ZCO-53-2: Galvanizing Experiments
as part of the project
ZCO-53: Hot-Dip Galvanizing of Third Generation (3G) AHSS

K. Bhadhon, E. McNally, D. Pallisco, V. Patel and J.R. McDermid

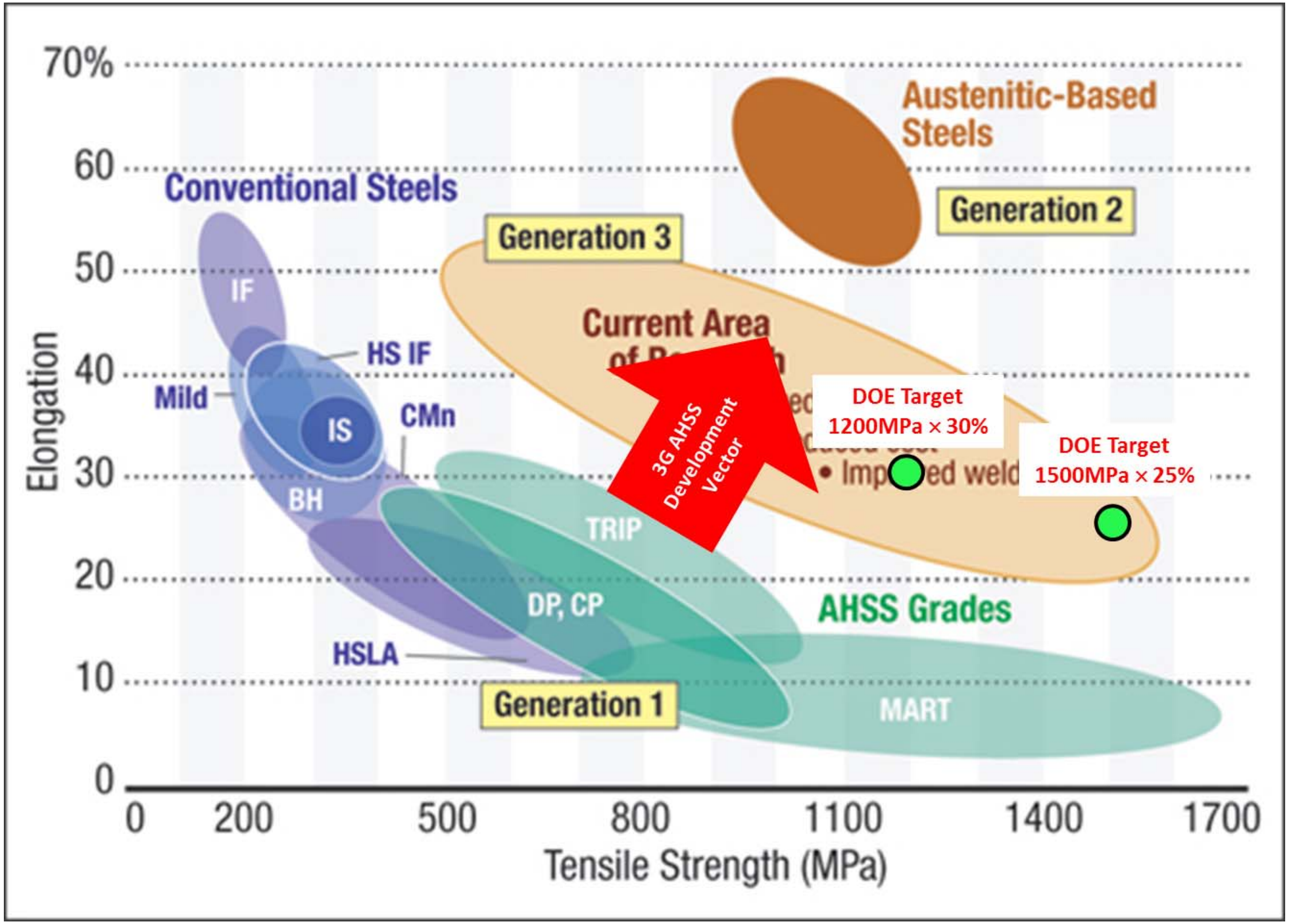
McMaster Steel Research Centre
Centre for Automotive Materials and Corrosion

October 2016 GAP Meetings

Project Objectives

- **Conduct galvanizing experiments using industrially solicited or custom fabricated medium Mn TRIP-assisted 3G-AHSS steels with the overall objectives of:**
  - **developing thermal processing treatments compatible with CGL capabilities which will;**
  - **meet 3G AHSS mechanical vs. US DOE targets 1200MPa UTS + 30% elongation (36,000 MPa%); 1500MPa UTS + 25% elongation (37,500 MPa%)**
  - **U.S. Auto targets: 1500MPa UTS + 20% elongation (30,000 MPa%)**
  - **developing a process window for the production of high quality galvanized coatings**

U.S. DOE Targets for 3G-AHSS Properties



C. Shutte: SAE Technical Paper 2015-01-0405 (2015), doi:10.4271/2015-01-0405

ZCO-53-2 Schedule

	M 1 – 6	M 6 – 12	M 12 – 18	M 18 – 24
Task 1: Microstructural and Mechanical Property Development				
steel A: 0.1C-6.3Mn-1.6Si-0.5Al				
steel B: 0.15C-6.5Mn-0.5Si-1.5Al				
steel D: 0.15C-6.1Mn-1Si-1Al				
steel E: 0.15C-6.1Mn-1Si-2Al				
steel F: 0.1C-6.3Mn-1.6Si-0.5Al-0.05Sn				
Task 2: Selective Oxidation and Hot-Dip Galvanizing Simulations				
Task 2a: Selective Oxidation				
steel A: 0.1C-6.3Mn-1.6Si-0.5Al				
steel B: 0.15C-6.5Mn-0.5Si-1.5Al				
steel D: 0.15C-6.1Mn-1Si-1Al				
steel E: 0.15C-6.1Mn-1Si-2Al				
Task 2b: Hot-Dip Galvanizing Simulations				
steel A: 0.1C-6.3Mn-1.6Si-0.5Al				
steel B: 0.15C-6.5Mn-0.5Si-1.5Al				
steel D: 0.15C-6.1Mn-1Si-1Al				
steel E: 0.15C-6.1Mn-1Si-2Al				
Task 3: Reports				

Steel Chemistries + Current Status

- **Actively working on Steels A, B and E (6Mn + 1Si + Al) grades**
- **Steel F = Steel A + 0.05Sn – oxide modification**

Steel	Type	C	Mn	Si	P	Al	Mo	Sn	Si/Mn
steel A	TRIP	0.21	6.26	1.55	0.007	0.52	0.077	--	0.25
steel B	TRIP	0.14	6.49	0.53	0.016	1.46	--	--	0.08
steel C	Q&P	0.2	1.8	1.5				--	0.83
steel D	TRIP	0.15	6.02	0.97	0.007	1.05	0.073	--	0.16
steel E	TRIP	0.15	6.09	0.97	0.007	1.89	0.084	--	0.16
steel F	TRIP	0.19	6.11	1.50	0.007	0.49	0.055	0.05	0.25

Steel A – Summary and Recent Progress

- **Have obtained high volume fractions of mechanically stable retained austenite consistent with 3G-AHSS mechanical properties and consistent with high UTS U.S. DOE 3G target properties**
  - **martensitic starting microstructures produced maximum 1640MPa × 23% (sub-size samples)**
  - **establishing elongation vs. full ASTM stds with U.S. Steel R&D (using U.S.S. CAL simulator material)**
- **Examined effect of 460°C IBT time on the properties of selected S-TM and S-M samples – sensitivity of mechanical properties to longer hold at bath temp**

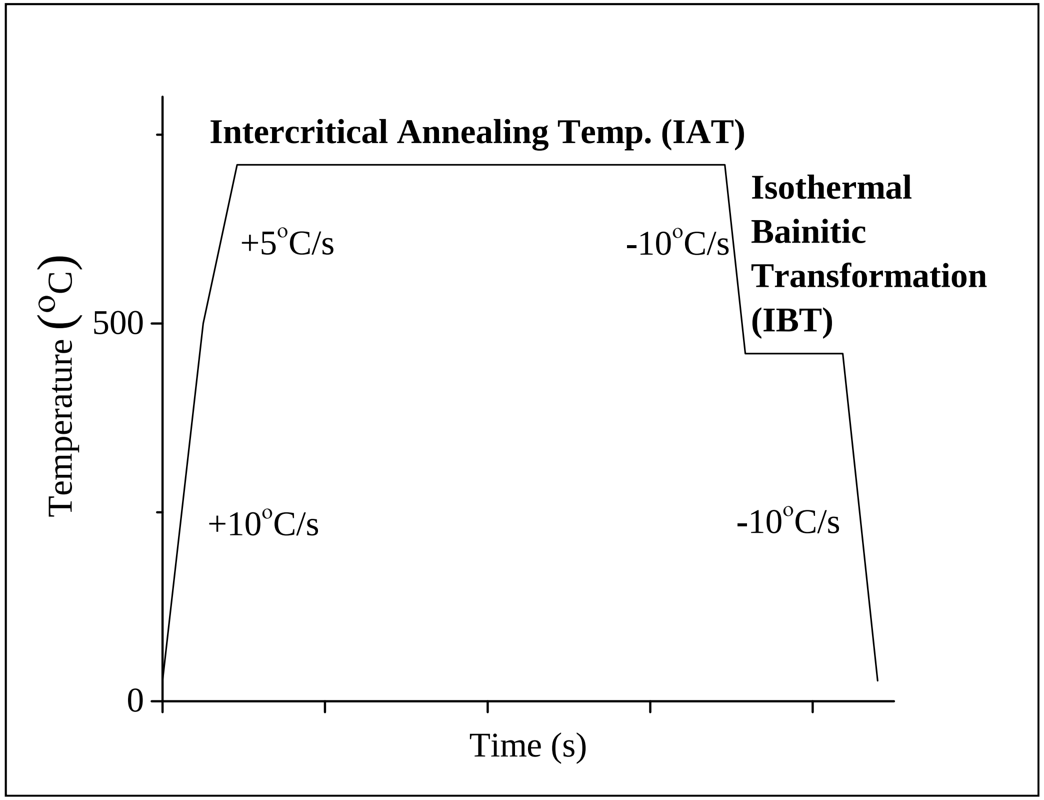
Steel A – Recent Progress

- Tensile and interrupted tensile tests of S-TM and S-M samples annealed with longer IBT holding times (60s, 120s)
- TEM analysis of selected S-M samples for fine scale microstructure and carbide chemistry analysis (20s IBT holding time)

