ZCO-79: Internal Oxidation Effects on Forming and Welding Behaviour of Advanced High Strength Steel

C. Thomsen, H. Xu, J.R. McDermid, E. Biro and Y.N. Zhou

Centre for Automotive Materials and Corrosion, McMaster University of Waterloo

McMaster University ENGINEERING

University of Waterloo

May 2018 GAP Meeting

camc Centre for Automotive Materials and Corrosion

Background

- **Resistance spot welding (RSW) is the most widely used sheet metal joining technique in the automotive assembly process**

- **The literature suggests that the use of internal oxidation or oxidation/reduction treatments to enhance the reactive wetting of AHSS by metallic coatings can also significantly affect RSW weld lobe curves**
  - **can dynamically change the electrical resistance – as characterized by the dynamic resistance profile (DRP)**

Background

- The summative literature further suggests that internal oxidation can affect not only the weld lobe curve and weld expulsion, but also mechanical properties and the formability of the resultant materials – needs investigation
- Global objective: determine the effect of various CGL treatment processes resulting in internal oxides on the RSW weld lobe curve and user properties of galvanized AHSSs

Specific Task Objectives

1. Quantify the degree and chemistry of the internal oxides generated during selected galvanizing pre-treatments consistent with obtaining high quality GI coatings.
2. Determine the effect of internal oxidization on RSW weld lobe curves and weld nugget expulsion.
3. Determine the static resistance and dynamic resistance profiles during the RSW of AHSS using the above galvanizing pre-treatments and correlate these with characteristics of the internal oxides present.
4. Evaluate the mechanical properties and formability of the welded coupons as a function of the various galvanizing surface pre-treatments in AHSS welds.

Research Tasks + Timeline

	M 1 – 6	M 6 – 12	M 12 – 18	M 18 – 24	M 24 – 30	M 30 – 36
Task 1a: Literature survey						
Task 1b: Solicitation, delivery and characterization of substrates from sponsors						
Task 1c: Thermal processing of substrates + characterization						
Task 2: Internal oxidation of the substrates + characterization						
Task 3: Weld lobe and expulsion behaviour						
Task 4:Static and dynamic resistance:						
Task 5: Mechanical properties						
Writing and submission of NSERC-CRD grant						
GAP Meeting Reports						
Mid-term and Final Reports						

Current Status – Project Staffing + CRD

- Han Xu (Waterloo) was hired to work on the welding portion of the project (Tasks 3, 4, 5) for his MASc project
- Chris Thomsen worked on this project for his senior thesis, but has subsequently decided to move to the ZCO-70 project
  - will move forward with a mixture of summer students and PDFs until a MASc student can be hired
- This project also comprises part of the larger NSERC CRD submitted in February 2018.

Current Status: Experimental Materials

- **Two industrial substrates have been recently received – two Mn-Si substrates with varying Mn and Si contents**
  - **steel A: Fe-0.05C-0.54Mn-0.47Si**
  - **steel B: under analysis, but reported to be higher Si**
- **We are currently working with two 0.1C-2Mn-xSi model alloys – x = 0.7, 1.8wt%**
  - **would require additional substrate if we were to move forward using these materials**

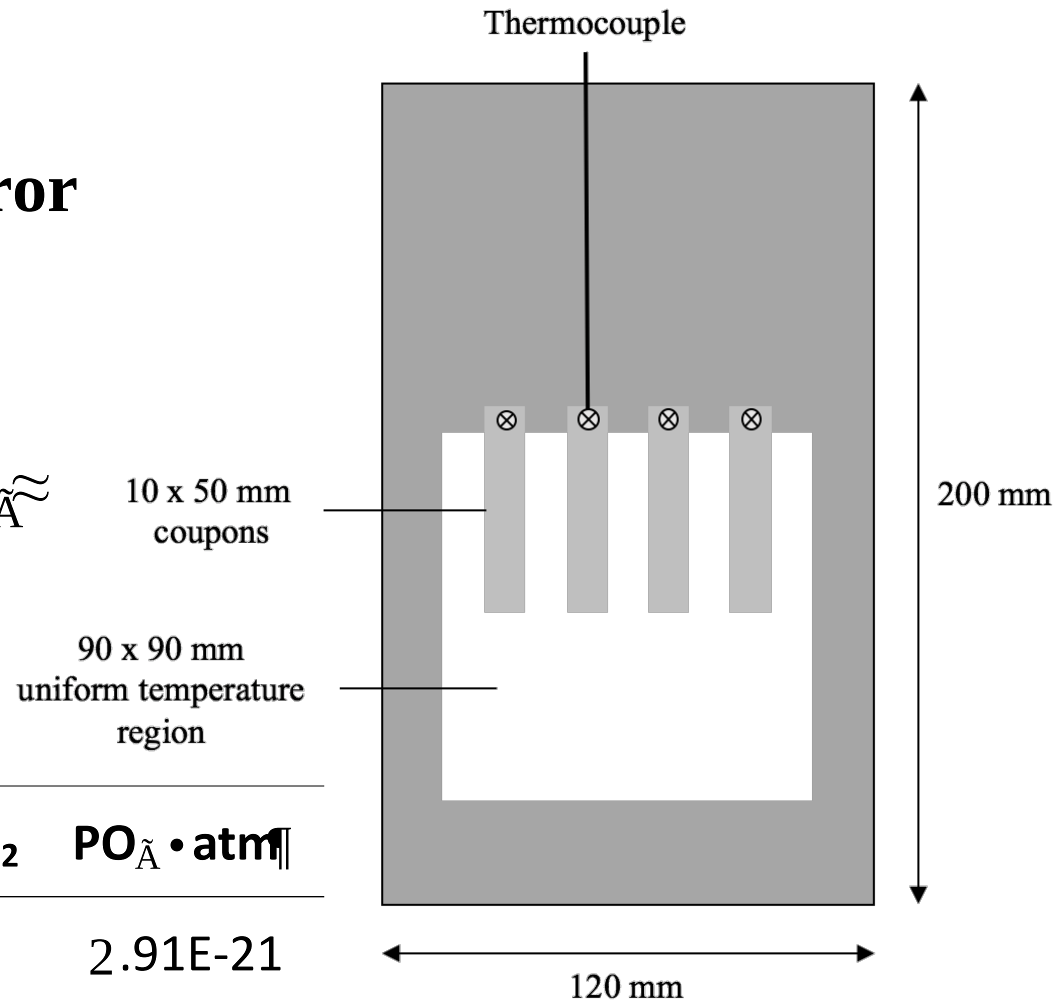
Task 1 and 2 – Materials

- **Two model Mn-Si alloys**
  - **9019: 2Mn-0.7Si**
  - **9088A: 2Mn-1.8Si**

Grade	C	Mn	Si	Al	Ti	N	S	Fe
9019	0.102	1.960	0.701	0.045	0.010	0.004	0.002	Bal
9088A	0.105	2.029	1.757	0.041	0.010	0.003	0.002	Bal

Task 2 – Selective Oxidation Experiments

- **10 x 50 mm coupons cut using waterjet**
- **Polished one side to mirror finish**
- **McMaster Galvanizing Simulator (MGS)**
- **All tests were run in a NÃ≈ 5%H_2 environment at a dew point of -5°C**

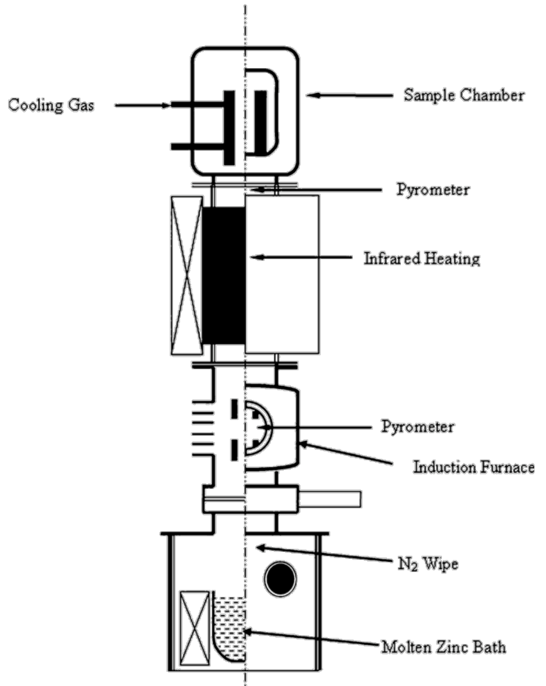


IAT (°C)	Dew point (°C)	pH_2O/pH_2	POÃ • atm¶
805 or 837	,4	0.083	2.91E-21

Experimental Parameters

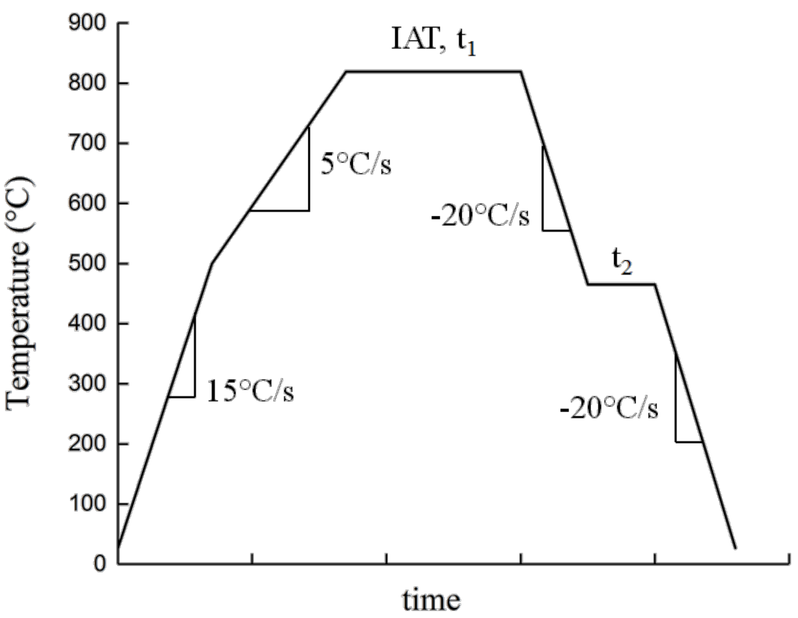
- Seven holding times: **30, 60, 90, 120, 240, 420, 600** seconds

