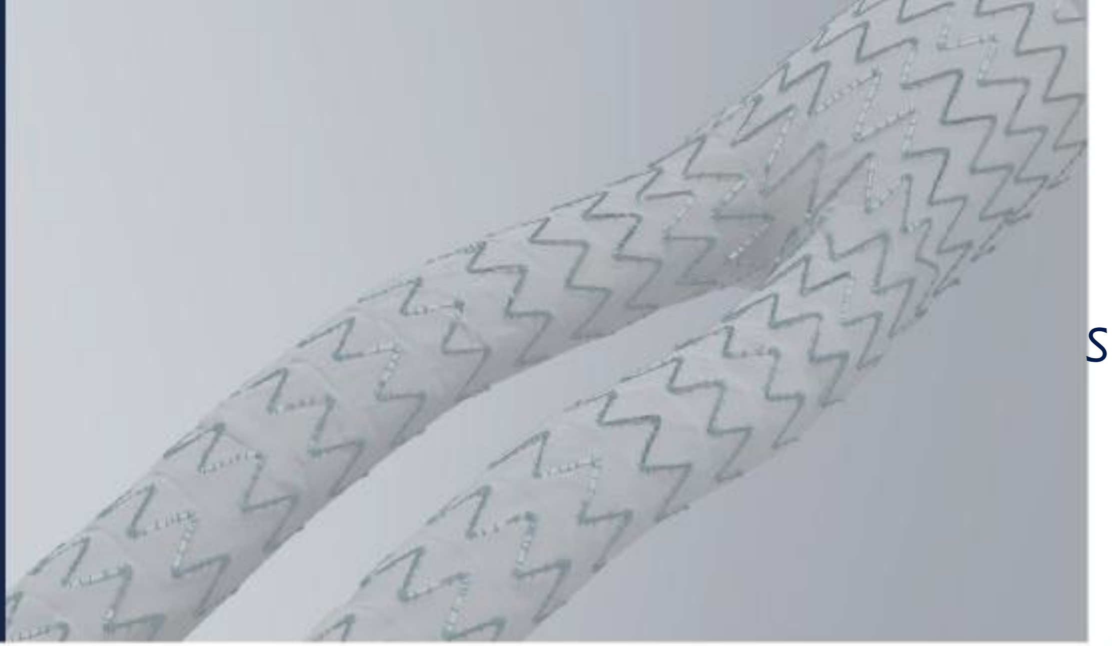

CONFLUENT

Effect of ePTFE sintering post processing steps on surface quality and pitting corrosion behavior of NiTi stents

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Pahul Singh, *Craig Bonsignore*

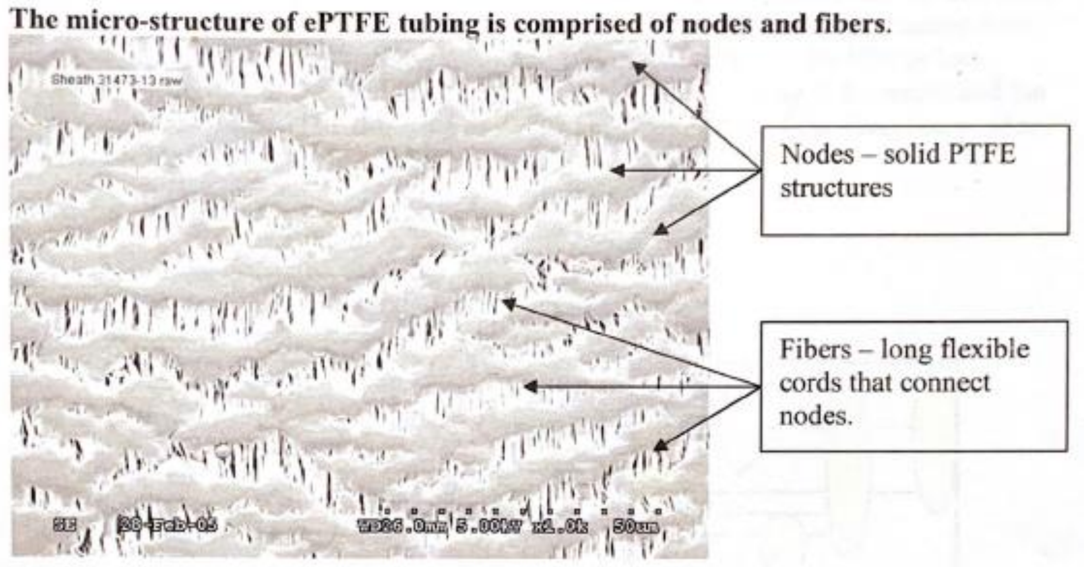
What is ePTFE?

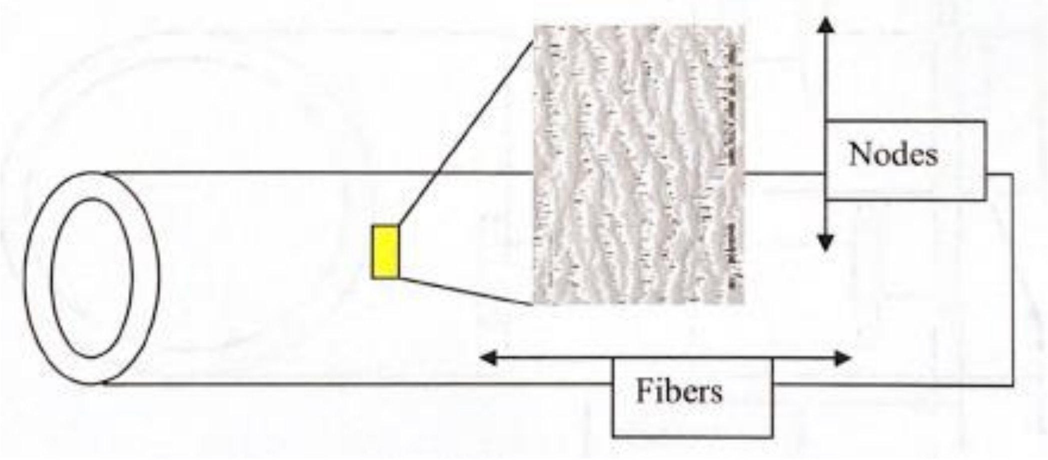
- Acts as a bio-inert barrier cover on medical stents
- Prevents or delays the tissue ingrowth within the stent

