Constant Stress Load Bias Tests

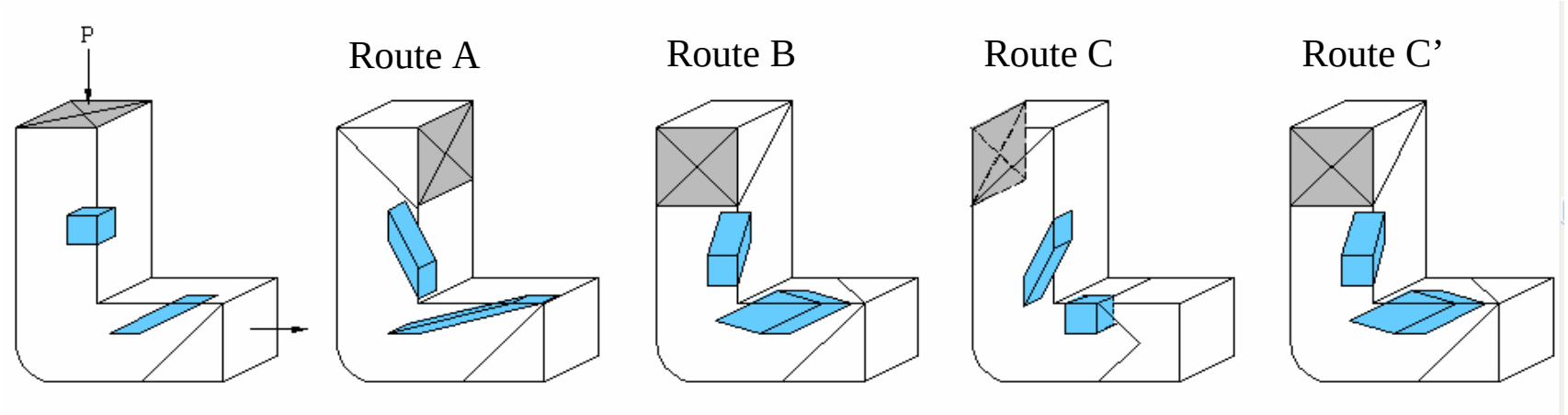
- Samples Tested:
  - As received 55NiTi (Hot rolled at 900°C)
  - ECAE processed 55NiTi
    - Route 4Bc, 350°C *
    - Route 4Bc, 400°
    - Route 8Bc, 400°C *

*: Two cycles were run at each stress level. Cooling of 1st cycle and heating of 2nd cycle was graphed.

Equal Channel Angular Extrusion Process at Texas A&M

First Pass (N=1)

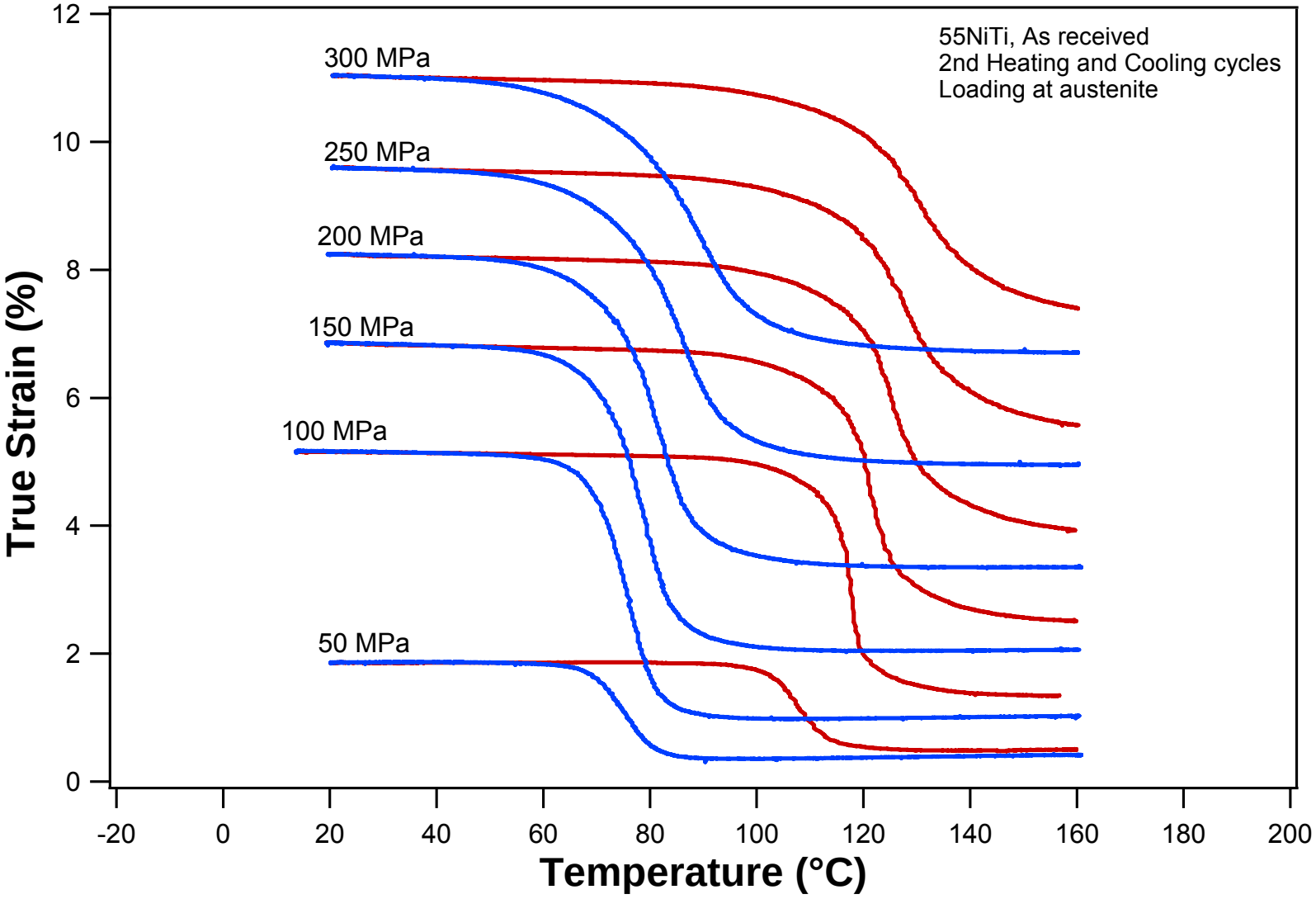
Second Pass (N=2)



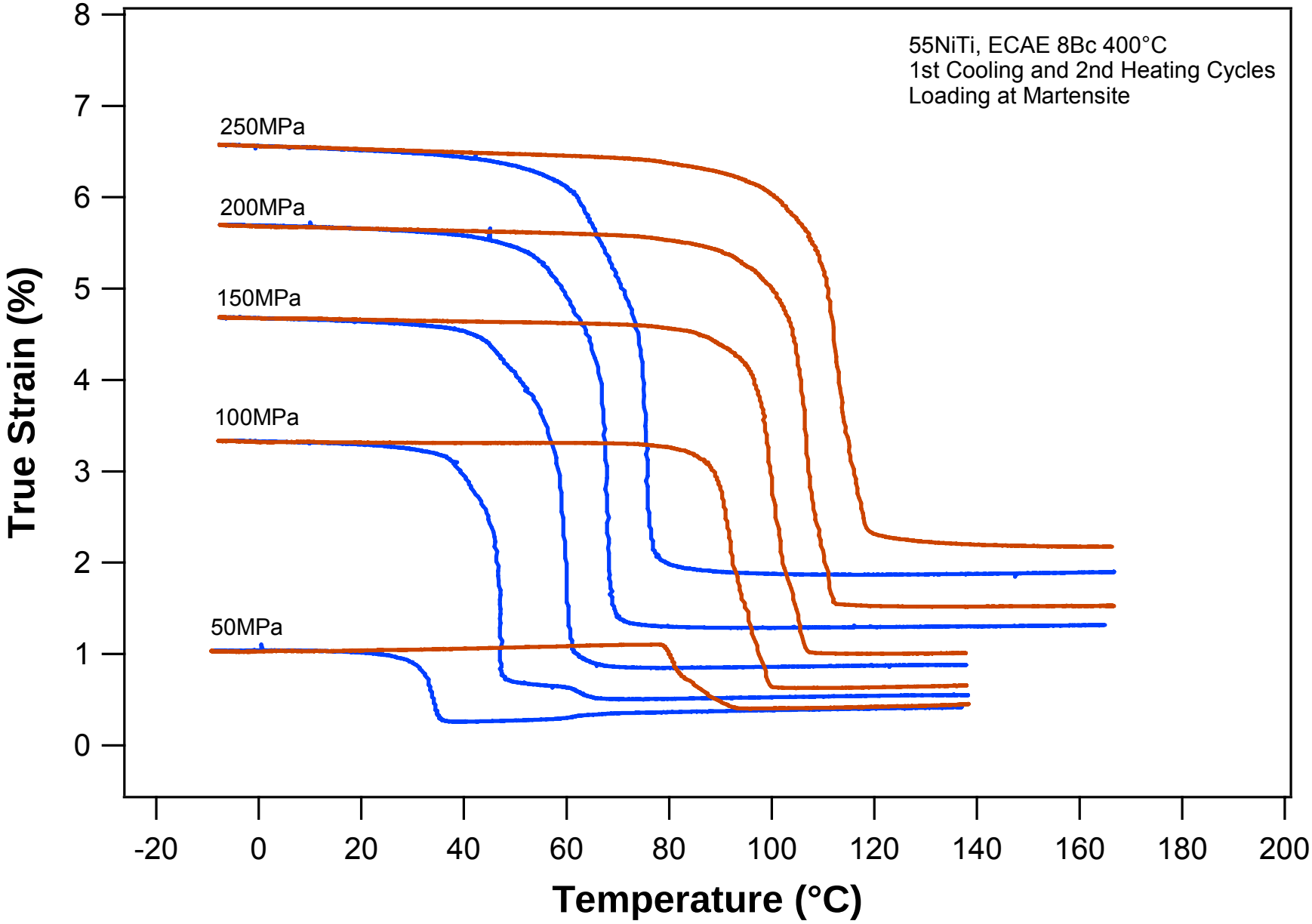
Route name	Min. # of passes	Billet rotations about the extrusion axis				Material Yield*	Effect on microstructure
		1 →	2 →	3 →	4 → N		
A	1	0°	0°	0°	etc.	0.58	elongation (lamellar)
B (B_A)	2	+90°	-90°	+90°	etc.	0.67	elongation (filamentary)
C	2	180°	180°	180°	etc.	0.83	back/forth shearing
C' (B_C)	4	+90°	+90°	+90°	etc.	0.67	back/forth cross-shearing
E	4	180°	90°	180°	etc.	0.78	back/forth cross-shearing