McMaster Galvanizing Simulator



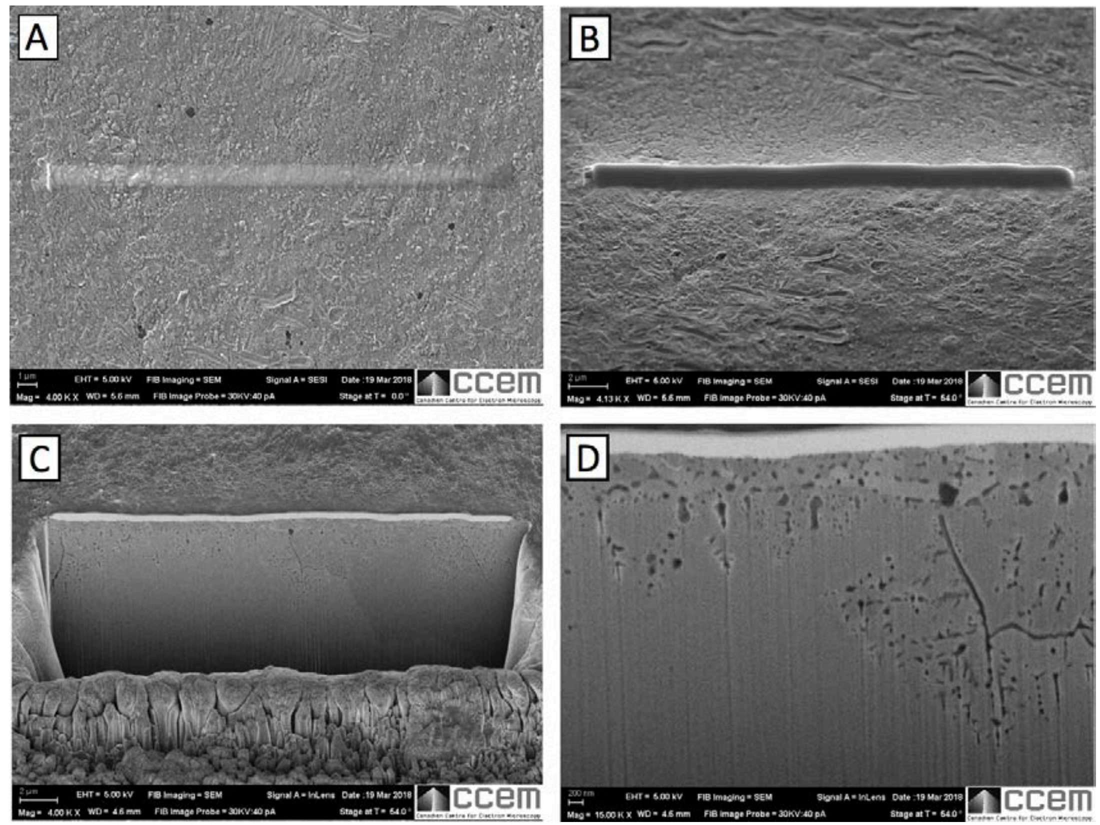
Heat treatment schematic

- 9088A IAT = 837 °C
- 9019 IAT = 805 °C



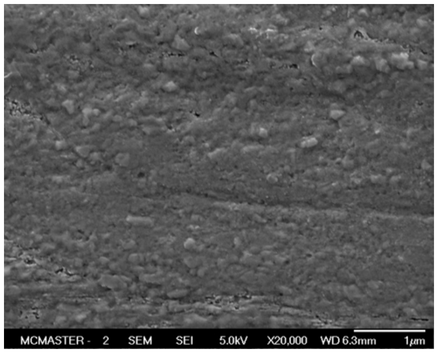
Methods – FIB trench cuts

- A: site of interest is selected
- B: tungsten “cap” is deposited
- C: Gallium ions bombard the surface to mill a smooth cross section (25 µm wide)
- D: higher mag images are taken across the width for oxide thickness measurements

