Surface oxidation of Fe-Si alloy

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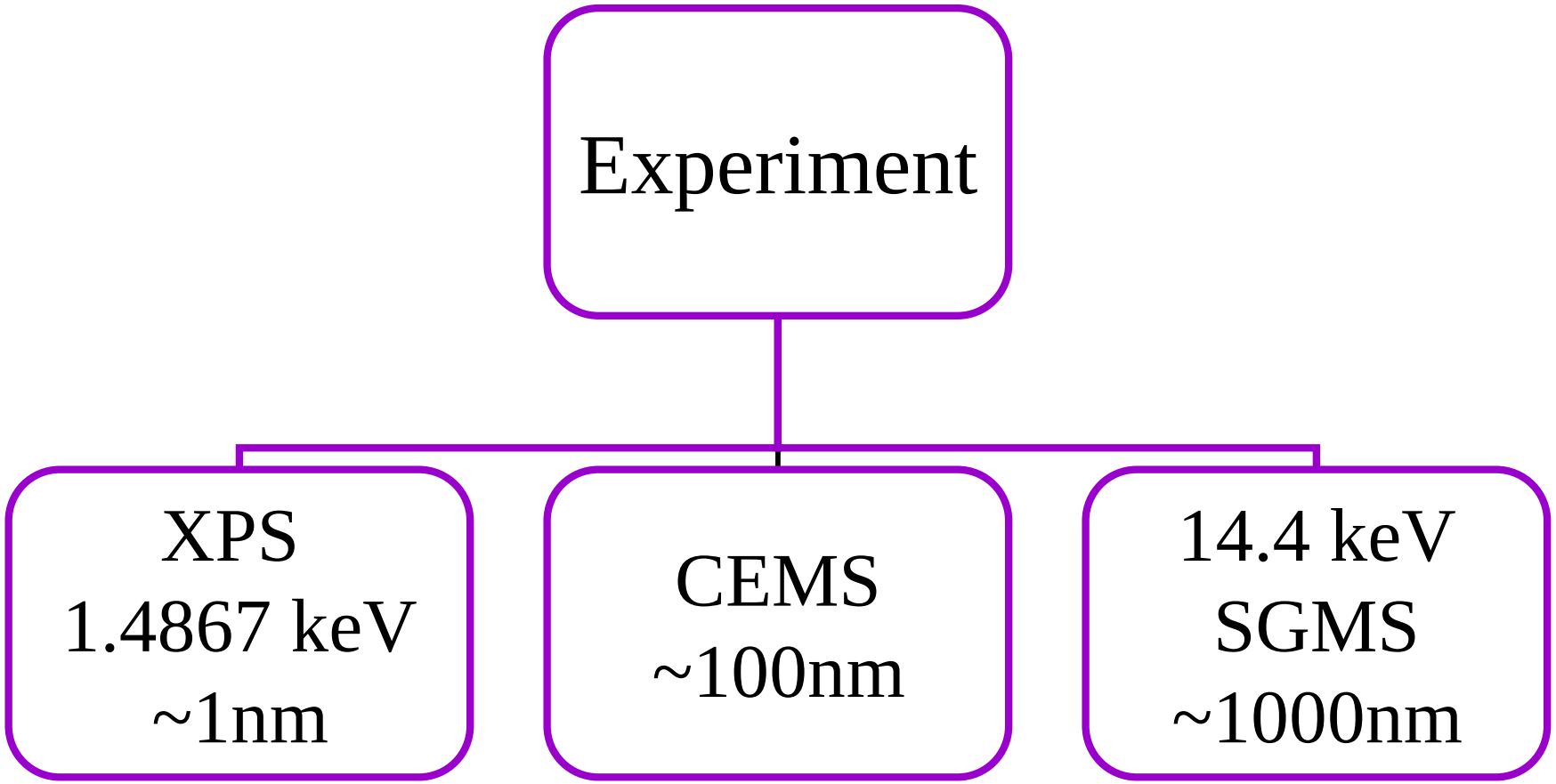
Aim

Investigation of phase composition at surface of bcc $Fe_{94}Si_6$ alloy based on heat treatment.


Experiment
XPS
1.4867 keV
~1nm
CEMS
~100nm
14.4 keV
SGMS
~1000nm

Samples:

11×11 × 0.3 mm plates of Fe–3wt.%Si

(grain oriented steel sheet) polished using the best metallography procedure.