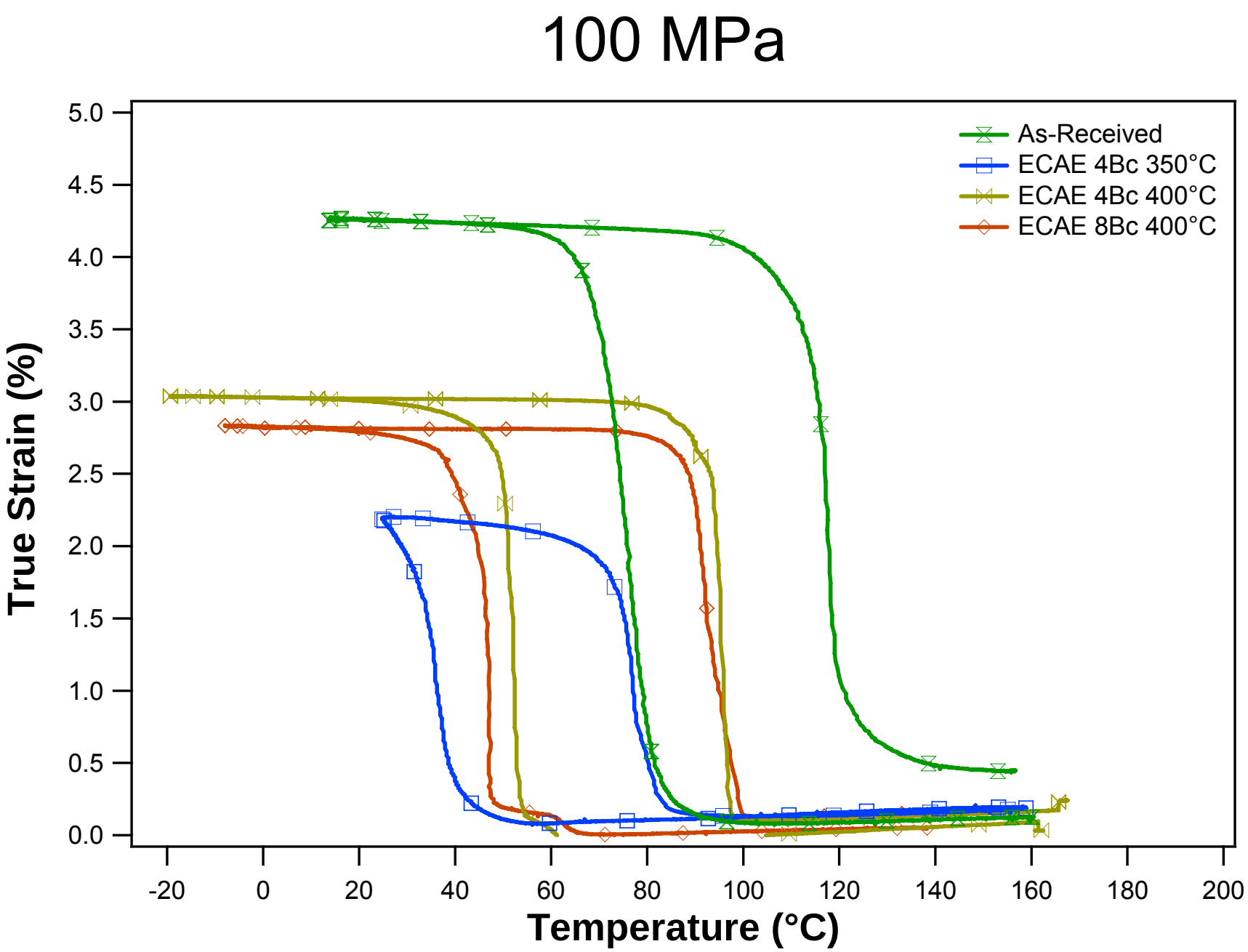
* Theoretical yield of fully deformed material after N=4 in billet with length/width ratio of 6

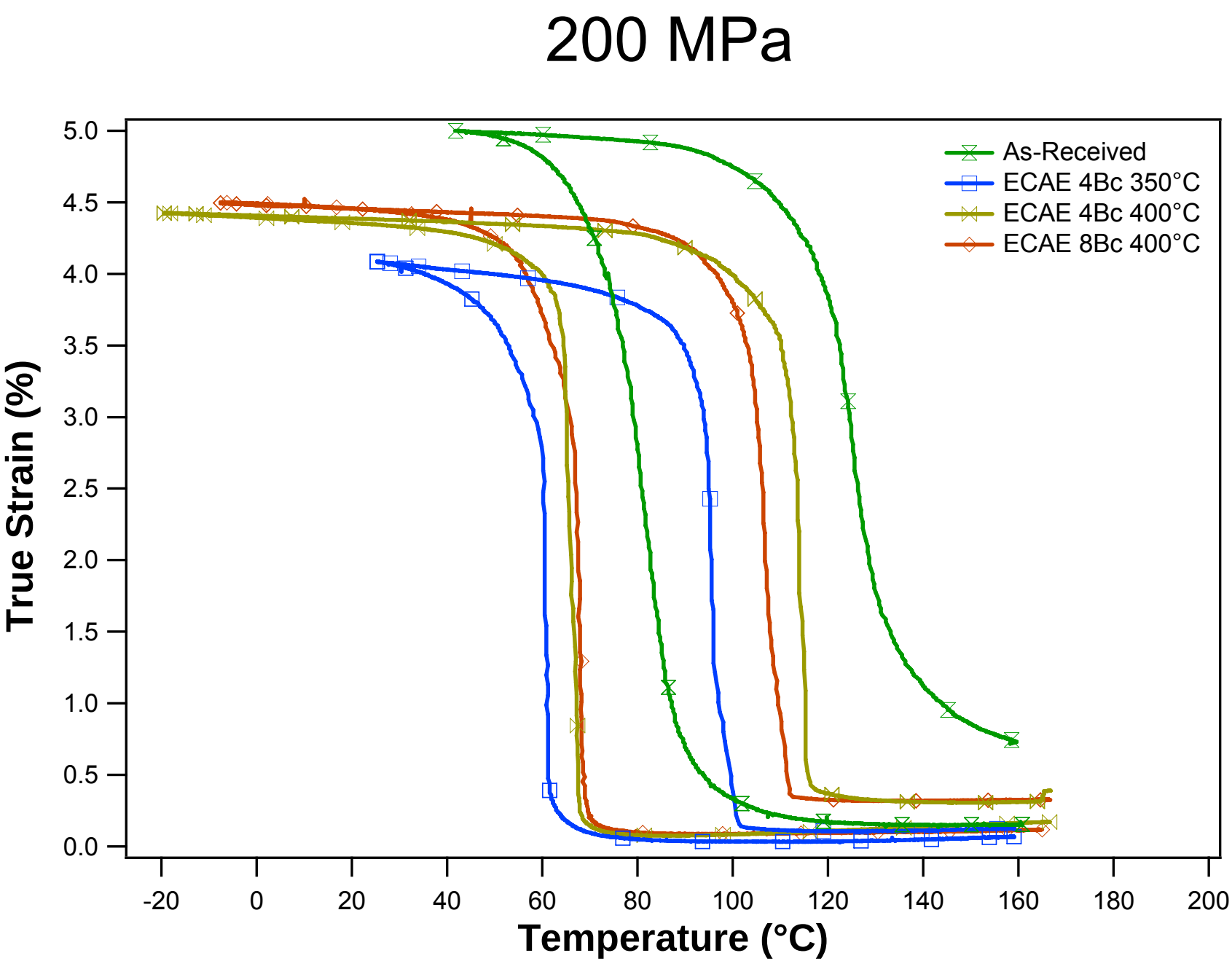
As-Received

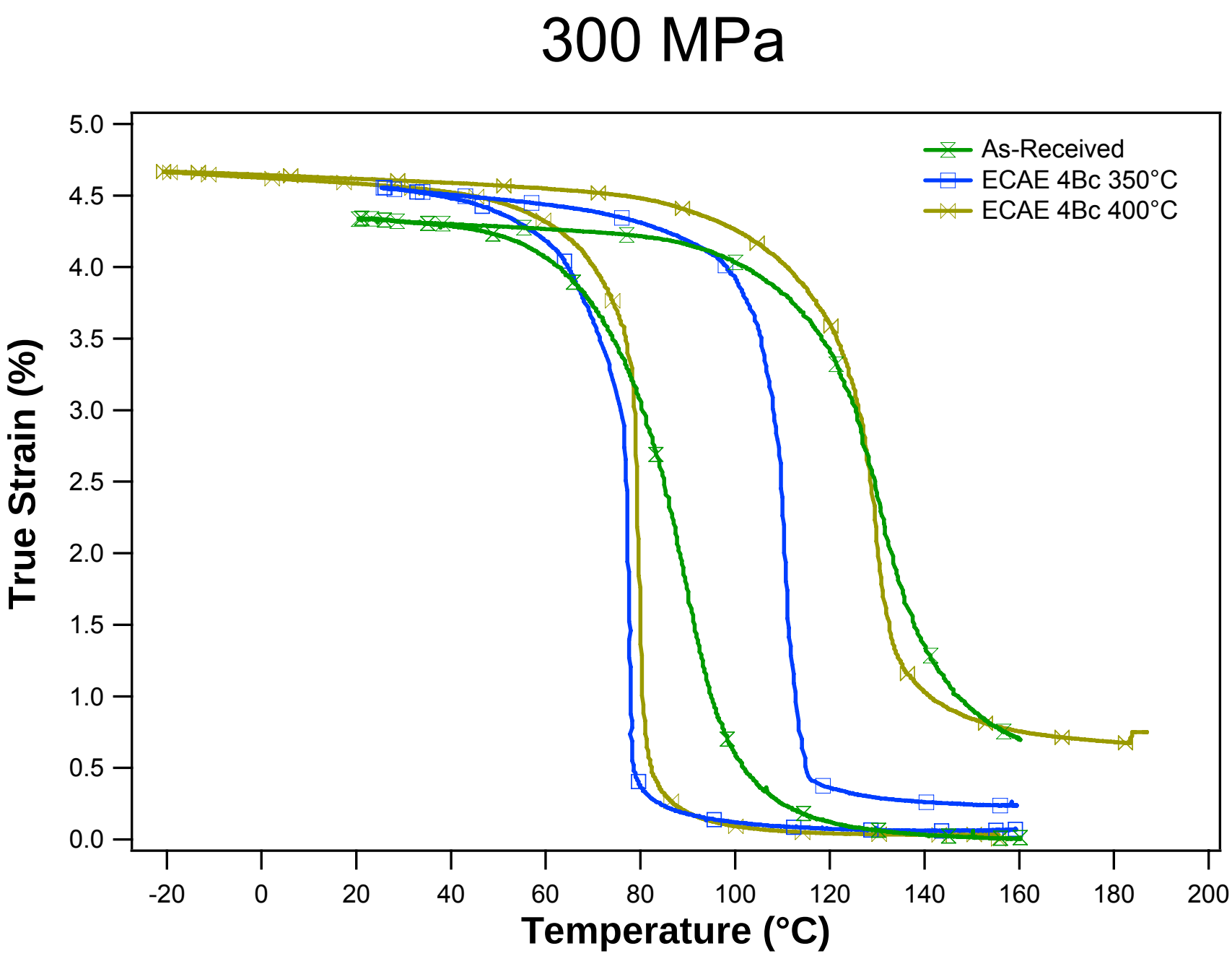

55NiTi, As received
2nd Heating and Cooling cycles
Loading at austenite
300 MPa
250 MPa
200 MPa
150 MPa
100 MPa
50 MPa
True Strain (%)
Temperature (°C)
0
2
4
6
8
10
12
-20
0
20
40
60
80
100
120
140
160
180
200