***expanded* Polytetrafluoroethylene**

- Properties include thermal stability, chemical resistance, electrical characteristics, and coefficient of friction
- Biocompatible implant material
- Its properties of porosity (inter-nodal distance), thickness, and crystallinity can be tuned for device performance

- Manufacturing process:
  1. Compounding PTFE fine powder resin
  2. Preforming, creating paste/billet for Ram extrusion
  3. Extrusion and tying
  4. Drying, to remove hydrocarbon lubricant
  5. Expansion, to create matrix of nodes and fibers
  6. Sintering, move from amorphous to crystalline





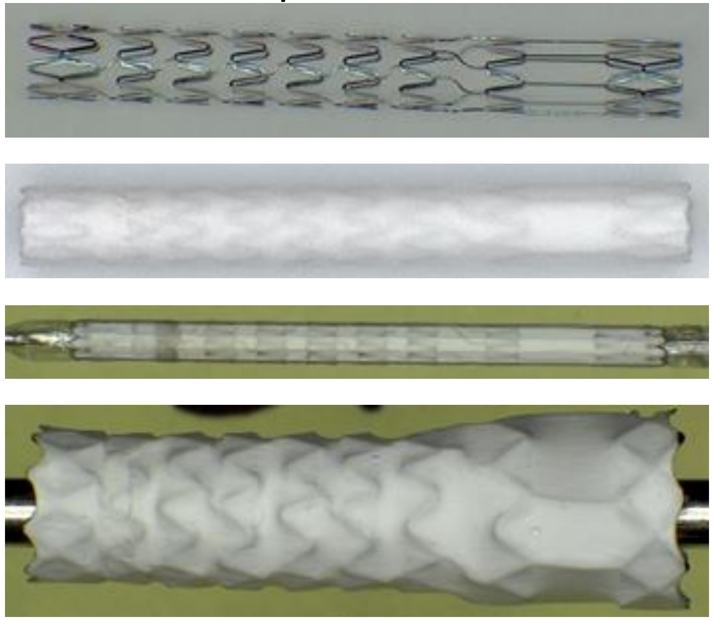
Images courtesy of IPE Inc.

ePTFE Processing with Frames

- Nitinol and S.S. frames can be encapsulated with a similar process
- The main steps of encapsulation:
  1. Raw ePTFE material (tube or sheet)
  2. Cover frame with ePTFE (ID / OD, Tie Layer, Mechanical)
  3. Compress layers and Sinter (>320C)
  4. Remove frame from tooling

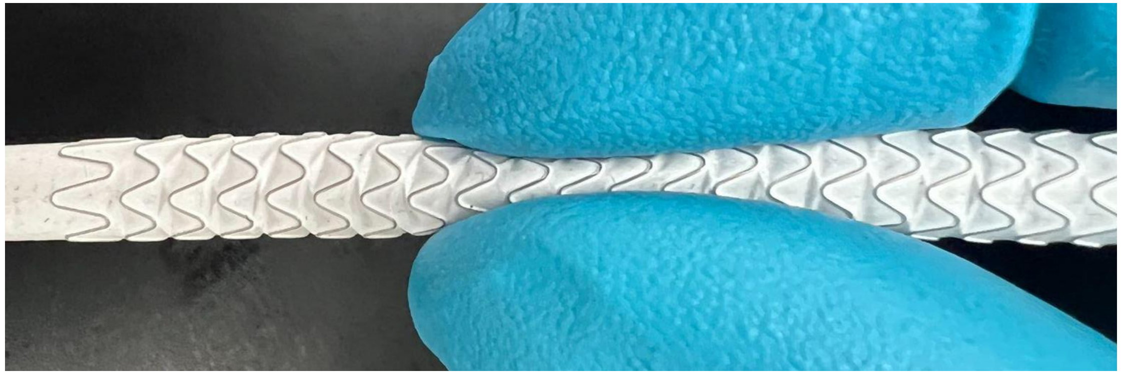
S.S. Frames:

- Crimped on balloon delivery system
- ePTFE can radially expand to accommodate plastic deformation



Nitinol Frames:

- Sheathed in a delivery system
- Flexible ePTFE that accommodates bending



In summary:
Sintering is done at (>320 C) after Electropolising the device so …..

What is the effect of post processing heat treatment on corrosion properties of the final device ?

Experimental details

CONFLUENT

- Use 0.6 mm $Ni_{50.8}Ti$ (at. %) wire

- Perform all manufacturing heat treatment steps as the NiTi stents undergo at around 500 C using salt pot furnace (i.e. shape setting, A_f tuning , etc)

- Perform manufacturing surface treatment steps (i.e. etching/ Electropolishing) CORROSION TEST

- Perform heat treatment at 360 C for 10 min and air cool using air furnace. CORROSION TEST