Steel A – Tensile Tests

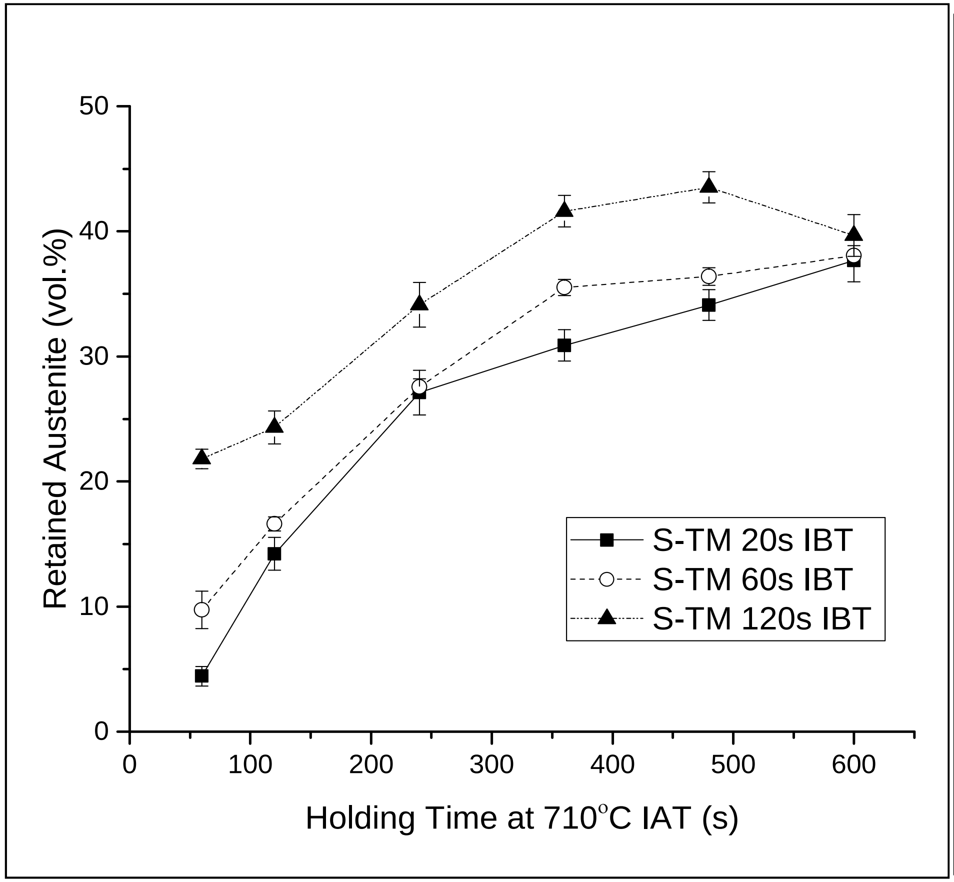
- **Conventional 100 kN tensile frame + ASTM E8M sub-size tensile samples**
- **EDM (electric discharge machining to prevent transformation of retained austenite)**
- **Crosshead speed of 1 mm/min + conventional strain gauge**
- **Interrupted tests comprised stopping the tensile test at $\varepsilon = 0.05$ intervals and performing XRD on sample gauge length cross-sections to determine γ(ret) vs. ε - assessment of mechanical stability/transformation kinetics during deformation**

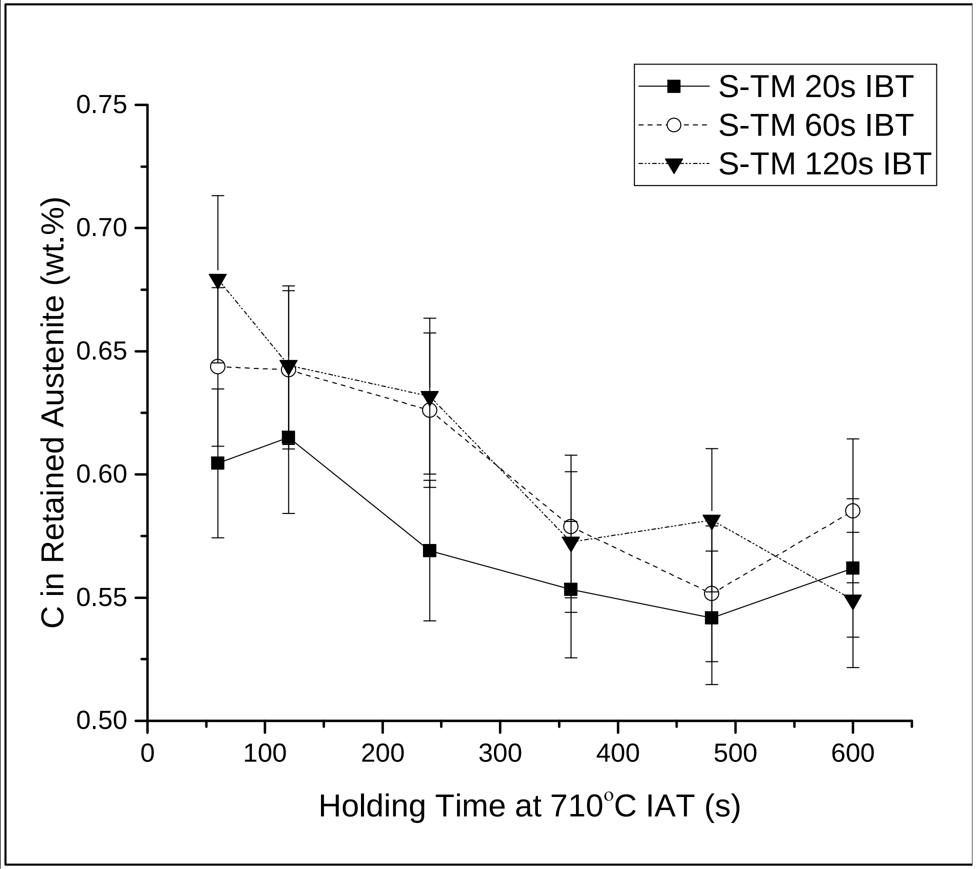
Steel A – Effect of IBT Holding Time



Steel ID.	IAT (°C)	IAT Holding Time (s)	IBT (°C)	IBT Holding Time (s)
S-TM	710	60, 120, 240, 360, 480, 600	460	20, 60, 120
S-M	675	60, 120	460	20, 60, 120

Steel A – Effect of IBT Holding Time (S-TM)

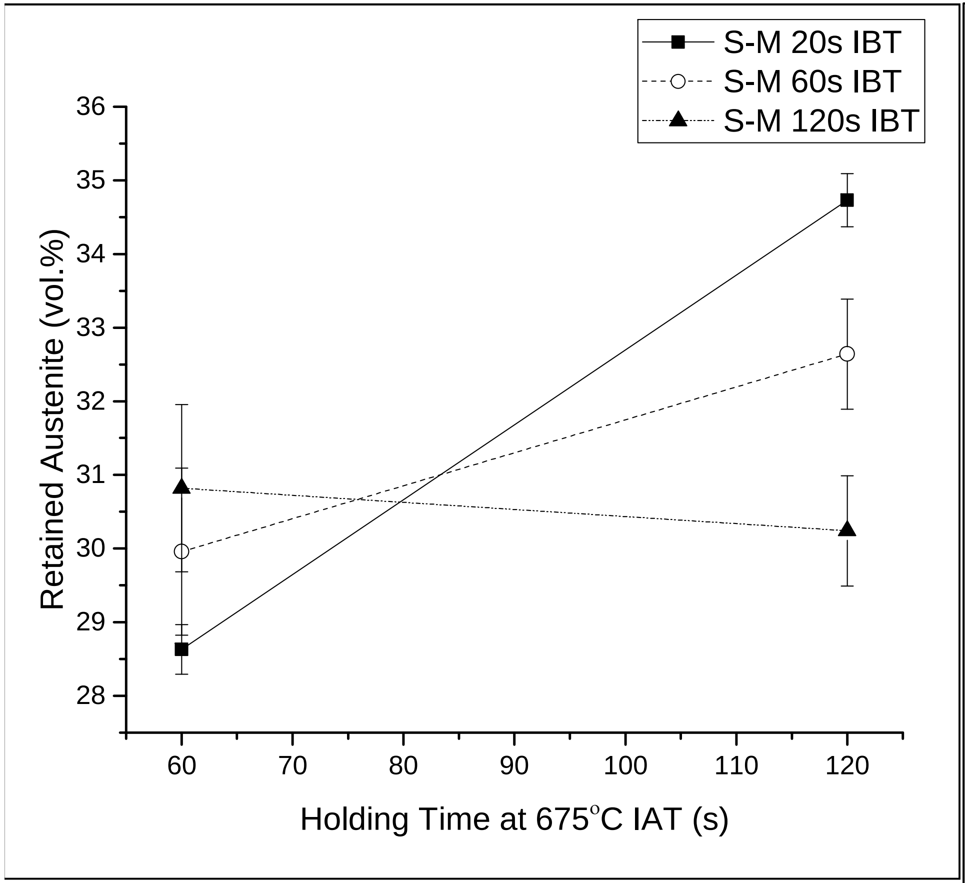


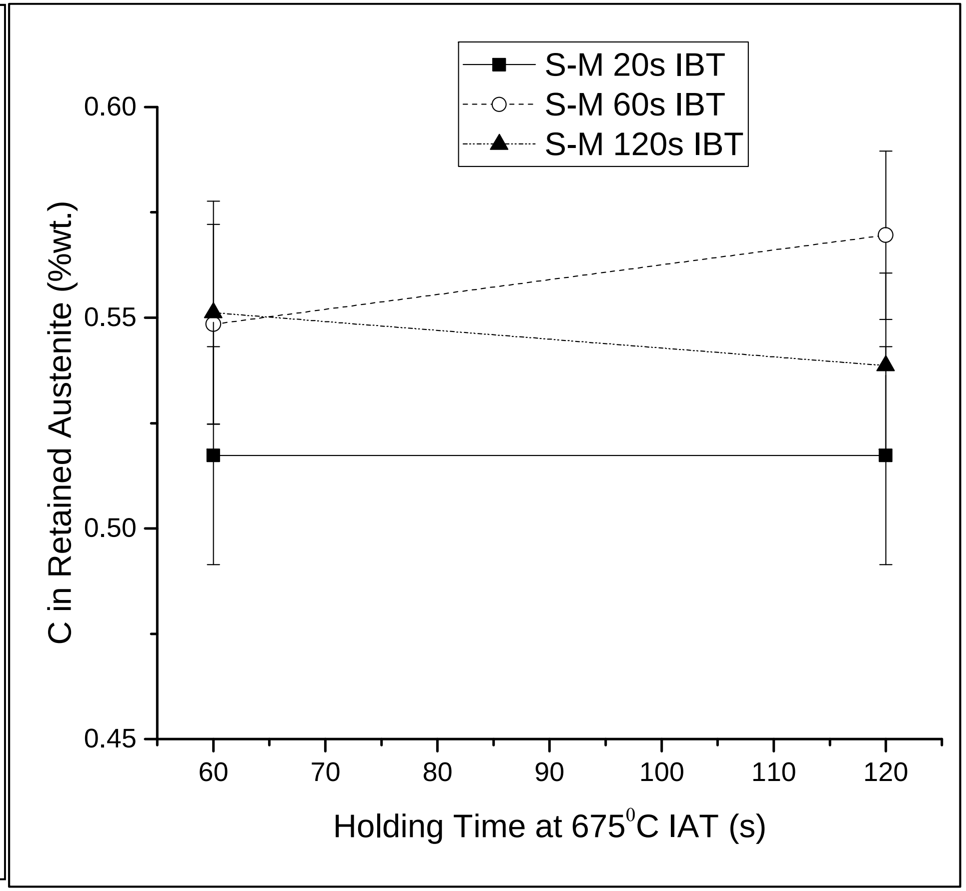


a) b)

XRD results a) retained austenite b) C in retained austenite as a function of IAT holding time for different IBT holding time

Steel A – Effect of IBT Holding Time (S-M)