ECAE 8Bc at 400°C

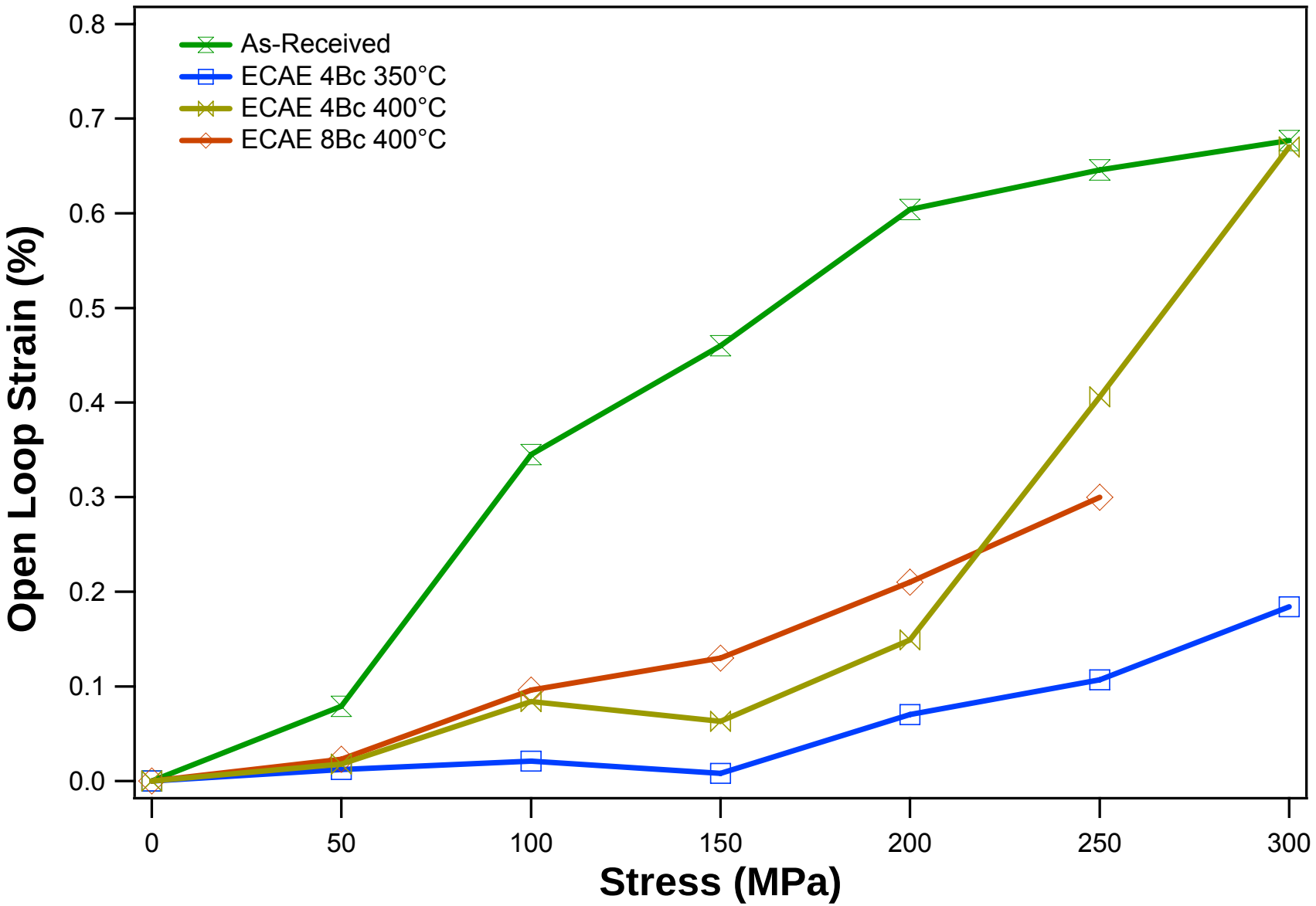


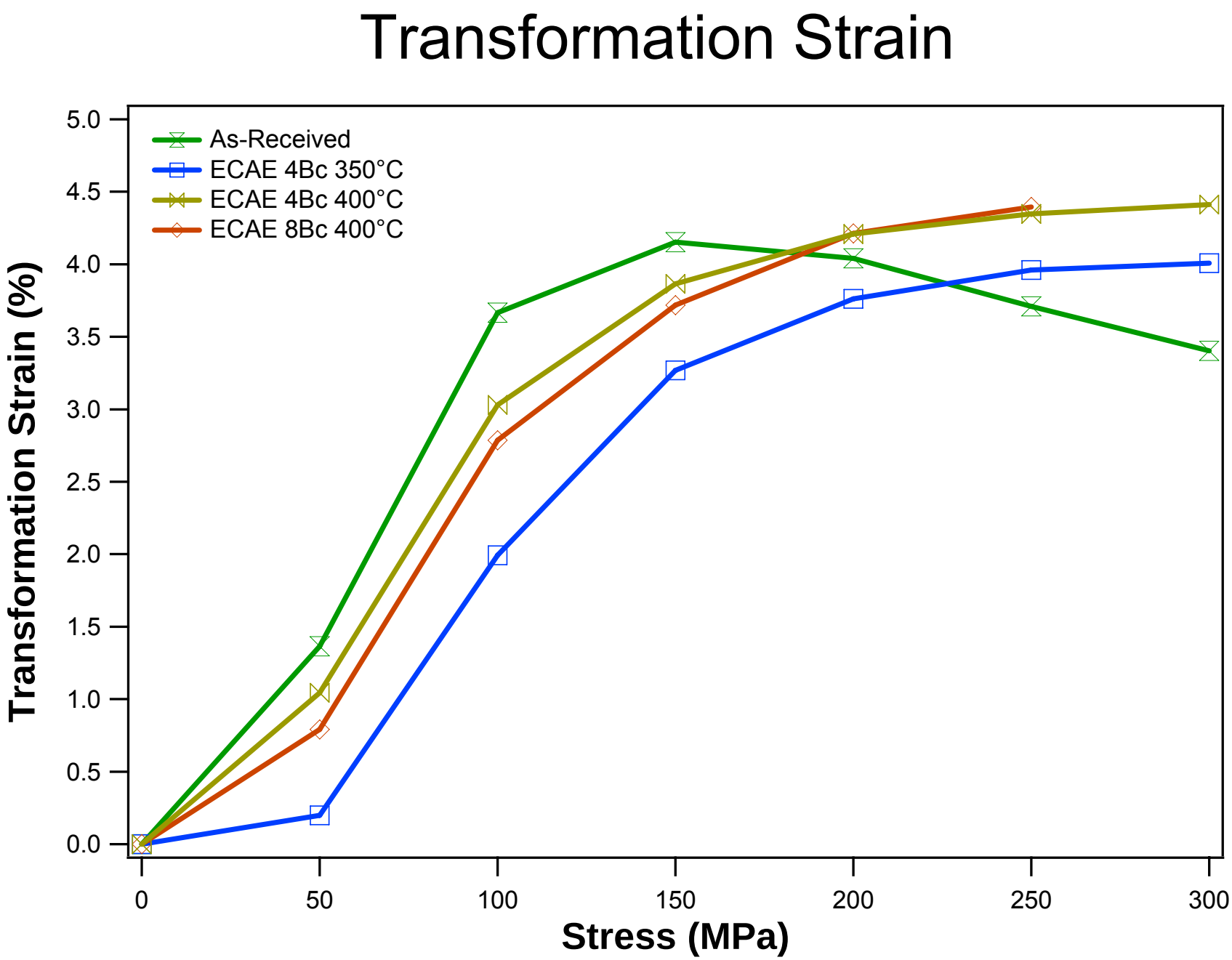

100 MPa
As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
ECAE 8Bc 400°C
True Strain (%)
Temperature (°C)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-20
0
20
40
60
80
100
120
140
160
180
200


200 MPa
As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
ECAE 8Bc 400°C
True Strain (%)
Temperature (°C)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-20
0
20
40
60
80
100
120
140
160
180
200

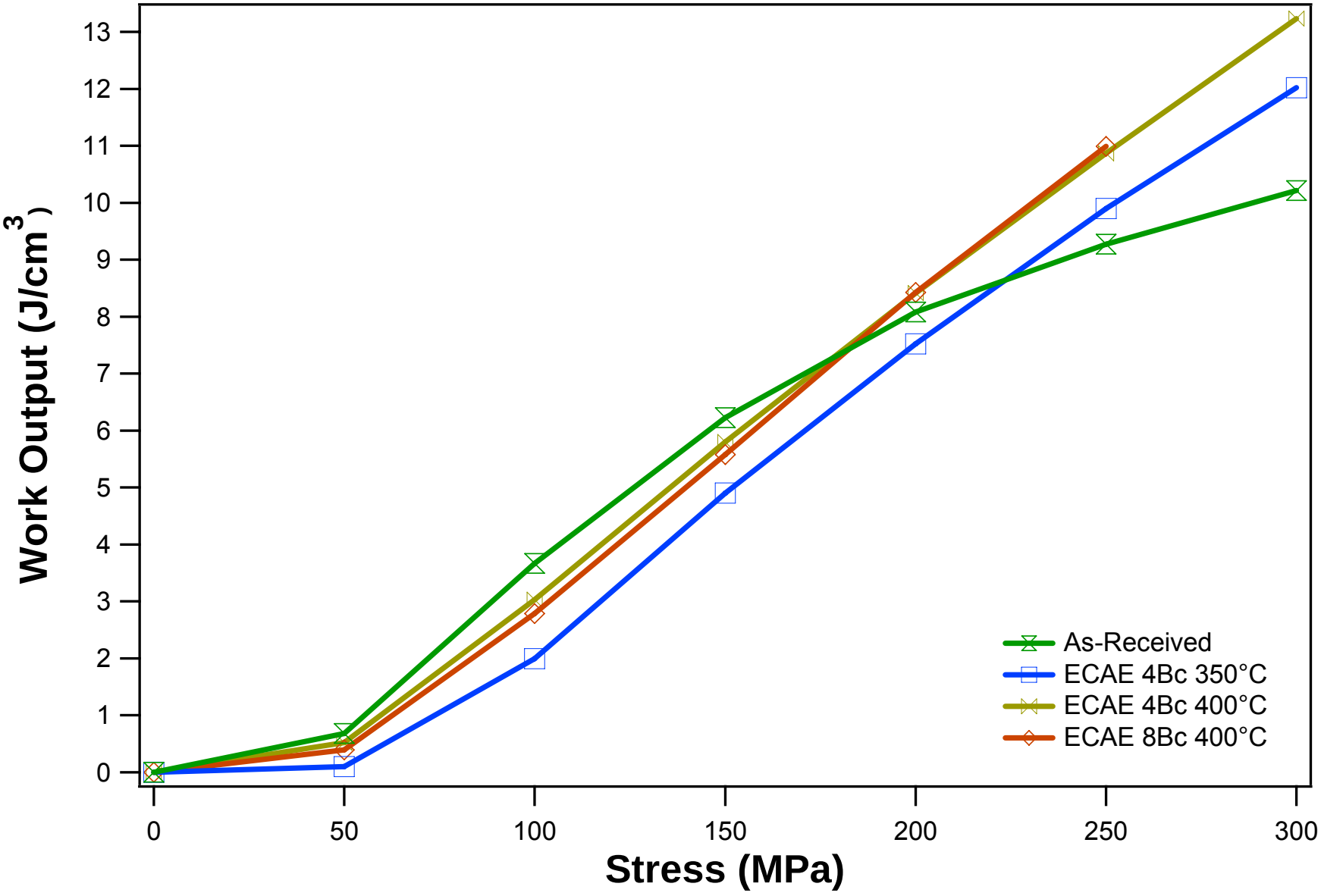

300 MPa
As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
True Strain (%)
Temperature (°C)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-20
0
20
40
60
80
100
120
140
160
180
200