- Apply 8 % strain on heat treated wire via wrapping around a mandrel for 1 min CORROSION TEST

to stimulate the crimping process

Potentiodynamic corrosion test

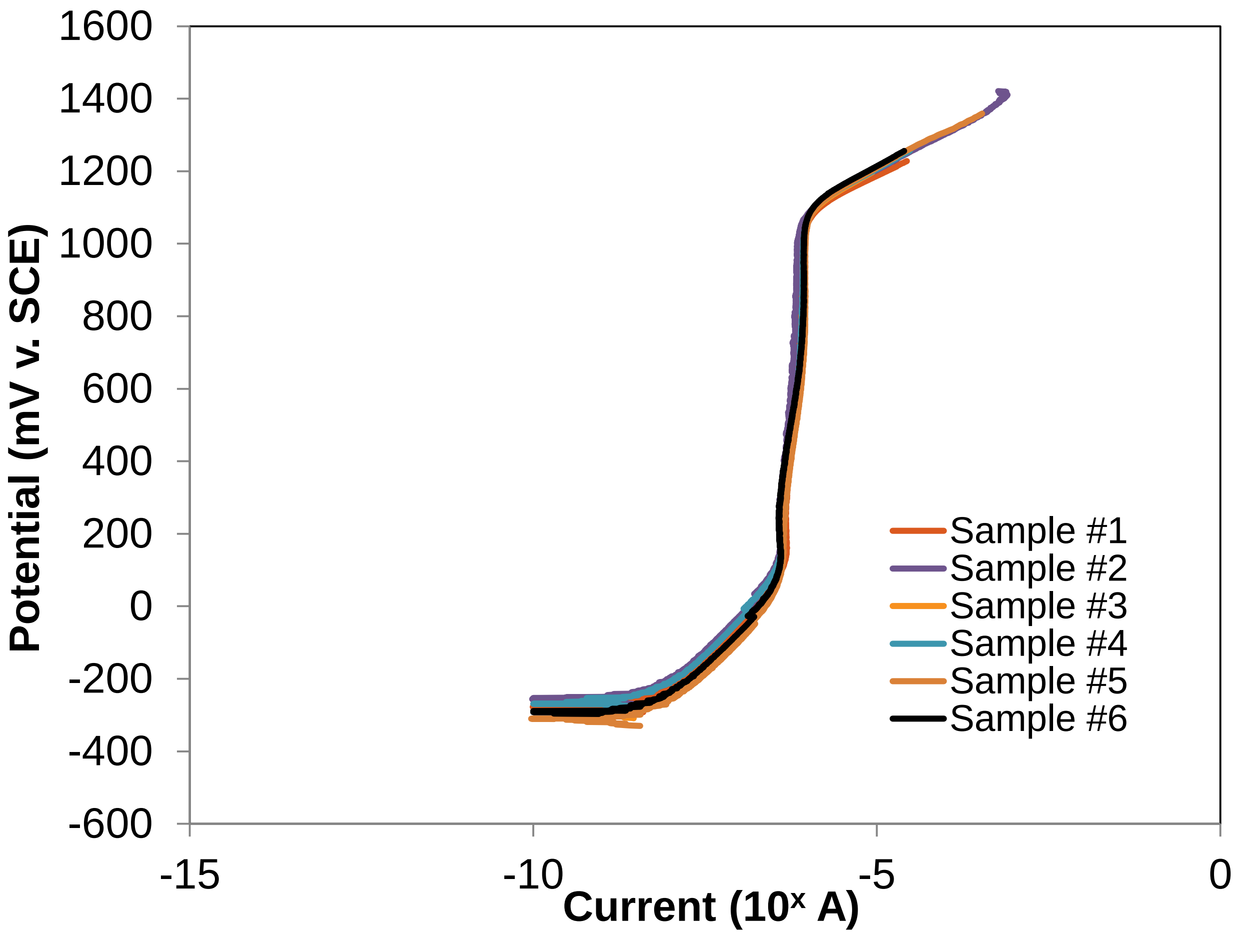
- Corrosion Test per ASTM F2129
- Phosphate Buffered Saline solution with pH of 7.4 ± 0.1.
- Saturated Calomel Electrode (SCE as reference electrode and 2x Platinum Auxiliary Electrodes as counter electrodes).
- 0.167mV/s scan rate
- Reverse scan was not performed to detect the pitting locations if occurred

1. As EPed- wire- SEM surface study
after heat treatment and etch/electropolish steps

Scanning electron microscope (SEM)

- SEM characterization of wire after EP process showed a well polished surface.
- No sign of any surface defects/ pitting was observed.

1. As EPed- wire- Potentiodynamic corrosion test
after heat treatment and etch/electropolish steps



Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
1	-290	n/a	1040
2	-270	n/a	1040
3	-308	n/a	1050
4	-277	n/a	1040
5	-329	n/a	1060
6	-295	n/a	1050

- All 6 as-EPed wires reached Oxygen evolution
- No pitting/break down was detected.

1. As EPed- wire- SEM surface study after corrosion test

after heat treatment and etch/electropolish steps

Scanning electron microscope (SEM)

- SEM characterization of as- EPed wire after corrosion testing showed similar polished surface as before corrosion testing.
- No sign of any surface defects/ pitting was observed.

