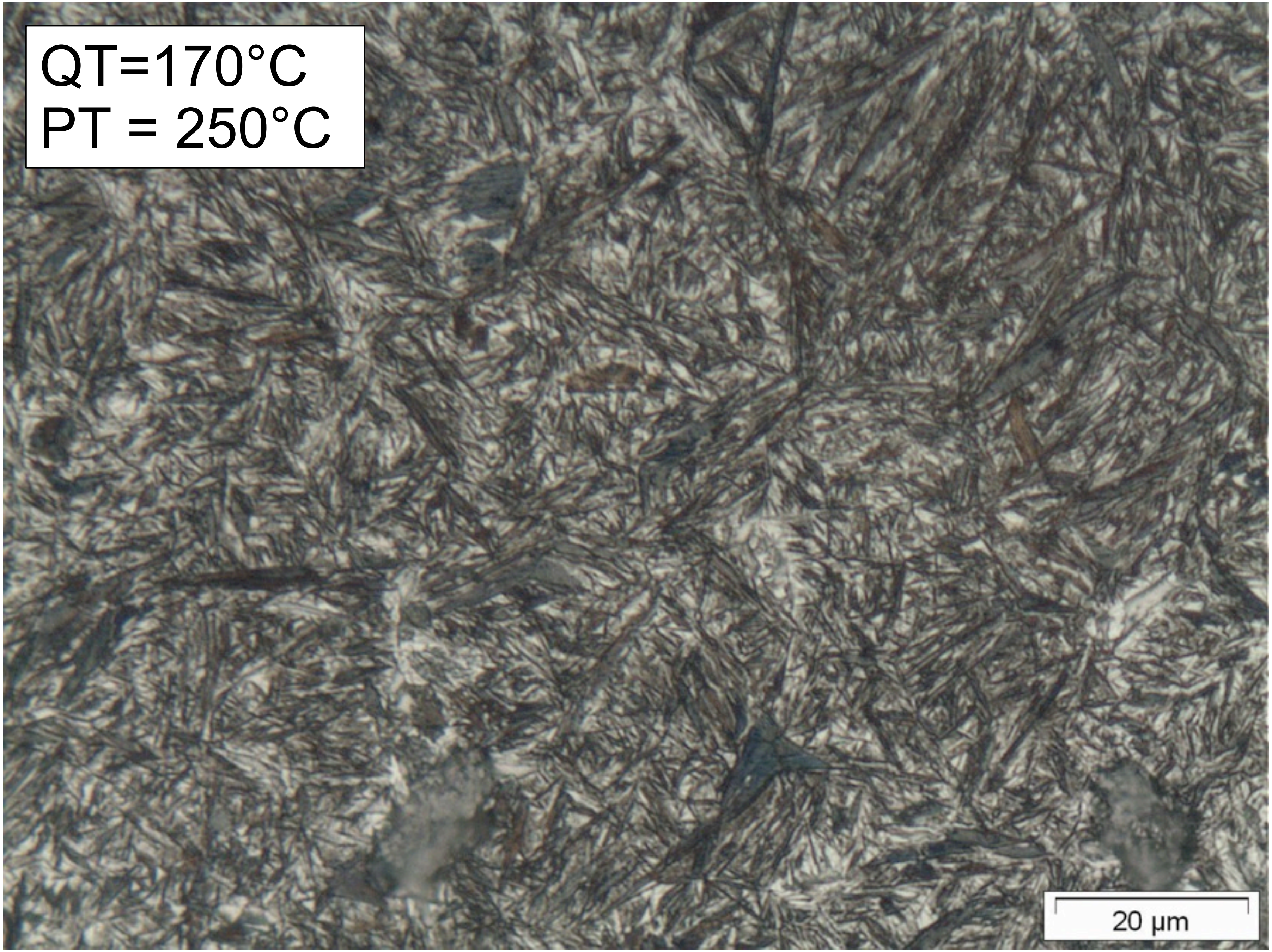


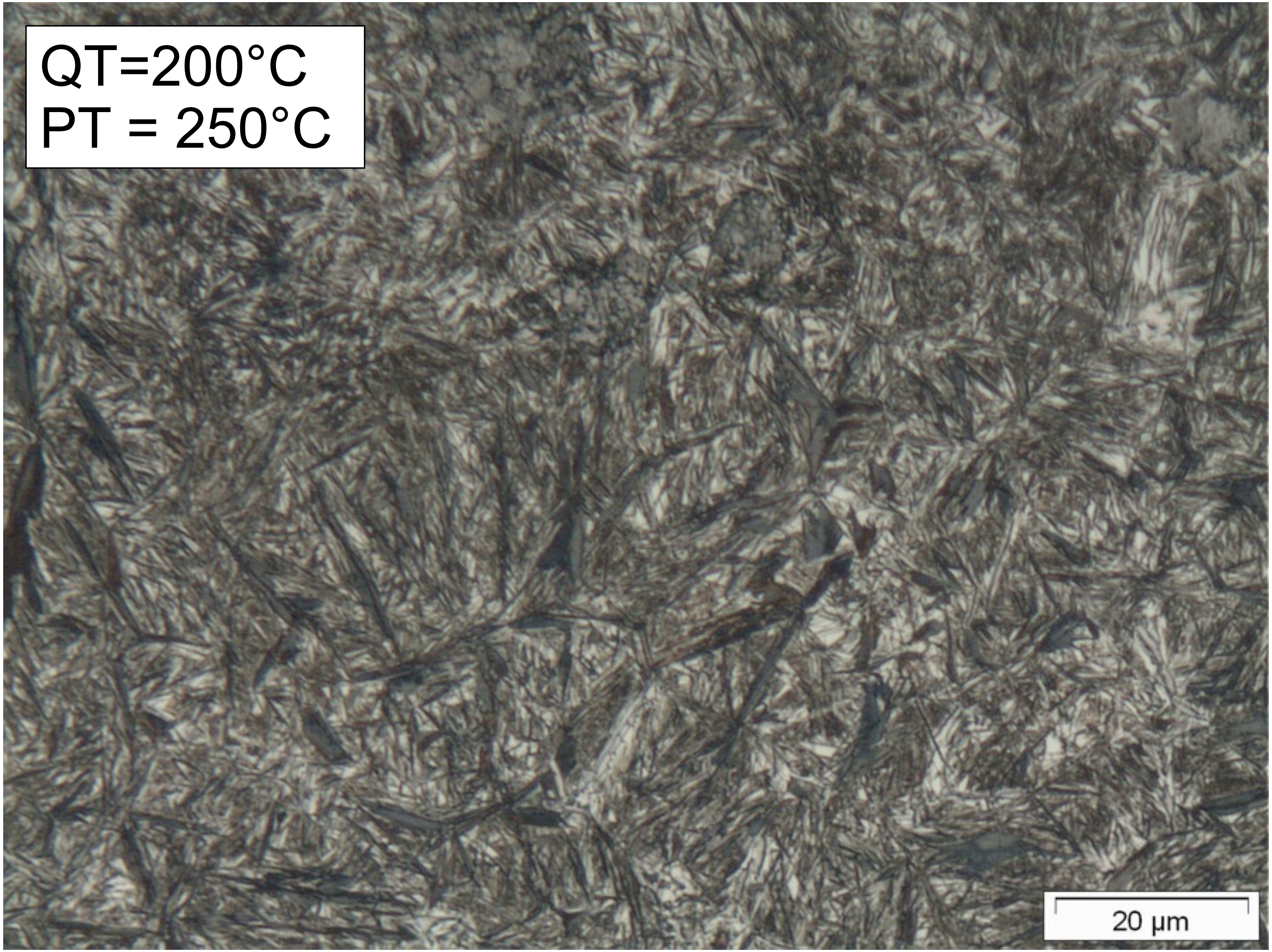

QT=140°C
PT = 200°C
20 µm

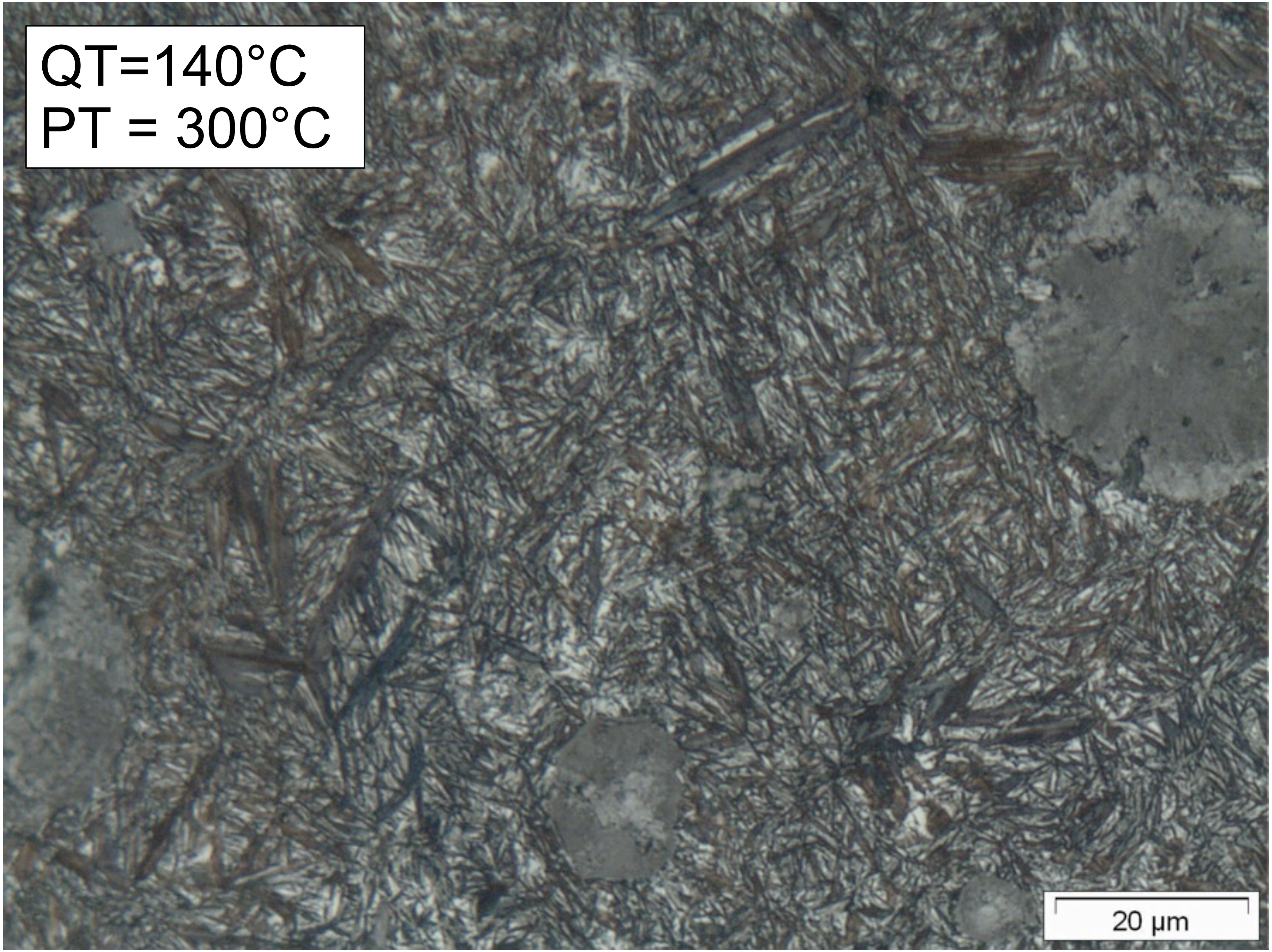

QT=170°C
PT = 200°C
20 µm

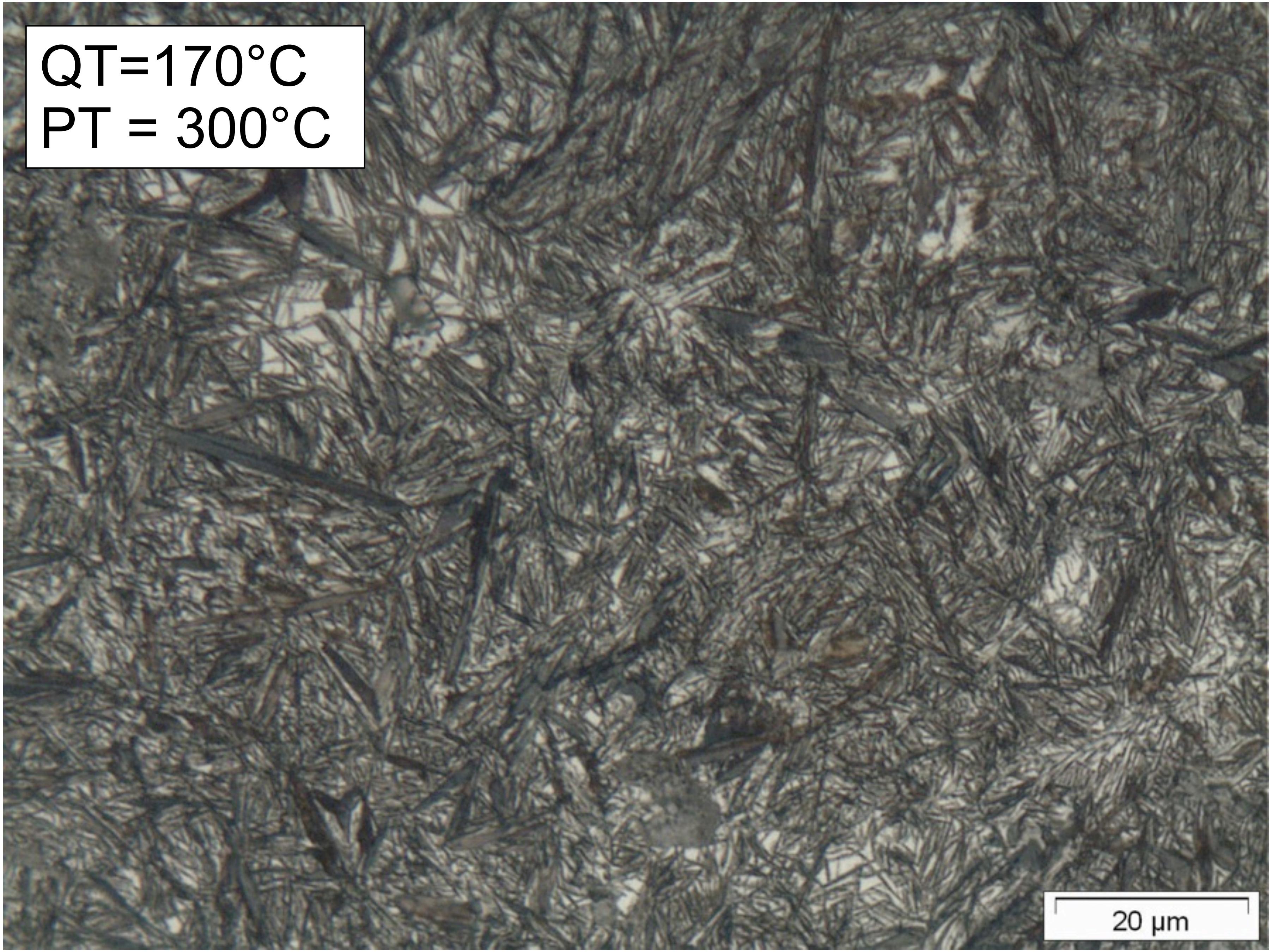

QT=200°C
