ancillary meeting times,
and much more.

M&M 2015
MICROSCOPY & MICROANALYSIS

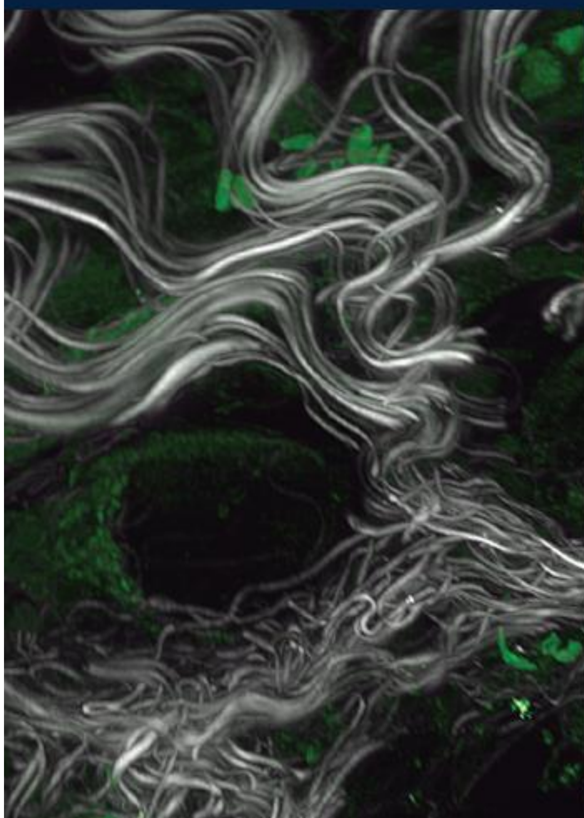
August 2-6 • Portland, OR

MSA Microscopy Society of America

MAS Microanalysis Society

IMS International Metallographic Society ASM INTERNATIONAL

www.microscopy.org/MandM/2015 for up-to-date meeting information



Microstructural Developments Leading to New Advanced High Strength Sheet Steels: *A Historical Assessment of Critical Metallographic Observations*

David K. Matlock, Larrin S. Thomas, Mark D. Taylor, Emmanuel De Moor, and John G. Speer
Advanced Steel Processing and Products Research Center*
Colorado School of Mines
Golden, Colorado

2015 IMS Henry Clifton Sorby Award
August 3, 2015

***An NSF Industry/University Cooperative Research Center - Est. 1984**

…Perspective…

"…More than half of the steels present in today's vehicles didn't exist 10 years ago and with breakthrough technologies and industry innovations, just imagine what steel will be able to accomplish tomorrow…" http://driveusingsteel.com/ (accessed July 2015)

…development of new steels facilitated by advanced microstructural analyses…!!

Henry Clifton Sorby (1826 – 1908)

Portrait in Mappin Hall
University of Sheffield

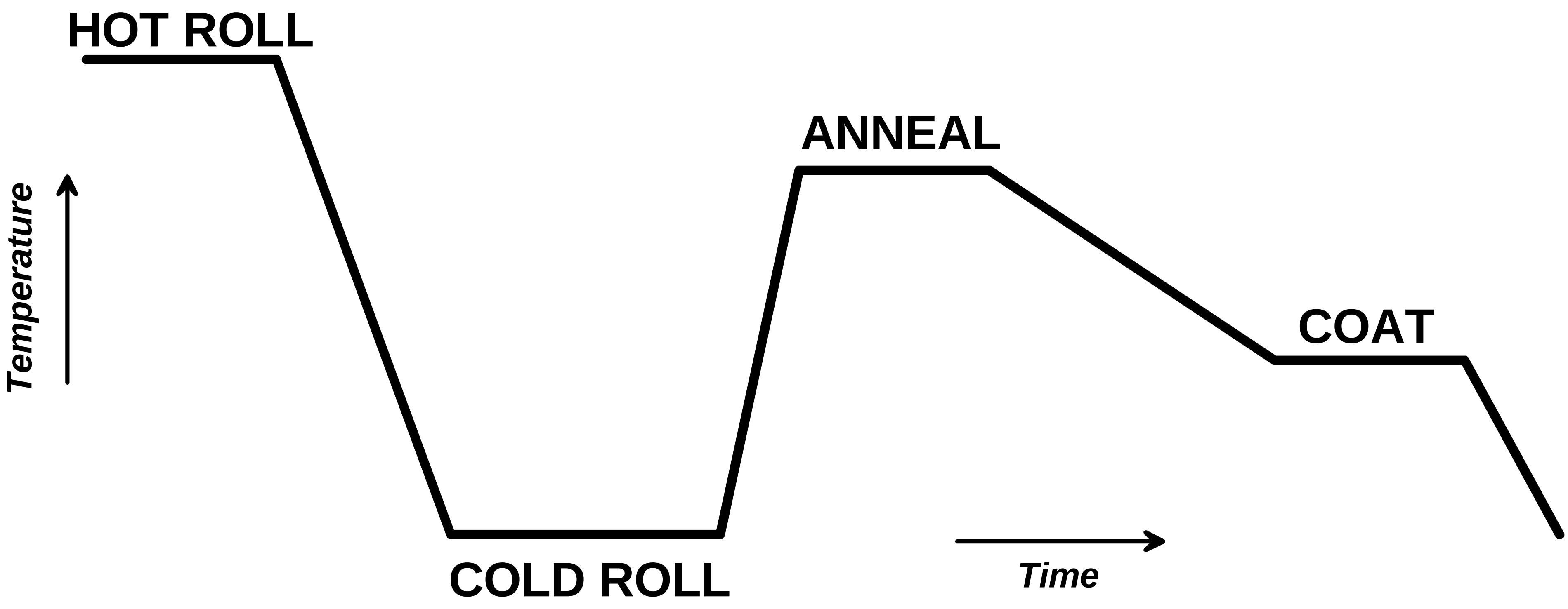
Pioneer of Ferrous Metallurgy Metallography

"In those early days, if railway accident had occurred and I had suggested that the company should take up a rail and have it examined with the microscope, I should have been looked upon as a fit man to send to an asylum. But that is what is now being done..."

http://www.sorby.org.uk/hcsorby.shtml

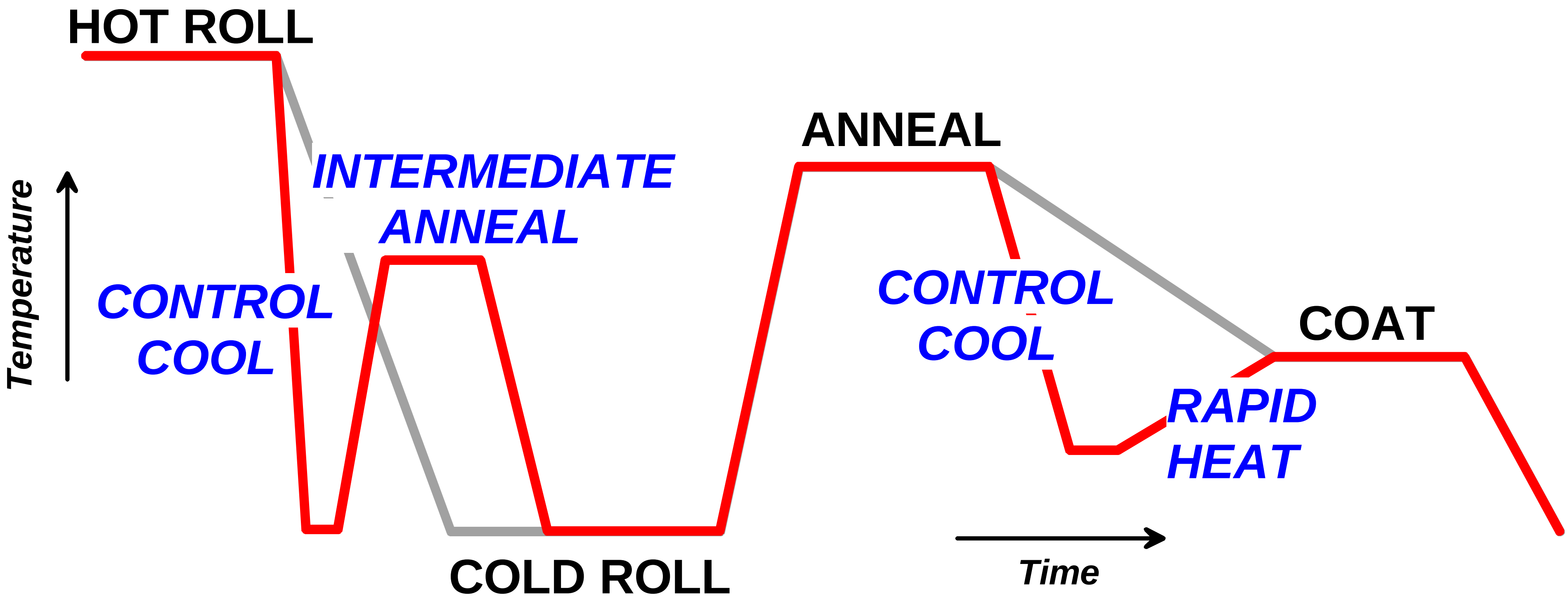
Theme: Process Control

Historical



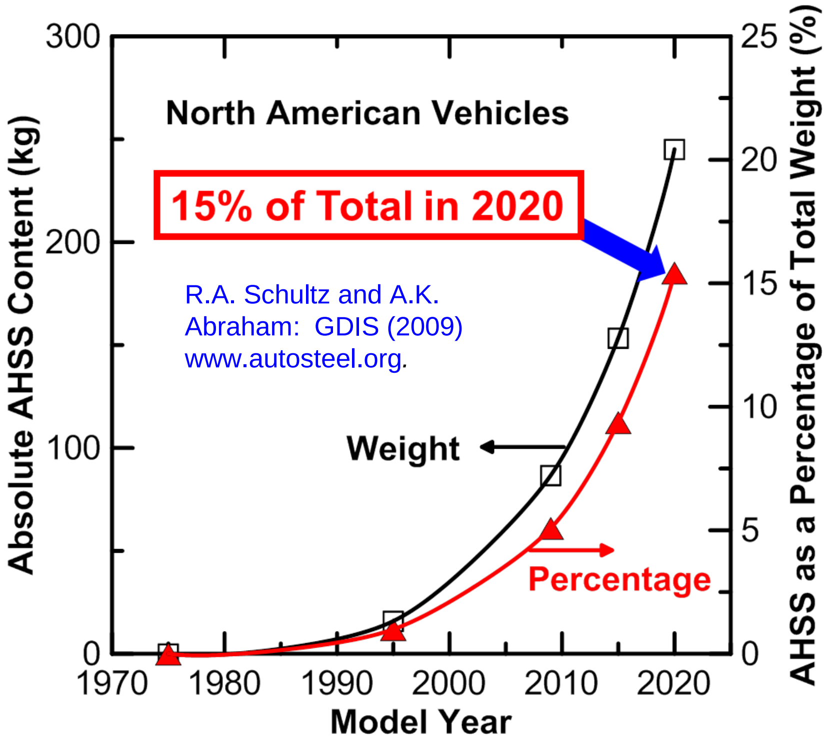
Theme: Process Control

Process Control and Alloying
= Advanced High Strength Sheet Steels (AHSS)



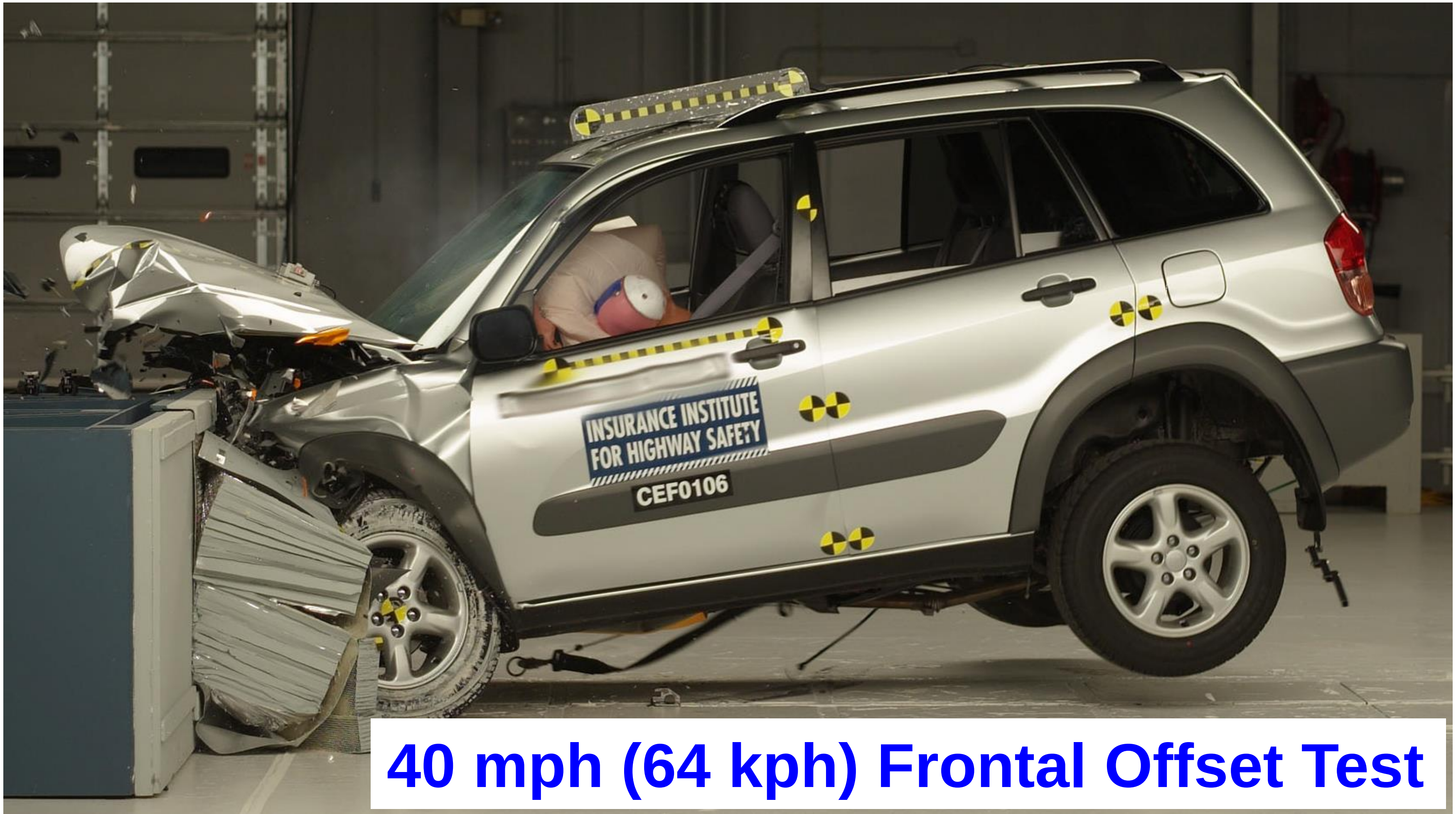
Why do we need AHSS?

High strength steels to enable safe, fuel-efficient lightweight designs



US Requirements for Automotive Steels

- **Design vehicle to produce "5-star" crash worthiness rating**
  - ***High toughness steels to absorb energy***



40 mph (64 kph) Frontal Offset Test

US Requirements for Automotive Steels

- **Roof crush resistance: increase to 4.0 times vehicle weight - (by 2017)**
  - ***High strength steels to maintain safety cage***

auto.howstuffworks.com (accessed Nov2013)

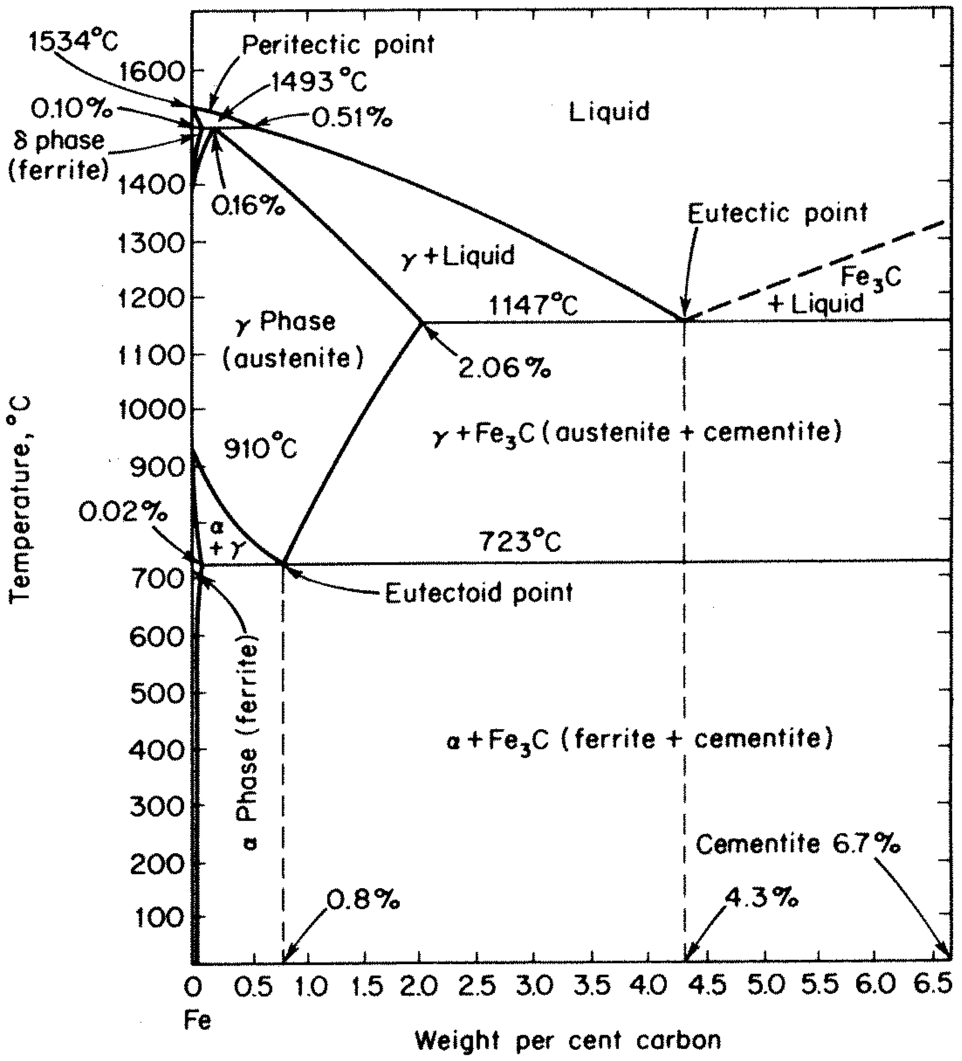
***wheels.blogs.nytimes.com (accessed Nov2013)**

US Requirements for Automotive Steels

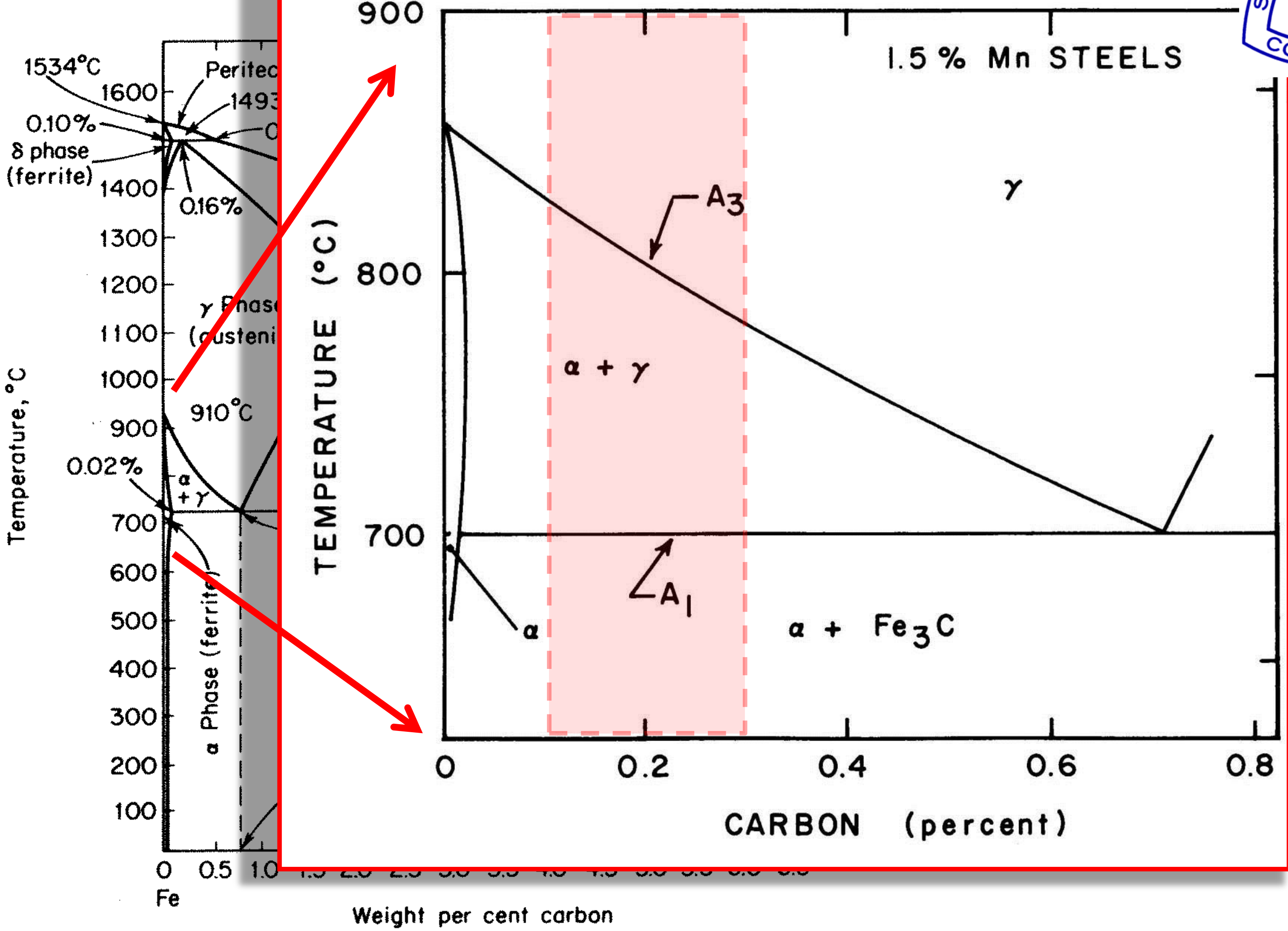
- CAFE (Corporate Average Fuel Economy)
  - Increase to 54.5 mpg (23.2 km/liter) by 2025
- Enhance competition to meet CAFE
  - Alternate vehicle technologies
  - Alternate materials
    - ***<u>High-strength steels for light-weight designs</u>***

What are Advanced High Strength Sheet Steels (AHSS)?

Background: Fe-C Phase Diagram

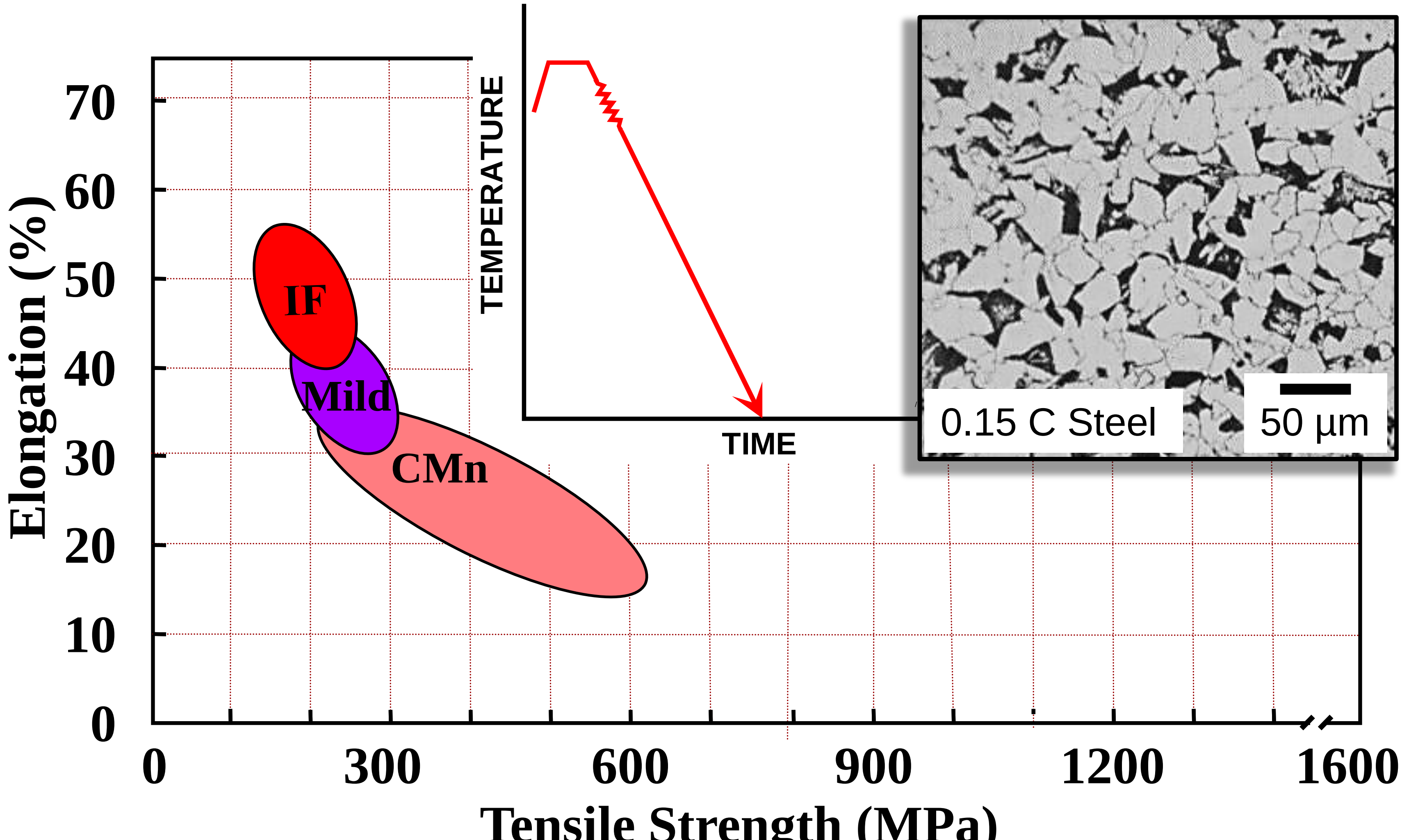


Background: Fe-C Phase Diagram



Background: AHSS Developments

- **Earlier times: Hot rolled low carbon steels**
  - **Ferrite – Pearlite**

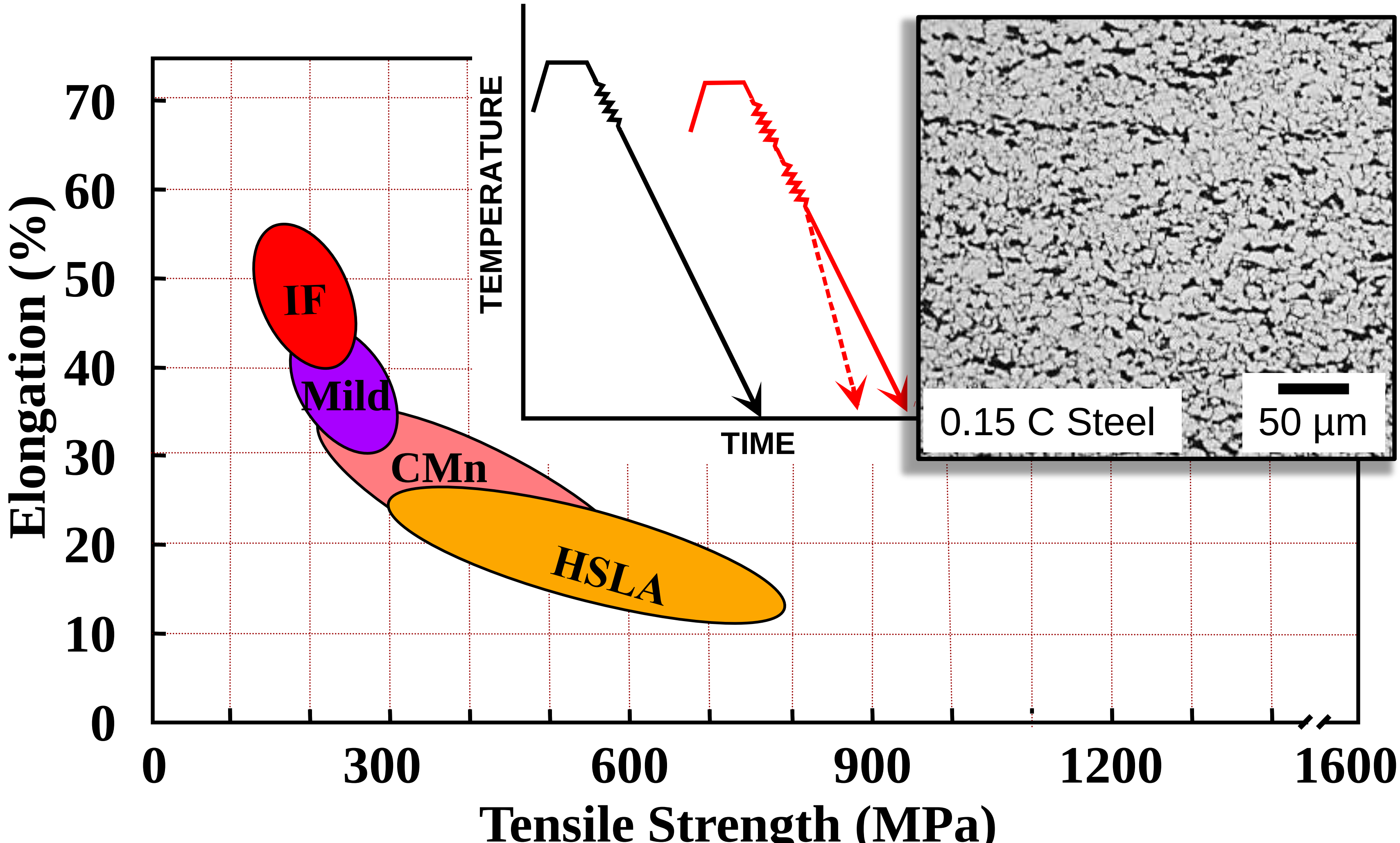


Plot adapted from - AISI: www.steel.org (2006)

Micrograph from: http://www.fgg.uni-lj.si/~/pmoze/ESDEP/master/wg02/l0100.htm (accessed July 2015)

Background: AHSS Developments

- 1960's – 1970's: <u>H</u>igh <u>S</u>trength <u>L</u>ow <u>A</u>lloy (HSLA) steels
  - Thermomechancial processing + microalloy additions

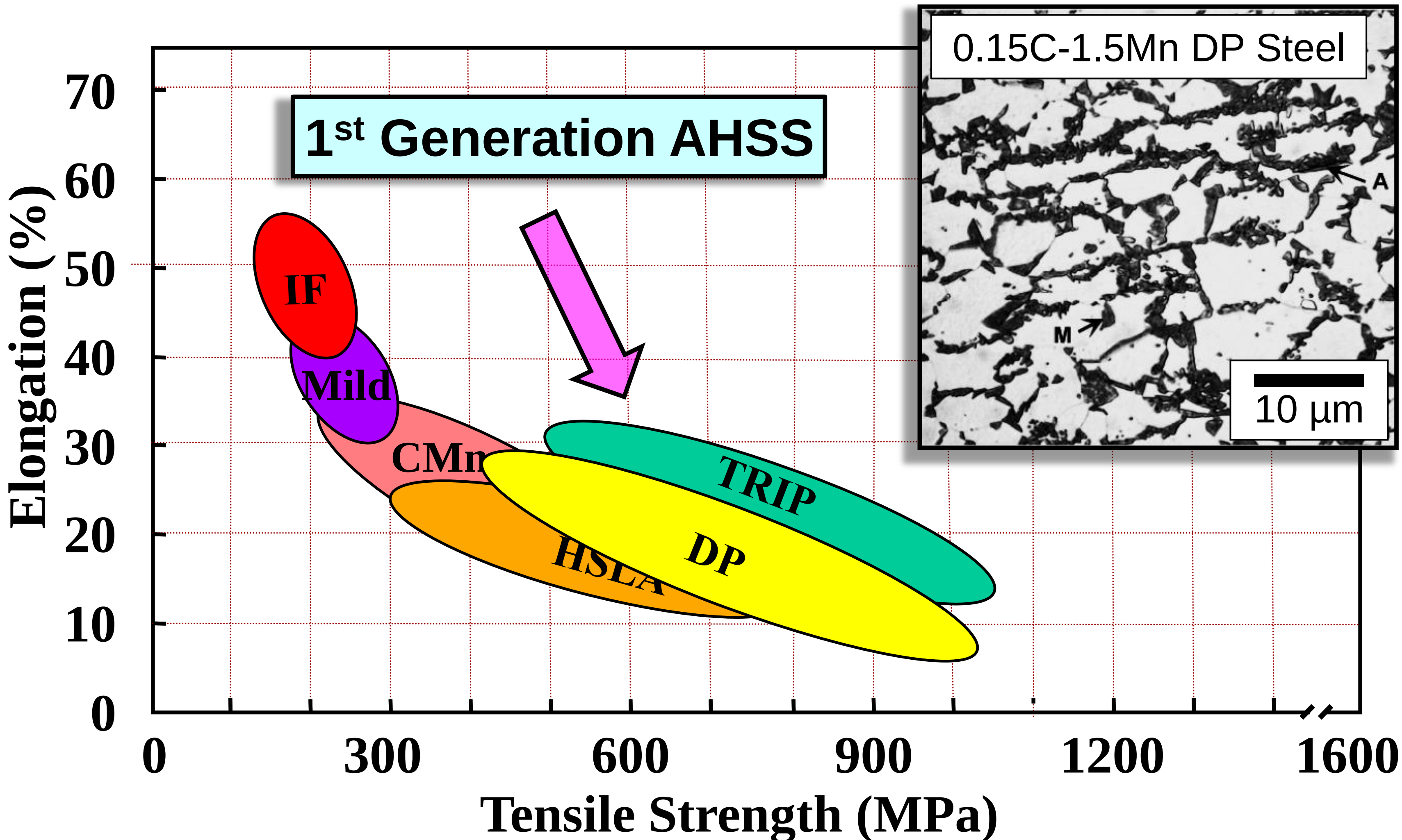


Plot adapted from - AISI: www.steel.org (2006)

Micrograph from: http://www.fgg.uni-lj.si/~/pmoze/ESDEP/master/wg02/l0100.htm (accessed July 2015)

Background: AHSS Developments

- **1970''s – 1990's: Dual-phase (DP) and TRansformation Induced Plasticity (TRIP) steels**

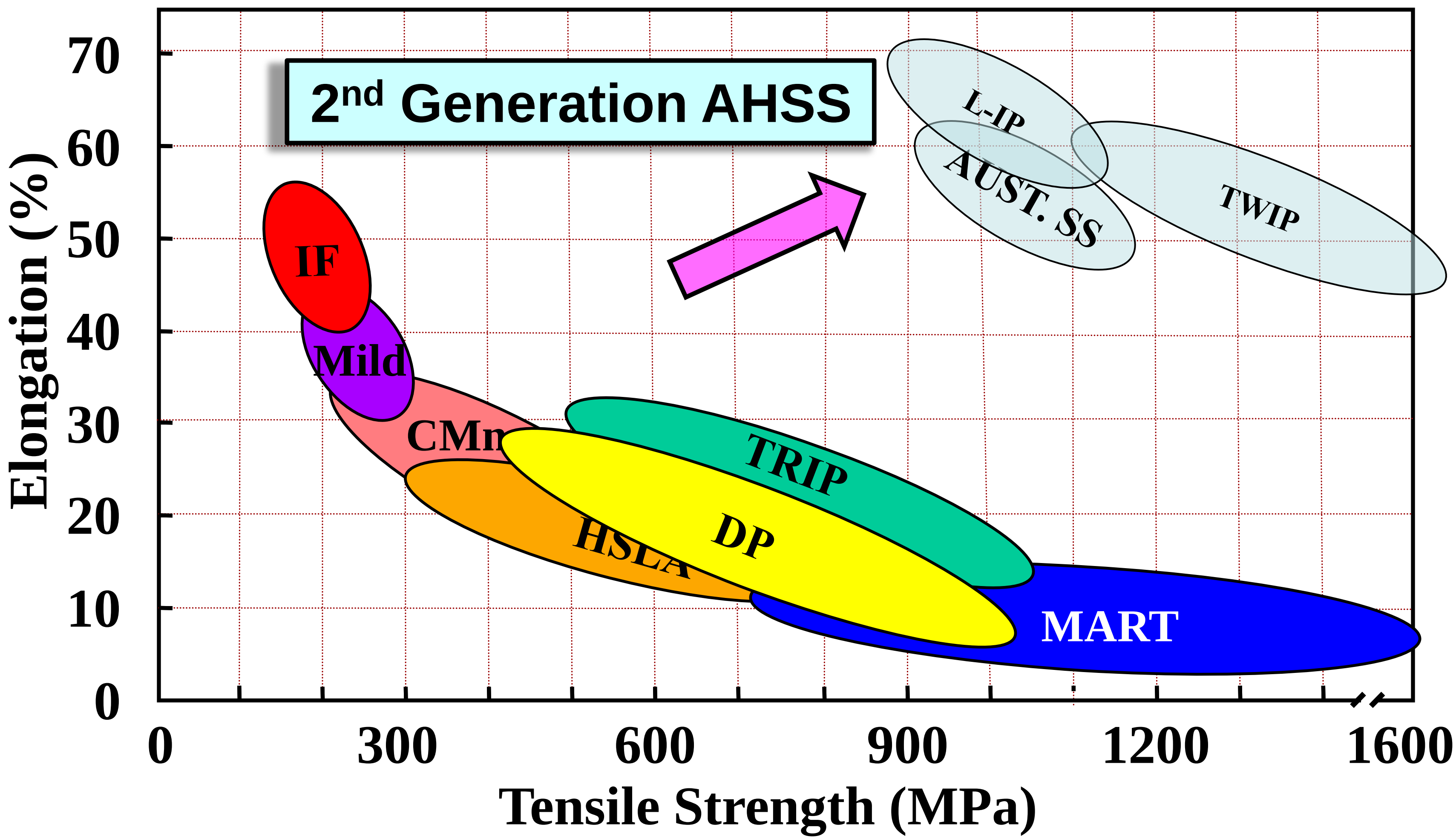


Plot adapted from - AISI: www.steel.org (2006)

Micrograph from: A.K. De, J.G. Speer, and D.K. Matlock, Applied Materials and Processes, vol. 161, Feb. 2003.