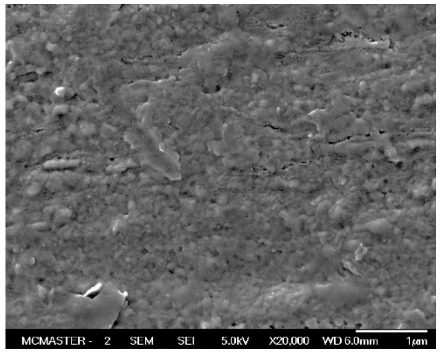


Task 2 - SEM of 2Mn-1.8Si Surfaces

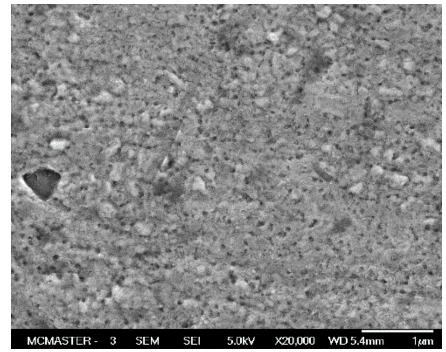
30 s



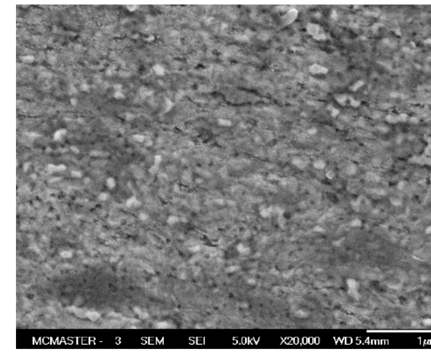
60 s



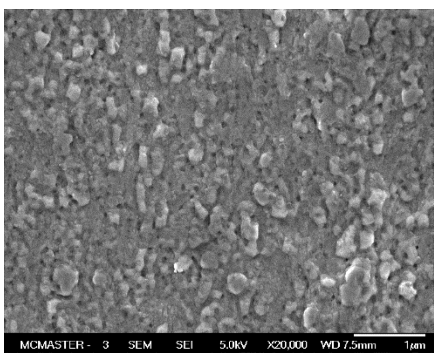
90 s



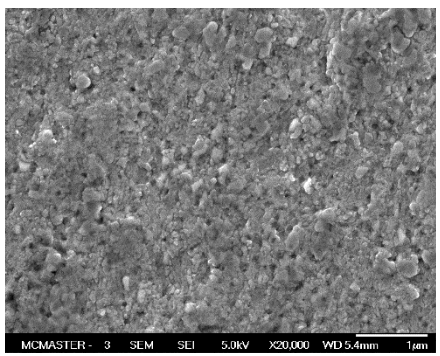
120 s



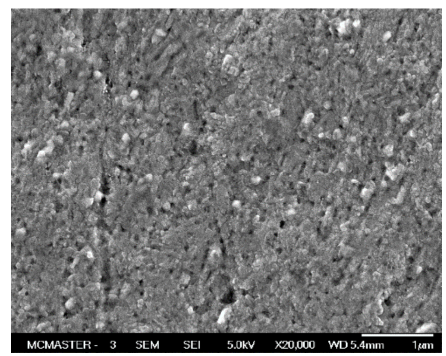
240 s



420 s



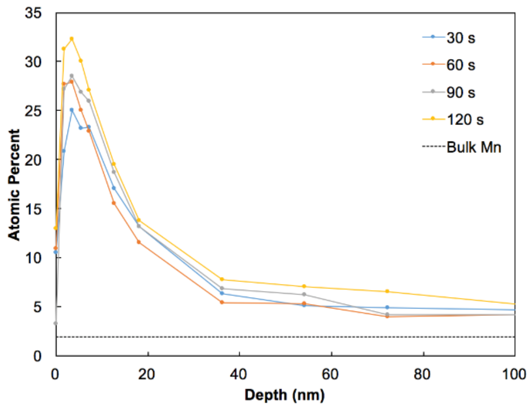
600 s

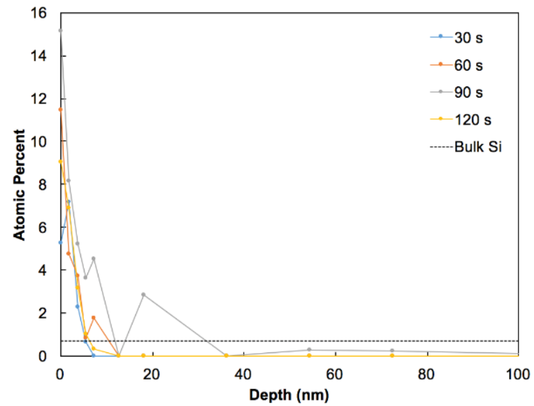


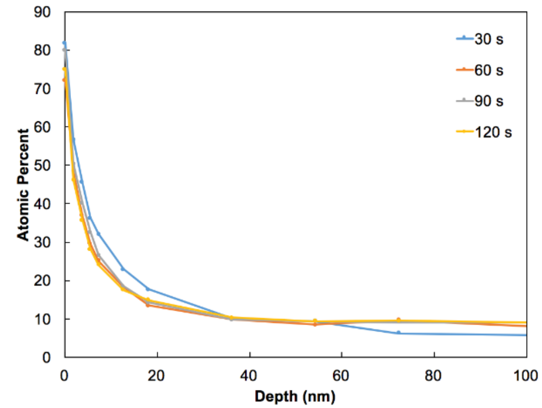
XPS Depth Profiles

Mn | Si | O

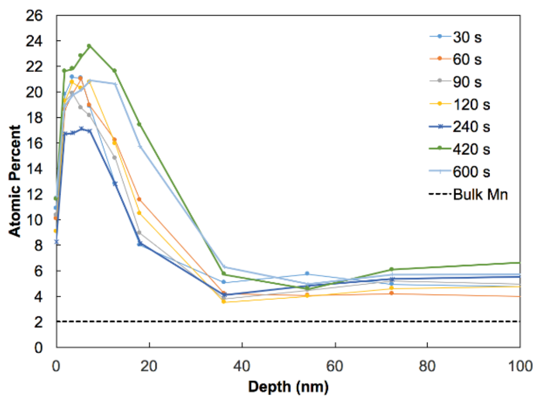
9019

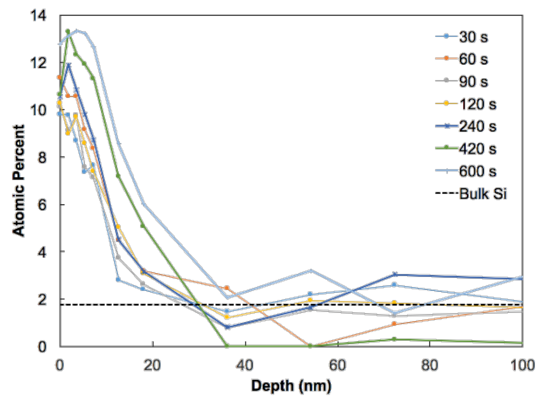


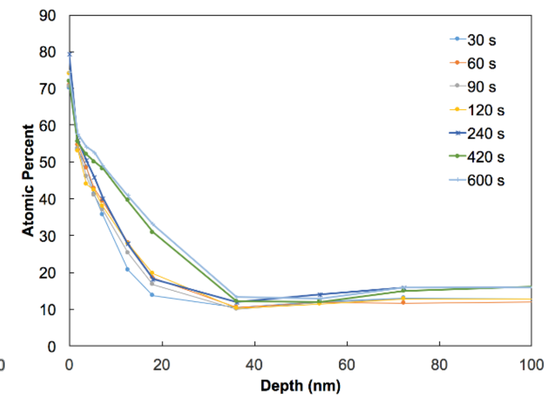




9088A





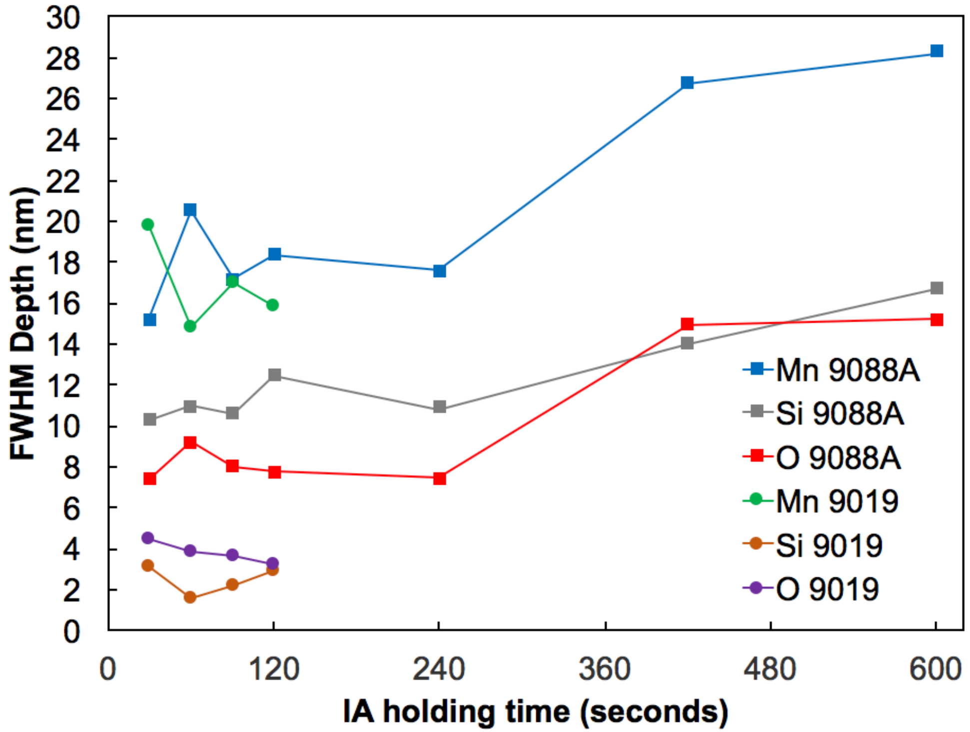


XPS Depth Profiles (cont.)

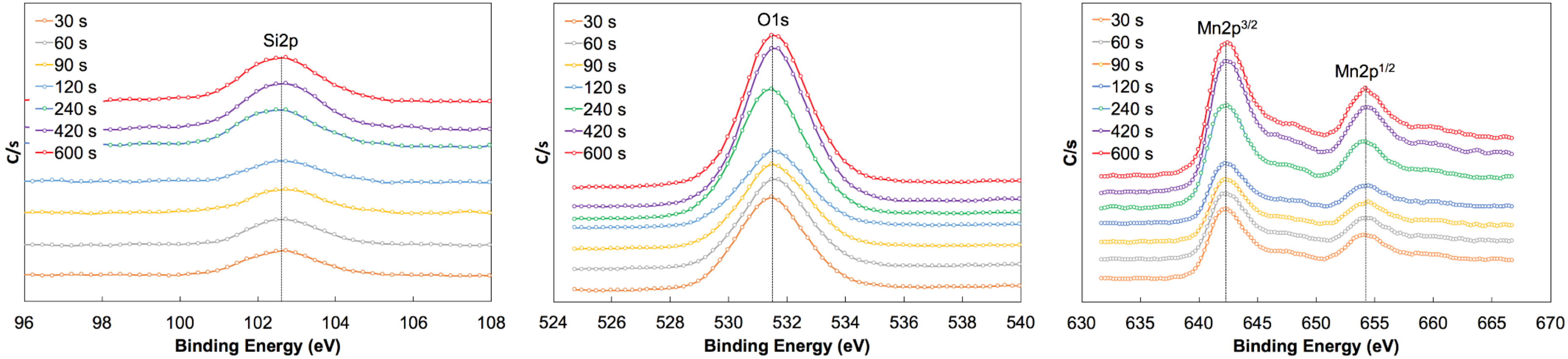
- **Between two alloys:**
  - **Mn similar**
  - **Si much higher in 9088A**
  - **O higher in 9088A**

Grade	Mn	Si
9019	1.960	0.701
9088A	2.029	1.757

Full width half maximum



XPS Binding Energies (2Mn-1.8Si)



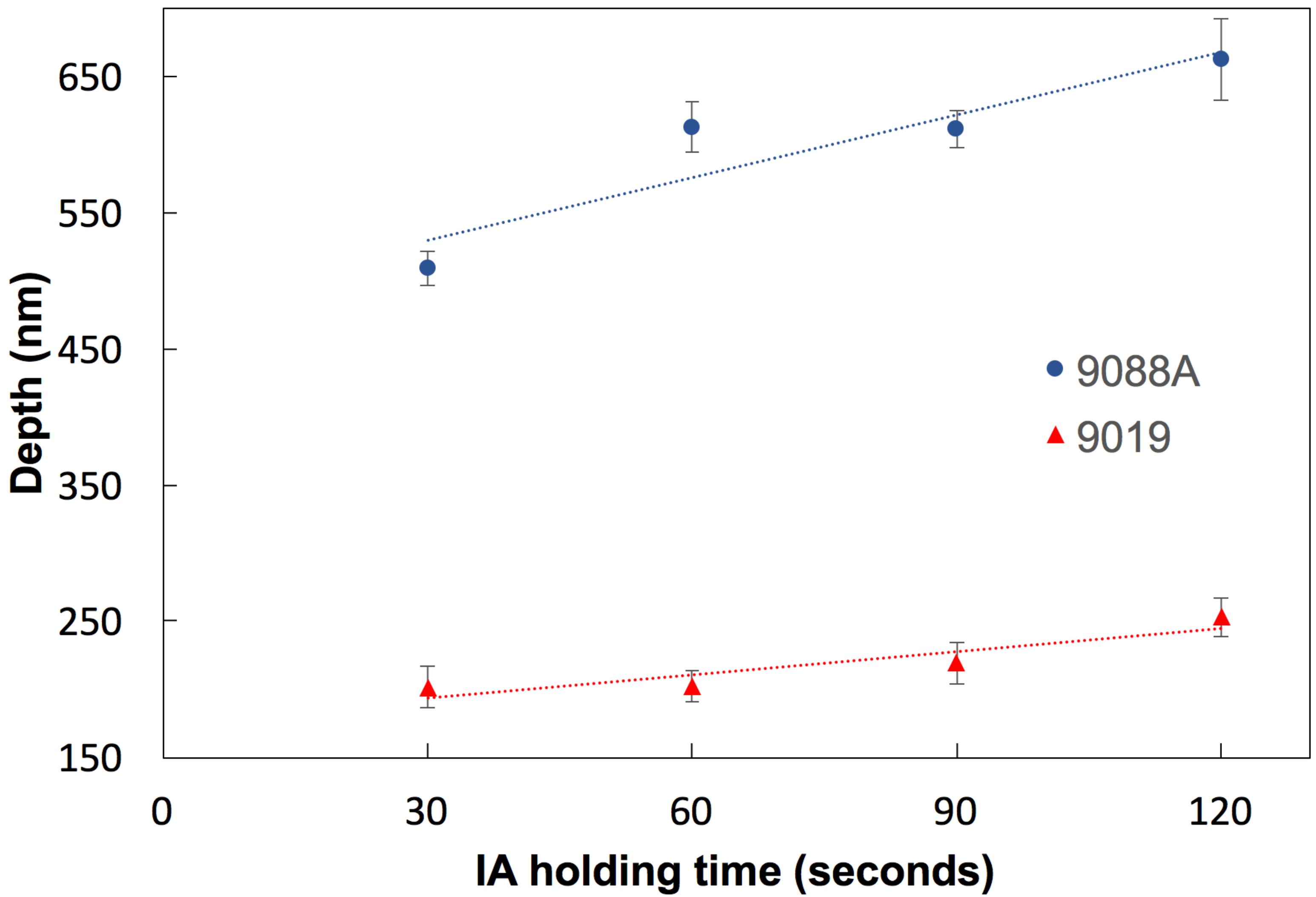
Measured from XPS

$Mn2p^{3/2}$	$Mn2p^{1/2}$	Si2p	O1s
642.3	654.3	102.5	531.5

Literature

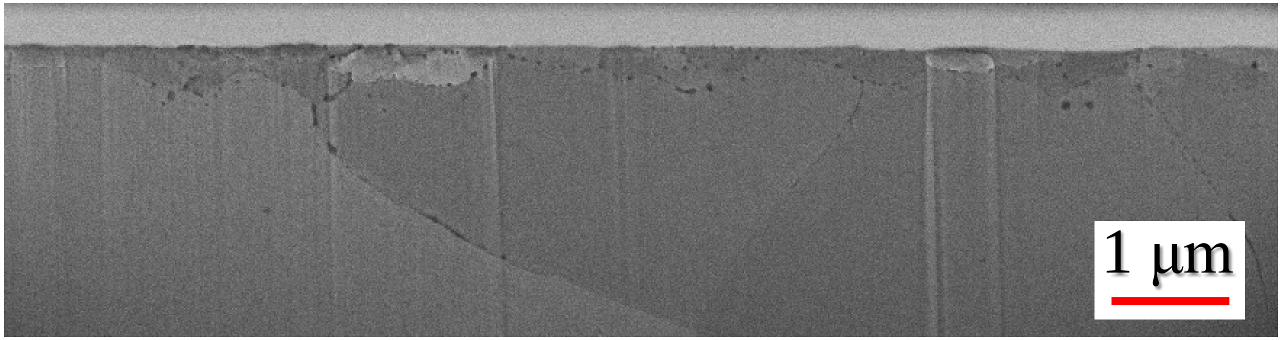
Compound	$Mn2p^{3/2}$	$Mn2p^{1/2}$	Si2p	O1s
MnO	641.7	653.3		530.2
$MnSiO_3$	641.9		102.1	530.9
Mn_2SiO_4	641.5		101.6	530.6

