



Alloys from Nb-W-Cr System for High Temperature Applications

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Acknowledgements

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In this presentation we are reporting the progress on the results of our study on the oxidation of alloys from Nb-W-Cr system



Overview

- # Introduction
 - # Experimental details
 - # Results & Discussion
 - # Conclusions
-



Physical Properties of Nb, W, Cr, and Ni

	<u>Nb</u>	<u>W</u>	<u>Cr</u>	<u>Ni</u>
Melting Point (°C)	2,469	3,422	1,863	1,453
Crystal Structure*	bcc	bcc	bcc	fcc
Density (gm/cm³)	8.57	19.3	7.9	8.9

*bcc = body-centered cubic; fcc = face-centered cubic



Experimental details

- ✚ The materials were fabricated by the Ames Laboratory of Iowa State University
 - ✚ Oxidation experiments from 700 to 1400°C in air were conducted with the use of a computerized temperature control tube furnace
 - ✚ Weight gain method was used for obtaining the oxidation curves
 - ✚ Optical Microscopy
 - ✚ X-Ray Diffractometer : XDS 2000
 - ✚ FESEM : Hitachi 8700
 - ✚ XPS & AES: PHI 560 ESCA/ SAM UHV system
-



Results & Discussion

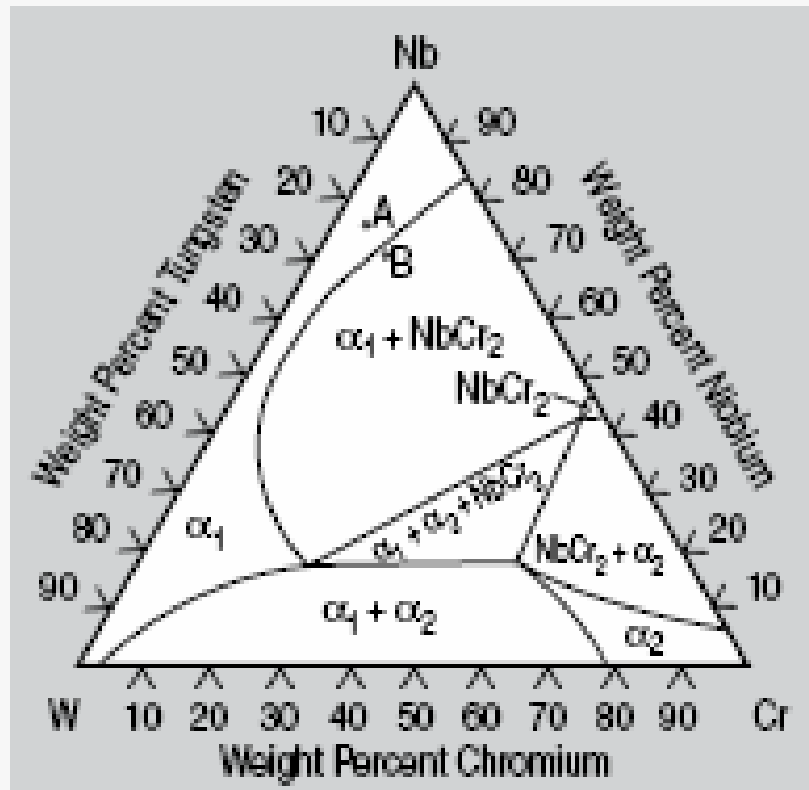


As Cast Structures





Isotherm of Nb-W-Cr system at 1000°C

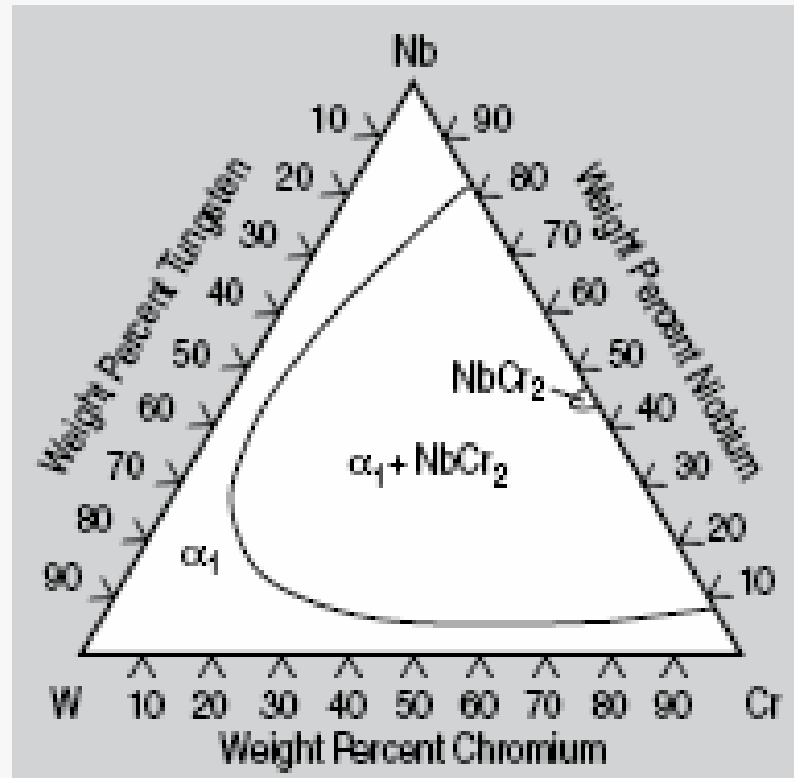


$\alpha_1 - cI2$
 $\alpha_2 - cI2$
 $NbCr_2 - hp12$ or $cF24$



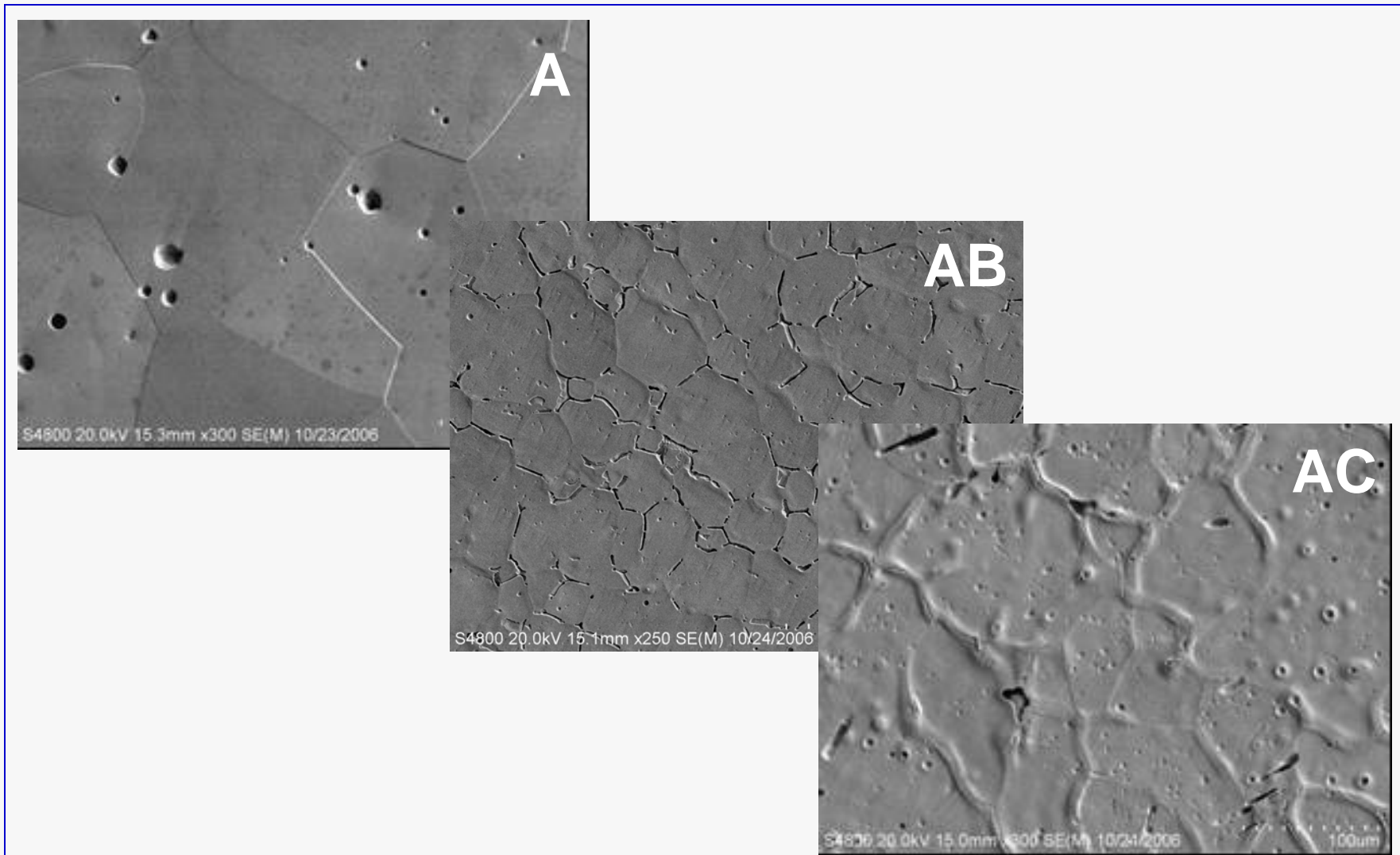


Isotherm of Nb-W-Cr system at 1500°C

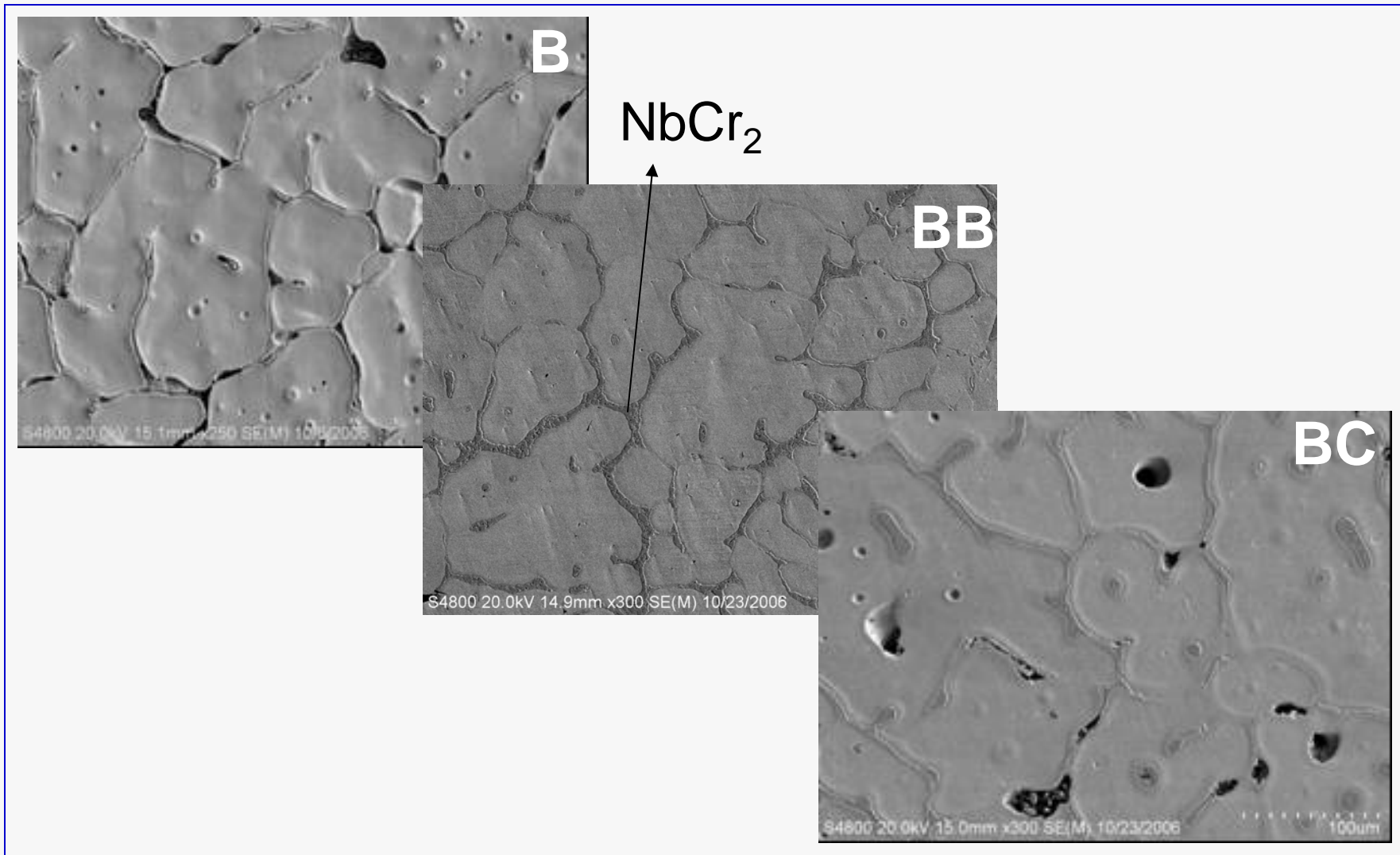




As-Cast SEM Microstructures

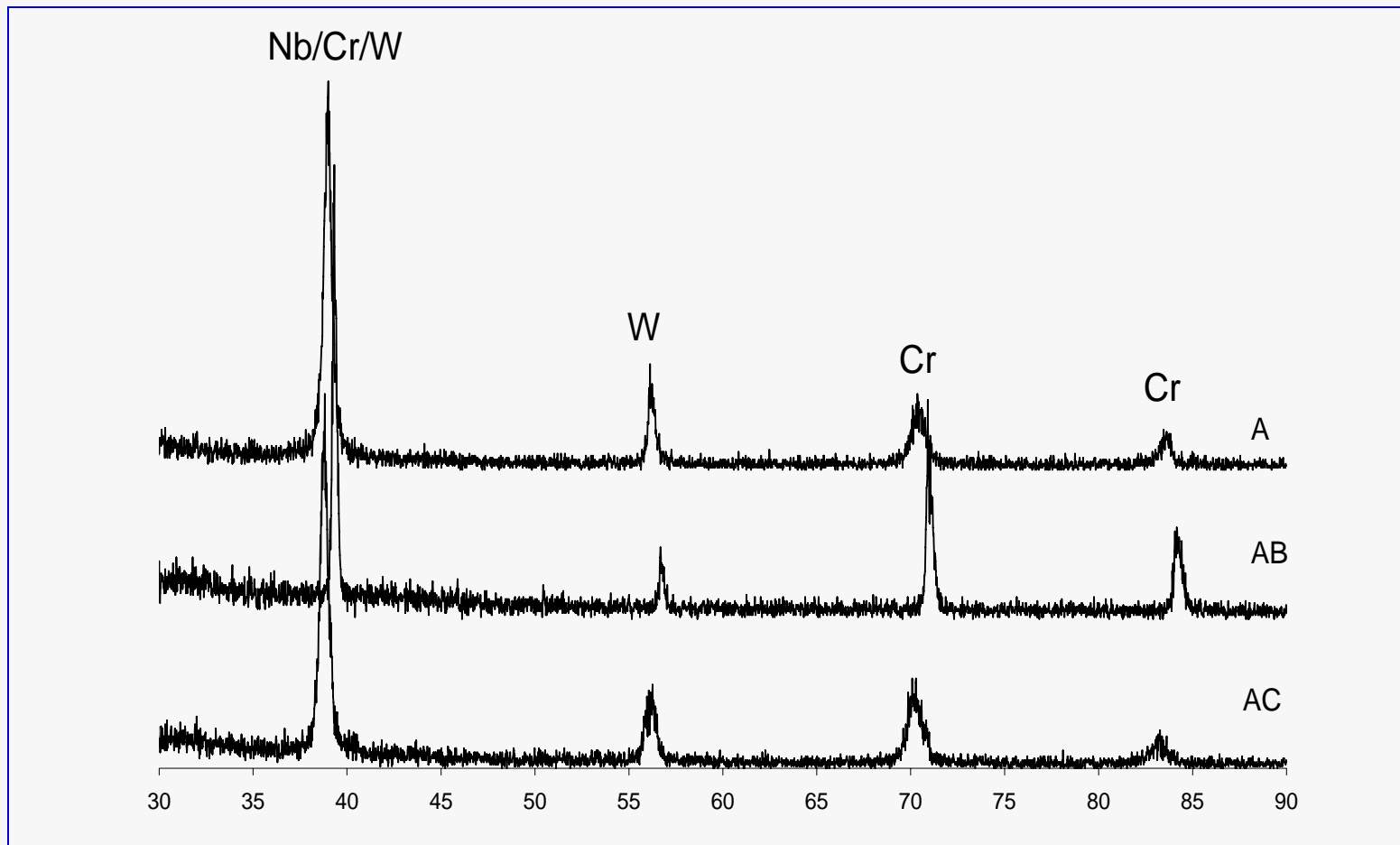


As-Cast SEM Microstructures



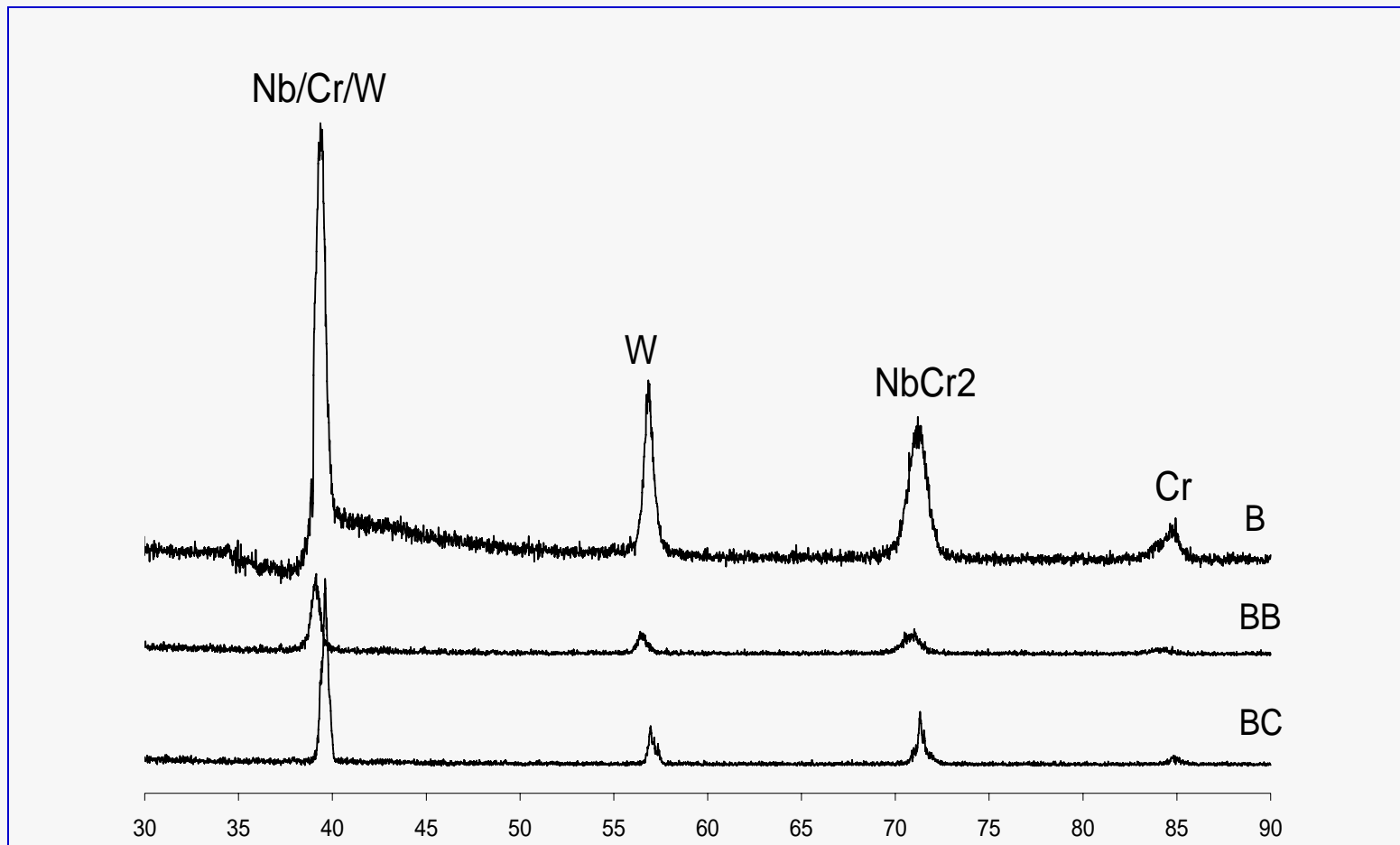


As-Cast XRD Patterns A alloys





As-Cast XRD Patterns B alloys



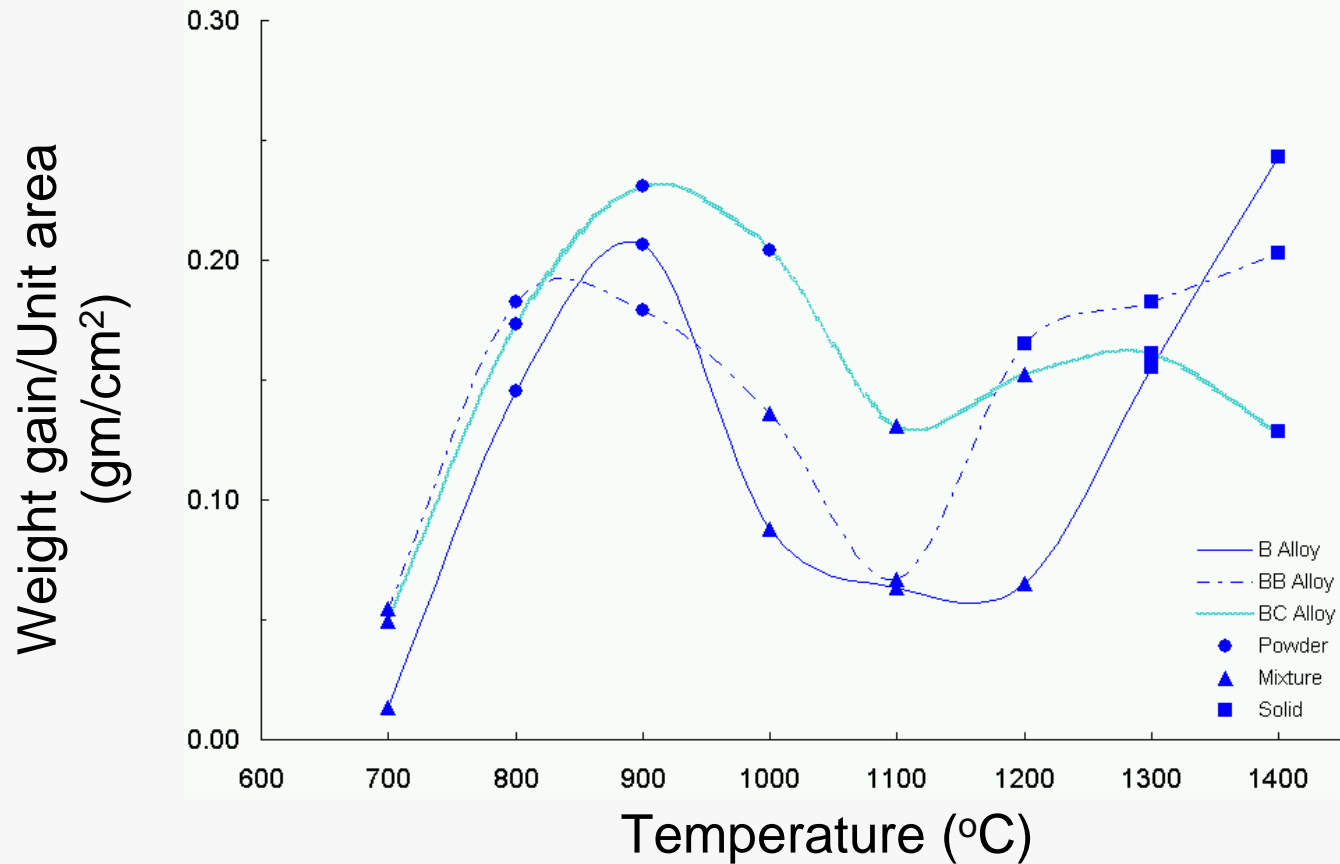


Short term oxidation



Oxidation curves

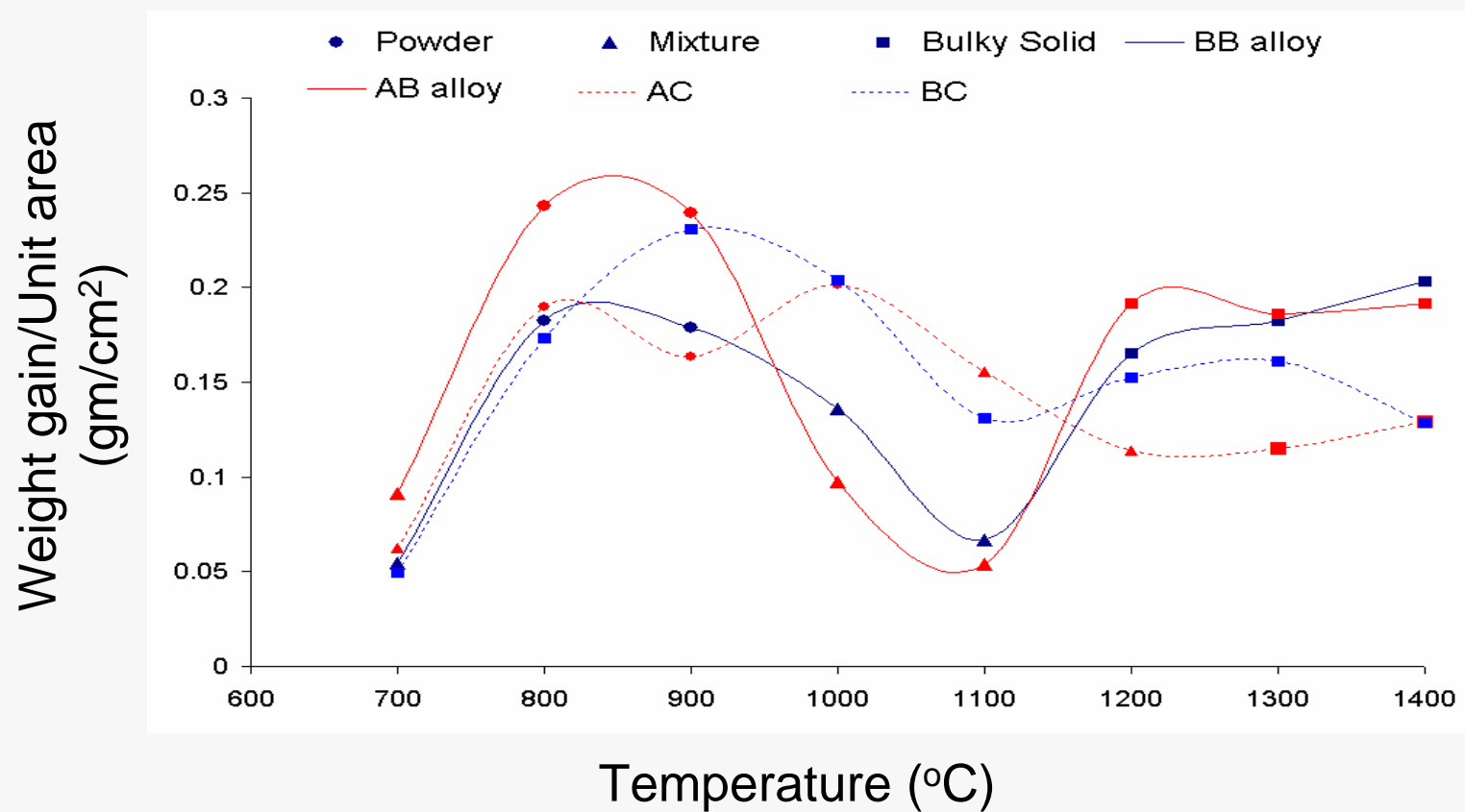
Oxidation curves of the alloys B, BB, BC oxidized at 700 -1400°C