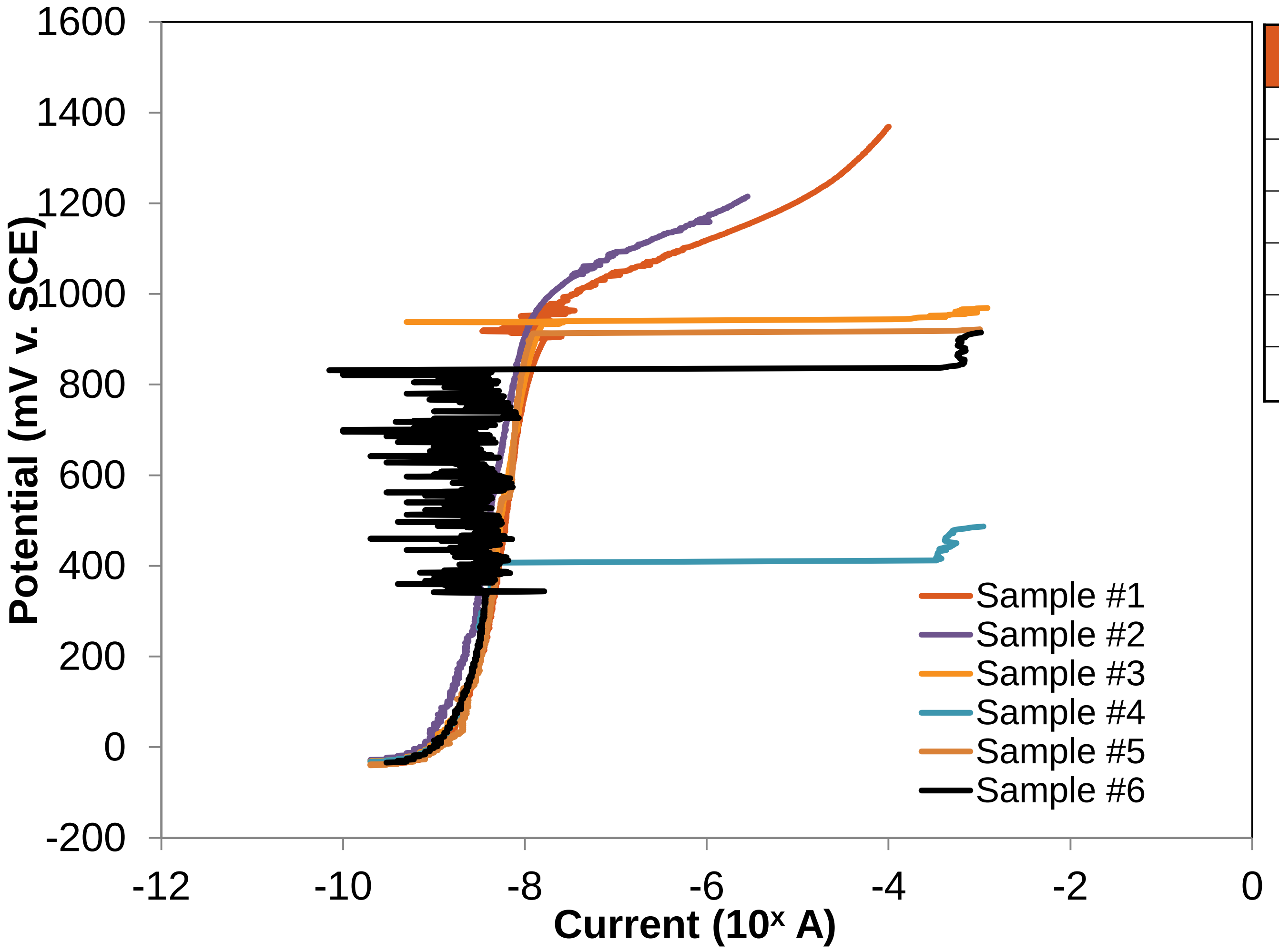
2. As sintered- wire- SEM surface study

after heat treatment of EPed wire at 360 C for 10 min in air furnace

- Heat treatment at 360 C for 10 min did not show any significant effect on the surface of the wire
- No sign of any surface defects/ crack was observed.
- The color of the wire turned to light gold after sintering heat treatment

2. As sintered- wire- Potentiodynamic corrosion test

after heat treatment of EPed wire at 360 C for 10 min in air furnace



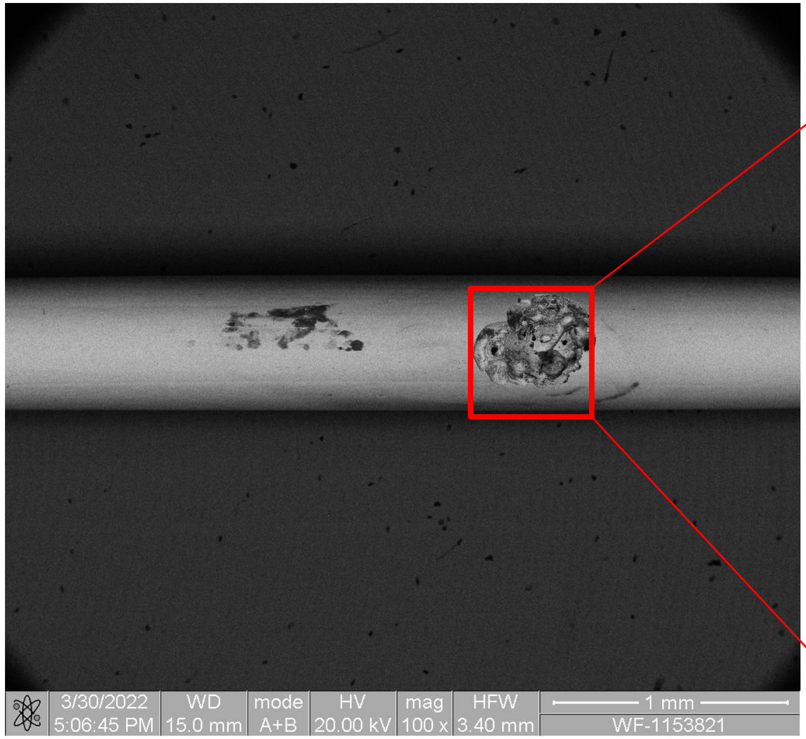
Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
1	-37	n/a	983
2	-26	n/a	963
3	-31	933	n/a
4	-30	407	n/a
5	-39	913	n/a
6	-32	832	n/a