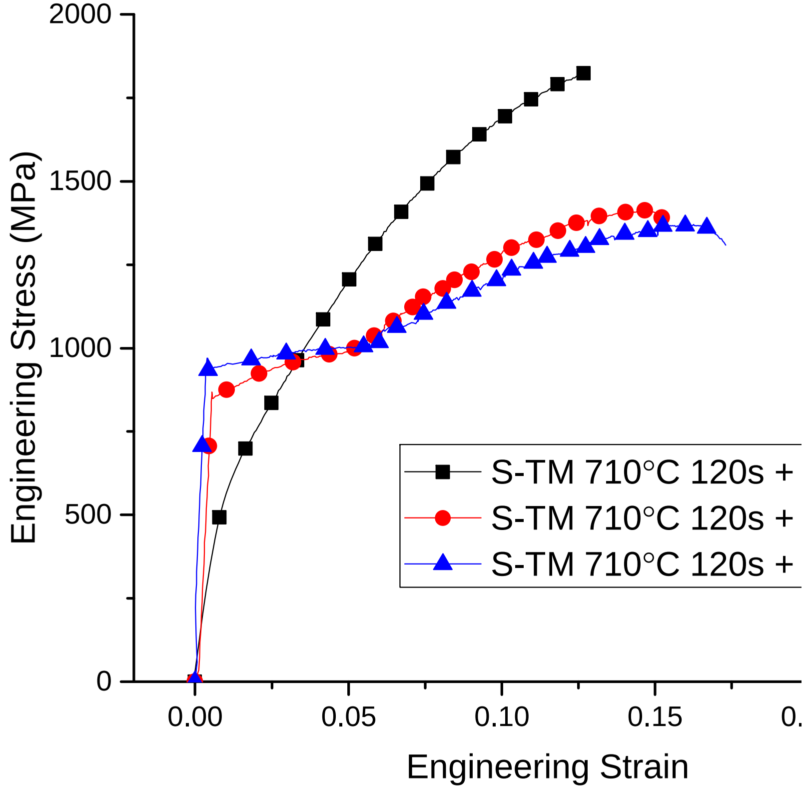


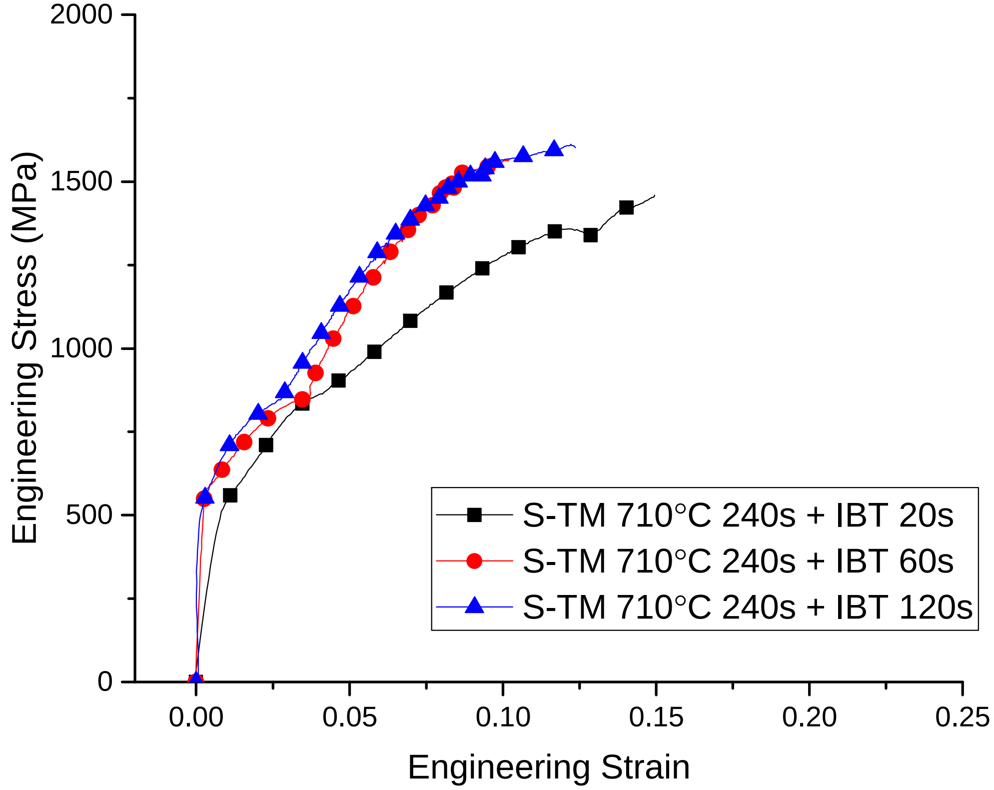


a) b)

XRD results a) retained austenite b) C in retained austenite as a function of IAT holding time at different IBT holding time

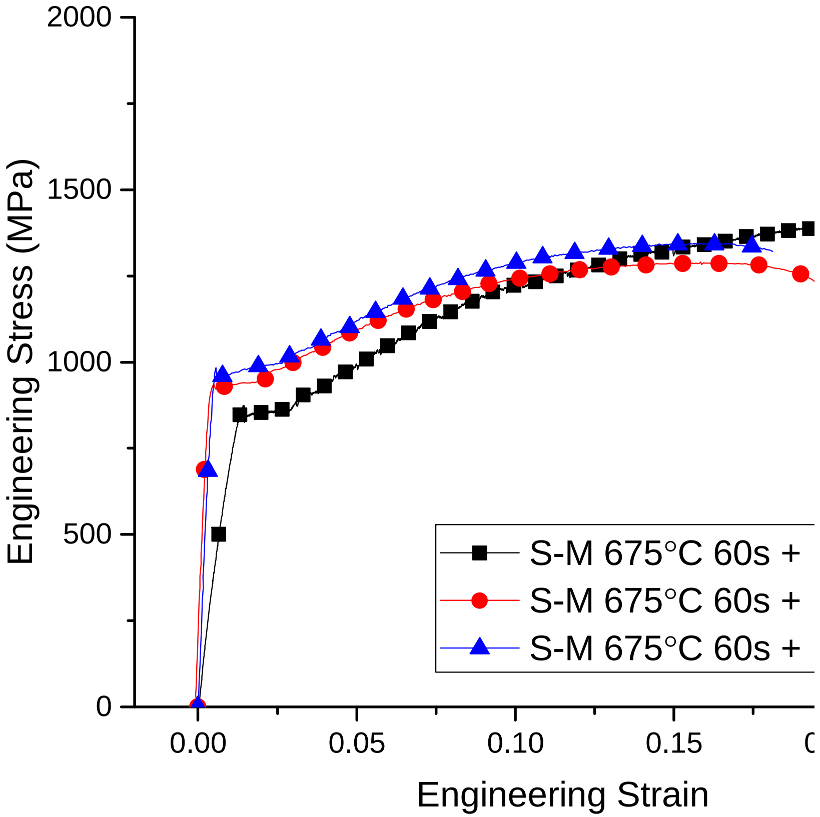
Steel A – Tensile Test Results (Effect of IBT)

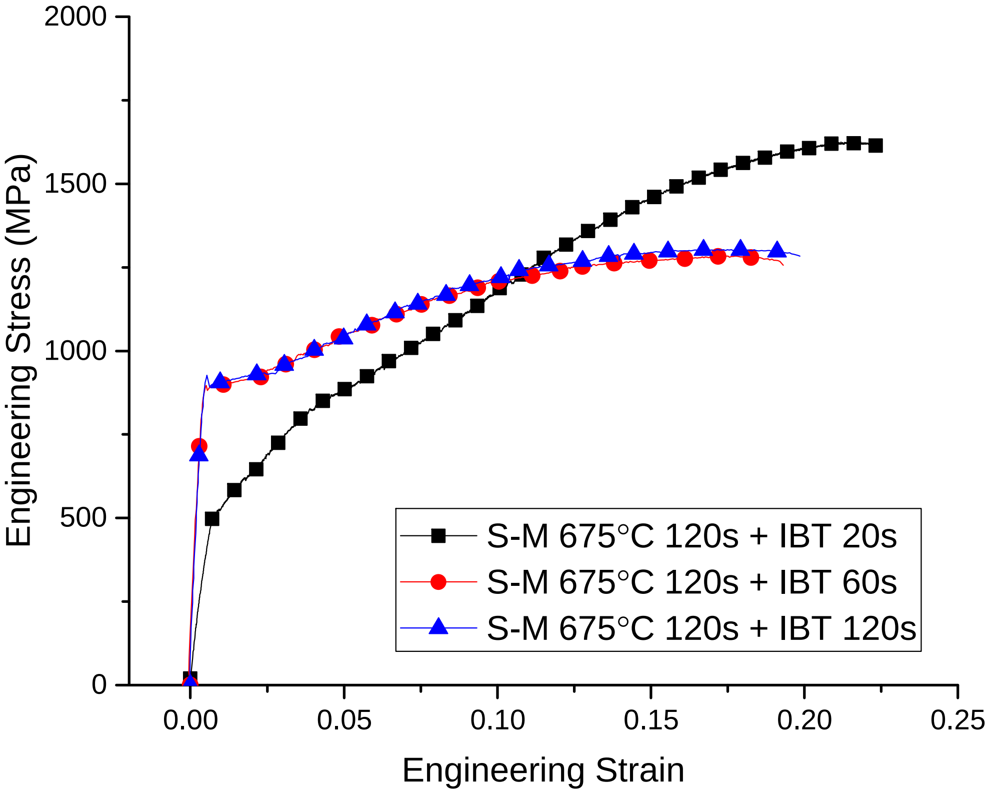




Effect of IBT holding times on tensile properties of S-TM samples annealed at 710°C IAT.

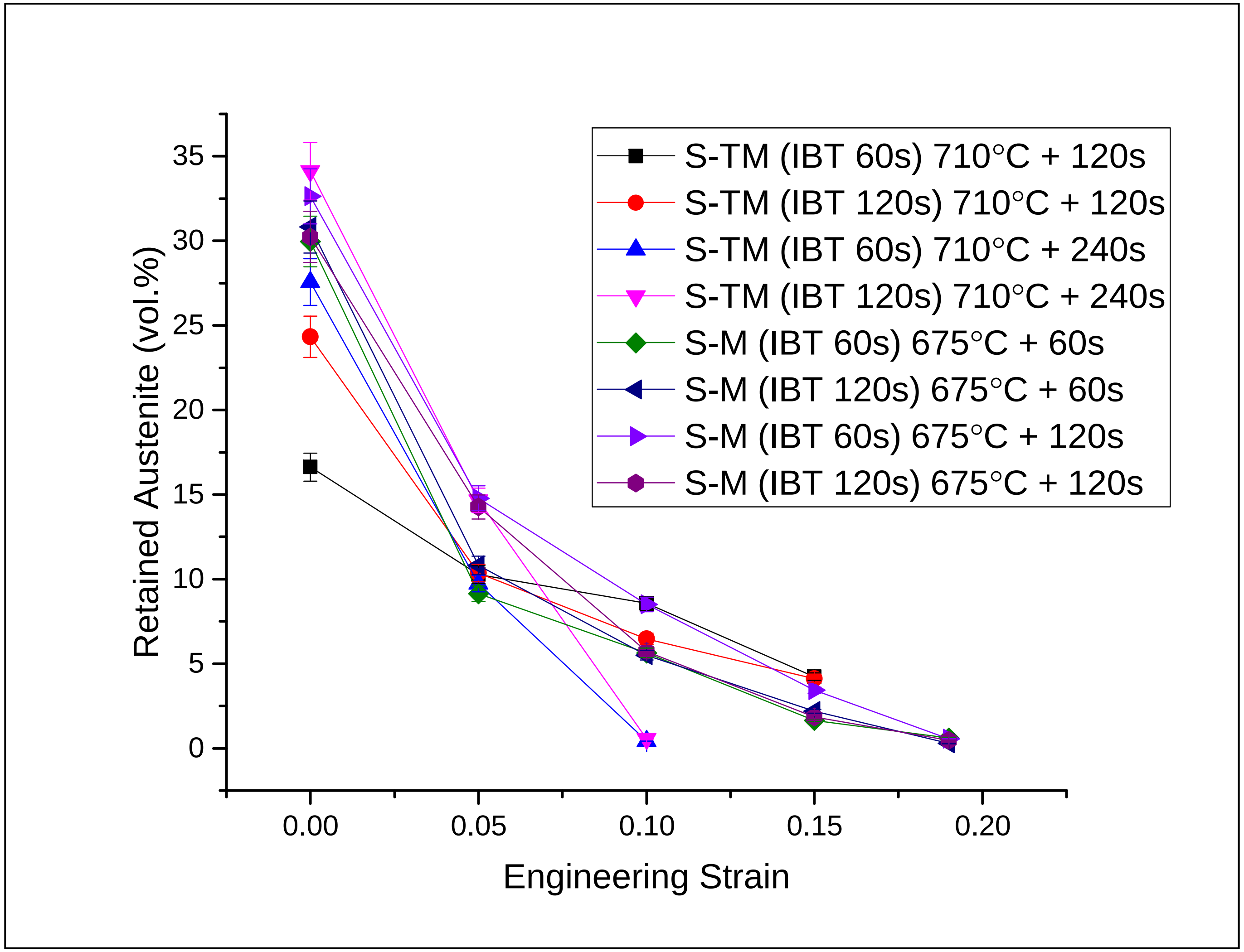
Steel A – Tensile Test Results (Effect of IBT)





Effect of IBT holding times on tensile properties of S-M samples annealed at 675°C IAT.