Oxidation curves

Oxidation curves of the alloys AB, AC, BB, BC oxidized at 700 -1400°C



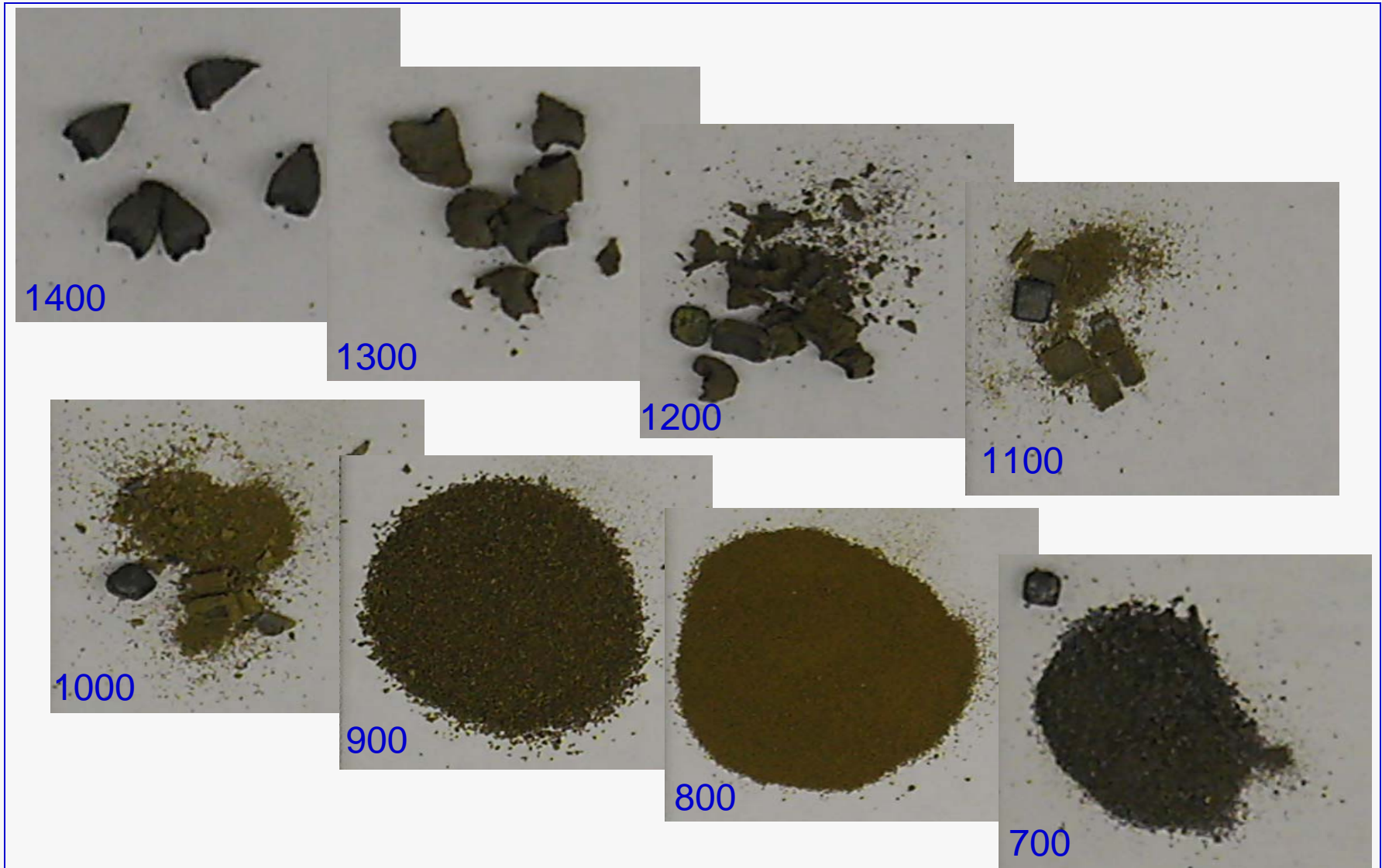


Faceting after STO of alloy B

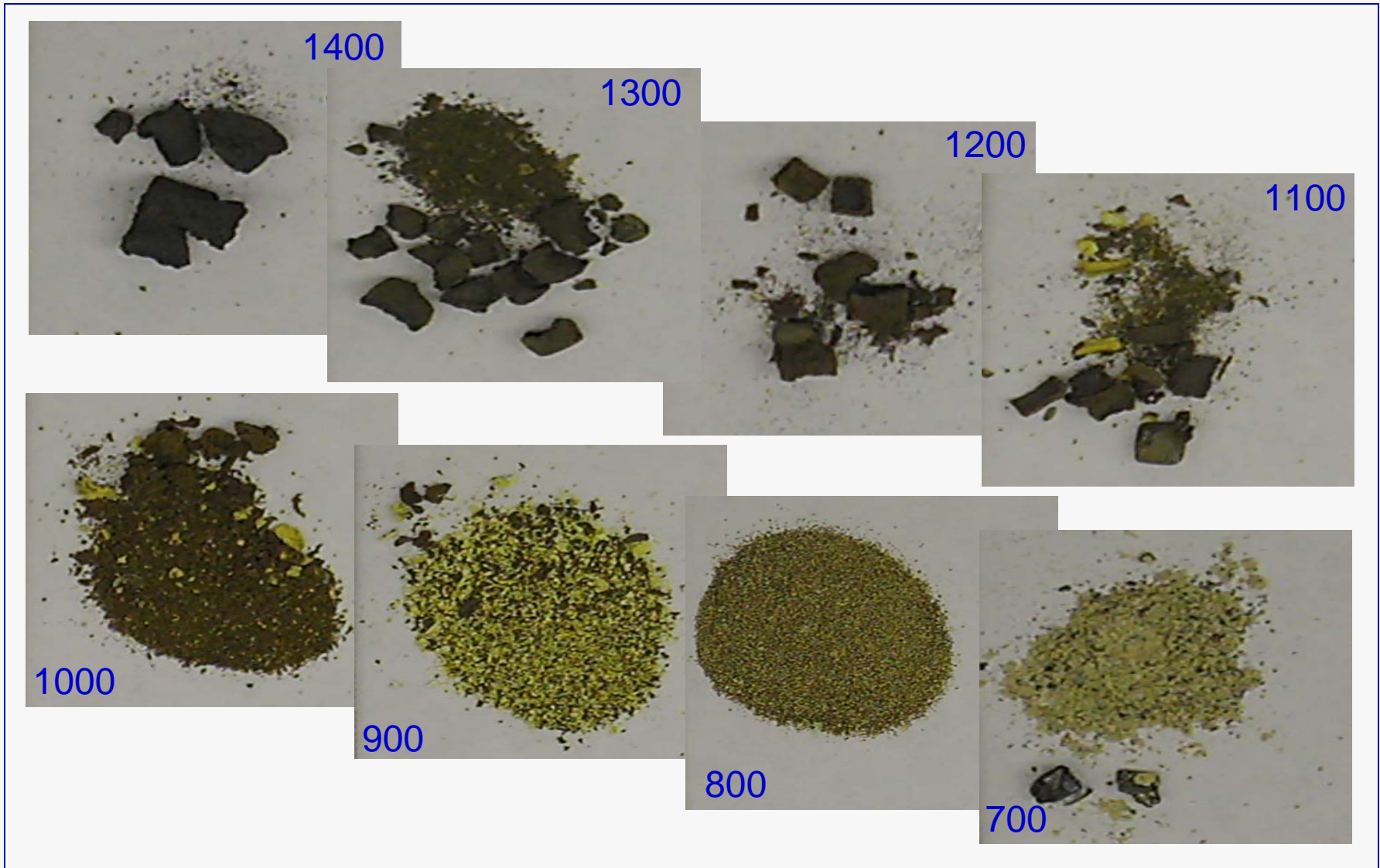


**1300°C after 24 hours of
oxidation**

Products of oxidation of alloy AB

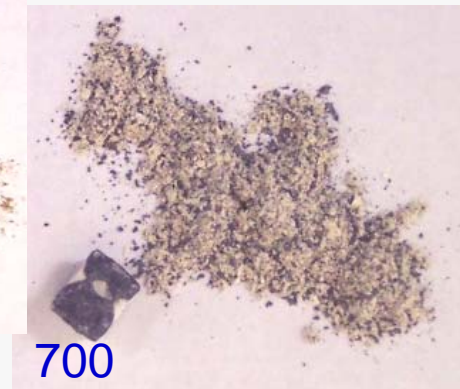
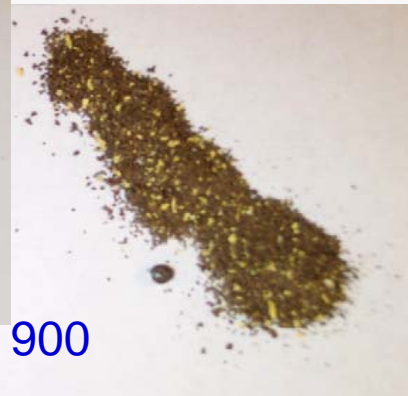
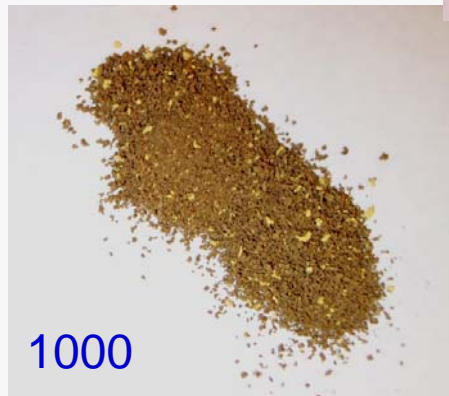
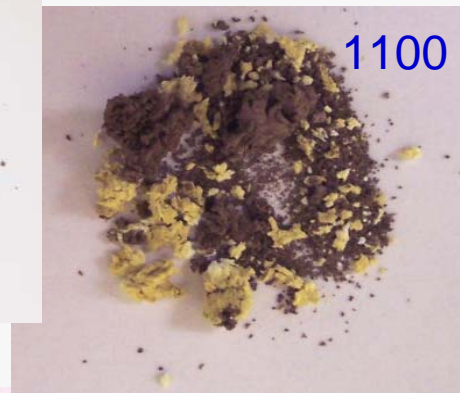
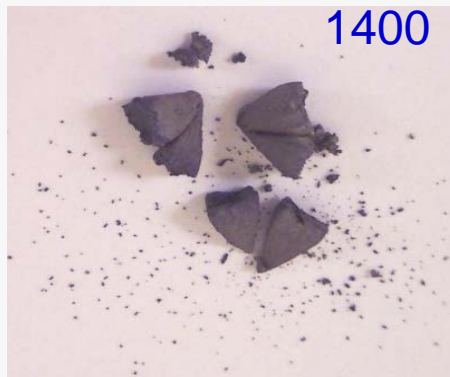


Products of oxidation of alloy BB

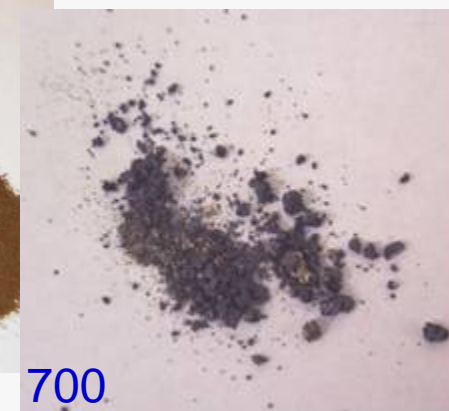
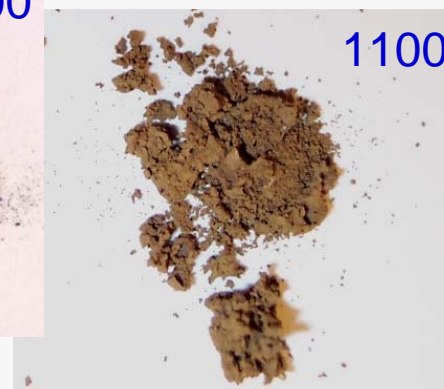
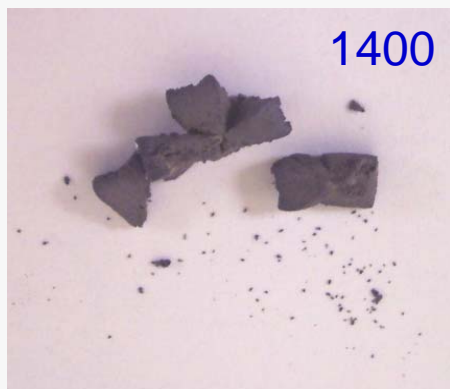




Products of oxidation of alloy AC



Products of oxidation of alloy BC



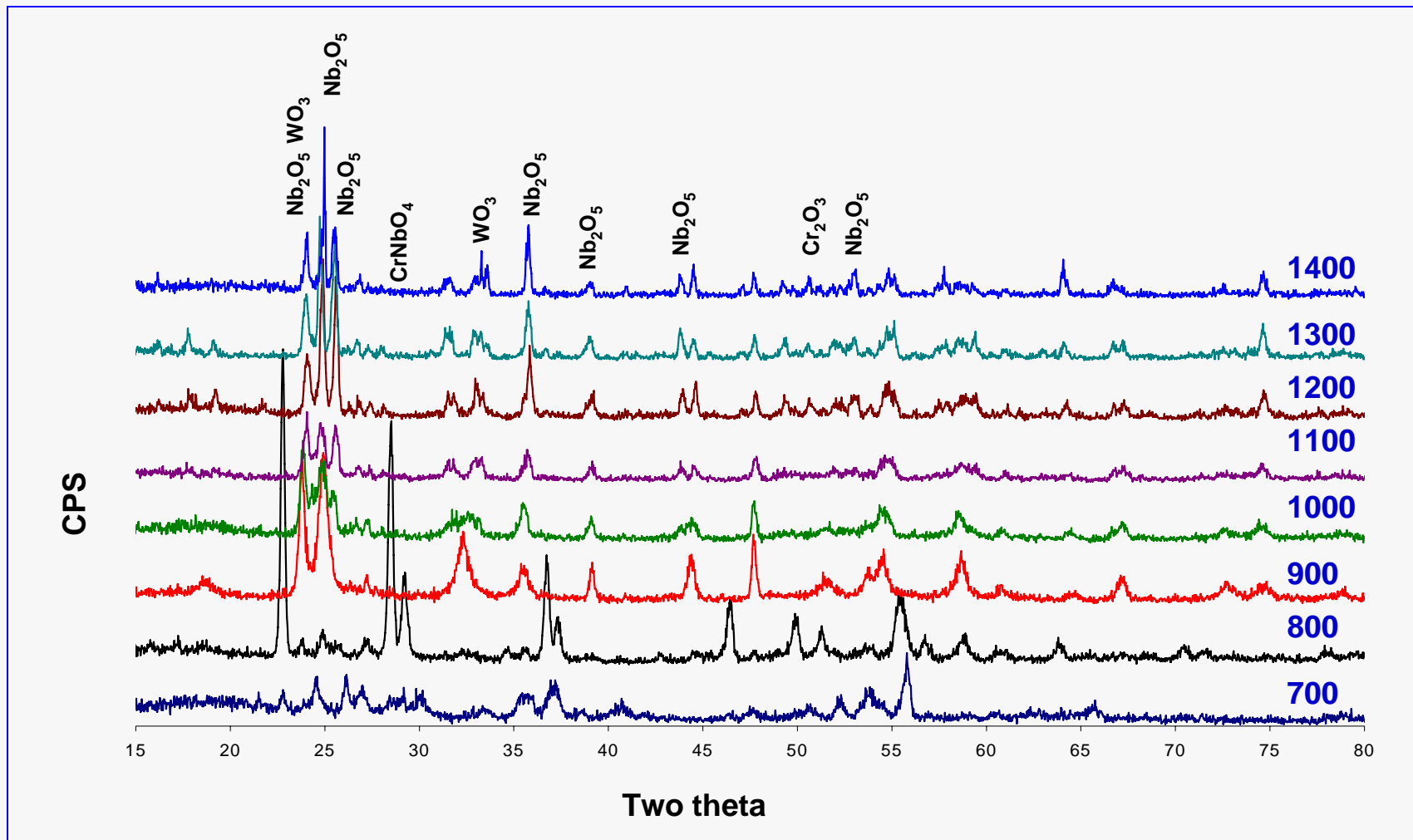


Properties of Select Metal Oxides

Oxide	Color	MP (°C)
NbO	Black	1,937
NbO ₂	Blue-Black, White	1,900
Nb ₂ O ₅	White	1,512
WO ₃	Yellow	1,473
WO ₂	Brown	1,500
Cr ₂ O ₃	Green	2,450