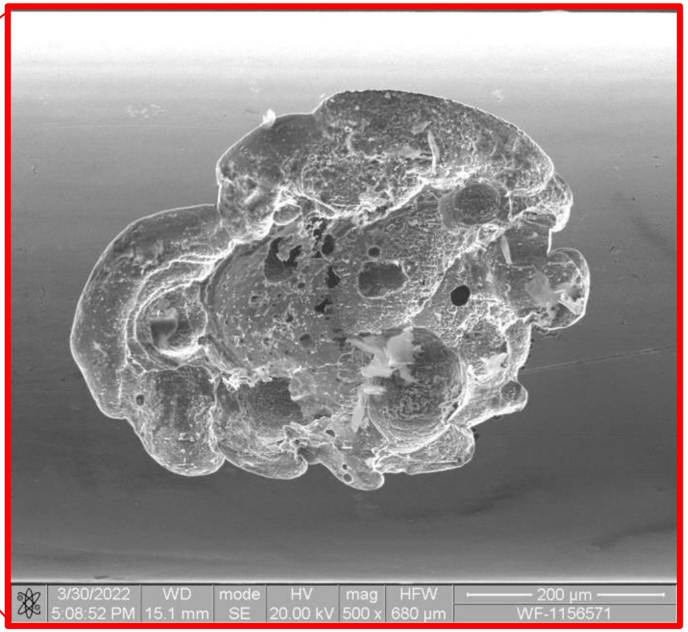
- 2 out of 6 as-sintered wires reached Oxygen evolution, however break down potential were high and one of the samples showed 407 mV break down potential

2. As sintered- wire- SEM surface study- after corrosion test

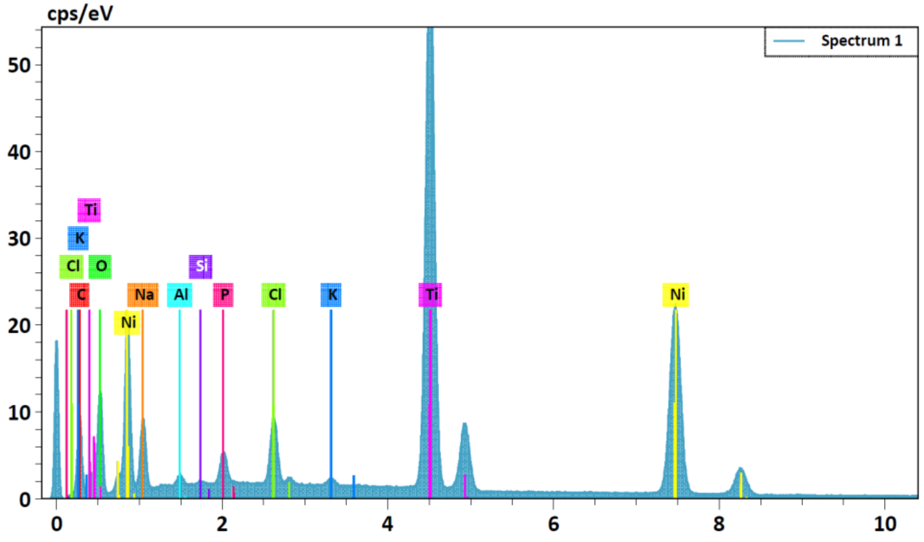
after heat treatment of EPed wire at 360 C for 10 min in air furnace