Steel A – Interrupted Tensile Test Results

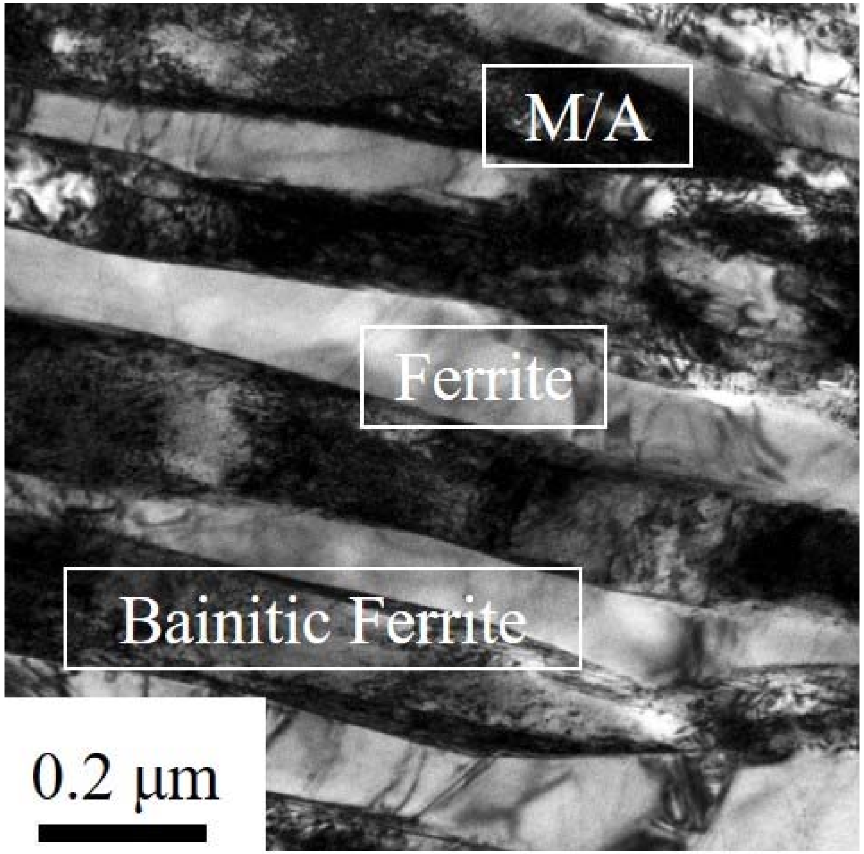


Interrupted tensile test results for different IBT holding times.

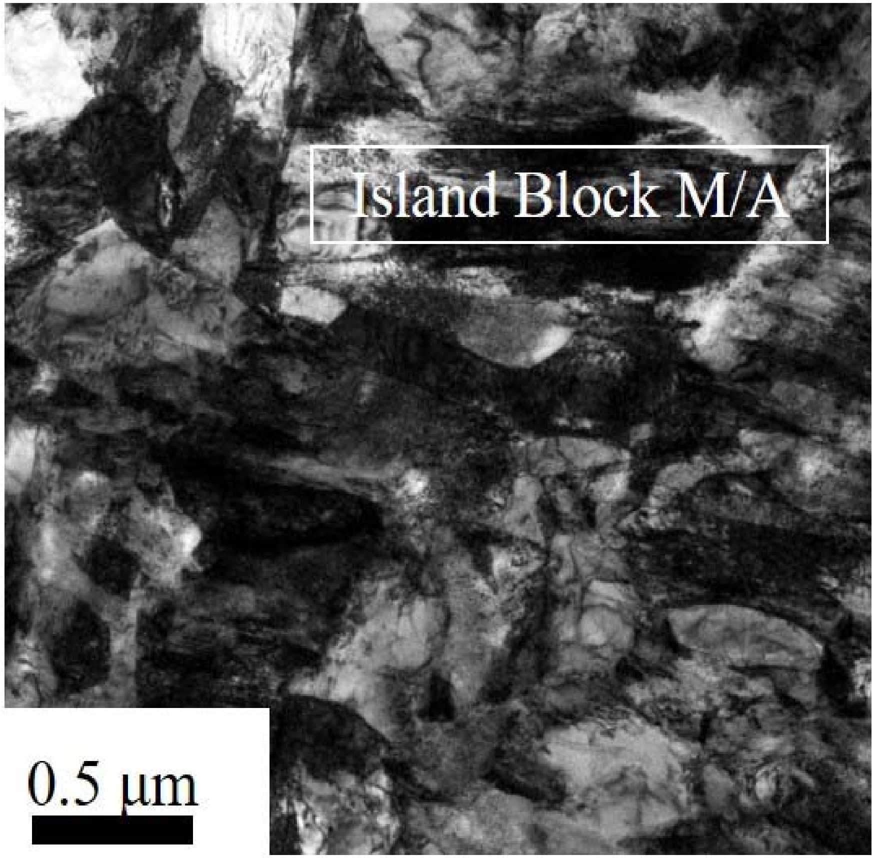
Steel A – Tensile Test Results (Effect of IBT)

Sample ID	Yield Strength (MPa)	Ultimate Tensile Strength, UTS (MPa)	% elongation	UTS × % elongation (MPa %)
S-TM(20s) 710°C +120s	600	1828	11.4	20,839
S-TM(60s)710°C+120s	859	1409	15.5	21,839
S-TM(120s)710°C+120s	960	1370	17.5	23,975
S-TM(20s) 710°C +240s	540	1458	13.6	19,828
S-TM(60s)710°C +240s	535	1566	10	15,660
S-TM(120s)710°C +240s	510	1612	12	19,344
S-M(20s) 675°C +60s	830	1465	20.5	30,032
S-M(60s) 675°C +60s	922	1290	19	24,510
S-M(120s) 675°C +60s	960	1345	18	24,210
S-M(20s) 675°C +120s	640	1535	28	42,980
S-M(60s) 675°C +120s	880	1285	19.5	25,057
S-M(120s) 675°C +120s	898	1303	19.5	25,408

Steel A – TEM Analysis



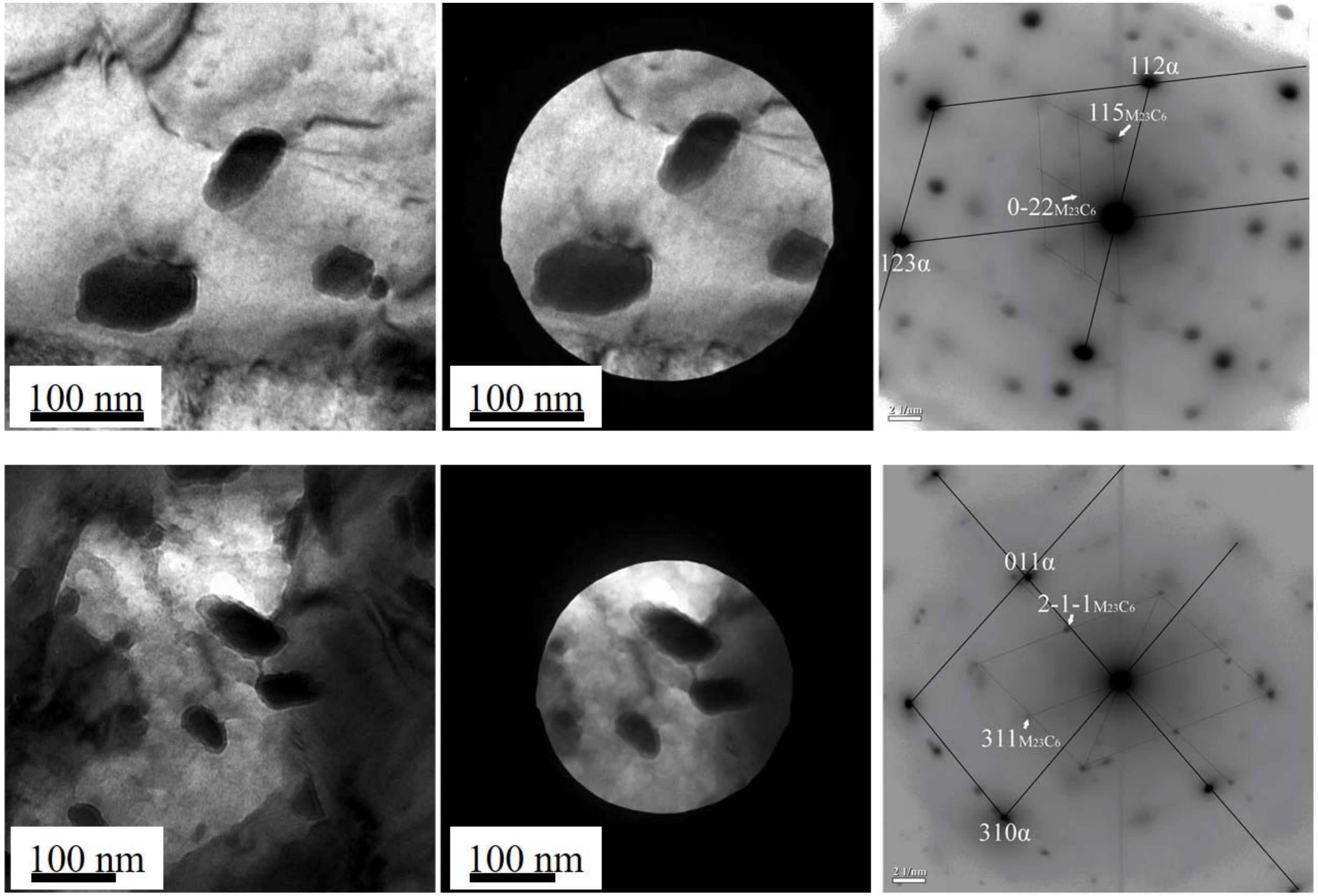
a)



b)

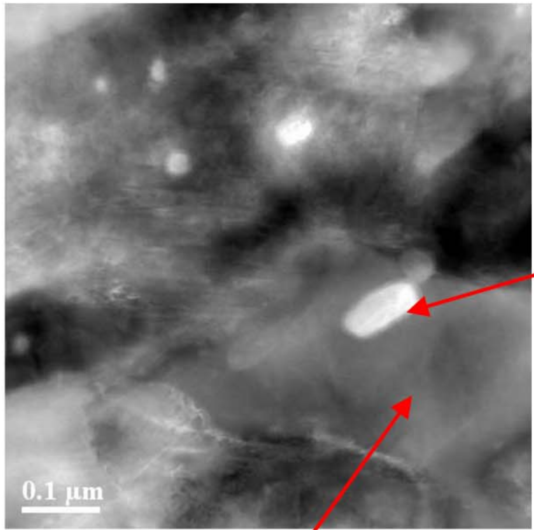
Different morphologies of the phases in a S-M sample annealed at a) 675°C IAT for 480s and b) 710°C IAT for 360s.