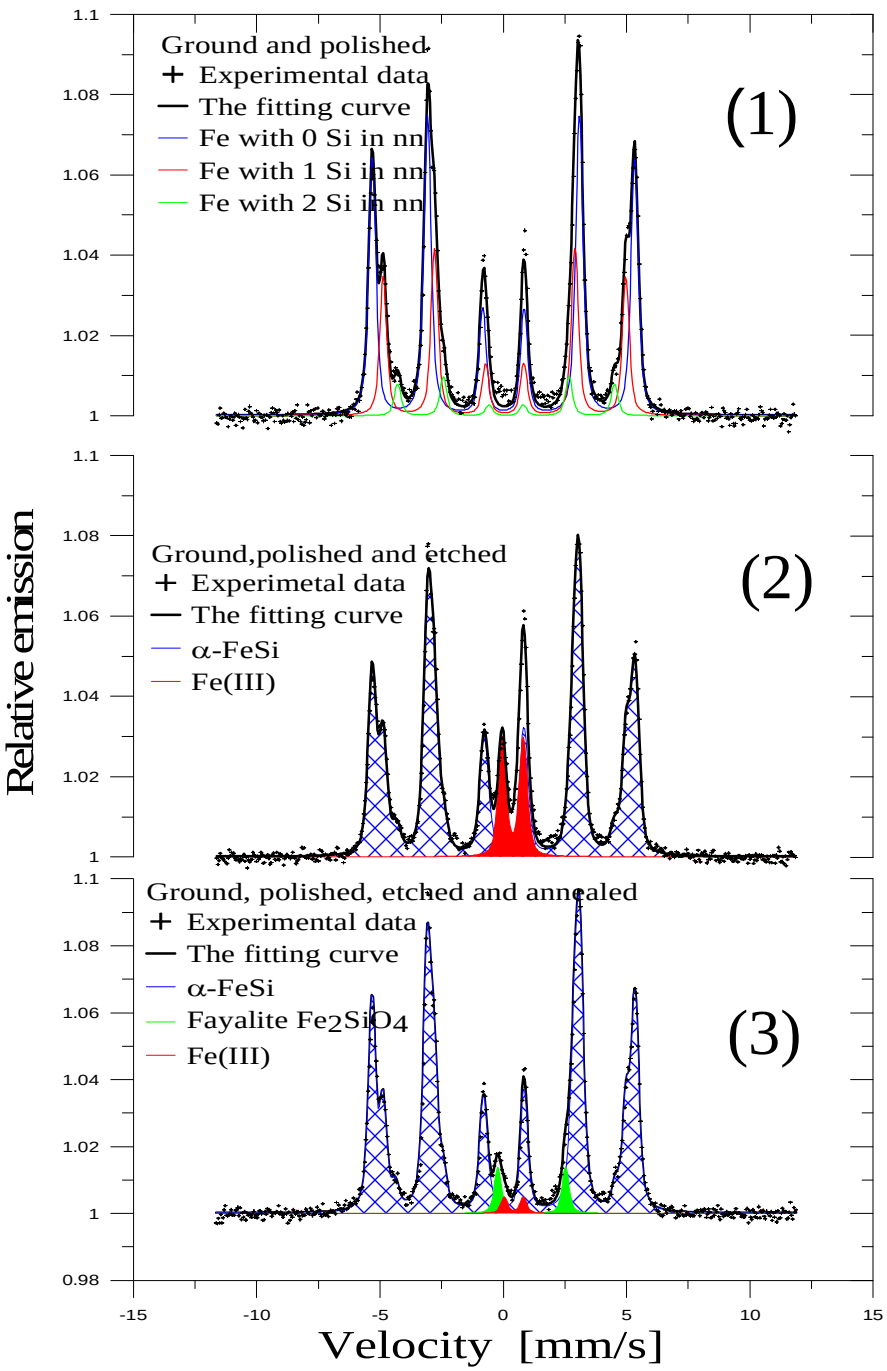
Mössbauer spectroscopy

1- Standard Mössbauer spectra were measured in scattering geometry with detection 14.4 keV gamma radiation using 2π proportional counter.

2- ^{57}Fe CEMS spectra were measured using ^{57}Co in Rh source at room temperature in gas-filled detector

XPS

spectra were taken in Institute of Chemical Technology, Department of Power Engineering, Prague, Czech Republic for this sample using ESCA equipment using 1.4867 keV X-ray radiation on Fe 2p edge. Surface cleaning by repeated Ar sputtering was carried out in situ.



1- CEMS spectra for the polished sample contains three sextets

S1: δ= 0.00 mm/s, σ=0.00 mm/s, B_{hf}=33.1 T

S1: δ= 0.05 mm/s, σ=0.00 mm/s, B_{hf}=30.9 T

S1: δ= 0.11 mm/s, σ=-0.04 mm/s, B_{hf}=27.4 T

2- CEMS spectra for the polished and etched sample (in $HF+H_2O_2$) showed:

a) Three sextets for the Substrate.