Background: AHSS Developments

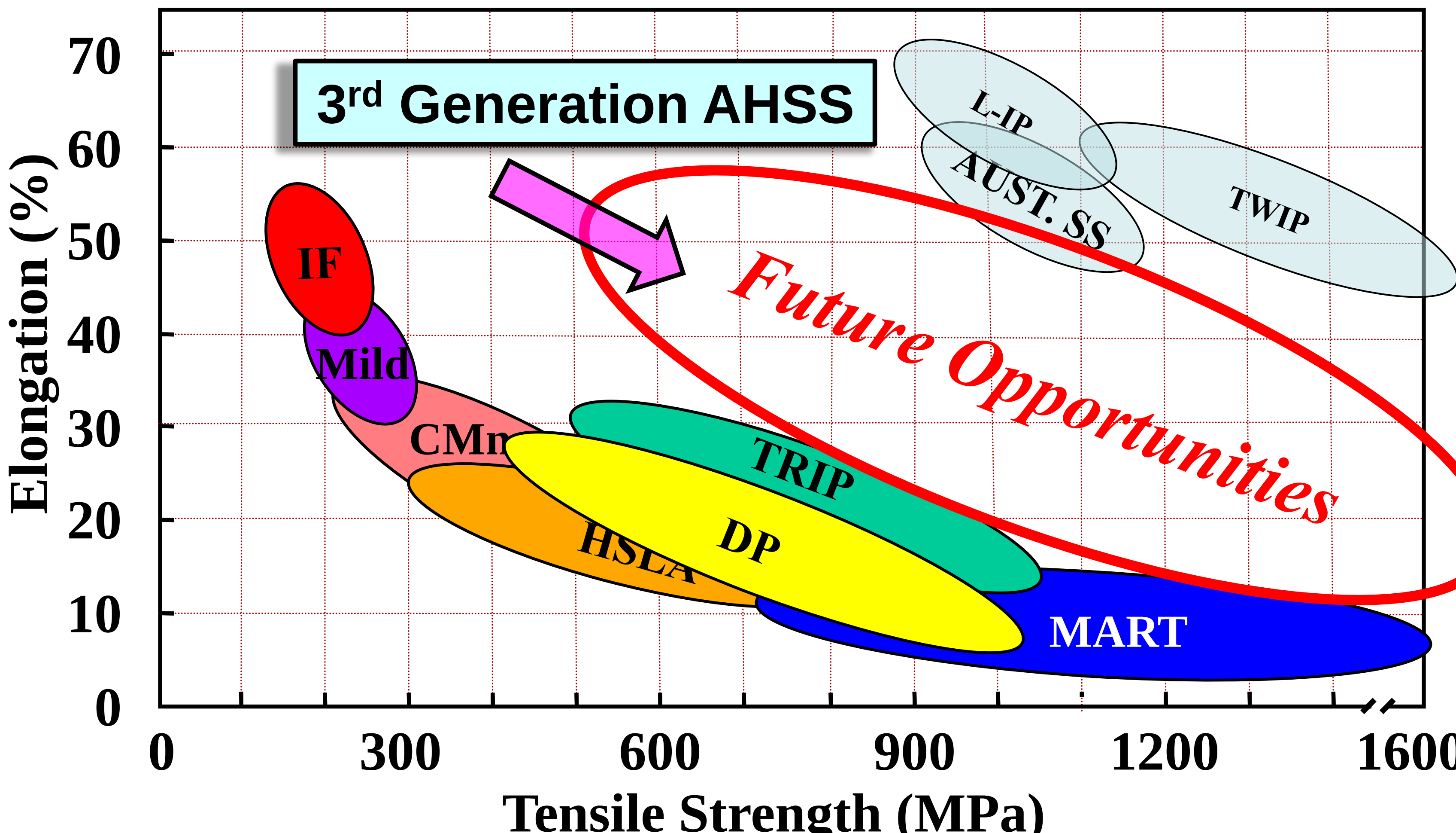
- Historical: Austenitic steels = expensive
  - Stainless and Twinning Induced Plasticity Steels (TWIP)



Plot adapted from - AISI: www.steel.org (2006)

Background: AHSS Developments

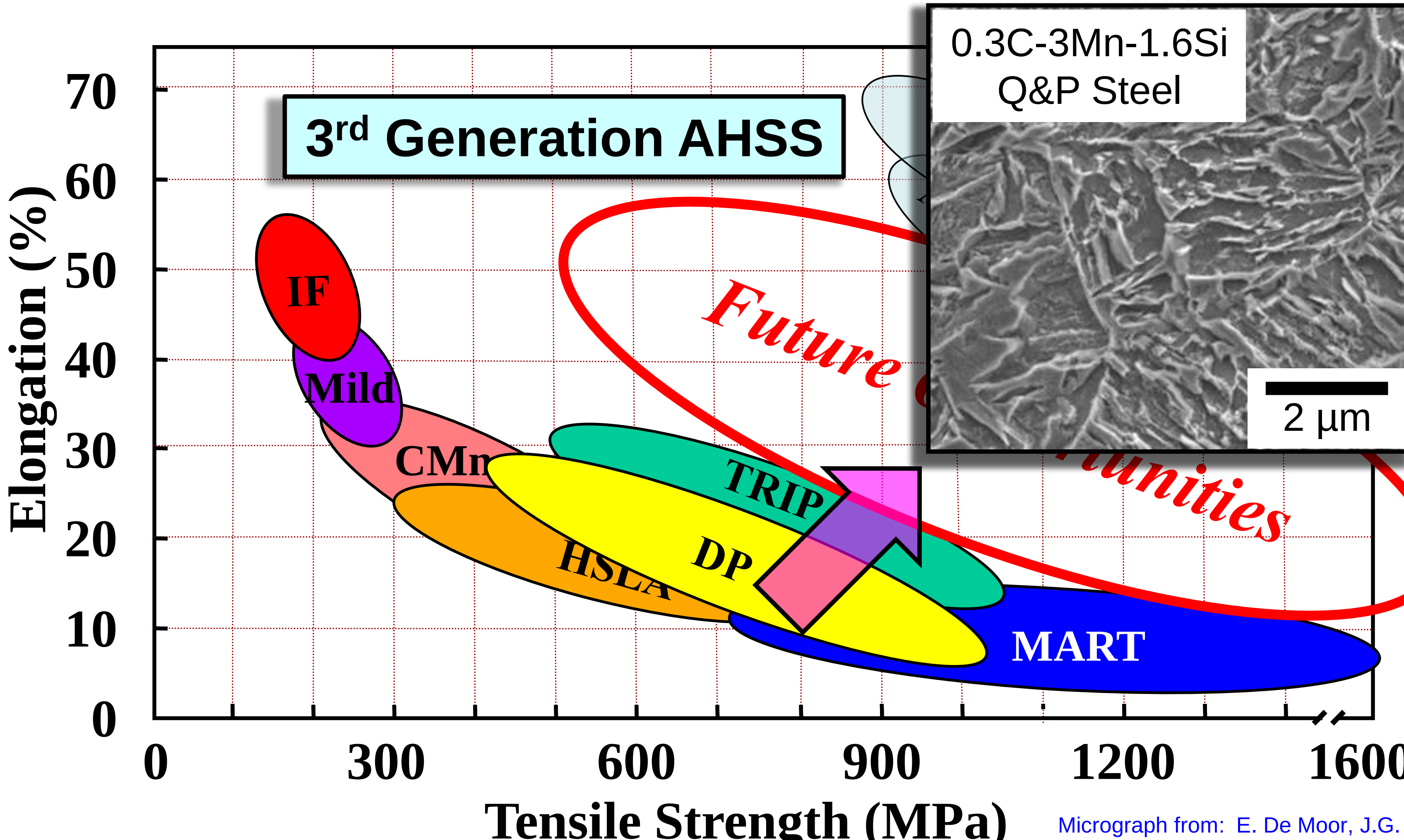
- 2000's and Beyond: Multiple new grades
  - Cost effective alternatives



Plot adapted from - AISI: www.steel.org (2006)

Background: AHSS Developments

- **2000's and Beyond: 3rd Generation AHSS**
  - **Example: Quenched and Partitioned Steel (Q&P)**



Plot adapted from - AISI: www.steel.org (2006)

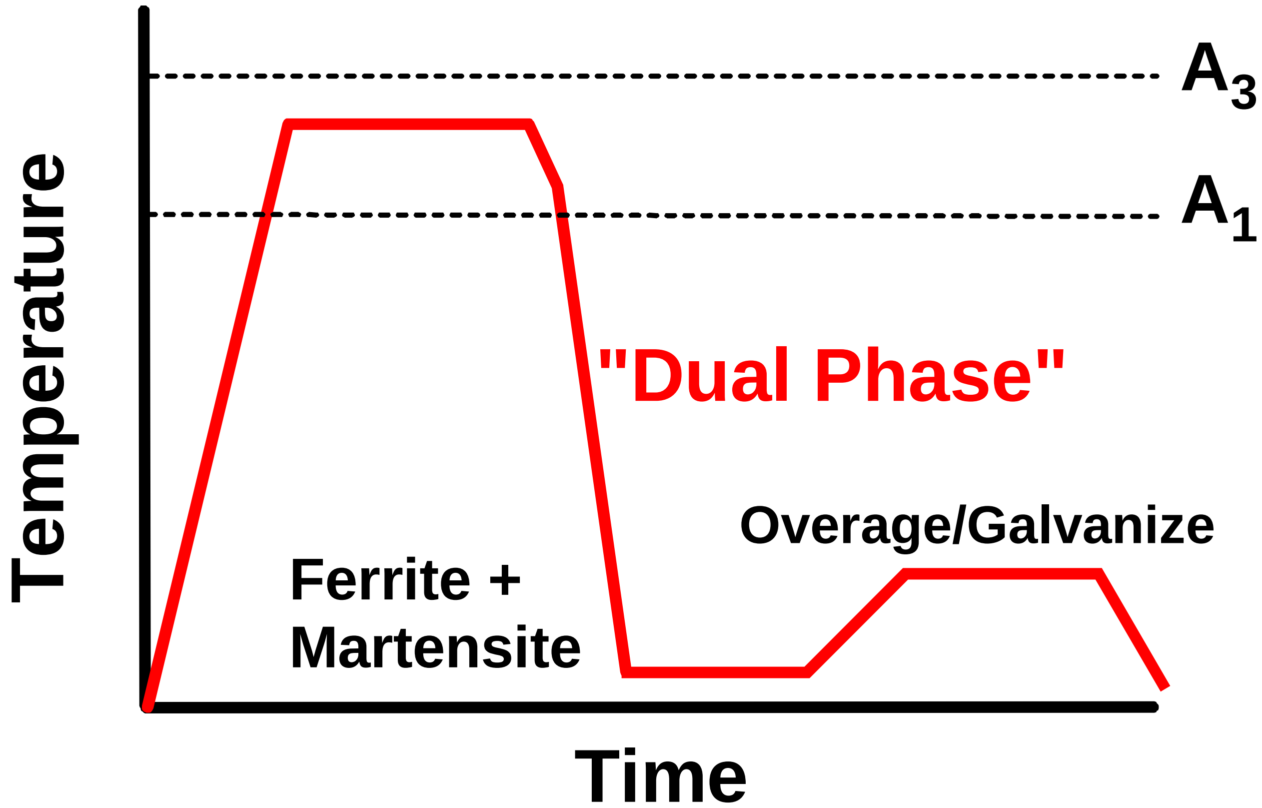
Micrograph from: E. De Moor, J.G. Speer, D.K. Matlock, J.H. Kwak, and S.B. Lee, *ISIJ Intl.* Vol. 51, no. 1, 2011, pp.137-144.

Dual-Phase Steel
"Fundamentals"

Hot Rolled DP Steels
Cold Rolled DP Steels

DP Steel Processing

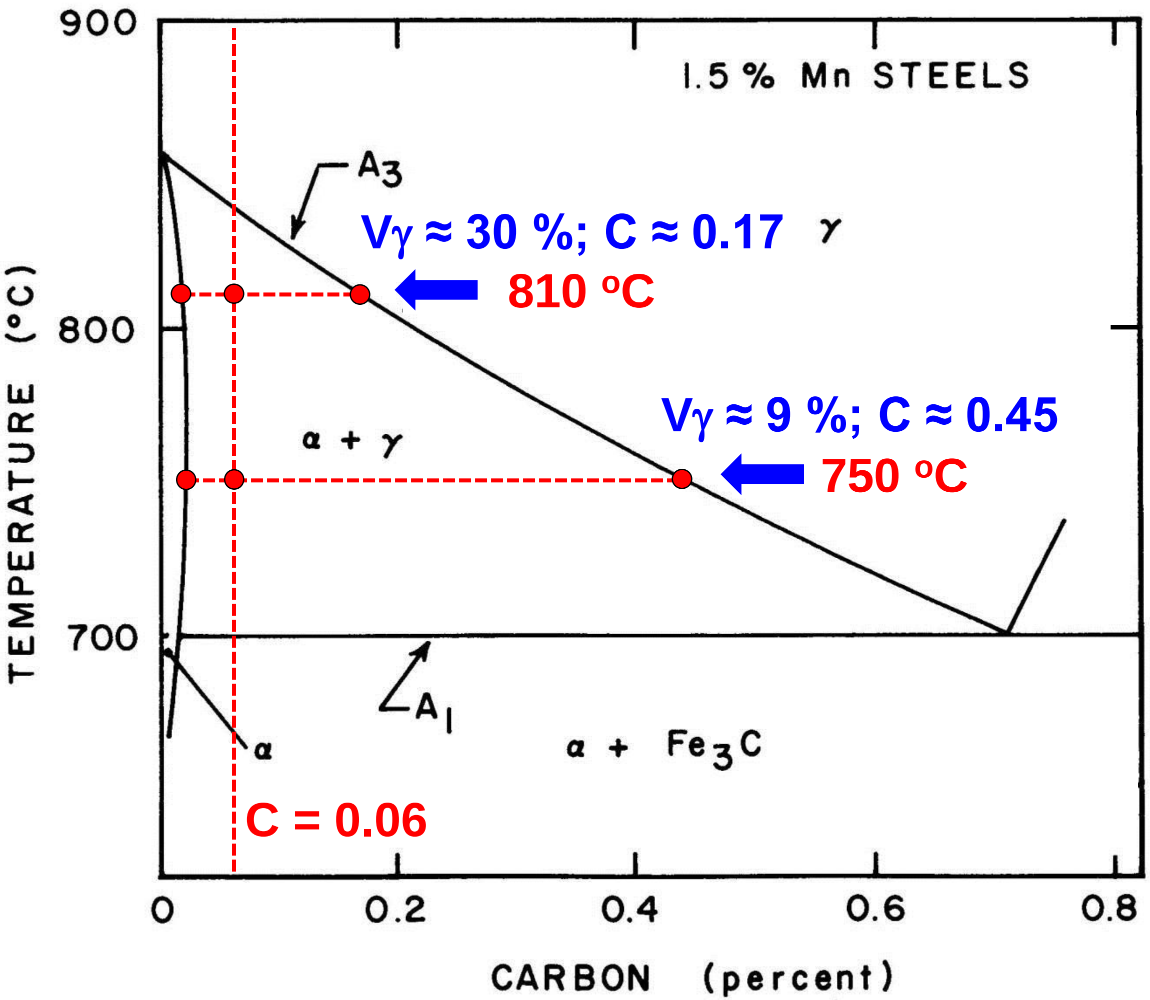
- Initially conceived based on "simple" routes for hot and cold rolled steels
  - Now, process controls offer significant opportunities to produce enhanced properties



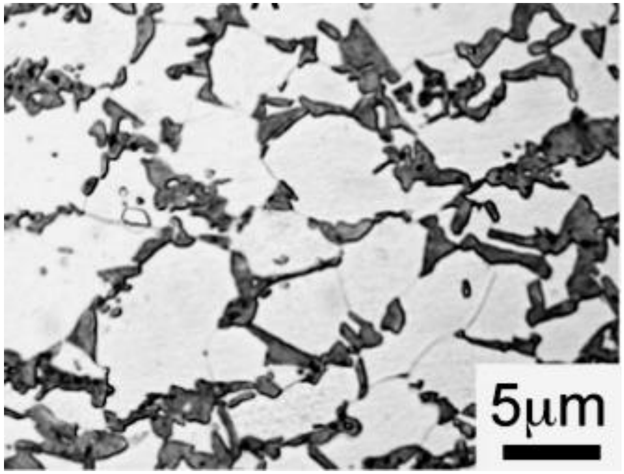
DP Steel Processing

"Typical:" **C: 0.05 - 0.15** **Mn: 1.0 – 2.0**

Others: Si, Cr, Ni, Mo, Nb, V



0.15C, 1.5 Mn, 1.5 Si
WQ from 775°C



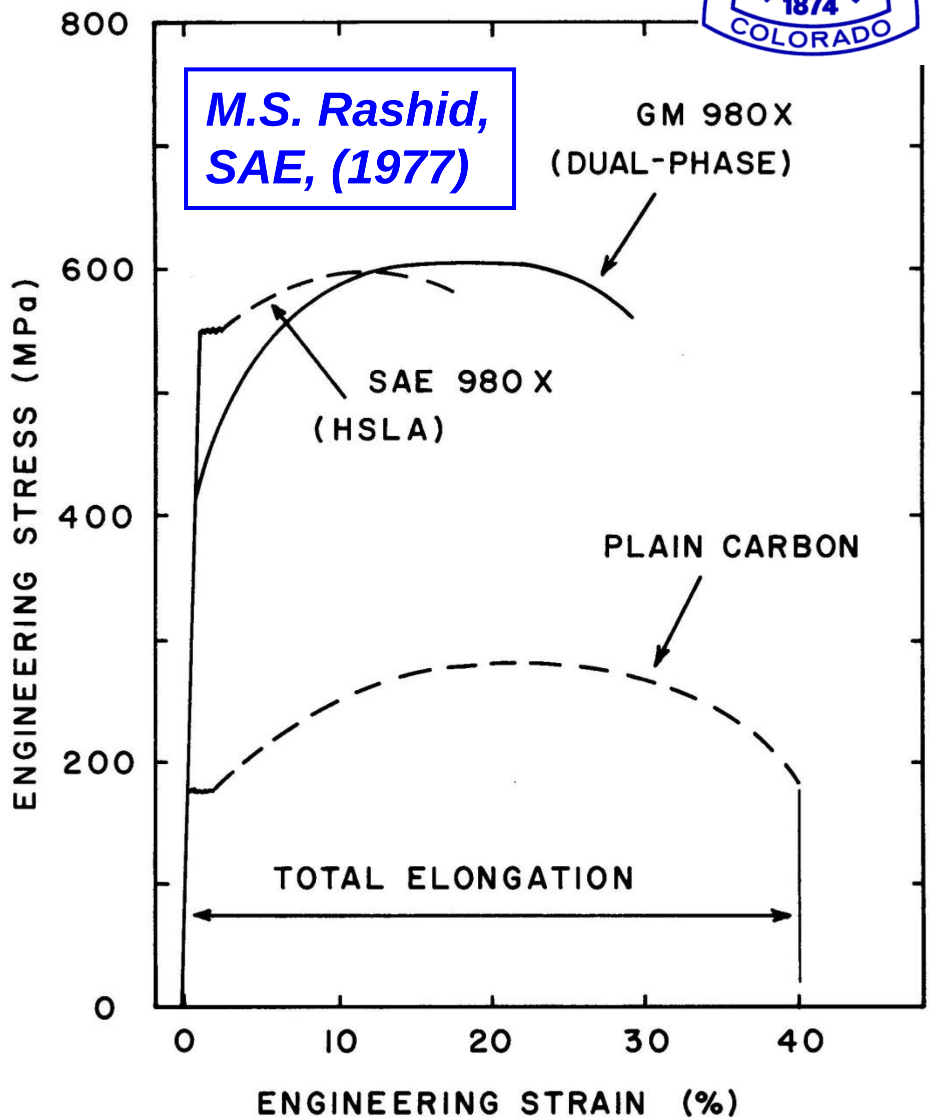
A.K. De, J.G. Speer, and D.K. Matlock, Applied Materials and Processes, vol. 161, Feb. 2003.

Dual-Phase Steels: "The Early Days"

Significant Parallel Research Activities

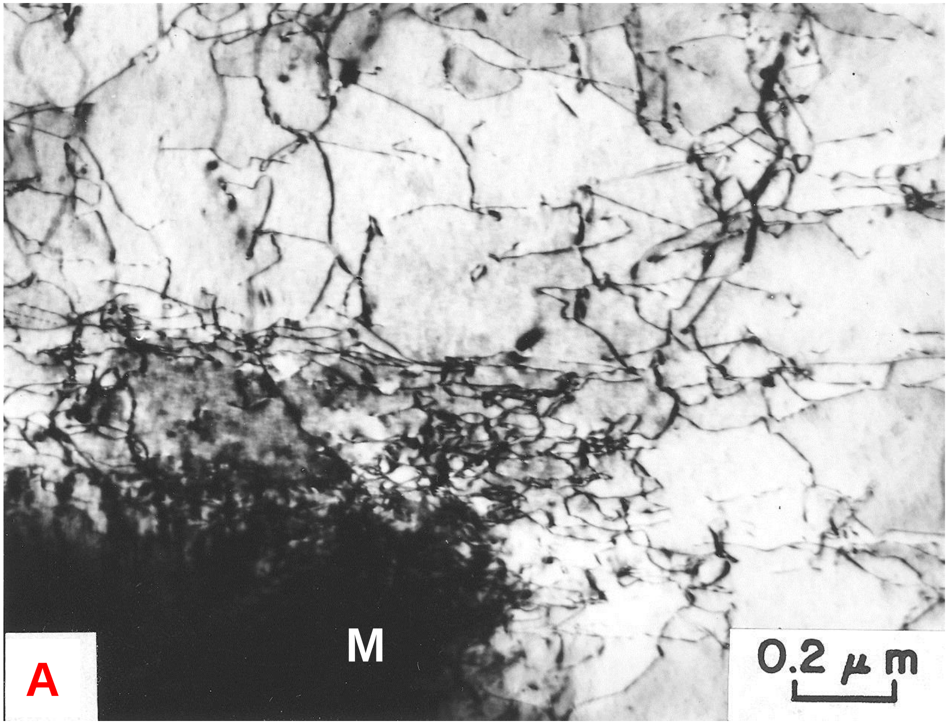
- **Nippon Steel**
  - **1975:** **S. Hayami and T. Furukawa, Microalloying '75**
- **GM**
  - **1976:** **M.S. Rashid, SAE + others**
- **Ford**
  - **1978:** **R.G. Davies, Met. Trans. + others**

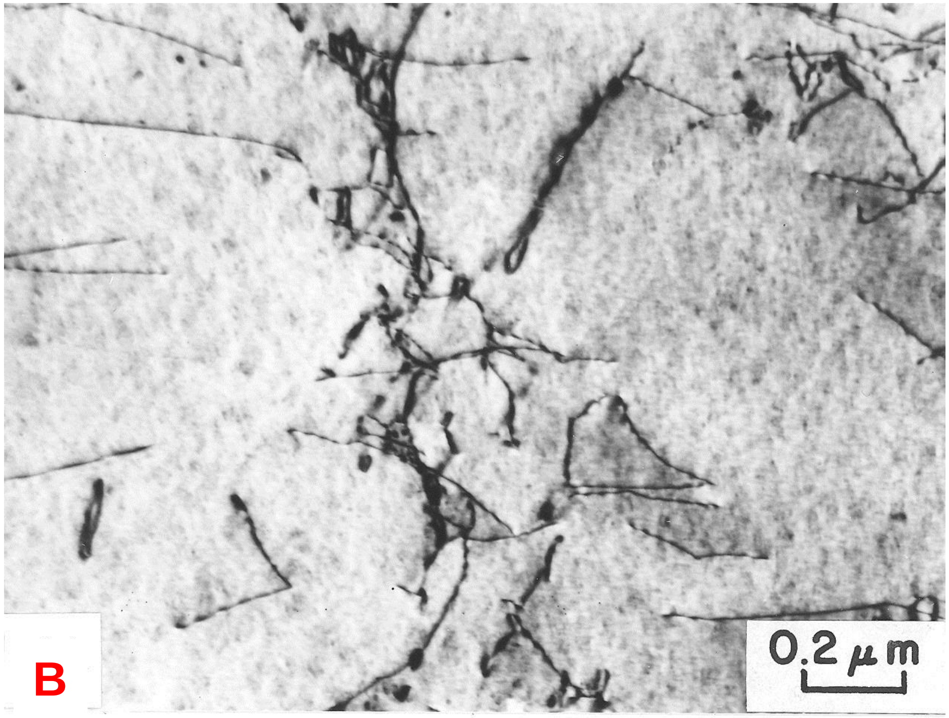
M.S. Rashid, SAE, (1977)

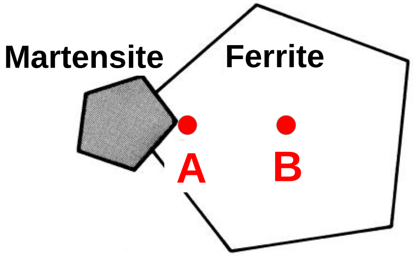


Strain Accumulation in Ferrite

- **C-Mn-Si DP Steel**
- **As heat treated**



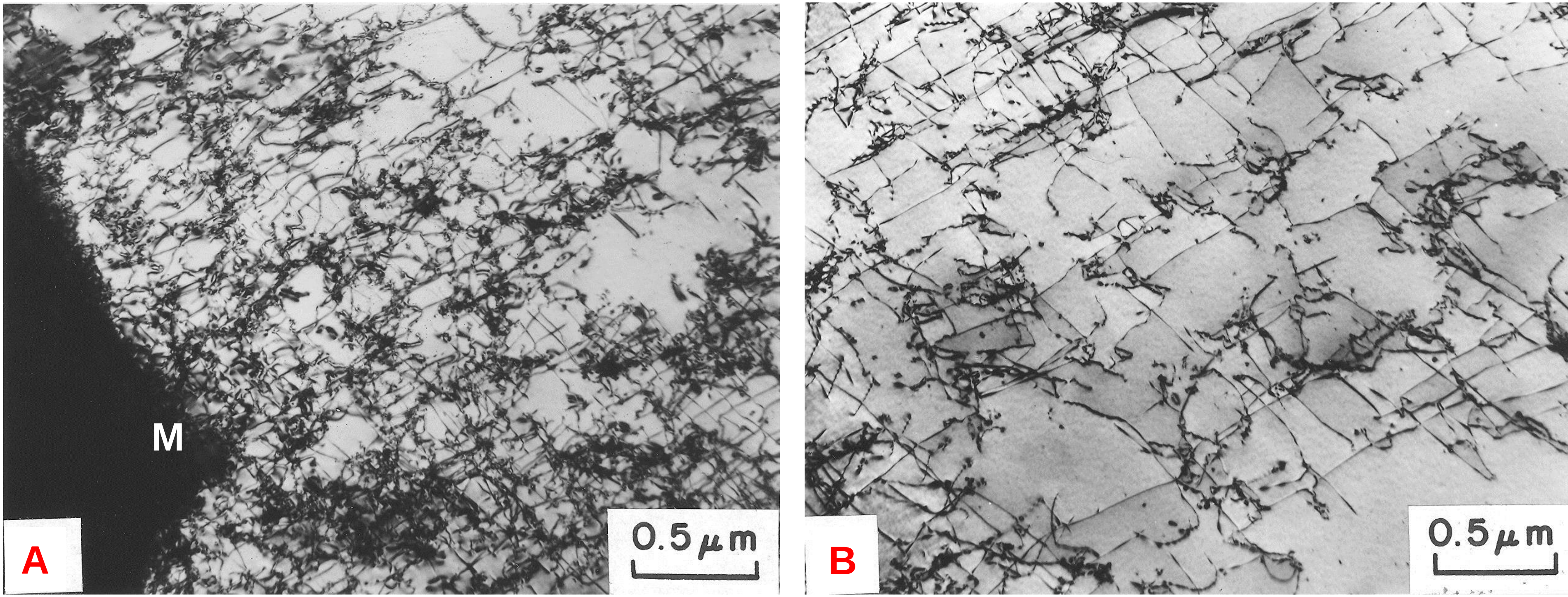


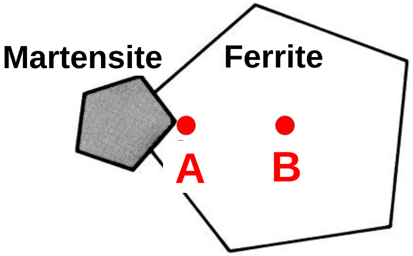


D.A. Korzekwa, D.K. Matlock, and G. Krauss, Metallurgical Transactions A, vol. 15A, 1984, pp. 122l 1228.

Strain Accumulation in Ferrite

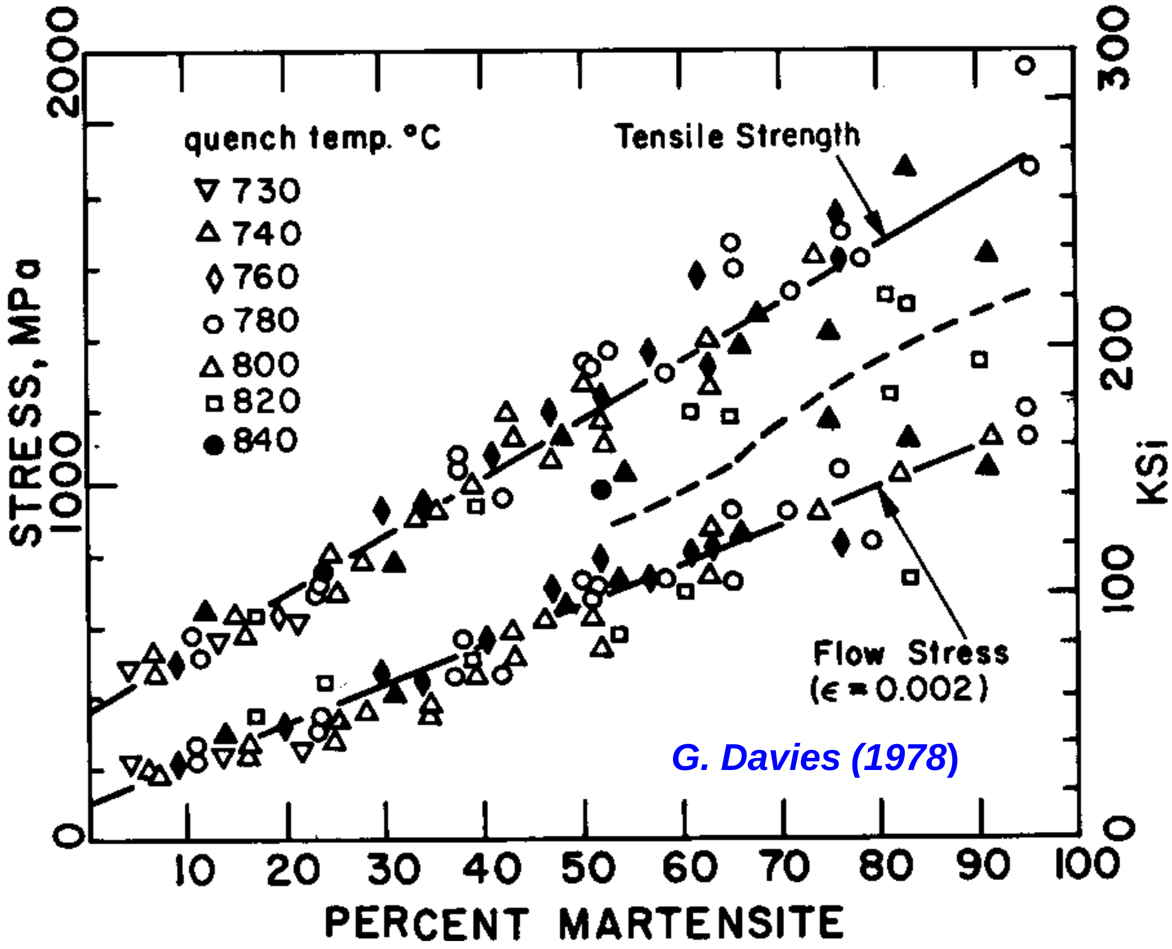
- **C-Mn-Si DP Steel**
- **2 % Tensile Strain**





D.A. Korzekwa, D.K. Matlock, and G. Krauss, Metallurgical Transactions A, vol. 15A, 1984, pp. 122l 1228.

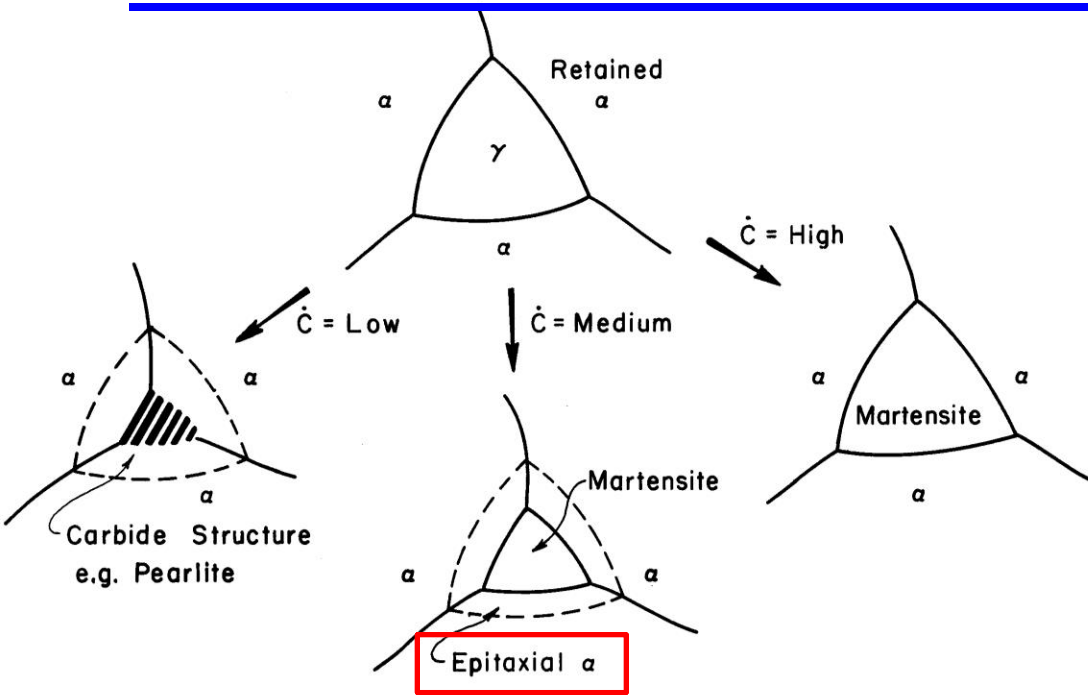
Strengthening in DP Steels



Strength increase follows rule of mixtures for composites: $\sigma_T = V_f\sigma_f + V_M\sigma_M$

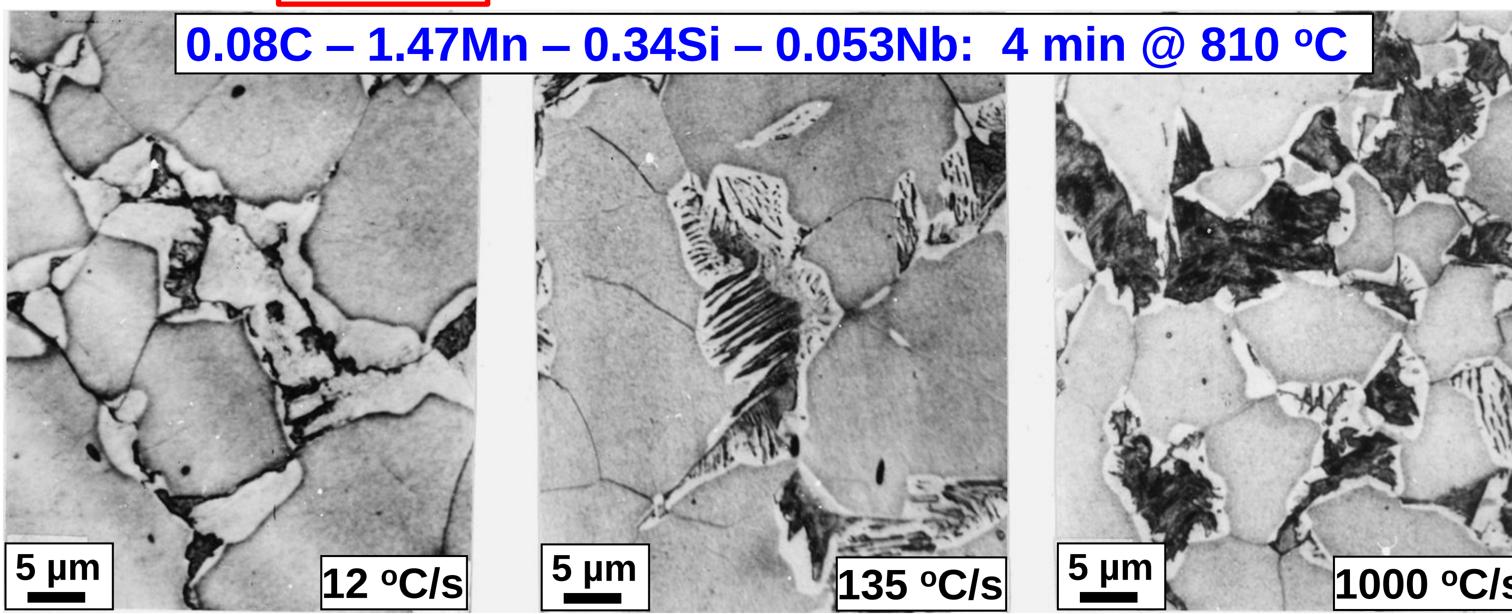
R.G. Davies, Metallurgical Transactions A, vol. 9A, 1978, pp. 671-679.