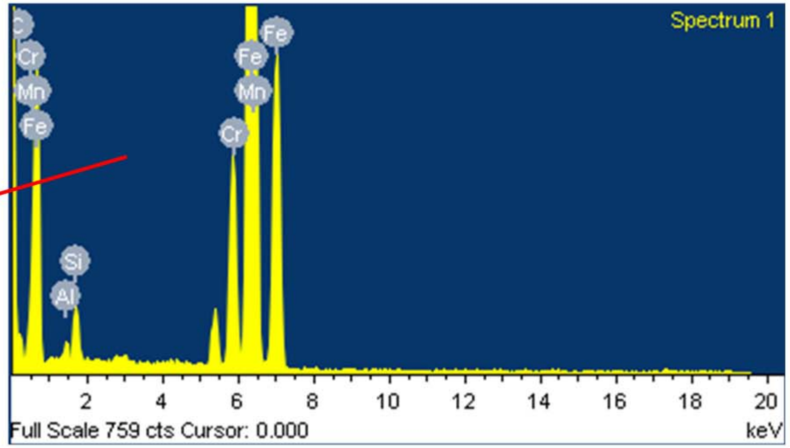
Steel A – TEM Analysis (Carbide Particles)

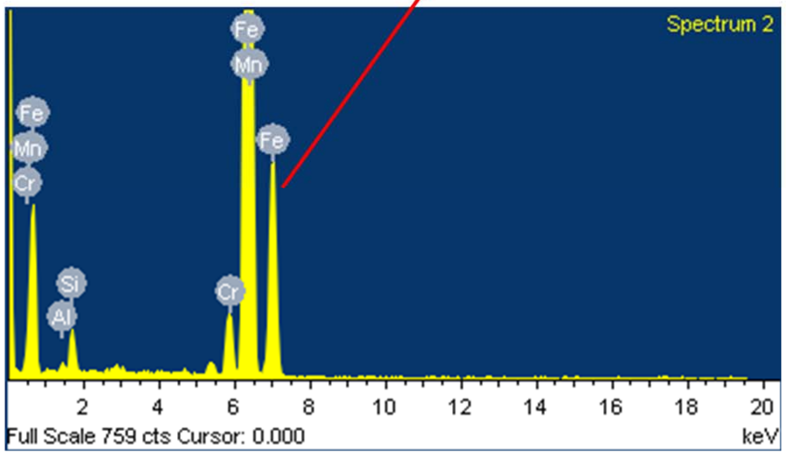


Particles and corresponding SAD patterns of S-M sample annealed at 675°C IAT for 480s.

Steel A – TEM Analysis (Carbide Particles)



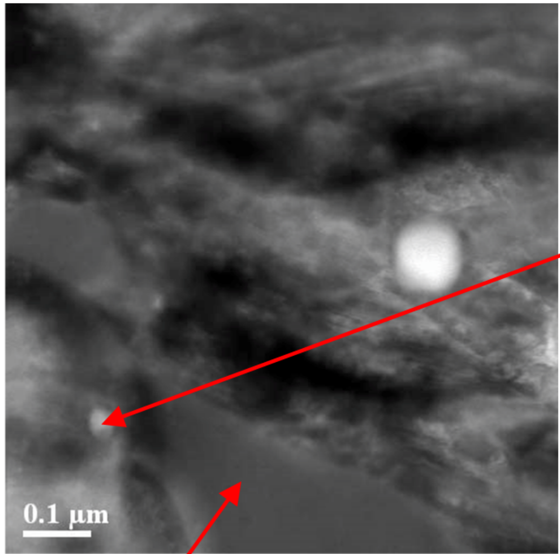


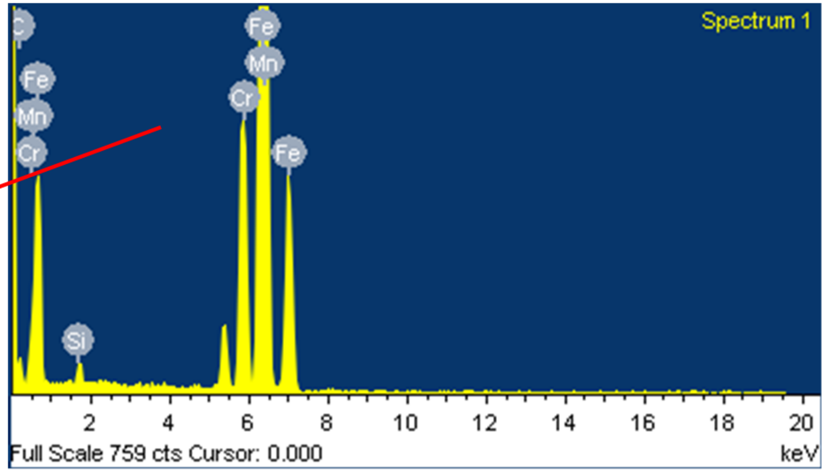


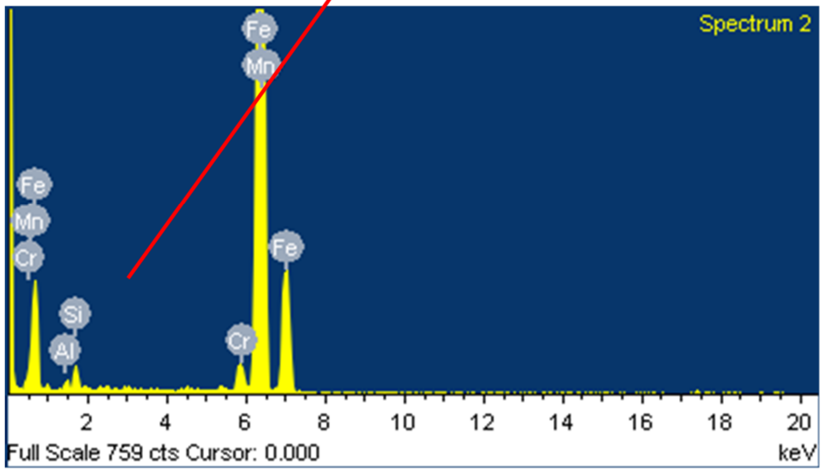
Element	C	Al	Si	Cr	Mn	Fe
Particle (wt. %)	1.65	0.19	0.84	3.92	13.83	79.58
Matrix (wt. %)	0	0.29	1.33	0.49	3.25	94.66

EDS spectra of S-M sample at 675°C IAT for 480s

Steel A – TEM Analysis (Carbide Particles)







Element	C	Al	Si	Cr	Mn	Fe
Particle (wt. %)	1.09	0	0.68	2.79	13.92	81.53
Matrix (wt.%)	0	0.46	1.05	0.38	2.62	95.48

EDS spectra of S-M sample at 710°C IAT for 360s

Steel A – TEM Analysis (Carbide Particles)

Average Composition of Particles and Matrix

Sample \ Element	C	Al	Si	Ti	Cr	Mn	Fe
Particle							
675°C for 480s	1.11	0.28	0.95	0.03	**2.8**	**13.47**	81.34
675°C for 600s	2.88	0.16	1.11	0	**3.49**	**15.75**	76.60
710°C for 480s	0.72	0.28	0.77	0.01	**2.99**	**11.62**	83.60
710°C for 600s	2.87	0.18	0.93	0	**4.29**	**17.26**	74.48

Matrix							
675°C for 480s	0.07	0.33	1.39	0	**0.57**	**3.82**	93.8
675°C for 600s	0.32	0.34	1.91	0	**0.55**	**5.32**	91.56
710°C for 480s	0.02	0.26	0.98	0	**0.54**	**5.09**	93.1
710°C for 600s	0.23	0.52	1.64	0	**0.46**	**4.38**	92.78

Steel A - Summary

- **Retained austenite more sensitive to IBT time than optimal for a TRIP steel**
  - **points towards marginal chemical stability of retained austenite using current thermal processing**
  - **confirmed by tests showing decreased stability of retained austenite with increasing IBT time**
  - **explore lowering IBT time to increase chemical and mechanical stability of retained austenite**
- **Film-like nature of retained austenite from martensitic starting microstructures is likely key to current stability and properties**

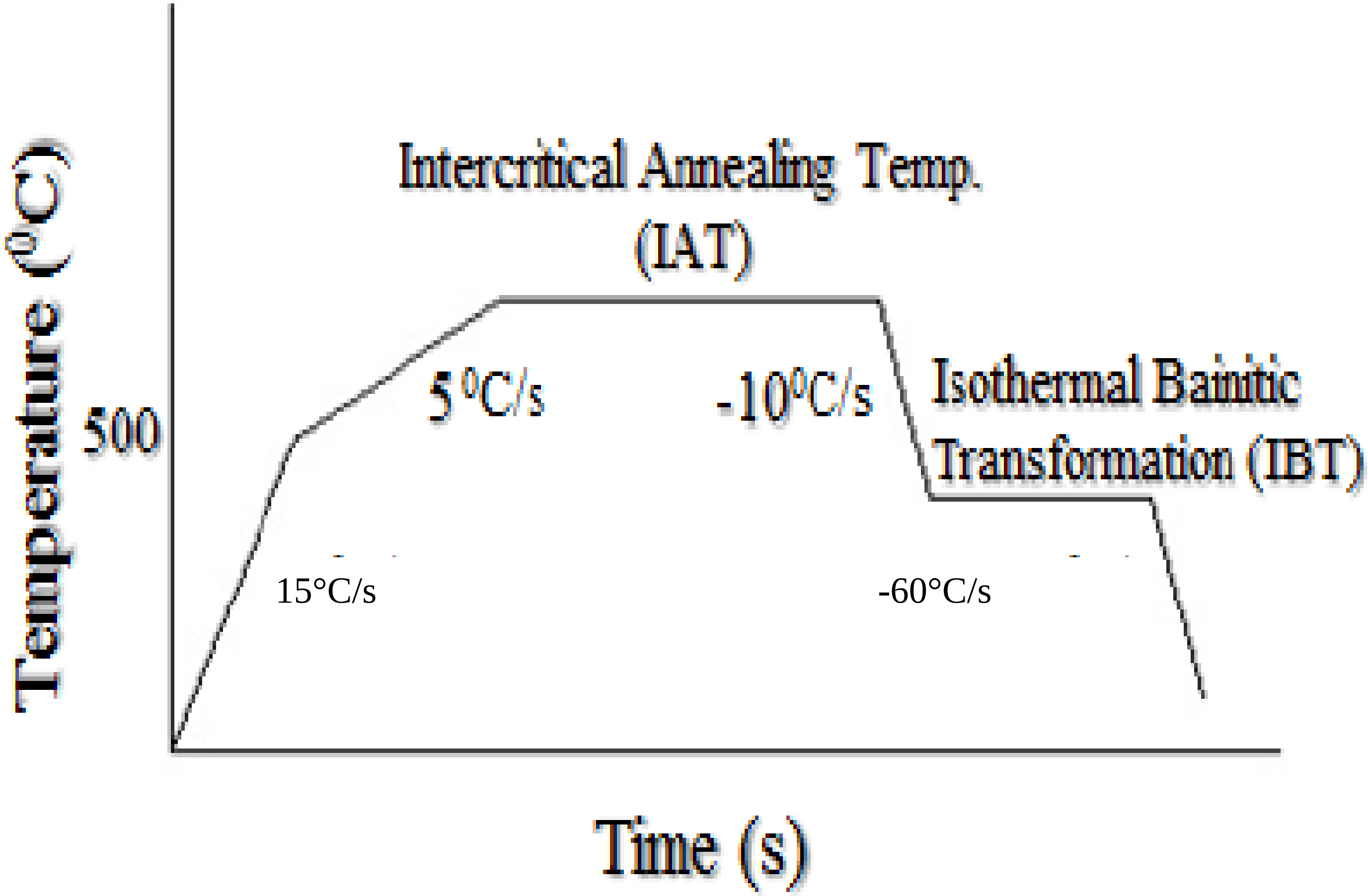
Steel A – Future Work

- Complete TEM for fine scale microstructure analysis and for longer IBT times – on-going
- TEM analysis to determine the effect of stacking fault energy on plasticity enhancing mechanisms (TWIP effect?) for short IBT time (20s) samples
- Effect of lower IBT temperature (420°C) on retained austenite evolution and stability – increase process robustness
- Selective oxidation and reactive wetting tests
- Complete study on elongation vs. sample geometry with U.S. Steel R&D
- Kazi to write and defend thesis – PhD start in January 2017