Short-time Internal Oxidation Kinetics

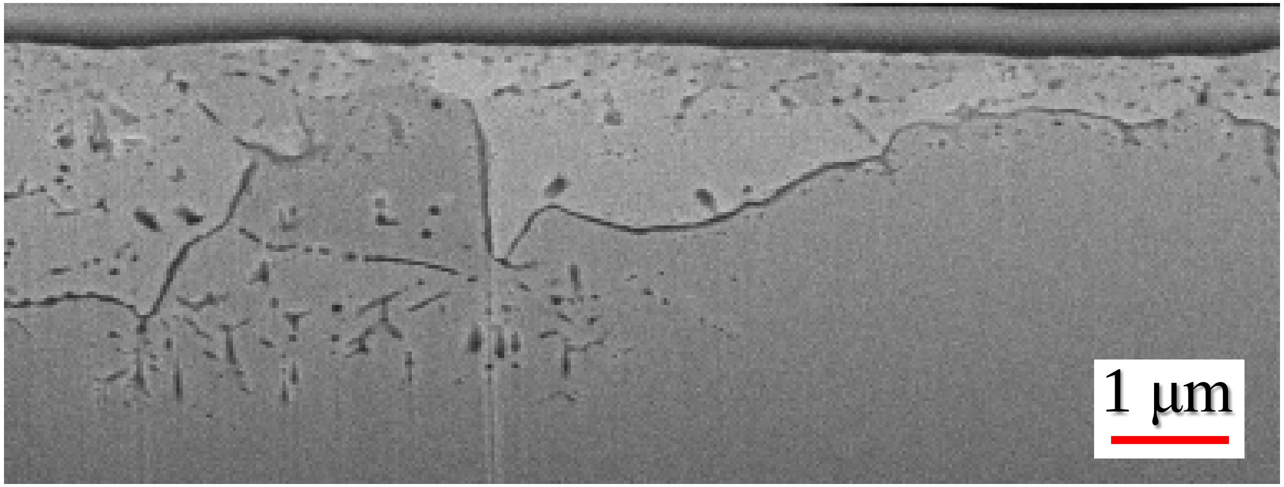


Comparison of microstructure after 120 s

2Mn-0.7Si

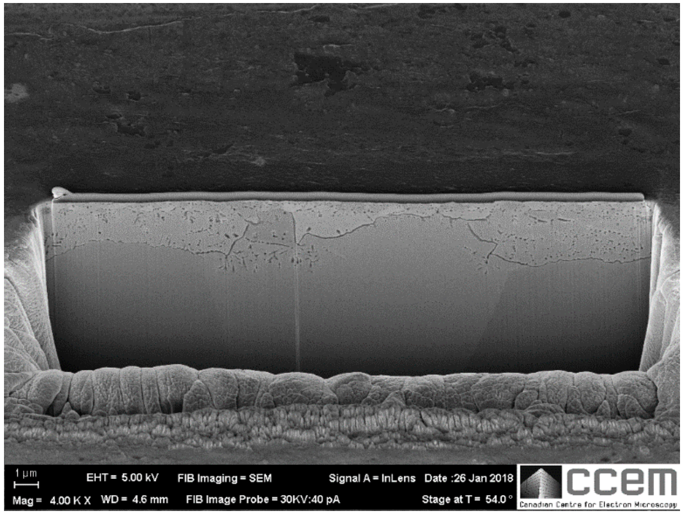


2Mn-1.8Si

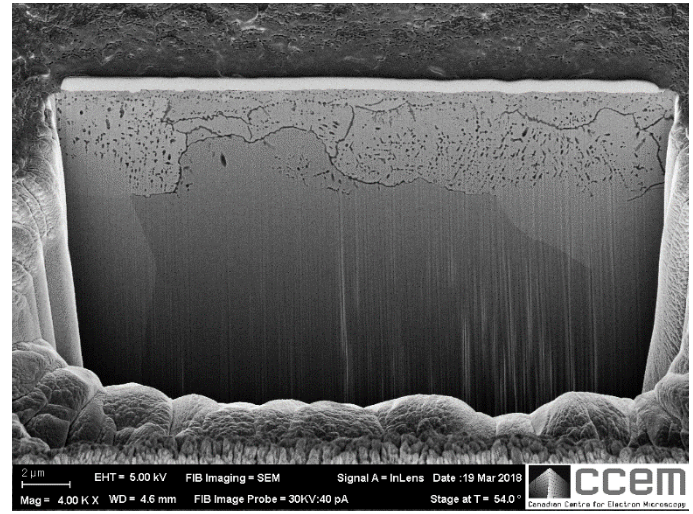


Evolution of Subsurface Oxidation (2Mn-1.7Si)

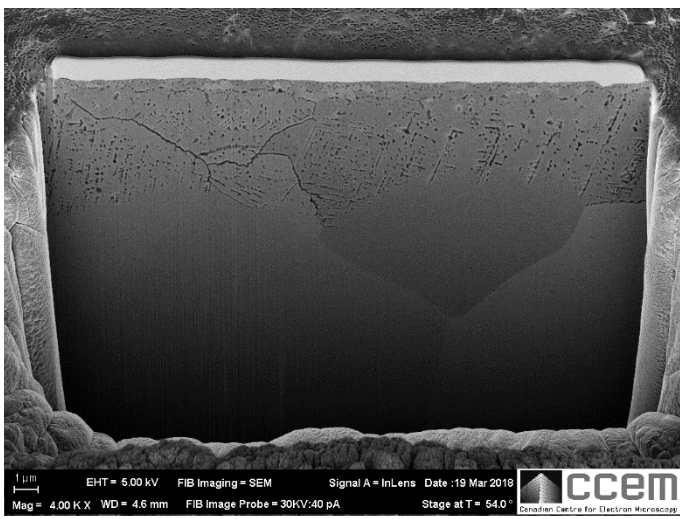
120 s



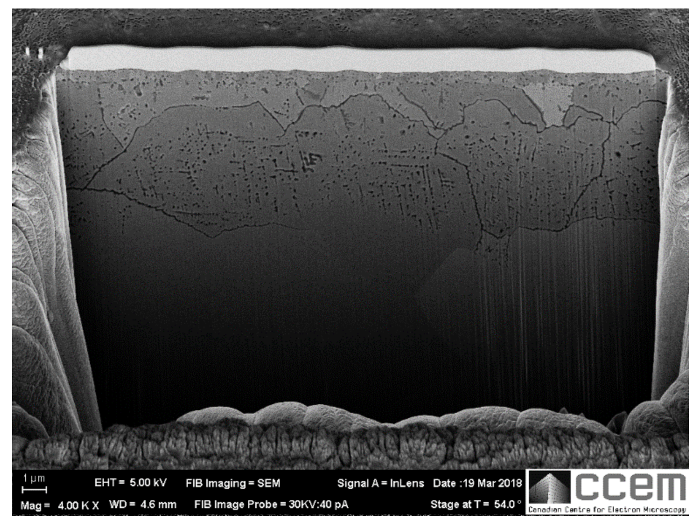
240 s



420 s



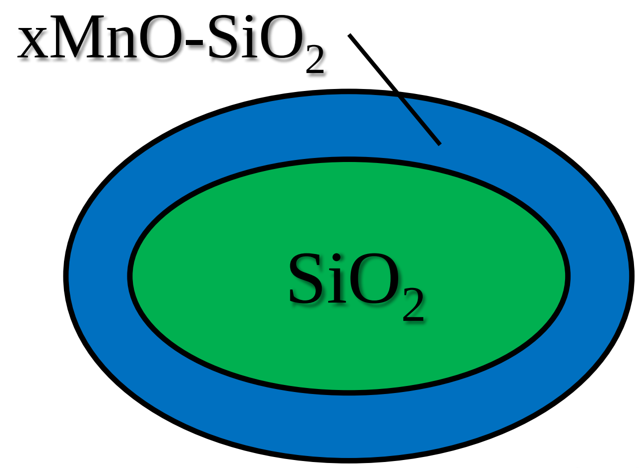
600 s

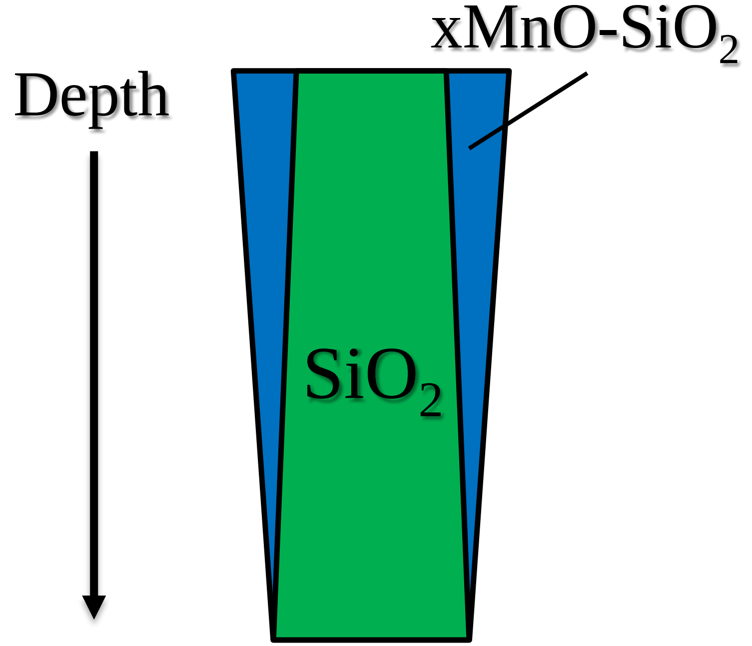


General Morphology of Subsurface Oxides

- **Core – shell structure**
  - **SiO_2 core, $xMnO\text{-}SiO_2$ shell**

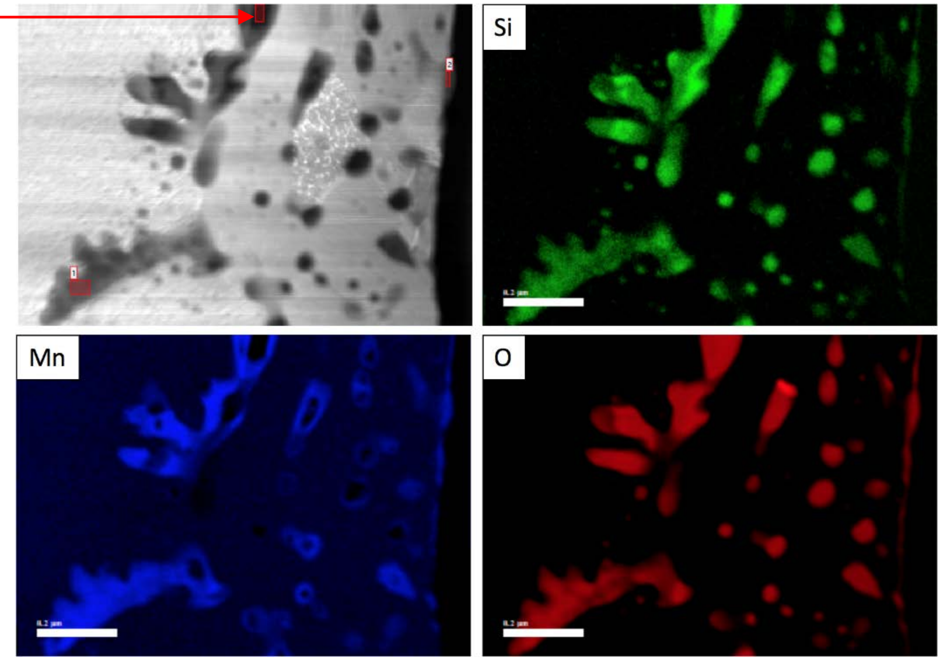
- **With increasing depth, thicker SiO_2 core and thinner $xMnO\text{-}SiO_2$ shell**



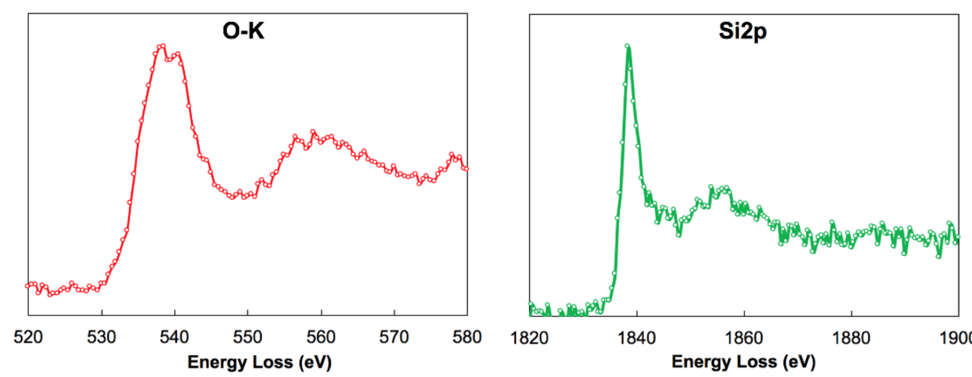


TEM + EELS – 2Mn-1.8Si × 120s

- SiO_2 core
  - Zero Mn present
  - In literature, well established that this is SiO_2