PT = 200°C
20 µm

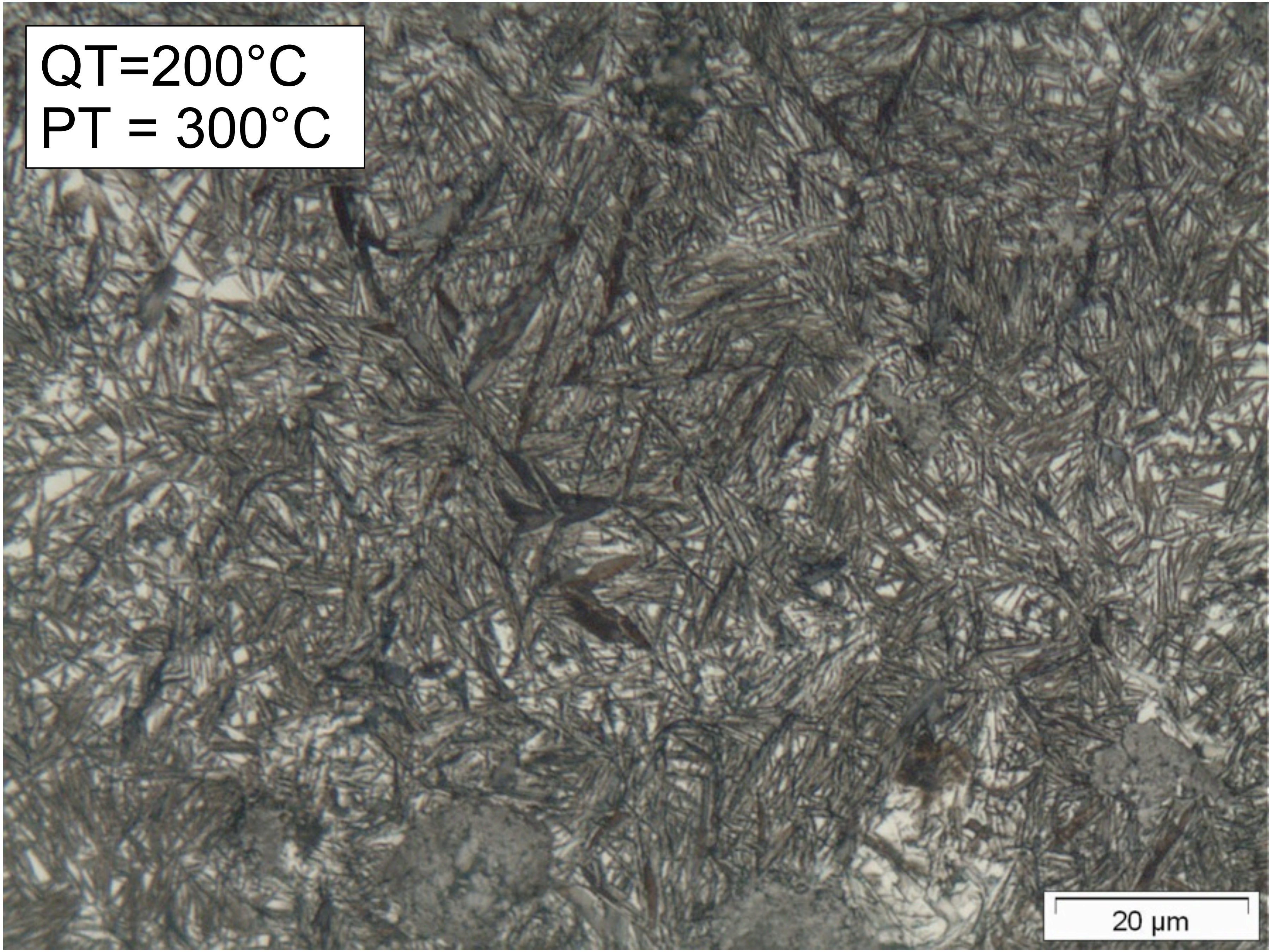

QT=140°C
PT = 250°C
20 µm


QT=170°C
PT = 250°C
20 µm

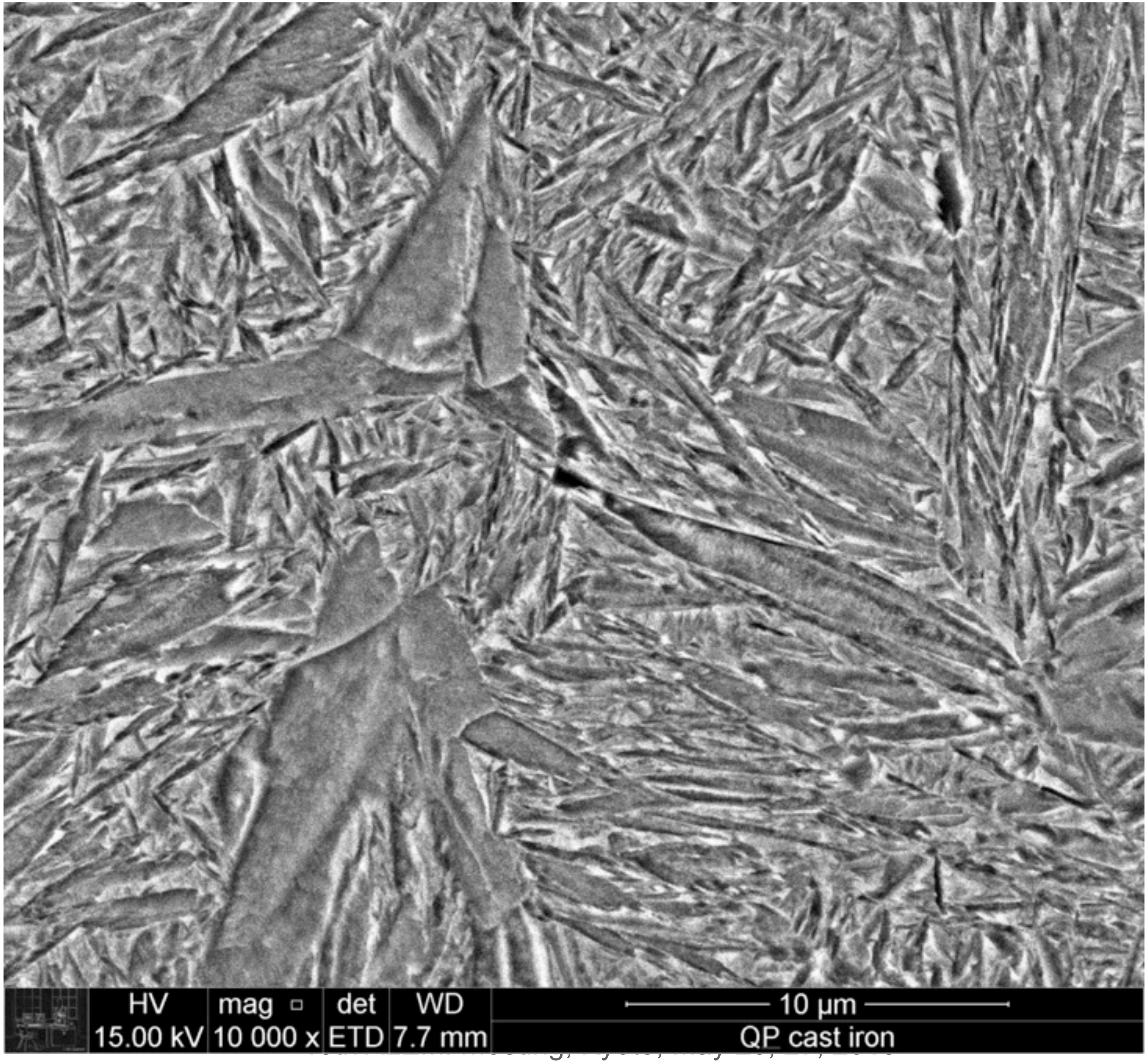

QT=200°C
PT = 250°C
20 µm


QT=140°C
PT = 300°C
20 µm