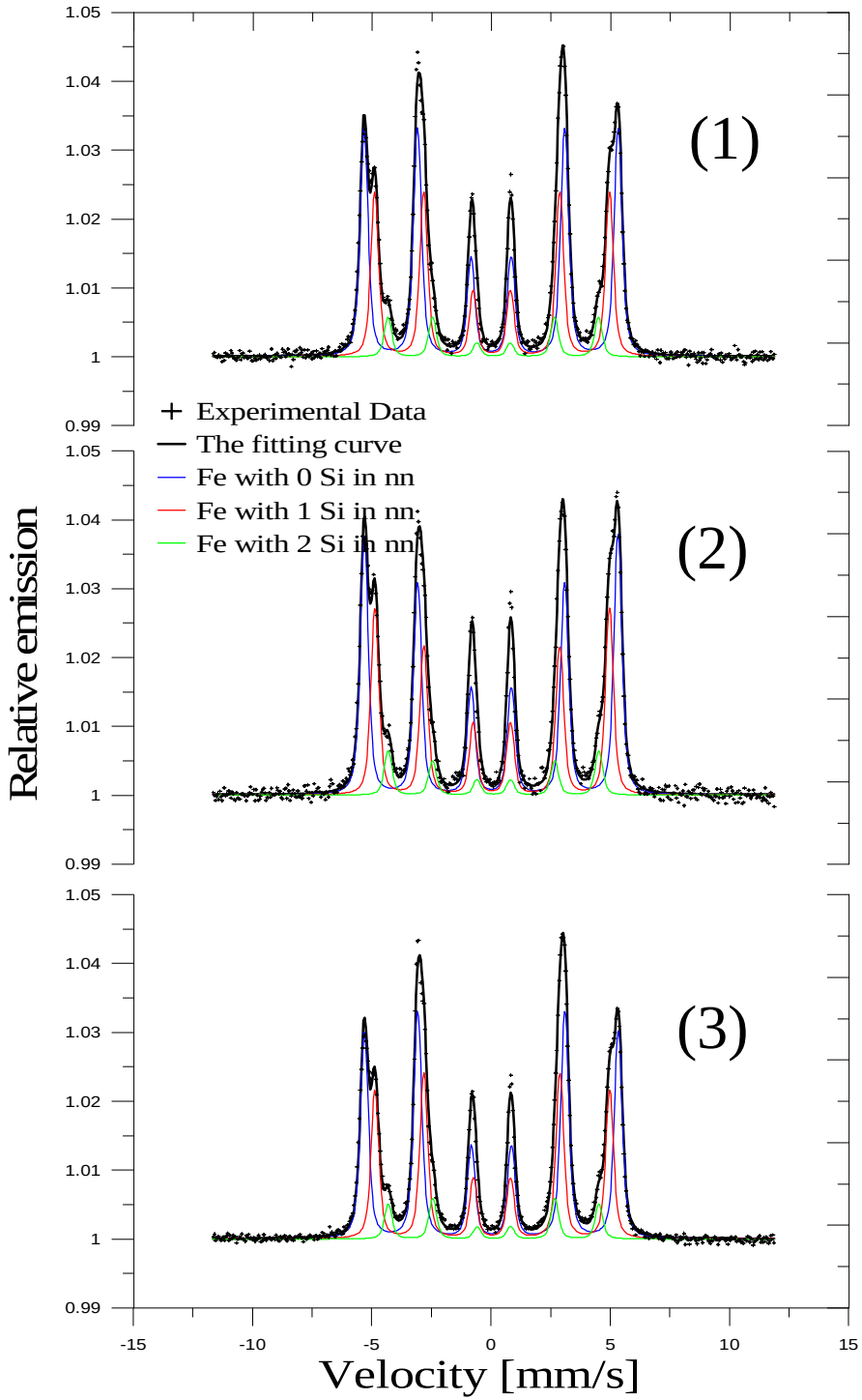
b) Fe (III), δ=0.36 mm/s and ε_Q=0.82 mm/s, with A=0.11.

3-CEMS spectra after annealing in vacuum for 2 hours at 780 ^oC showed:

a) Three sextets for the substrate.

b) Fayalite Fe_2SiO_4, δ=1.15 mm/s and ε_Q=2.72 mm/s, with A=0.04.

c) Fe(III) δ=0.42 mm/s and ε_Q=0.78 mm/s , with A=0.01.