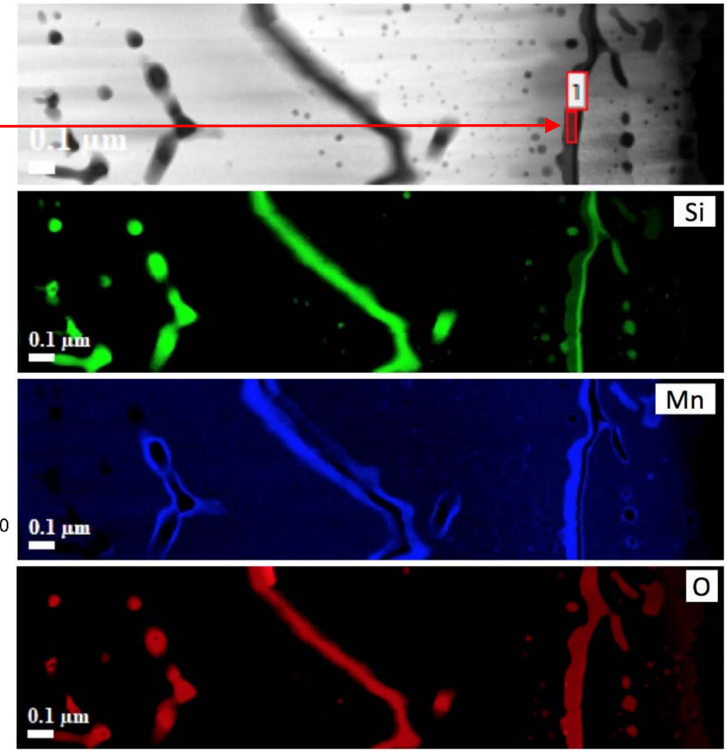


Element	Si	O	Mn
At. %	38.1	61.9	0.0

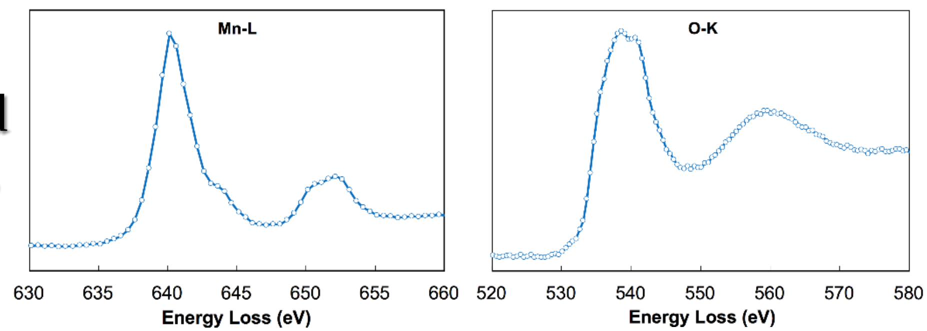


TEM + EELS (cont.) – 2Mn-1.8Si

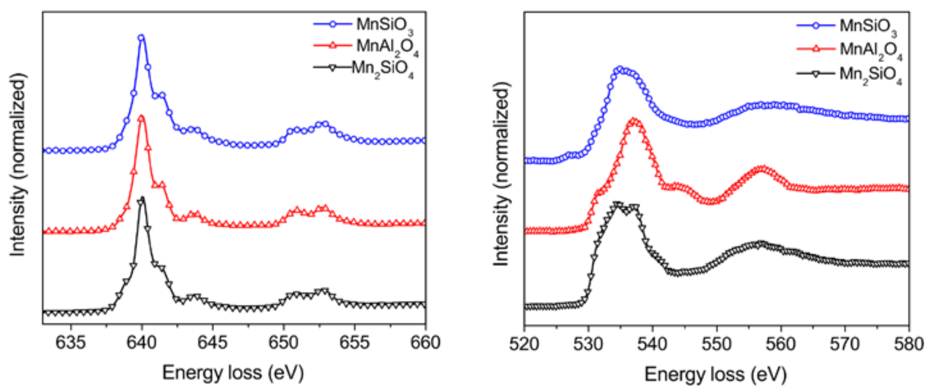
- xMnO-SiO_2 shell: Fine structure most closely match $MnSiO_3$



Measured by EELS



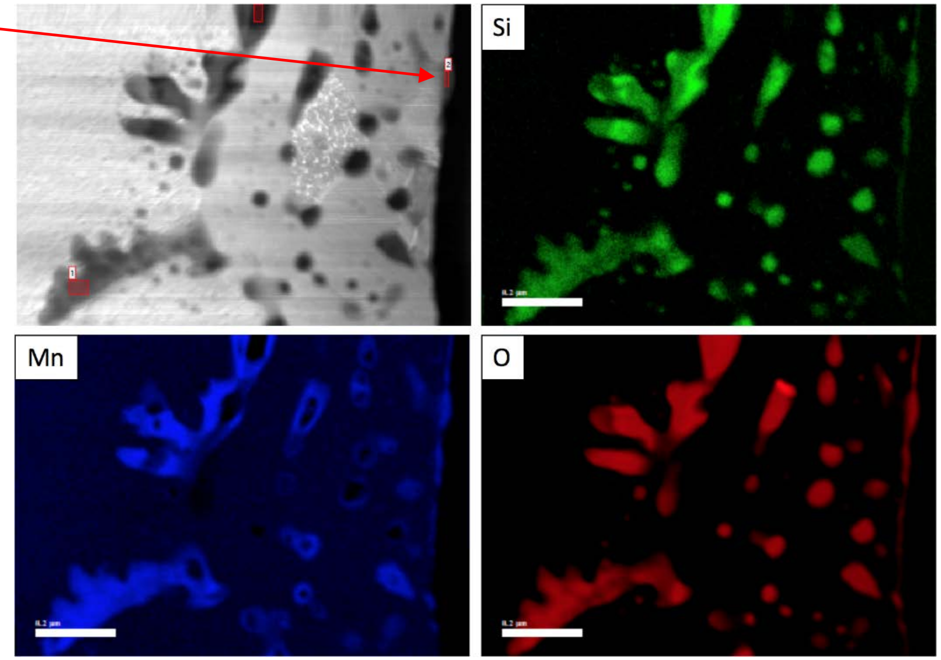
From Literature

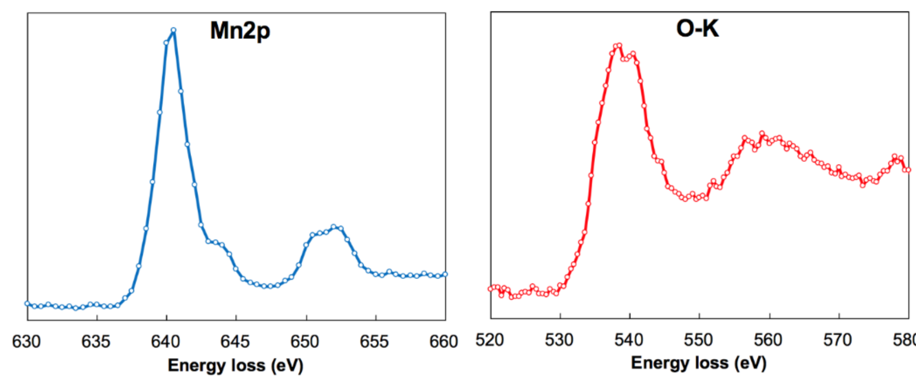


Element	Si	Mn	O	Fe
At. %	14.6	19.0	58.5	8.0

TEM + EELS (cont.) – 2Mn-1.8Si

- **External oxide**
  - **Very little external oxidation found – thin and discontinuous**
  - **Composition: xMnO-SiO_2**

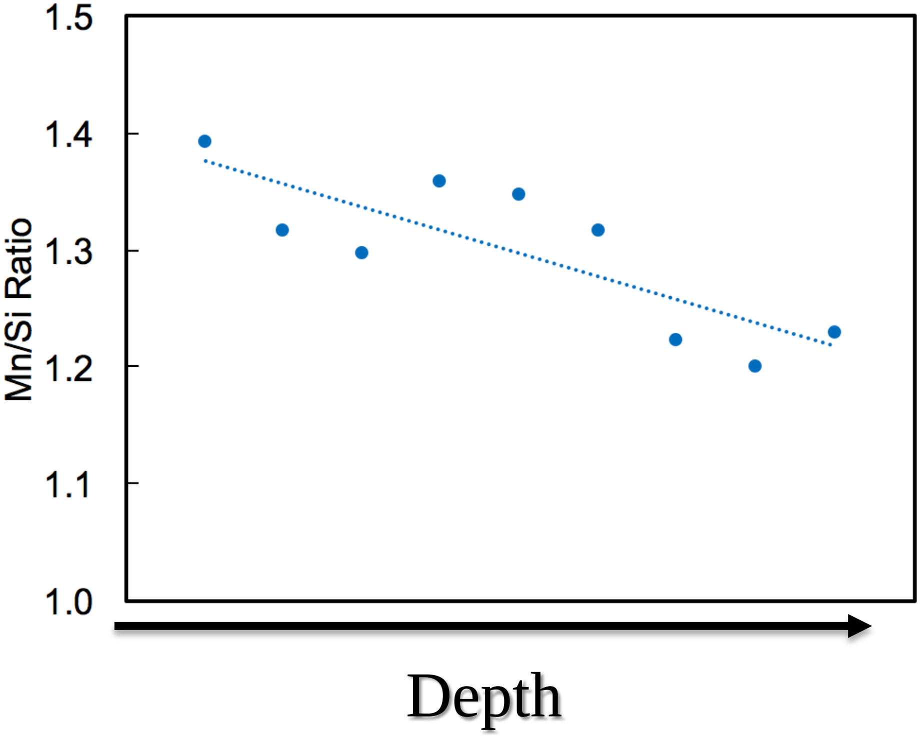


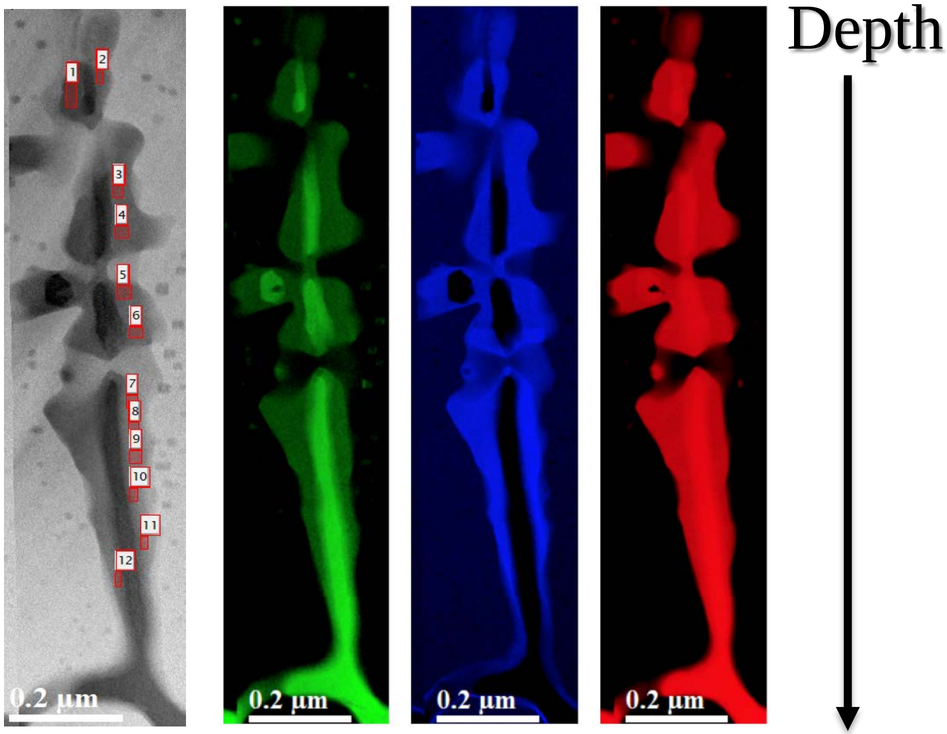


Element	Si	Mn	O	Fe
At. %	14.2	18.6	58.3	8.9

TEM + EELS (cont.) – 2Mn-1.8Si (120s)