Sample #6 , E_b= 832 mV





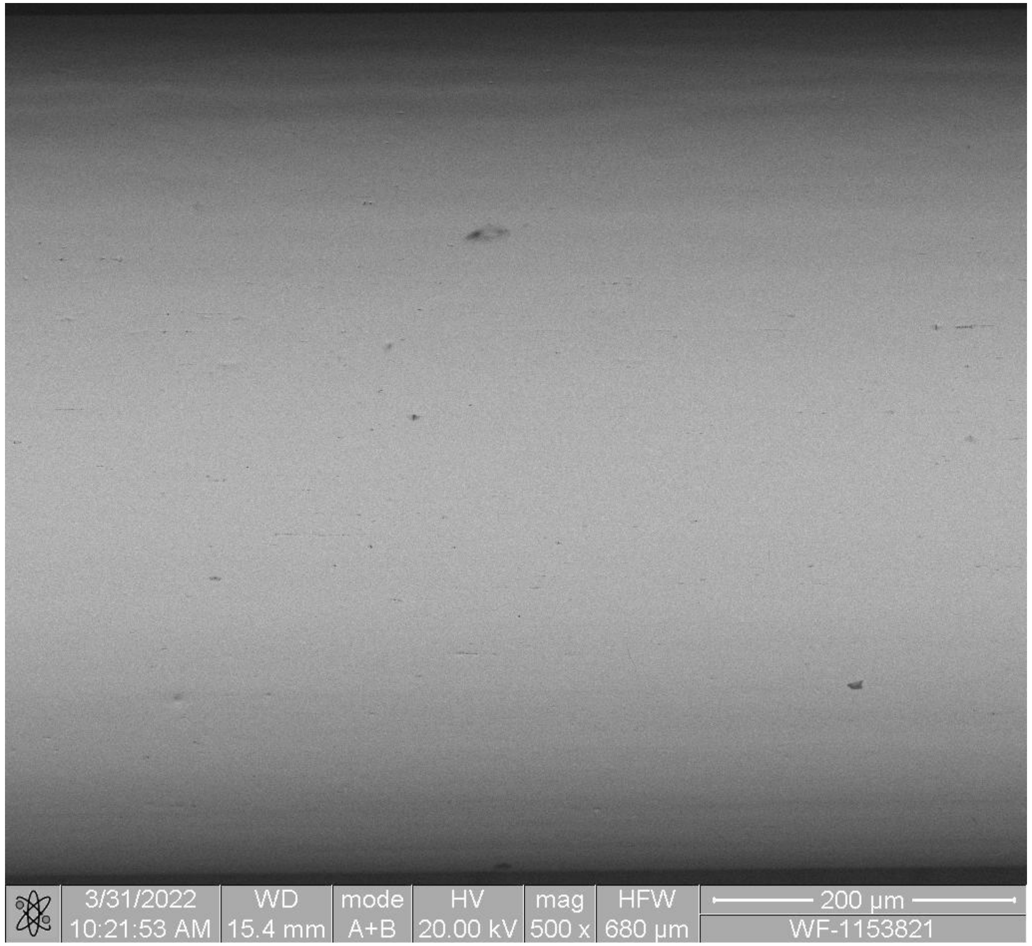
SEM-EDX



- Pitting region was characterized using SEM/EDX techniques

3. Applying 8 % strain on the sintered- wire- SEM surface study

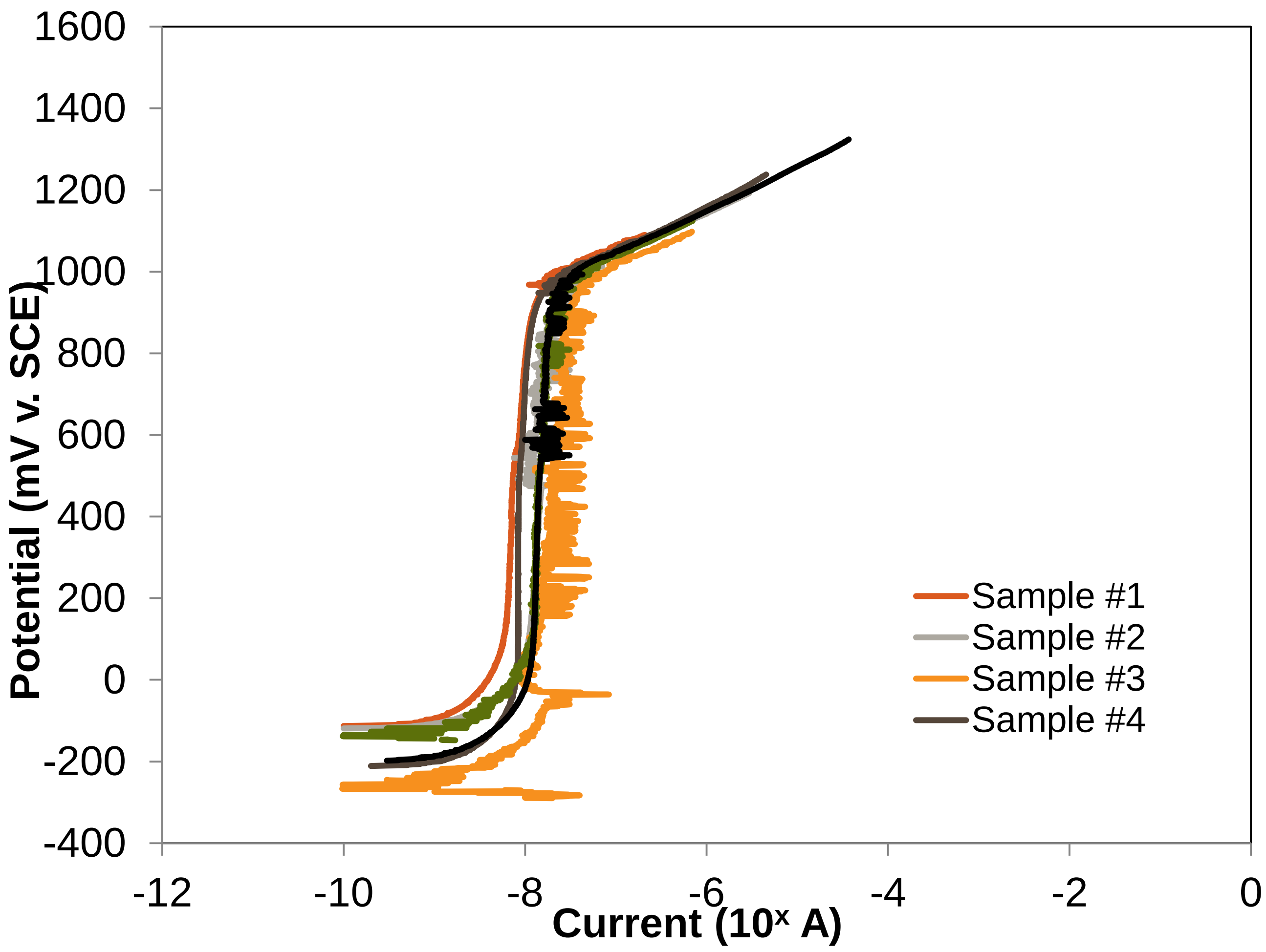
after heat treatment of EPed wire at 360 C for 10 min in air furnace, the wire was deformed at 8 % strain for 1 min



- Applying 8% strain on sintered wire did not show any significant effect on the surface of the wire
- No sign of any surface defects/ crack was observed.

3. Applying 8 % strain on the sintered- wire- corrosion test

after heat treatment of EPed wire at 360 C for 10 min in air furnace, the wire was deformed at 8 % strain for 1 min



Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
1	-111	n/a	942
2	-374	n/a	991
3	-288	n/a	963
4	-209	n/a	923
5	-146	n/a	954
6	-197	n/a	959