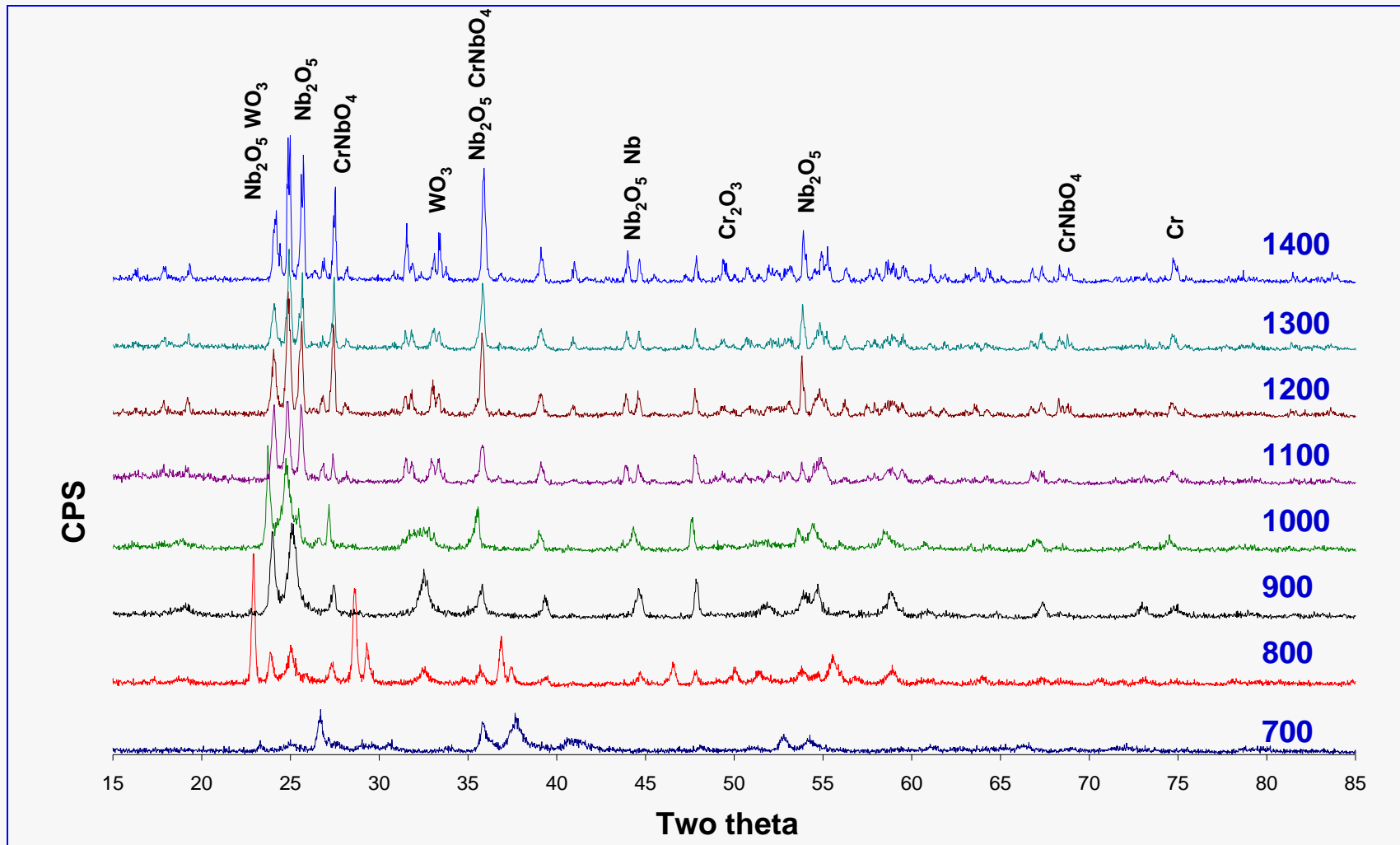


XRD results on STO powders A



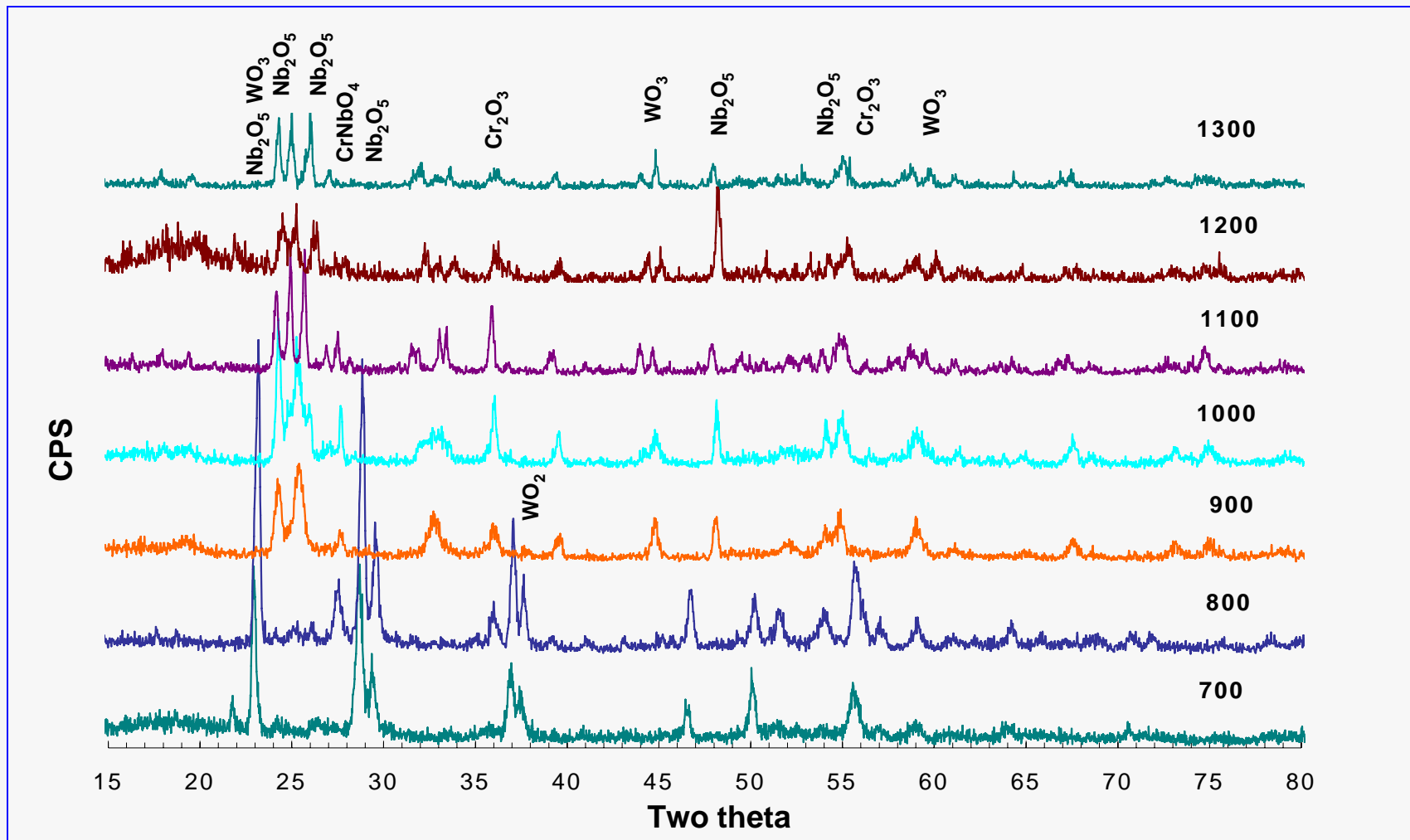


XRD results on STO powders AB



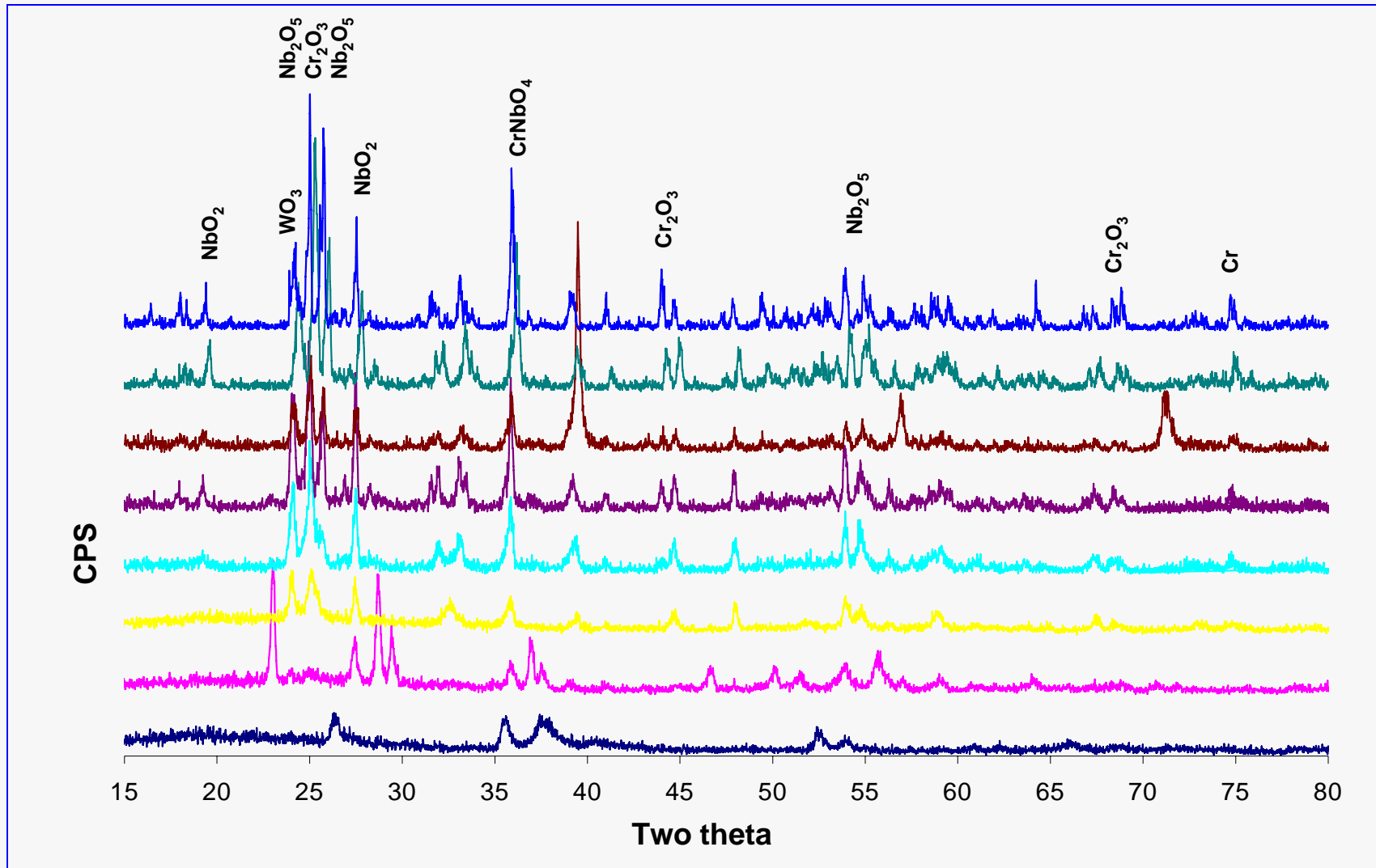


XRD results on STO powders AC



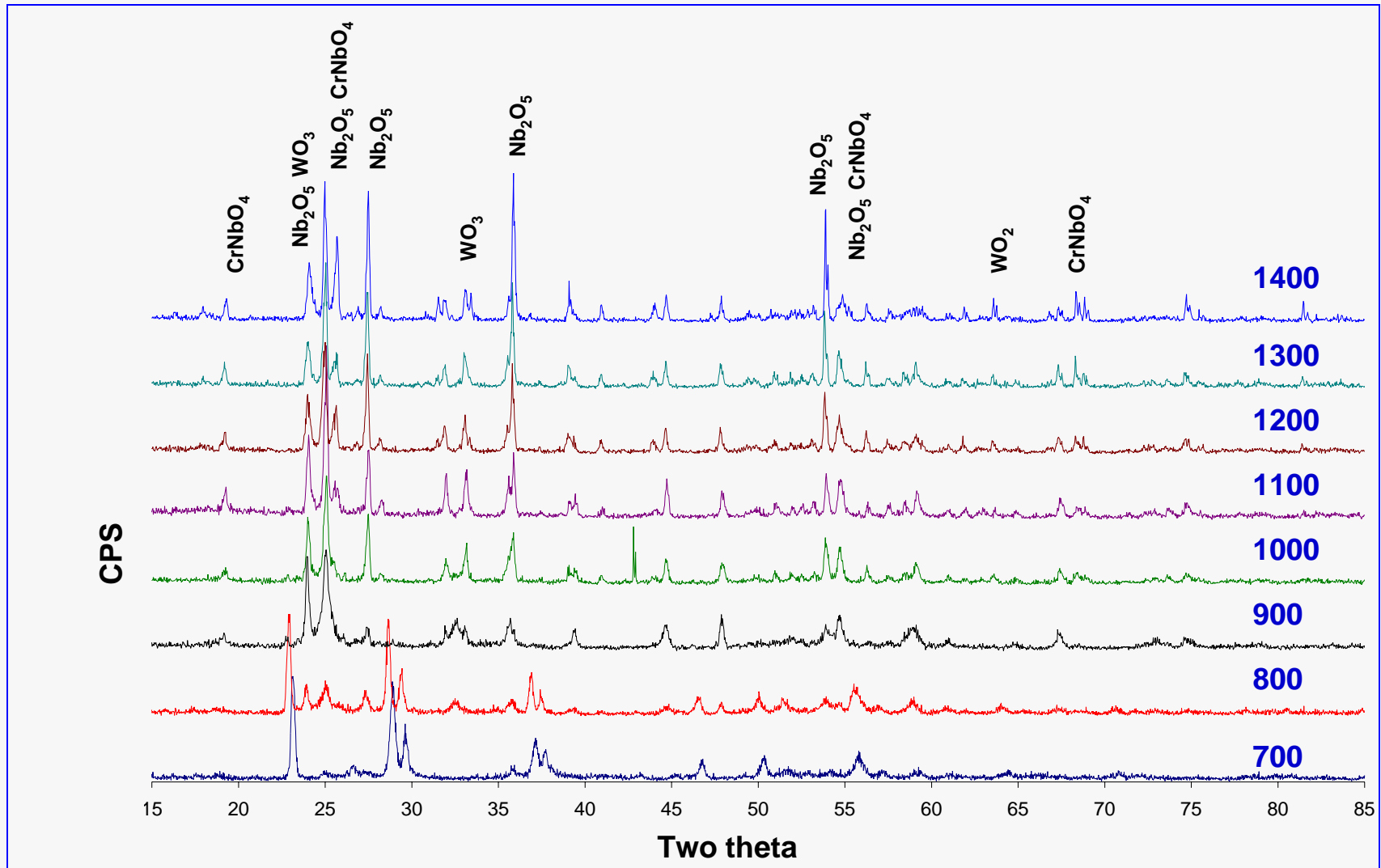


XRD results on STO powders B



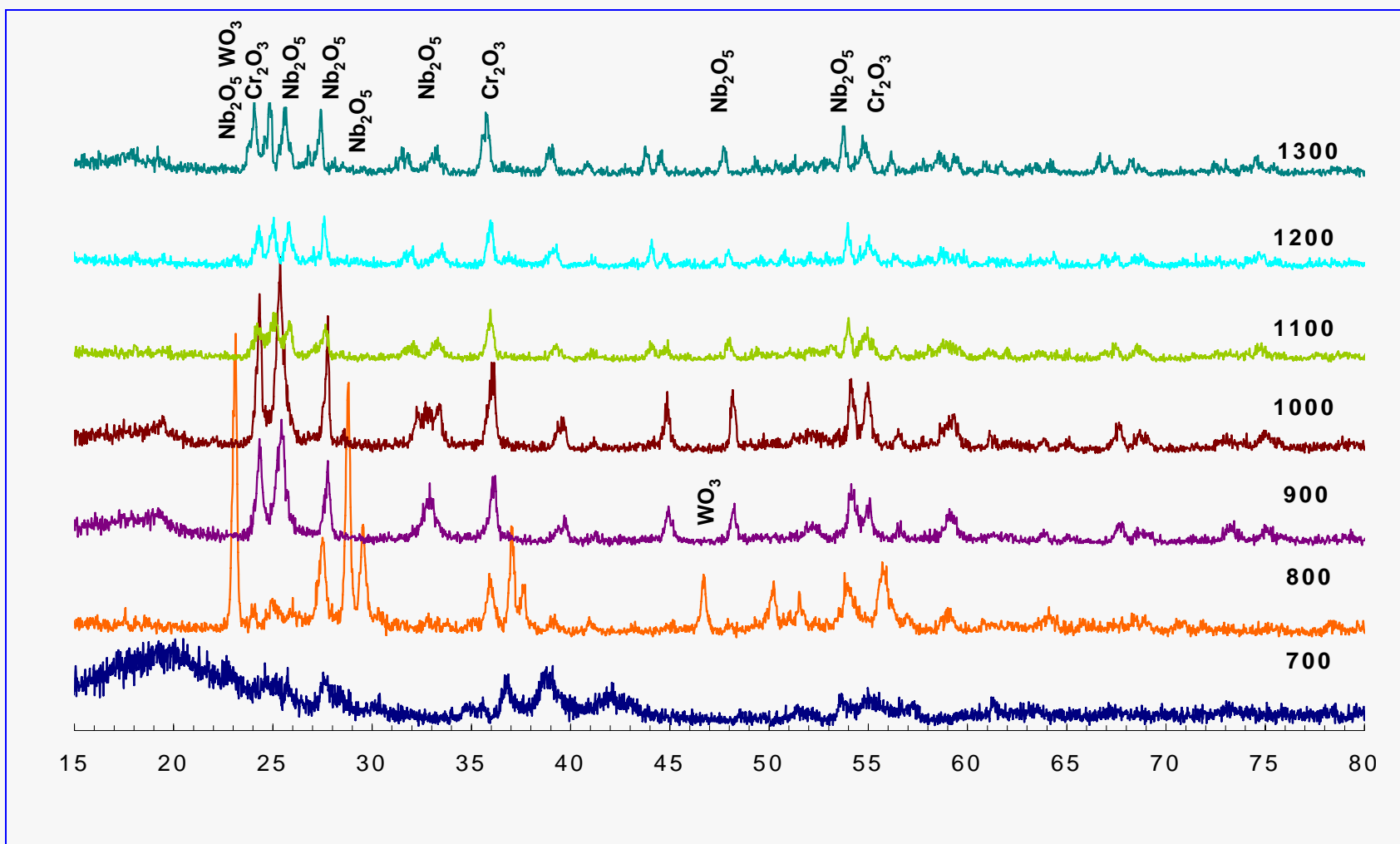


XRD results on STO powders BB

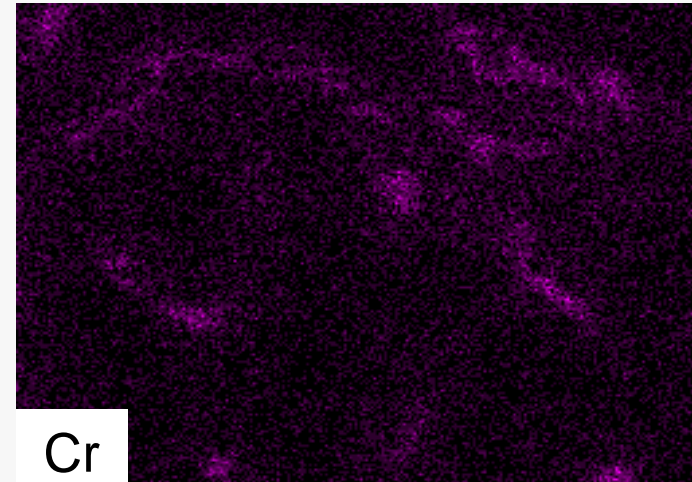
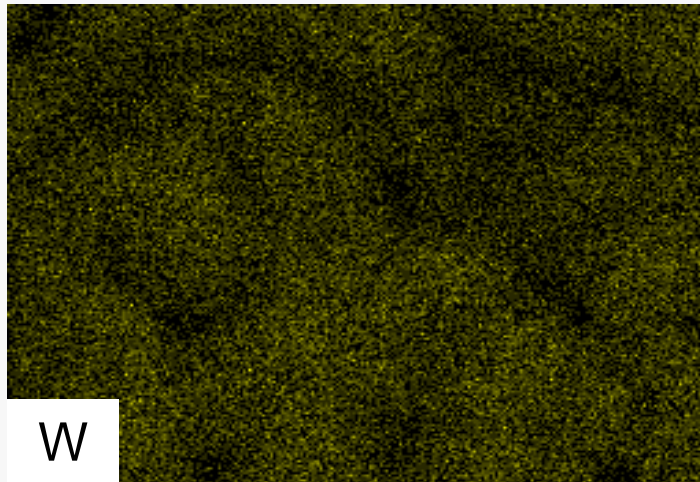
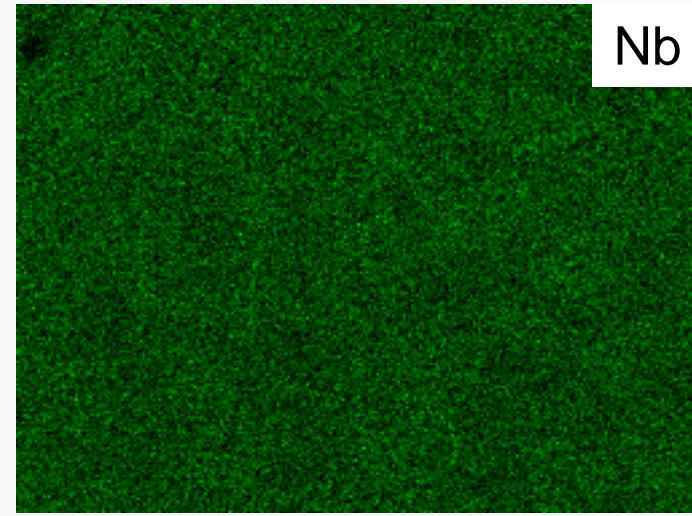
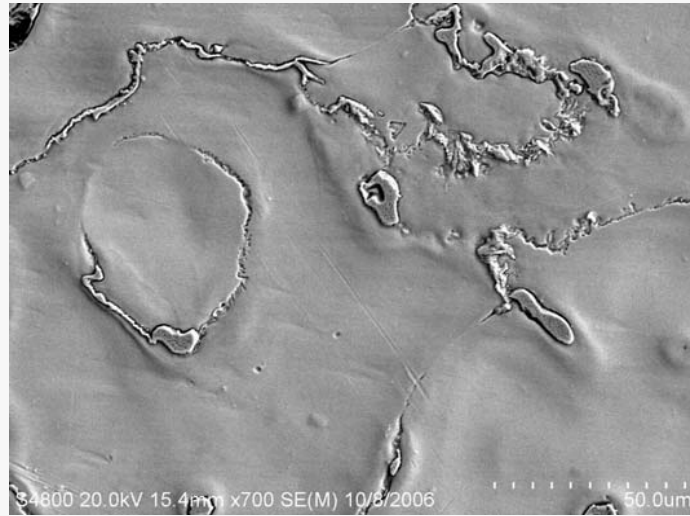




XRD results on STO powders BC

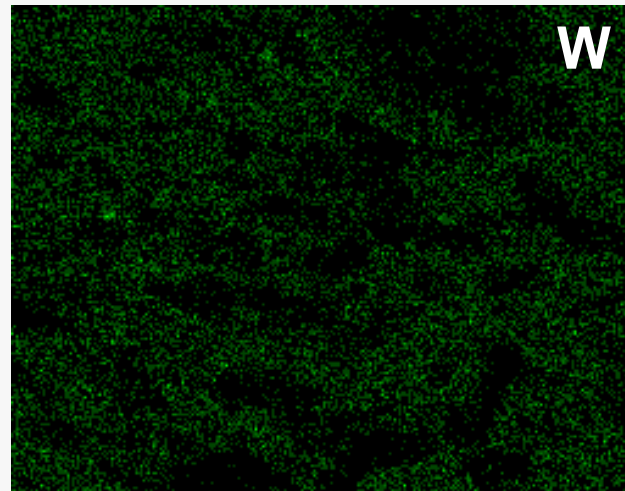
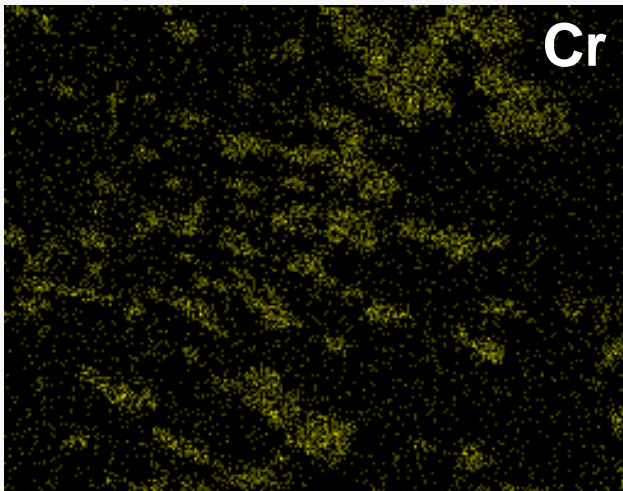
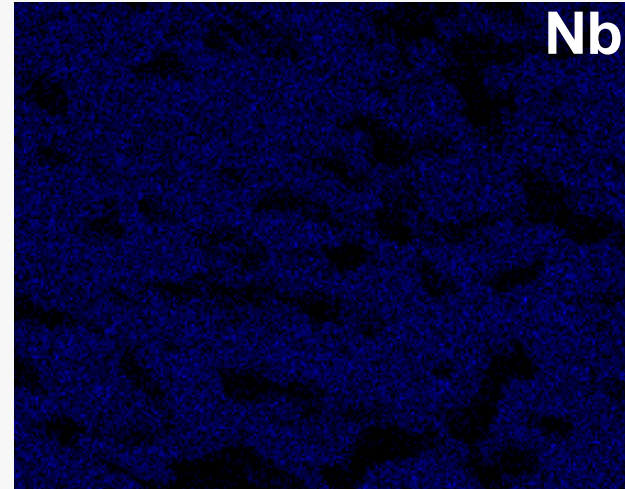
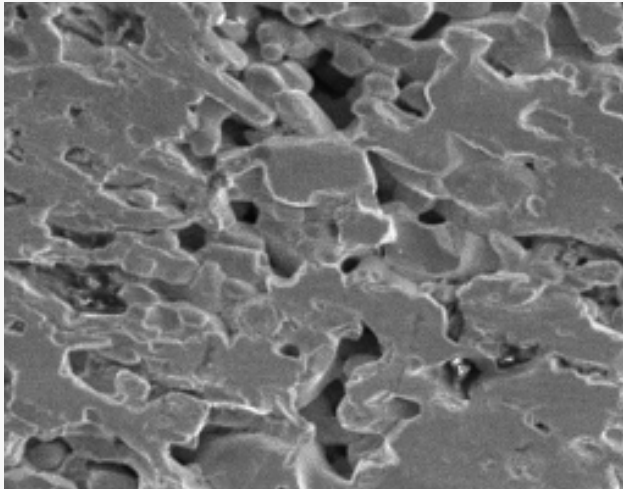


