Synchrotron Radiation Research to control scale and rust layer in steel surface

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SR applied Research Targets

Field	Part	Specimen	Method (Aim)	Section
Steel	Surface scale Layer (practical process)	Steel plate	*In heating* XRD (structure analysis on heating process)	Analysis of Surface scale layer –part1
		Steel plate surface	XAFS (local structure/chemical state analysis) XRD (structure analysis)	Analysis of Surface scale layer –part2
	Surface rust layer (formation mechanism)	Synthesized Metal Oxide	XAFS (local structure/chemical state analysis)	Analysis of rusts –part1 & 2
	Nano cluster in micro alloy	Steel plate	XAFS (microstructure analysis) SAXS (cluster size analysis)	
Car	Crushable body	Porous Al block	X-ray Radiography & CT (image analysis for foam size, strain, 3D-structure)	
Electronics	Circuit material	Thin layer (Cu alloy, Al alloy, Ag alloy)	*In plane* XRD (Residual stress analysis) XAFS (microstructure analysis) DS-XAFS (microstructure analysis for surface details)	
Environment	Soil	Soil contained heavy metal artificially	XAFS (Chemical state analysis)	
other	Medical Building structure Hard coating layer		XAFS (microstructure analysis) XRD (structure analysis) *In plane* XRD (Residual stress analysis)	

Approaches for study

- Surface scale layer
  - Specimens: High temperature scale layer
    - Iron oxide scale layer on the surface of Si-contained steels during heating process
  - Methods and Aims
    - *In heating* XRD (direct observation of scale formation on heating process)
    - XAFS (local structure/chemical state analysis)
    - XRD (structure analysis to certify the existence of fayalite)
- Surface rust layer
  - Specimens: Artificially-synthesized rusts
    - Fe oxide rusts contained an alloying element (Al, Ti, Co, Ni)
    - Zn oxide rusts contained an alloying element (Al, Fe, Co, Ni)
    - Zn oxide rusts made from $ZnSO_4$ solution and iron compound species
  - Methods and Aims
    - XAFS (local structure/chemical state analysis)

- Final Purpose
  - Analyzing rusts and scales on practical processes
  - Finding scales and rusts suitable as material
  - Decision of the treating condition/process of scales and rusts

Study of the microstructure in scale layer on steel surface

- Task
  - To clarify process parameters for producing steel to form the easier spalled scale layer

Pic.1 Hot rolling process

- Problem
  - Scales are consisted of lamellar metal oxides
  - Each scale layer is a mixture of different metal oxides

- Aim
  - To establish evaluation methods for scale study with SR technique
  - To elucidate microstructures and formation processes of scale on steel

Analysis of surface scale layer -part1

- Structure analysis of surface scale layer on Si-contained steel : *in heating* XRD (transmission mode, BL16B2)
  - Results
    - Fayalite (Fe_2SiO_4) forms over 1123K in oxidation atmosphere on the surface of 3wt% silicon contained steel
    - The adding silicon affects the phase transition point, the grain growth behavior and the formation of metal oxide species