- Mn/Si ratio as a function of depth



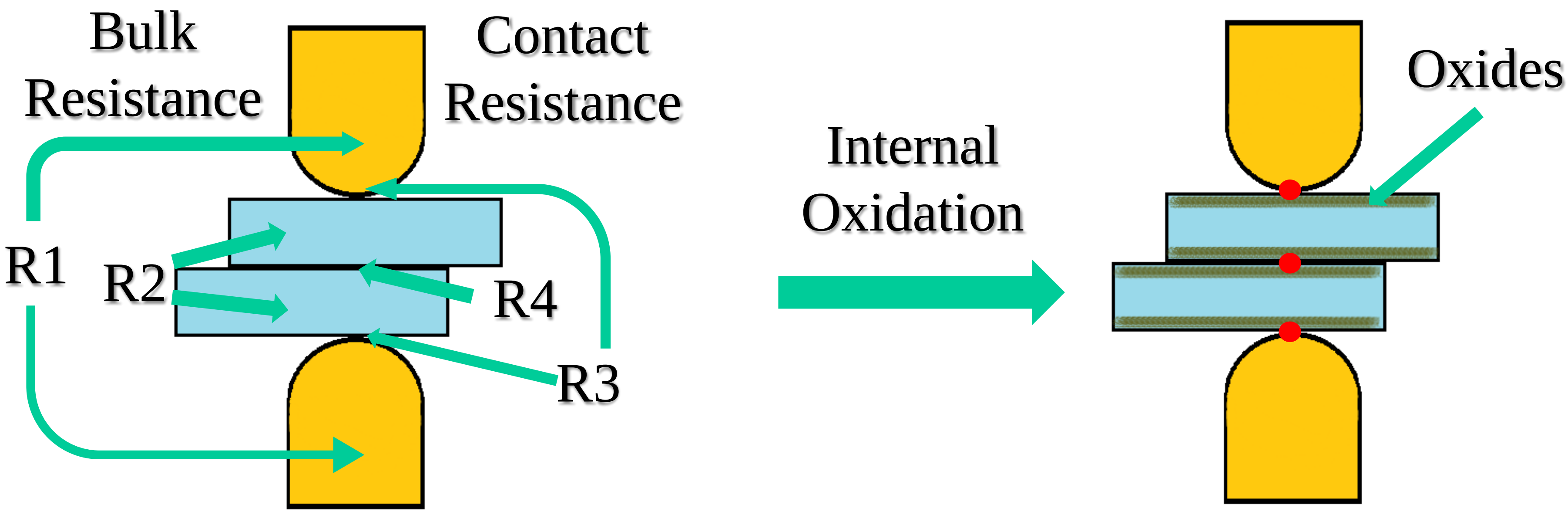


Task 1 + 2 – Summary

- **Both the 2Mn-0.7Si and 2Mn-1.8Si substrates displayed internal oxidation when annealed in a N2-5H_2 × –5°Cdp process atmosphere**
- **Internal oxides comprised mixtures of (Mn,Si)-oxides surrounding a SiO_2 core**
  - **consistent with observations of Seyed Mousavi**
- **Internal oxide kinetics not parabolic, but largely linear – will require further investigation as this is in disagreement with literature (including our own!)**

Task 3 – Internal Oxidation in RSW

- Formation of internal oxides increase contact resistance R3 and R4
- Increase heat input into the weld region

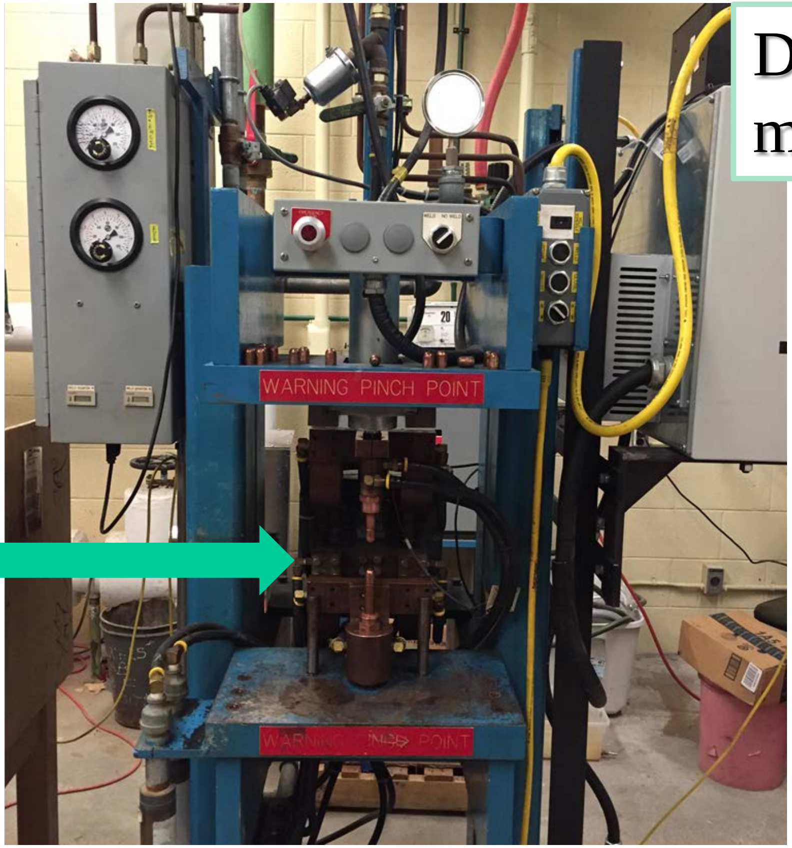


R3 and R4 increase

Task 3 – RSW Equipment and Parameters

- Medium Frequency Direct Current (MFDC) welder
- AWS 8.9 standard used for welding schedule