Open Loop Strain

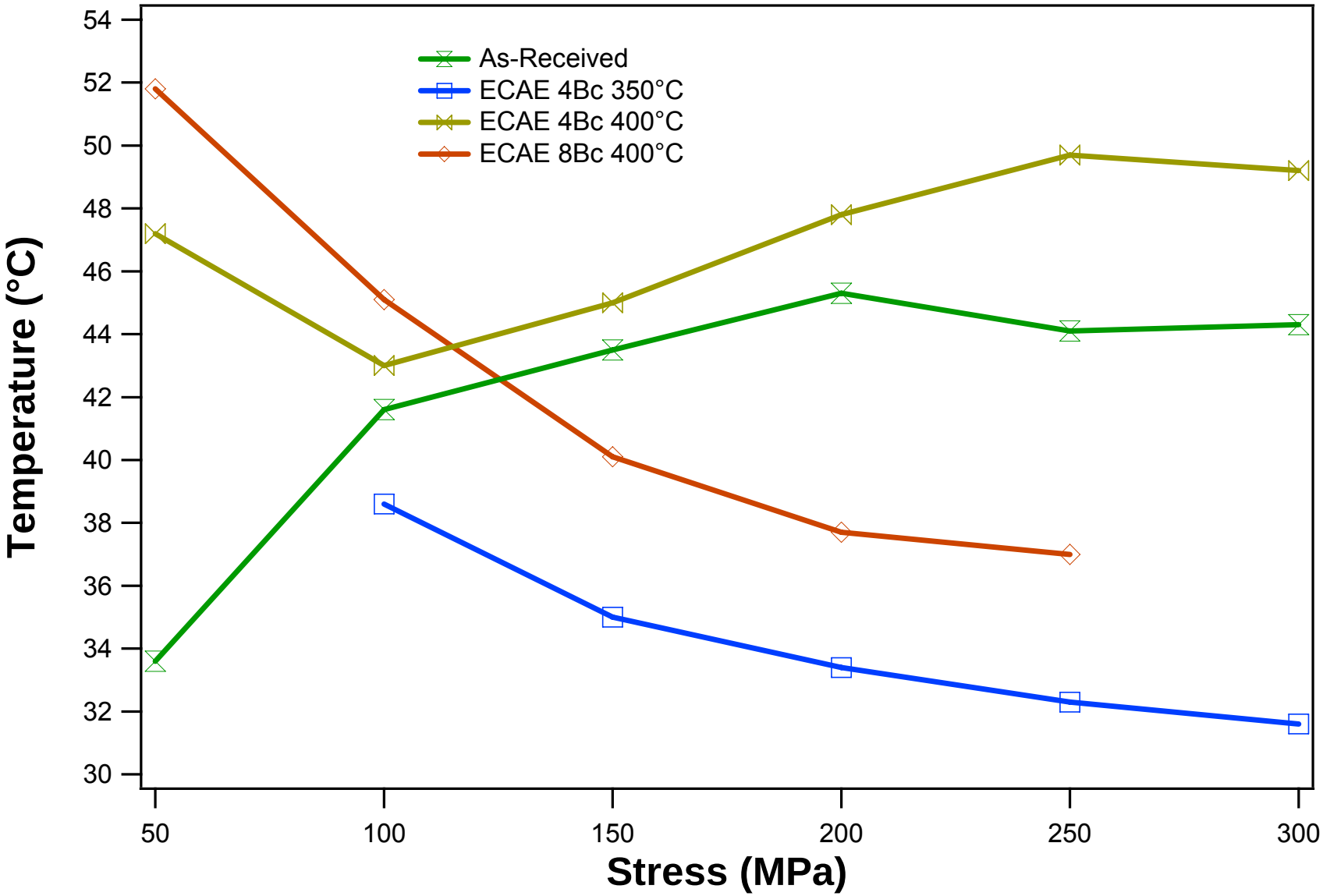



Transformation Strain
As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
ECAE 8Bc 400°C
Transformation Strain (%)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
50
100
150
200
250
300
Stress (MPa)

Work Output

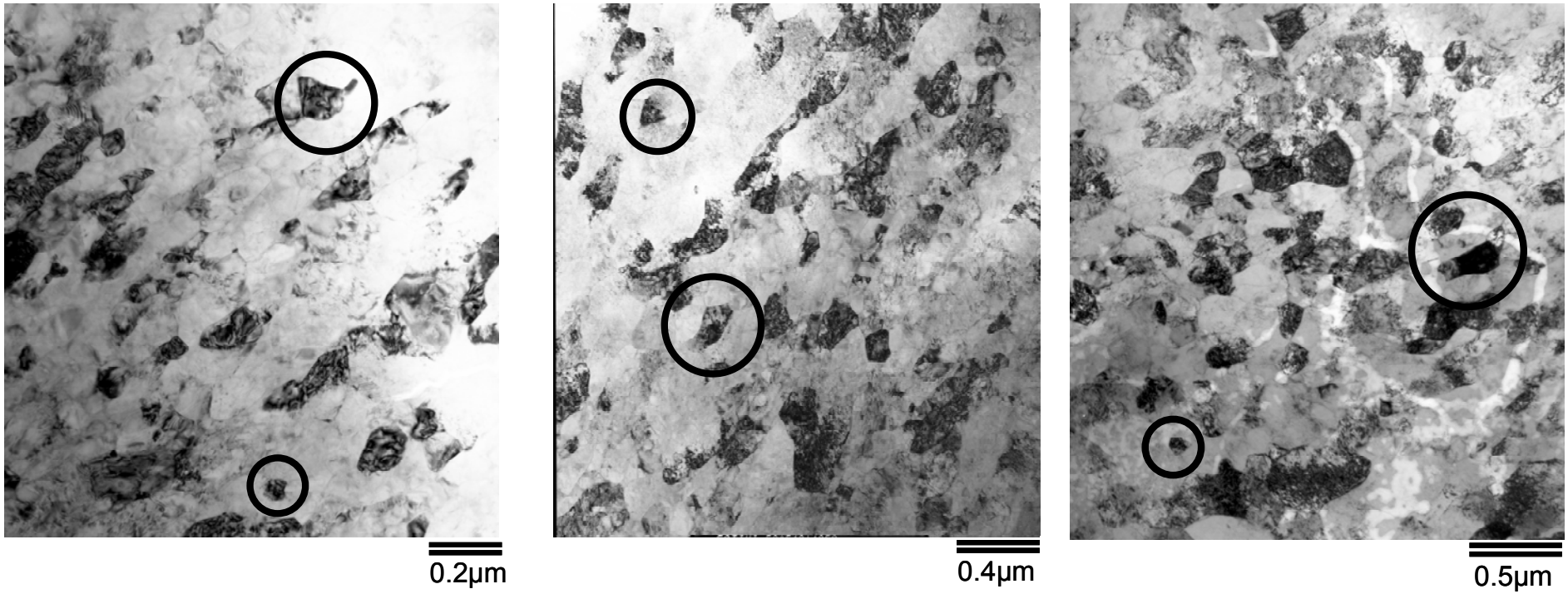

Work Output (J/cm3)
Stress (MPa)
0
1
2
3
4
5
6
7
8
9
10
11
12
13
0
50
100
150
200
250
300
As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
ECAE 8Bc 400°C

Hysteresis


As-Received
ECAE 4Bc 350°C
ECAE 4Bc 400°C
ECAE 8Bc 400°C
Temperature (°C)
54
52
50
48
46
44
42
40
38
36
34
32
30
50
100
150
200
250
300
Stress (MPa)

Grain Size Comparison

Old Results



(a) (b) (c)

Figure 3. Bright field TEM images recorded above A_f at 175°C showing the grain size reduction due to ECAE processing. Four ECAE passes following (a) route B_c at 400°C b) route B_c at 425°C and (c) route E at 450°C.

$NiTiPd_{25}$ Characterization Results

As-received material

<u>Nominal compositions</u>

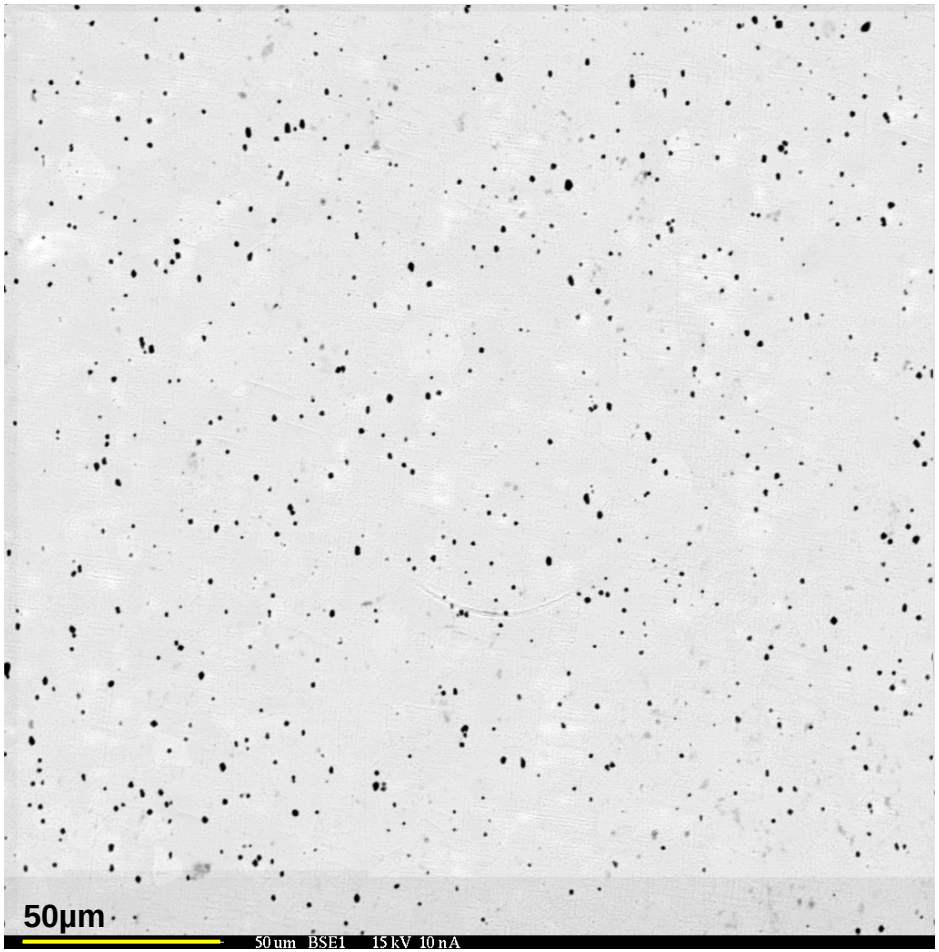
$Ni_{25}Ti_{50}Pd_{25}$ (at. %)

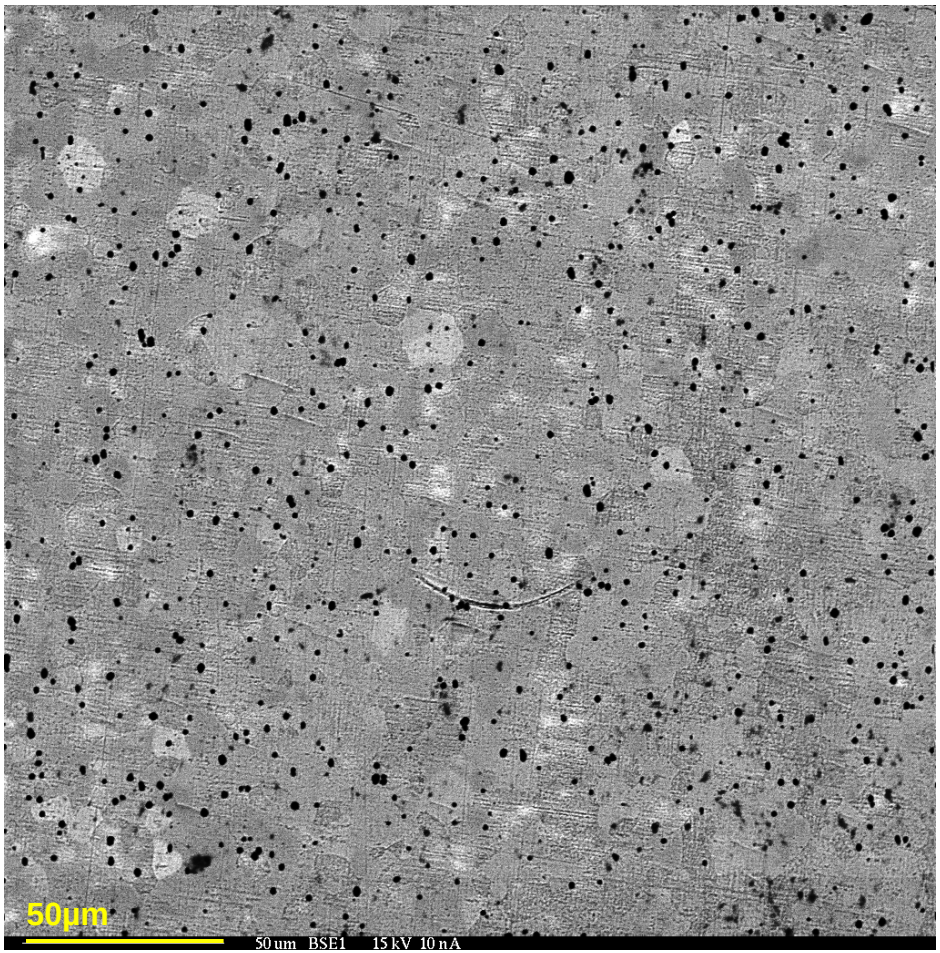
$Ni_{22.50}Ti_{36.71}Pd_{40.79}$ (wt. %)