Austenite Transformation in DP Steels



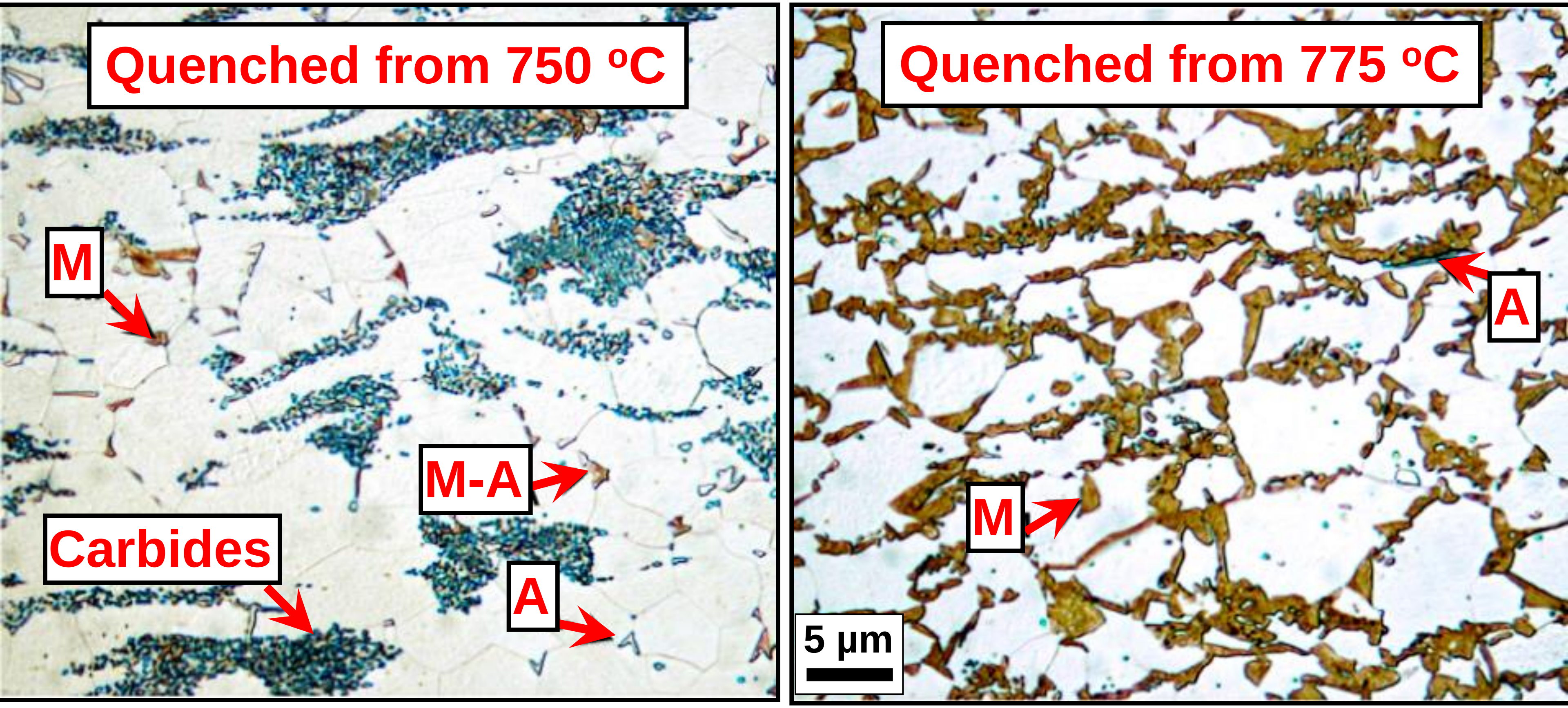
R.D. Lawson, D.K. Matlock, and G. Krauss, "An Etching Technique for Microalloyed Dual Phase Steels," Metallography, Vol. 13, 1980, pp. 71 87.

0.08C – 1.47Mn – 0.34Si – 0.053Nb: 4 min @ 810 ^oC



DP Microstructures…..

Special color tint etching: 0.15C – 1.5Mn – 1.5Si

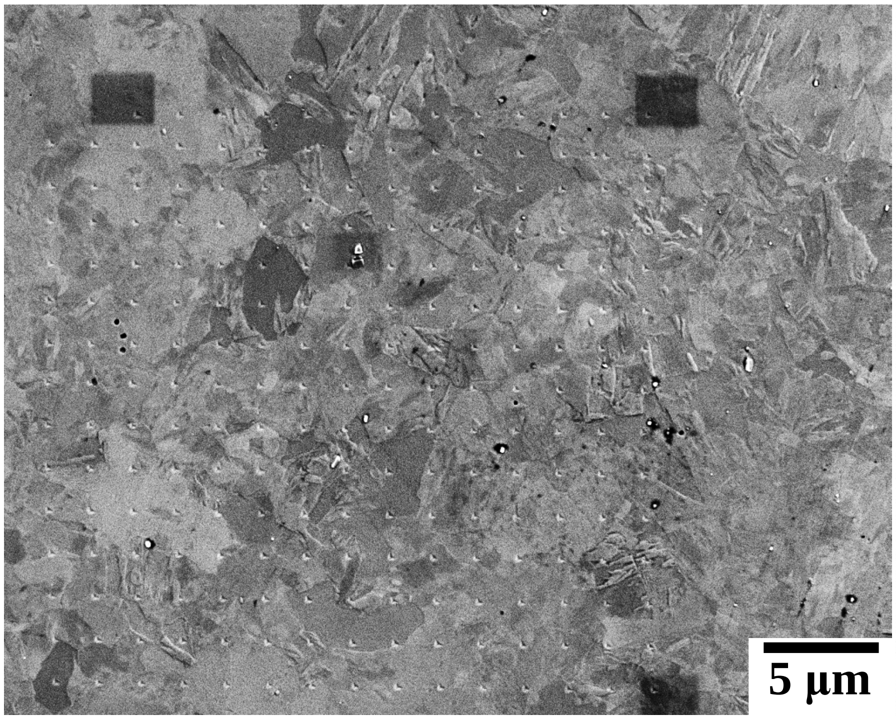


DP Steels = “*More” than martensite and ferrite*

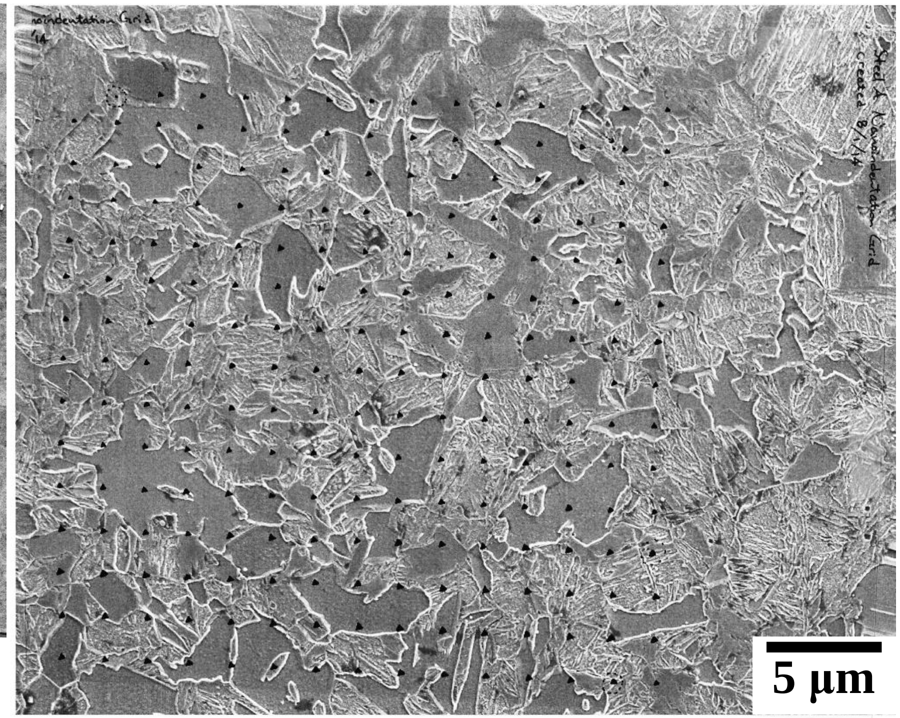
Amar K. De, J.G. Speer and D.K. Matlock, “Color Tint-Etching for Characterization of Complex Microstructures in Multiphase Steels,” Advanced Materials and Processes, vol. 161, no. 2, 2003, pp. 27-30..

Martensite and Ferrite Properties

- **Nanoindentation**
- **DP Steel: 0.14 C, 2.04 Mn – cold rolled and heat treated**
- **YS = 920 MPa; UTS = 1220 MPa**



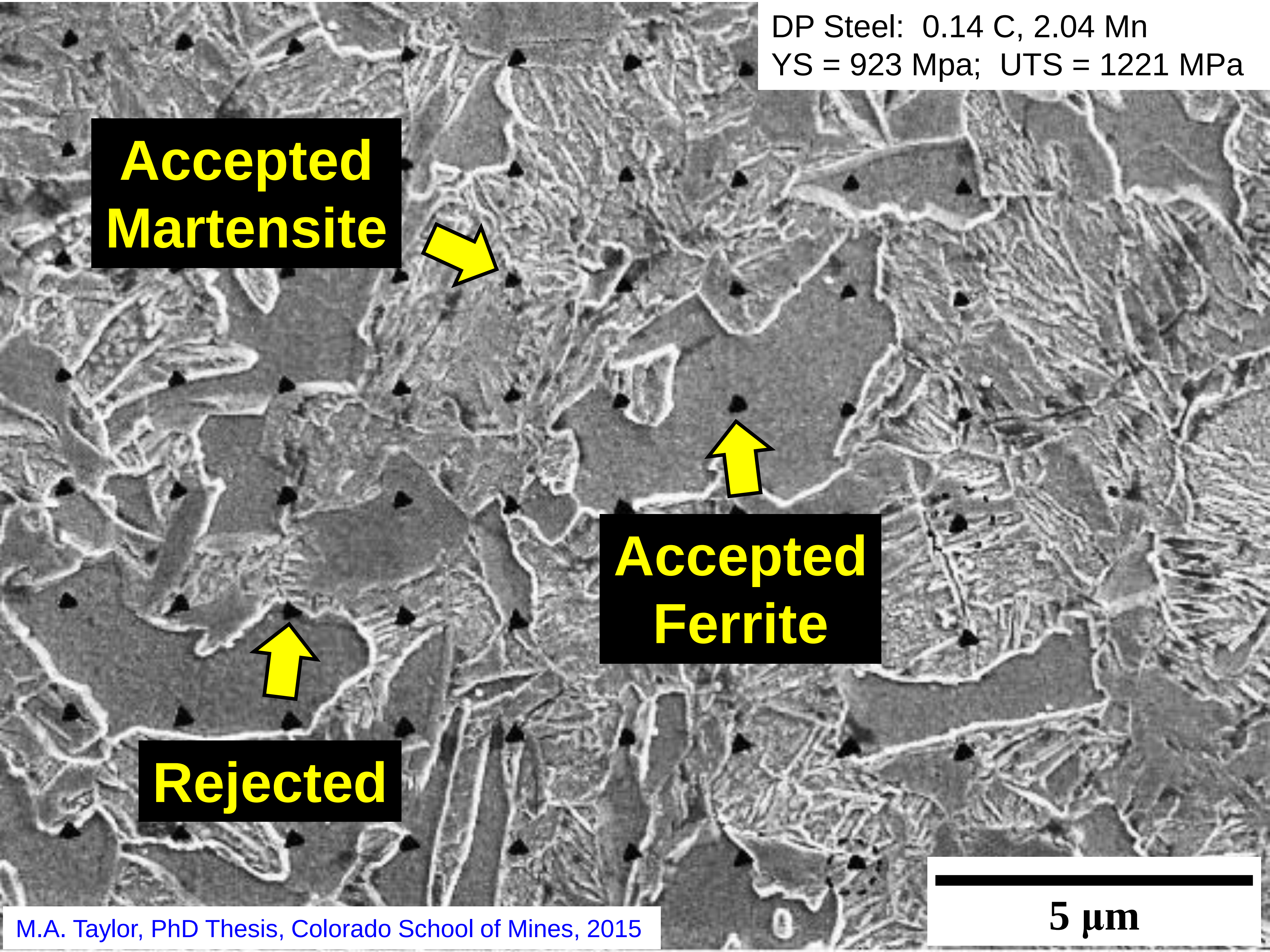
SEM Micrograph: Unetched



SEM Micrograph: 2 pct nital etch

Black reference boxes = 4 minutes of SEM beam raster
View = normal to rolling plane

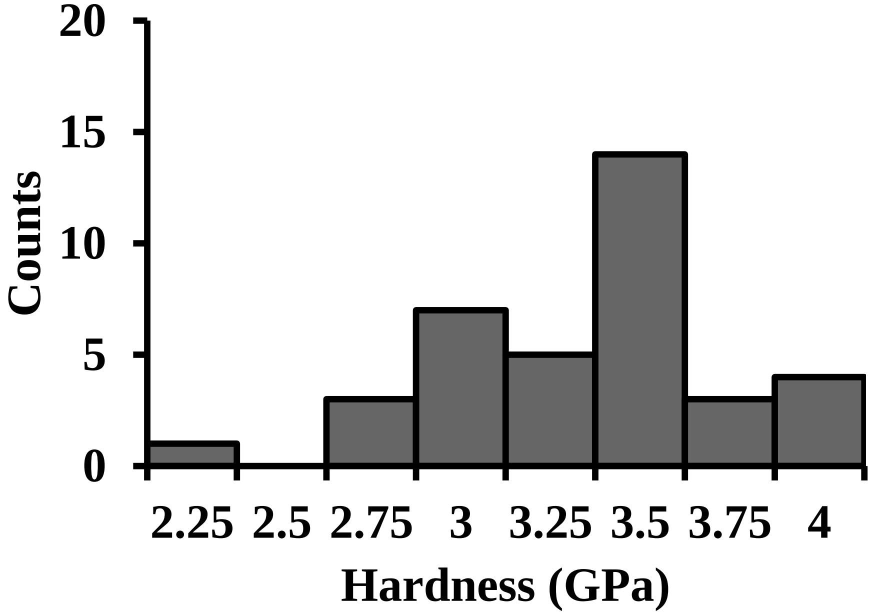
M.A. Taylor, PhD Thesis, Colorado School of Mines, 2015


DP Steel: 0.14 C, 2.04 Mn
YS = 923 Mpa; UTS = 1221 MPa
Accepted Martensite
Accepted Ferrite
Rejected
5 µm
M.A. Taylor, PhD Thesis, Colorado School of Mines, 2015

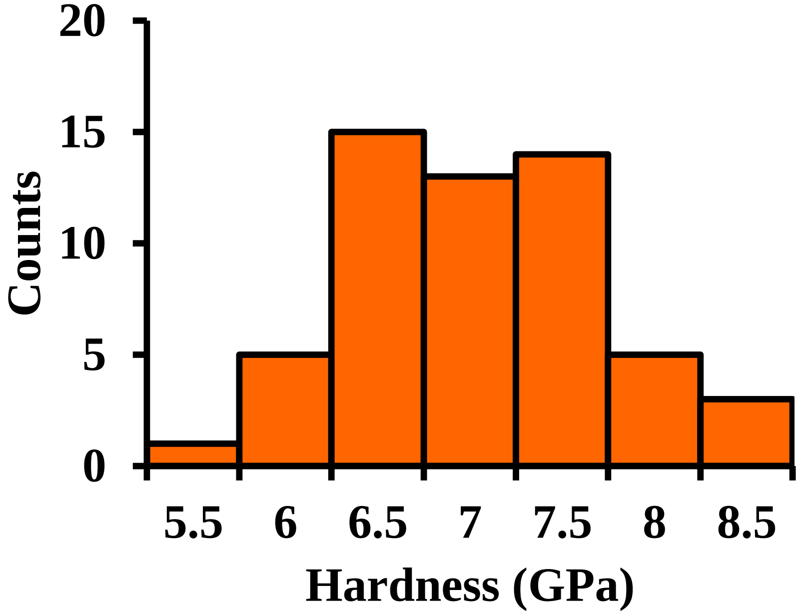
Martensite and Ferrite Properties

- Nanoindentation
- DP Steel: 0.14 C, 2.04 Mn – cold rolled and heat treated
- YS = 920 MPa; UTS = 1220 MPa

Ferrite: Avg = 3.2 GPa



Martensite: Avg = 7.3 GPa

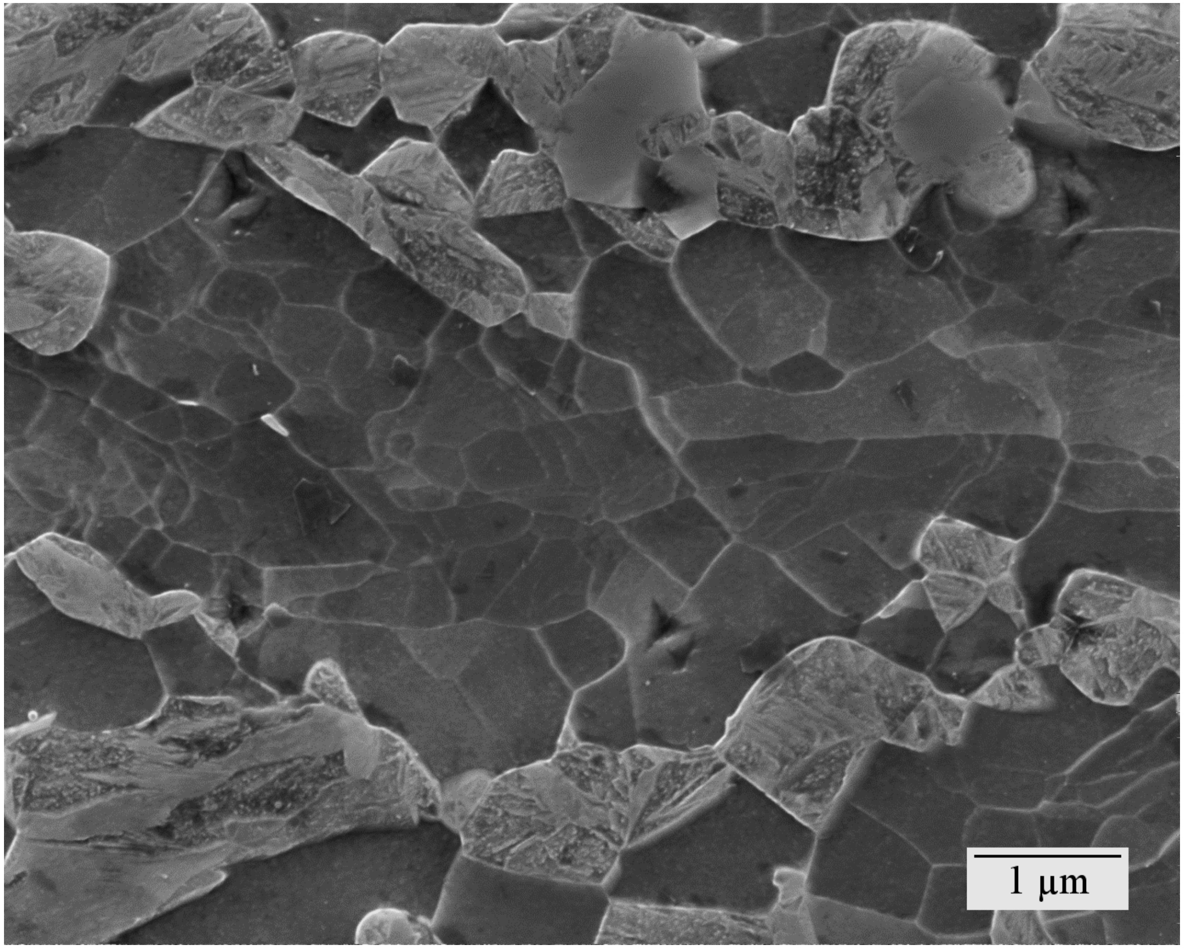


Data approximate a normal distribution

M.A. Taylor, PhD Thesis, Colorado School of Mines, 2015

Why Hardness Variability?

Influenced by the mixture of recovered (in example) and recrystallized ferrite in the samples

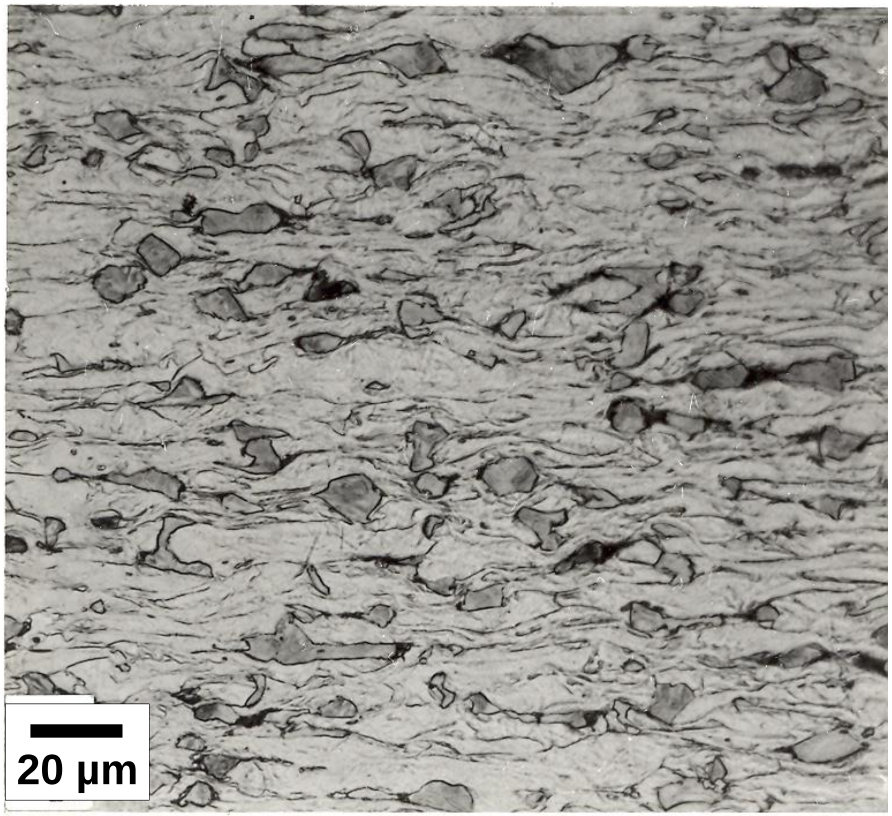


T.W. Brown, PhD Thesis, ASPPRC/CSM, 2014

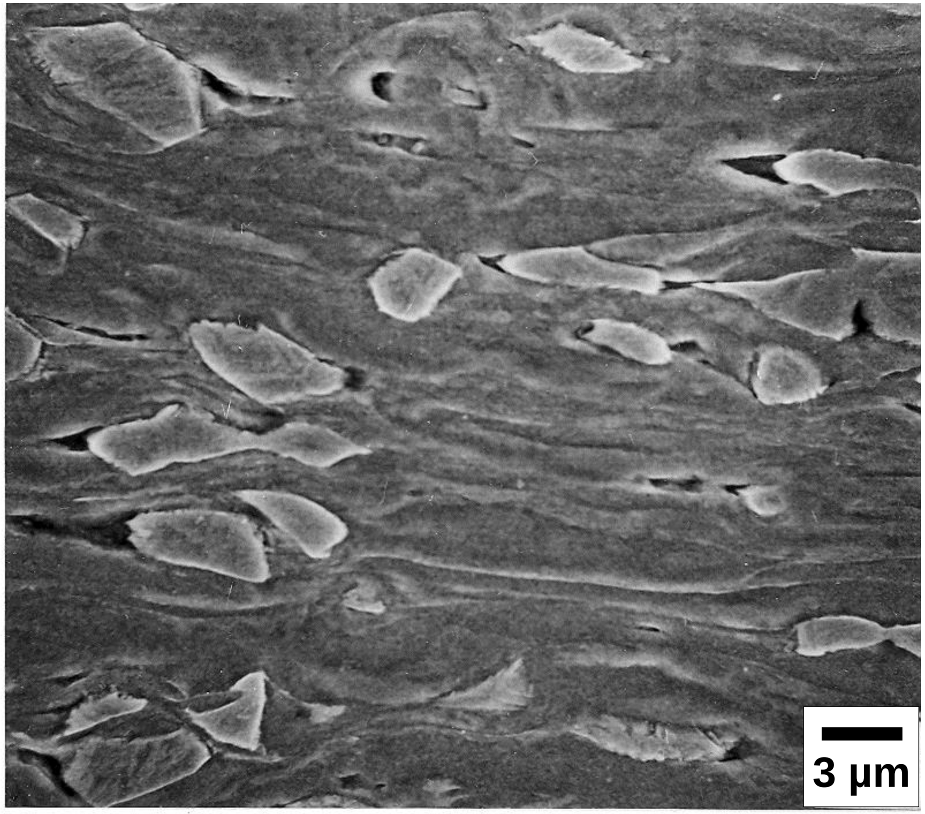
DP Steel: 0.1 C, 2.0 Mn, 0.3 Cr, 0.3 Mo (wt-%)

Consequence of Hardness Differences

- **1008 steel heat treated to DP microstructure**
- **Cold drawn to wire – true strain = 1.3**



Light Optical Micrograph



SEM Micrograph

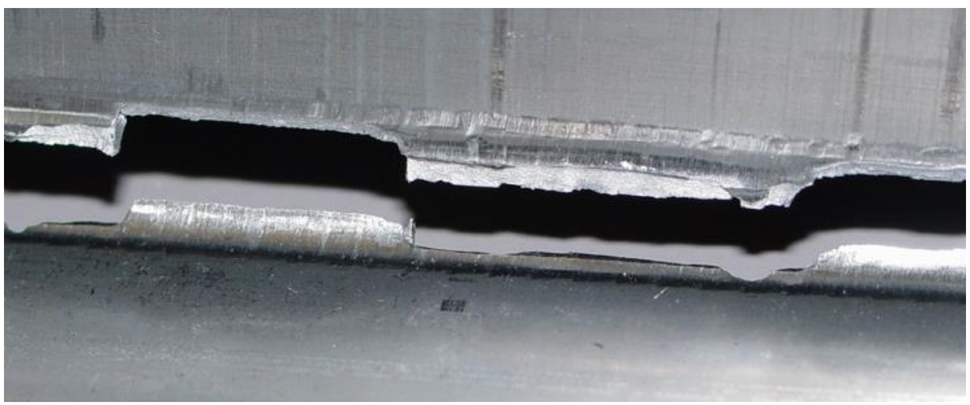
Dual-Phase Processing:
30 min @ 760 °C + WQ
25 vol % Martensite

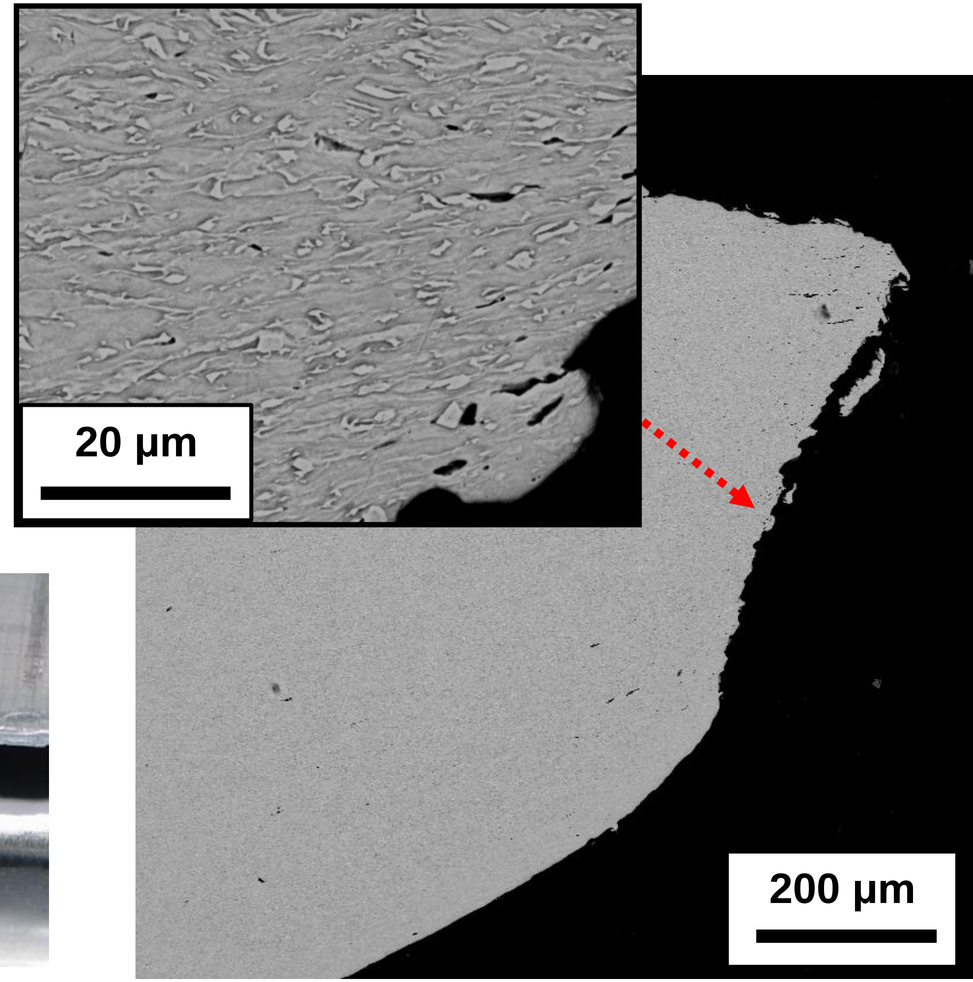
2 pct nital etch

D.A. Korzekwa, R.D. Lawson, D.K. Matlock, and G. Krauss, *Scripta Met.,* Vol. 14, 1980, pp. 1023-1028.

Example Failures: Formability Limited by Fracture

DP Steels





Fractures often referred to as "Shear Fractures"

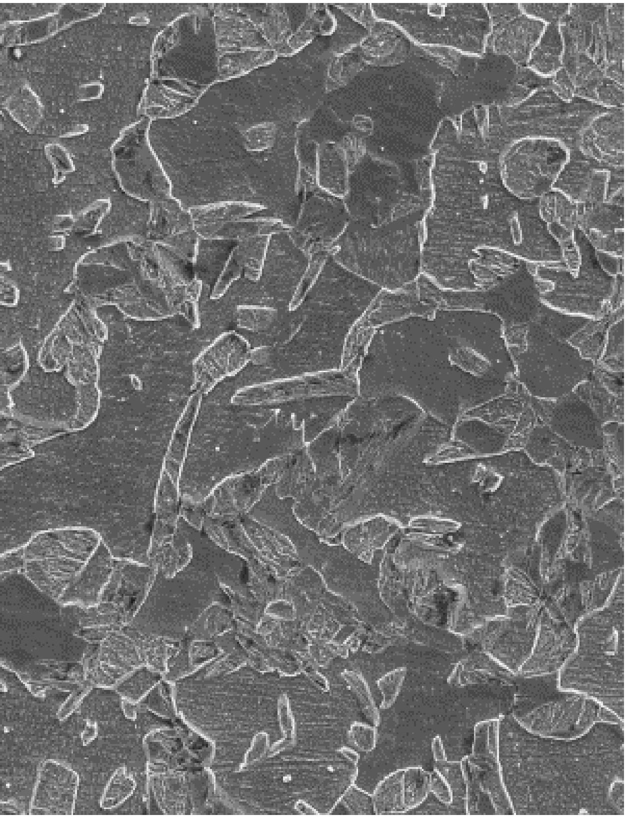
Images Courtesy of Jim Fekete, GM,
and Matt Walp, DaimlerChrysler

A.W. Hudgins, PhD Thesis,
Colorado School of Mines, 2010.