Dynamic resistance measurement

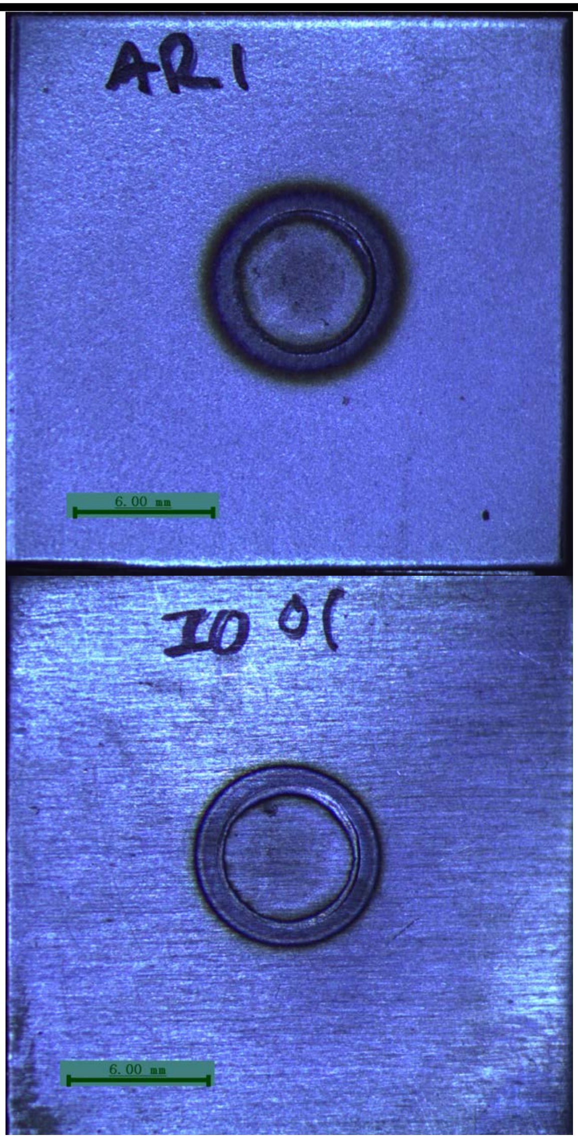


Adjustable weld schedule and electrode force

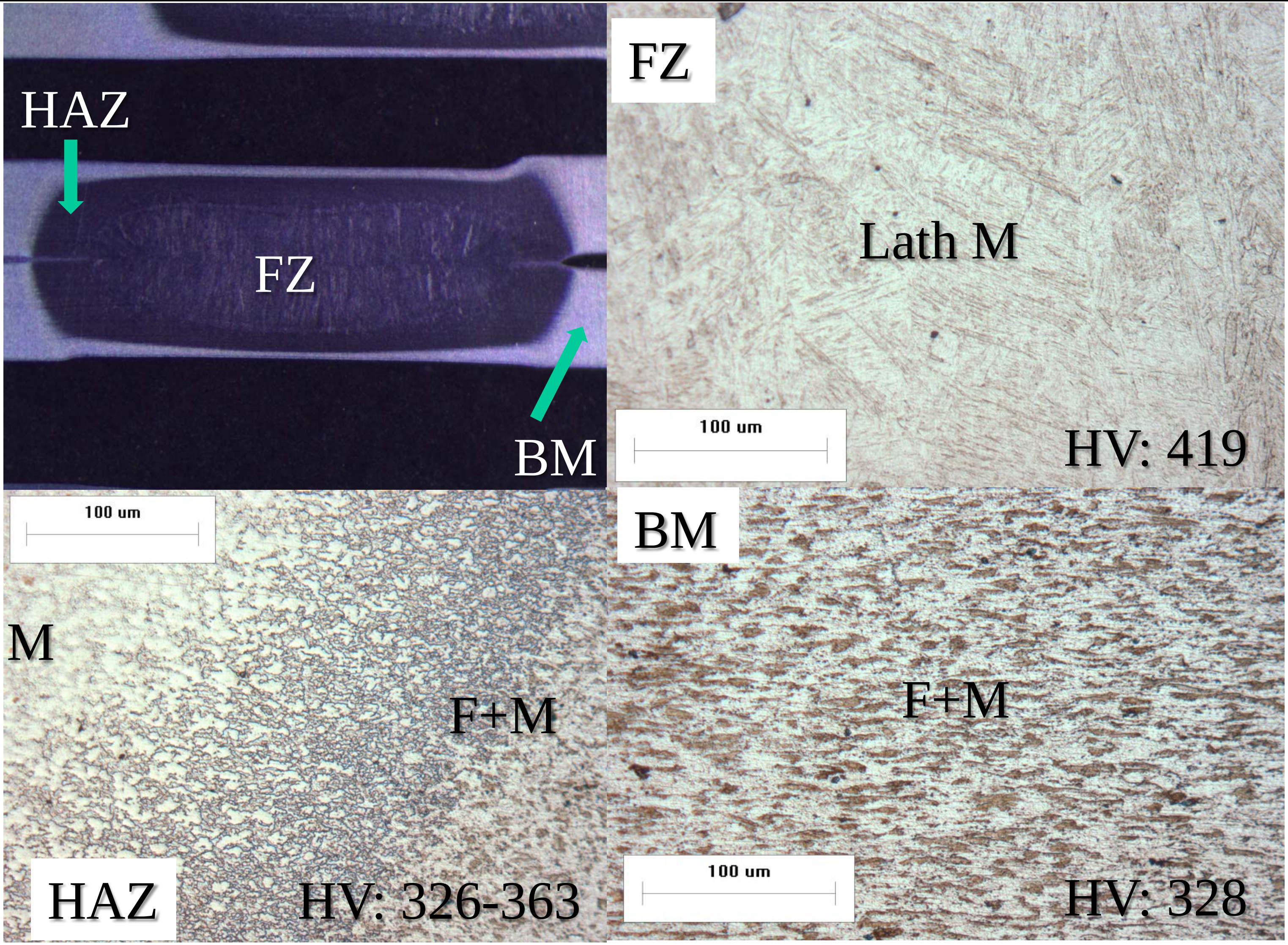
Material Studied

- **As-received 2Mn-0.7Si steel**
  - **thickness of 1.2 mm**

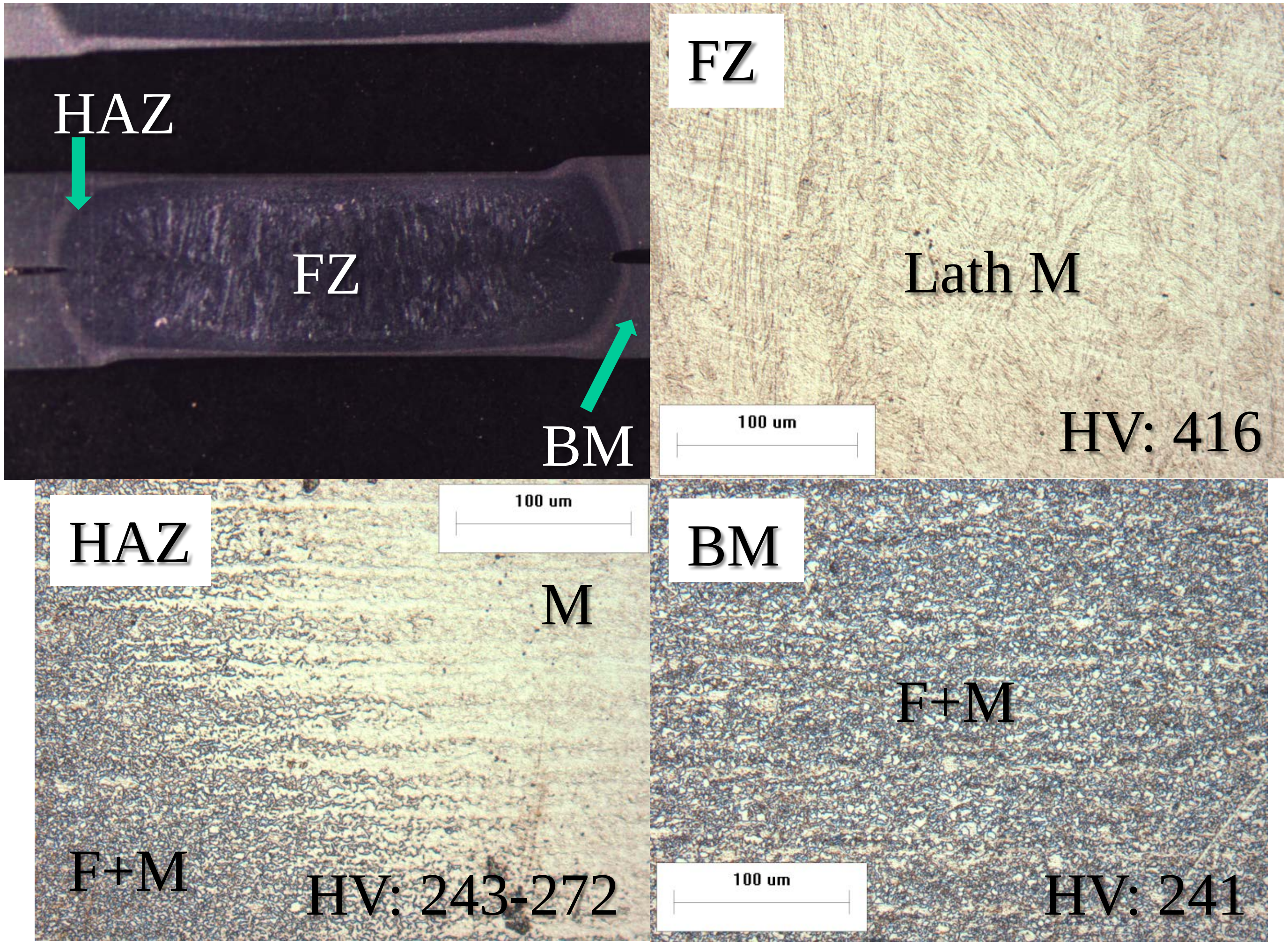
- **Internally oxidized 2Mn-1.8Si**
  - **time: 120 seconds**
  - **dew point: -5°C**
  - **atmosphere: N_2-5H_2**



Microstructure: RSW AR 2Mn-0.7Si

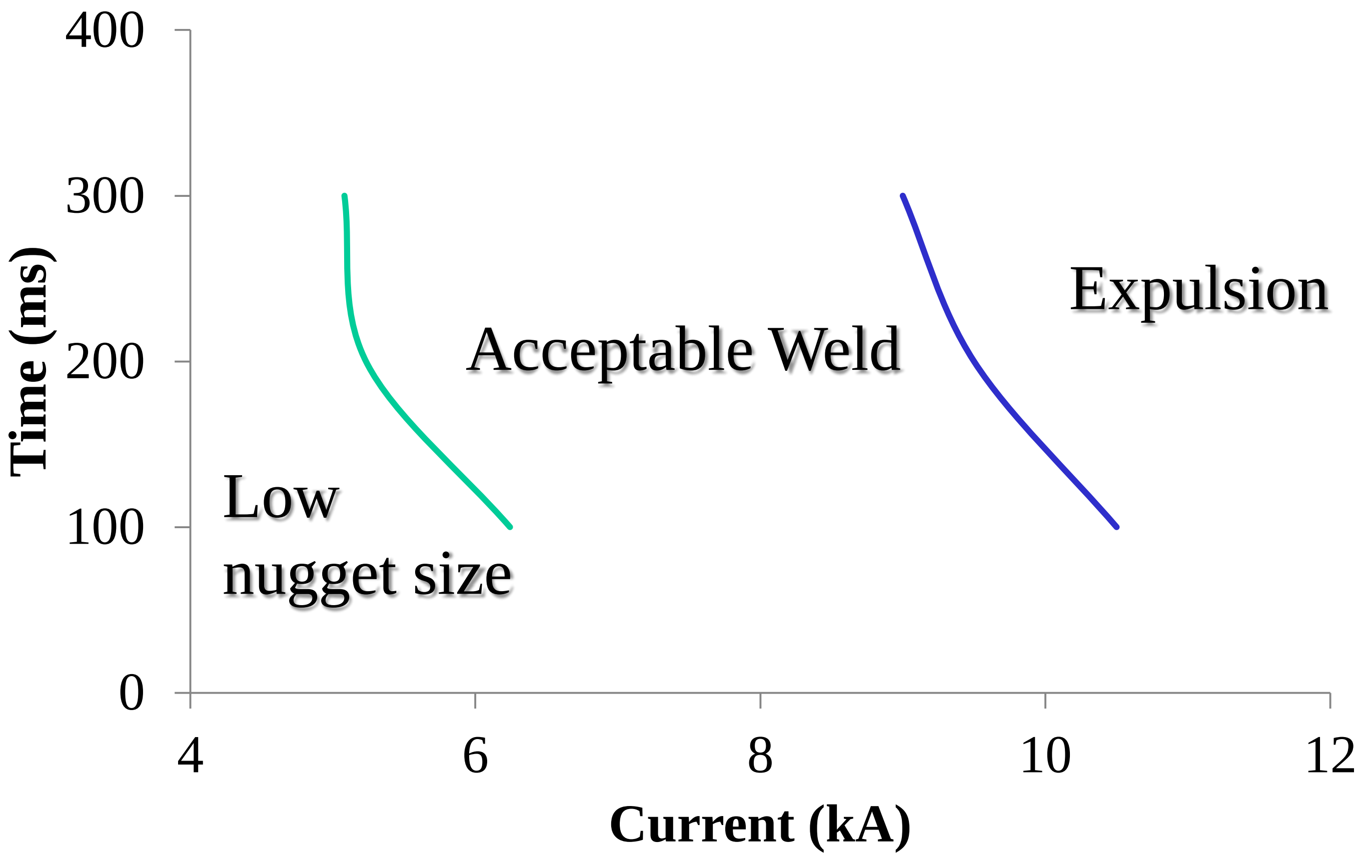


Microstructure: RSW IO 2Mn-1.8Si



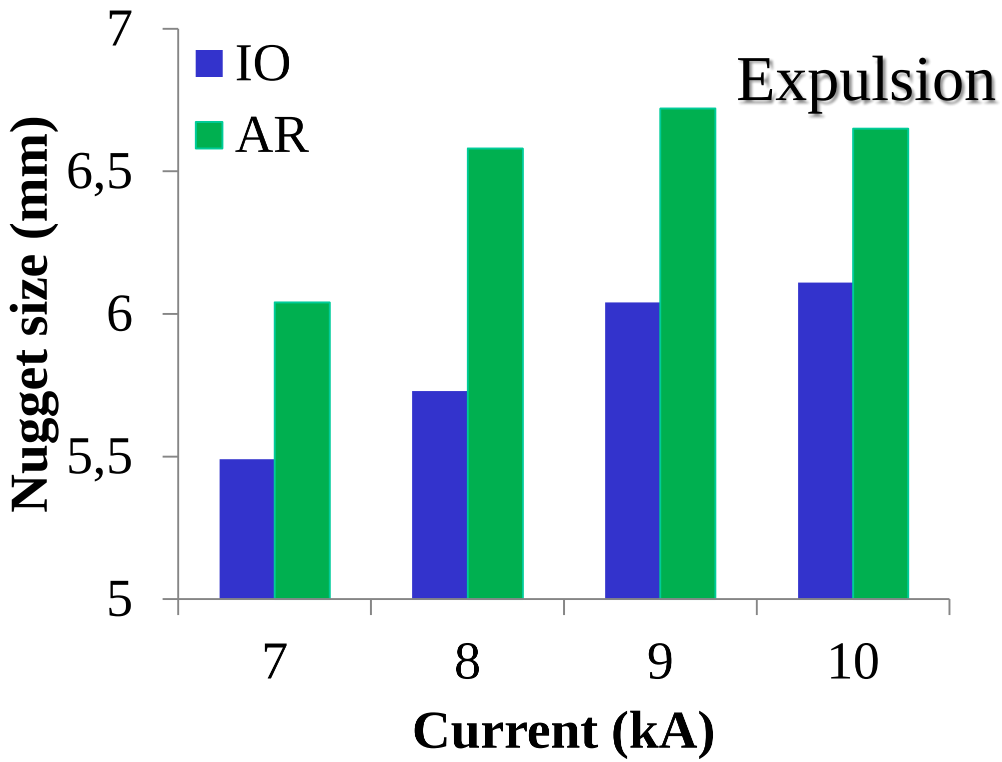
RSW Weld lobe

- **Weld lobe indicates process window of RSW based on nugget size and expulsion criteria**