1-SGMS spectra for the polished sample showed:

Three sextets for the substrate

S1: δ= 0.00 mm/s, σ=0.00 mm/s, B_{hf}=33.1T

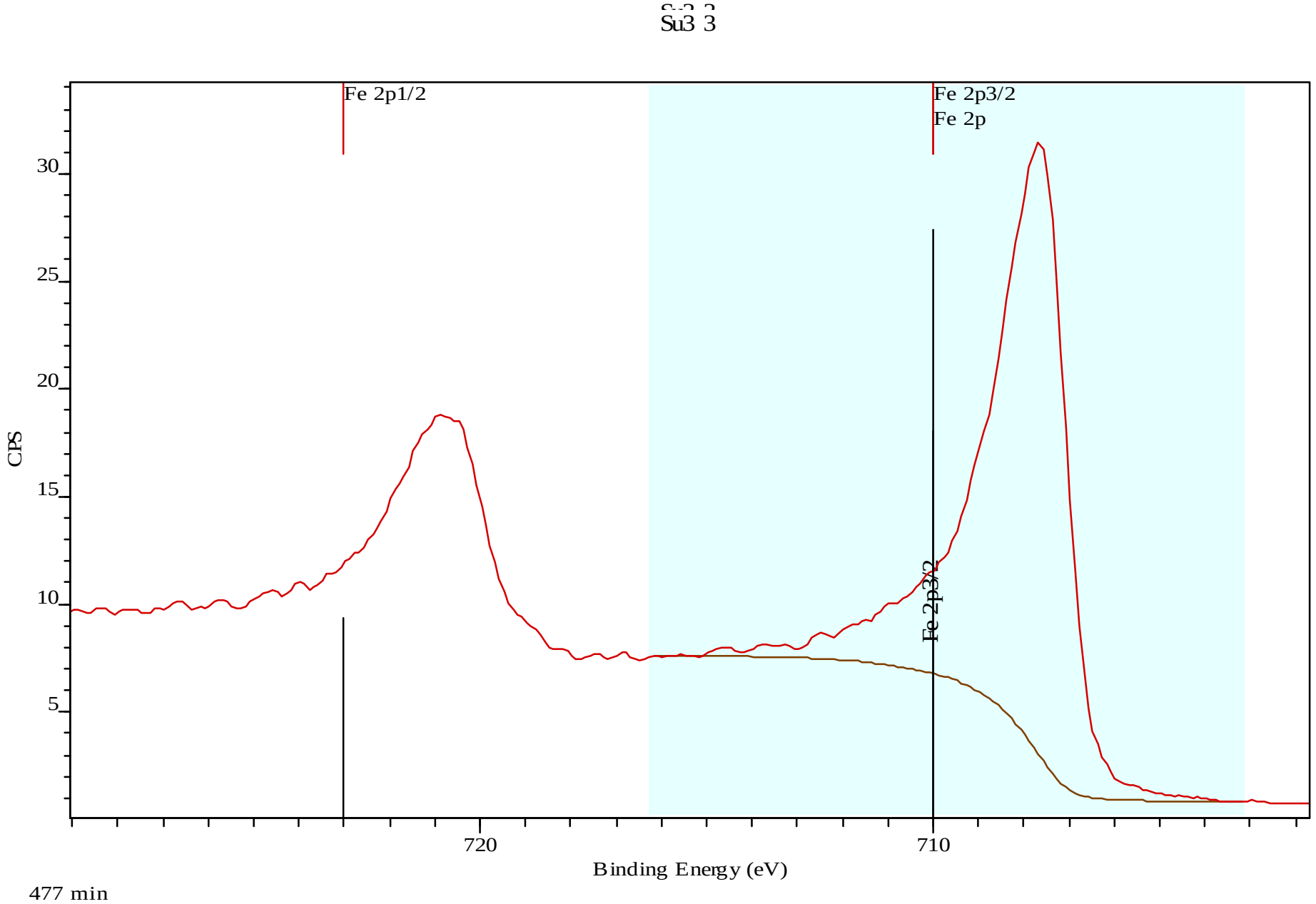
S1: δ= 0.04 mm/s, σ=0.00 mm/s, B_{hf}=30.6T