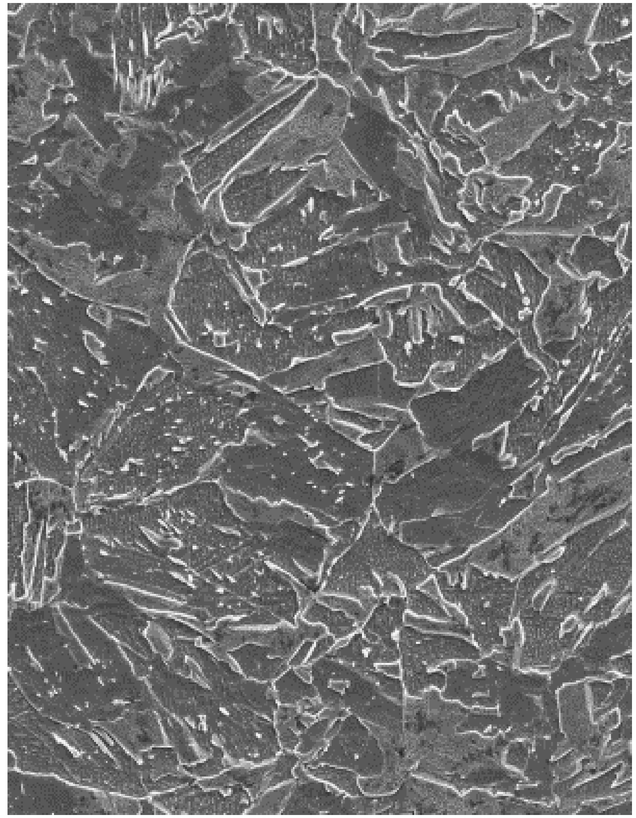
Commercially-Produced DP980 Steels

SEM Micrographs; Secondary Electron Images

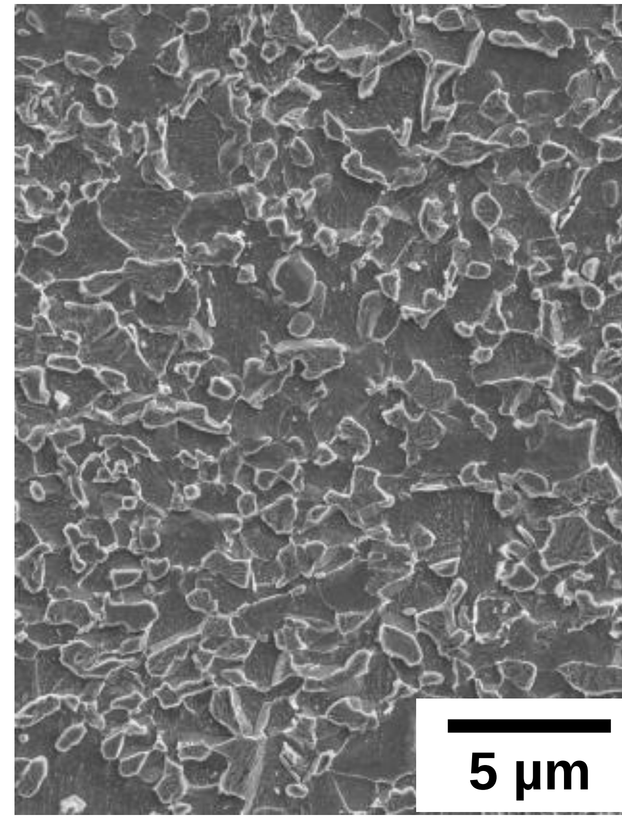
2 pct nital etch



Steel A:
0.09 C, 2.13 Mn, 0.57 Si
0.02 Cr, 0.07 Mo



Steel D:
0.12 C, 2.47 Mn, 0.03 Si
0.25 Cr, 0.36 Mo

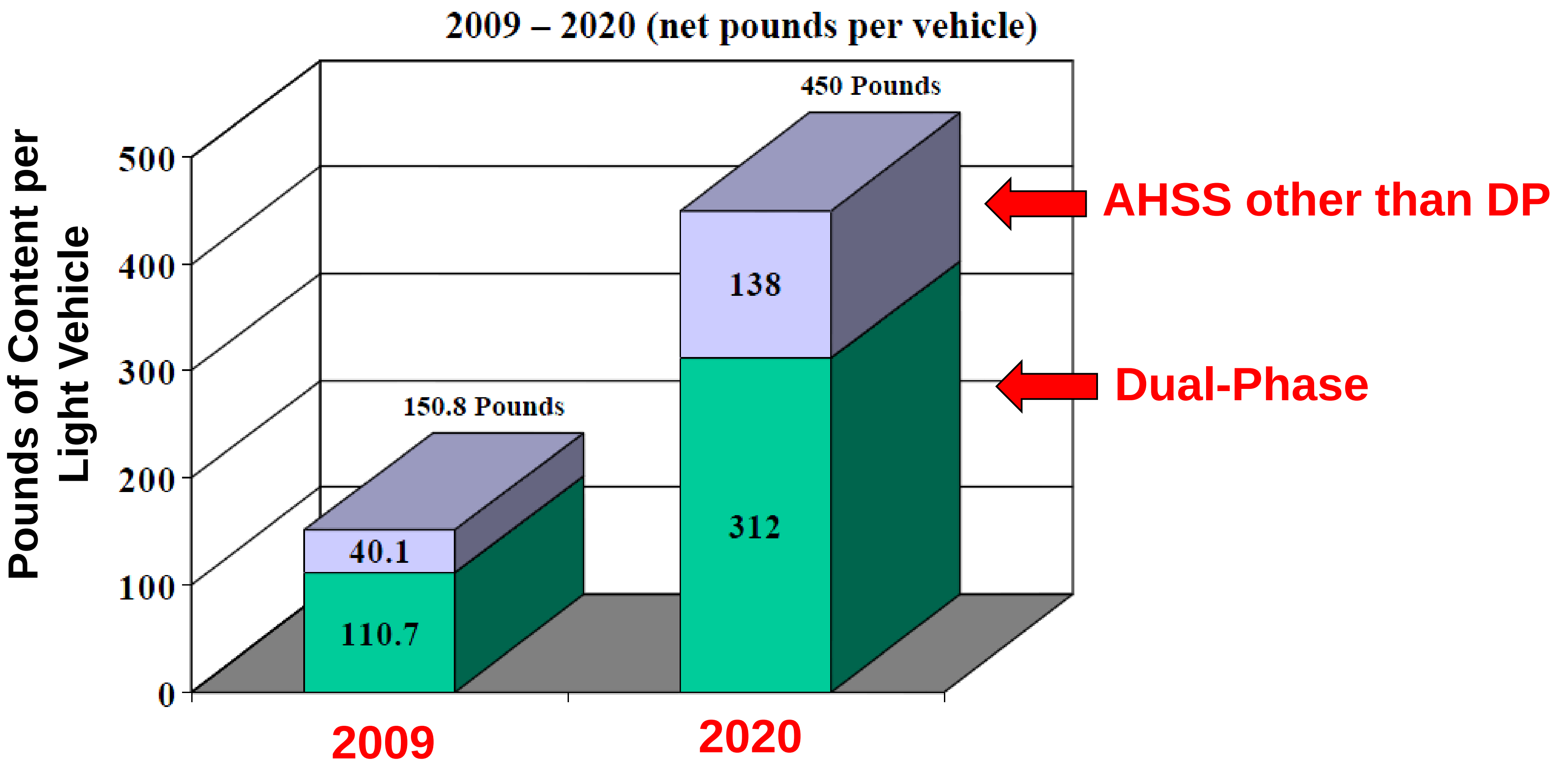


Steel H:
0.15 C, 1.93 Mn, 0.64 Si
0.32 Cr, 0.01 Mo

M.D. Taylor, K.S. Choi, X. Sun, D.K. Matlock, C.E. Packard, L. Xu, and F. Barlat, “Correlations Between Nanoindentation Hardness and Macroscopic Mechanical Properties in DP980 Steels,” Materials Science and Engineering A, vol. 597, 2014, pp. 431-439..

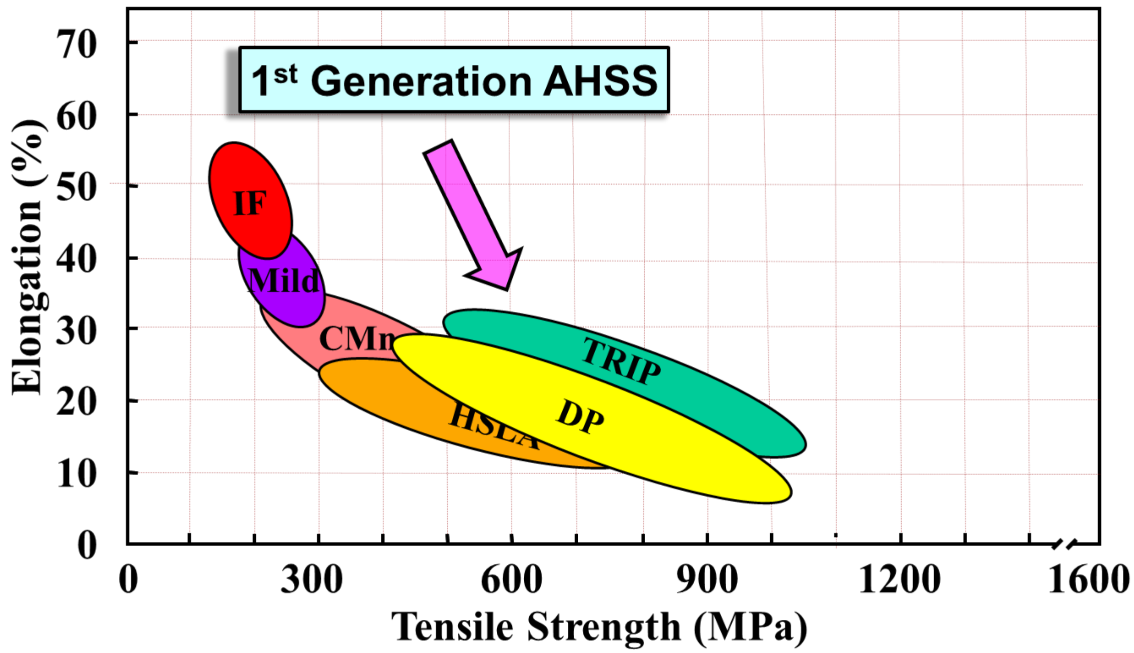
Trends: AHSS use in North America

- **Dual-phase steels, AHSS of choice**



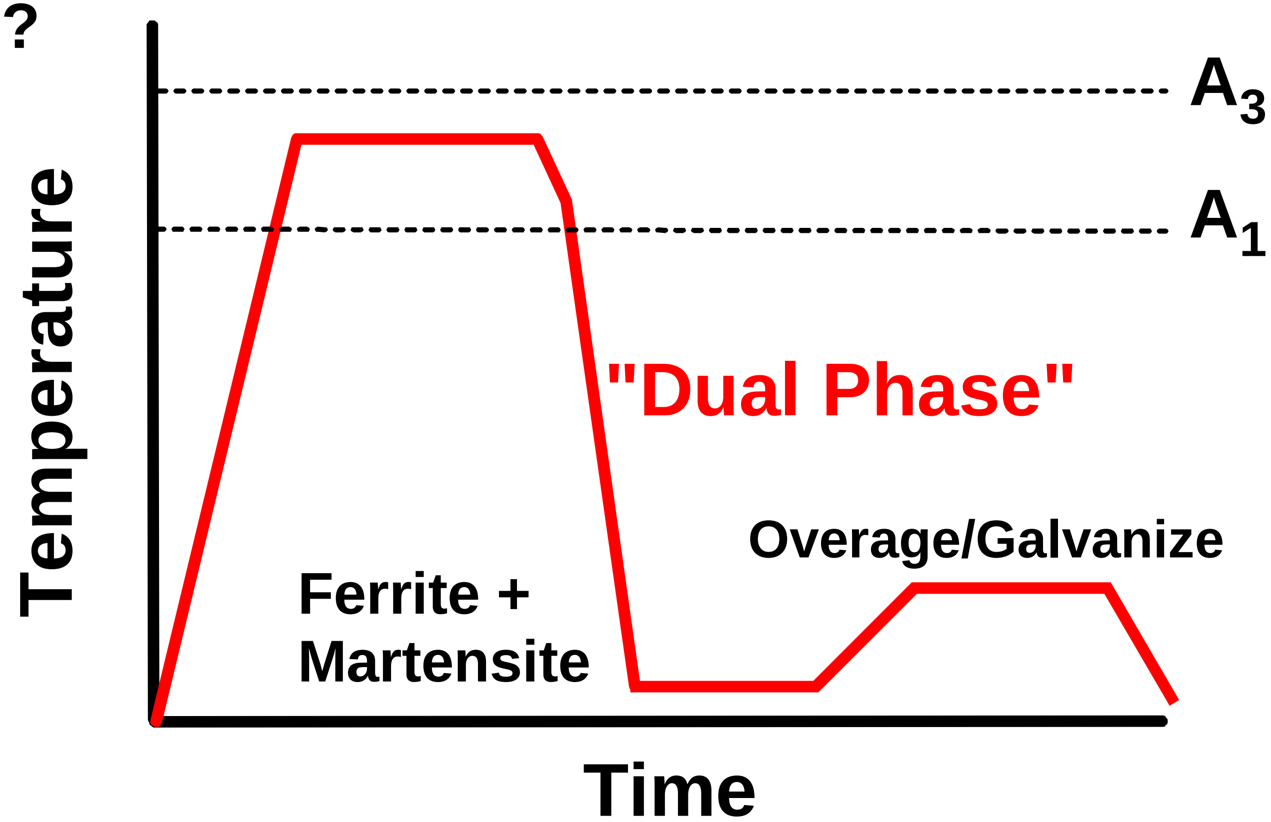
R.A. Schultz and A.K. Abraham, Ducker Worldwide @ GDIS, 2010. www.autosteel.org

Fundamentals of TRIP (TRansformation Induced Plasticity Steels)



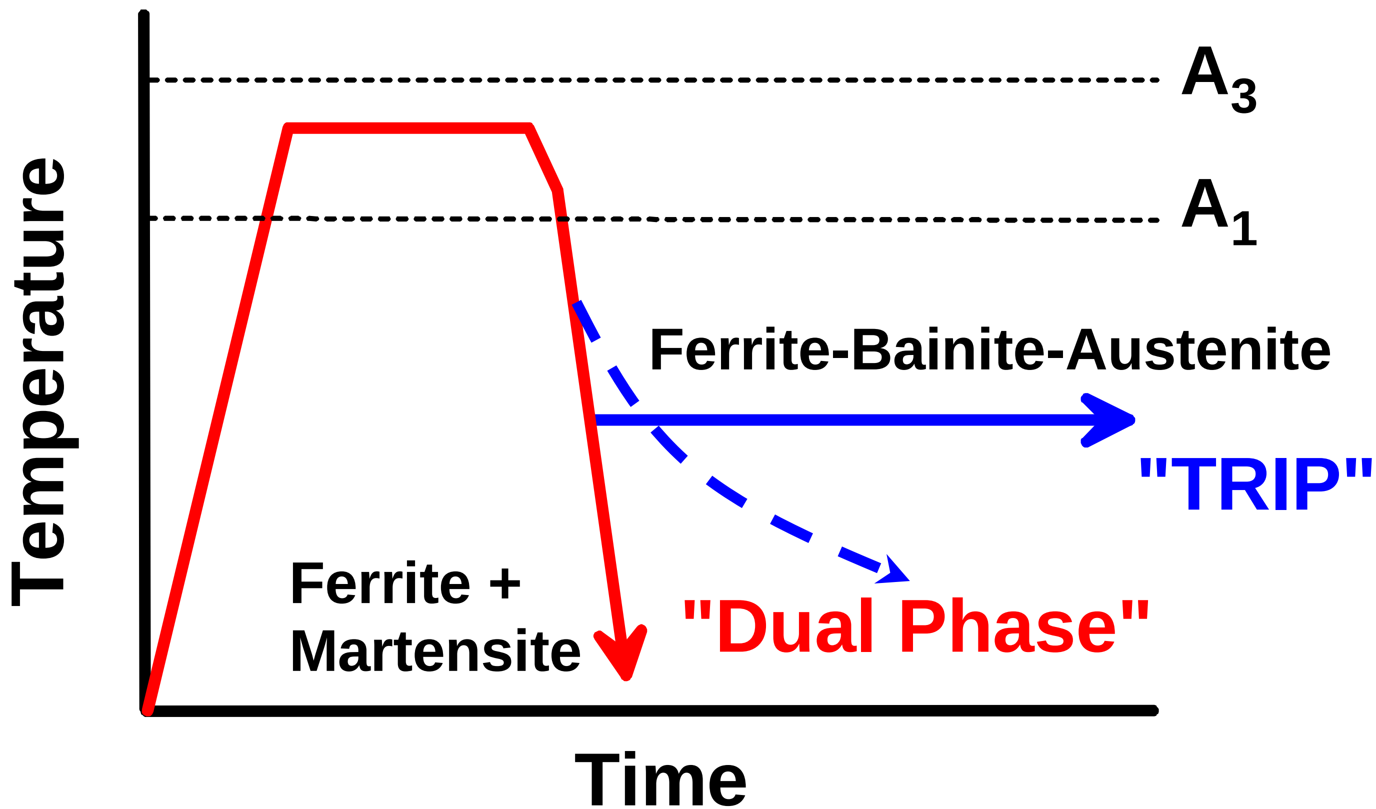
DP Steel Processing

- **Initially conceived based on “simple” routes for hot and cold rolled steels**
  - **Microstructures more complex than simply ferrite + martensite and contained retained austenite + ??**



TRIP Steel Processing

- **Isothermally transform or control cool**
- **Austenite in high strength matrix (e.g. fine grain ferrite, martensite, bainite, …)**

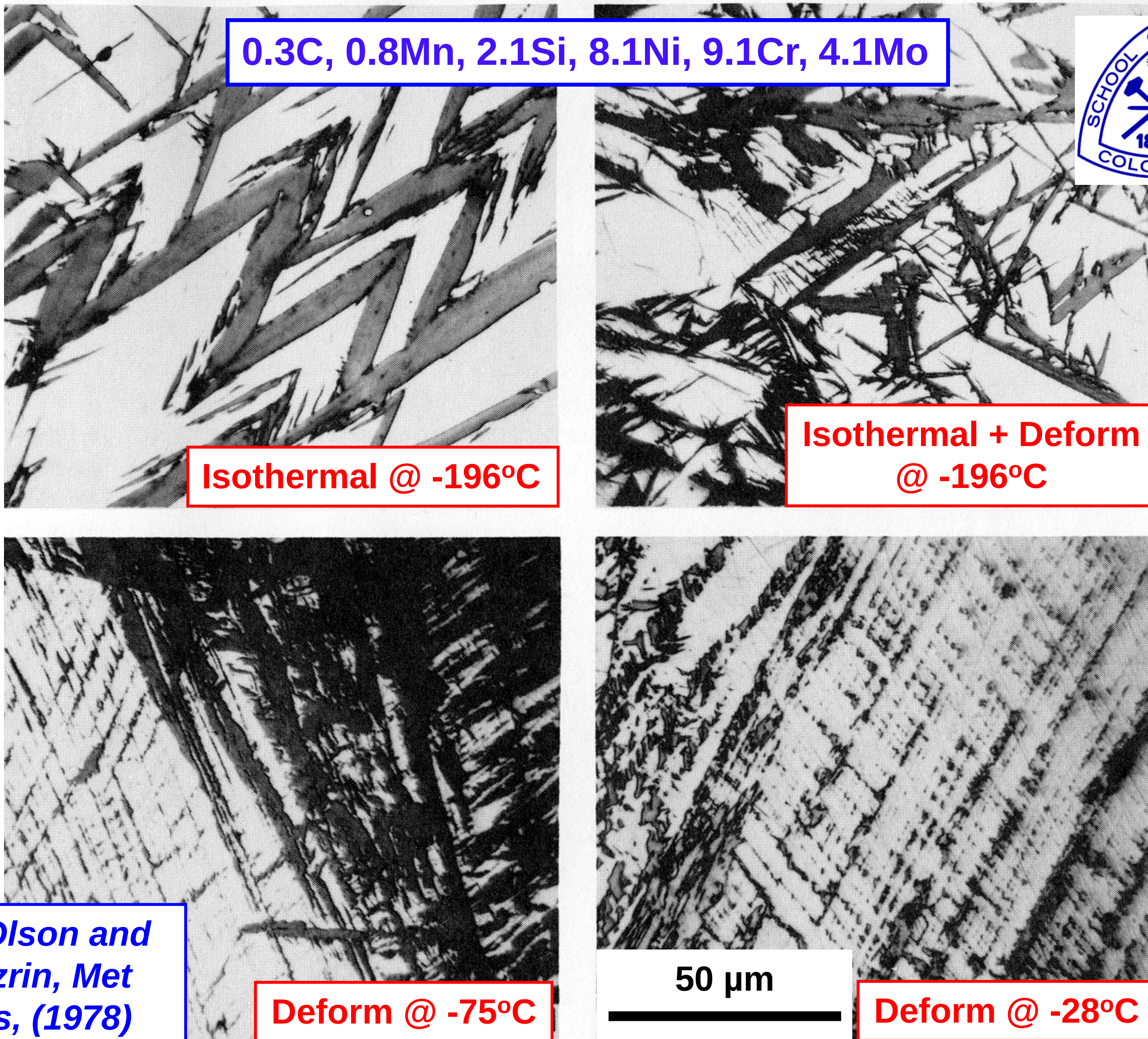


The "TRIP EFFECT"

- **"Stress-Assisted"**
  - **Heterogeneous martensite nucleation on the same sites responsible for transformation on cooling but assisted by stress – *(e.g. at prior austenite grain boundaries; lattice defects…)***
- **"Strain-Assisted" or "Strain-Induced"**
  - **Martensite nucleates on new sites created by plastic deformation -- *(e.g. at slip band intersections)***
- **Important consequences of the "TRIP Effect:"**
  - **Volume expansion: austenite to martensite**
  - **Strength of martensite > austenite**

G.B. Olson, in Deformation, Processing, and Structure, ed. by G. Krauss, ASM, Metals Park, OH, pp. 391-425, 1984.

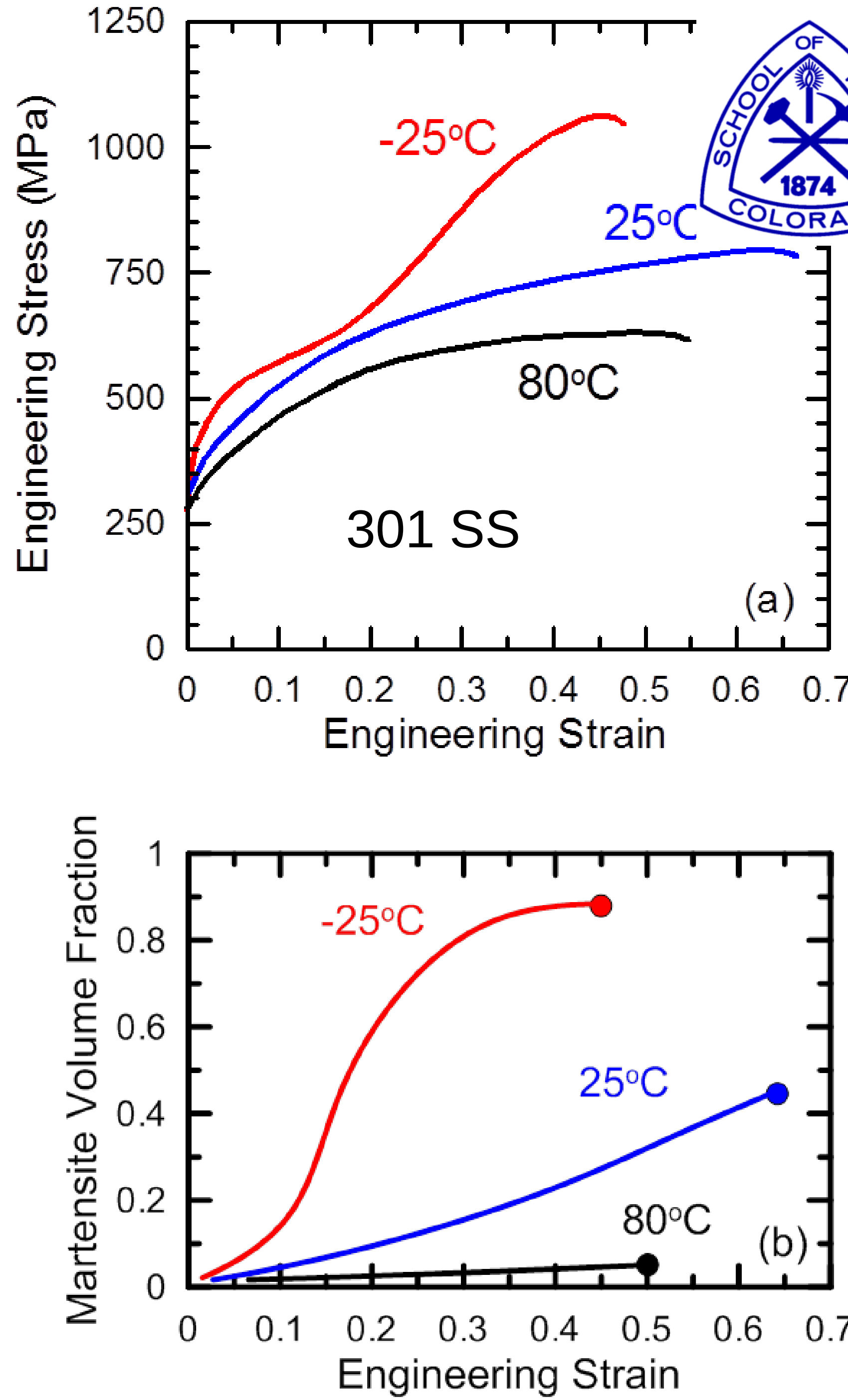
0.3C, 0.8Mn, 2.1Si, 8.1Ni, 9.1Cr, 4.1Mo



G.B. Olson and M. Azrin, Met Trans, (1978)

Strain-Assisted TRIP: Metastable Austenitic Stainless Steel

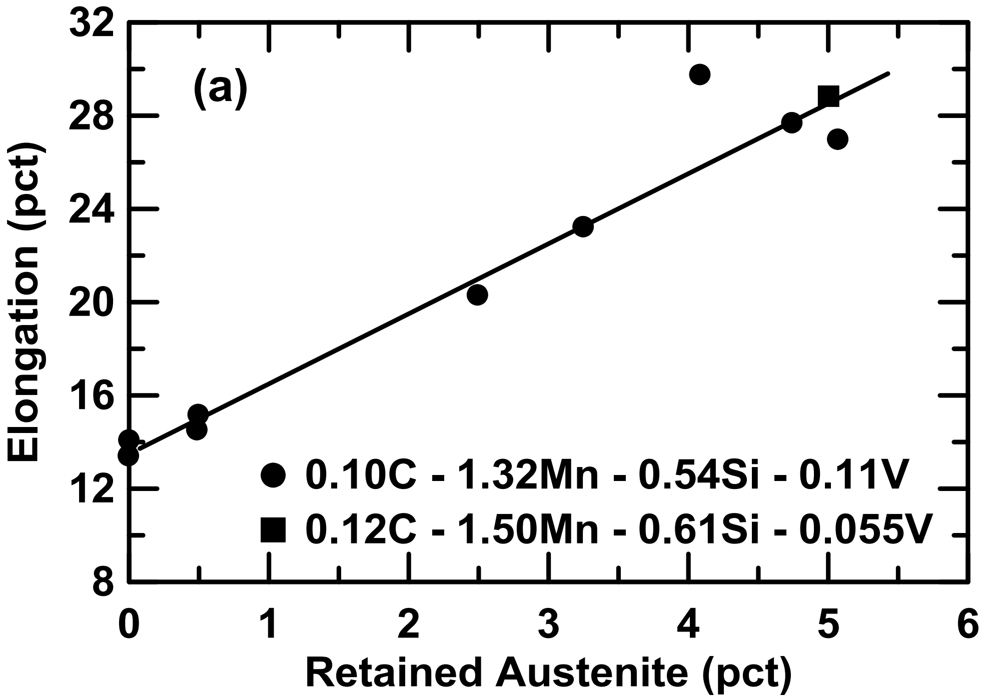
- **301 Stainless Steel**
- **0.096C-1.85Mn-17.33Cr-6.66Ni**
- **Isothermal tensile tests**
- **MVF measurements based on x-ray diffraction and magnetic inductance**
- **Peak ductility at intermediate temperature**

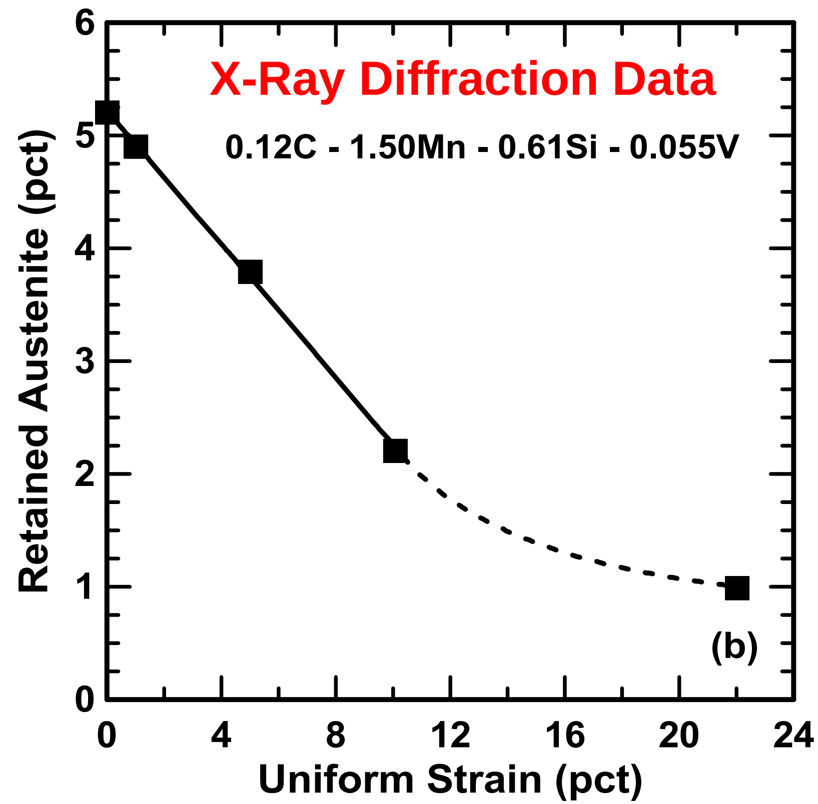


Adapted from S. Peterson, MS Thesis, ASPPRC, (1996)

…Lessons Learned from "Early" DP Steels

- **Processed as dual-phase steel**
- **Observed microstructures more complex than ferrite + martensite**





"…the ductility could be further improved by increasing the amount of retained austenite…" A. Marder (1977)

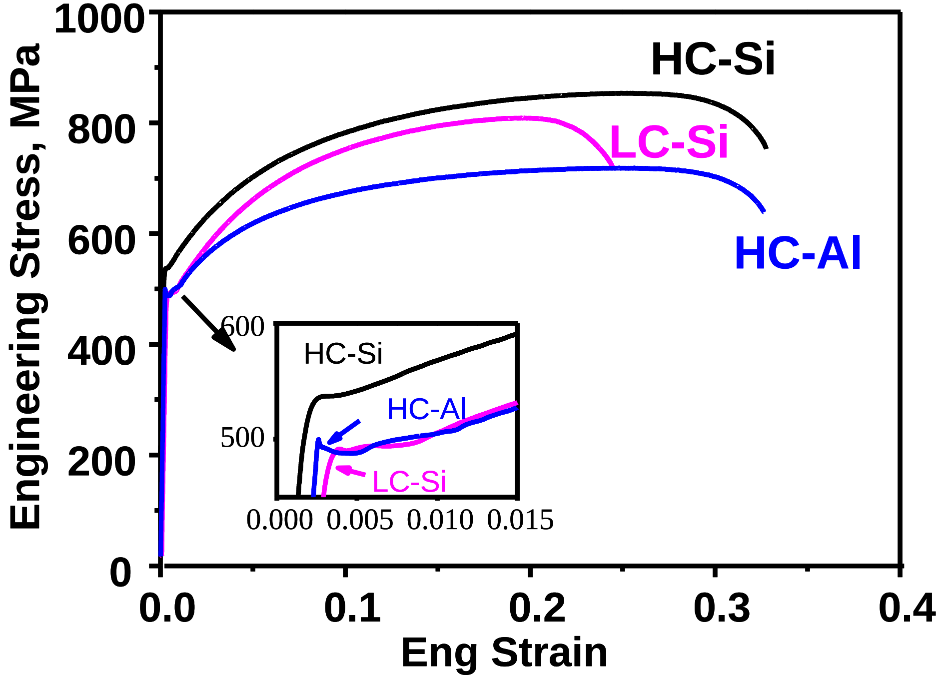
A. Marder, "Factors Affecting the Ductility of Dual-Phase Alloys," TMS, (1979)

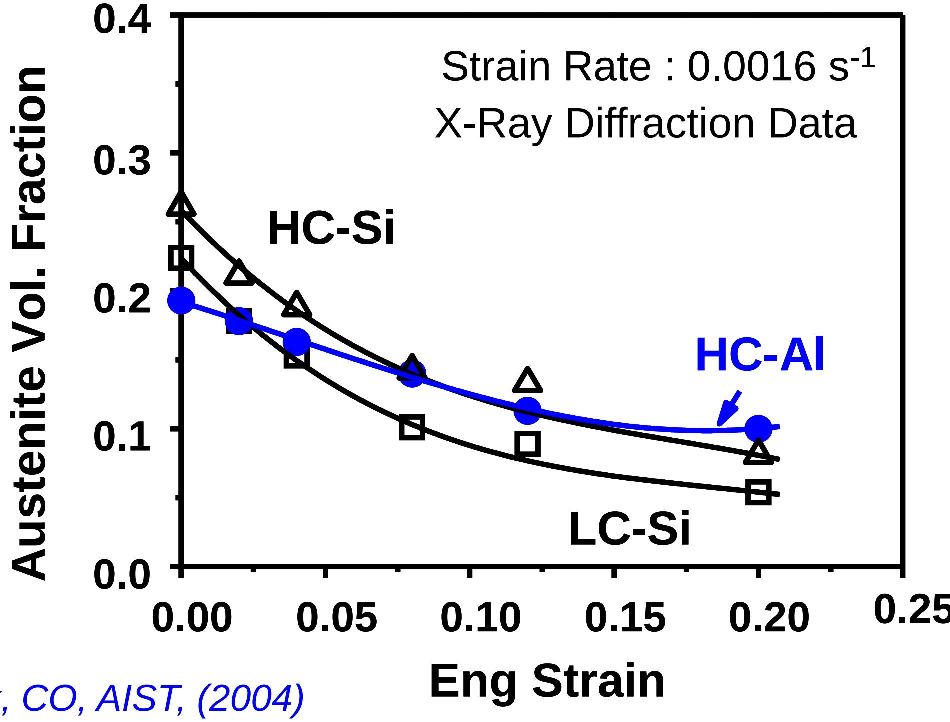
Austenite Stability: Alloying Effects

Steel	C	Mn	Si	Al
LC-Si	0.14	1.5	1.46	0.03
HC-Si	0.21	1.5	1.47	0.03
HC-Al	0.21	1.5	0.37	1.00

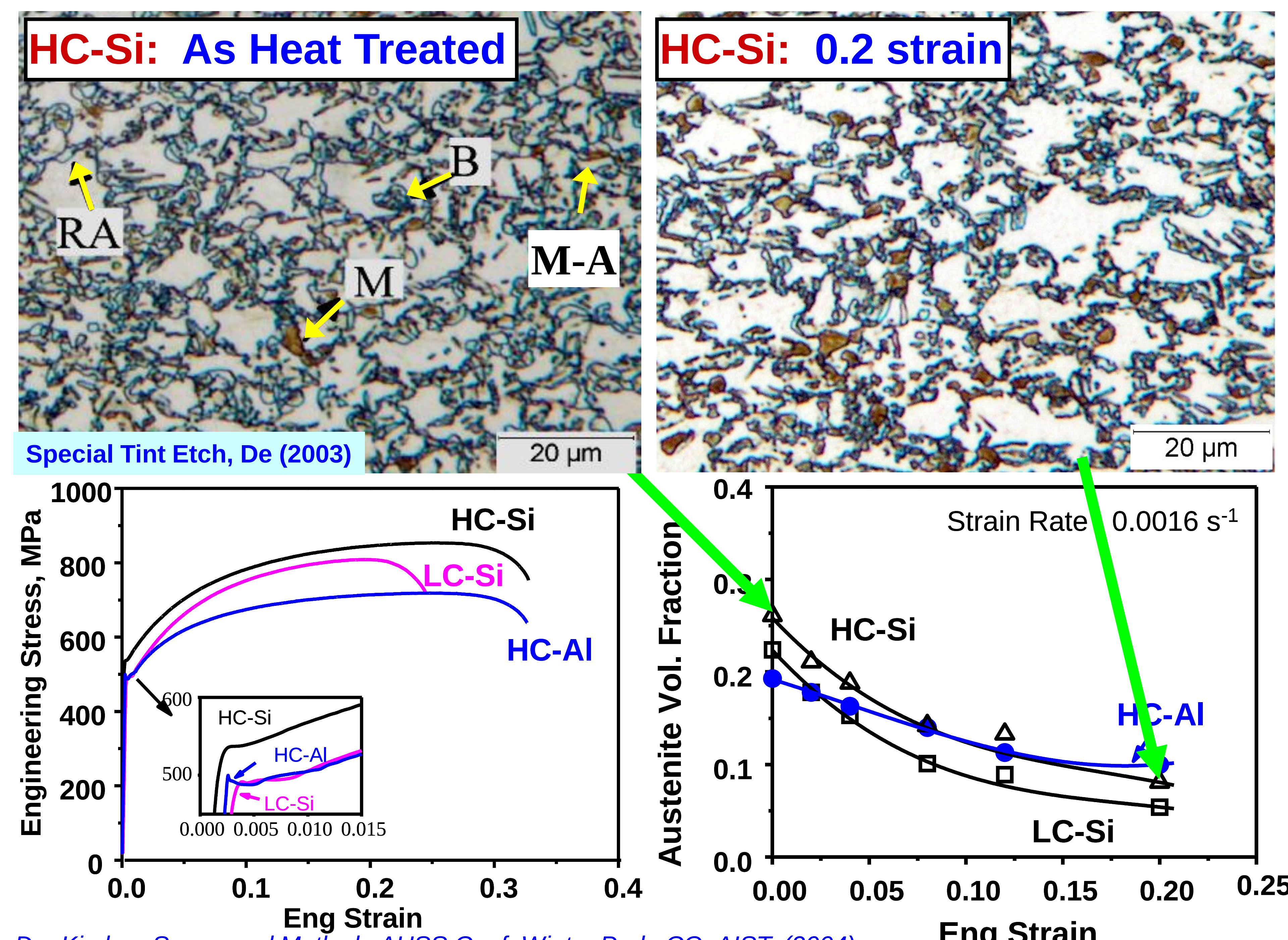
De, Kircher, Speer, and Matlock, AHSS Conf, Winter Park, CO, AIST, (2004)

(760 ^{o}C - 6 min) + (420 ^{o}C - 5 min) + WQ





De, Kircher, Speer, and Matlock, AHSS Conf, Winter Park, CO, AIST, (2004)

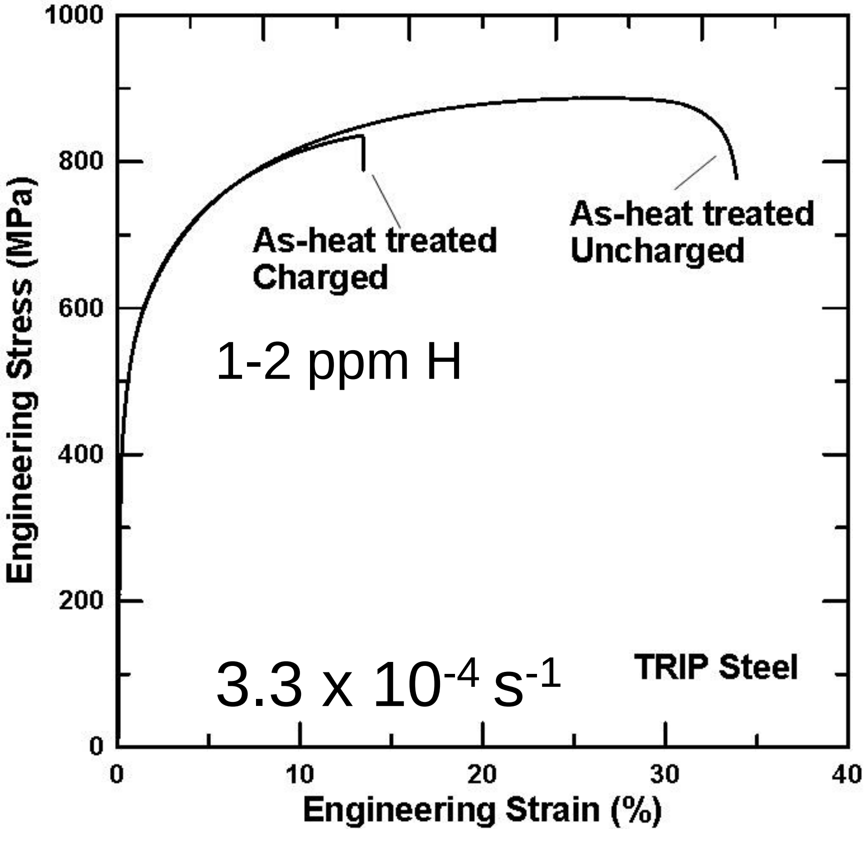


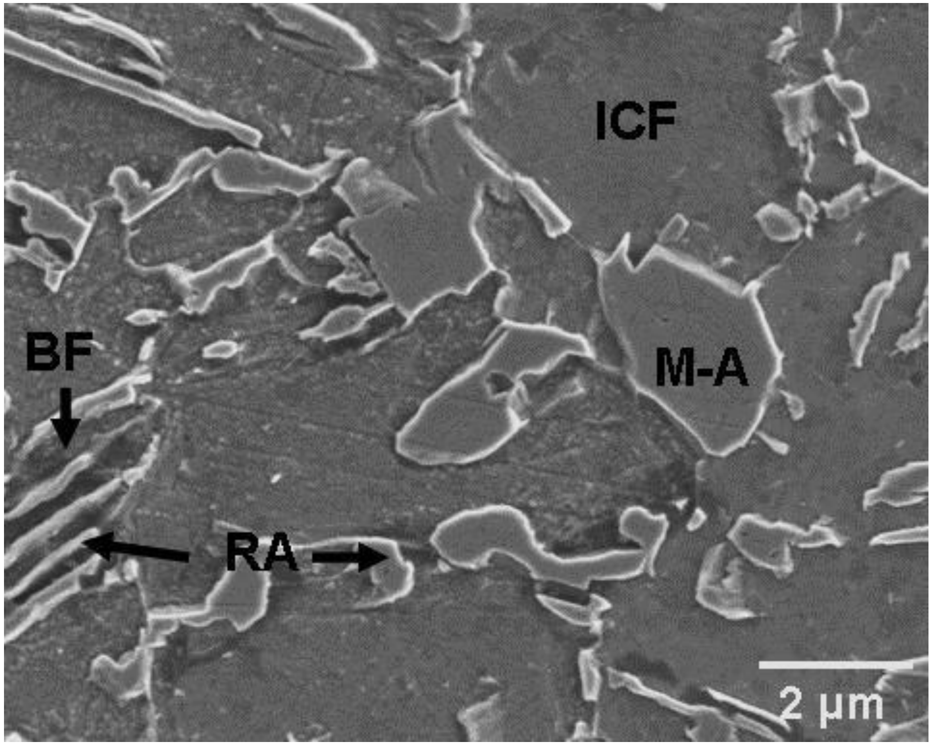
De, Kircher, Speer, and Matlock, AHSS Conf, Winter Park, CO, AIST, (2004)

The Hydrogen Microprint Technique to Reveal Hydrogen Distributions in TRIP Steels

Hydrogen in TRIP Steels

0.19C, 1.59Mn, 1.63Si (wt-%)





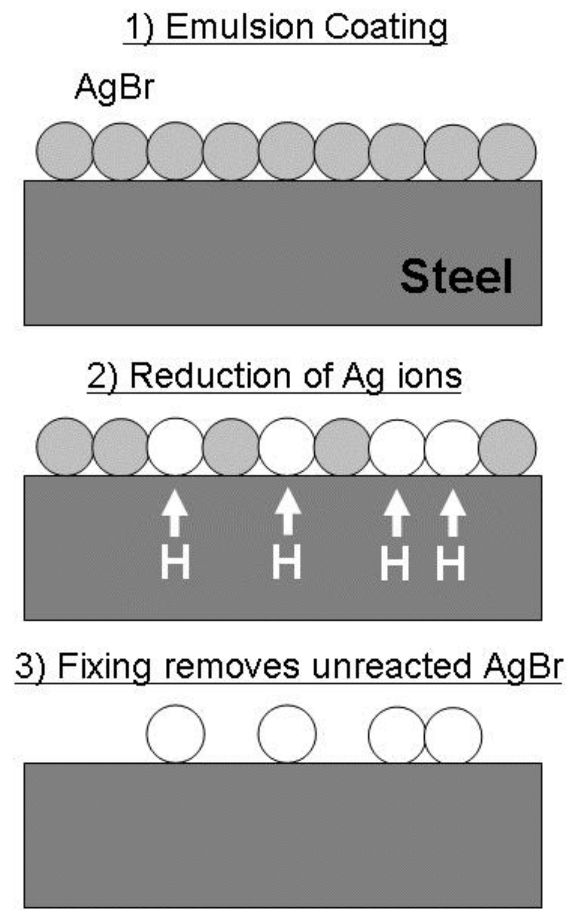
J. A. Ronevich, J. G. Speer, G. Krauss and D. K. Matlock, "Improvement of the Hydrogen Microprint Technique on AHSS Steels," Metallography, Microstructure, and Analysis, vol. 1, 2012, pp. 79-84.