<u>Processing conditions</u>

Extruded at 900°C (7:1 area reduction)

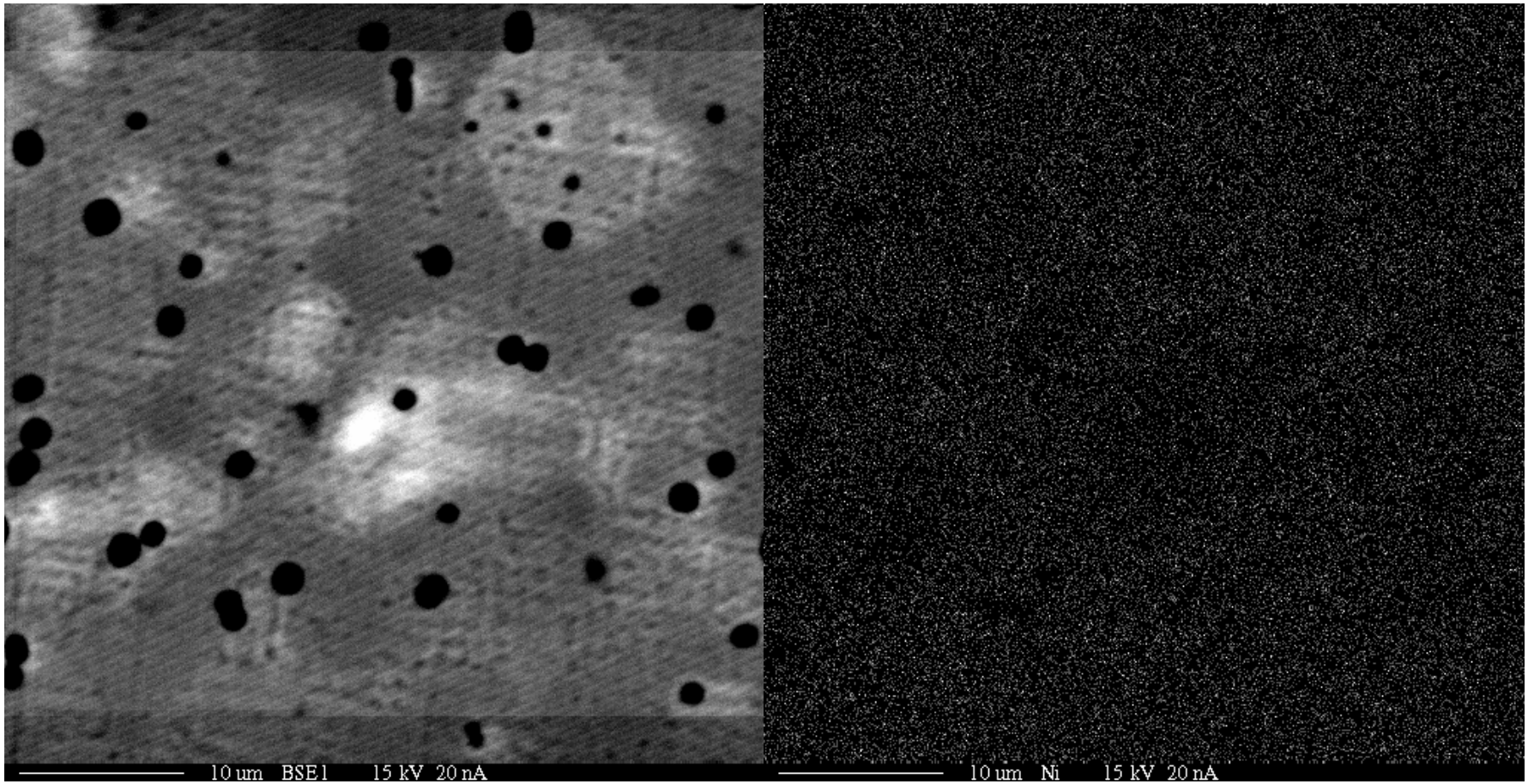
BSE Images – Ext.65





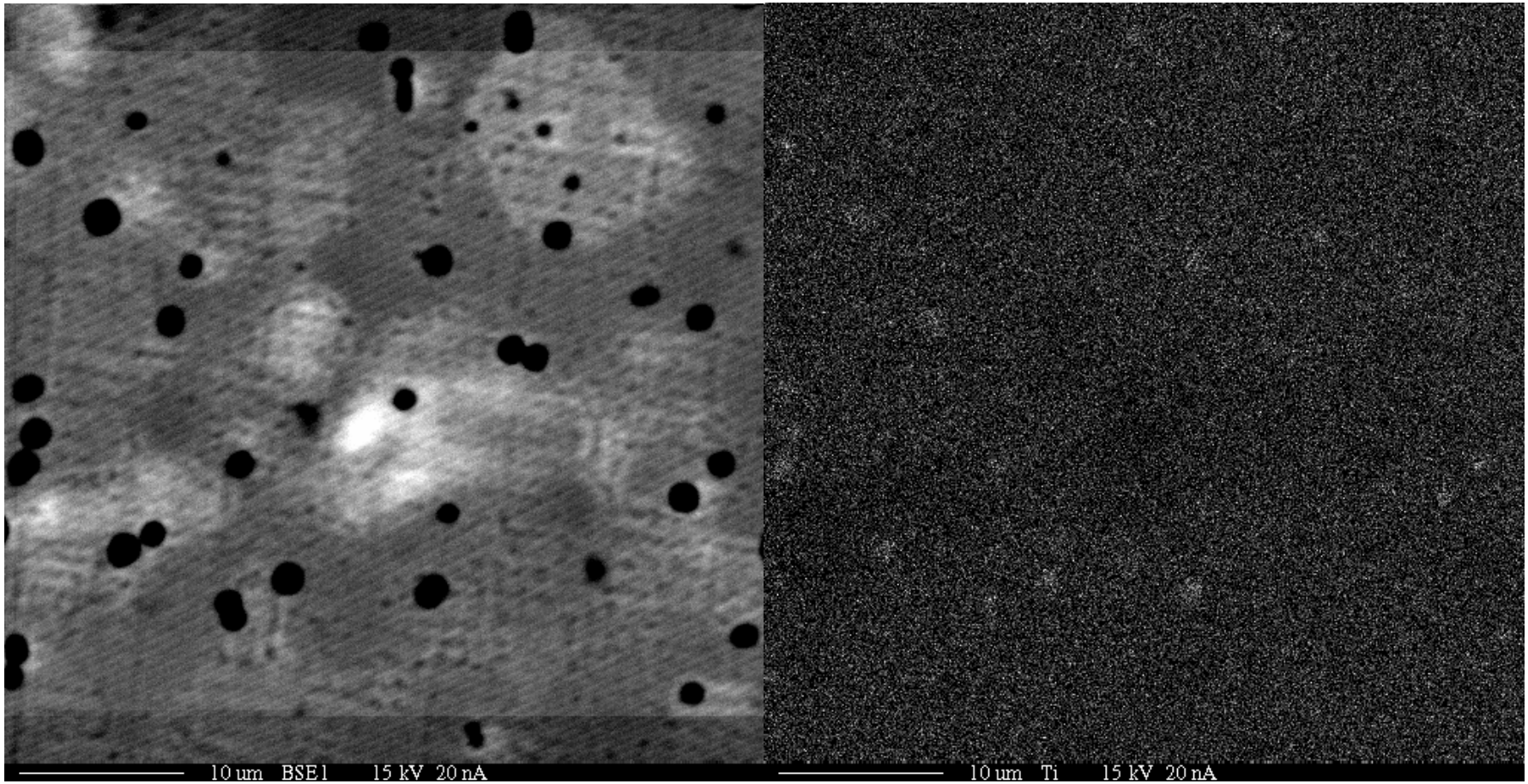
Images taken from etched samples

WDS X-Ray Mapping for Ni – Ext. 65



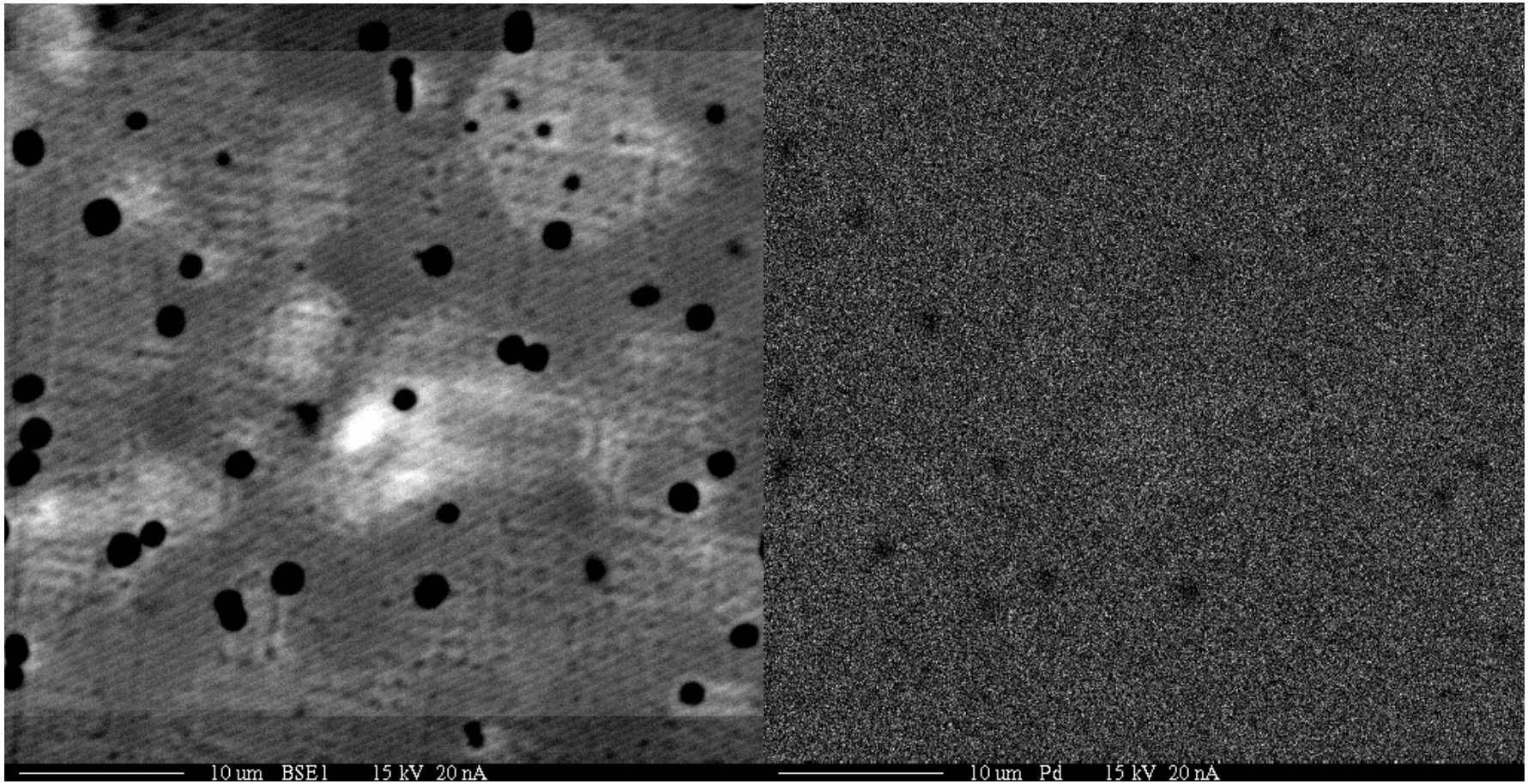
Short time (5 minute) WDS X-ray map of the etched sample