Steel E – Current Status

- Three samples have achieved the required mechanical property targets:
  - AR-665°C-120s (31,200 MPa%)
  - AR-665°C-240s (33,600 MPa%)
  - MF-665°C-240s (30,900 MPa%)
- Revised heat treatment matrix for XRD/SEM coupons is complete
  - Many samples have achieved ~30% retained austenite
- Selected heat treatments were performed on panels for tensile samples
  - Panels have been sent for machining, tensile tests to be performed by the end of October

Steel E – Thermal Profile



- **IBT of 30s at 460°C for all samples.**

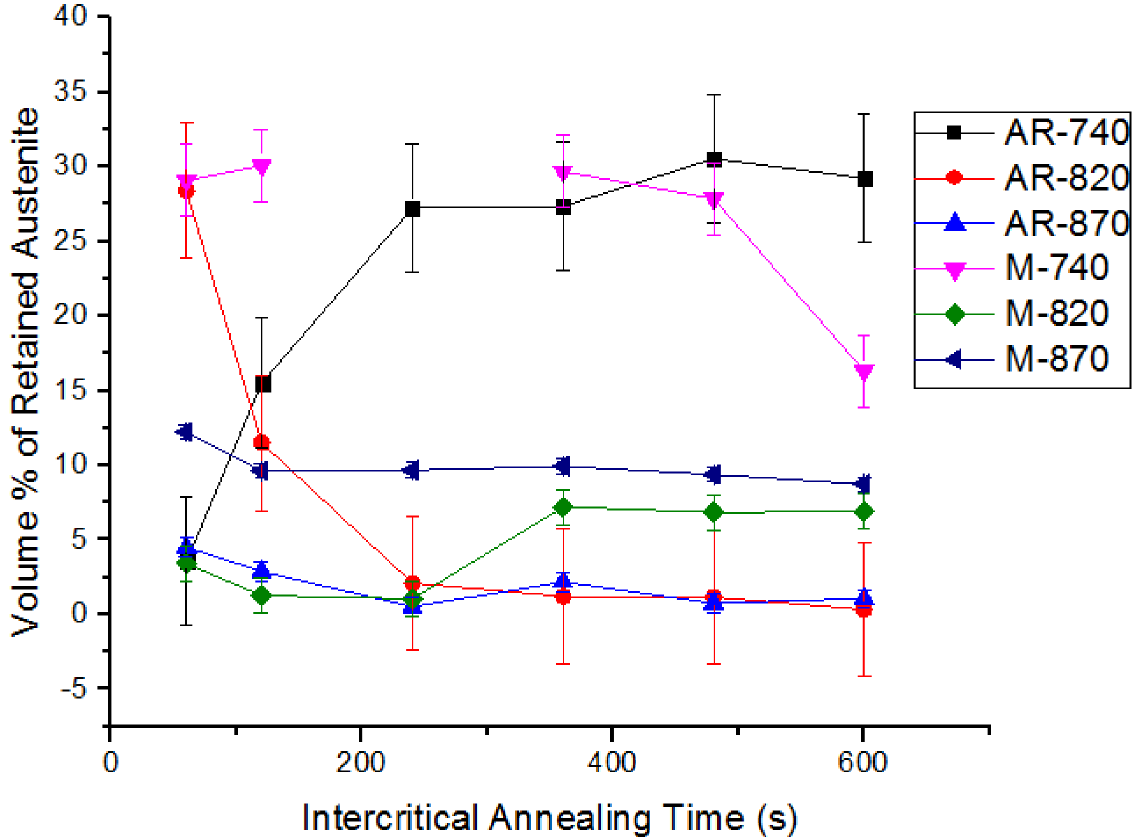
Steel E – Experimental Plan

Target Vol% of Intercritical Austenite	Intercritical Annealing Temperature (°C)	Intercritical Annealing Time (s)
25	740	60, 120, 240, 360, 480, 600
50	820	60, 120, 240, 360, 480, 600
75	870	60, 120, 240, 360, 480, 600

- **IBT of 30s at 460°C.**
- **All treatments performed on as-received and martensitic starting microstructures.**
- **Martensitic structure obtained by annealing at 890°C for 10 minutes and then quenching.**

Steel E – Retained Austenite Content

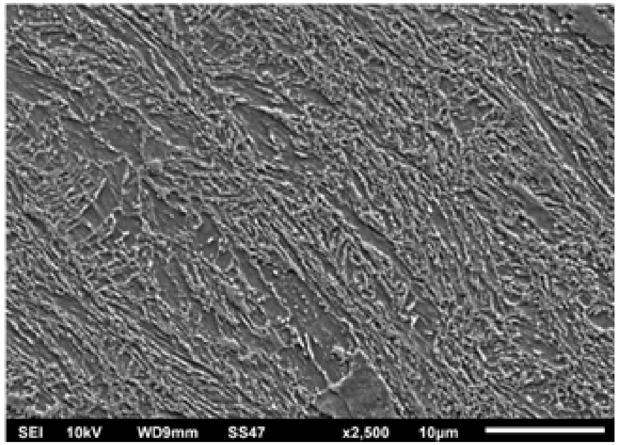
- **Promising results for samples annealed at 740°C.**
  - **Longer times required for AR samples.**
- **IA temperatures of 820°C and 870°C likely too high to achieve target.**