Hydrogen Microprint Technique

Image-before-etch method … for fine microstructures

- Utilizes AgBr → Ag reduction with H
  - AgBr emulsion gel

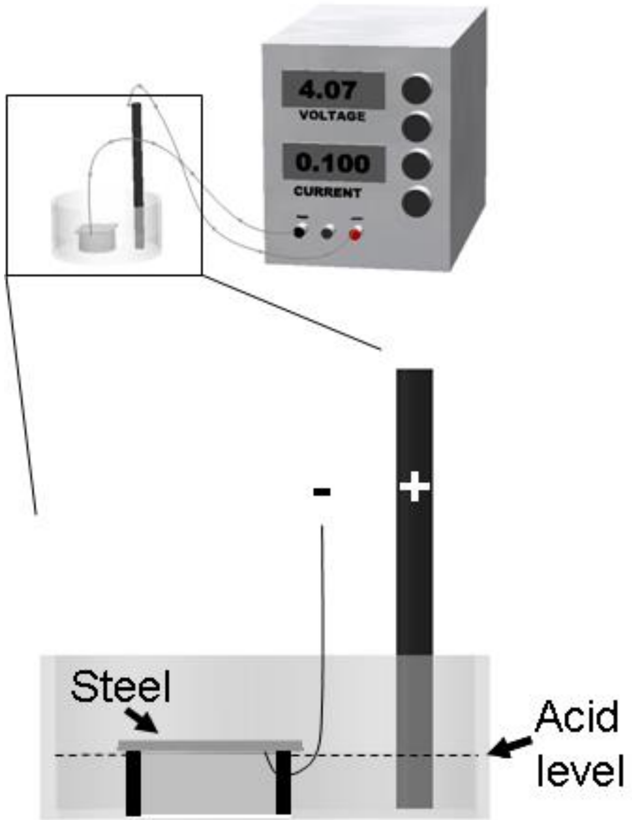
1) Hydrogen diffuses through steel (one way)
2) Reduces Ag^+ → Ag
3) Fixing removes excess AgBr
4) SEM analysis reveals Ag grains where H has exited steel
5) Observe microstructural features where H is located



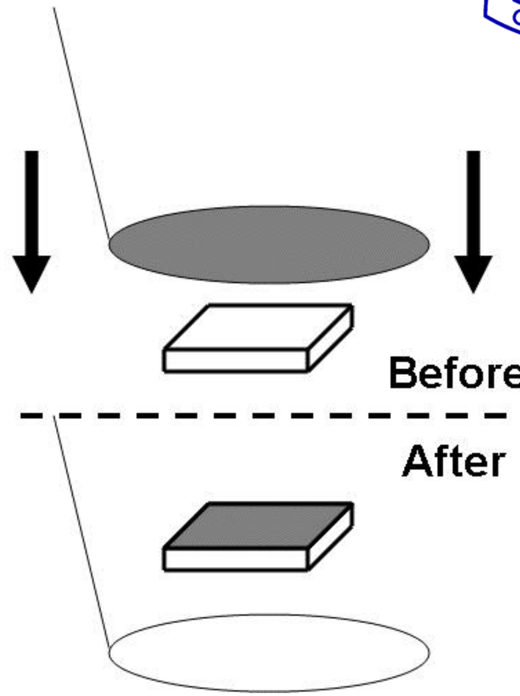
J. A. Ronevich, J. G. Speer, G. Krauss and D. K. Matlock, "Improvement of the Hydrogen Microprint Technique on AHSS Steels," Metallography, Microstructure, and Analysis, vol. 1, 2012, pp. 79-84.

Hydrogen Microprint Technique

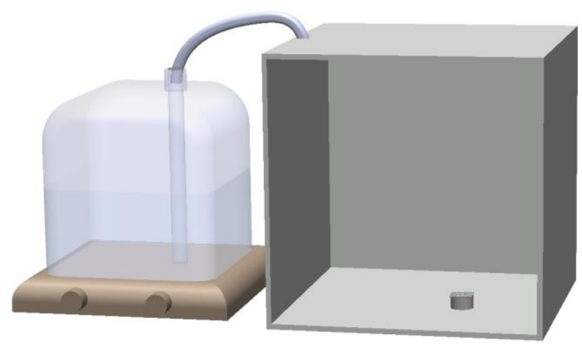
1)



2)

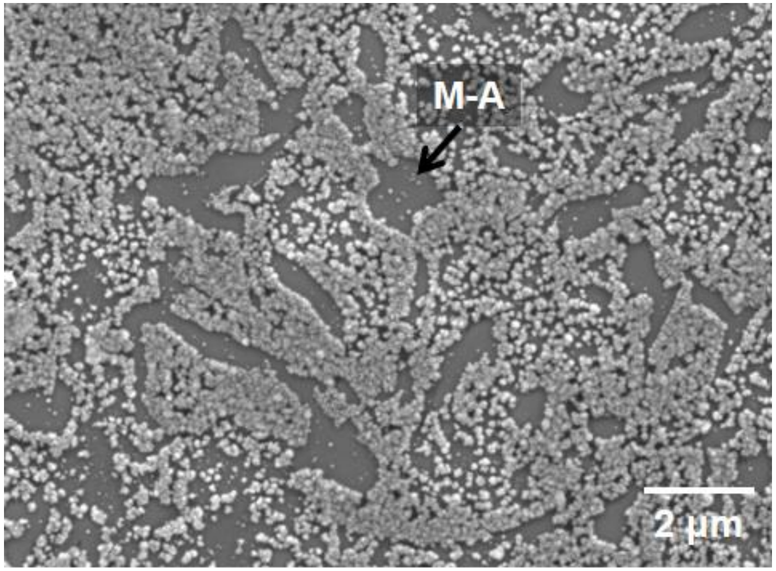


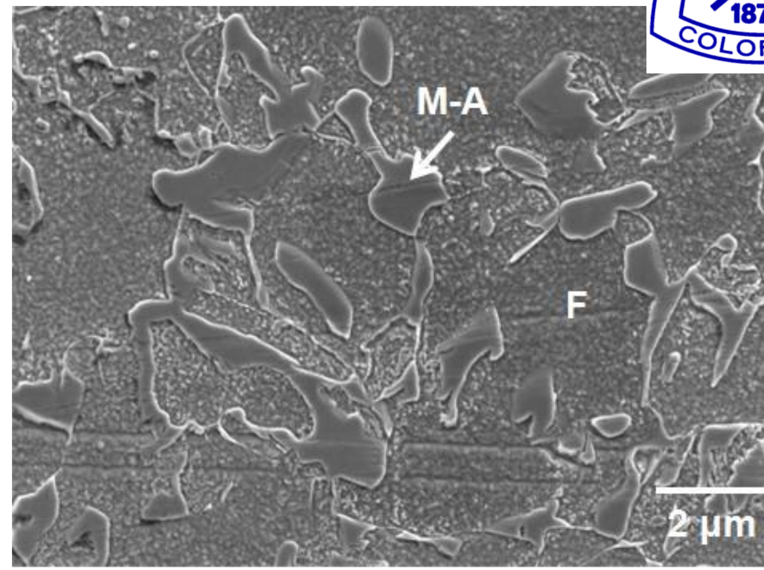
3)

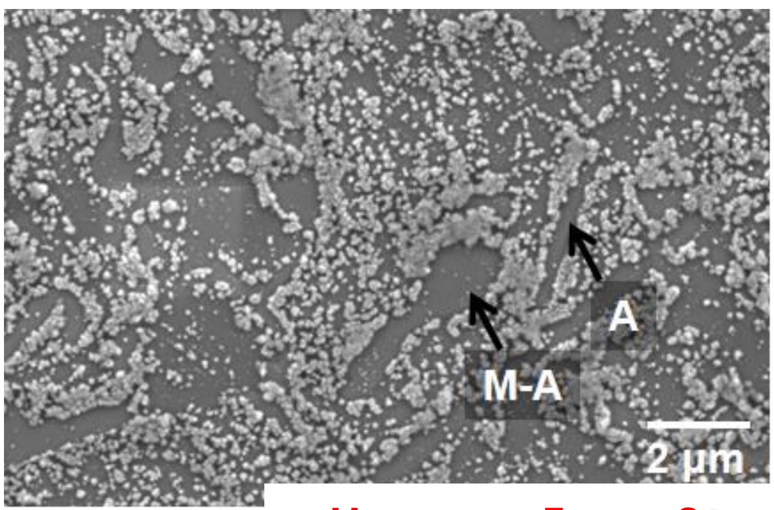


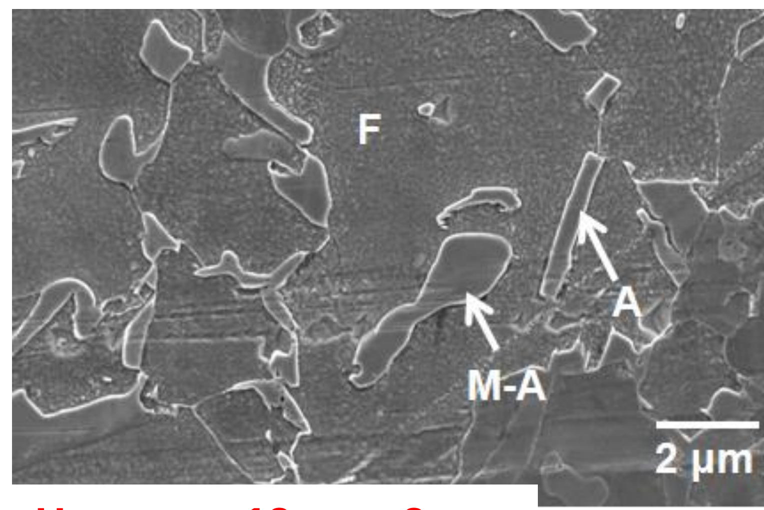
J. A. Ronevich, J. G. Speer, G. Krauss and D. K. Matlock, "Improvement of the Hydrogen Microprint Technique on AHSS Steels," Metallography, Microstructure, and Analysis, vol. 1, 2012, pp. 79-84.

Hydrogen Microprint Technique









$D^{H}_{\alpha} \sim 10^{-5}$ cm²/s **$D^{H}_{\gamma} \sim 10^{-12}$ cm²/s**

J. A. Ronevich, J. G. Speer, G. Krauss and D. K. Matlock, "Improvement of the Hydrogen Microprint Technique on AHSS Steels," Metallography, Microstructure, and Analysis, vol. 1, 2012, pp. 79-84.

TRIP Steel Summary

- **TRIP effect enhances strength and ductility**
  - **….requirements - austenite with controlled stability**
- **Assessment of stability**
  - **Color tint etching**
  - **Diffraction: x-ray and neutron**
  - **EBSD**
  - **Mechanical properties (stress-strain curve shape)**
  - **….**

Alloying and Processing Strategies to Produce 3rd Generation AHSS

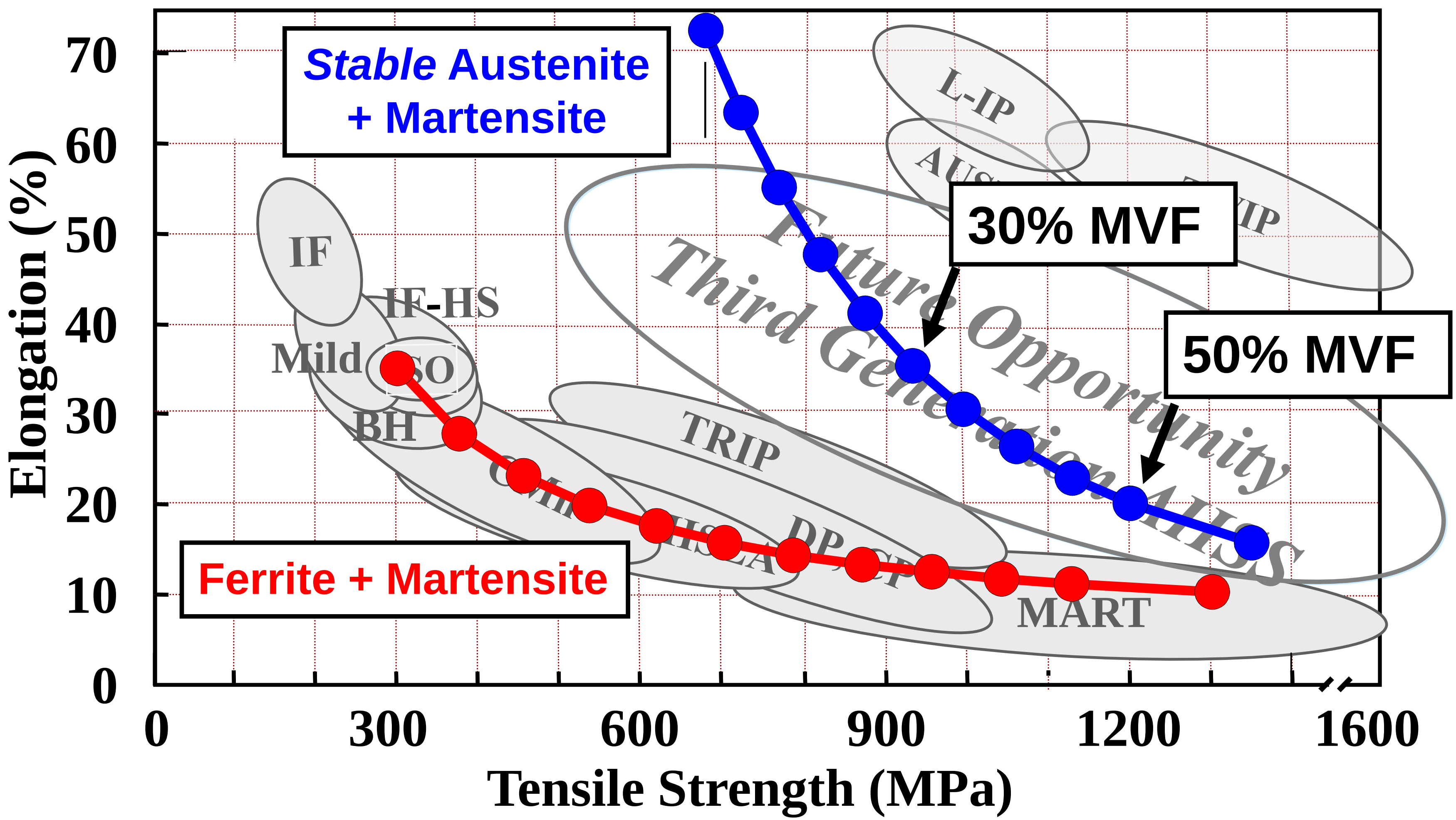
Example:

Quenched and Partitioned (Q&P) Steels

....a fundamentally new processing approach to produce high strength sheet steels with significant amounts of austenite....

Predictions Based on Composite Modeling

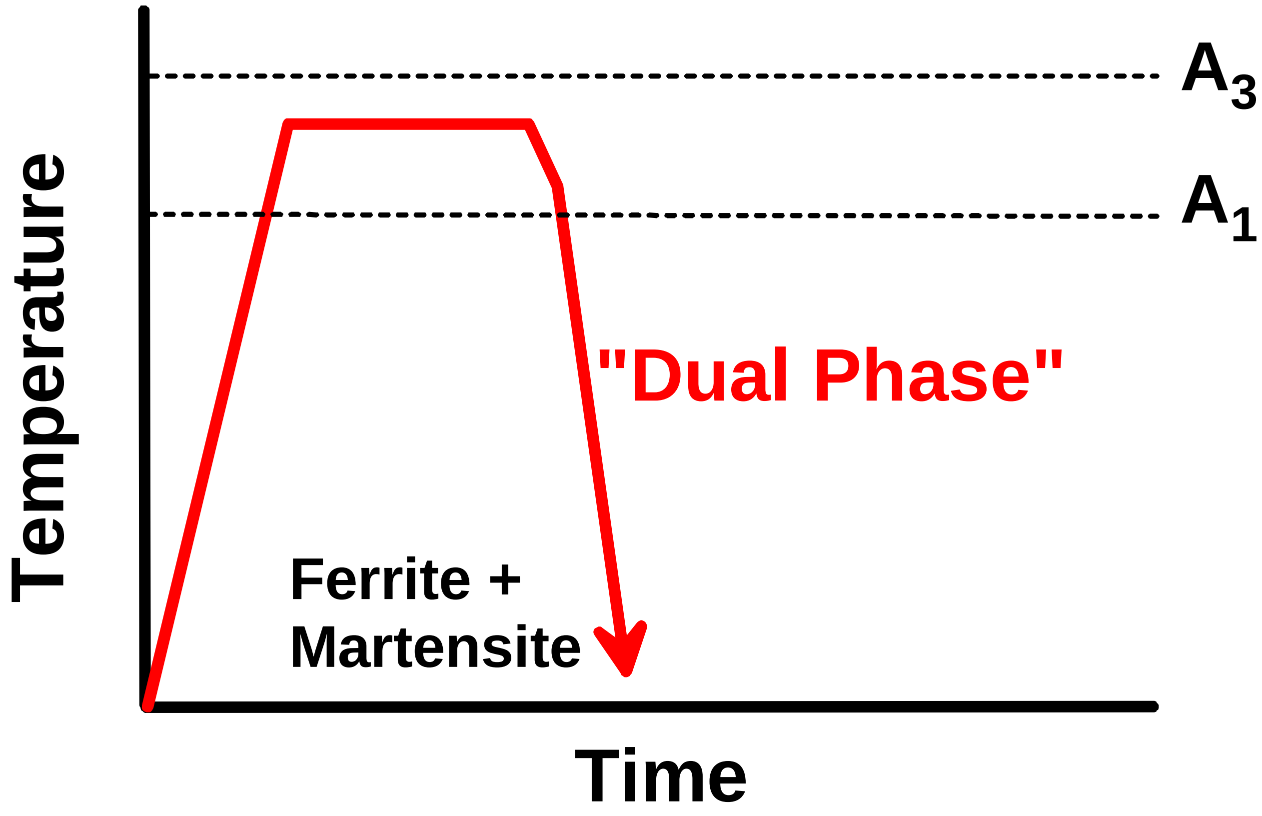
Austenite is a key enabler for new AHSS



D.K. Matlock and J.G. Speer, "Design Considerations for the Next Generation of Advanced High Strength Sheet Steels," Proceedings of the 3rd Int. Conf. on Structural Steels, ed. by H.C. Lee, The Korean Institute of Metals and Materials, Seoul, Korea, 2006, pp. 774-781.

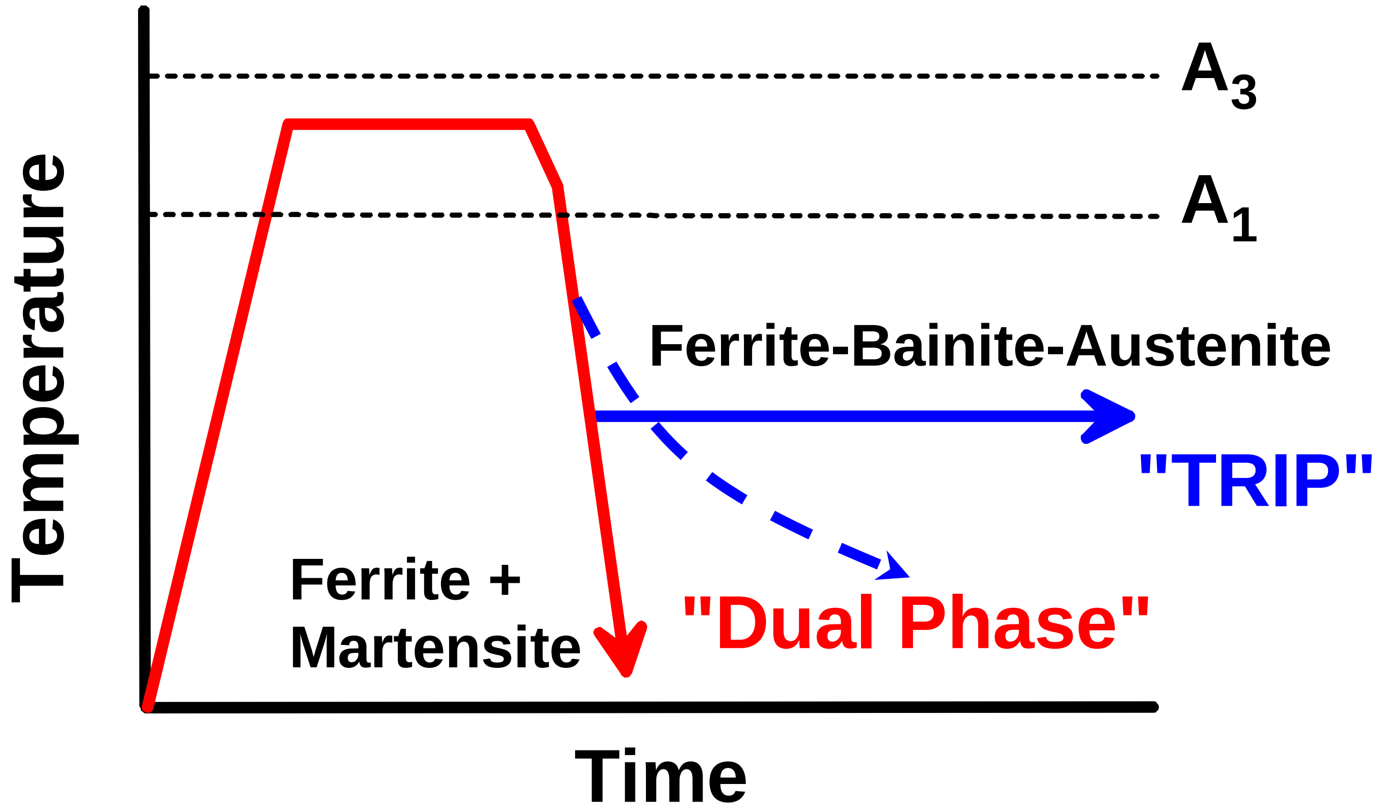
DP Steel Processing

- **Initially conceived based on "simple" routes for hot and cold rolled steels**
  - **Microstructures may be more complex than simply ferrite + martensite**



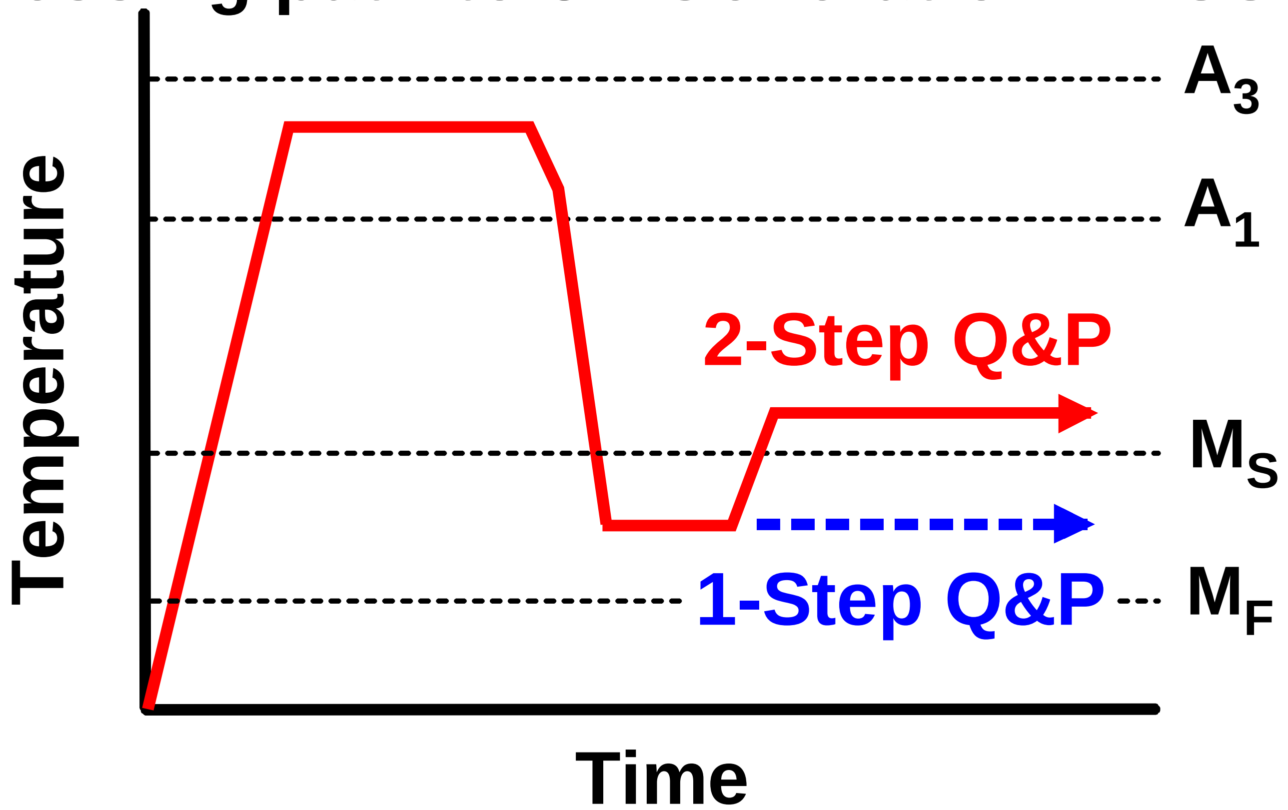
TRIP Steel Processing

- **Isothermally transform or control cool**
- **Austenite in high strength matrix (e.g. fine grain ferrite, martensite, bainite, …)**



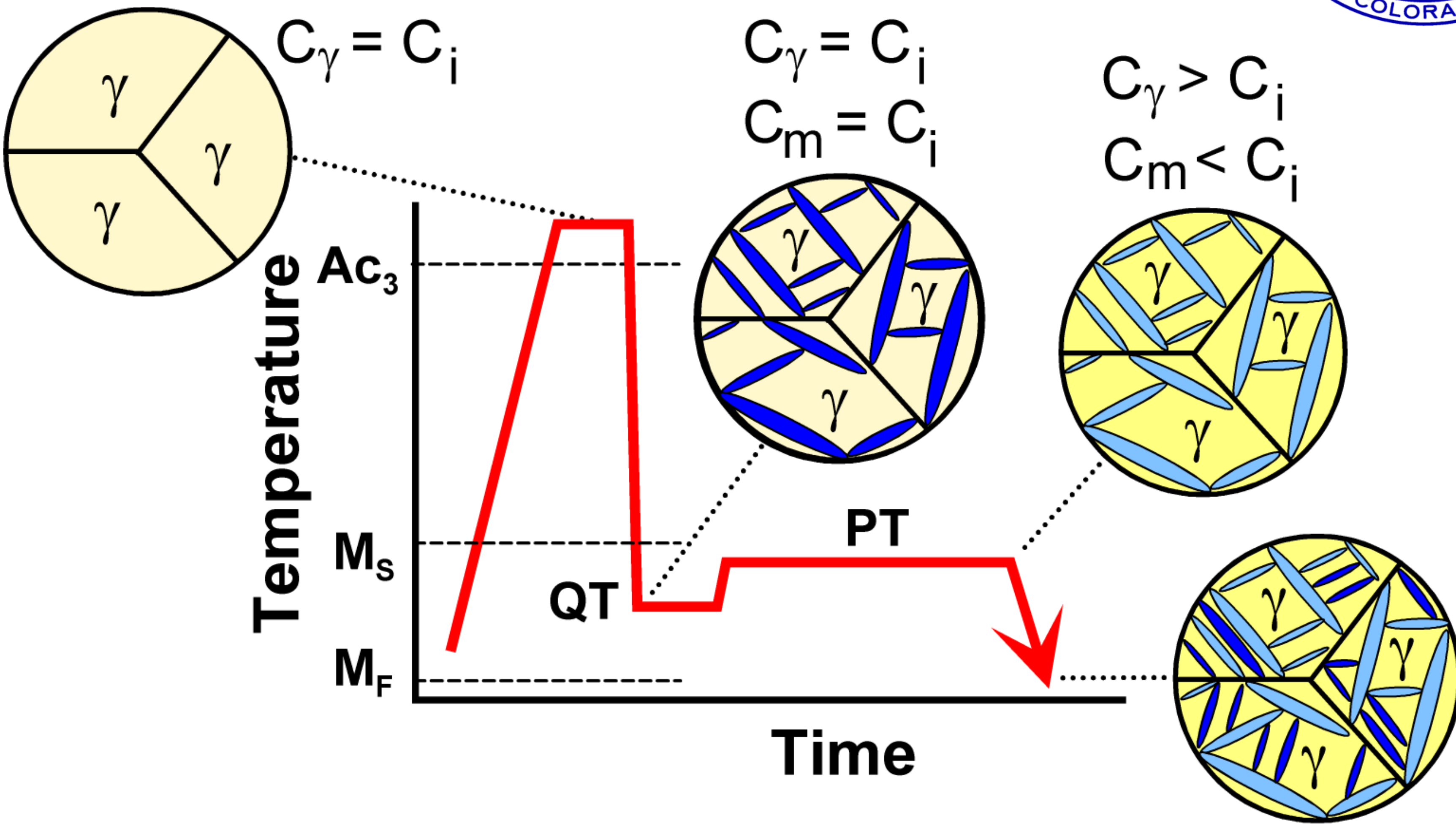
Interrupted Cooling: The "Q&P" Process

- **Novel method to produce high strength material with significant amounts of retained austenite**
- **Processing path to 3rd Generation AHSS**



J.G. Speer, et al. ASPPRC, Colorado School of Mines, (2003 - ongoing)

Q & P Processing - Overview



Final Austenite Amount Modified by Additional Martensite Formation on Cooling to Room Temperature

J.G. Speer, et al. ASPPRC, Colorado School of Mines, (2003 - ongoing)

Q&P Selection Methodology

- **Evaluate austenite stability**

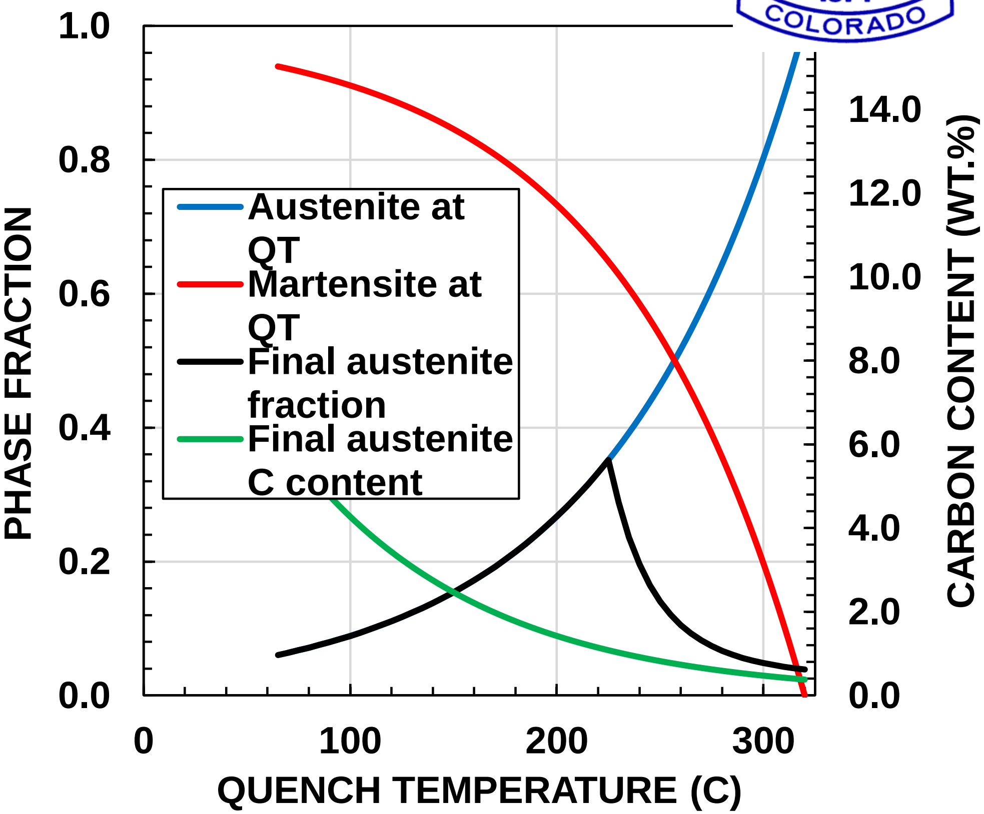
  $M_s = 539 - 423C - 30.4Mn - 7.5Si + 30Al$