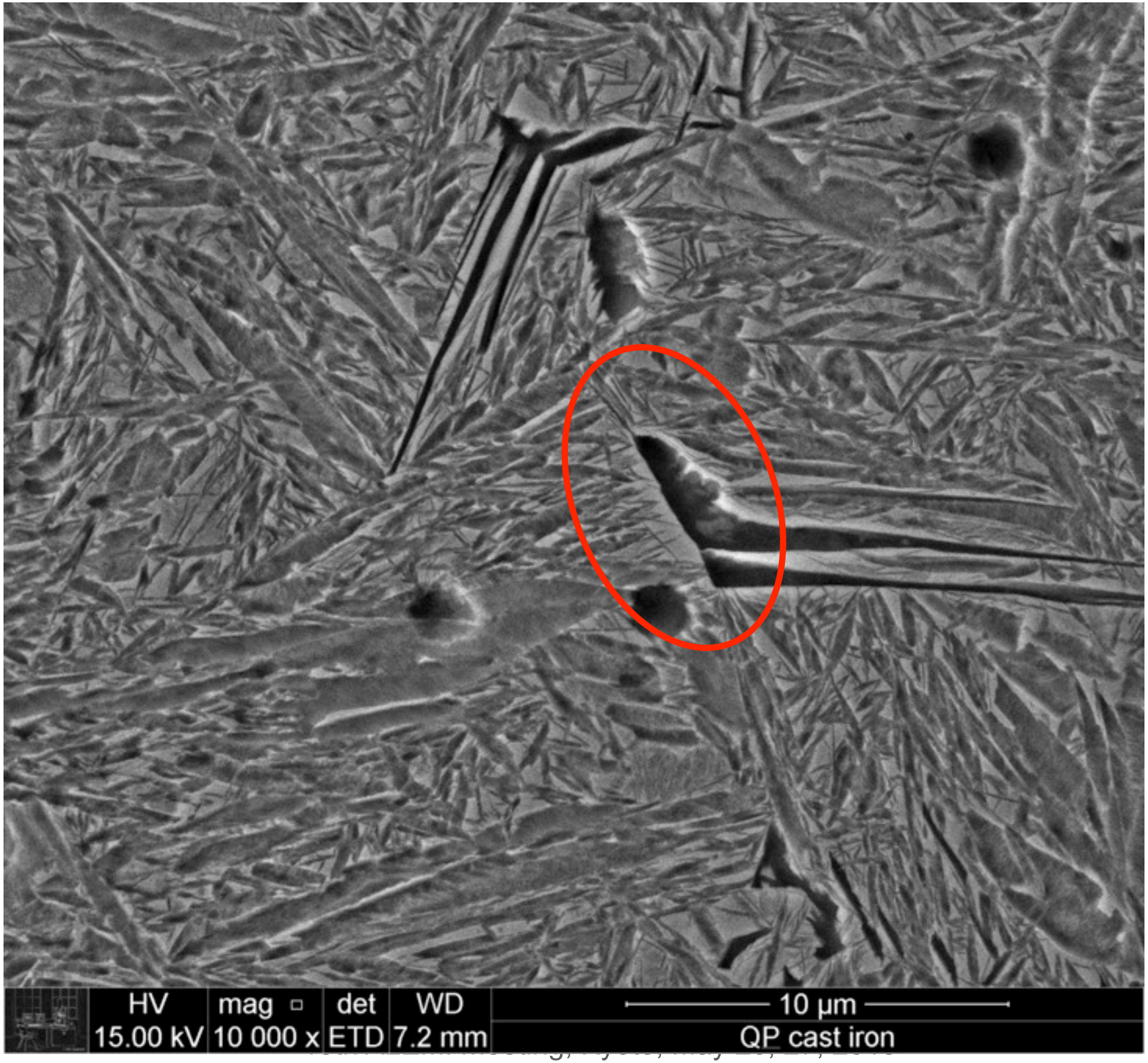

QT=170°C
PT = 300°C
20 µm


QT=200°C
PT = 300°C
20 µm

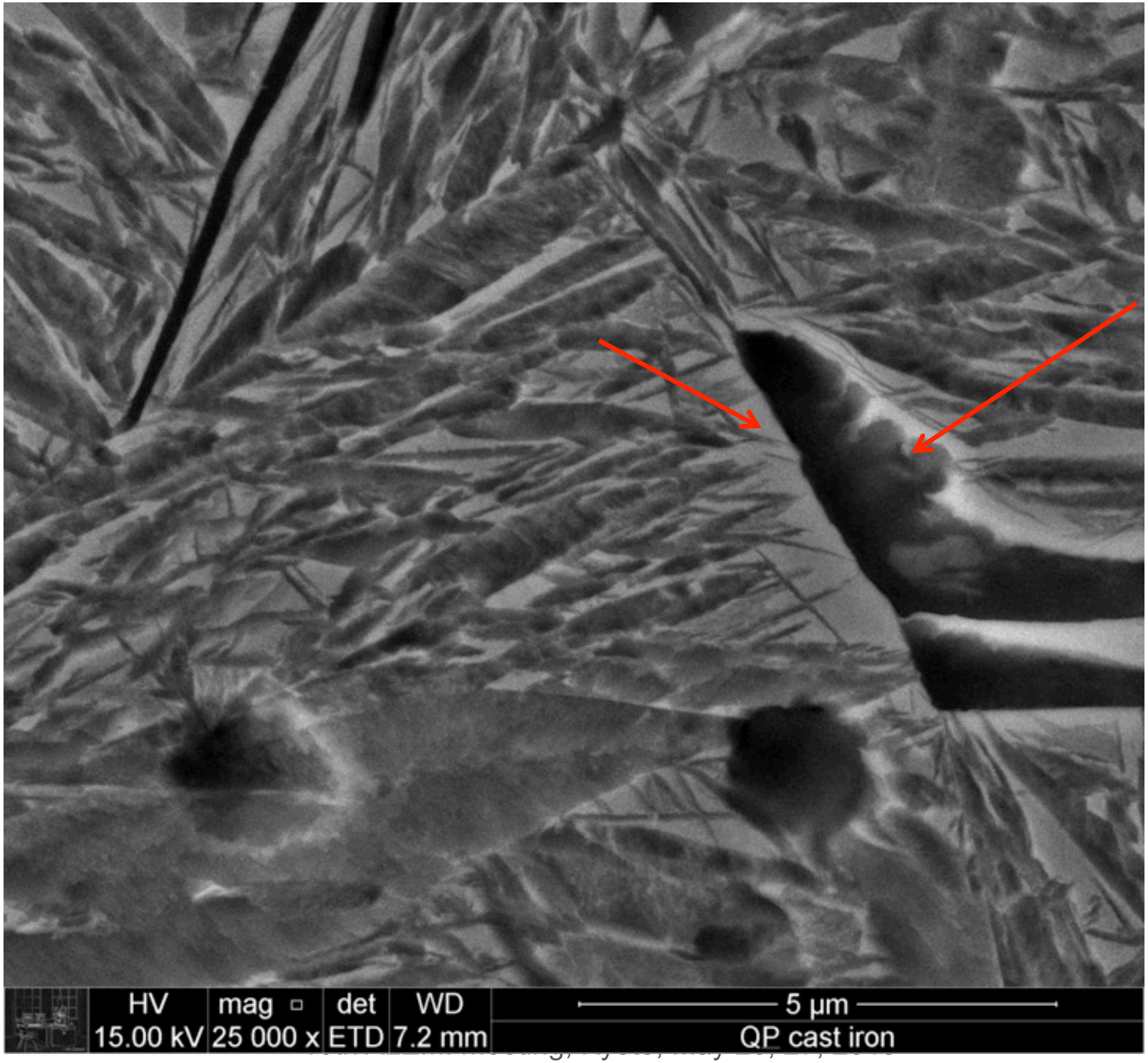
QT=140°C & PT=200°C



QT=140°C & PT=250°C

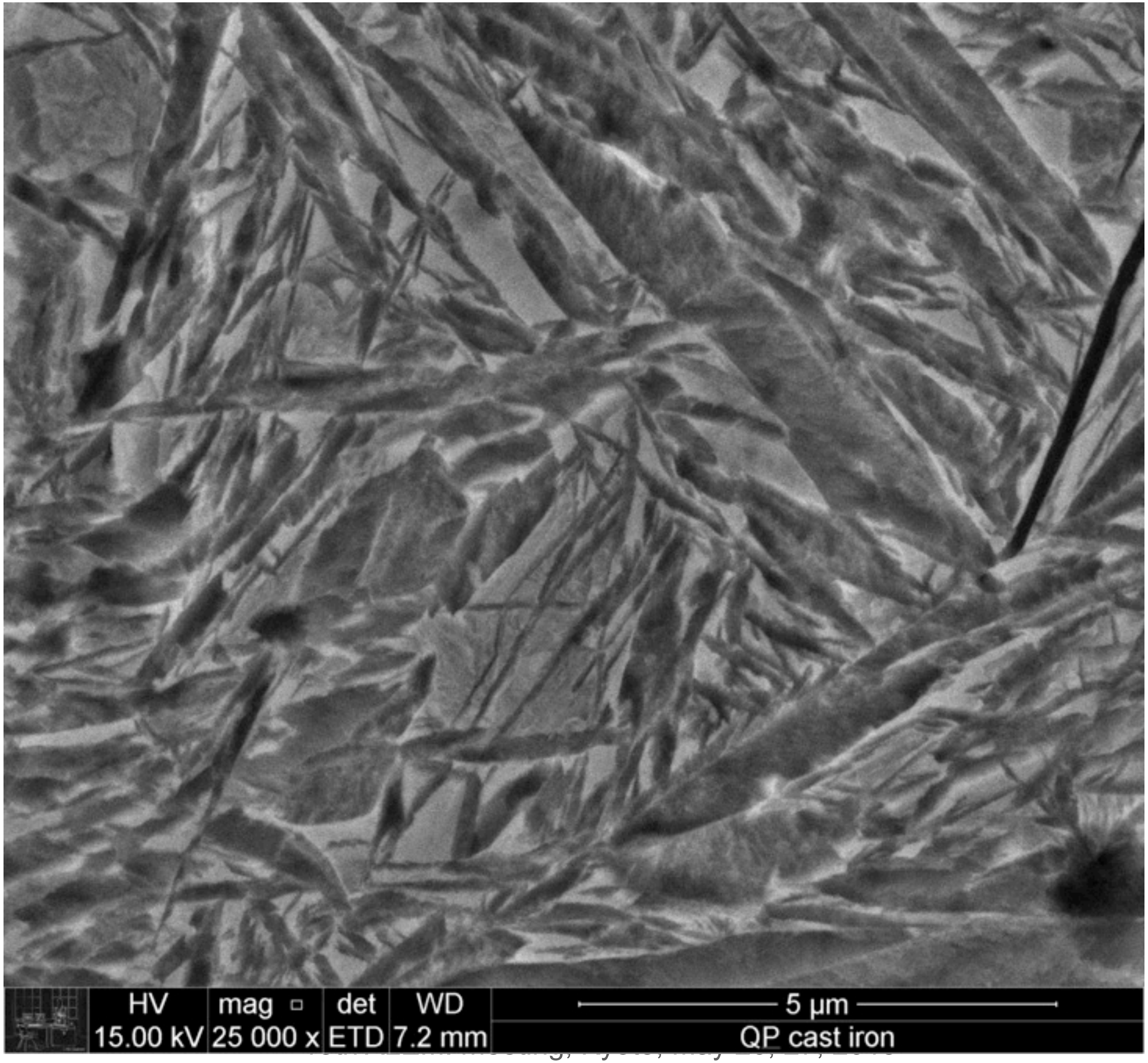


QT=140°C & PT=250°C