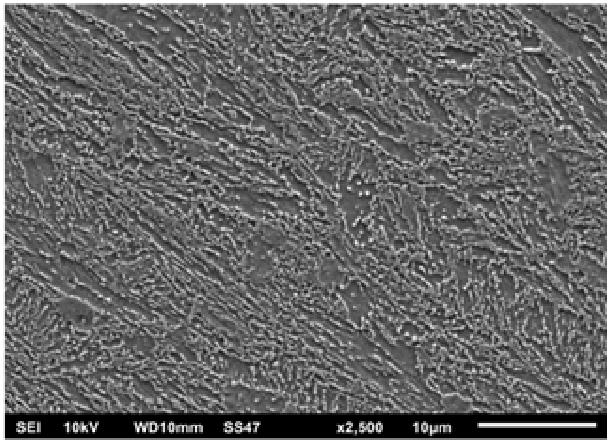
Steel E – Microstructural Analysis

➢ AR-740 series

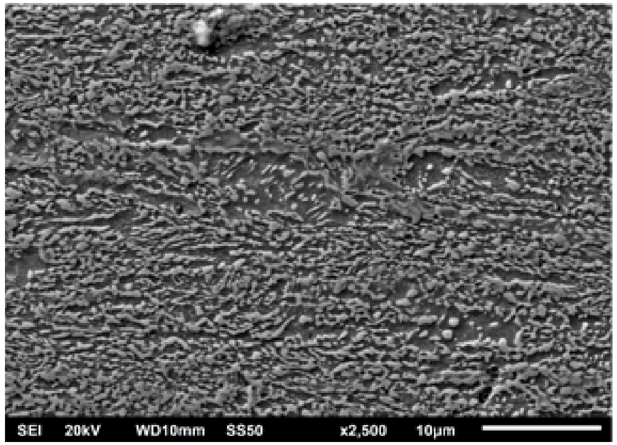
AR-740-60



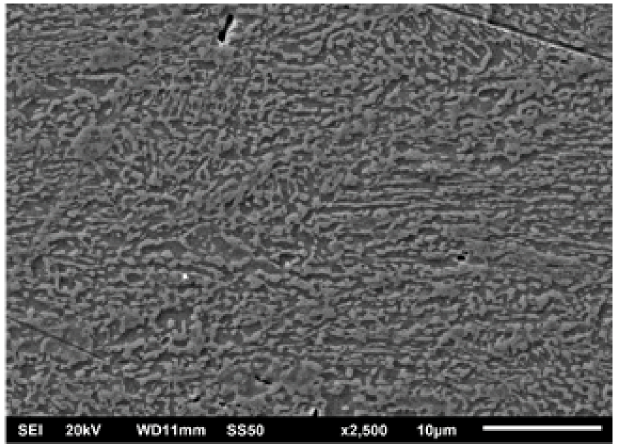
AR-740-120



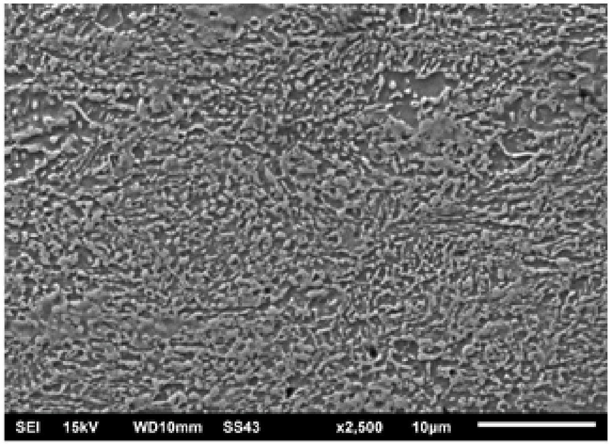
AR-740-240



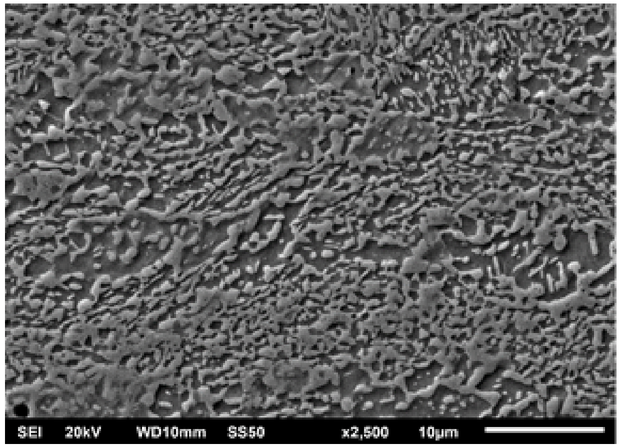
AR-740-360



AR-740-480



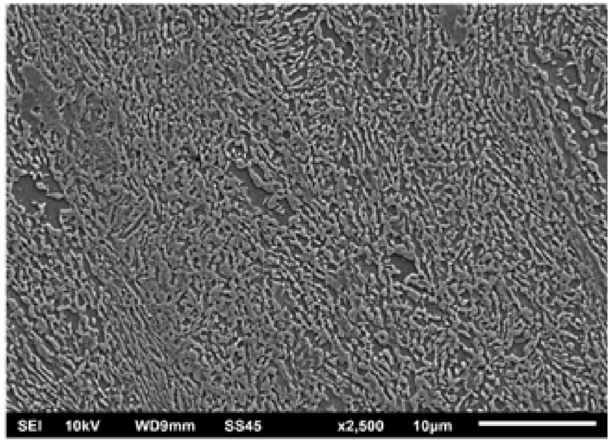
AR-740-600



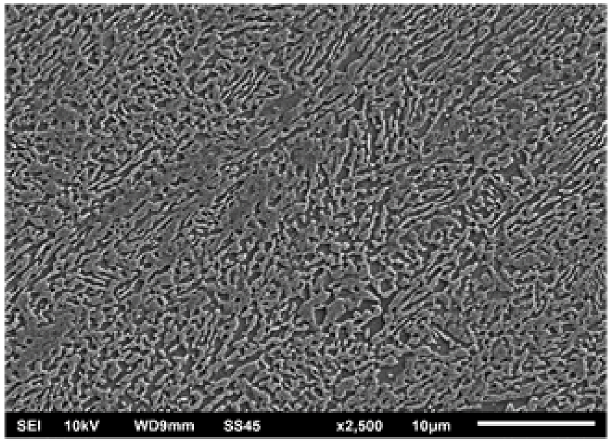
Steel E – Microstructural Analysis

- **AR-820 series**

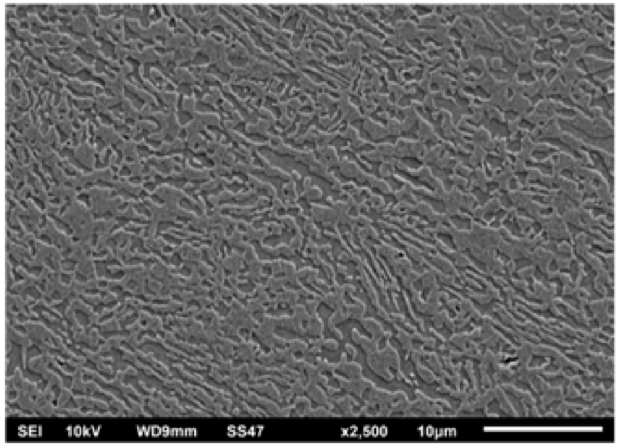
AR-820-60



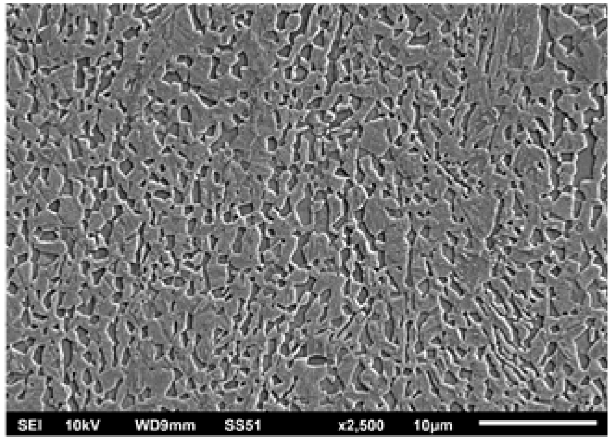
AR-820-120



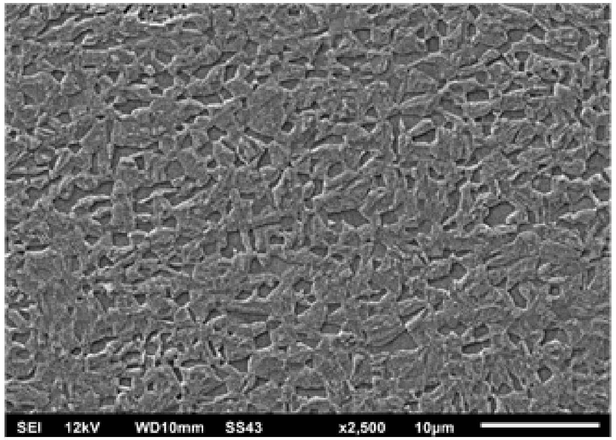
AR-820-240



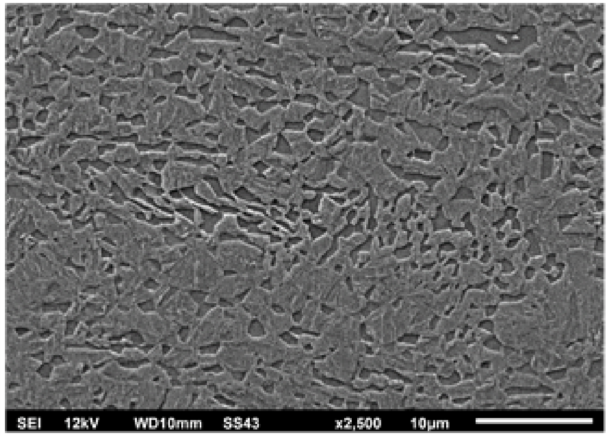
AR-820-360



AR-820-480



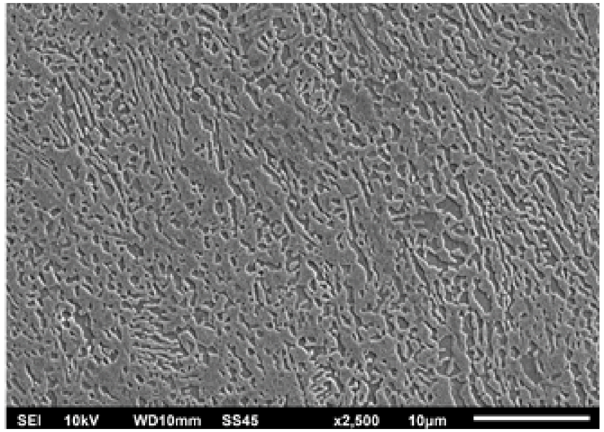
AR-820-600



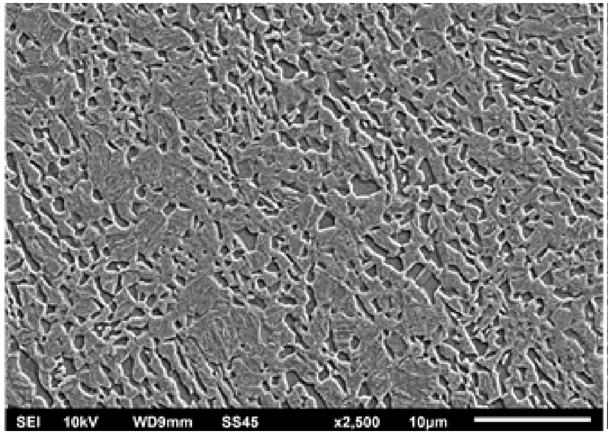
Steel E – Microstructural Analysis

- **AR-870 series**

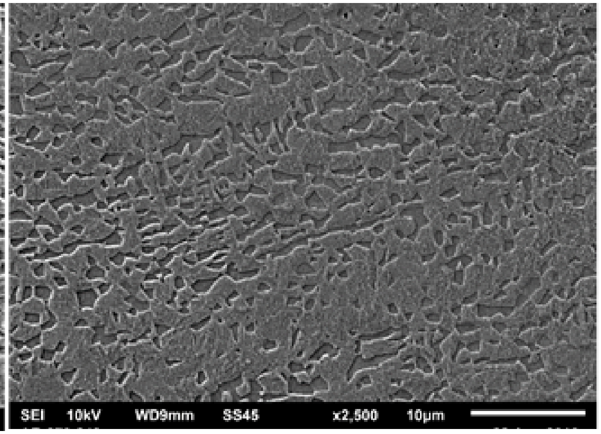
AR-870-60



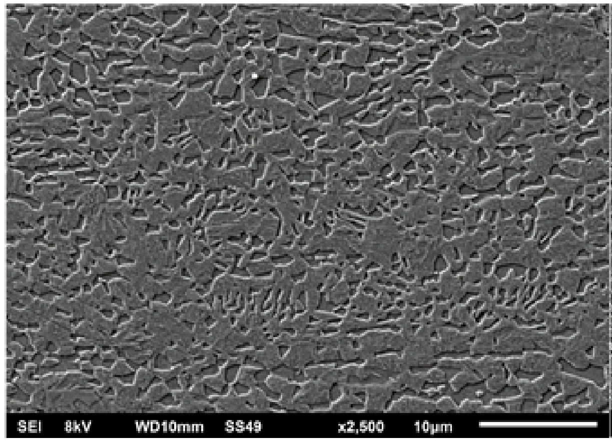
AR-870-120



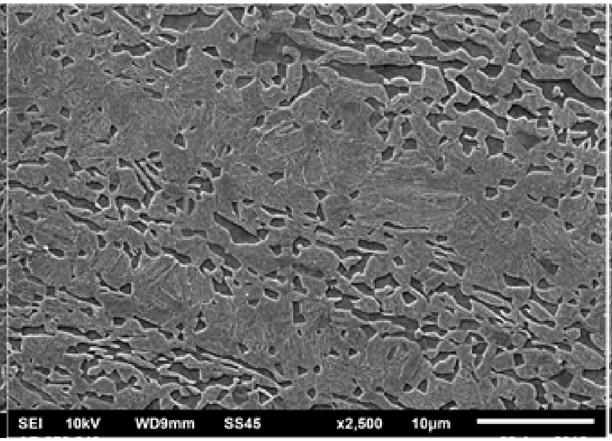
AR-870-240



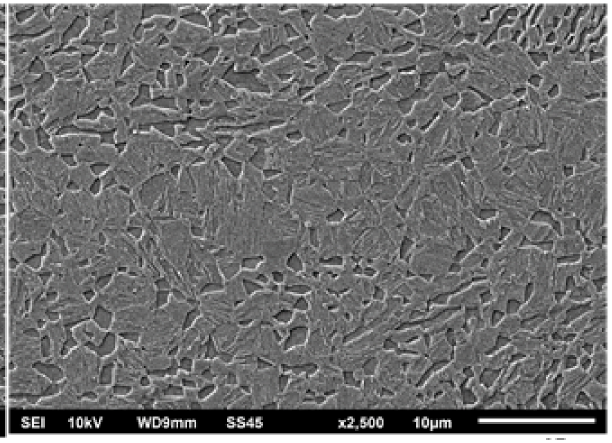
AR-870-360



AR-870-480



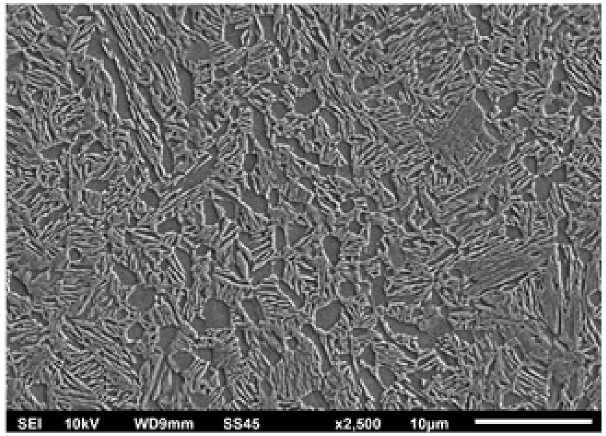
AR-870-600



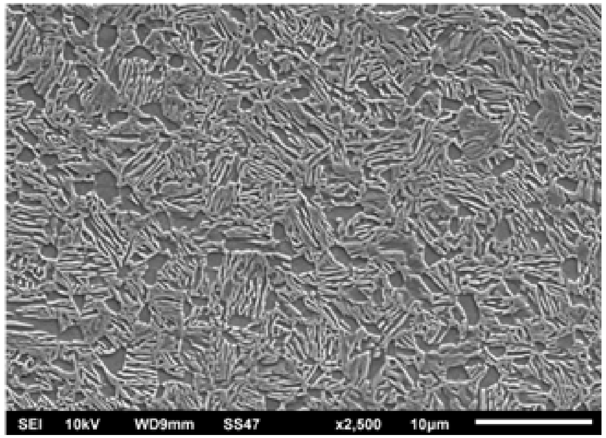