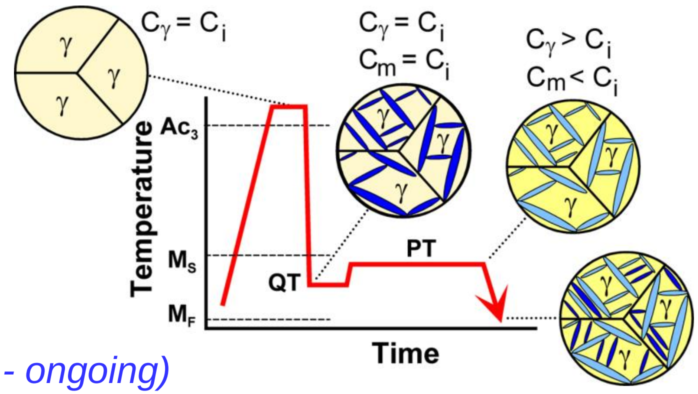
- **Predict fraction transformed on cooling**
  (Koistinen-Marburger equation)

  $$f_M = 1 - e^{-0.011(Ms - T)}$$

Assumptions:

- **No** austenite decomposition
- Assumes ***all*** C partitions from martensite to austenite
- **No** carbide formation

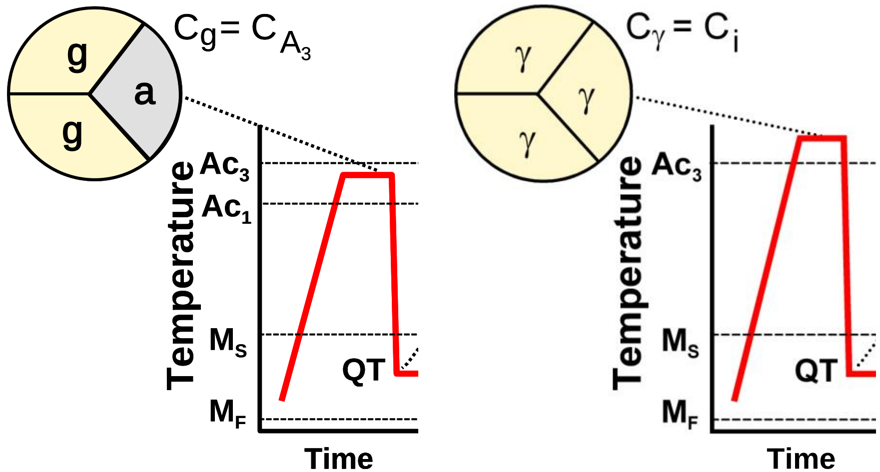




J.G. Speer, et al. ASPPRC, Colorado School of Mines, (2003 - ongoing)

Q&P: Unique Designed Microstructures

(1) Annealing temperature controls amount of initial ferrite



(2) Quench temperature (QT): controls amount of initial martensite

(3) Alloying controls carbide stability – e.g. add Si

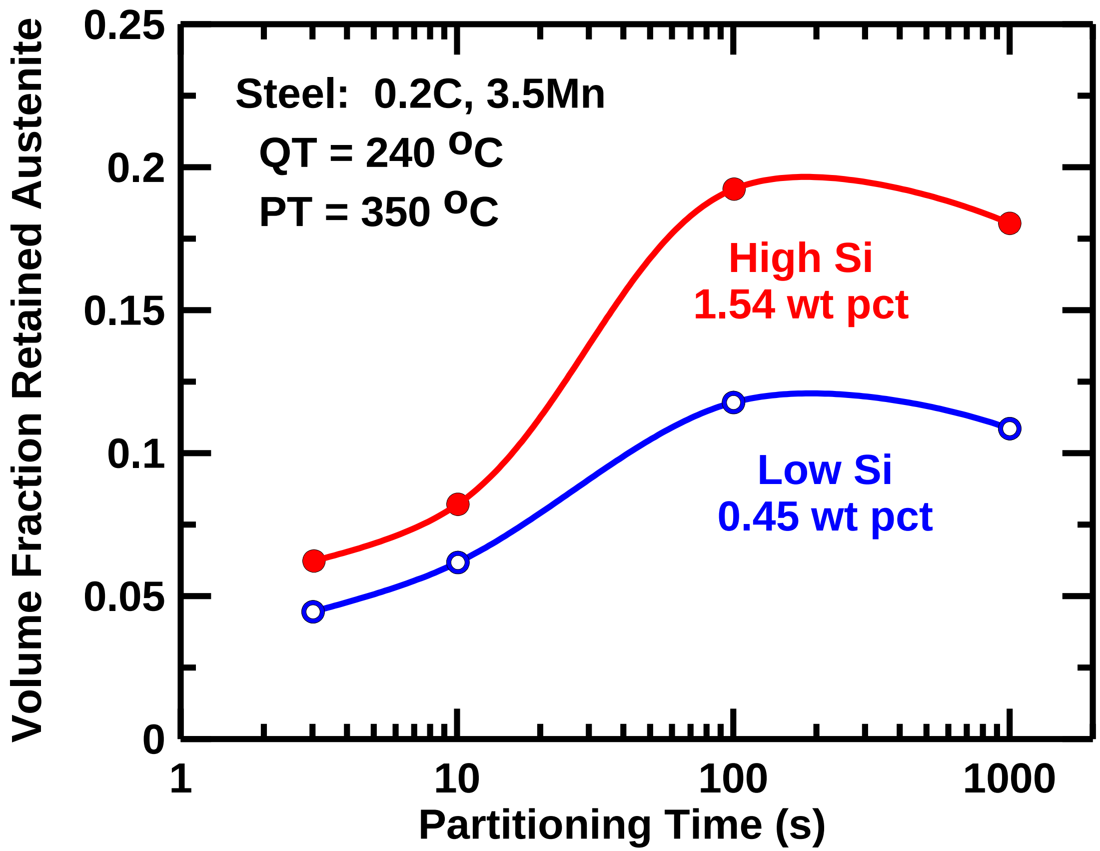
(4) Partitioning temperature and time: control austenite stability

(5) Result: unique combinations of martensite + austenite + ferrite + ..other..

J.G. Speer, D.K. Matlock, B.C. De Cooman, and J.G. Schroth, "Carbon Partitioning Into Austenite after Martensite Transformation," *Acta Materialia*, vol. 51, 2003, pp. 2611-2622.

"Q&P" Alloying and Processing

- **Alloying: e.g. Mn Si Al C Mo**
- **… to control critical temperatures: M_s M_f …..**
- **… to suppress cementite formation**

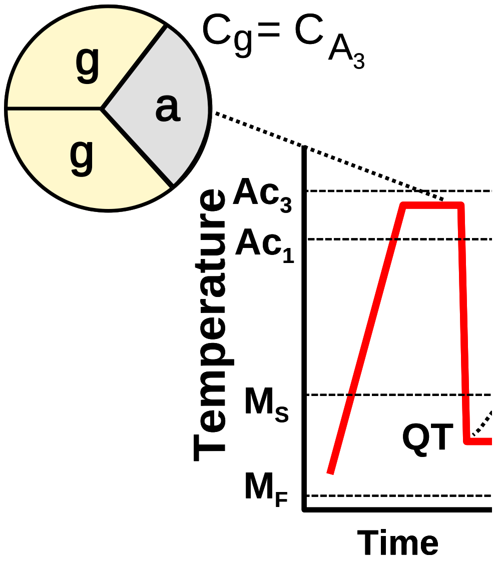


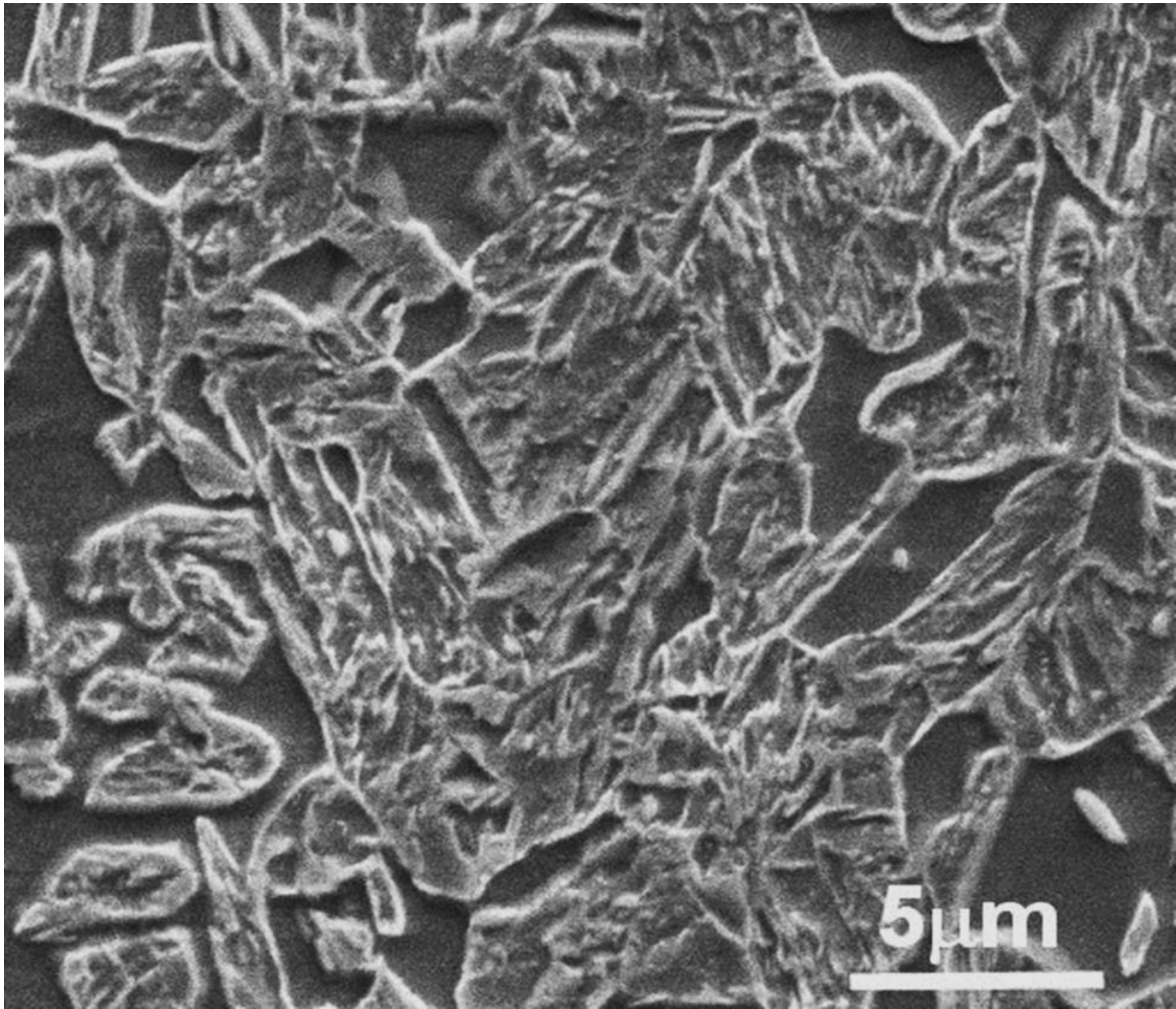
M.J. Santofimia, et al., "Experimental Investigation of Q&P on Two Low-Carbon Steels with Different Silicon Contents," Proc. Int. Conf. on New Developments in AHSS, ed. by J.G. Speer, B. Nelson, and R. Pradhan, AIST, Warrendale, PA, 2008.

Example Q&P Microstructure:

- **Intercritically annealed: α_{IC}=25%**
- **0.19C, 1.59Mn, 1.63Si (wt-%)**
- **QT = 200 ^{o}C, 10 s**
- **PT = 400 ^{o}C, 10s**
- **Austenite = 11.4%**

SEM Micrograph: 2% nital etch

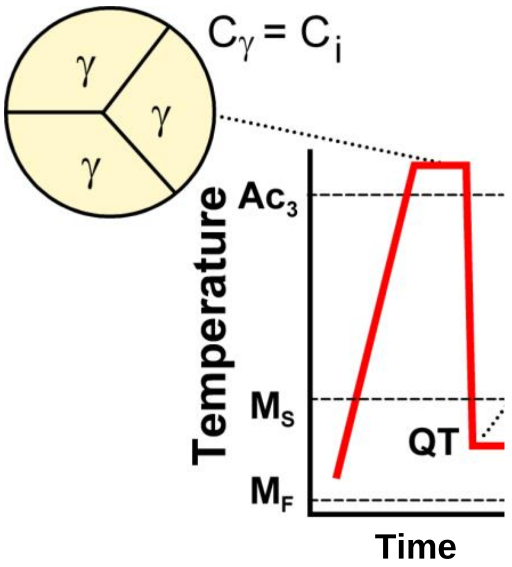




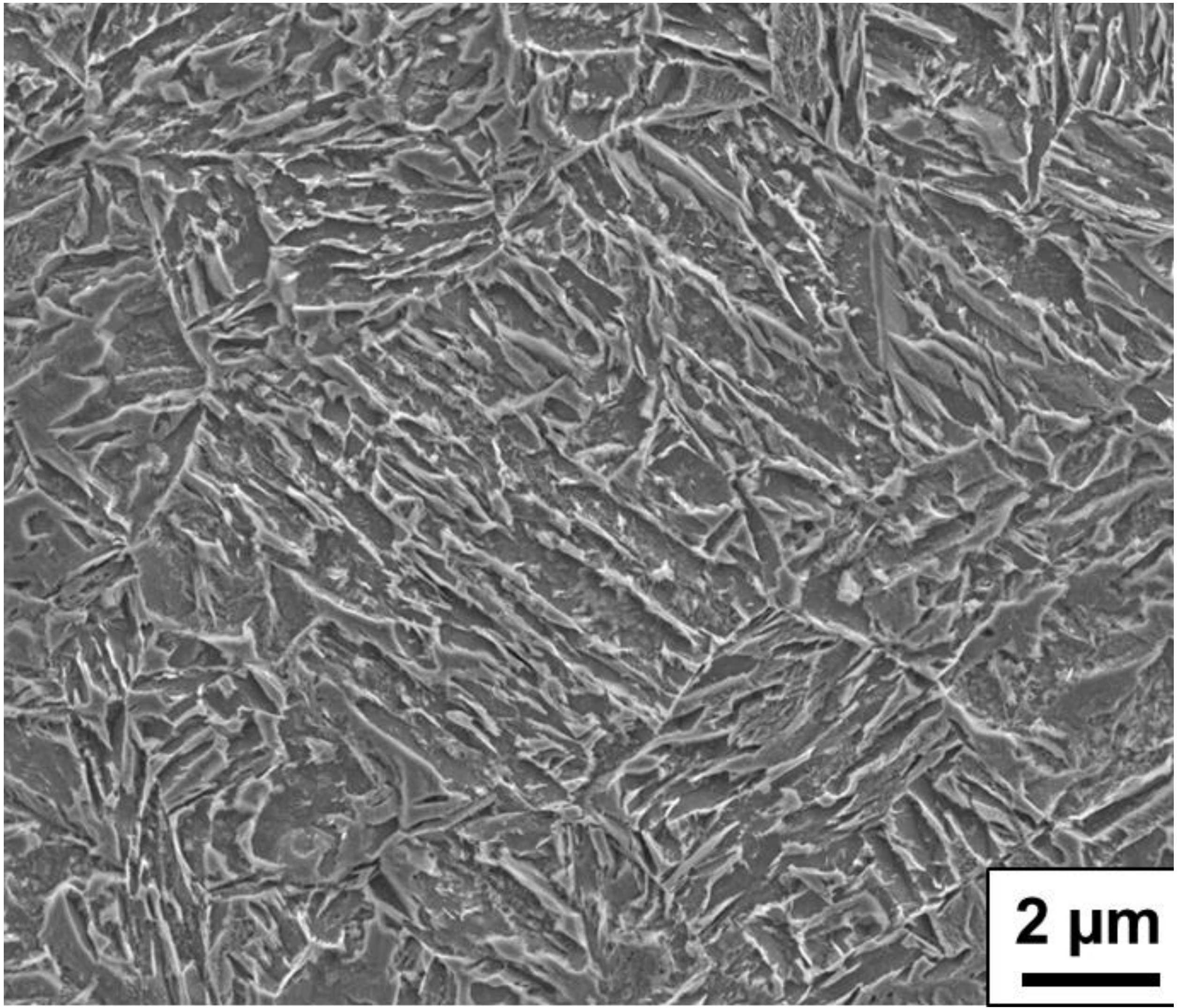
A. Clarke, PhD Thesis, ASPPRC/CSM, 2006.

Example Q&P Microstructure:

- **Annealed as austenite: $\alpha_{IC} = 0\%$**
- **0.3C, 3Mn, 1.6Si (wt-%)**
- **QT = 200 °C**
- **PT = 400 °C, 30 s**
- **Austenite = 10.9%**

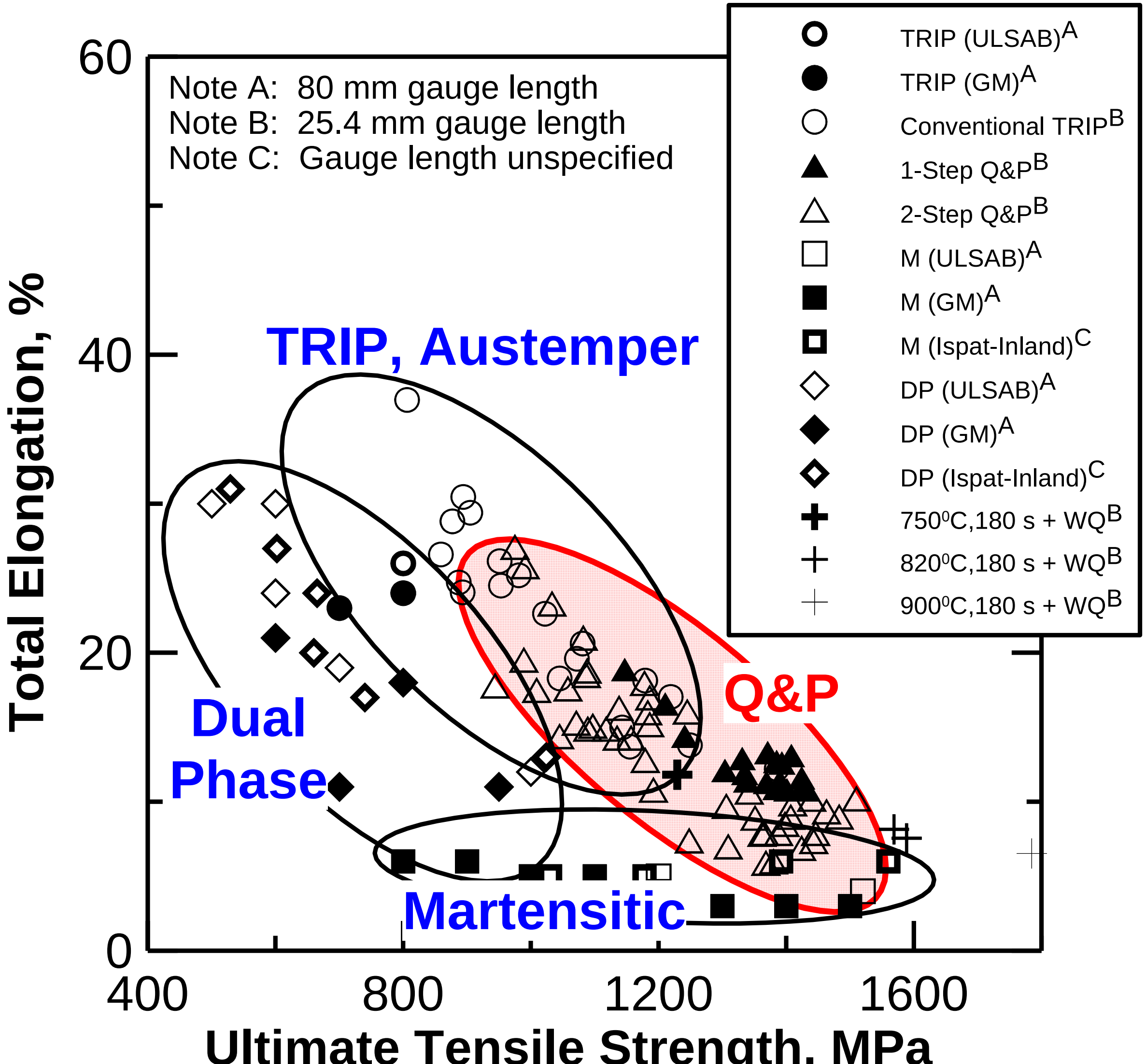


SEM Micrograph: 2% nital etch



E. De Moor, J.G. Speer, D.K. Matlock, J.H. Kwak, and S.B. Lee, *ISIJ Intl.* Vol. 51, no. 1, 2011, pp.137-144.

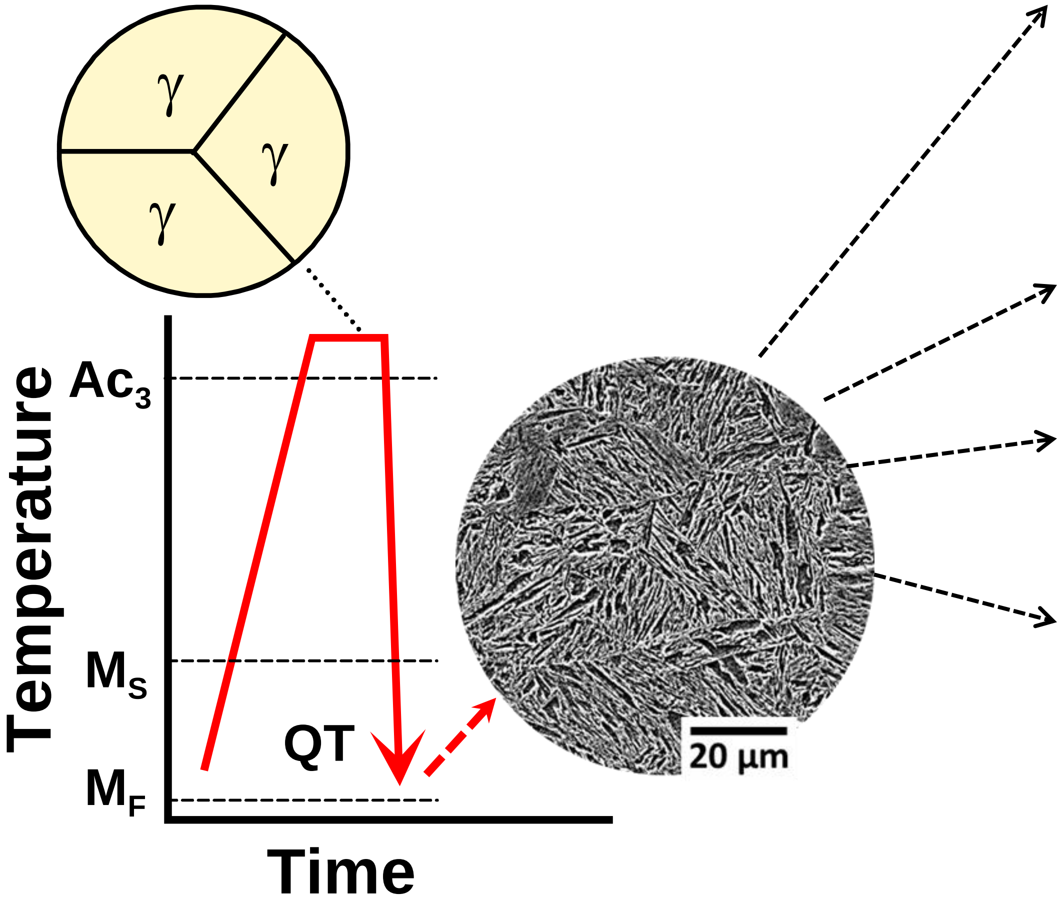
Properties of Q&P
Summary and Comparison with AHSS



Clarke, Speer, et al. ASPPRC, Colorado School of Mines (2006)

Assessment of Q&P Microstructures

Competing Reactions:



Carbon Partitioning

Compositions change:
Austenite and martensite

Martensite Tempering

Stage 1: **Formation of transition carbides, epsilon carbide**

Stage 2: **Transformation of retained austenite to ferrite and cementite**

Stage 3: **Replacement of transition carbides and martensite by cementite and ferrite**

Micrograph: 0.2 C, 1.2 Mn, Direct Quenched:
N. Muckelroy, MS Thesis, ASPPRC/CSM, 2010.

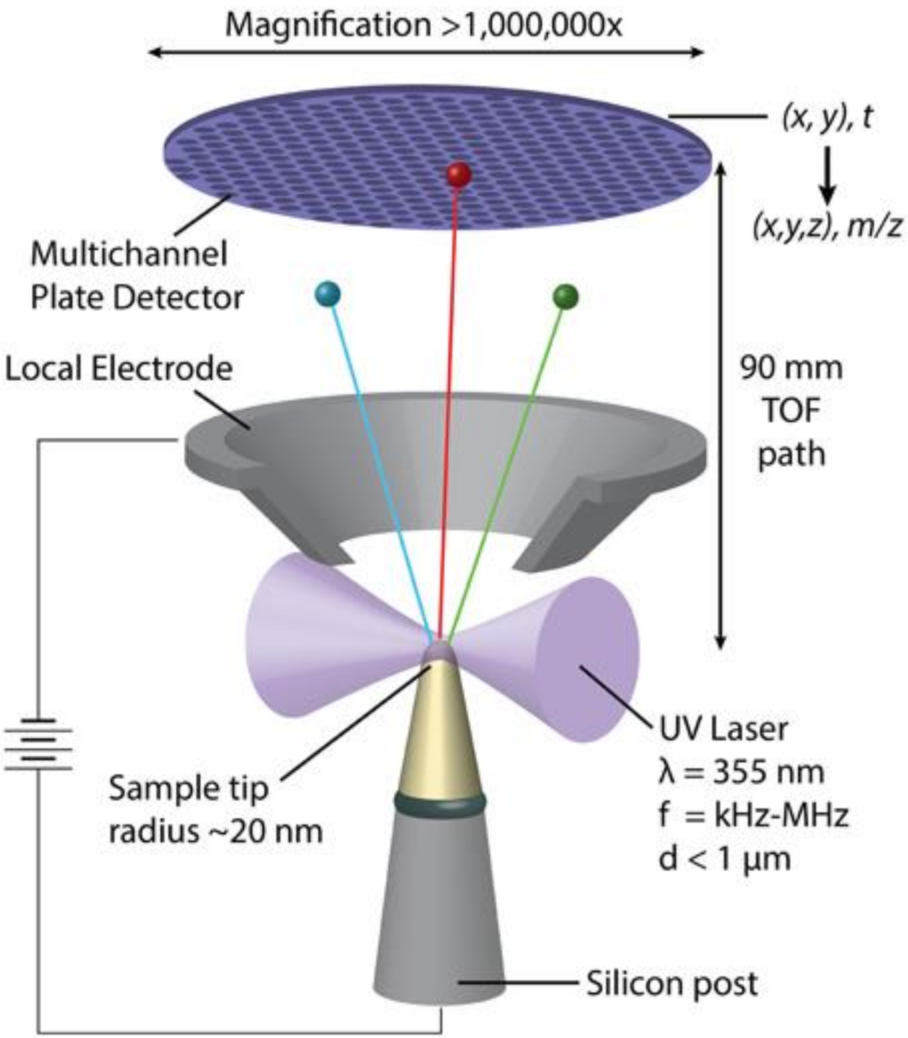
Application of Advanced Analytical Techniques to Assess Q&P and AHSS Microstructures

Atom Probe

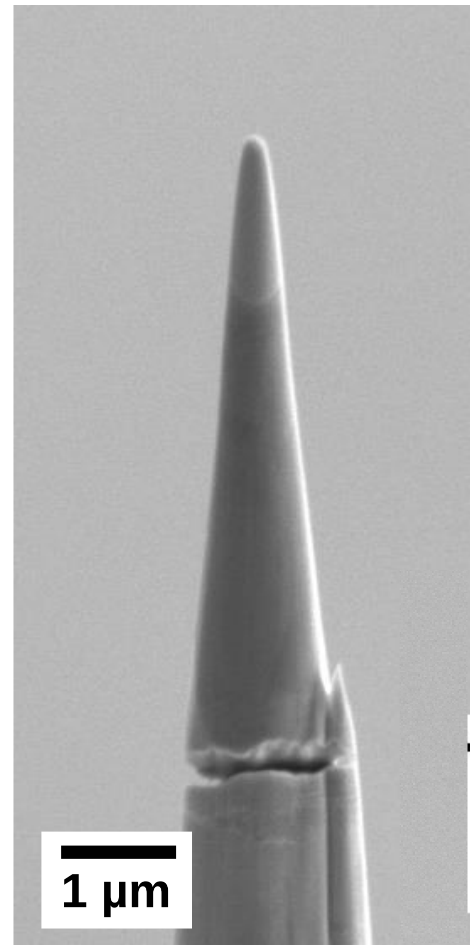
Atom Probe Tomography

- **3D imaging and chemical composition measurements at the atomic scale**
- **Assess carbon (and nitrogen) partitioning and local compositions**
  - **DP Steel - 0.10 C, 2.0 Mn, 0.3 Cr, 0.3 Mo (wt-%)**
  - **Q&P Steels**
    - **0.19 C, 1.59 Mn, 1.63 Si (wt-%) -- M_s = 399 ^{o}C**
    - **Special alloys designed for QT = RT**
      - **0.32 C, 1.91 Si, 13.95 Ni, 0.25 Mo (wt-%)**
        - **M_s = 258 ^{o}C**
      - **0.27 C, 8.31 Mn, 1.94 Si, 0.24 Mo (wt-%)**
        - **M_s = 142 ^{o}C**

Atom Probe Tomography

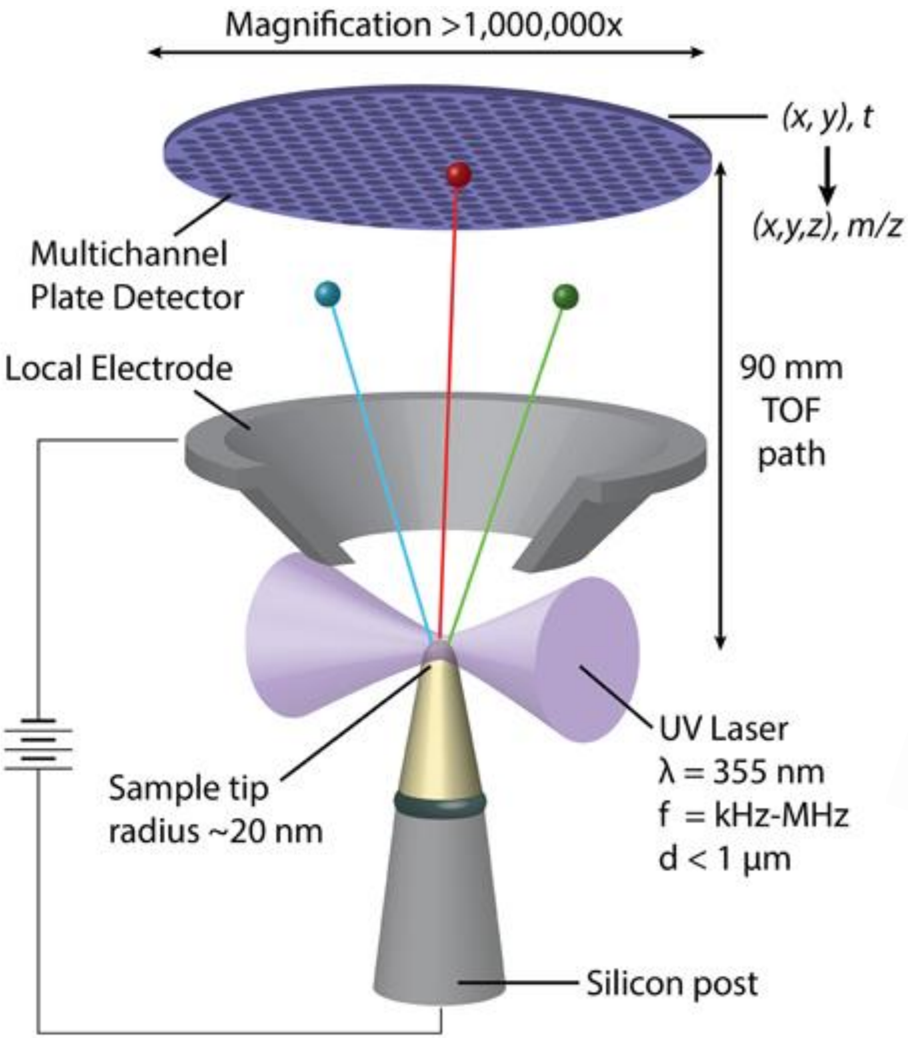


L.M. Gordon and D. Joester,
Front. Physiol. Vol. 6, 2015, p.:57.



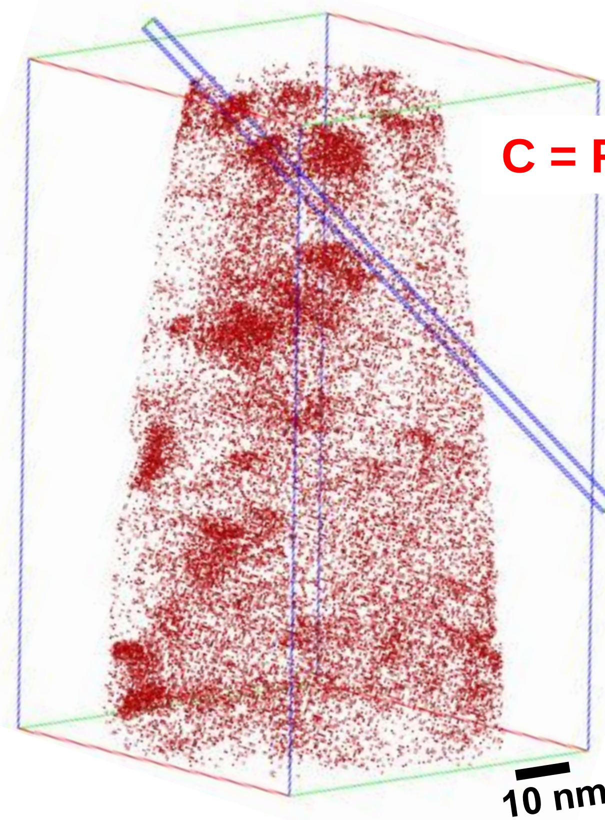
T.W. Brown, PhD Thesis,
ASPPRC/CSM, 2014

Atom Probe Tomography



Evidence of Carbide Formation

0.19C, 1.59Mn, 1.63Si (wt-%)

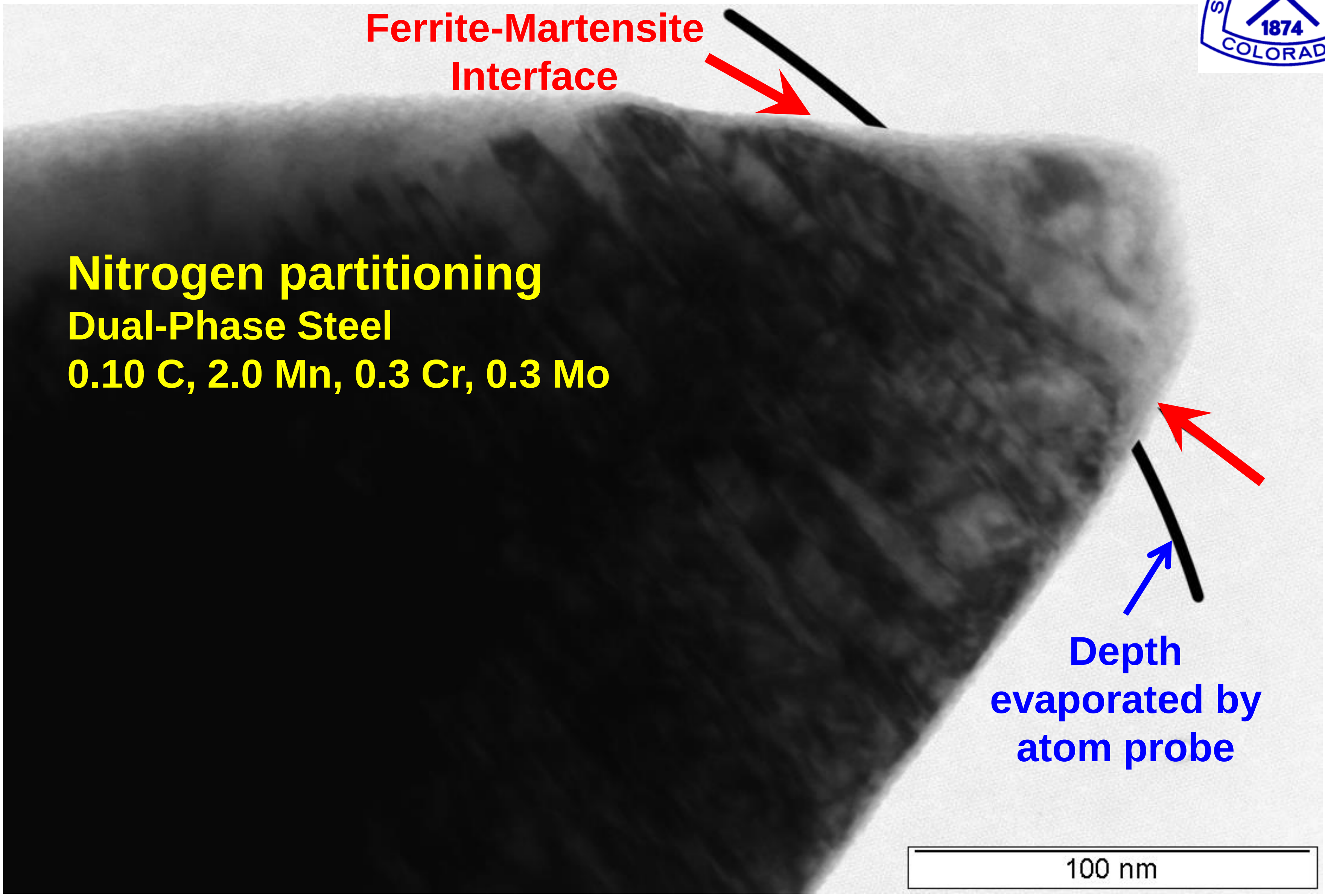


Anneal = 900 ^{o}C, 180 s
QT = 240 ^{o}C, 10 s
PT = 400 ^{o}C, 30 s

L.M. Gordon and D. Joester,
Front. Physiol. Vol. 6, 2015, p.:57.