X-Ray Mapping on SEM : alloy B

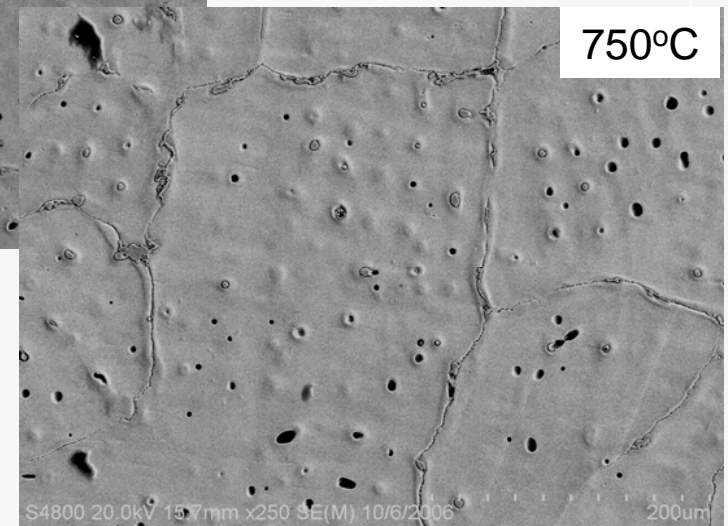
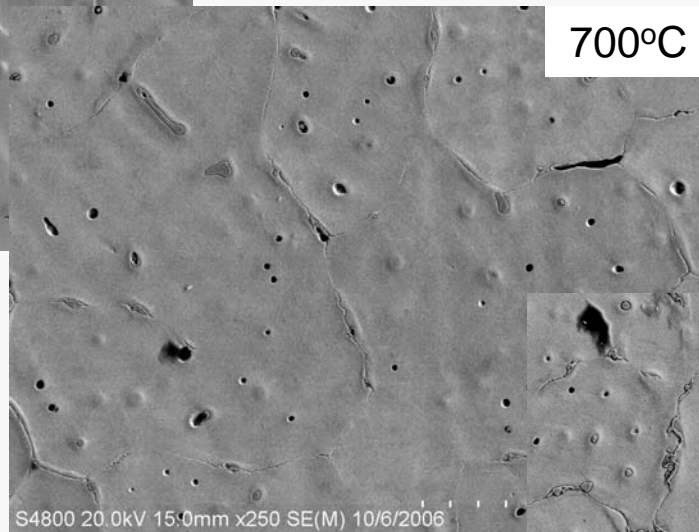
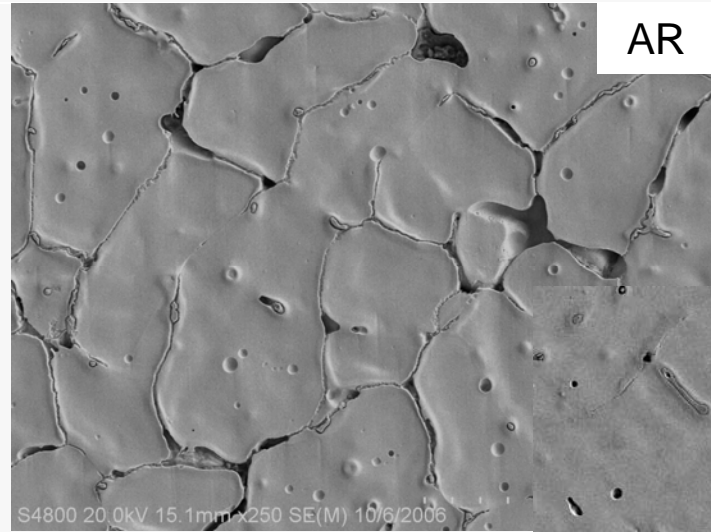


oxidation in air at 750°C after 24hrs

X-ray mapping on AB 1400

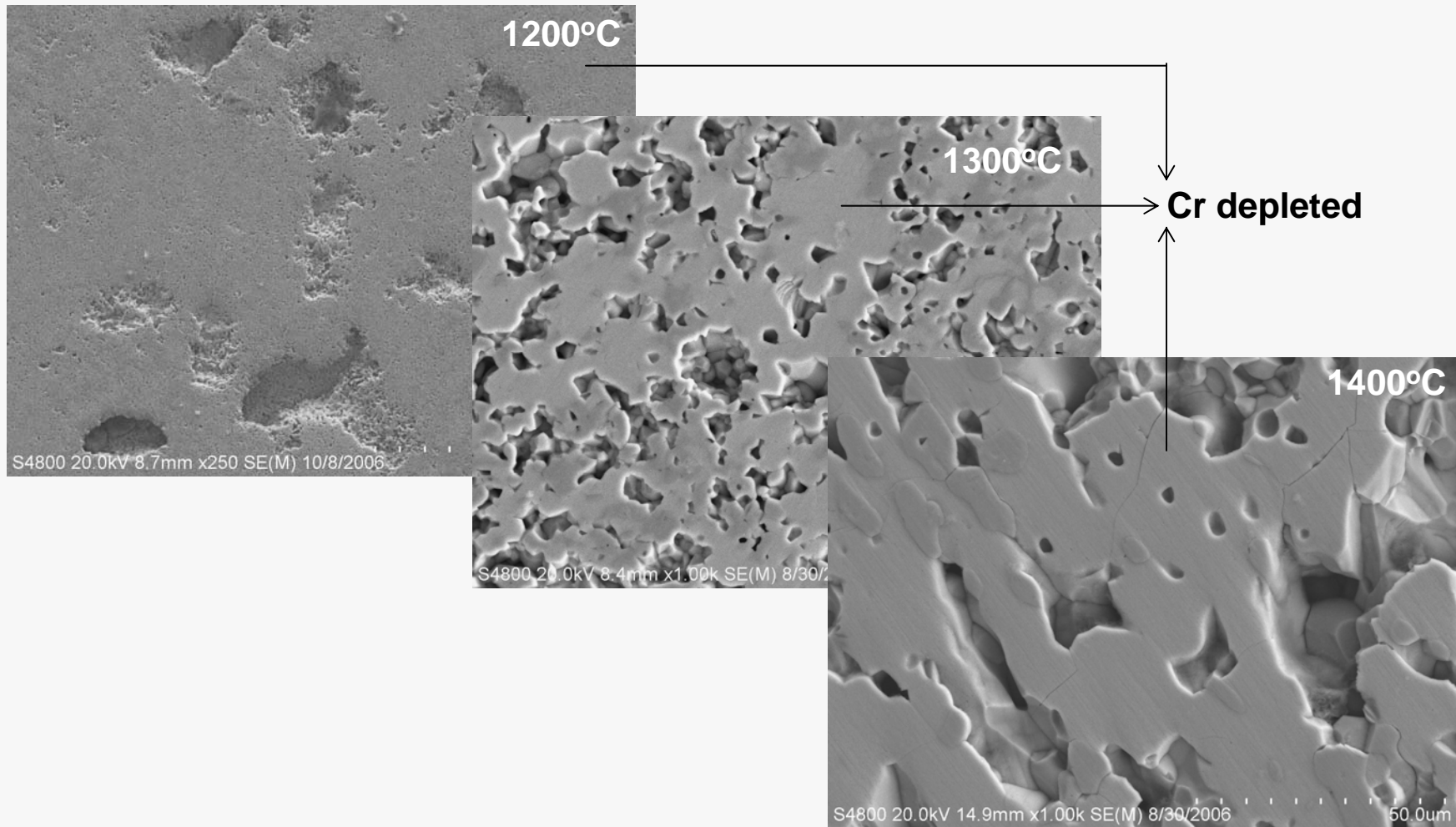


SEM on alloy B



SEM on polished surface of B alloy

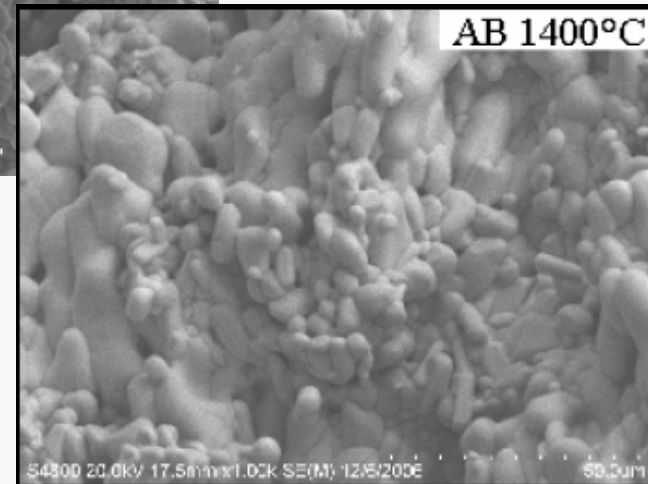
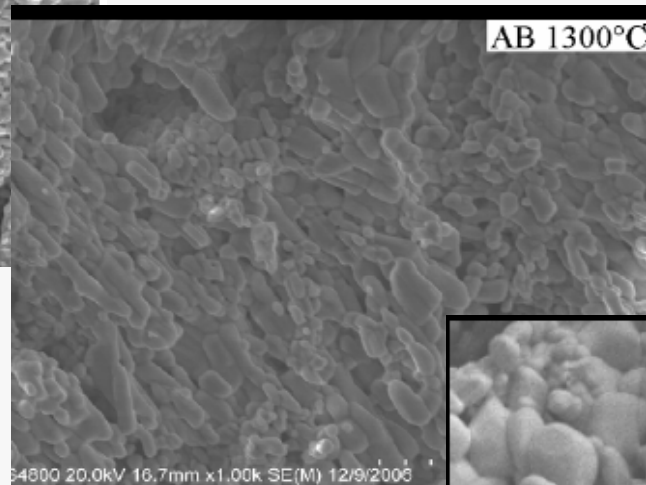
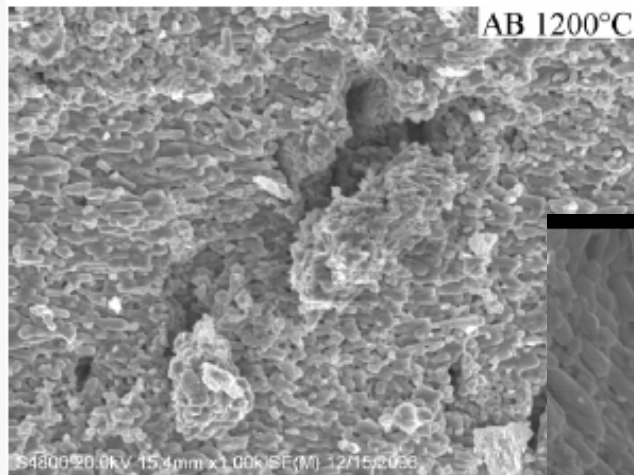
Oxidized in air at 1200, 1300, and 1400°C for 24hrs 1000x





SEM on unpolished surface of AB alloy

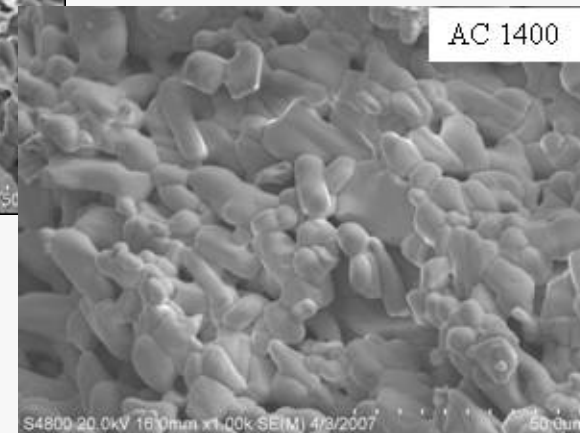
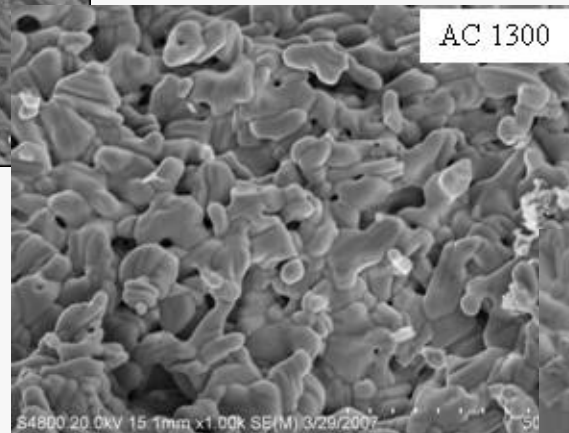
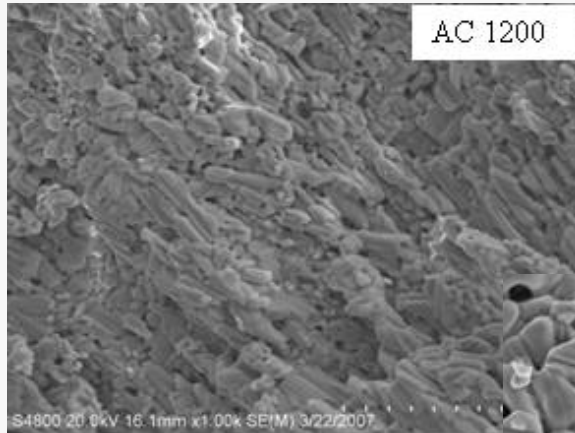
Oxidation in air at 1200, 1300 and 1400°C for 24hrs 1000x



SEM on unpolished surface of AC alloy



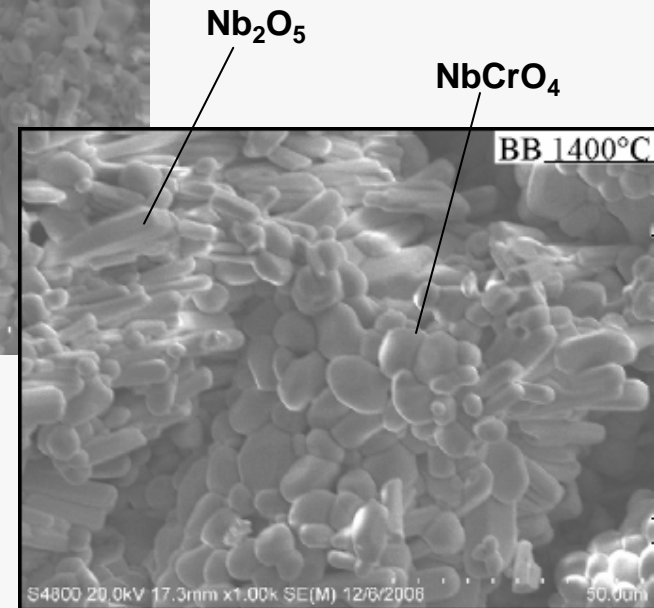
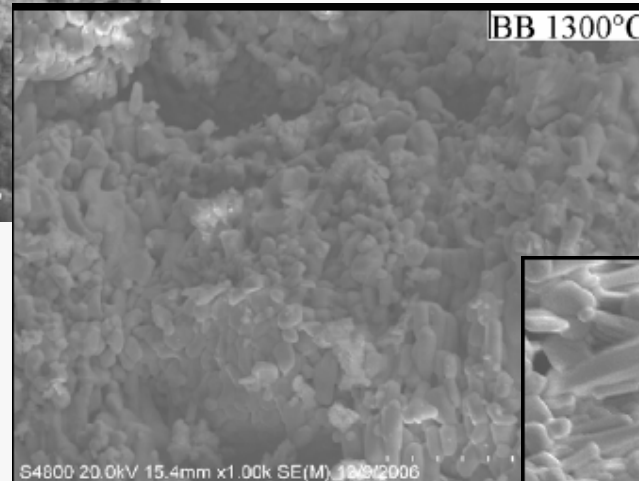
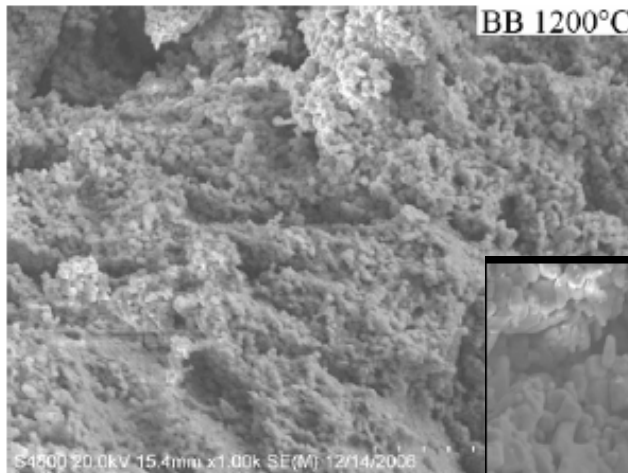
Oxidation in air at 1200, 1300, and 1400°C for 24hrs 1000x