Steel E – Microstructural Analysis

➢ M-740 series

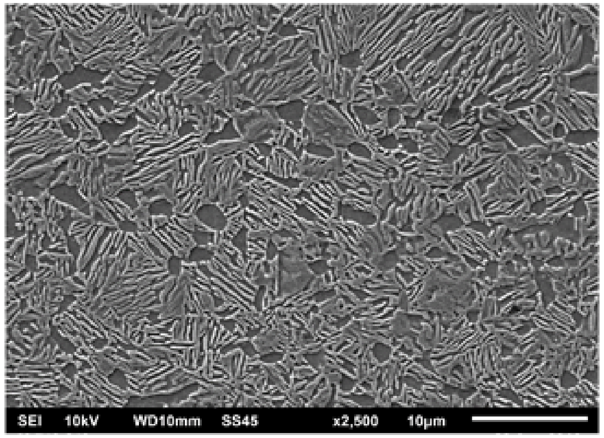
M-740-60



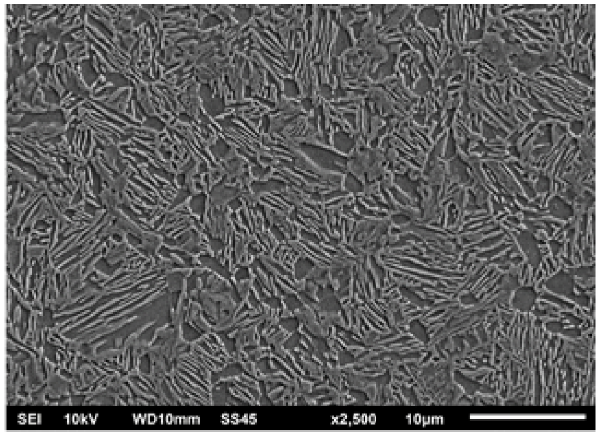
M-740-120



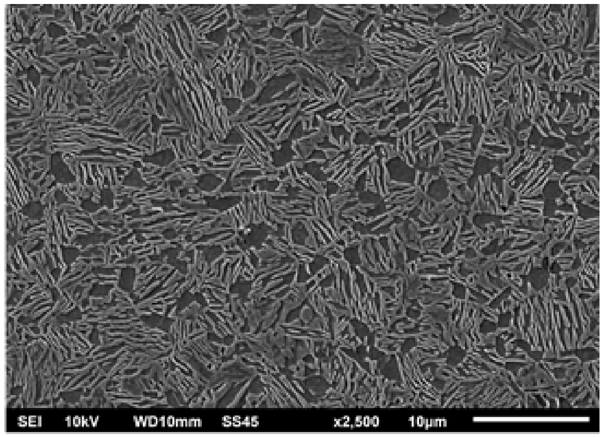
M-740-240



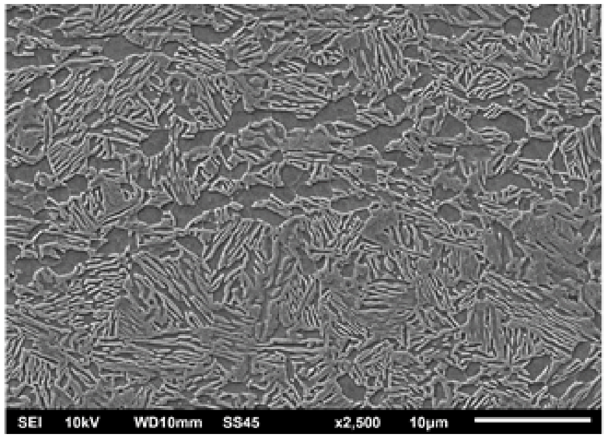
M-740-360



M-740-480



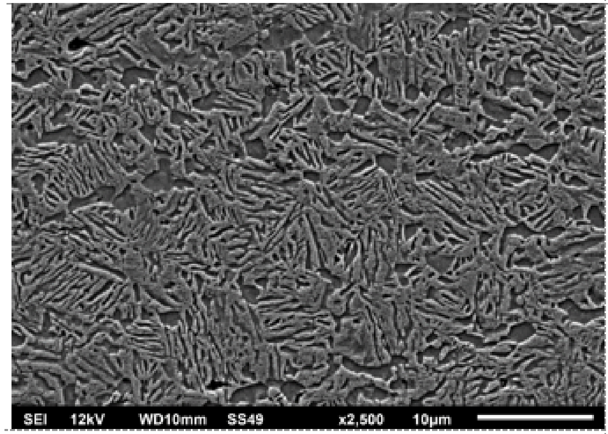
M-740-600



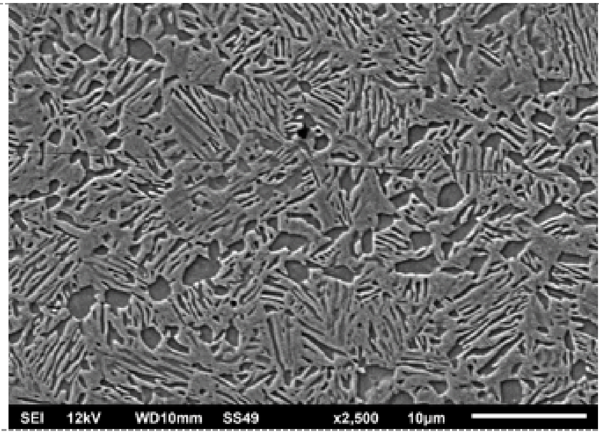
Steel E – Microstructural Analysis

➢ M-820 series

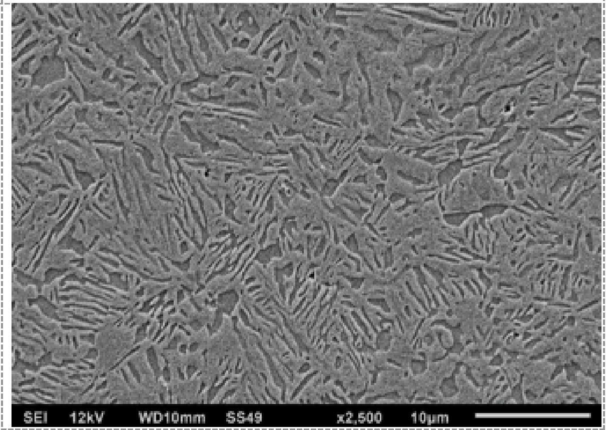
M-820-60



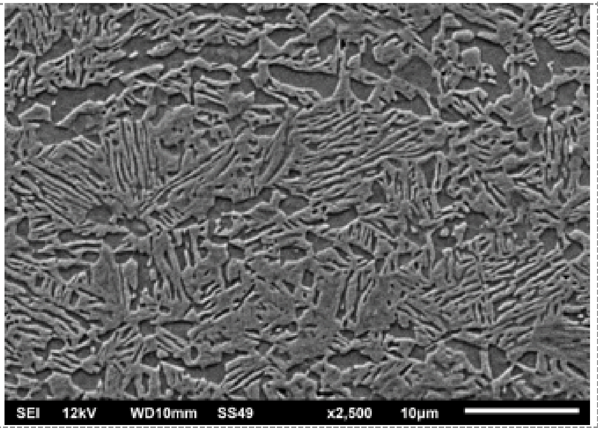
M-820-120



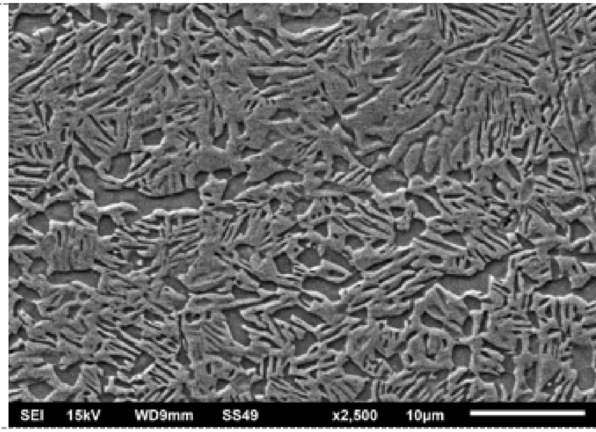
M-820-240



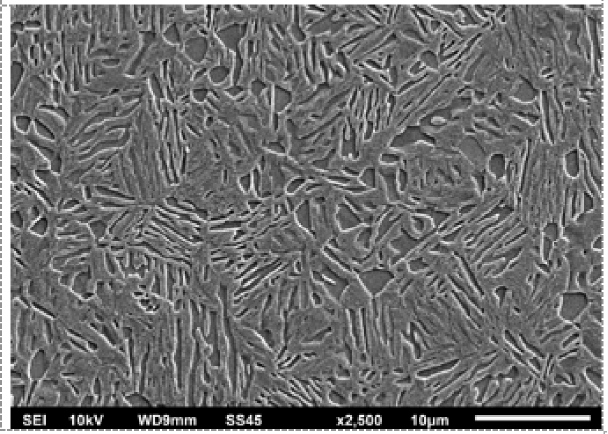
M-820-360



M-820-480

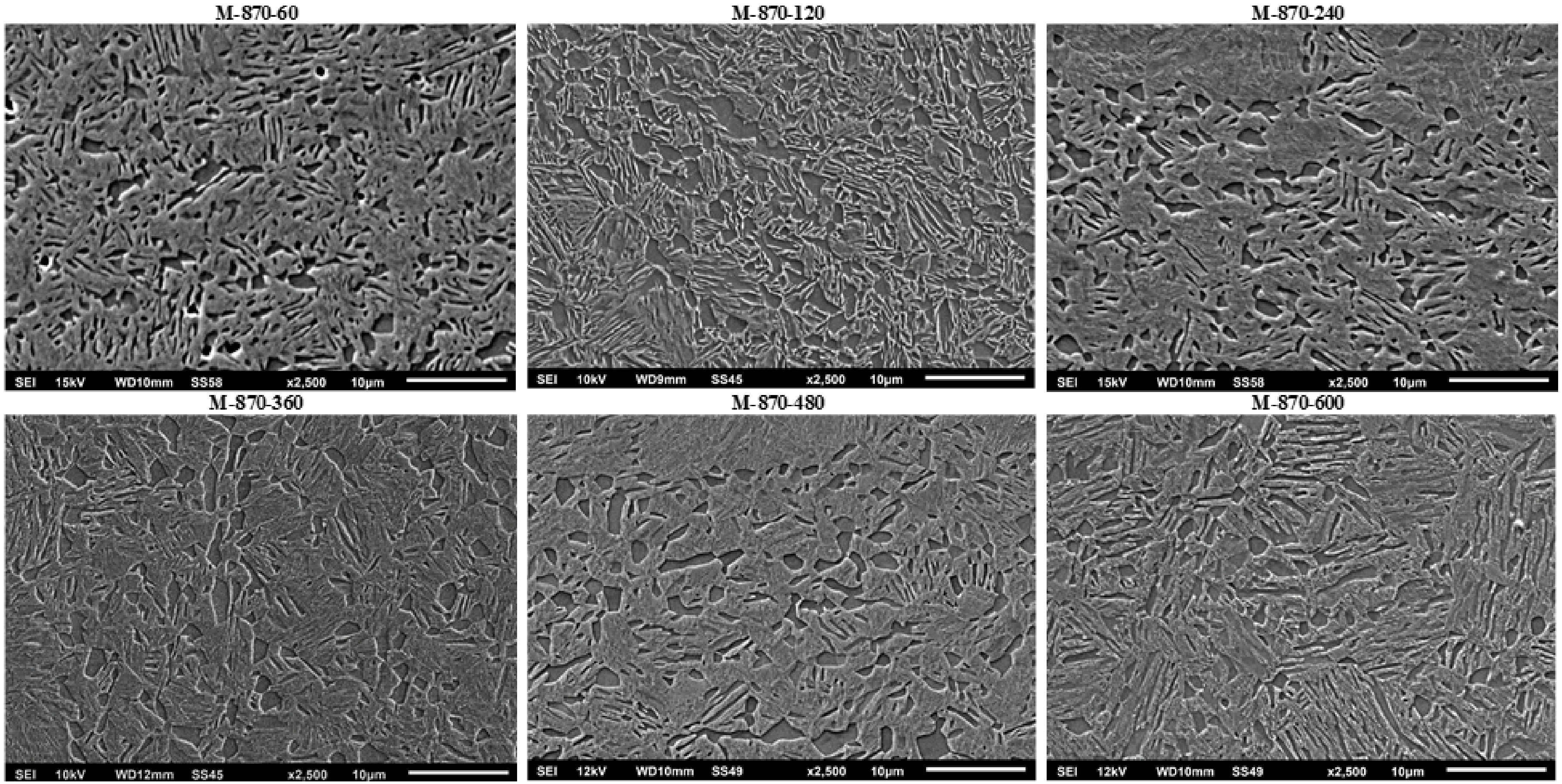


M-820-600



Steel E – Microstructural Analysis

- **M-870 series**



Steel E – Future Work

- Perform tensile tests on the following:
  - AR-740-240/360
  - AR-820-60
  - M-740-60/120/240/360
- Assess mechanical properties vs DOE targets and 3G window of $30{,}000 \le UTS \times \%telong \le 40{,}000MPa\%$
- TEM to evaluate fine scale microstructure/carbide morphology.
- Begin selective oxidation tests of the samples that meet mechanical property targets.