S1: δ= 0.10 mm/s, σ=0.00 mm/s, B_{hf}=27.4T

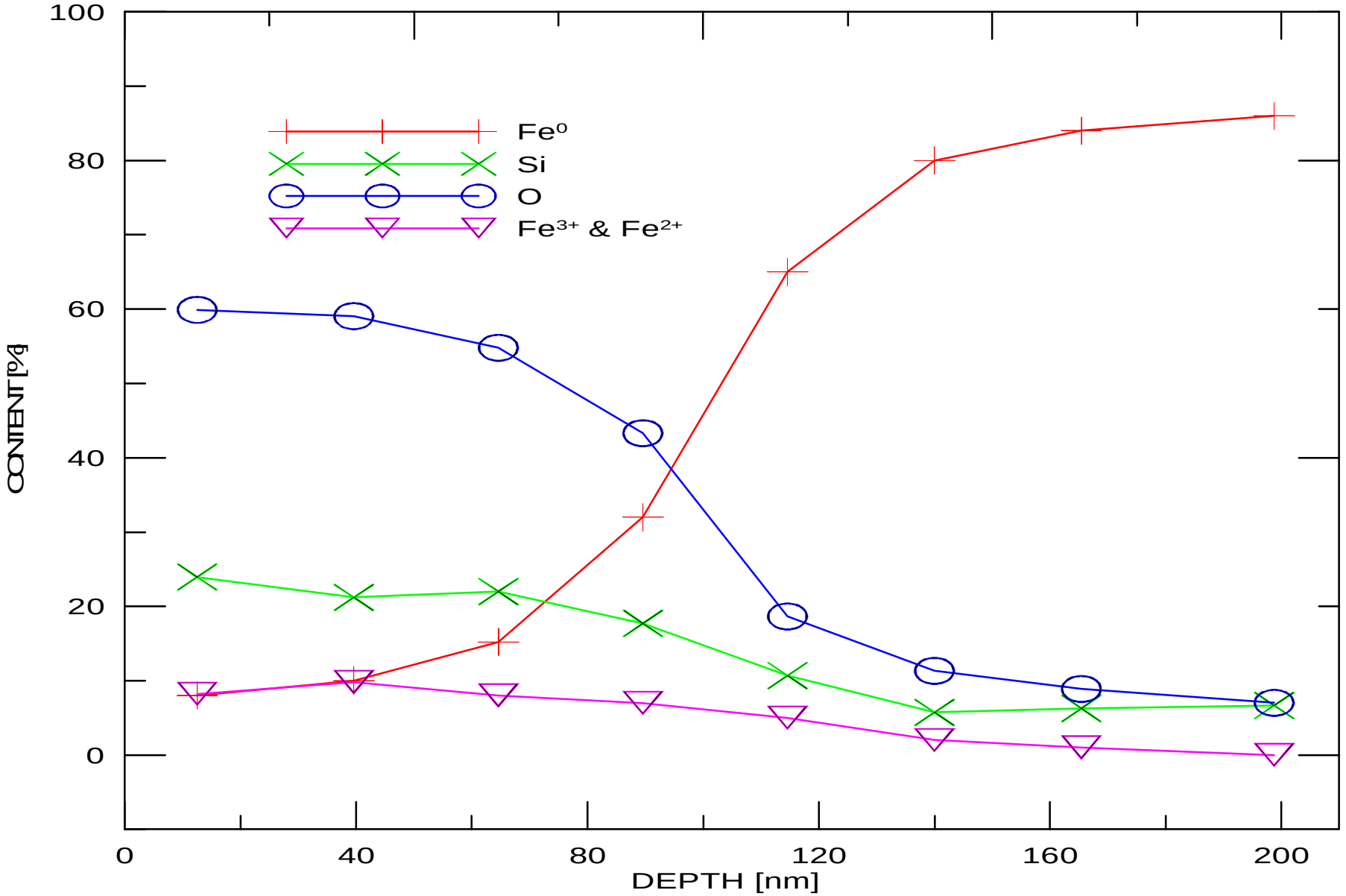
2- SGMS spectra for the polished and etched sample (in $HF+H_2O_2$)