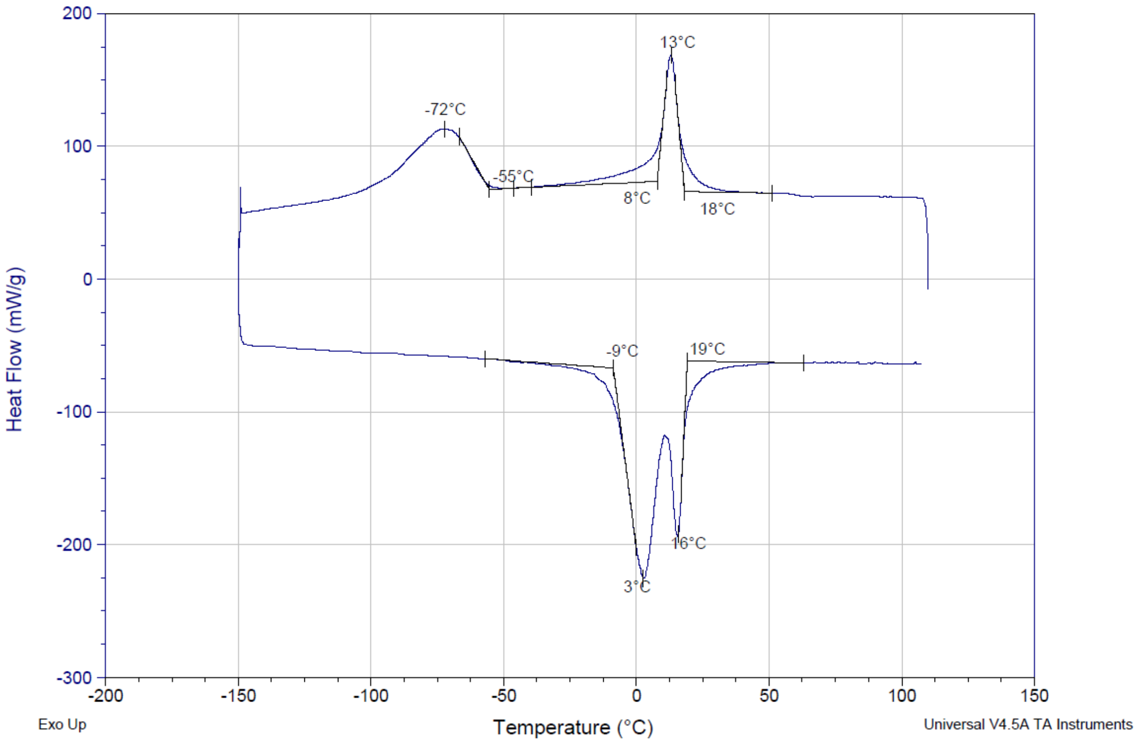
- All 6 wires reached Oxygen evolution
- No pitting/break down was detected.

Conclusions

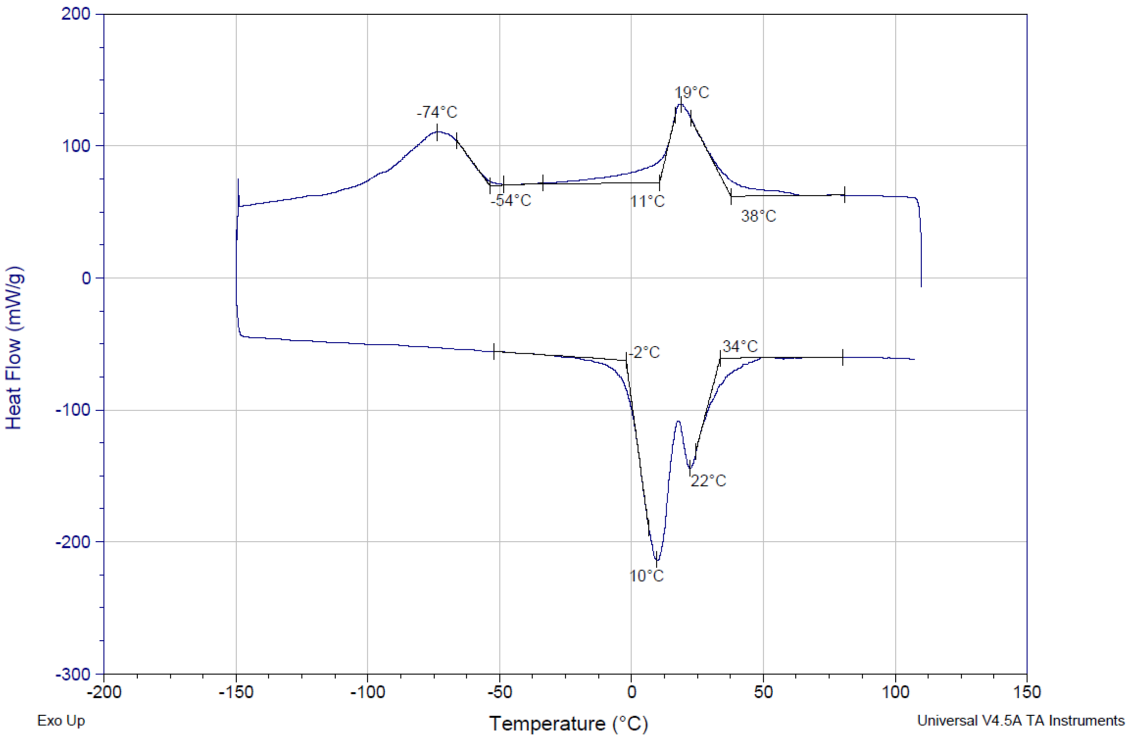
- Post processing heat treatment during ePTFE cover, slightly degrades the potentiodynamic corrosion properties of NiTi wire.
- Lowest beakdown potential was 407 mV which is still higher than accepted criteria for NiTi devices (300 mV)
- Applying 8 % strain (stimulating the crimping procedure) did not have any effect on corrosion properties of the device