WDS X-Ray Mapping for Ti – Ext. 65

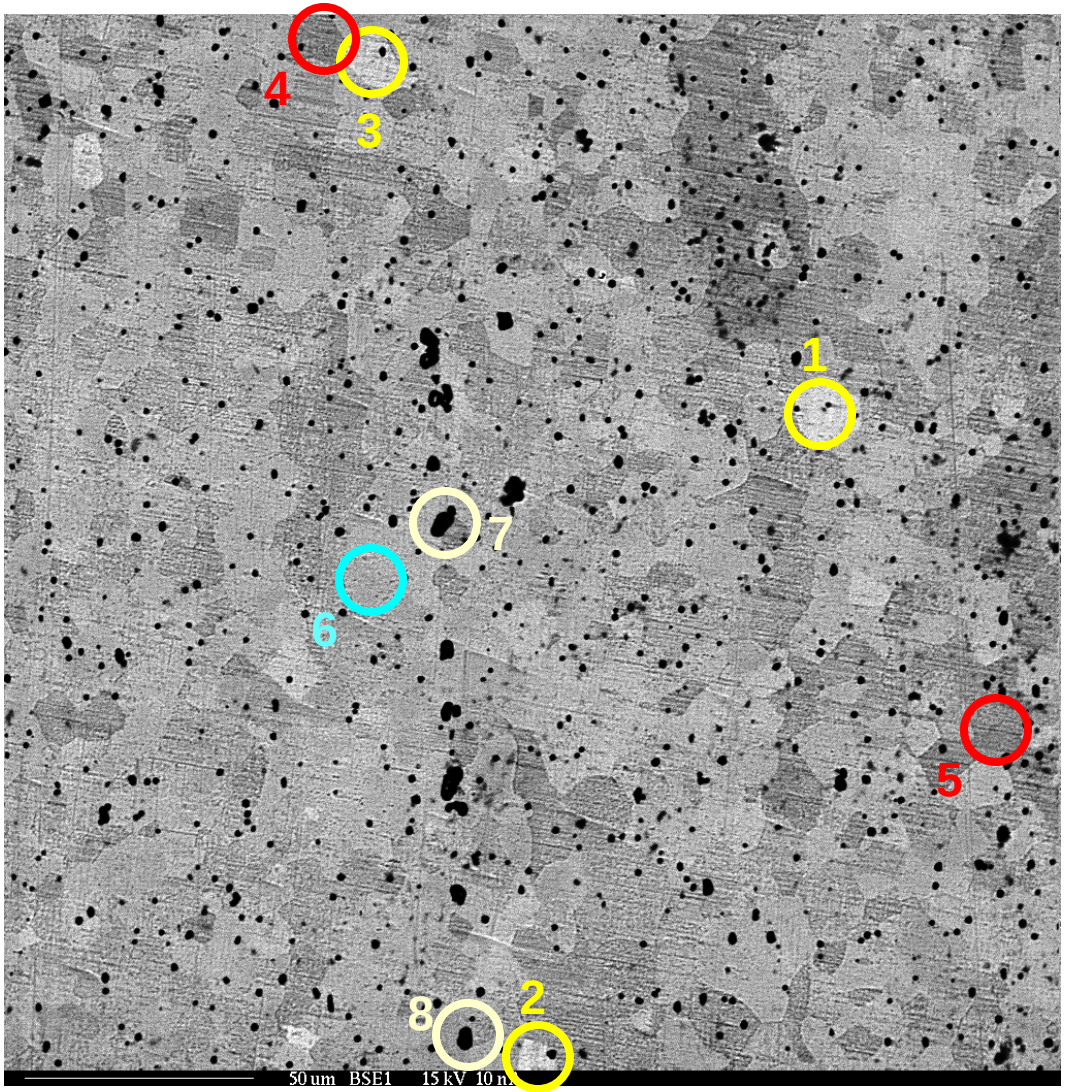


Short time (5 minute) WDS X-ray map of the etched sample

WDS X-Ray Mapping for Pd – Ext. 65



Short time (5 minute) WDS X-ray map of the etched sample



SEM:BSE (etched sample)

WDS Analyses of Different Regions

	Pd (at%)	Ti (at%)	Ni (at%)	Total (wt%)
1	27.17	47.17	25.66	100.21
2	27.19	47.81	25.01	99.69
3	28.65	45.94	25.41	99.54
4	31.29	44.02	24.69	96.67
5	31.83	43.96	24.21	96.90
6	27.77	46.90	25.34	99.90
7	0.29	99.29	0.42	82.10
8	1.25	97.88	0.87	82.61

1-3: Bright regions
4-5: Dark regions
6: Matrix
7-8: Precipitate

Chemical Analyses (Ext 64)

Analysis No	Ni (at. %)		Ti (at. %)		Pd (at. %)	
	TAMU	NASA	TAMU	NASA	TAMU	NASA
1		24.9		49.2		25.6

NASA GRC results from Inductively Coupled Plasma (ICP) Emission Spectrometry

Chemical Analyses (Ext 65)

Analysis No	Ni (at. %)		Ti (at. %)		Pd (at. %)	
	TAMU	NASA	TAMU	NASA	TAMU	NASA
1	24.4	25.3	48.0	47.8	27.7	26.7
2	24.7	24.9	47.9	47.9	27.4	26.4
3	24.7		47.7		27.5	
4*	24.8		49.0		26.2	
5*	24.6		49.1		26.2	
6*	24.7		48.9		26.3	

NASA GRC results from Inductively Coupled Plasma (ICP) Emission Spectrometry

TAMU results from Wavelength dispersive X-ray Spectrometry (WDS) (Average values)

* Analysis done on **unetched** sample

Chemical Analyses
(Inductively Coupled Plasma (ICP) Emission Spectrometry)

	Ext. 65 Analysis 1		Ext. 65 Analysis 2		
Element	Measured wt.%	Measured at.%	Measured wt.%	Measured at.%	Aim at.%
Ni	22.3	25.3	22.2	24.9	25
Pd	42.9	26.7	42.7	26.4	25
Ti	34.6	47.8	34.9	47.9	50
O	0.03065	0.13	0.0303	0.12	
C	ND		0.11	0.6	
N	0.00185	0.009	0.0019	0.009	

	Ext. 64 Analysis 1		
Element	Measured wt.%	Measured at.%	Aim at.%
Ni	22.2	24.9	25
Pd	41.6	25.6	25
Ti	36	49.2	50
O	0.06525	0.26	
C	ND		
N	0.00185	0.009	

DSC Results (Ext 64 & 65)

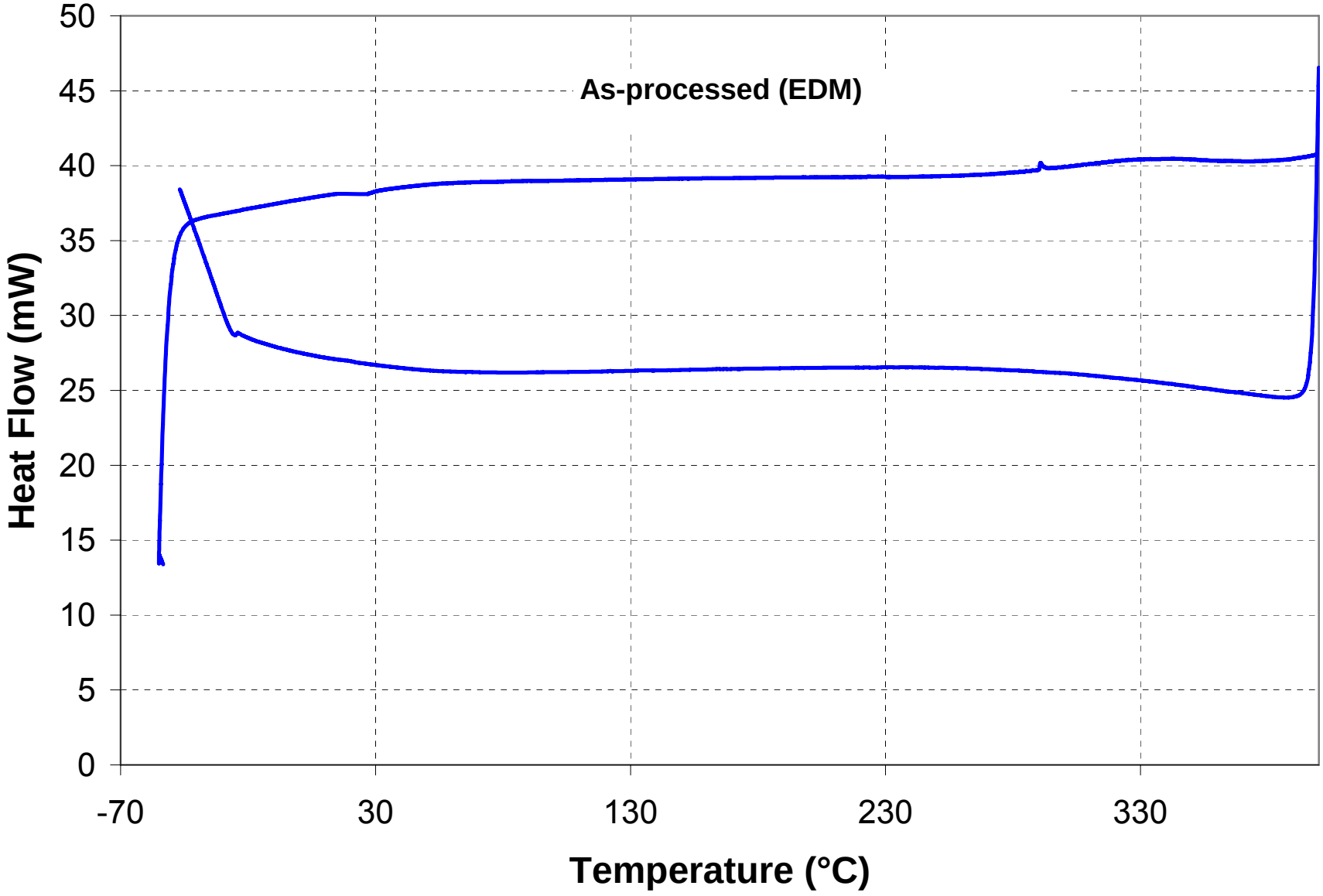
on as-received and heat treated samples

Heat treatment conditions for Ext 64 and Ext 65

Batch	Temperature (°C)	Time (hours)
Ex 64	300	1
	400	10
	500	24
	600	24
Ex 65	300	1
	400	10
	500	24
	600	24