3-SGMS spectra after annealing in vacuum for 2 hours at 780 ^oC

XPS measurement



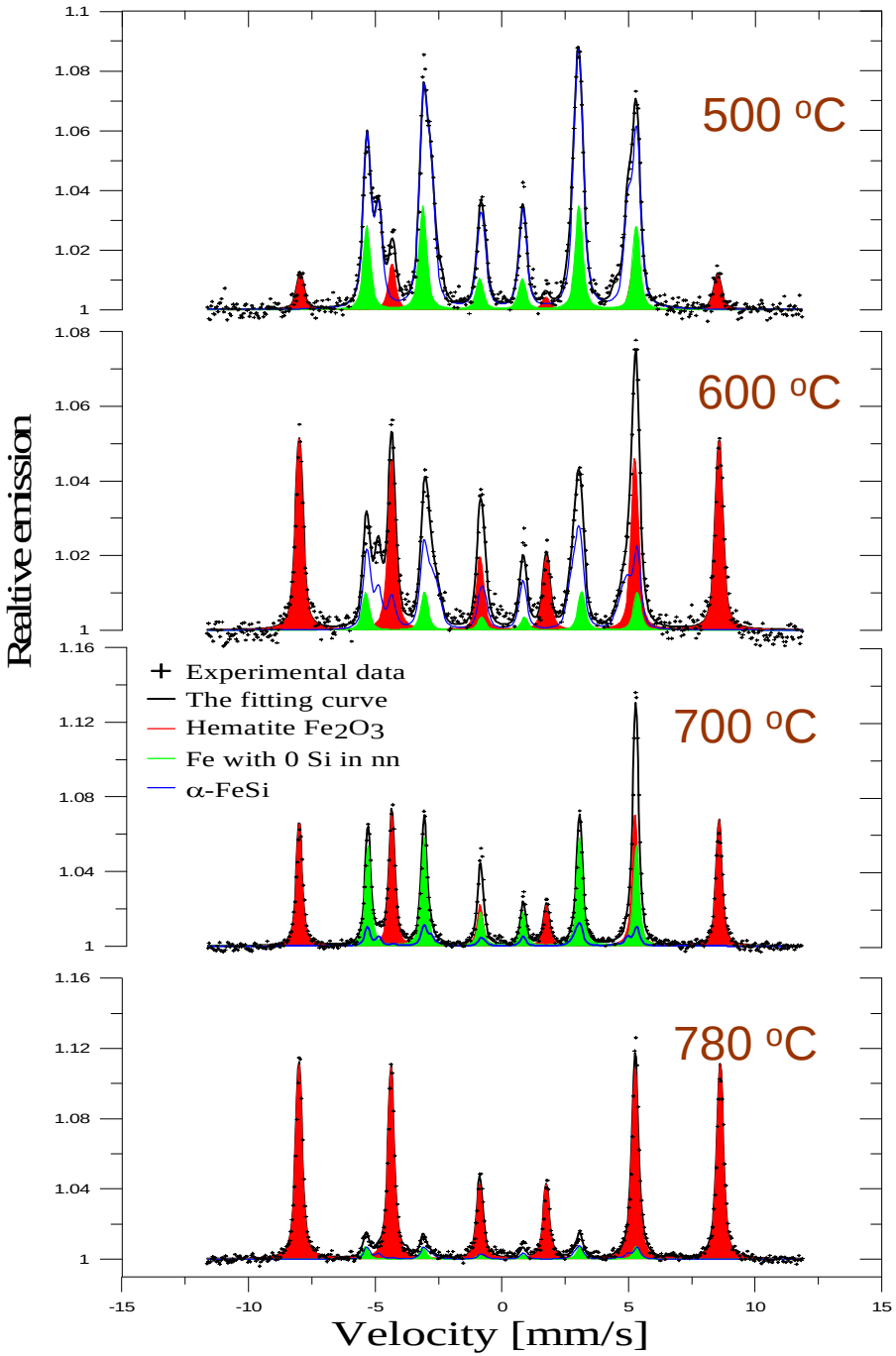
The depth profile



Annealing in oxygen

Four samples have been ground and polished both sides and then annealed in oxygen for 10 minutes at 500 ^{o}C, 600 ^{o}C, 700 ^{o}C and 780 ^{o}C respectively.

The CEMS measurements for the four samples showed hematite α-Fe_2O_3 , Fe with 0 Si in nn (pure iron) on the surface of these samples in addition to the substrate which contains α-FeSi.



The CEMS measurements for the four samples showed:

1-Hematite α-Fe_2O_3 (—)