SEM on unpolished surface of BB alloy

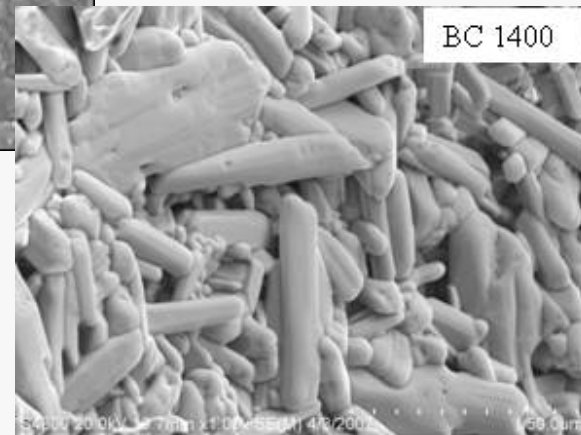
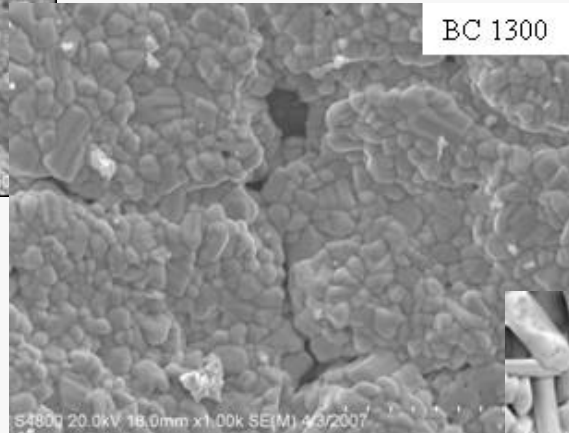
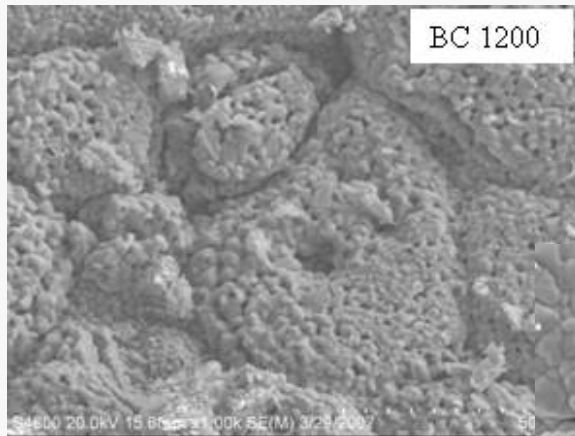
Oxidation in air at 1200, 1300, and 1400°C for 24hrs 1000x



SEM on unpolished surface of BC alloy



Oxidation in air at 1200, 1300, and 1400°C for 24hrs 1000x



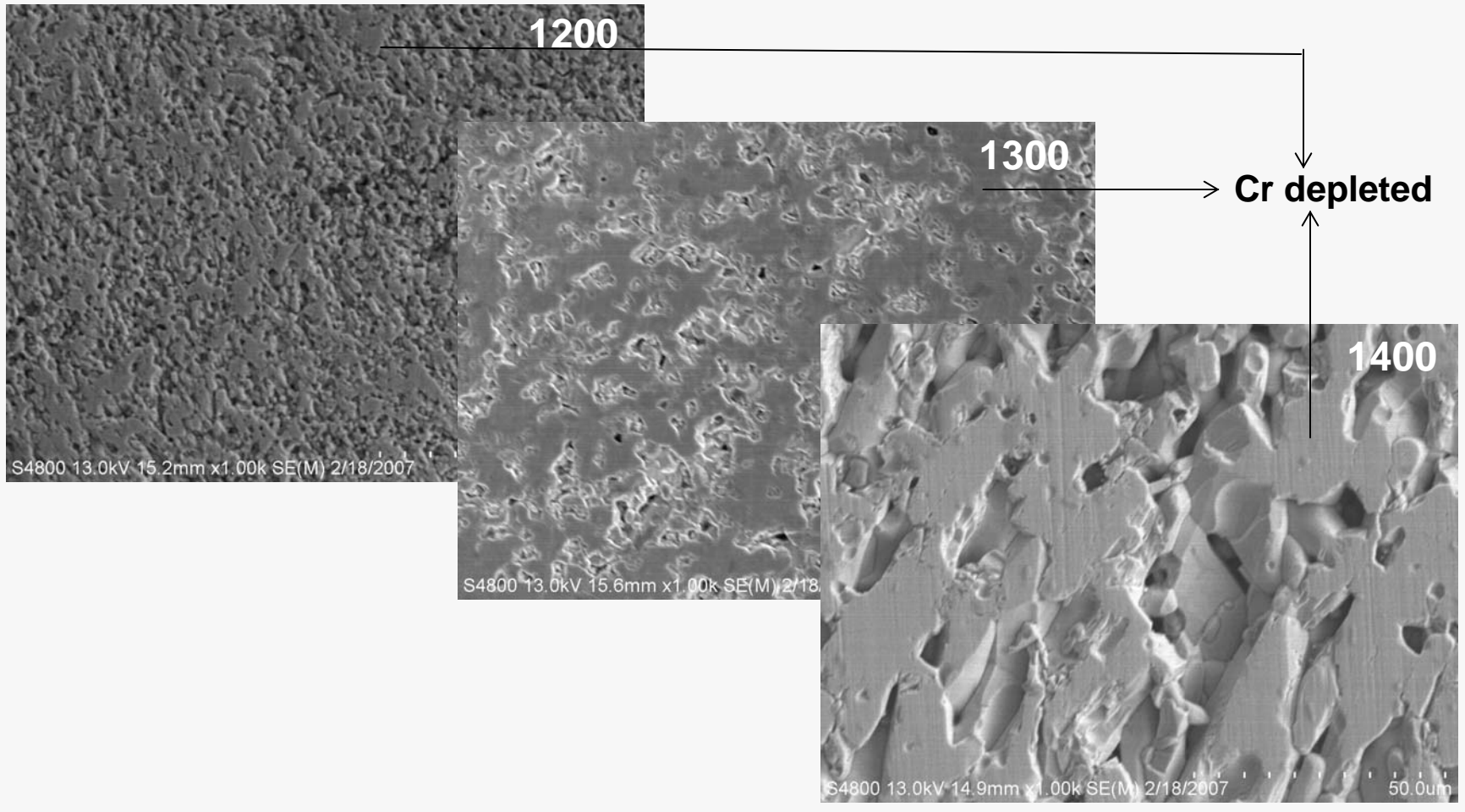


EDS results for BB alloy

BB 24 hrs	T (°C)	Oxide	O at. %	Nb at. %	Cr at. %	W at. %
Round Particles	1400	CrNbO ₄	69.84	16.19	13.43	0.54
	1300	CrNbO ₄	65.94	18.03	15.06	1.42
Cylindrical Particles	1400	Nb ₂ O ₅	67.61	26.6	1.82	3.97
	1300	Nb ₂ O ₅	72.48	23.73	1.01	2.77

SEM on polished surface of AB alloy

Oxidized in air at 1200, 1300, and 1400°C for 24hrs 1000x





EDS analysis on BB and AB alloys

Oxidized in air at 1200, 1300, and 1400°C for 24hrs

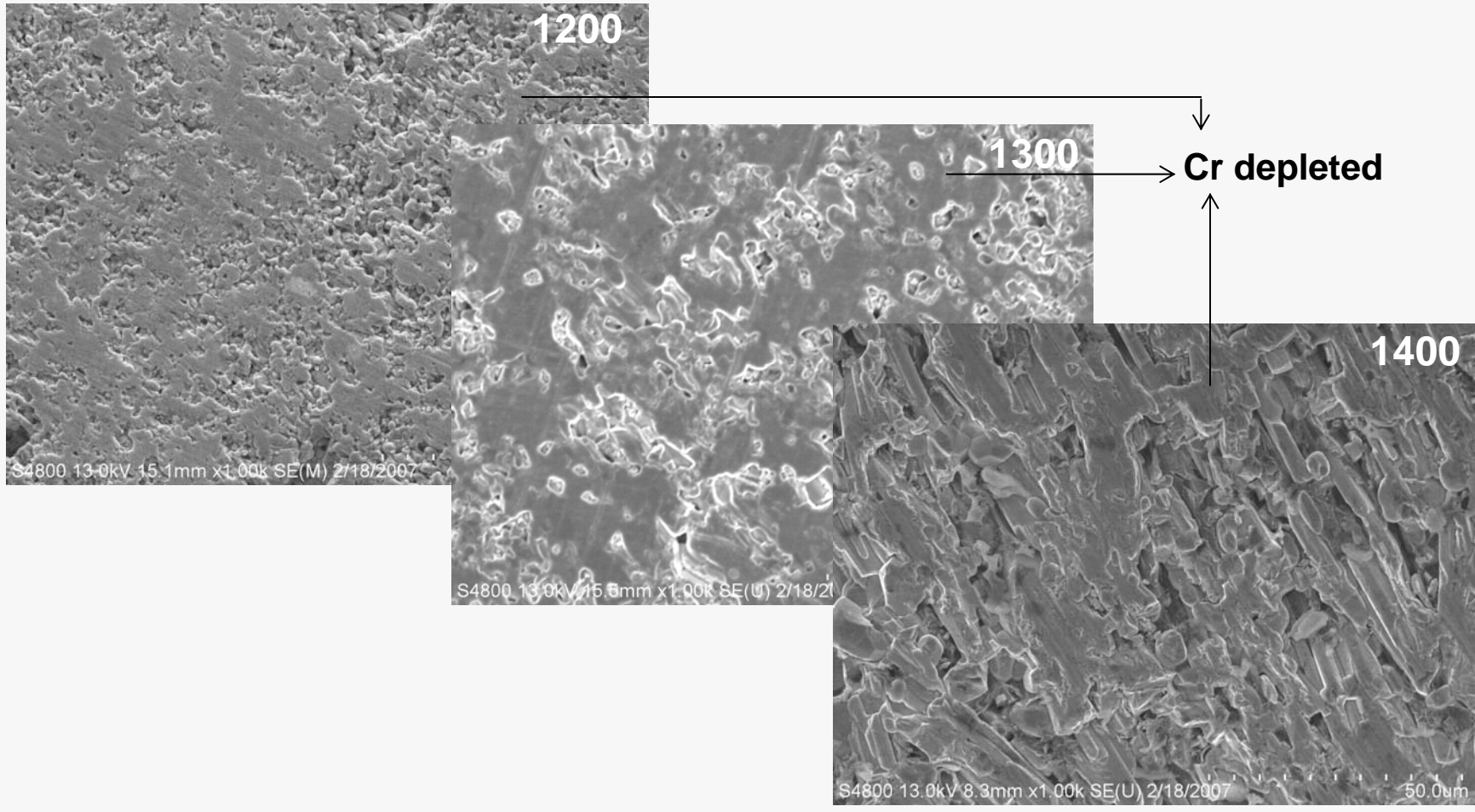
AT.%	Nb		W		Cr		O	
	F*	H*	F	H	F	H	F	H
BB1200	26.5	24.1	5.61	4.24	5.81	8.84	62.08	62.78
BB1300	27.1	22.8	6.43	4.28	4.92	12.25	61.53	60.67
BB1400	25.0	25.6	3.08	3.71	7.29	8.3	64.6	62.36
AB1200	29.8	42.3	8.17	5.8	1.47	11.5	60.5	40.3
AB1300	33.6	26.2	5.68	4.33	0.94	13.8	59.69	55.6
AB1400	32.8	18.9	5.25	2.5	1.19	11.07	60.73	67.5

*F = Flat regions in micrograph; H = Hollow regions in micrograph

SEM on polished surface of BB alloy



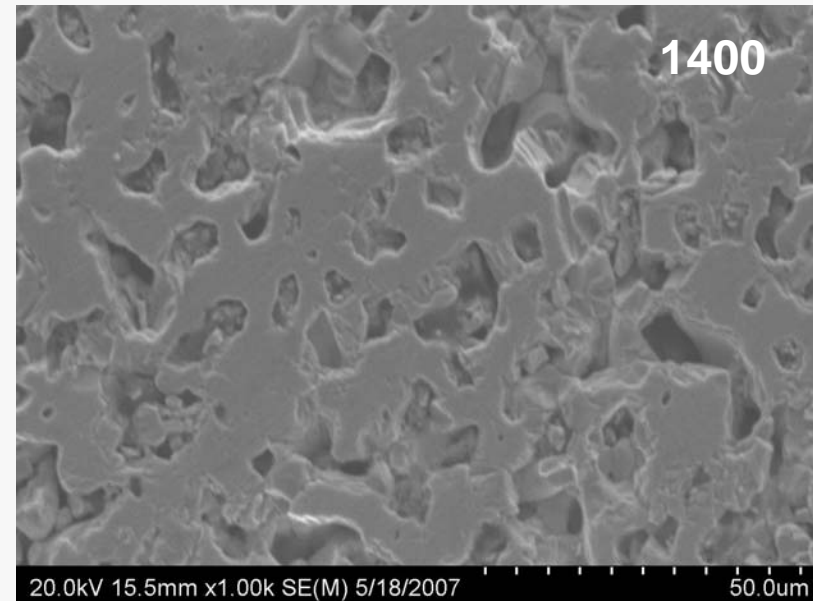
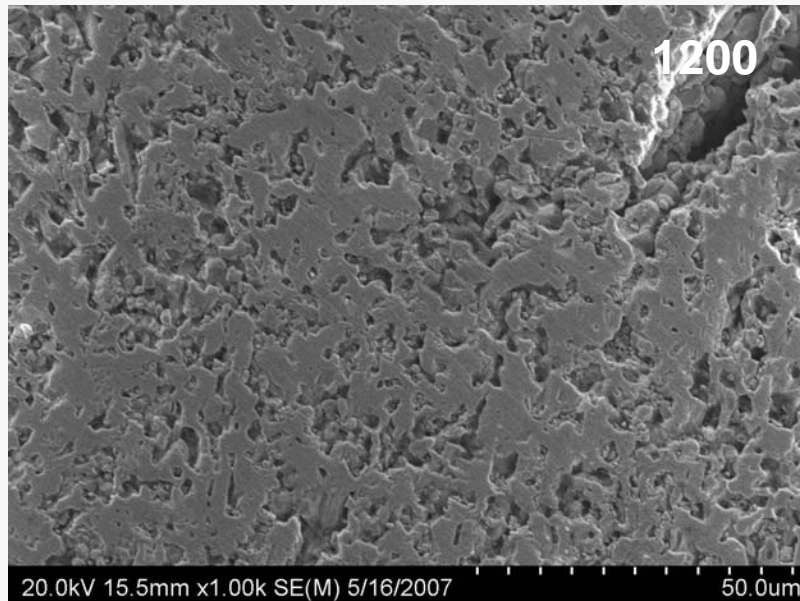
Oxidized in air at 1200, 1300, and 1400°C for 24hrs 1000x



SEM on polished surface of AC alloy



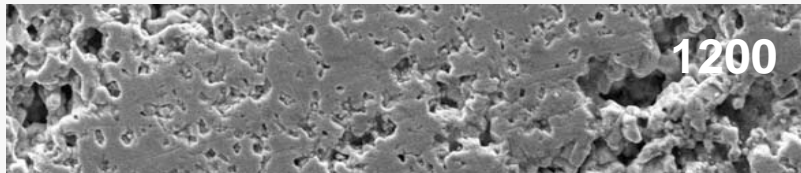
Oxidized in air at 1200 and 1400 for 24hrs 1000x