Ex65 – DSC Results

DSC Results - Ext 65, As-processed material



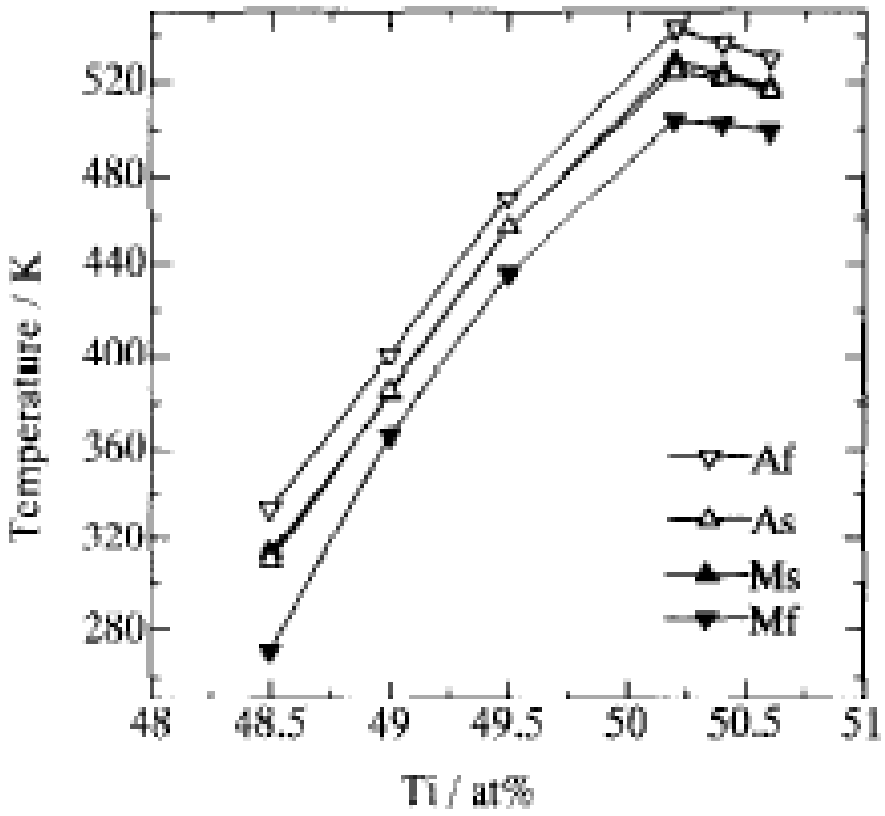
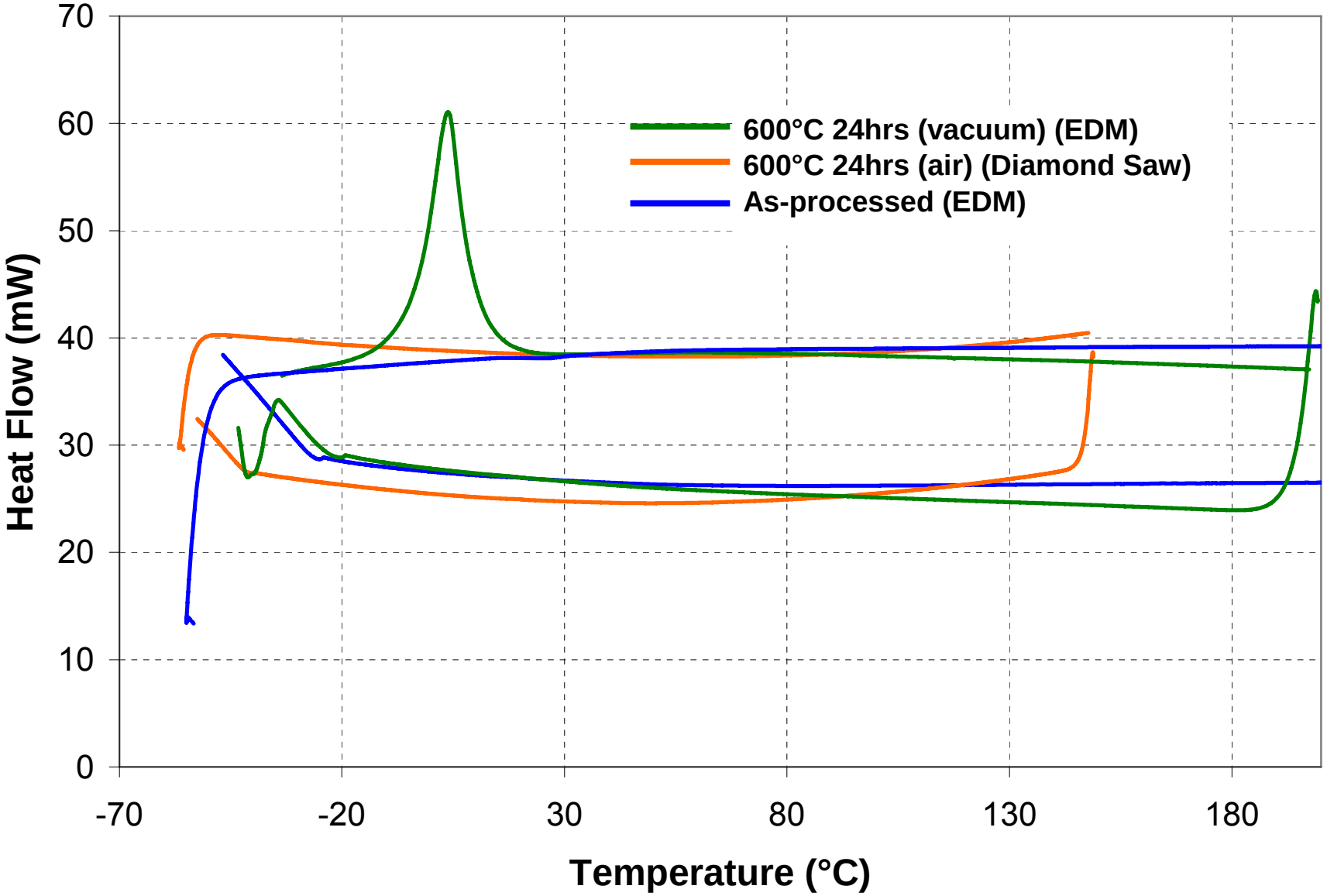
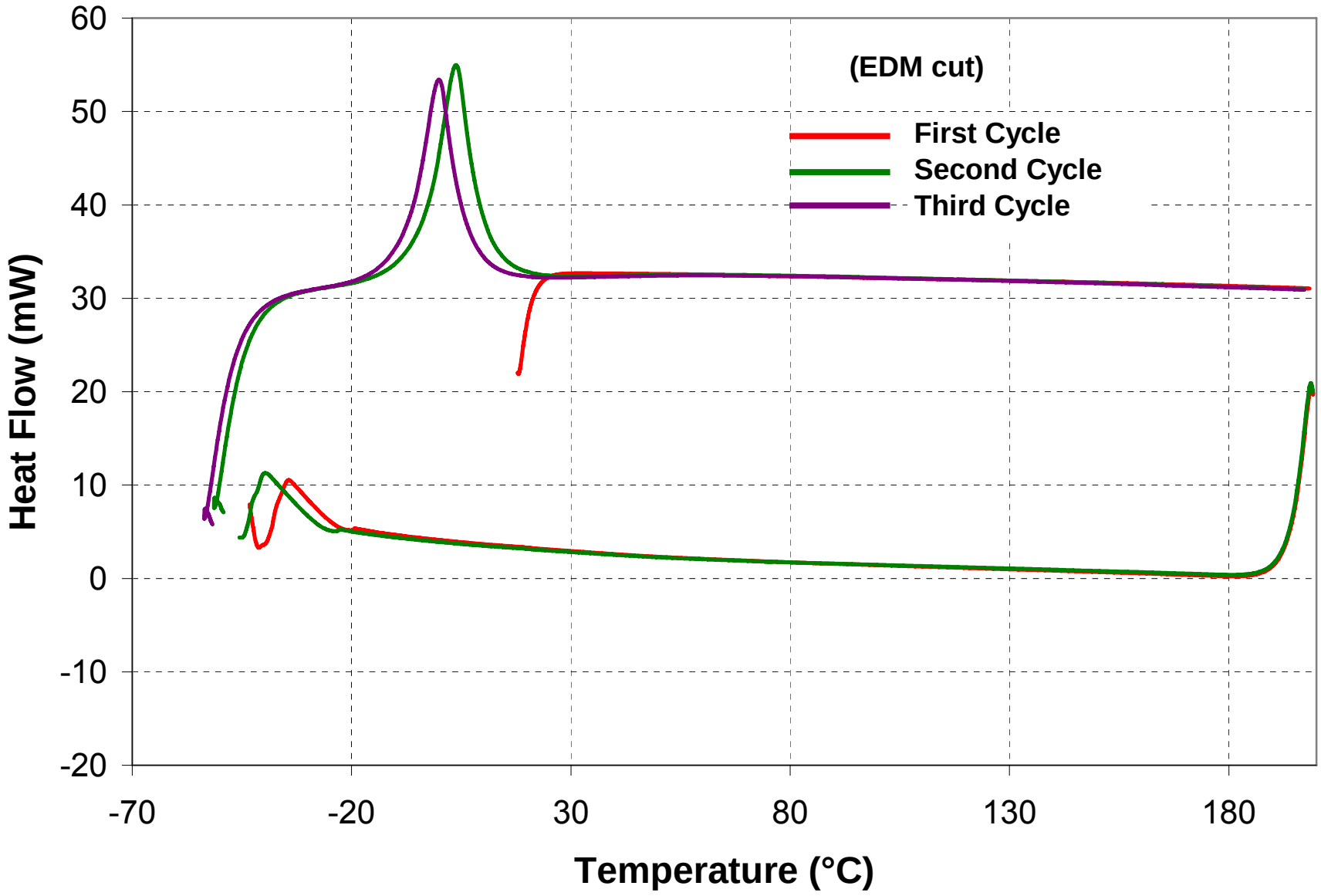


Fig. 2. Plots of martensitic transformation temperatures vs. composition for $Ti_{50-x}Pd_{30}Ni_{20+x}$ ($x = -0.6$–1.5) alloys.