a) At 500 ºC

δ= 0.36 mm/s, σ=-0.14 mm/s, B_{hf}=51.2 T

b) At 600 ºC

δ= 0.37 mm/s, σ=-0.16 mm/s, B_{hf}=51.5 T

c) At 700 ºC

δ= 0.37 mm/s, σ=-0.16 mm/s, B_{hf}=51.5 T

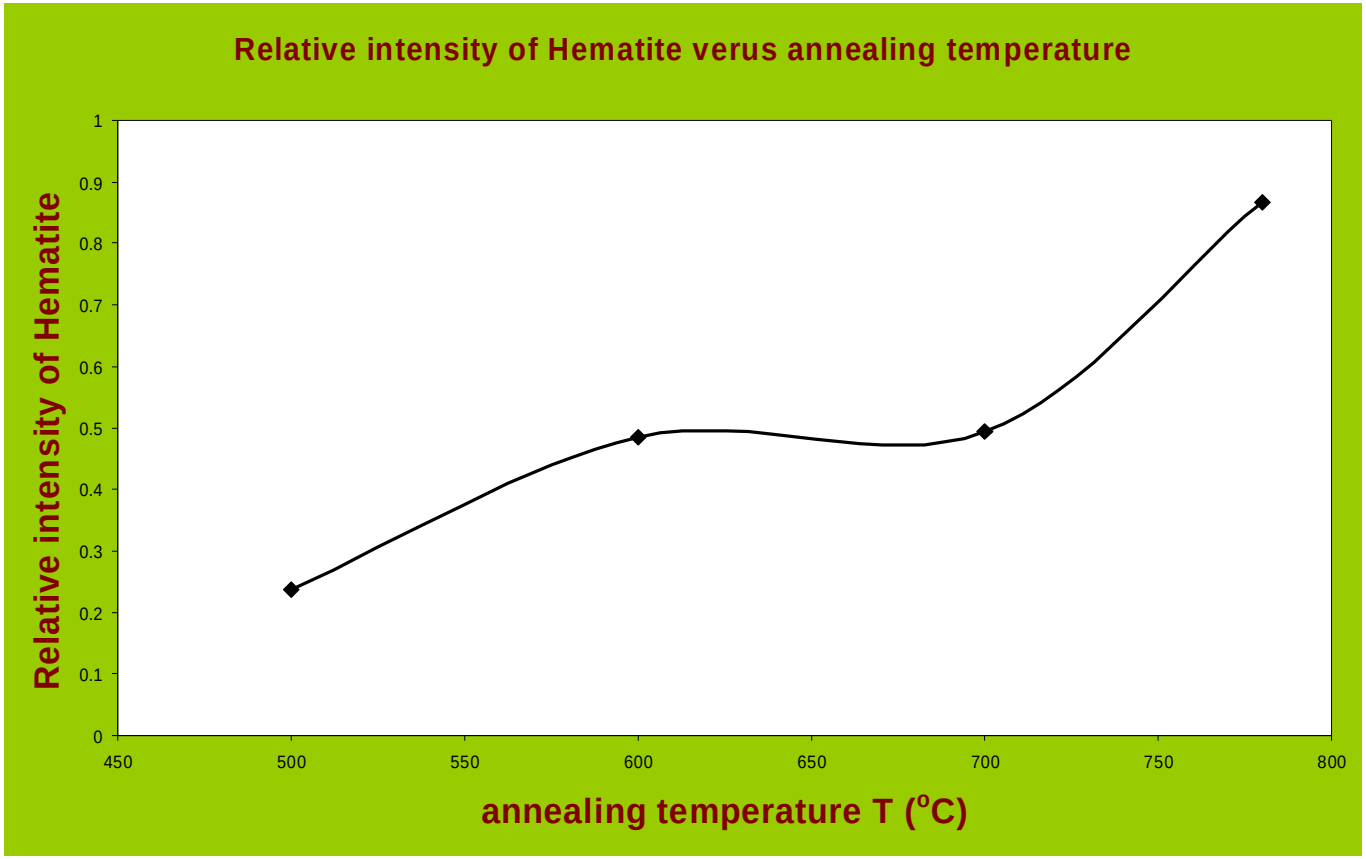
d) At 780 ºC

δ= 0.37 mm/s, σ=-0.16 mm/s, B_{hf}=51.6 T

2- Fe with 0 Si in nn (—)

3- α-FeSi (—)

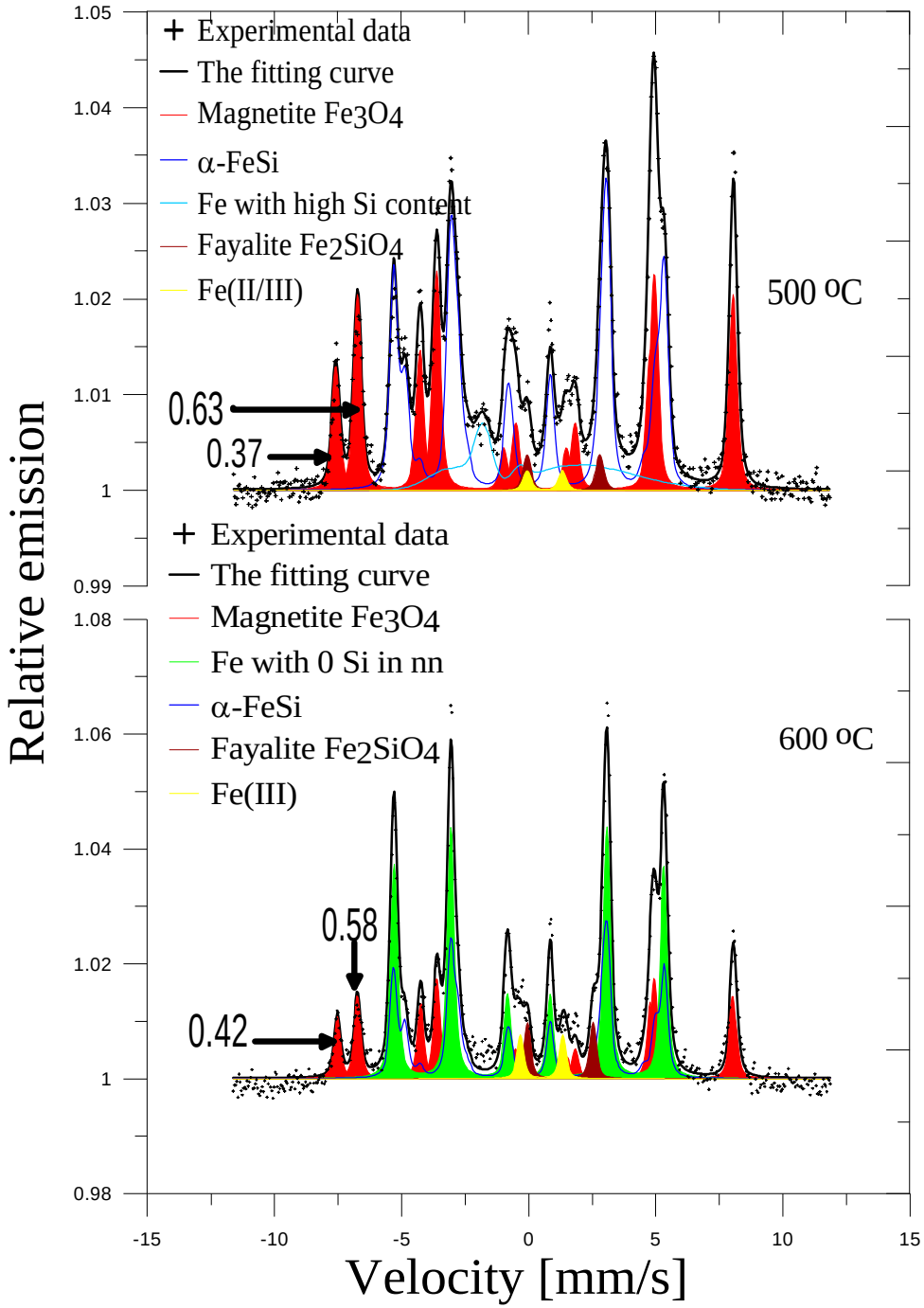
The concentration of hematite which has been detected on the surface of these samples increases by increasing the annealing temperature