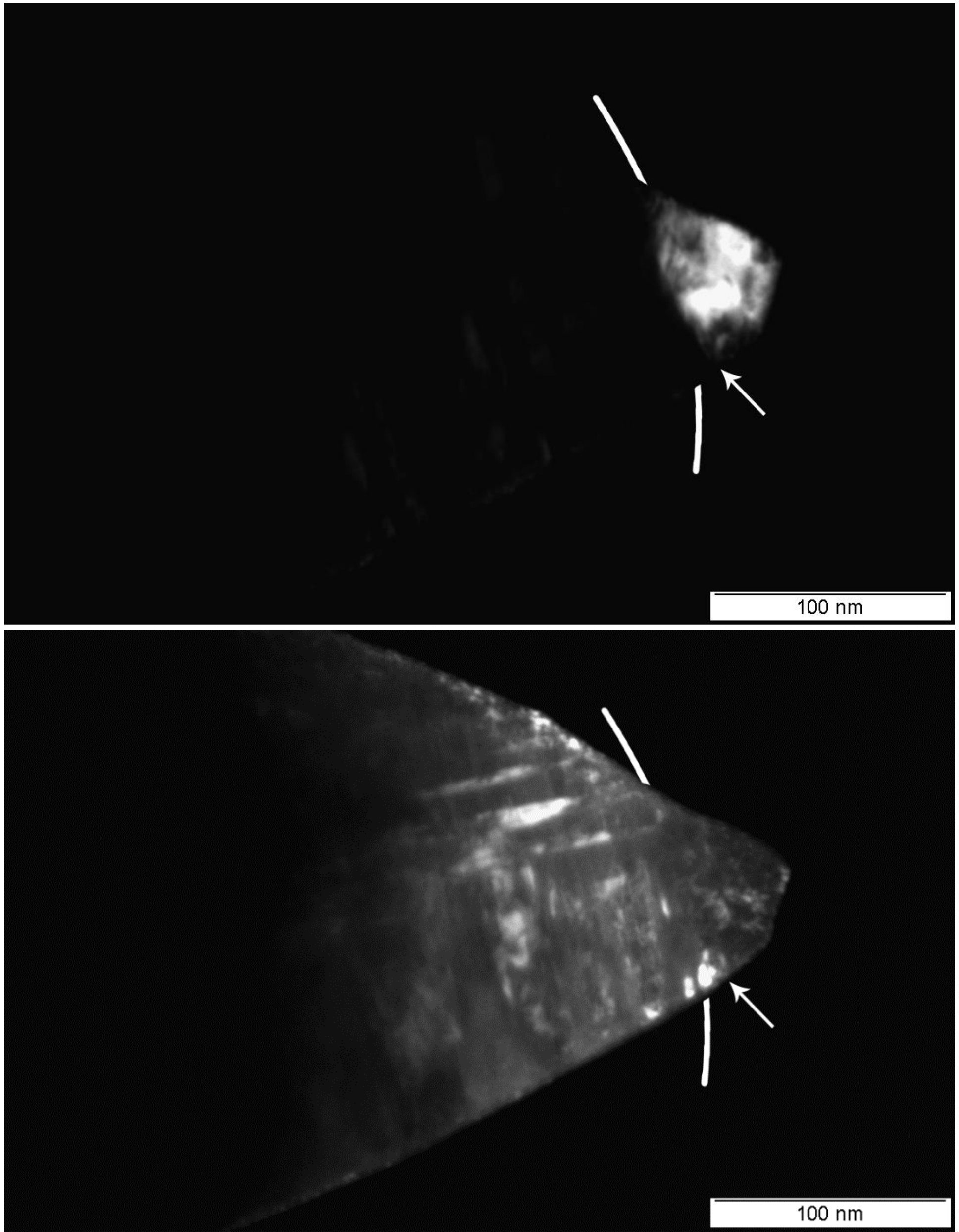
A. Clarke, PhD Thesis, Colorado School of Mines, 2006.

APT DP Steel Specimen –TEM



T.W. Brown, PhD Thesis, ASPPRC/CSM, 2014

APT DP Steel Specimen – Dark Field TEM



Nitrogen partitioning

Dual-Phase Steel

0.10 C, 2.0 Mn, 0.3 Cr, 0.3 Mo

Ferrite illuminated at tip

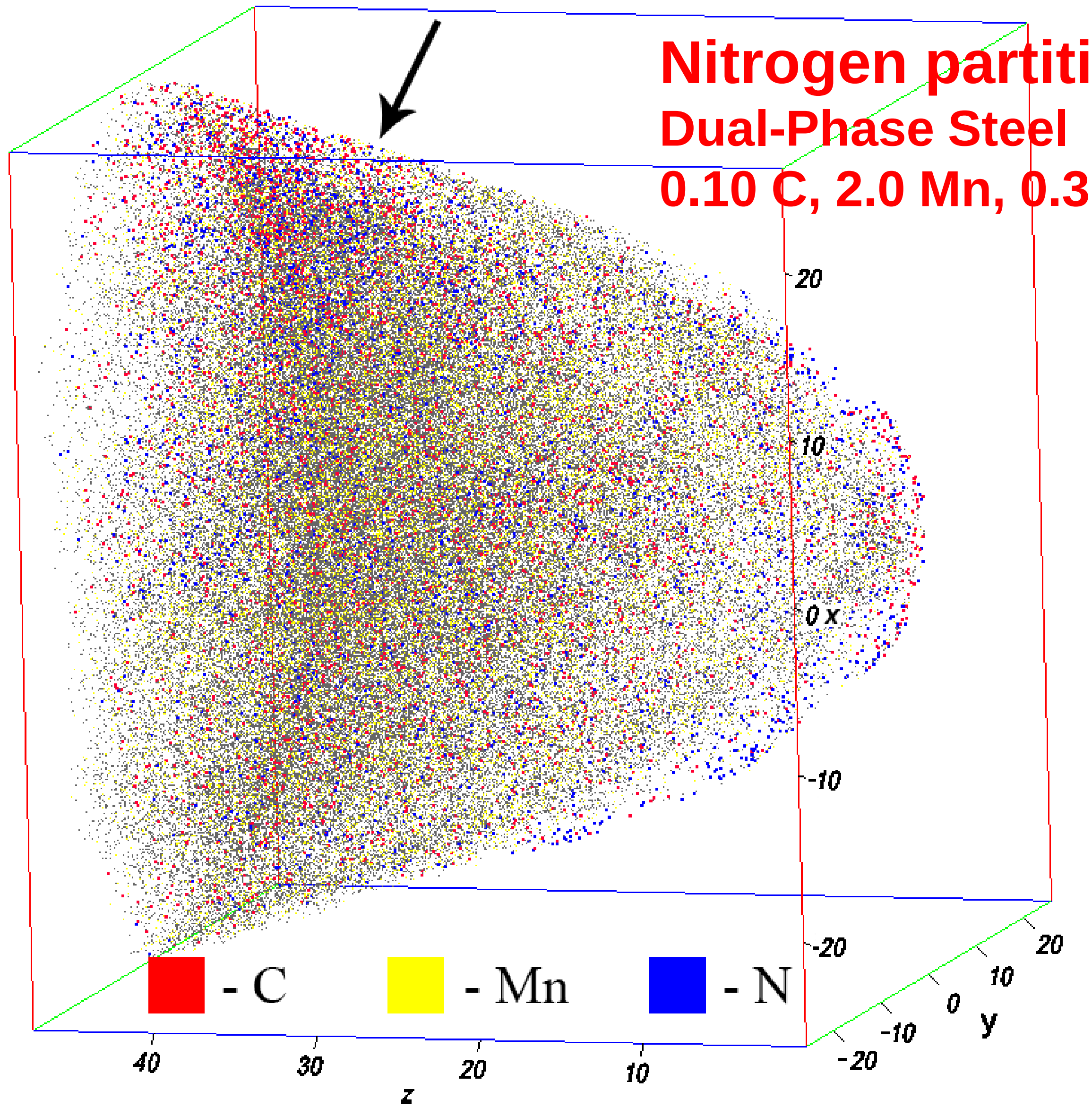
Martensite identified by lath structure

One of two variants illuminated

T.W. Brown, PhD Thesis, ASPPRC/CSM, 2014

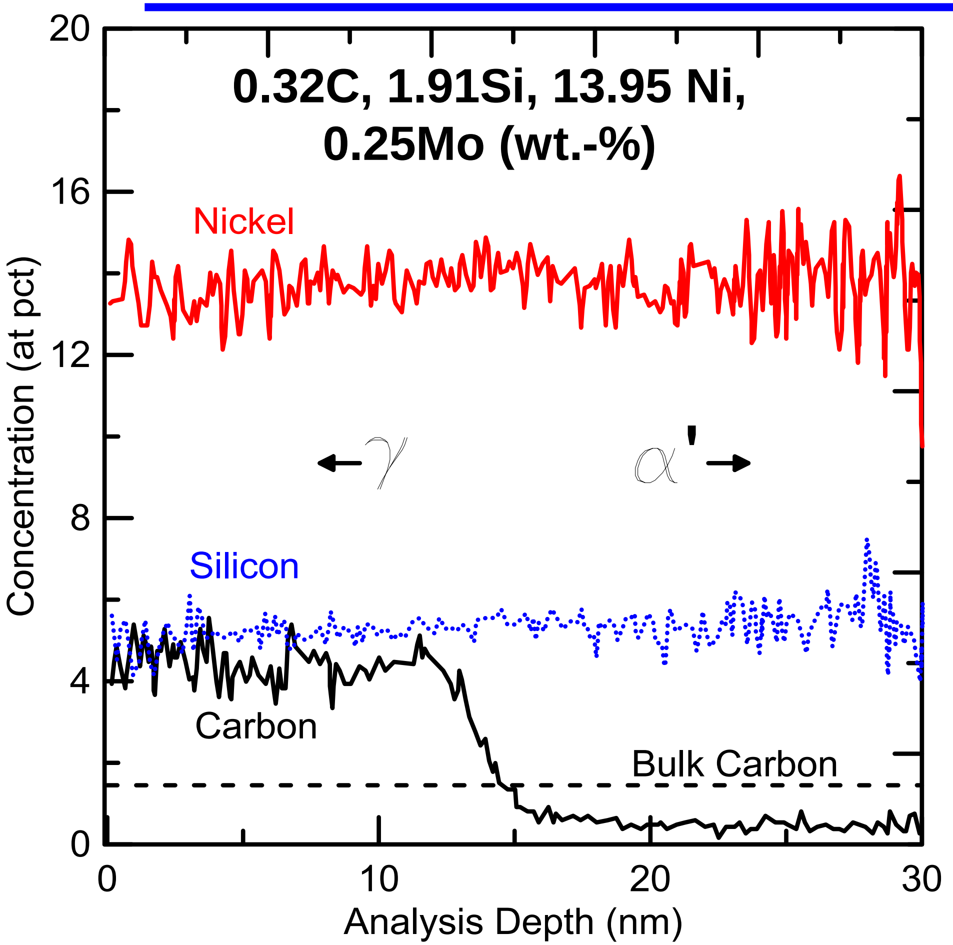
APT DP Steel: Atom Probe Reconstruction

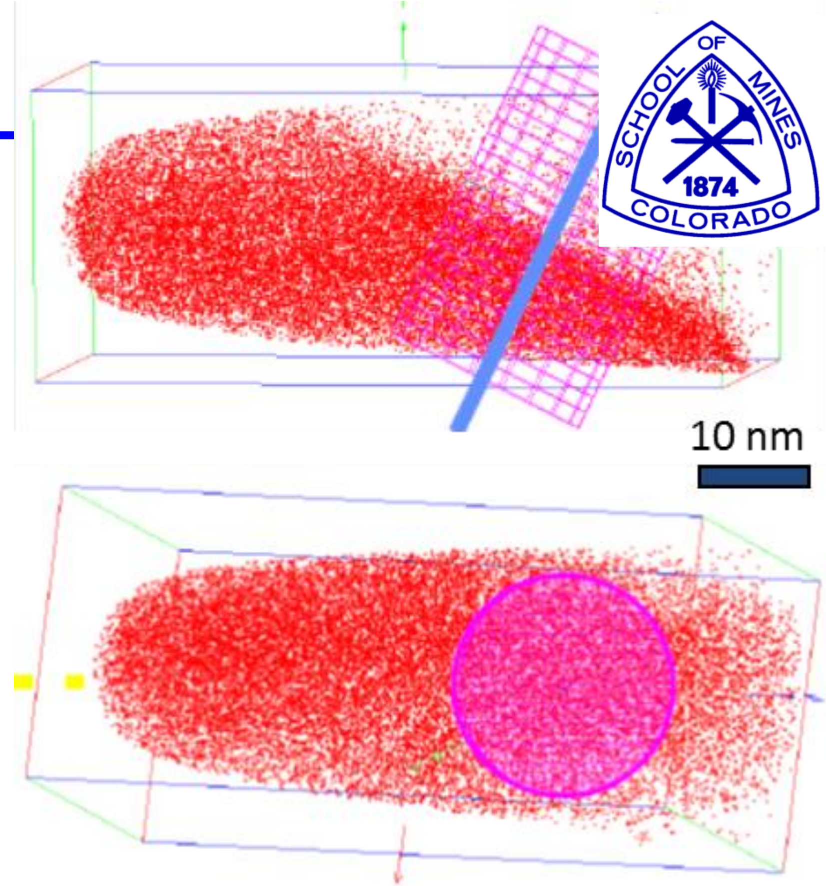
Nitrogen partitioning
Dual-Phase Steel
0.10 C, 2.0 Mn, 0.3 Cr, 0.3 Mo



T.W. Brown, PhD Thesis, ASPPRC/CSM, 2014

APT: High Ni Alloy





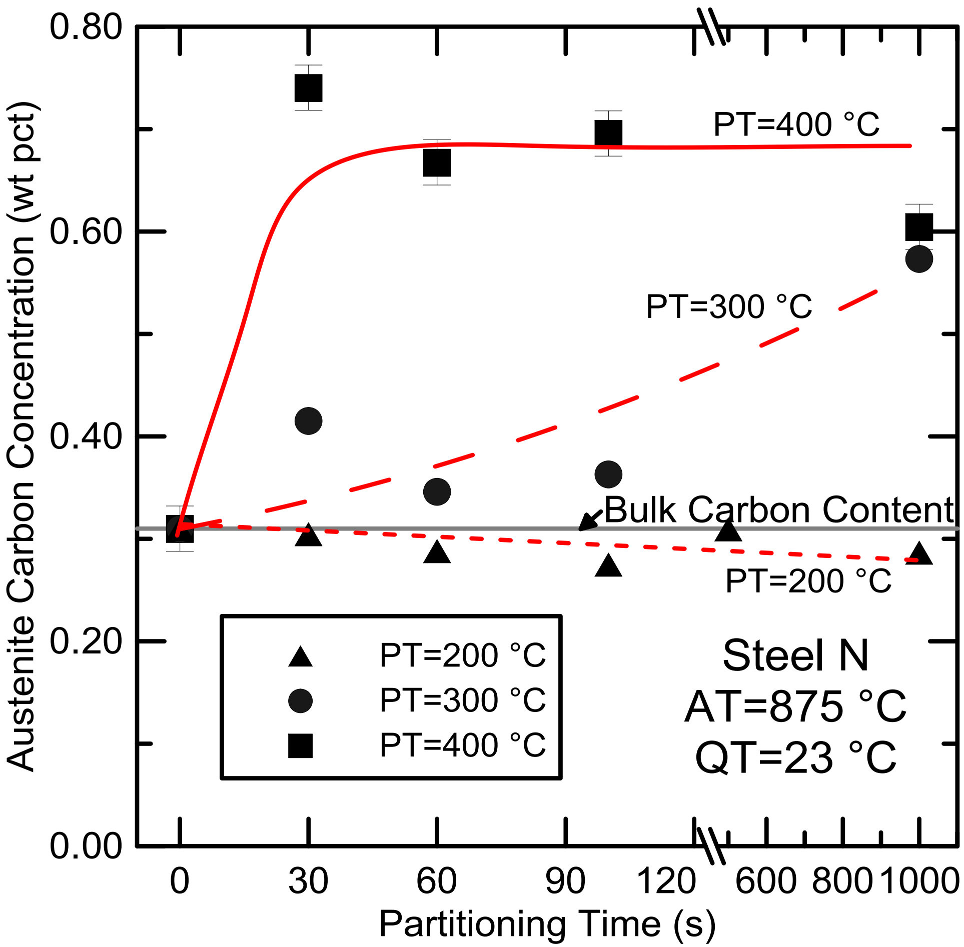
- **Austenitize = 875 ºC**
- **QT = Room T (23 ºC)**
- **PT = 400 ºC, 100 s**

Austenite (at.-%)			
C	Si	Ni	Fe
4.51	5.1	14.2	76.3
Martensite (at.-%)			
0.18	5.3	13.7	80.9
Bulk Steel (at.-%)			
1.45	3.7	13.0	81.9

G.A. Thomas, F. Danoix, J.G. Speer, S.W. Thompson and F. Cuvilly, *ISIJ International*, Vol. 54, No. 12, 2014, pp. 2900-2906.

High Ni Alloy: X-Ray Diffraction + APT

0.32C, 1.91Si, 13.95 Ni, 0.25Mo



Austenite				
	C	Si	Ni	Fe
at pct	4.51	5.06	14.2	76.3
wt pct	1.05	2.69	15.8	80.5

Martensite				
	C	Si	Ni	Fe
at pct	0.18	5.27	13.7	80.9
wt pct	0.04	2.71	14.7	82.6

Bulk Steel				
	C	Si	Ni	Fe
at pct	1.45	3.71	13	81.9
wt pct	0.32	1.91	14	83.8

- Austenitize = 875 ^{o}C
- QT = Room T (23 ^{o}C)
- PT = 400 ^{o}C, 100 s

G.A. Thomas, F. Danoix, J.G. Speer, S.W. Thompson and F. Cuvilly, *ISIJ International*, Vol. 54, No. 12, 2014, pp. 2900-2906.

Atom Probe Tomography: Summary

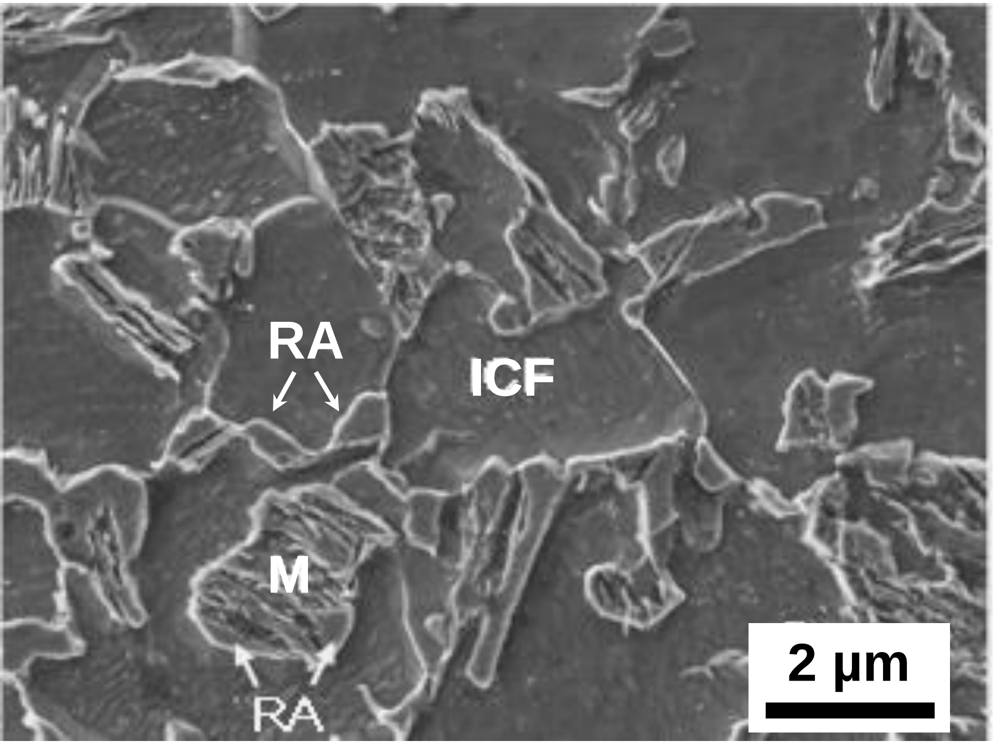
- APT technique applicable to Q&P steels
- APT confirmed C partitioning based on XRD analyses
- APT provided evidence of carbon atom "clustering" in martensite during partitioning
  - Clustering related to carbide formation
  - Carbon partitioning and martensite partitioning occur simultaneously

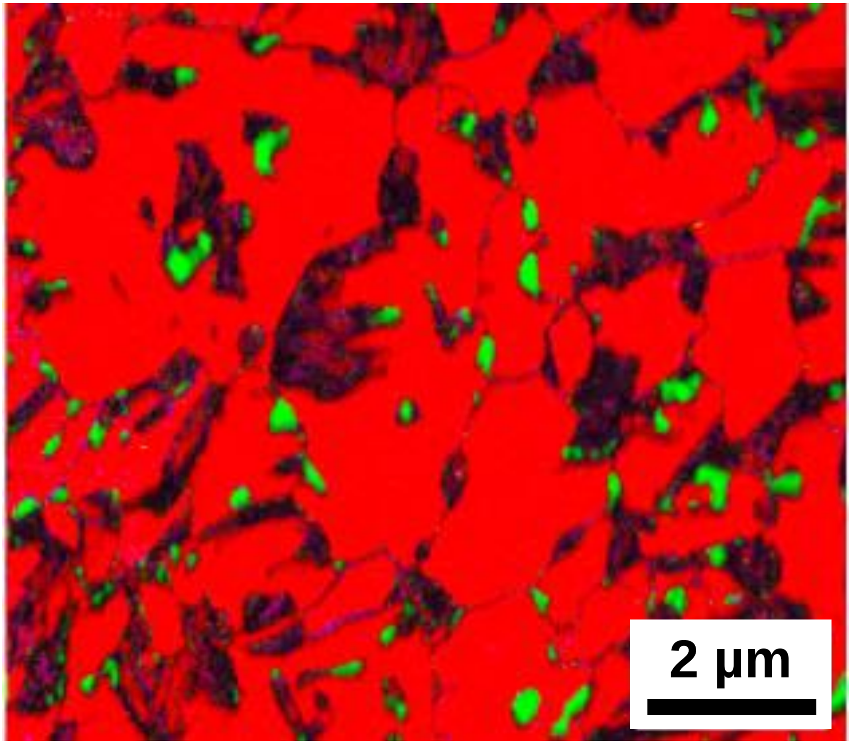
EBSD

Grant Thomas, John Speer, David Matlock and Joseph Michael, "Application of Electron Backscatter Diffraction Techniques to Quenched and Partitioned Steels," *Microscopy and Microanalysis, vol.* 17, pp 368-373.

Example: EBSD of Q&P Microstructure

- 0.19C, 1.59Mn, 1.63Si (wt-%) steel
- Intercritically annealed at 820 °C
- QT = 250 °C
- Partitioned on cooling to simulate processing during hot rolling

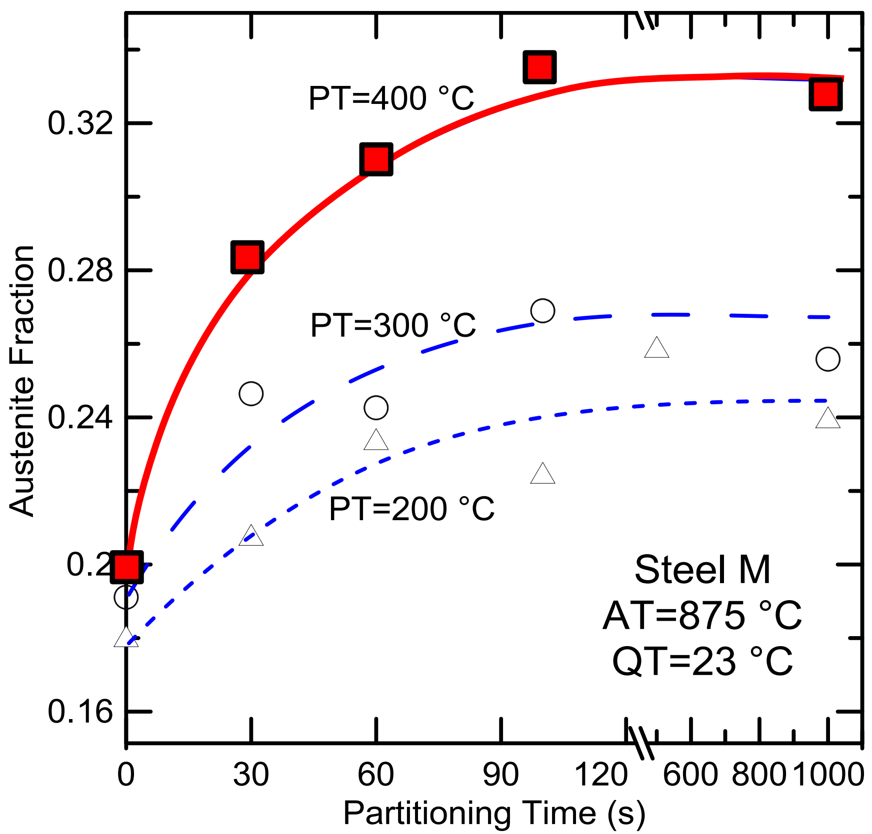




Grant Thomas, John Speer, David Matlock and Joseph Michael, "Application of Electron Backscatter Diffraction Techniques to Quenched and Partitioned Steels," *Microscopy and Microanalysis, vol.* 17, pp 368-373.

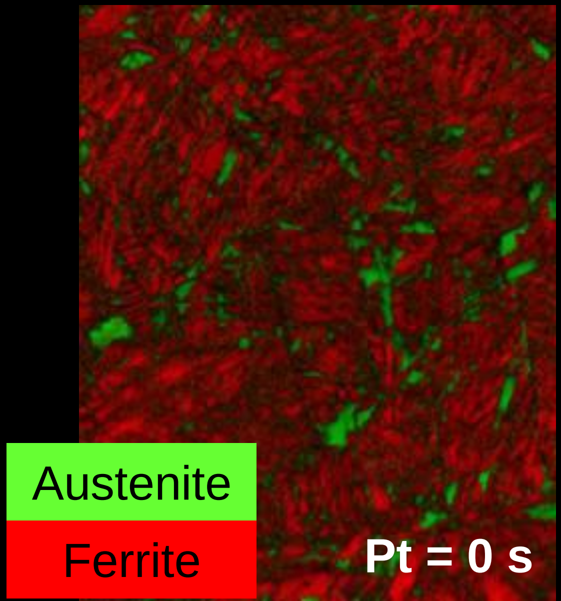
With partitioning time…..Growth of Existing Austenite or Nucleation of New?

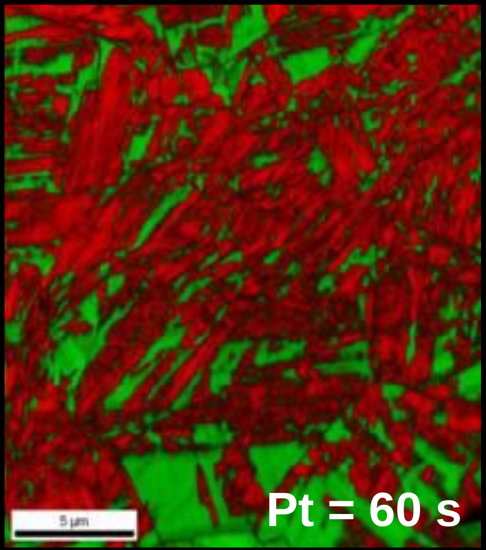
- **Application of EBSD to assess austenite contents and density = f(t).**
- **Evaluate special steel with QT = Room T**
- **0.27 C, 8.31 Mn, 1.94 Si, 0.24 Mo (wt-%)**
  - **M_s = 142 °C**

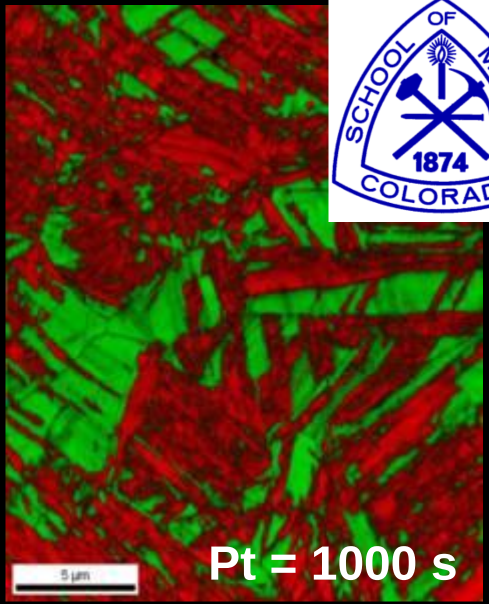


G.A. Thomas, PhD Thesis, ASPPRC/CSM, 2012

EBSD: IQ + Phase Maps





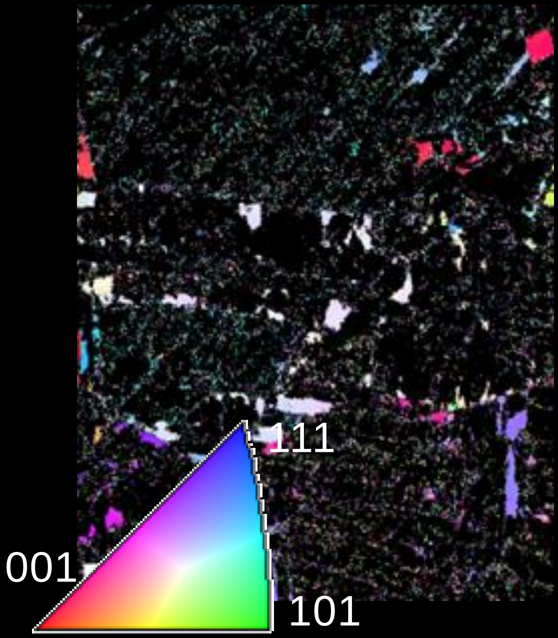


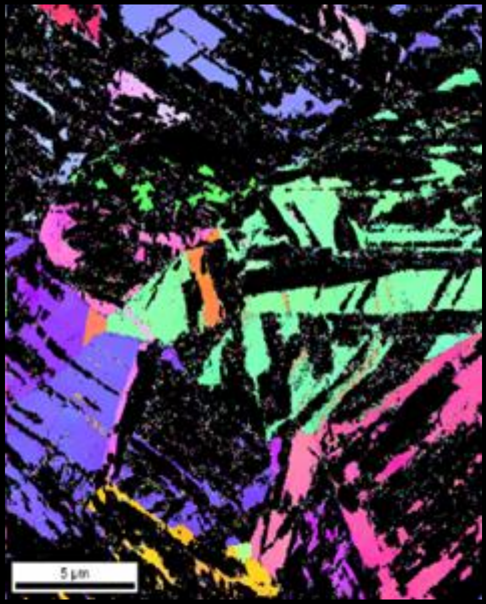
0.27 C - 8.31 Mn - 1.94 Si - 0.24 Mo

AT=875, QT=23 °C, PT=400°C

10 µm

EBSD: Austenite IPF Maps





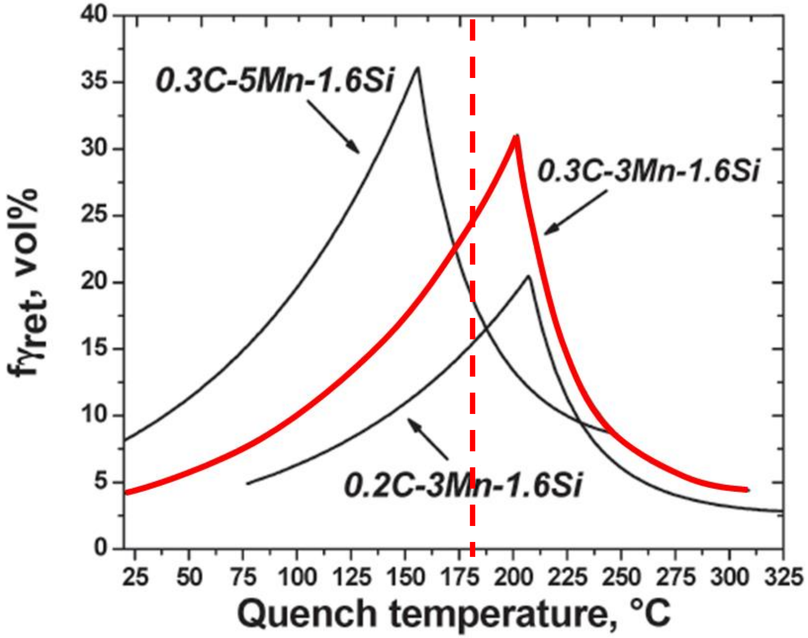
G.A. Thomas, PhD Thesis, ASPPRC/CSM, 2012

Nanoscale Secondary Ion Mass Spectroscopy (NanoSIMS)

Kyoo Sil Choi, Zihau Zhu, Xin Sun, Emmanuel De Moor, Mark D. Taylor, John G. Speer, and David K. Matlock, "Determination of Carbon Distributions in Quenched and Partitioned Microstructures using Nanoscale Secondary Ion Mass Spectroscopy," Scripta Mater., Vol. 104, 2015, pp.79-82.

NanoSIMS - Experiment

- **Steel 0.3C-3Mn-1.6Si**
- **Q&P**
  - **Anneal at 830 ºC**
  - **QT = 180 ºC (10 s)**
  - **PT = 400 ºC (100 s)**
- **Assess C distribution in microstructure**
- **Evaluate with SEM, EBSD, NanoSIMS****

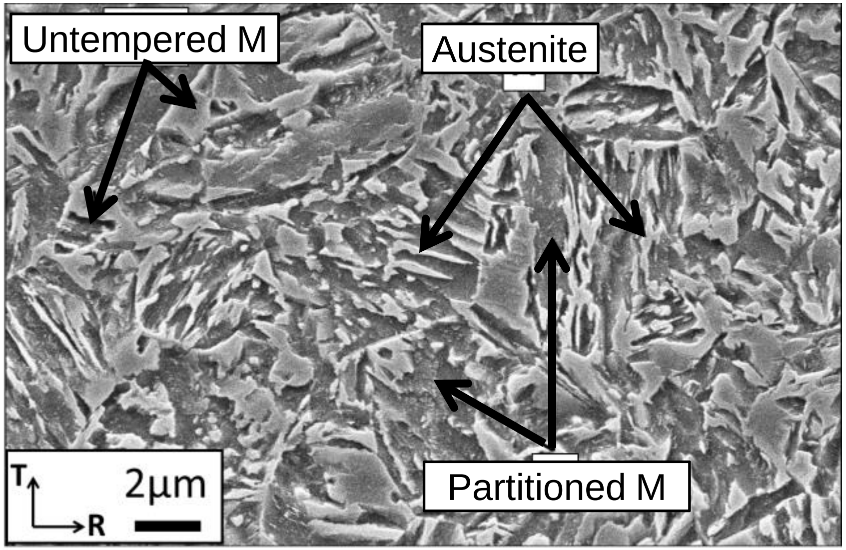


E. De Moor, J.G. Speer, D.K. Matlock, J.H. Kwak, and S.B. Lee, "Effect of Carbon and Manganese on the Quenching and Partitioning Response of CMnSi Steels," *ISIJ Intl.* Vol. 51, no. 1, 2011, pp.137-144.