Fig.1-1 *In heating* XRD setting

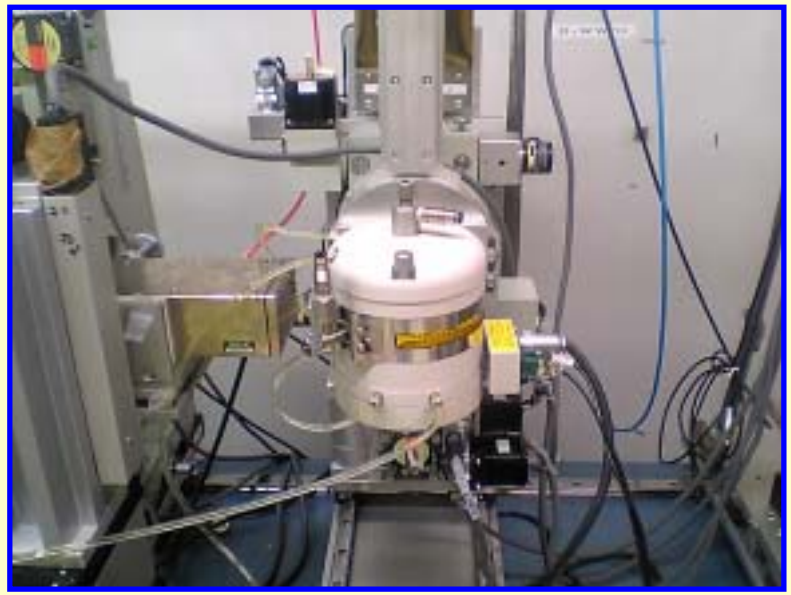
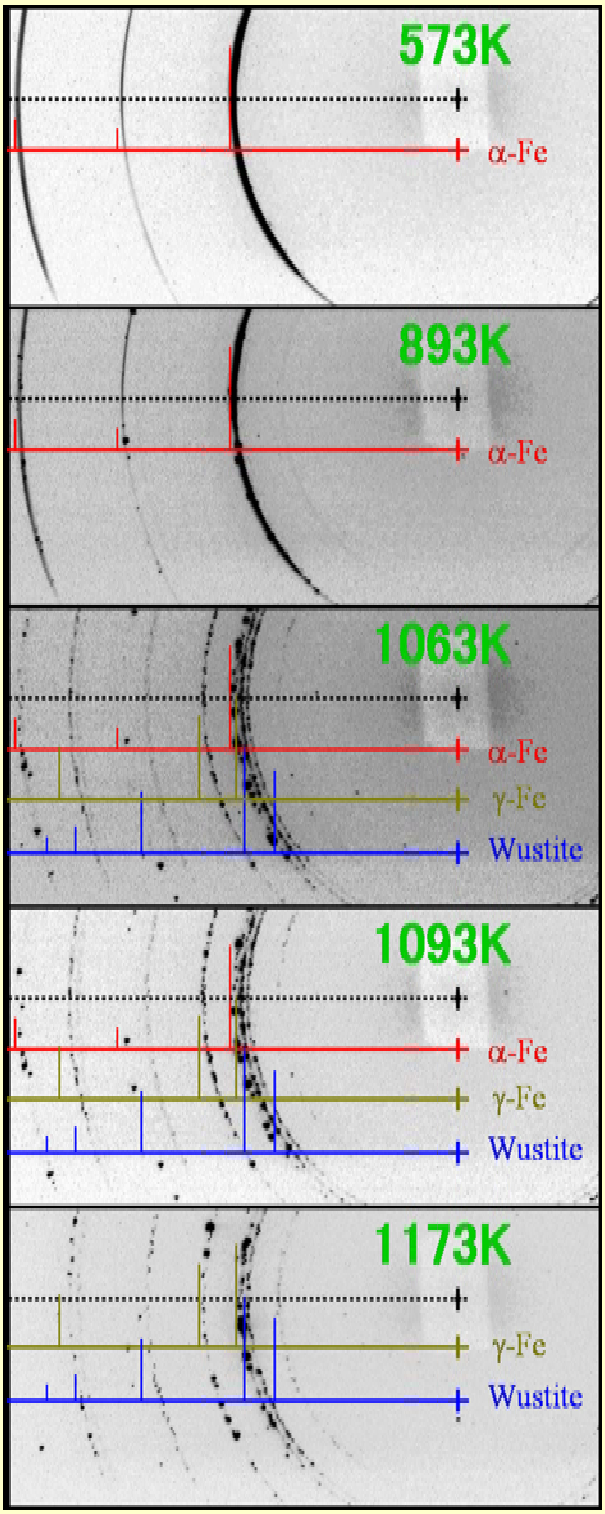
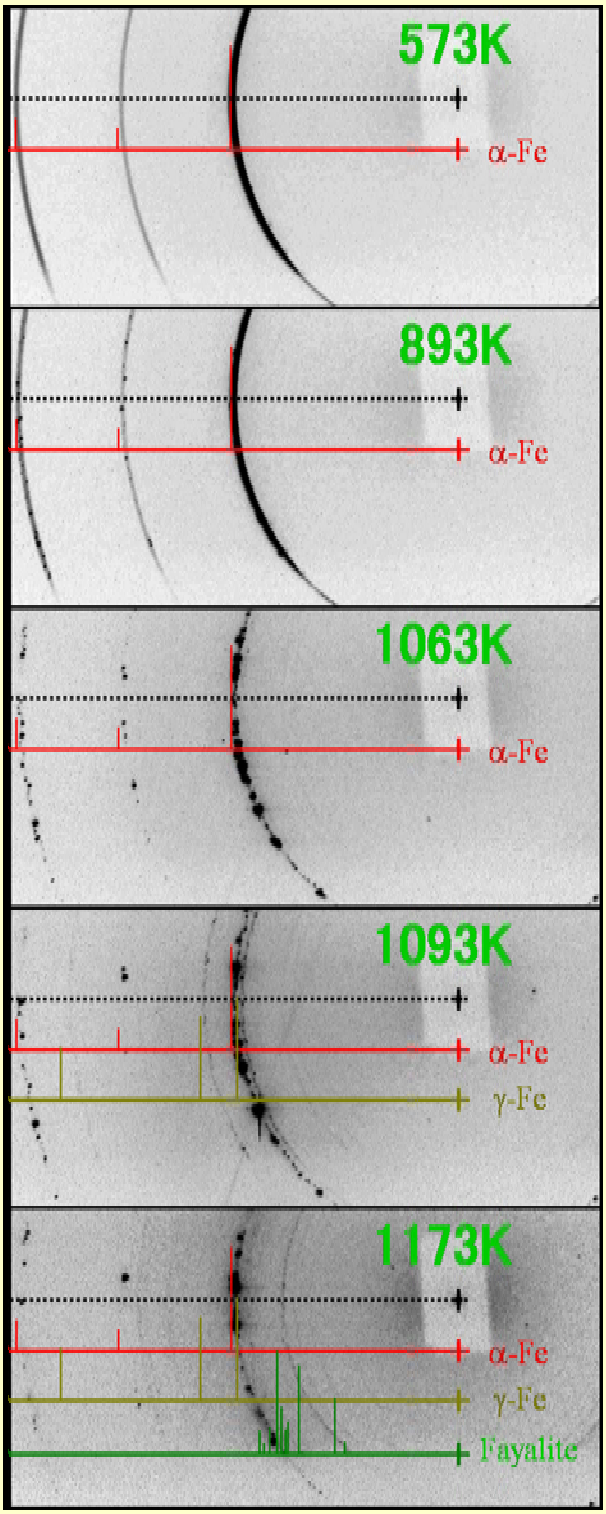