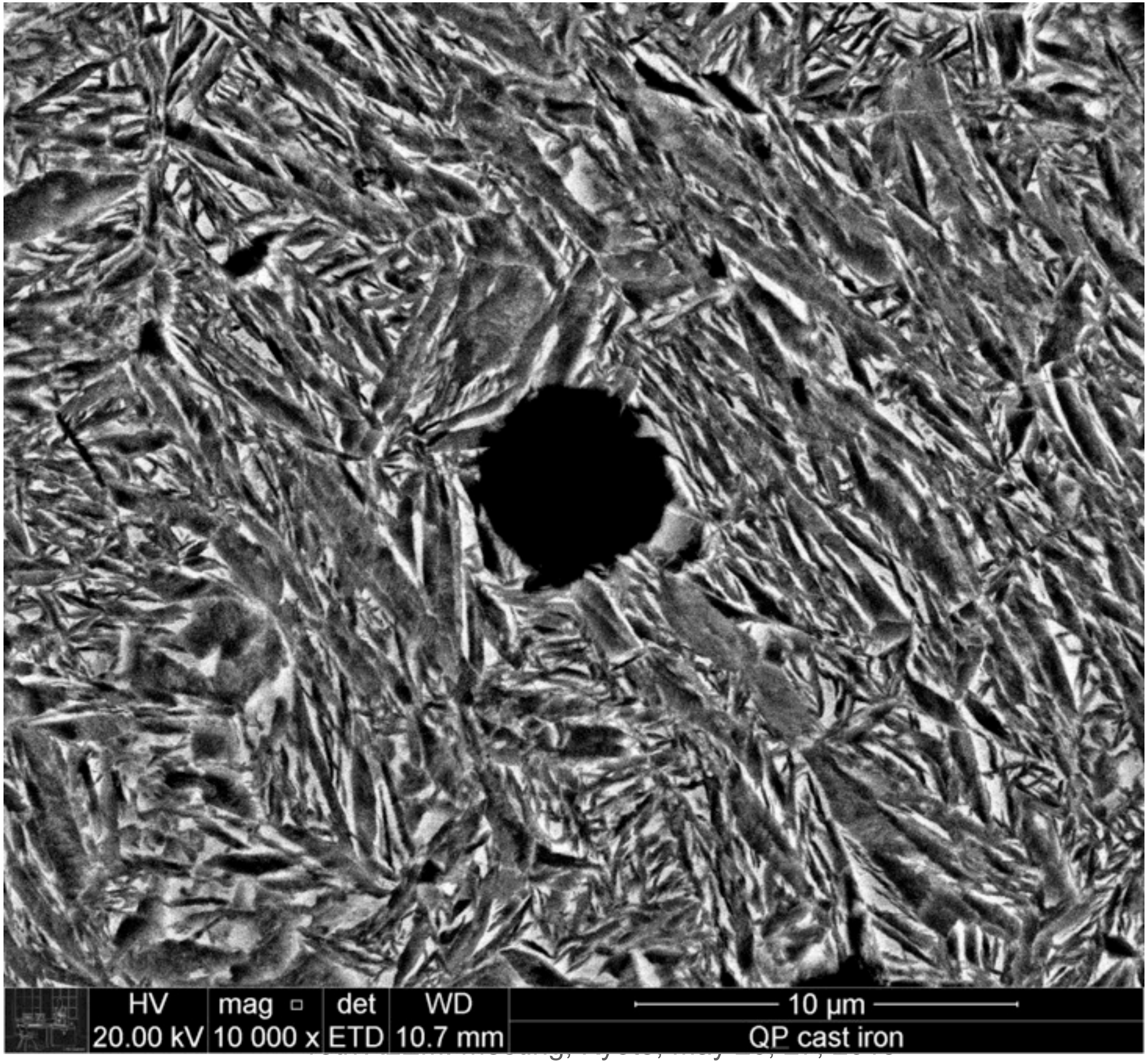


Ferrite formed
during quenching

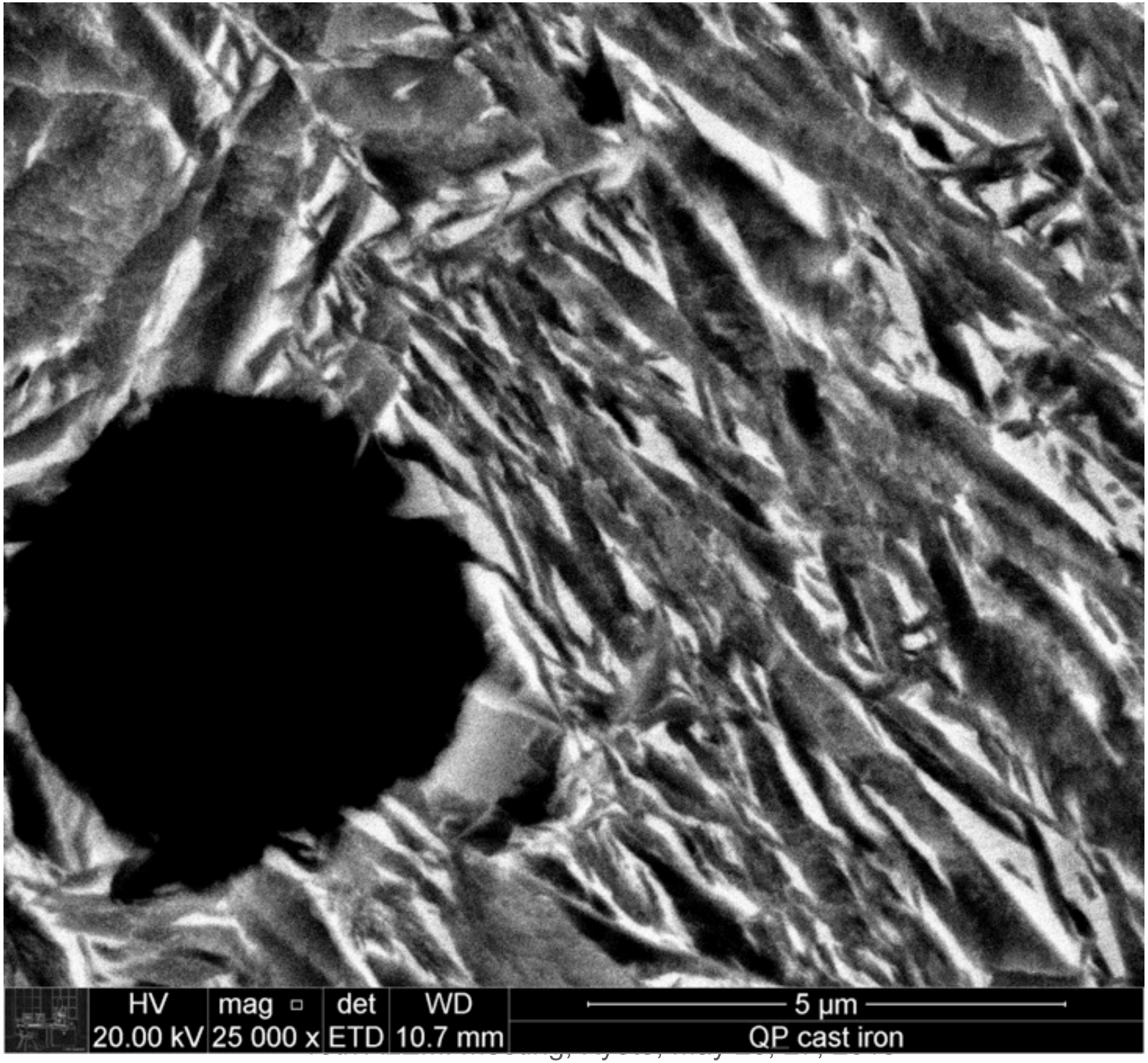
QT=140°C & PT=250°C

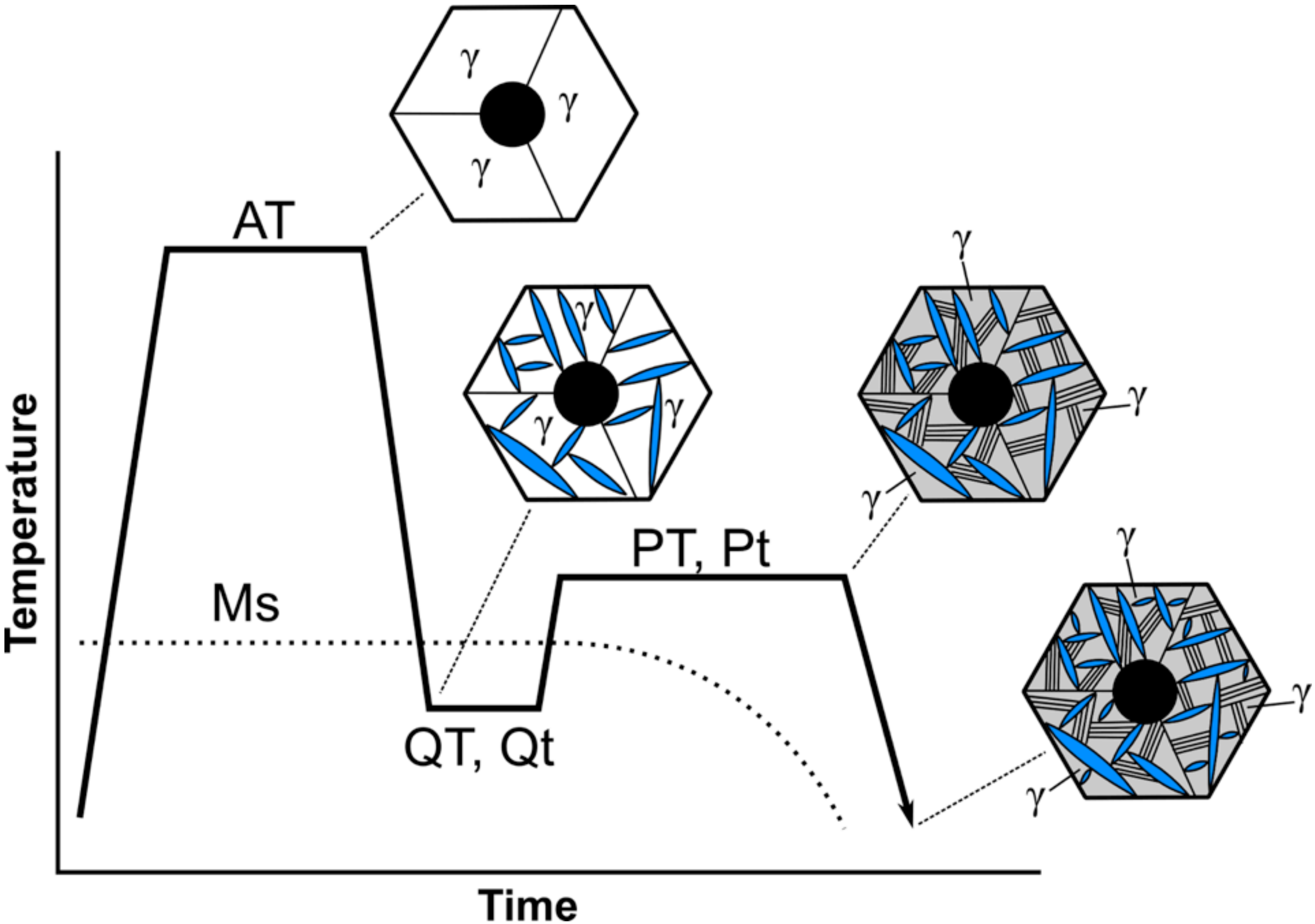


QT=140°C & PT=300°C



QT=140°C & PT=300°C




γ
γ
γ
AT
γ
γ
γ
γ
PT, Pt
Ms
QT, Qt
Temperature
Time

Concluding remarks

- The in situ DRX of the samples during Q&P, using synchrotron radiation was able to follow in real time the C enrichment of austenite.
- Surprisingly a large amount of α (or α’) bcc Fe precipitation was also detected during partition at 250 and 300ºC. The large increase in γ C-content is actually due to this α precipitation.
- Samples partitioned at 300°C all converge to approximately the same amount of austenite, even though the amount of isothermal ferrite and athermal martensite formed during the initial quench is different for different quenching temperatures.
- The morphology of the α formed is very similar to the martensite plates, unlike bainite usually found in ADI. More careful characterization work is needed to determine if we have isothermal martensite, thin plate martensite or just conventional carbide free bainite.