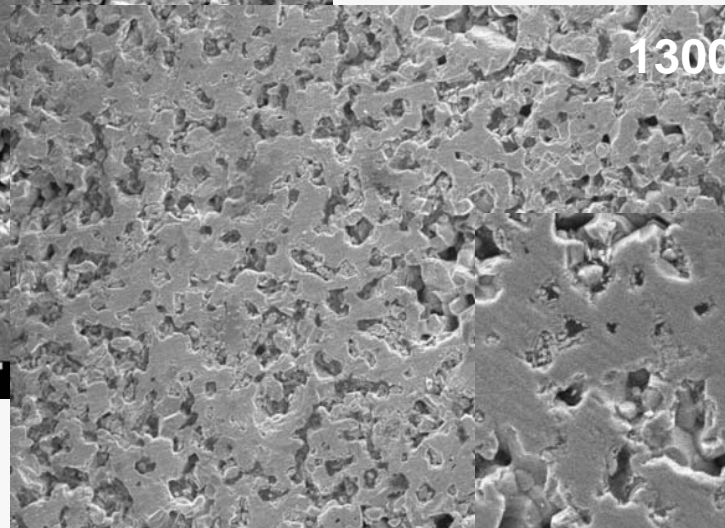
SEM on polished surface of BC alloy



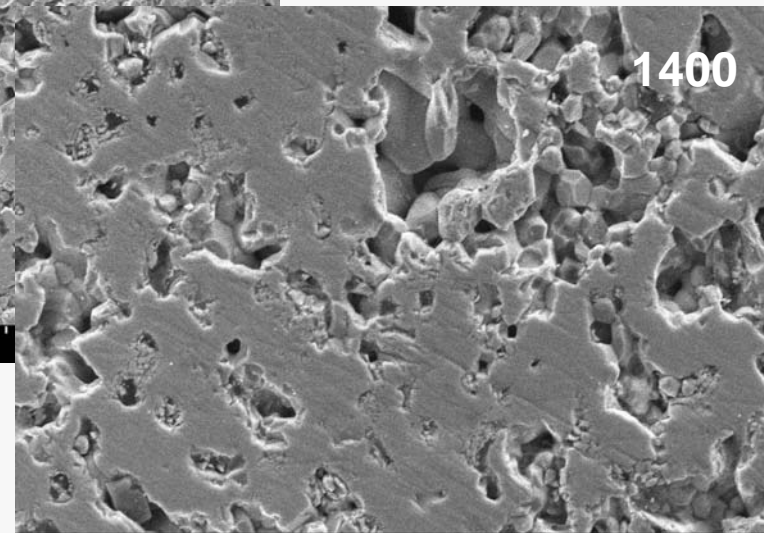
Oxidized in air at 1200, 1300, and 1400 for 24hrs 1000x



20.0kV 15.5mm x1.00k SE(M) 5/16/2007



20.0kV 15.5mm x1.00k SE(M) 5/18/2007



20.0kV 15.5mm x1.00k SE(M) 5/18/2007

50.0um



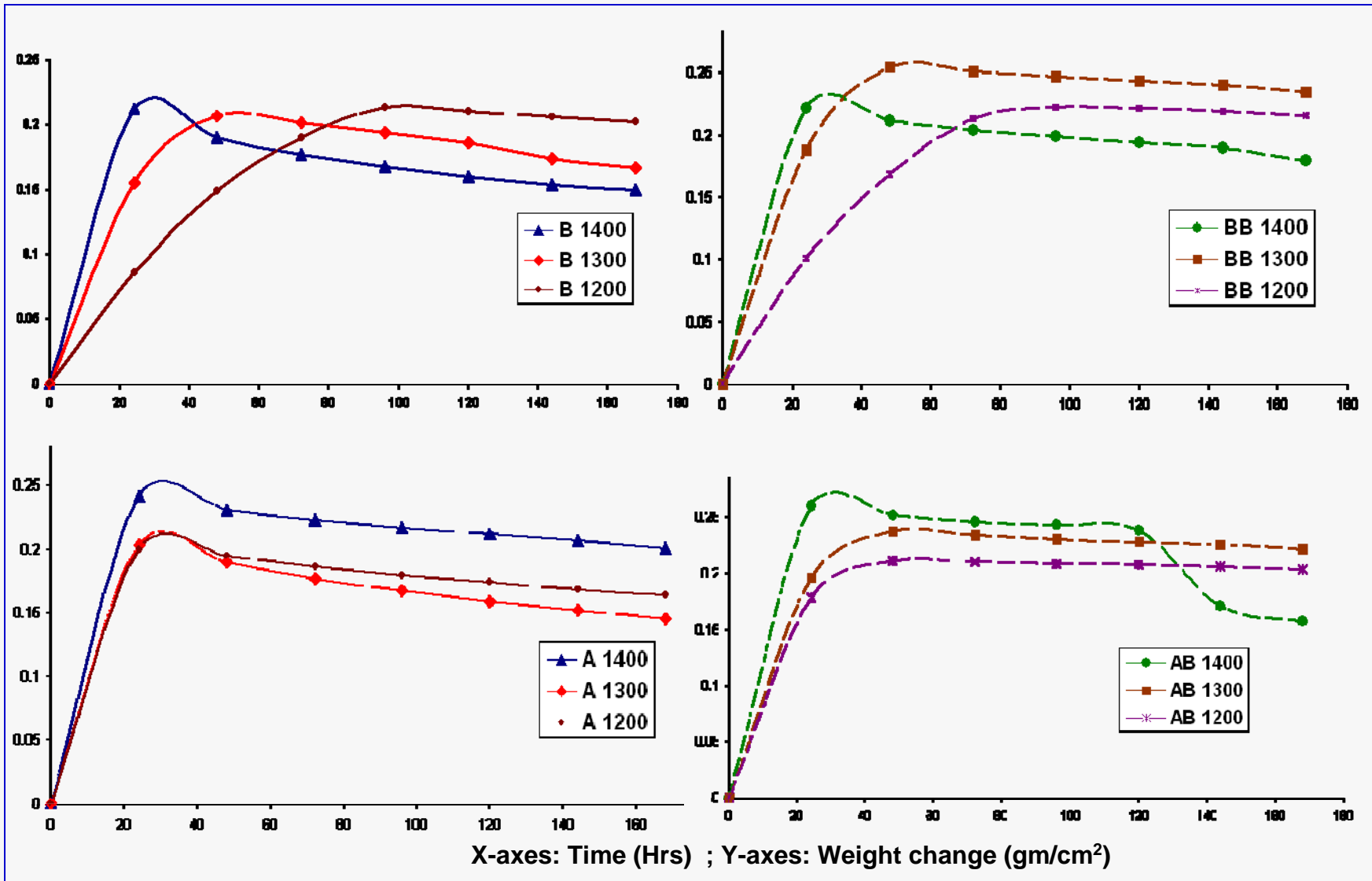
Thermal Linear Expansion Values (%) for Metal Oxides

Oxide	700	800	900	1,000
Nb_2O_5	0	0.02	0.04	0.07
Cr_2O_3	0.54	0.60	0.68	0.76
WO_3	0.98	1.02	1.14	--



Long term oxidation

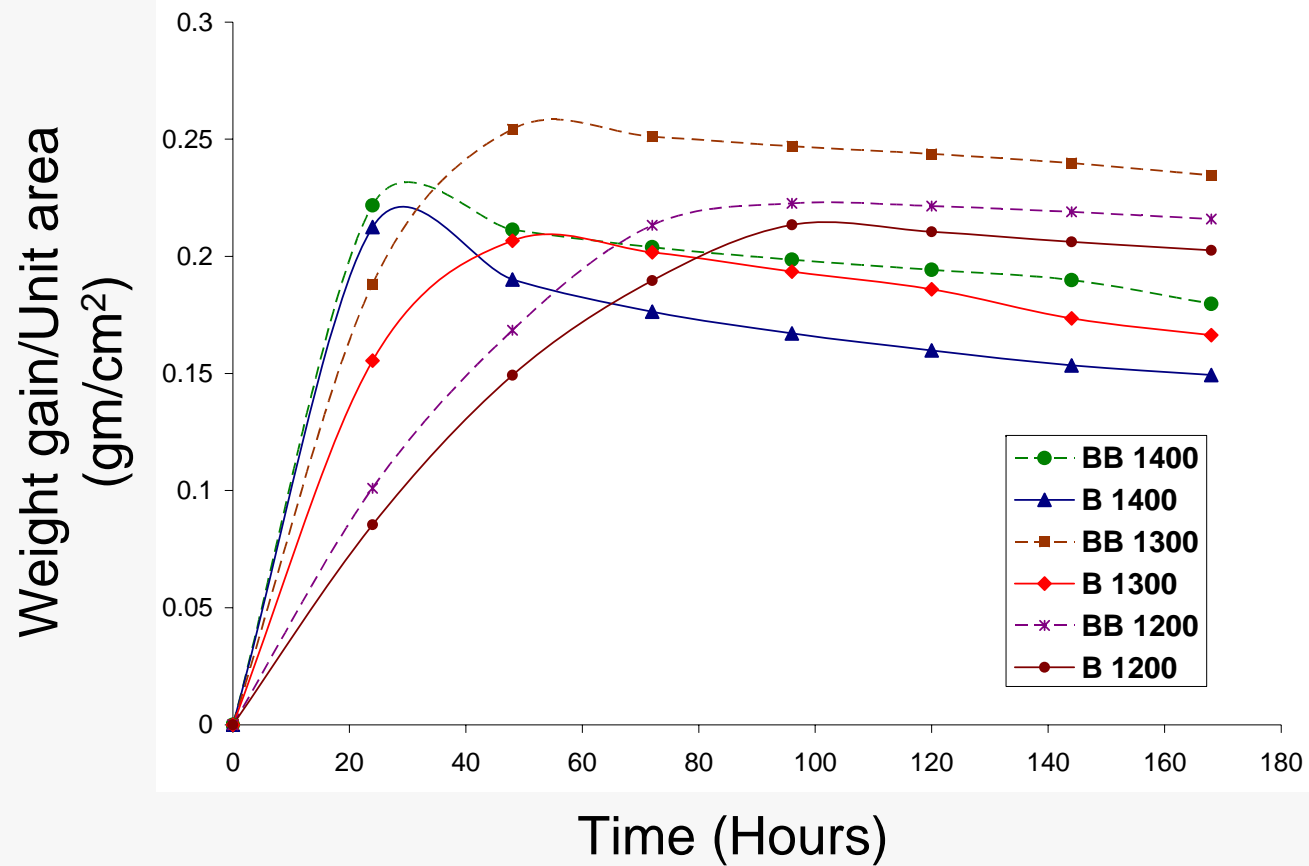
Oxidation behavior of alloys B, BB, A, and AB





Weight gain chart - LTO

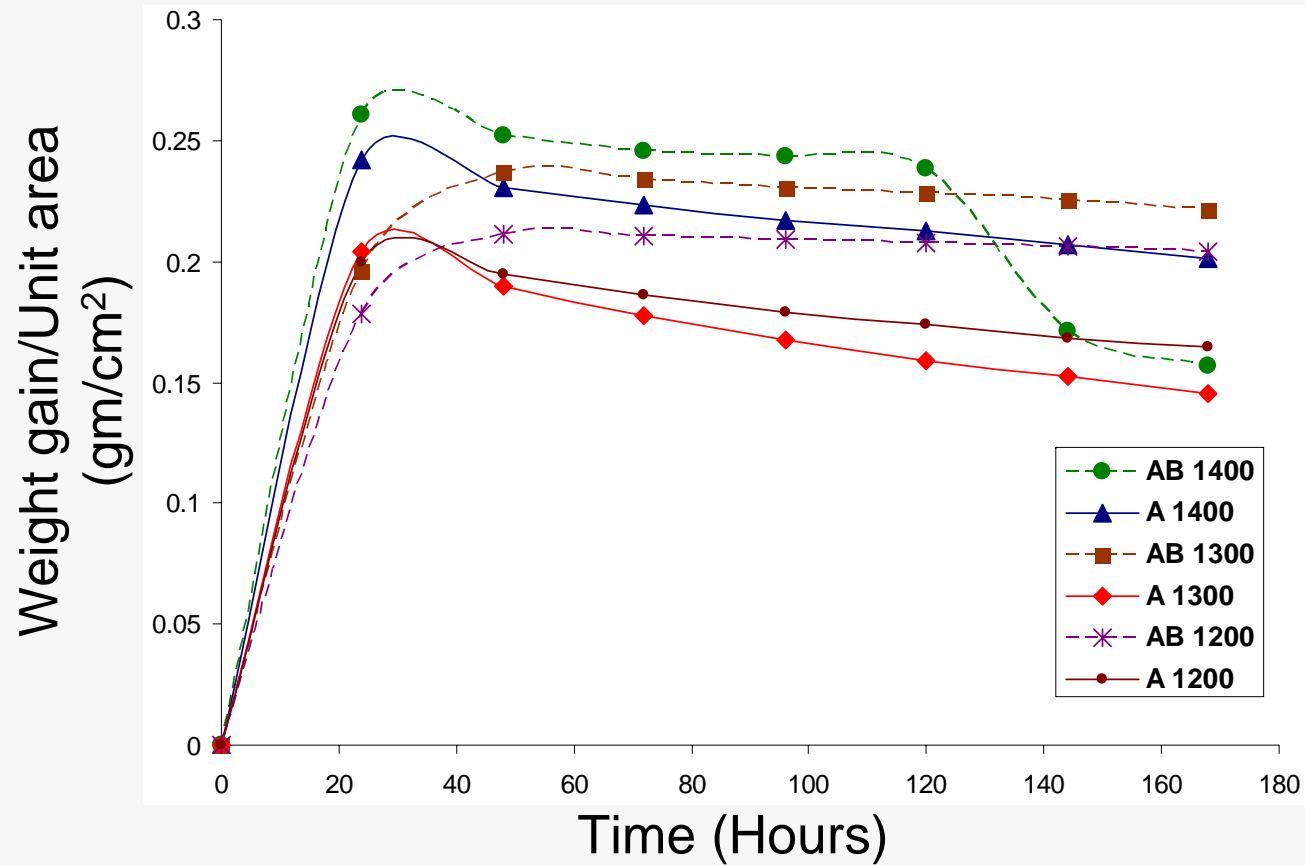
Oxidation curves of the alloys B and BB at 1200 -1400°C





Weight gain chart - LTO

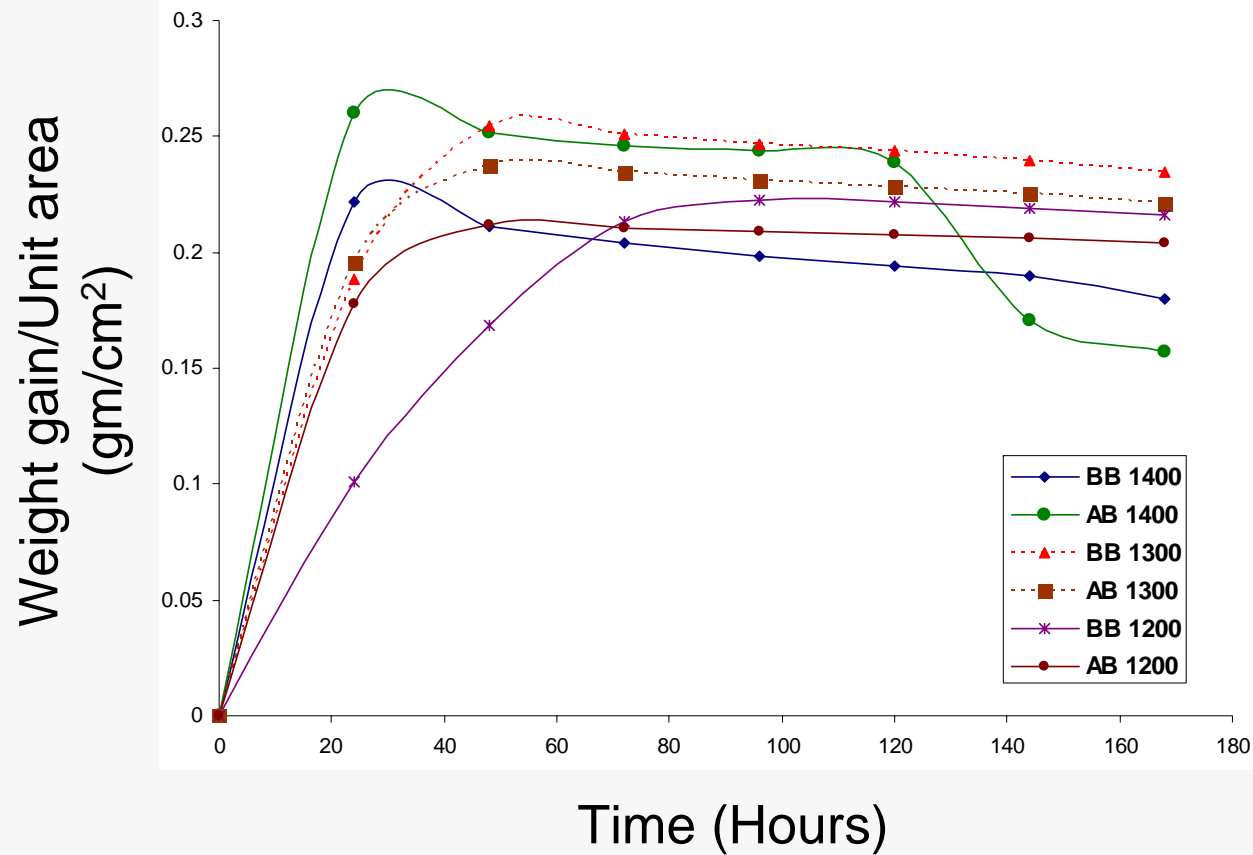
Oxidation curves of the alloy A and AB at 1200 -1400°C





Weight gain chart - LTO

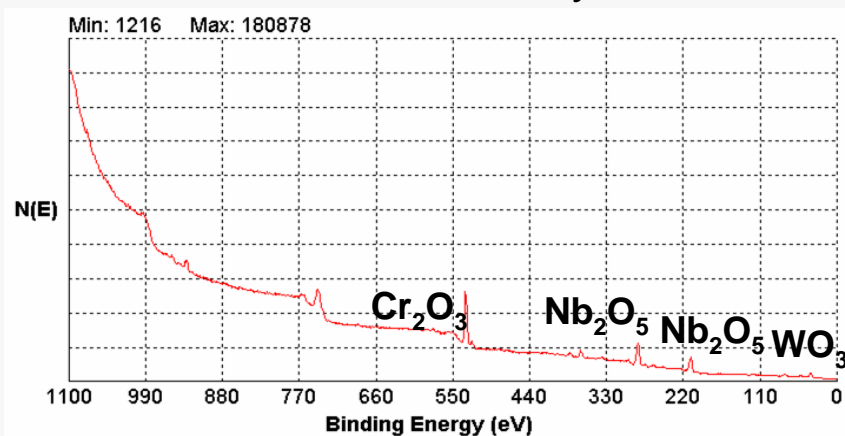
Oxidation curves of the alloy AB and BB at 1200 -1400°C