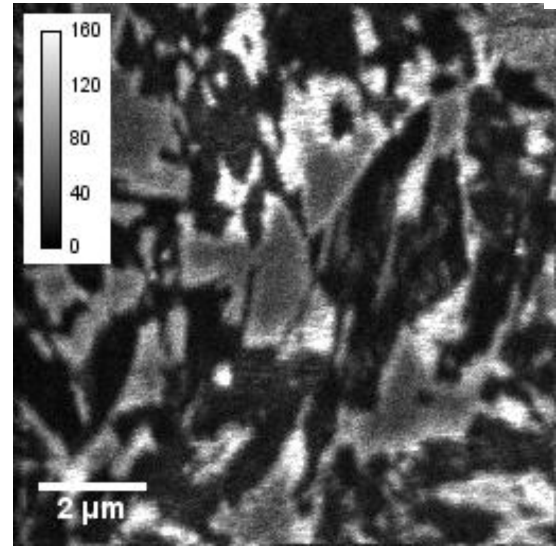
**Acknowledge collaboration with PNNL

NanoSIMS - Experiment

SEM Image (2 pct nital

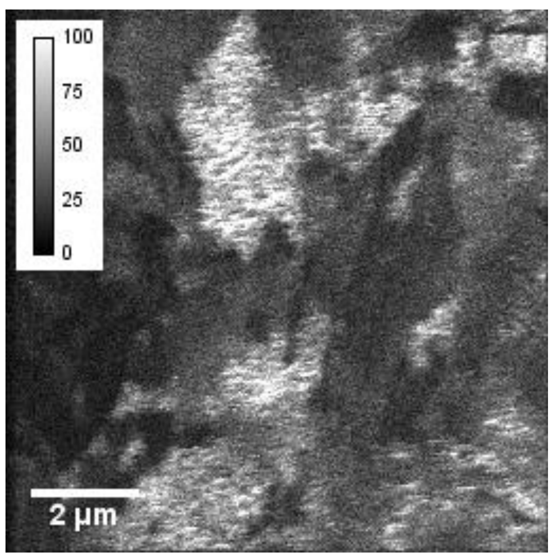


Carbon ($^{12}C^-$) counts

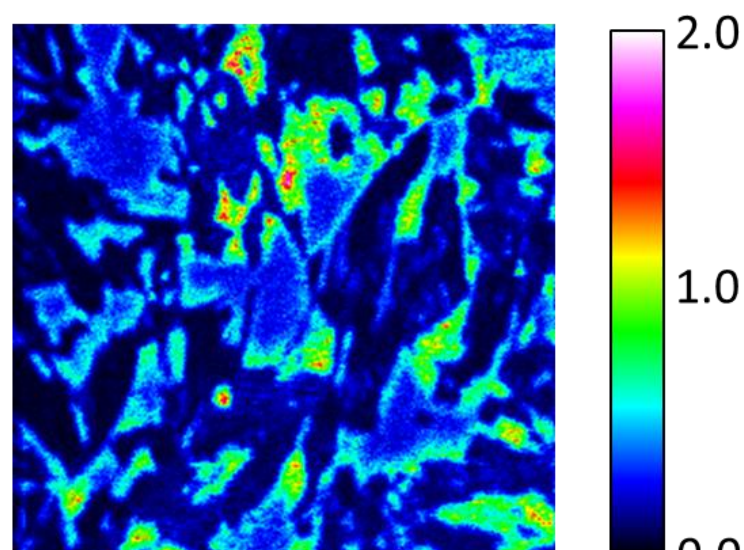


Kyoo Sil Choi, Zihau Zhu, Xin Sun, Emmanuel De Moor, Mark D. Taylor, John G. Speer, and David K. Matlock, "Determination of Carbon Distributions in Quenched and Partitioned Microstructures using Nanoscale Secondary Ion Mass Spectroscopy," Scripta Mater., Vol. 104, 2015, pp.79-82.

Silicon ($^{28}Si^-$) counts

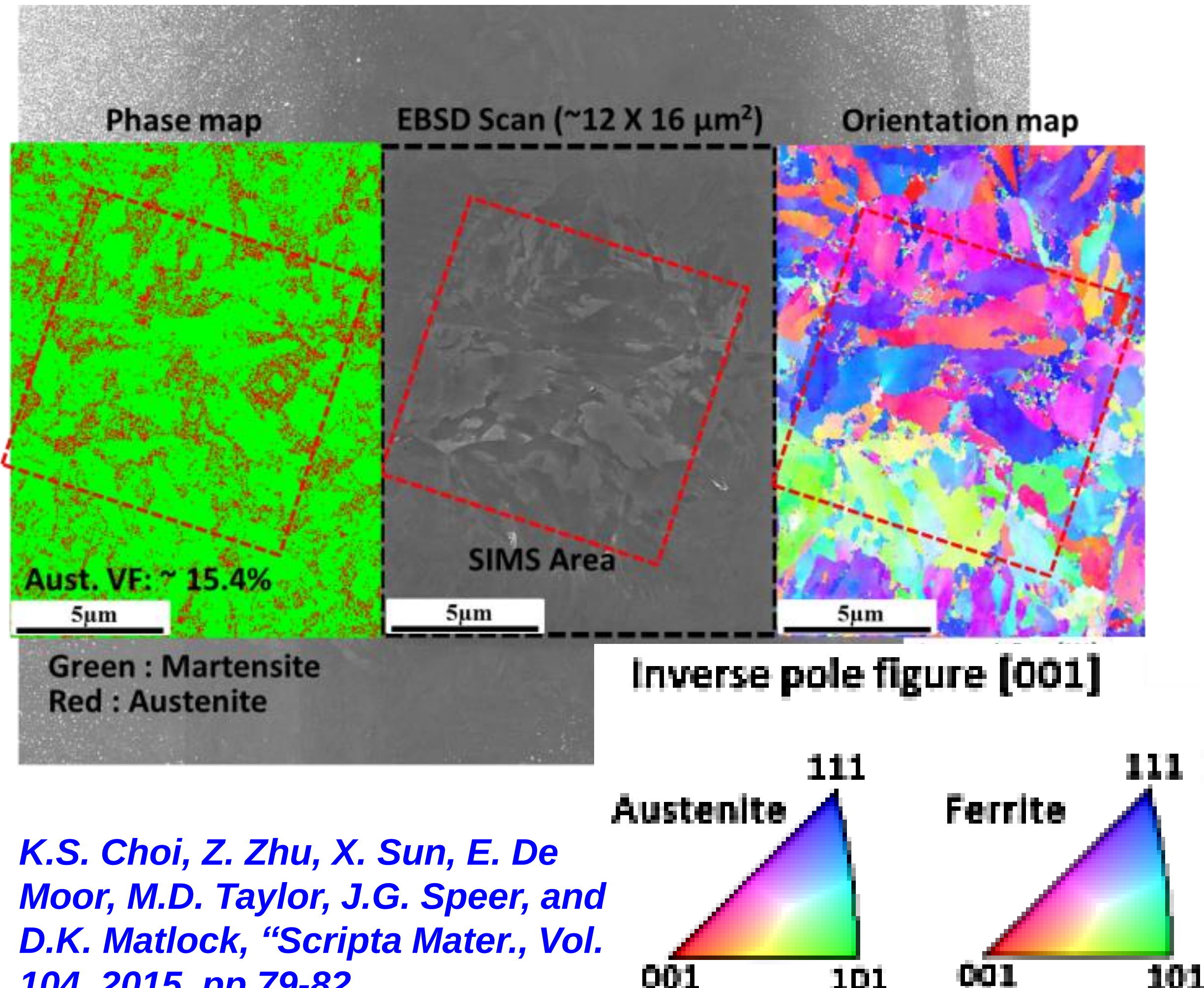


Carbon distribution in wt-%

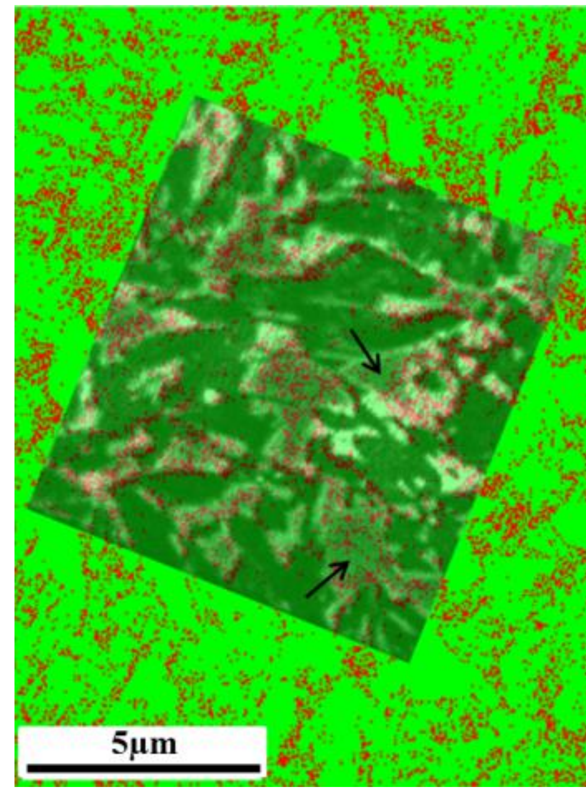


NanoSIMS - Experiment

EBSD results for location investigated using Nano-SIMS



Overlay of carbon count image on EBSD phase map



K.S. Choi, Z. Zhu, X. Sun, E. De Moor, M.D. Taylor, J.G. Speer, and D.K. Matlock, "Scripta Mater., Vol. 104, 2015, pp.79-82.

NanoSIMS Summary

- Significant carbon enrichment of austenite confirmed
- Carbon content of martensite less than bulk
- NanoSIMS capable of evaluating C and Si distributions in very fine AHSS microstructures

Kyoo Sil Choi, Zihau Zhu, Xin Sun, Emmanuel De Moor, Mark D. Taylor, John G. Speer, and David K. Matlock, "Determination of Carbon Distributions in Quenched and Partitioned Microstructures using Nanoscale Secondary Ion Mass Spectroscopy," Scripta Mater., Vol. 104, 2015, pp.79-82.

Mössbauer Spectroscopy: Characterization of Transition Carbides in Q&P Steel

*DT Pierce, DR Coughlin, DL Williamson, KD Clarke, AJ Clarke, JG Speer, DK Matlock, E De Moor, "Mössbauer Spectroscopy and Transmission Electron Microscopy Analysis of Transition Carbides in Quenched and Partitioned Steel," Session P07.06, - **Metallography and Microstructural Characterization of Metals, Location C123, Thursday, Aug 6 @ 2:15 PM***

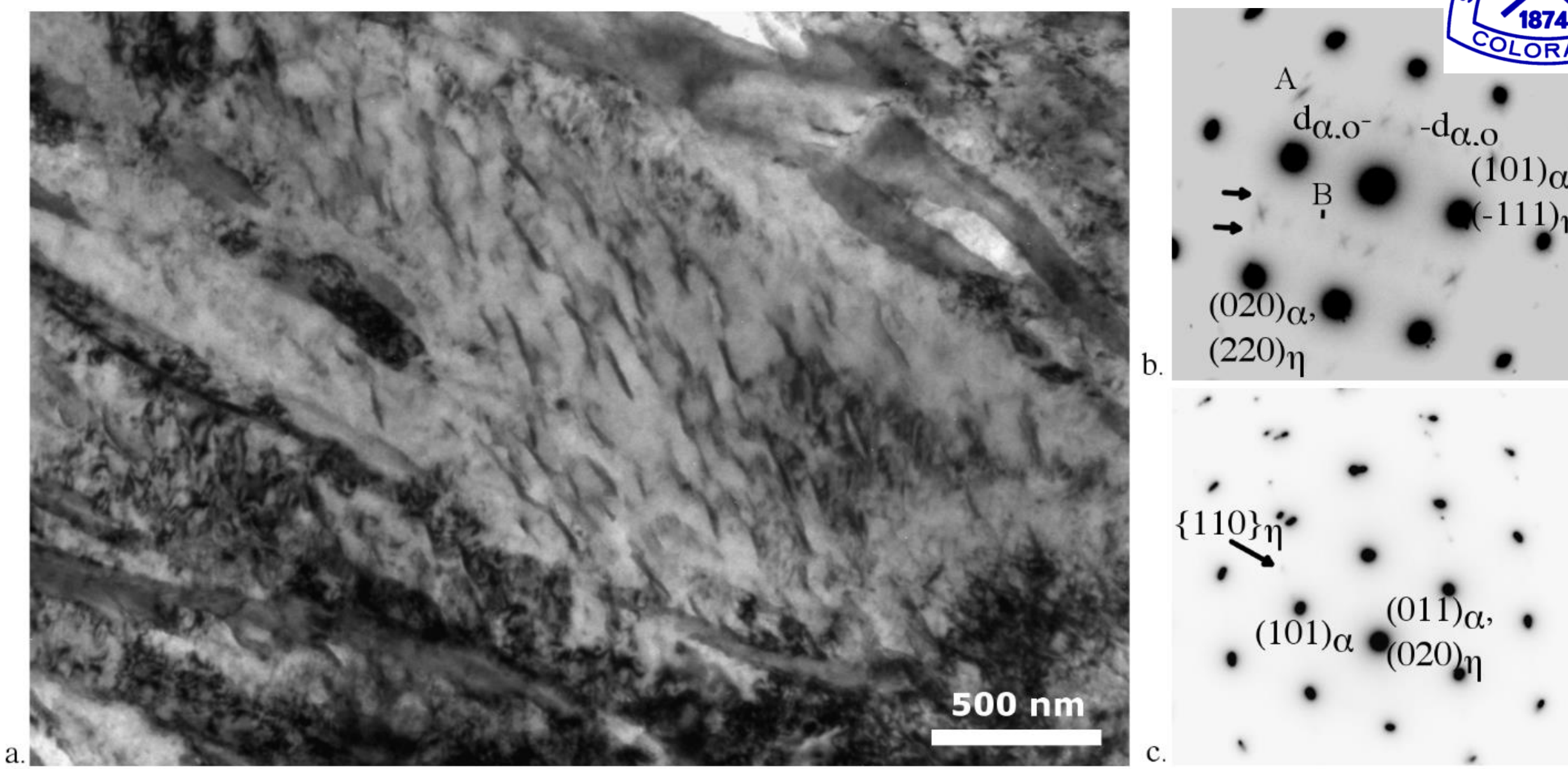
Mössbauer Spectroscopy

- **Develop Mössbauer methodology for Q&P**
- **Characterize evolution of carbide formation during Q&P.**
- **Mössbauer spectra sensitive to atom nearest neighbors and can be used to assess structure and composition**
- **Previously applied to binary high-C steels**
- **Experiment:**
  - **Evaluate effects of Q&P processing parameters and alloy content**

C	Mn	Si	Al	N
0.38	1.54	1.48	0.070	0.0022

DT Pierce, DR Coughlin, DL Williamson, KD Clarke, AJ Clarke, JG Speer, DK Matlock, E De Moor, "Mössbauer Spectroscopy and Transmission Electron Microscopy Analysis of Transition Carbides in Quenched and Partitioned Steel," Session P07.06, - Metallography and Microstructural Characterization of Metals, Location C123, Thursday, Aug 6 @ 2:15 PM

TEM Evidence for η-carbide Formation



C	Mn	Si	Al	N
0.38	1.54	1.48	0.070	0.0022

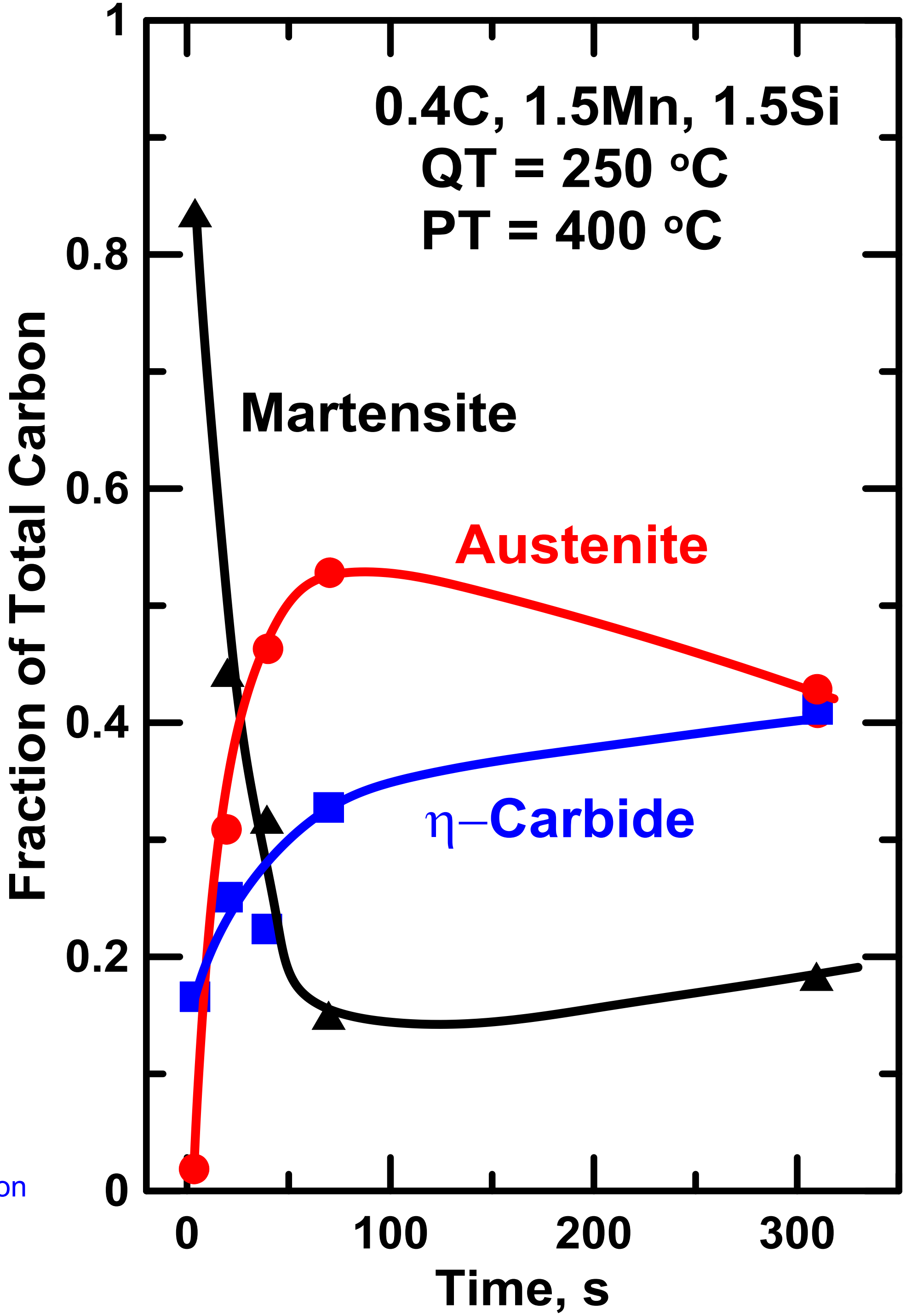
Q&P Processed: QT = 225°C PT = 400°C for 300 s

DT Pierce, DR Coughlin, DL Williamson, KD Clarke, AJ Clarke, JG Speer, DK Matlock, E De Moor, "Mössbauer Spectroscopy and Transmission Electron Microscopy Analysis of Transition Carbides in Quenched and Partitioned Steel," Session P07.06

Mössbauer

- **With partitioning time**:
  - Carbon in martensite decreases
  - Carbon partitioned to austenite
  - Up to 40% of available carbon tied up as η-carbide

C	Mn	Si	Al	N
0.38	1.54	1.48	0.07	0.0022



D.T. Pierce, D.R. Coughlin, D.L. Williamson, K.D. Clarke, A.J. Clarke, J.G. Speer and E. De Moor, "Characterization of Transition Carbides in Quench and Partitioned Steel Microstructures by Mössbauer Spectroscopy and Complementary Techniques" Acta Mater., Vol. 90, 2015, pp. 417-430.

Mössbauer Summary

- **Significant carbon enrichment of austenite confirmed**
- **η-carbide formation consumes a portion of available carbon**
- **Carbon content of martensite is depleted**

D.T. Pierce, D.R. Coughlin, D.L. Williamson, K.D. Clarke, A.J. Clarke, J.G. Speer and E. De Moor, "Characterization of Transition Carbides in Quench and Partitioned Steel Microstructures by Mössbauer Spectroscopy and Complementary Techniques" Acta Mater., Vol. 90, 2015, pp. 417-430.

DT Pierce, DR Coughlin, DL Williamson, KD Clarke, AJ Clarke, JG Speer, DK Matlock, E De Moor, "Mössbauer Spectroscopy and Transmission Electron Microscopy Analysis of Transition Carbides in Quenched and Partitioned Steel," Session P07.06, - Metallography and Microstructural Characterization of Metals, Location C123, Thursday, Aug 6 @ 2:15 PM

Status Update: AHSS Developments

Government Initiatives and Industrial Commercialization

ENERGY.GOV
Office of Energy Efficiency & Renewable Energy

U.S. DEPARTME
ENER

SERVICES | EFFICIENCY | RENEWABLES | TRANSPORTATION | ABOUT US | OFFICES

SCHOOL OF MINES 1874 COLORADO

Integrated Computational Materials Engineering (ICME) Development of Advanced Steel for Lightweight Vehicles

- **"…develop 3rd Generation Advanced High Strength Steel (3GAHSS) technology .. Advance ICME techniques to support a reduced development-to-deployment lead time in all lightweight materials systems."**
- **$8.5 million US for 4 years - steel and automobile companies, national laboratories, and universities.**

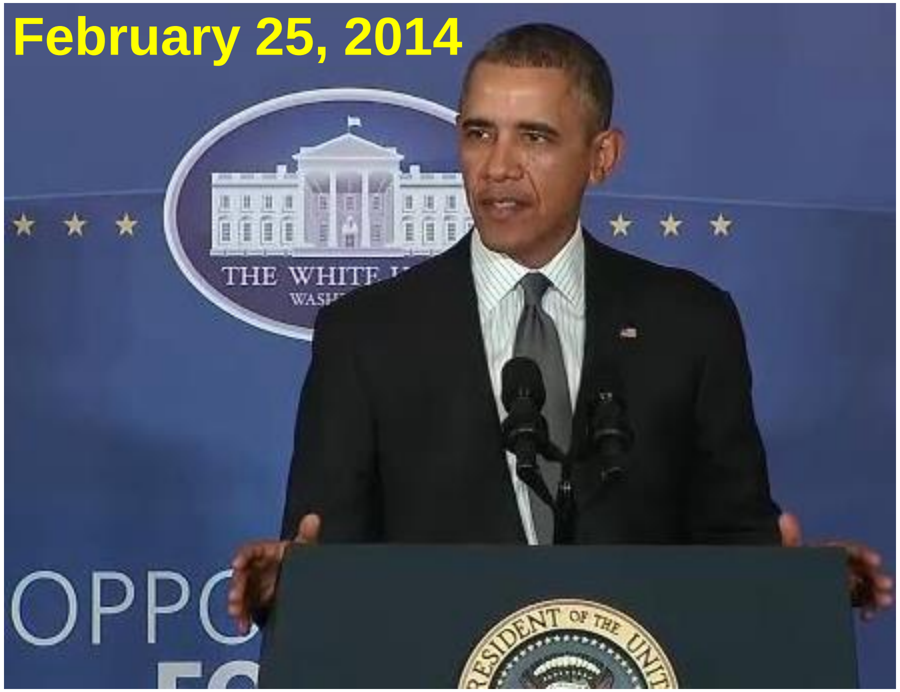
Advanced Manufacturing Office - Innovative Manufacturing Initiative

- **CSM received $1.2 million USD for 3-year program on new Q&P steels**

energy.gov/eere/vehicles/

National Network for Manufacturing Innovation

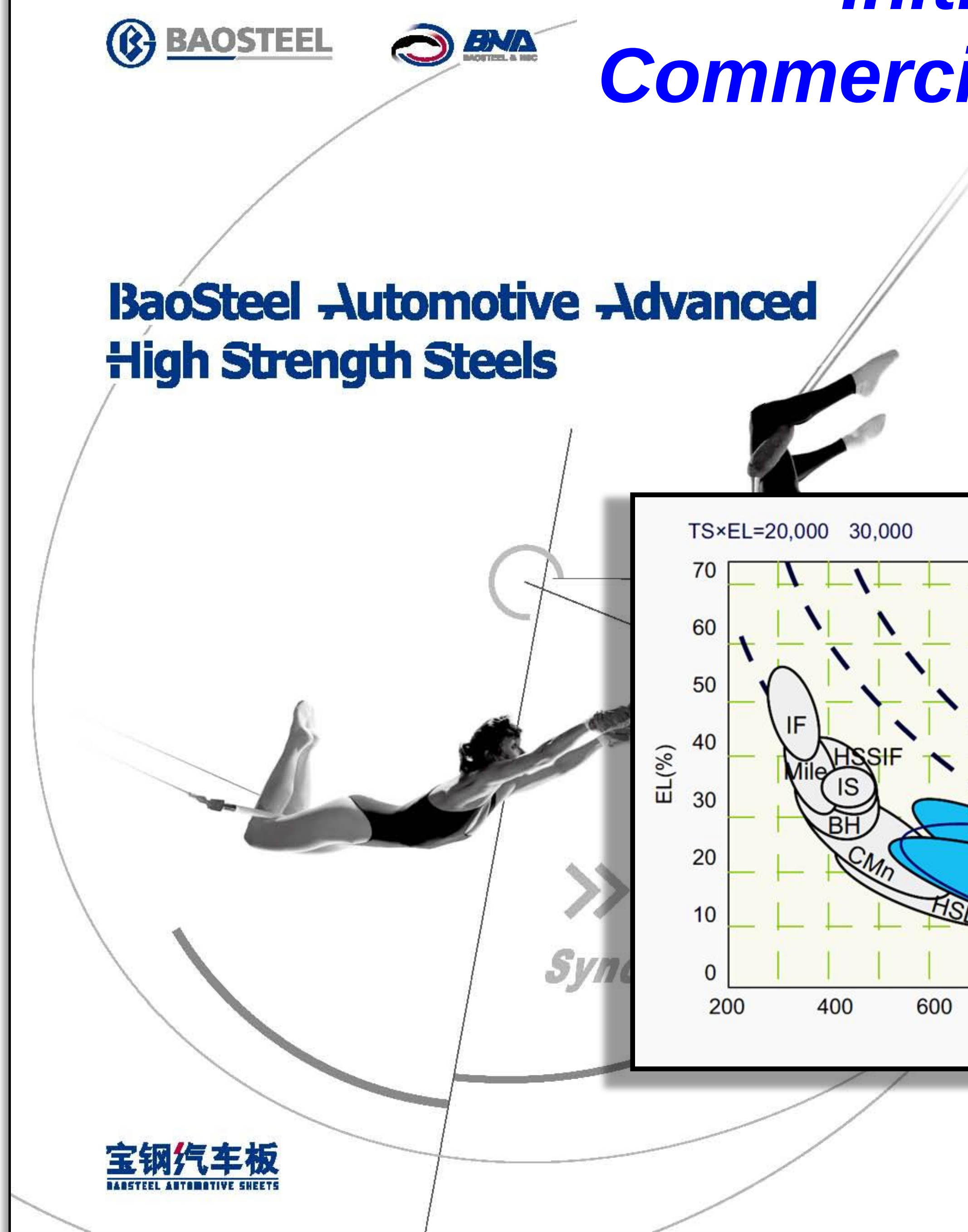
- **Lightweight Modern Metals Manufacturing Innovation (LM3I) Institute**
  - **Focus:**
    - **High Strength Steel**
    - **Mg, Ti, Al**
  - **$140M+ for 5 years**

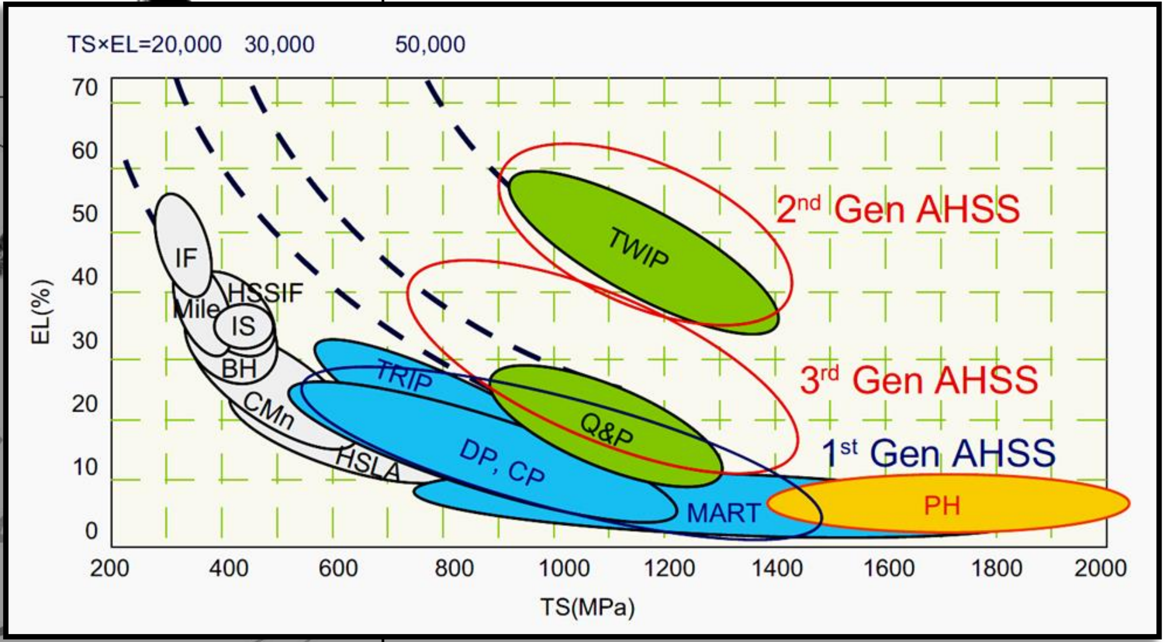


http://lift.technology/

A program operated by the American Lightweight Materials and Manufacturing Innovation Institute (ALMMII)

Initial Commercialization





www.baosteel.com/group_en/contents/2915/40092.html

New Pro-Tec CAL in Leipsic, Ohio

Joint Venture USS/Kobe

Opened May 2013

- **“..*state-of-the-art line ….*”**
- **“…*equipped with both advanced water quench …and rapid gas jet cooling*…”**
- **“…*to make next-generation high-strength steel products*…”**

ArcelorMittal – July 2015

Industry today

Search...

MAGAZINE ISSUE July 2015

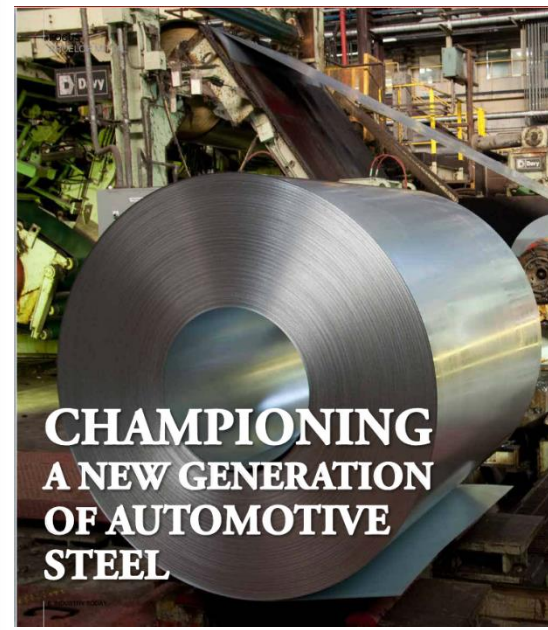
Volume 18, Issue 4

ARCELORMITTAL

Championing a New Generation of Automotive Steel

Preparing for future regulations

Automotive manufacturers face an immense challenge in the coming years.



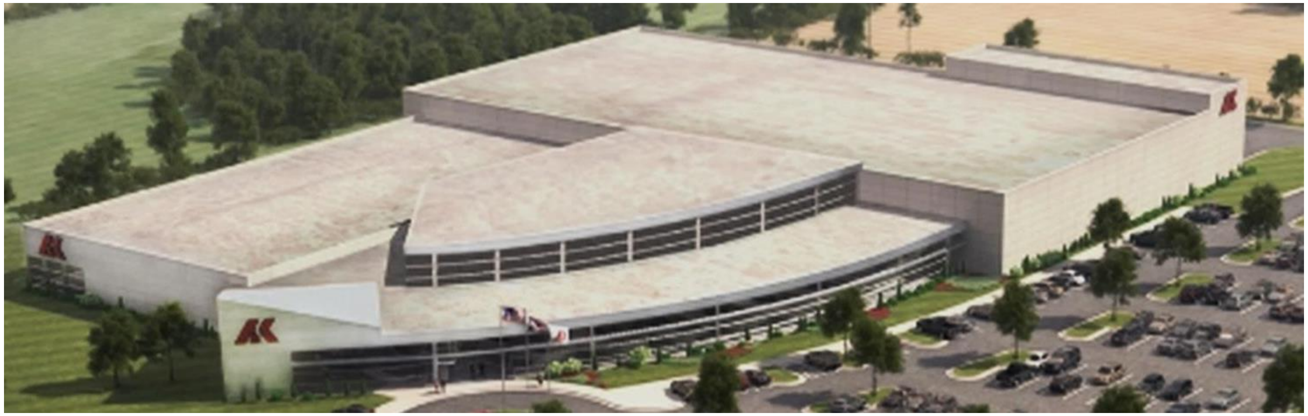
"ArcelorMittal …has been developing new, lightweight solutions using a range of advanced high strength steels (AHSS)"

http://industrytoday.com/currentissue.asp (accessed July 2015)

AK Steel – June 2015

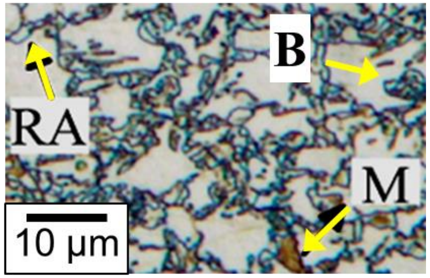
AK Steel to Produce Next-Generation Advanced High Strength Steels: West Chester, OH, June 01, 2015—AK Steel today announced plans to introduce one of the first commercially available Next-Generation Advanced High Strength Steels in the world -- $29 million project …at AK Steel's Dearborn Works, using new process technology..

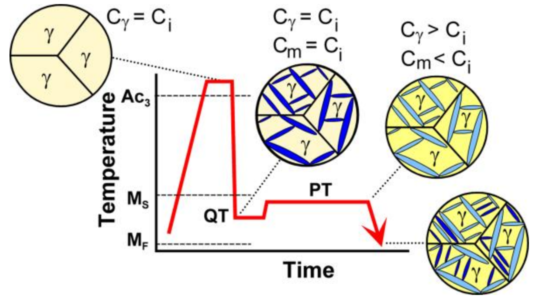
AK Steel Breaks Ground for New World-Class Research and Innovation Center: West Chester, OH, June 19, 2015

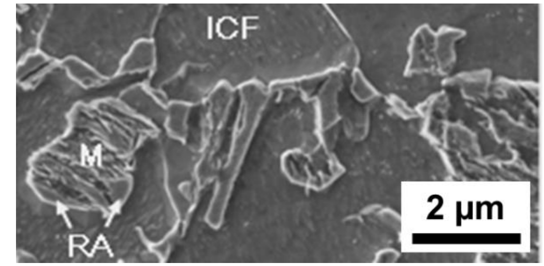


http://www.aksteel.com/news (accessed July 2015)

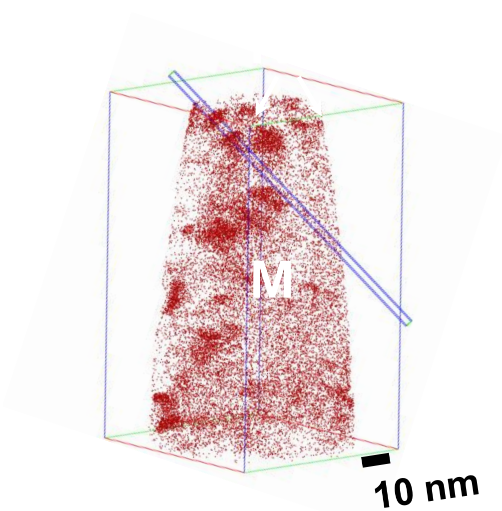
Closing Comments

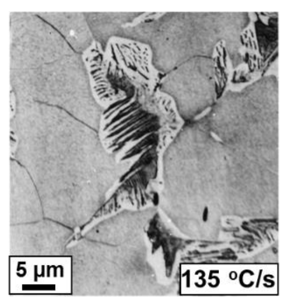


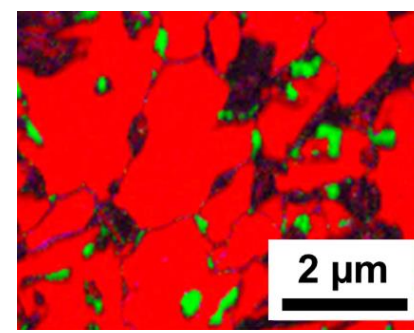




Results based on advanced metallographic techniques provide critical insight to facilitate developments of new AHSS products







Acknowledgements

- **This presentation is based in part upon work supported by the Department of Energy Advanced Manufacturing Office Innovative Manufacturing Initiative under Award Number DE-EE0005765 and by the Department of Energy National Energy Technology Laboratory under Award Number DE-EE0005976.**

- **The support of the sponsors of the Advanced Steel Processing and Products Research Center, an industry-university cooperative research center at the Colorado School of Mines is gratefully acknowledged**