DSC results

As -EPed

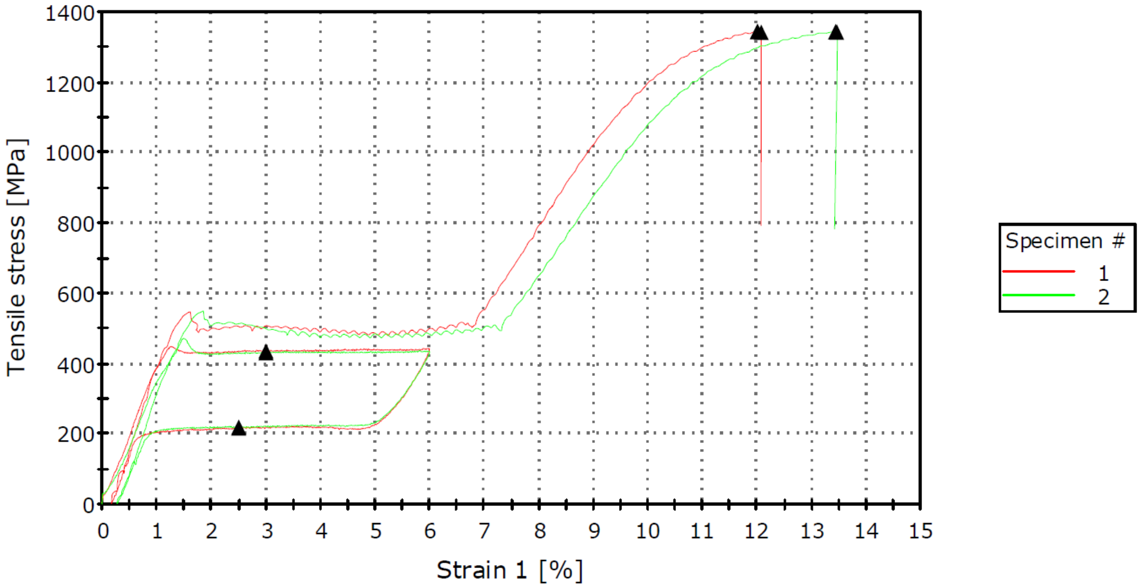


As –Eped+ 360 C for 10 min



Tensile tests

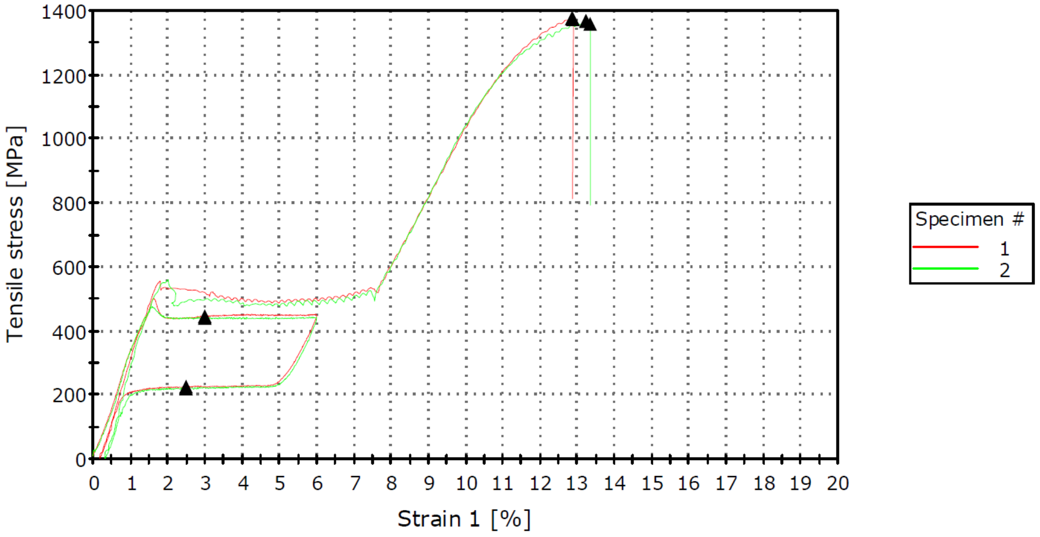
As -EPed



Results table 1

	Specimen label	UPS @ 3% [MPa]	LPS @ 2.5% [MPa]	UTS [MPa]	Elr [%]	Elu [%]	El at Break [%]	Comment
1	Test1	438	219	1344	0.2	12.0	12.1	
2	Test2	433	222	1344	0.3	13.4	13.4	
Mean		436	221	1344	0.2	12.7	12.8	
Standard deviation		3	2	0	0.1	1.0	1.0	

As –Eped+ 360 C for 10 min



Results table 1

	Specimen label	UPS @ 3% [MPa]	LPS @ 2.5% [MPa]	UTS [MPa]	Elr [%]	Elu [%]	El at Break [%]	Comment
1	Test1	448	228	1376	0.0	12.8	12.9	OD= .56mm
2	Test2	443	223	1368	0.3	13.2	13.3	OD= .56mm
Mean		445	226	1372	0.2	13.0	13.1	
Standard deviation		4	4	6	0.2	0.3	0.3	

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Questions ?

TableI: Corrosion results SMST Group1

Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
As Is	-74	212	n/a
HT 360c/10mins AC	5	1040	n/a
HT 550c/10mins WQ	-411	24	n/a
550c + 360c/10mins WQ+AC	-86	123	n/a

TableII: Corrosion results SMST Group2

Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
EP As Is	-307	n/a	978
EP+HT 360c/10mins AC	43	n/a	980
HT 550c/10mins WQ+ EP	-349	n/a	969
550c/10mins WQ+ EP + 360c/10mins AC	63	n/a	1000

TableIII: Corrosion results SMST Group3 Strain @4%

Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
EP+4% Strain, 360c/10mins Air Cool	-2	721	n/a
EP+4% Strain, 550c/10m SP+WQ	-324	409	n/a
EP+4% Strain+ SP 550c/10m+360c/10m AC	-261	n/a	1420
EP+ST wire SP 550c/10m WQ,+4% strain +360c/10m AF AC	-234	n/a	1020

TableIV: Corrosion results SMST Group4 Strain @3%

Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
EP+3% Strain, 360c/10mins Air Cool	17	n/a	968
EP+3% Strain, 550c/10m SP+WQ	-318	n/a	961
EP+3% Strain+ SP 550c/10m+360c/10m AC	-144	1290	n/a
EP+ST wire SP 550c/10m WQ,+3% strain +360c/10m AF AC	-286	n/a	1010

TableV: Corrosion results SMST Group4 Strain @2%

Sample #	E_r (mV v. SCE)	E_b (mV v. SCE)	$E_{ox\ ev}$ (mV v. SCE)
EP+2% Strain, 360c/10mins Air Cool	3	n/a	995
EP+2% Strain, 550c/10m SP+WQ	-34	n/a	1393
EP+2% Strain+ SP 550c/10m+360c/10m AC	-325	n/a	1020
EP+ST wire SP 550c/10m WQ,+2% strain +360c/10m AF AC	-280	n/a	987