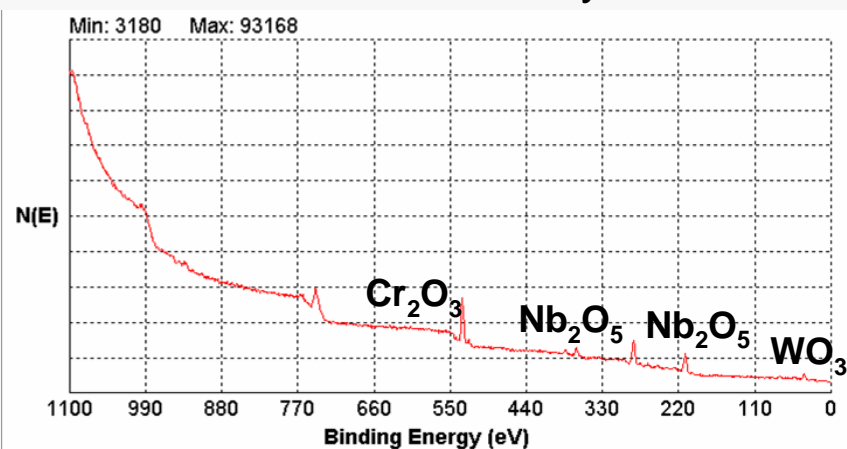


XPS on alloy B

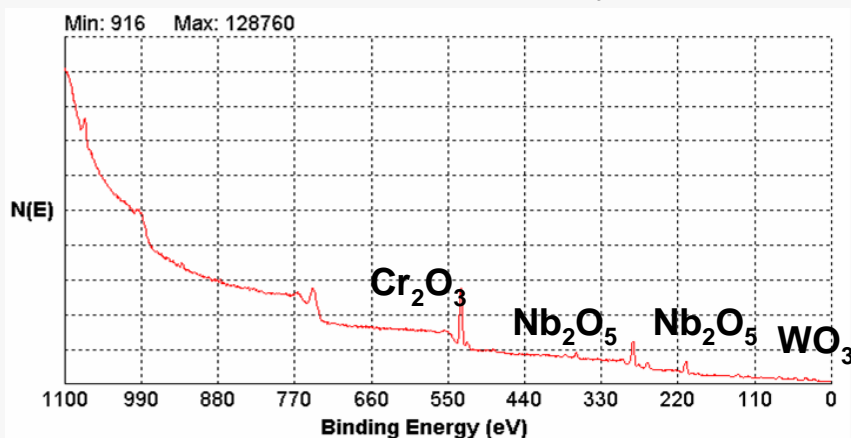
XPS survey on 1100°C



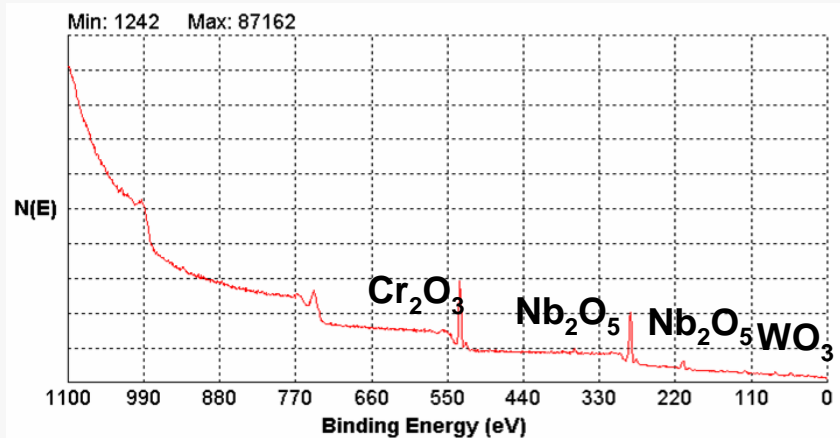
XPS survey on 1200°C



XPS survey on 1300°C



XPS survey on 1400°C





Features of Oxidation Resistance

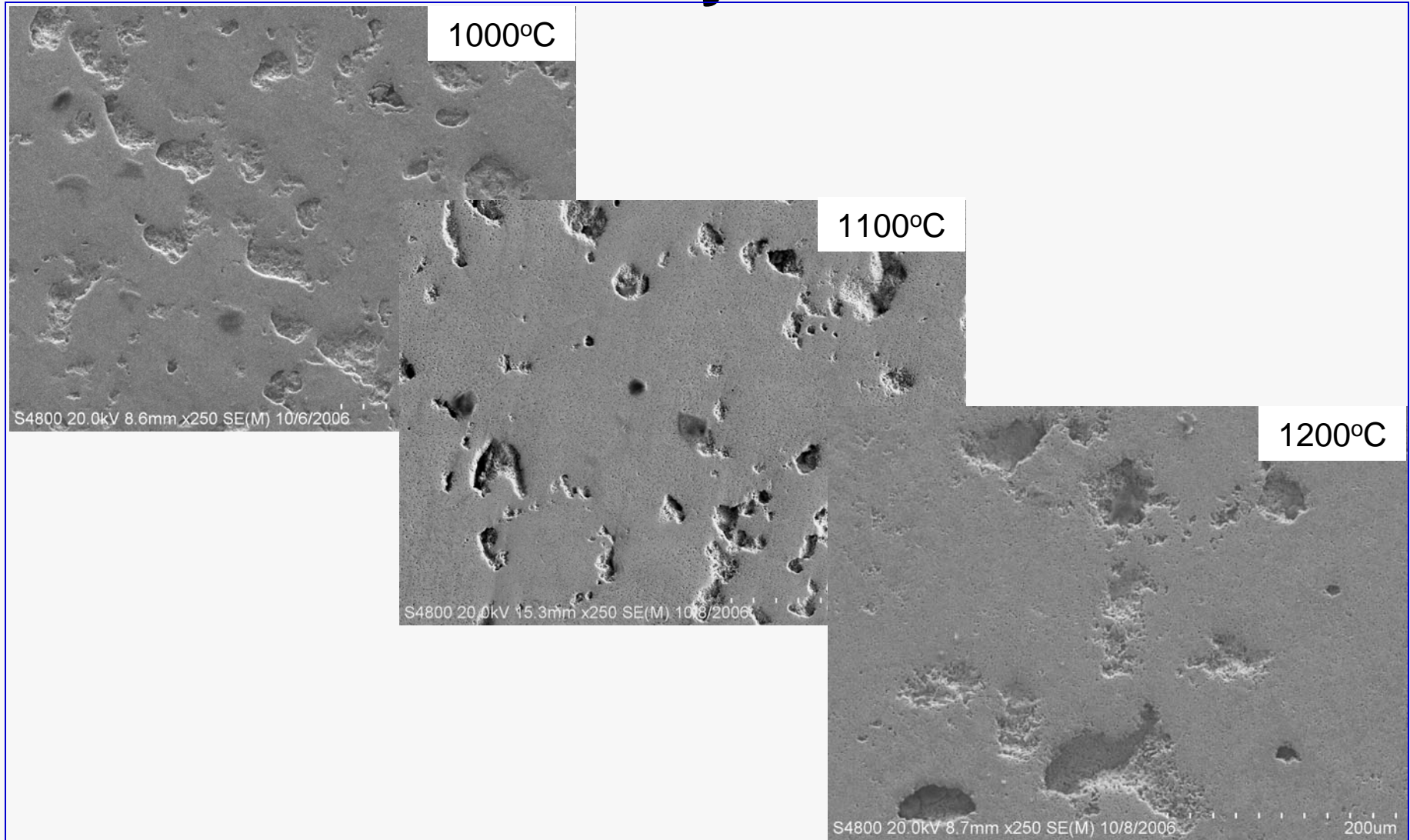
- ✚ Complete powders at 800 and 900°C and significant amounts at 700 and 1000°C
 - ✚ Oxidation resistance increases with the increase in temperature during LTO
-



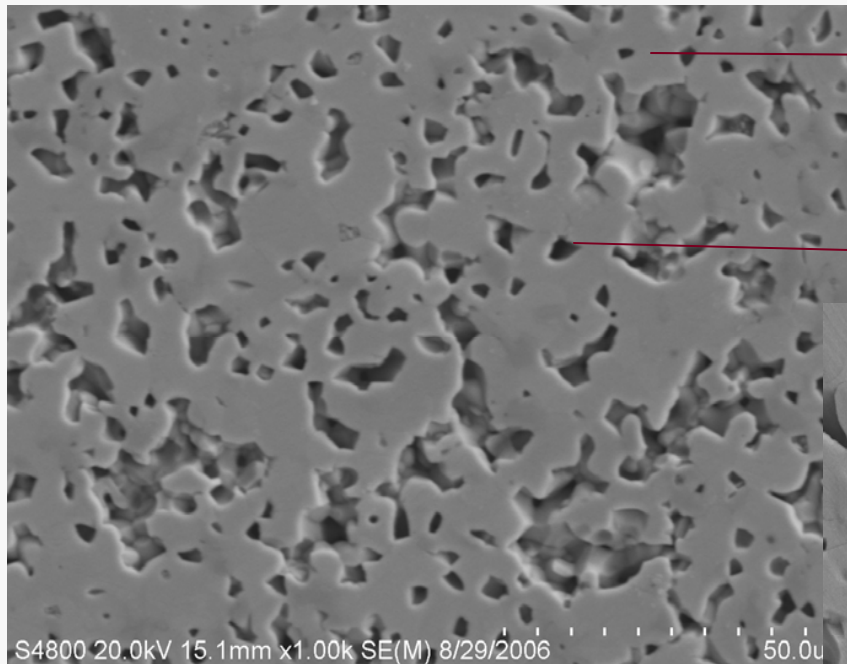
Solubility of Oxygen in Nb and Cr

	Temperature (°C)	Solubility (at.%)
Cr	1,100	0.0025
	1,500	0.043
Nb	700	1
	1,915	9

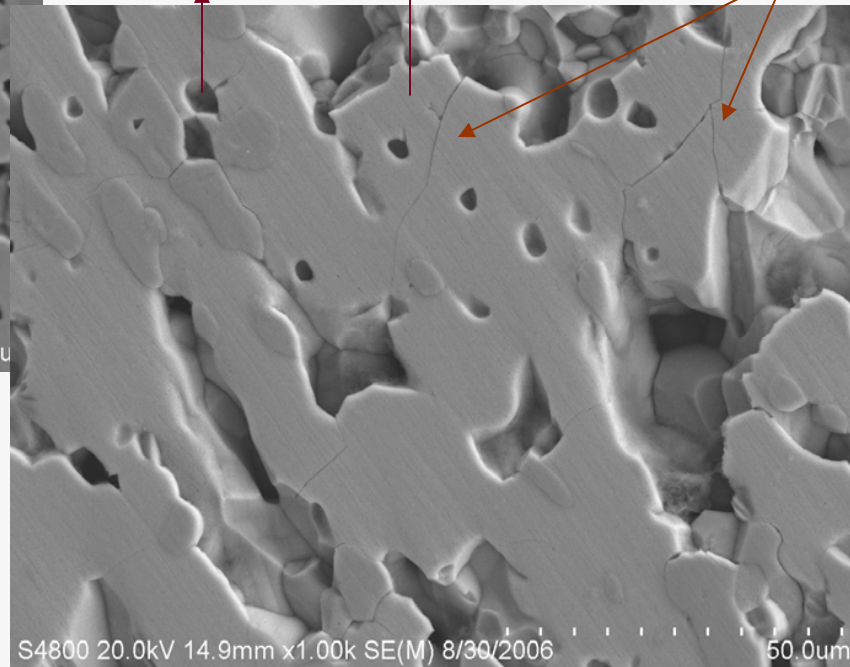
SEM – High temperature oxidation of alloy B



SEM – High temperature oxidation of alloy B

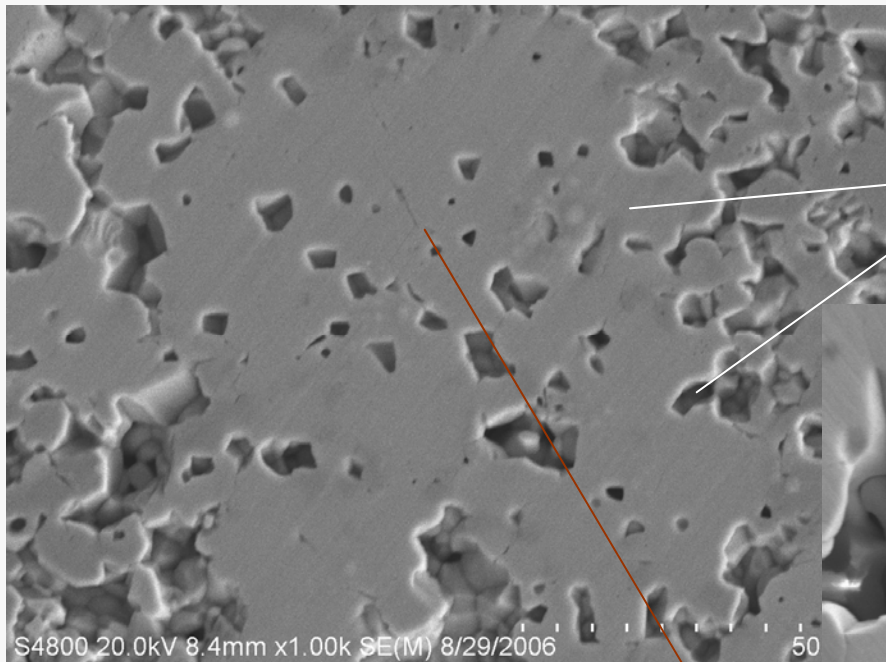


SEM micrograph of alloy oxidized at 1300°C after 24hrs

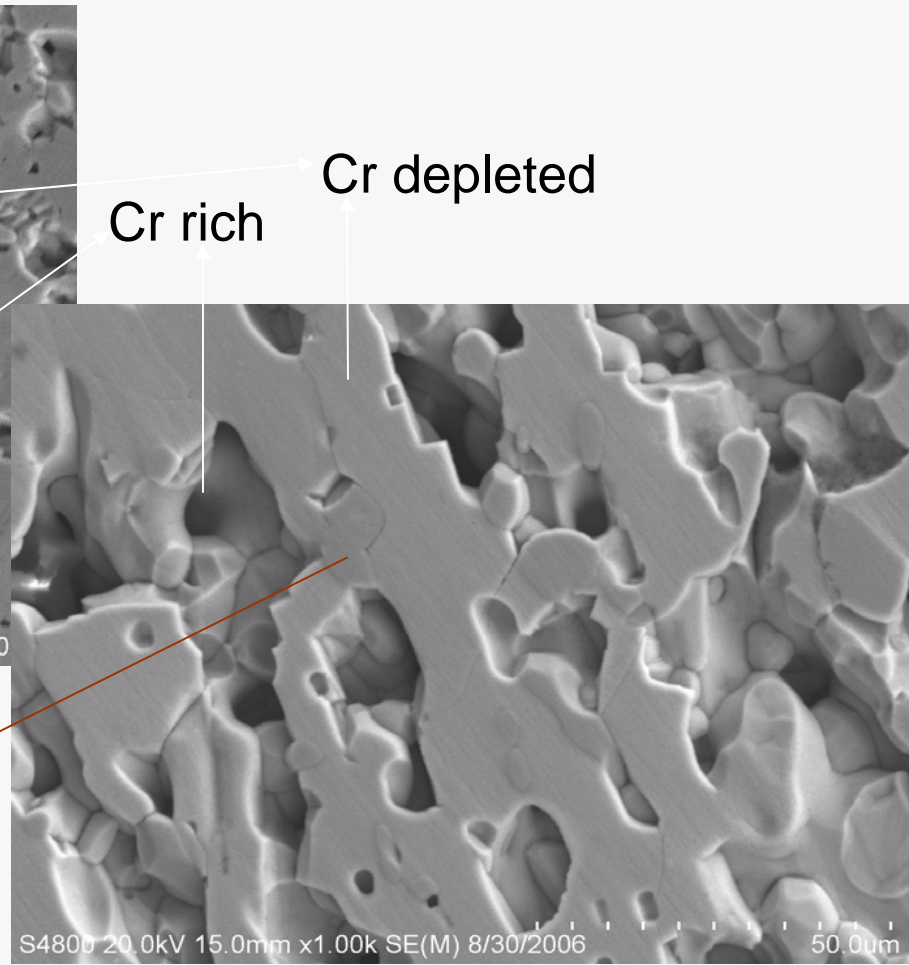


SEM micrograph of alloy oxidized at 1400°C after 24hrs

SEM – High temperature oxidation (LTO)



Alloy oxidized at 1300°C
for 168 hrs



Cr rich Cr depleted

Cracking

Alloy oxidized at 1400°C
for 168 hrs



Conclusions

- ✚ Powder formation or fragmentation during oxidation in air is perhaps due to formation of WO_3 because of its extremely high thermal linear expansion.
 - ✚ $NbCr_2$ may be the micro constituent responsible for enhanced oxidation. The amount of it decreases with an increase in temperature in the range of 1100 to 1400°C which may contribute to the improved oxidation resistance.
-



Conclusions

- ✚ It appears that Cr concentration controls the oxidation resistance at elevated temperature.
 - ✚ An increase in oxidation resistance with an increase in temperature at elevated temperatures can be attributed to the unusual increase in oxygen solubility in Nb.
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Conclusions

- ✚ NbCrO_4 and Nb_2O_5 are falling off from the alloy during high temperature oxidation.
 - ✚ B addition is not beneficial for improving the oxidation resistance of high Cr alloys. Role of B in these alloys is not well delineated from the results of this study.
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THANK YOU

JNB