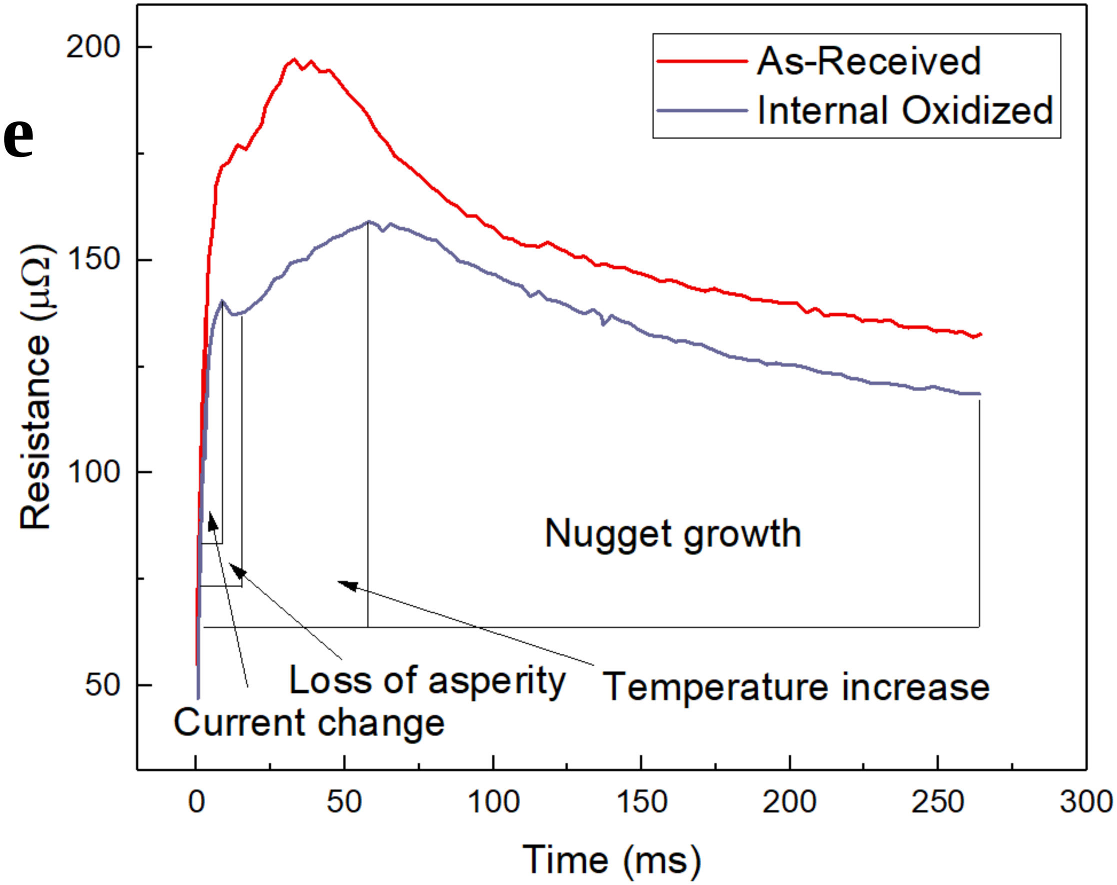
Nugget Size

- **Weld time of 266ms per AWS standard D8.9**
- **Nugget growth stopped at 10kA due to expulsion**
- **As-received (AR) had larger nugget than internal-oxidized (IO) in similar welding setting**



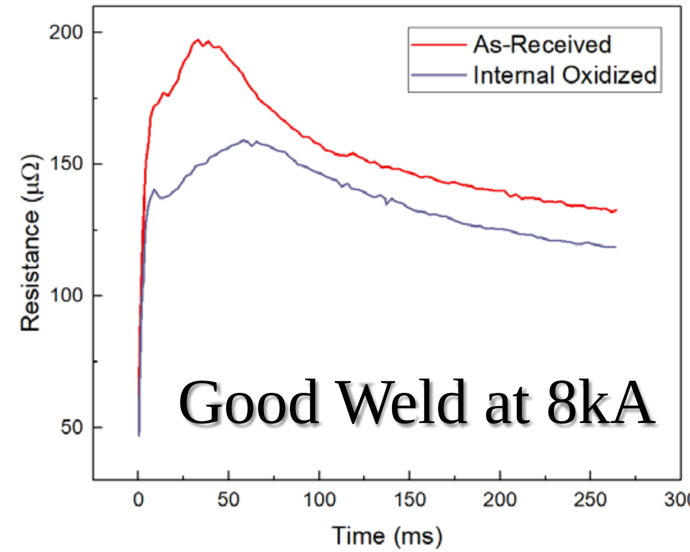
Dynamic Resistance

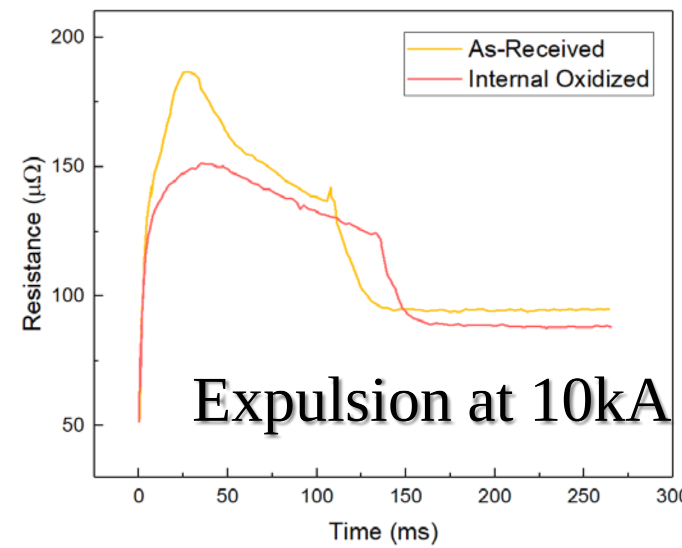
- Higher resistance leads to higher heat input and earlier nugget growth
- Heat and asperity increases resistance
- Growth of liquid nugget reduces resistance



Dynamic Resistance

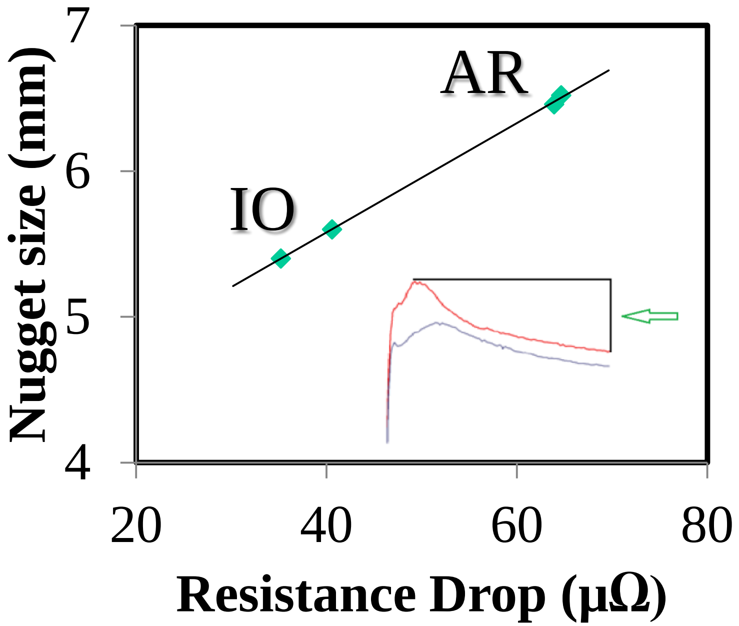
- AR 2Mn-0.7Si had higher resistance vs IO 2Mn-1.8Si leading to faster heating rate
- Weld pool started to form earlier and have more time to grow
- Under extreme welding condition, expulsion occurred earlier in AR sample

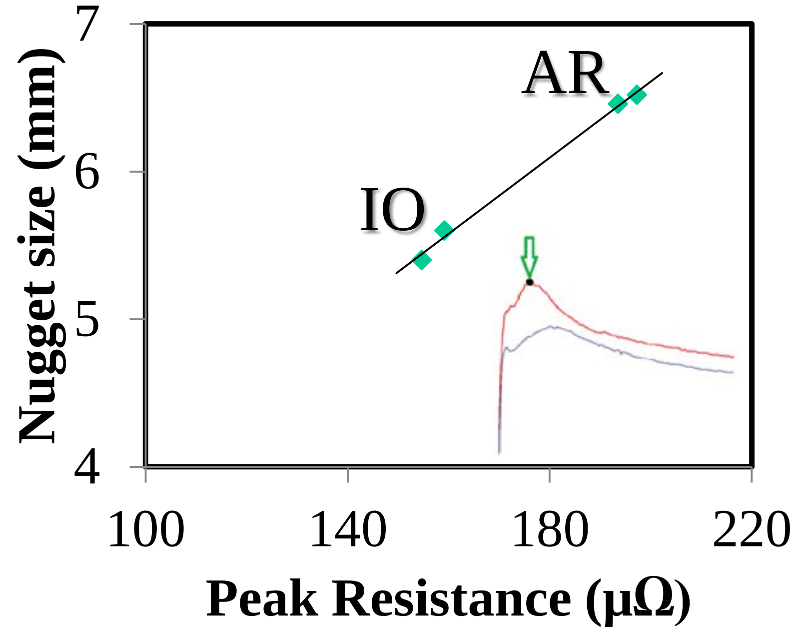




Dynamic Resistance and Nugget Size

- **Nugget size is correlated to peak and drop of dynamic resistance in good weld condition**
- **Nugget size in expulsion condition are less predictable due to expulsion of molten metal**





Task 3 – RSW Conclusions + Next Steps

- **AR 2Mn-0.7Si alloy had a higher resistance vs the IO 2Mn-1.8Si alloy, leading to earlier nugget expulsion**
  - **currently assessing AR 2Mn-1.8Si RSW and IO 2Mn-0.7Si alloys to allow direct comparison**
- **Next steps**
  - **complete internal oxidation kinetic assessment and RSW of model 2Mn-xSi alloys**
  - **move onto recently received industrial alloys – currently assessing thermal processing and oxidation mode transition for the alloys**