Annealing in Ar

Two samples have been ground, polished and annealing in oxygen for 10 minutes at 500 ^{o}C and 600 ^{o}C and then polished once more and annealed in Ar for 10 minutes at 500 ^{o}C and 600 ^{o}C respectively.

CEMS spectra for these two samples showed magnetite Fe_3O_4, fayalite Fe_2SiO_4, and Fe(III) where the concentration of magnetite at 500 ^{o}C was greater than that at 600 ^{o}C.



1-At 500 ºC, CEMS showed:

(a) Magnetite Fe_3O_4 ( ) (~ 1:2)

S1: δ=0.24 mm/s, σ=0.00 mm/s, B_{hf}=48.6 T

S2: δ=0.65 mm/s, σ=0.00 mm/s, B_{hf}=45.8 T

(b) Fe with higher content of Si ( )

δ=0.28 mm/s, σ=0.10 mm/s B_{hf}=21.3 T

(c) α-FeSi ( )

(d) Fayatite Fe_2SiO_4 ( )

δ=1.36 mm/s, ε_Q=1.42 mm/s, with A=0.02

(e) Fe (II/III) ( )

δ=0.62 mm/s, ε_Q=0.69 mm/s with A=0.01

2-At 600 ºC, CEMS showed:

(a) Magnetite Fe_3O_4 ( ) (0.42: 0.58)

S1: δ=0.26 mm/s, σ=0.00 mm/s B_{hf}=48.4 T

S2: δ=0.65 mm/s, σ=0.00 mm/s B_{hf}=45.8 T

(b) Fe with 0 Si in nn ( )

δ=0.00 mm/s, σ=0.00 mm/s B_{hf}=32.9T

(c) α-FeSi ( )

(c) Fayalite Fe_2SiO_4( )

δ=1.22 mm/s and ε_Q=2.62 mm/s, with A=0.04

(d) Fe(III) ( )

δ=0.49mm/s, ε_Q=0.83mm/s, with A=0.02

Summary

1-Influence of Surface preparation

a) Ground and polished sample showed only α-FeSi

b) After etching this sample in $HF+H_2O_2$, Fe^{3+} formed

c) After annealed in vacuum fayalite Fe_2SiO_2 and $Fe^{3+}$are formed.

d) Oxide and fayalite phases were confirmed by XPS measurement.

2-Annealing in Oxygen

a) In the temperature range 500 ^oC to 780 ^oC exhibiting the formation of hematite α-Fe_2O_3 on the surface of the samples.

b) The relative concentration of hematite increases with increasing temperature.

3-Annealing in Argon

a) Annealing in Ar at 500 ^oC caused the formation of magnetite, fayalite Fe_2SiO_4 and Fe^{3+}.

b) Annealing at 600 ^oC caused the formation of magnetite, Fe with 0 Si in nn and fayalite on the surface.

c) The relative concentration of magnetite ate 500 ^oC is higher than that at 600 ^oC.

Acknowledgements

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Thankyou