Ex65 - Consolidated DSC data, 1st cycle

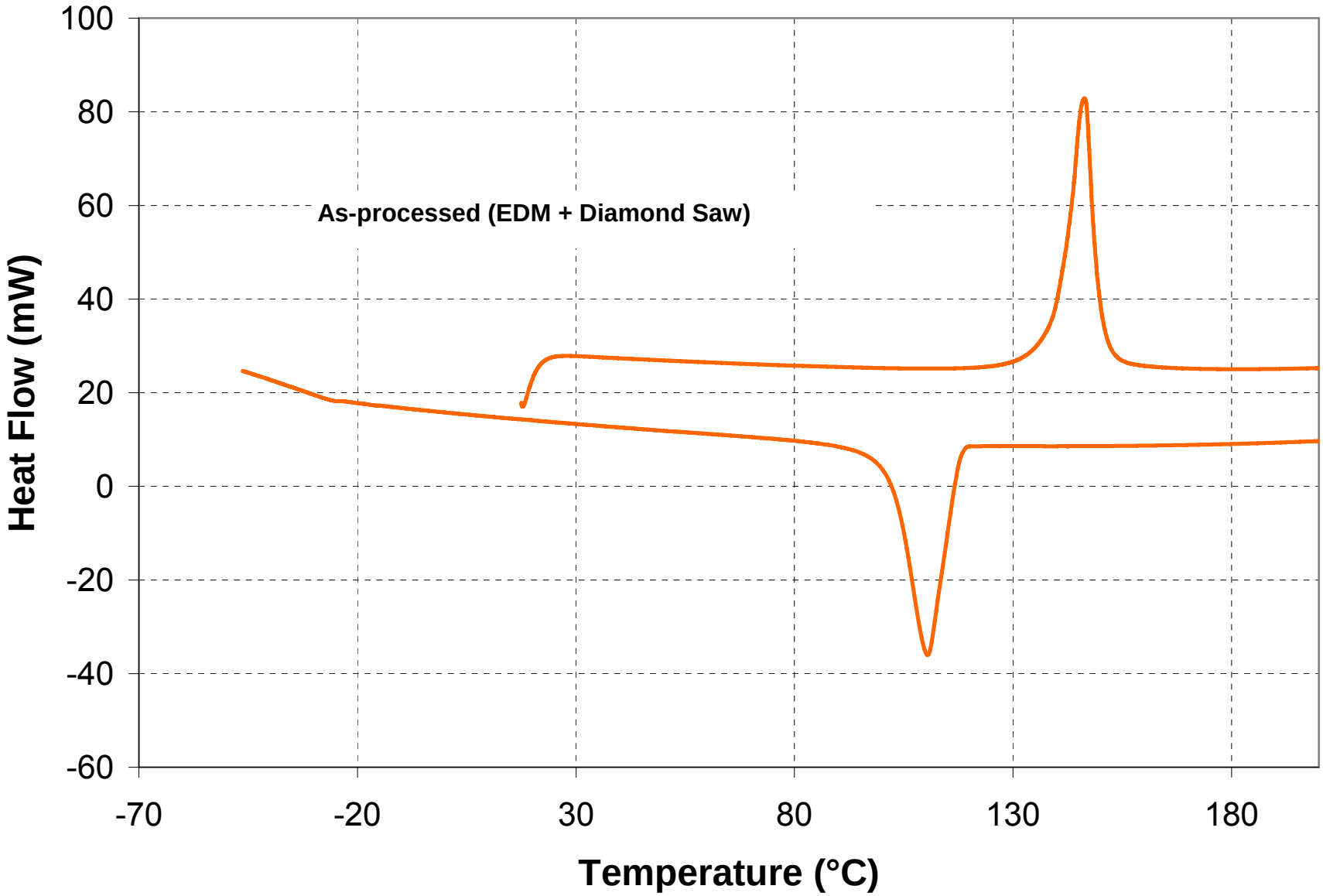


Ex65 – 600°C, 24 hour (vacuum) heat treated

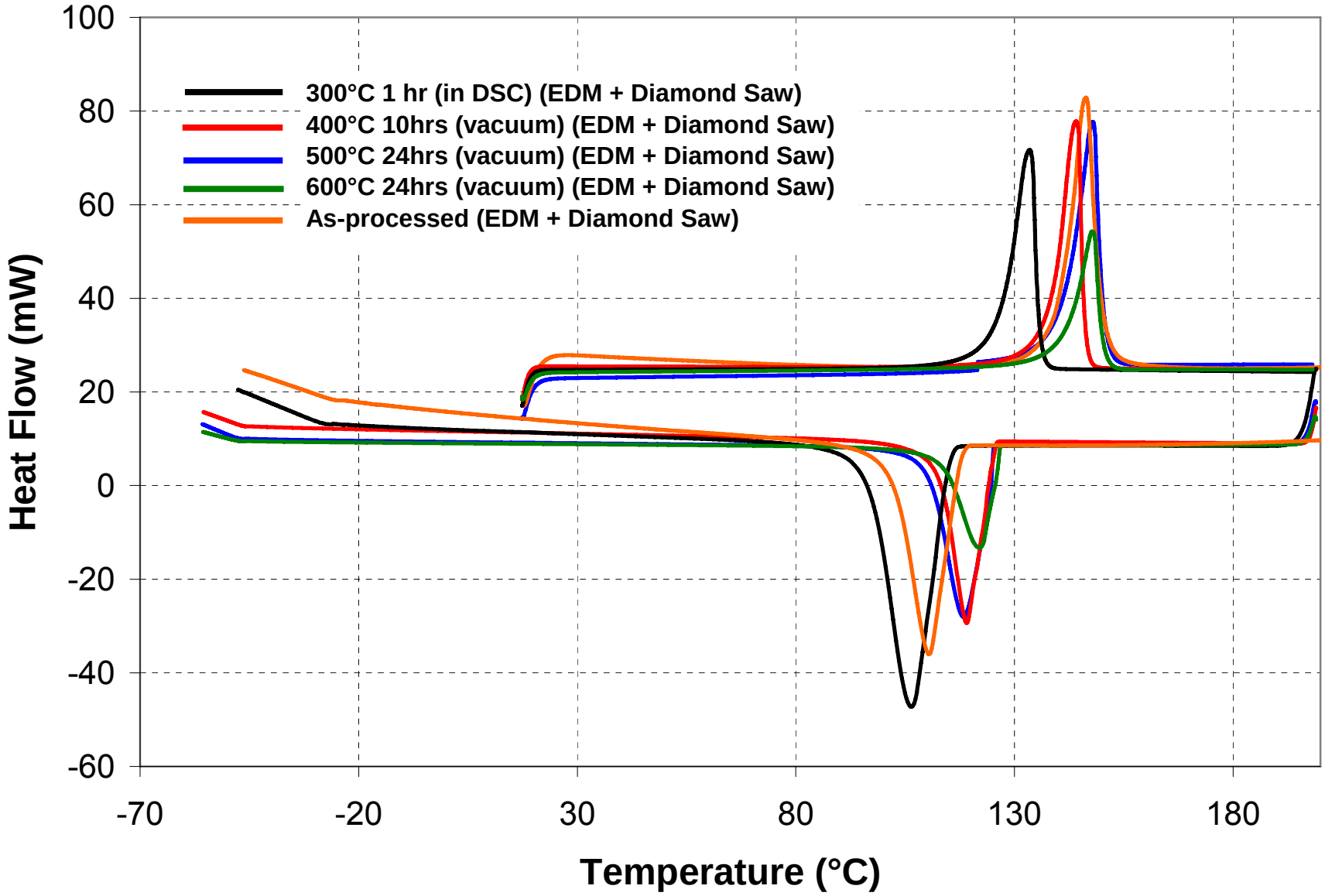


Ex64 – DSC Results

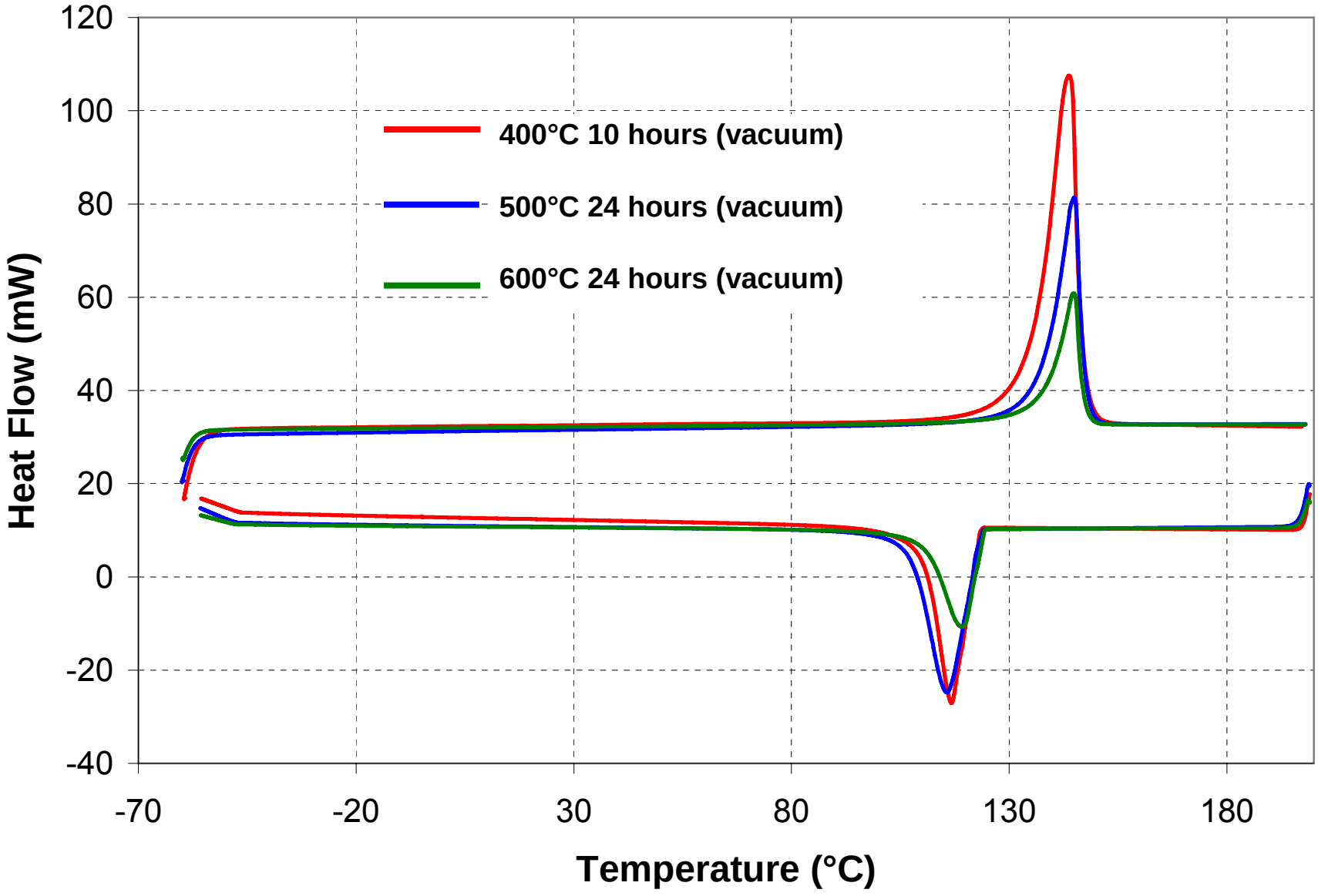
DSC Results - Ext 64, As-processed material



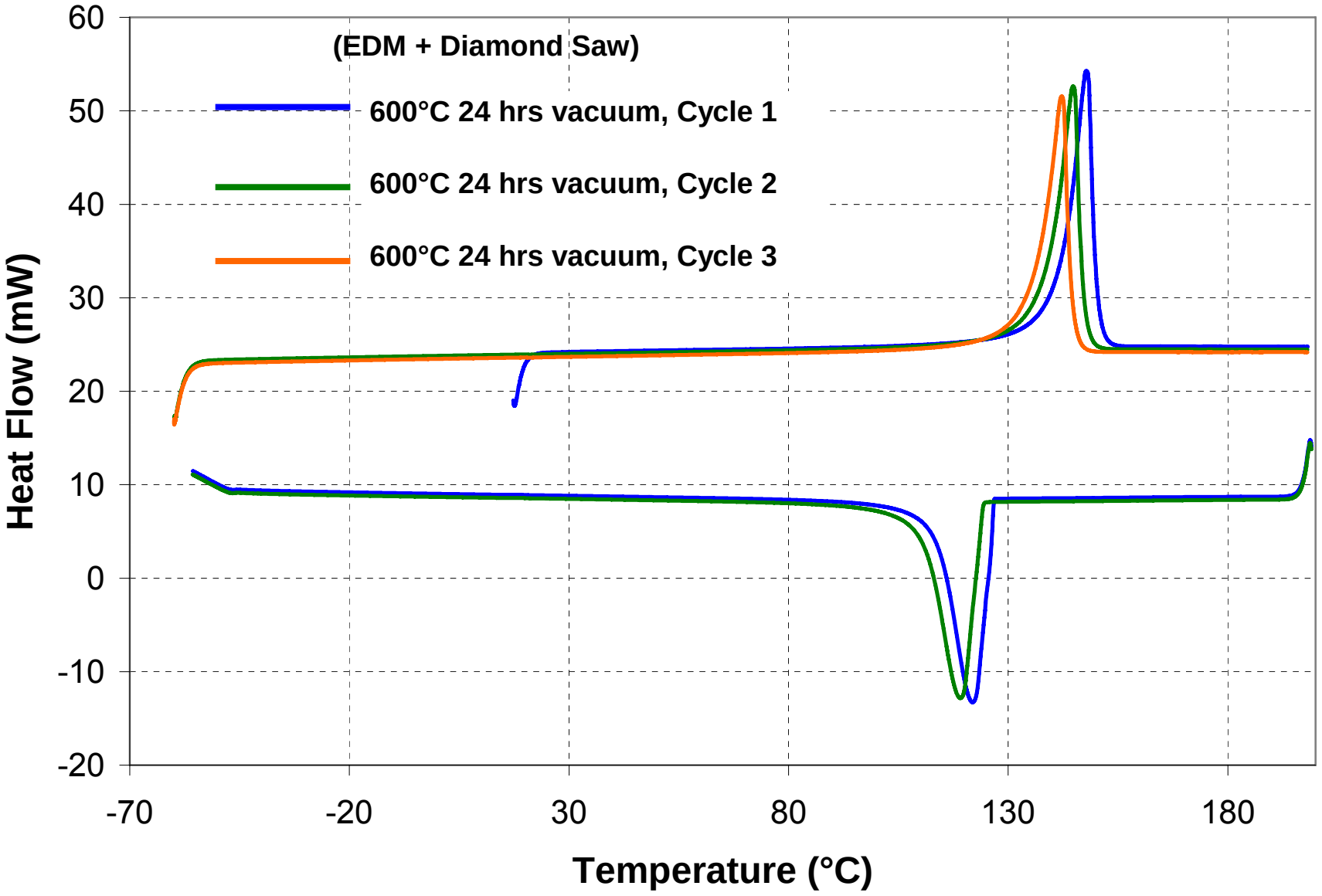
Ex64 - Consolidated DSC data, 1st cycle



Ex64 - Consolidated DSC data, 2nd cycle



Ex64 - Comparison of 1st 2nd and 3rd cycle data



Ex64 – 300°C, 1hour heat treated in DSC