Fig.1-2 *In heating* XRD setting (a heating furnace)



(a)



(b)

Fig.1-3 X-ray diffraction patterns during heating process
(a) Silicon free steel, (b) 3wt% silicon contained steel

Analysis of surface scale layer -part2

- Structural analysis of surface scale layer on Si-contained steel : XAFS(BL16B2), XRD(BL16XU)
  - Results
    - Mixing ratio of iron oxide compounds (i.e. Fe oxidation state) was varied by annealed temperatures and Si content.
    - Thickness of fayalite (Fe_2SiO_4) layer increased with increasing Si content.

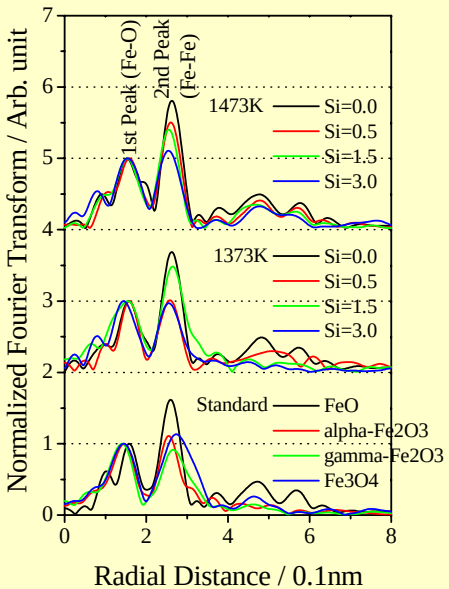


Fig.2-1 RDF of scale samples annealed at 1374K/1474K and standard iron oxide

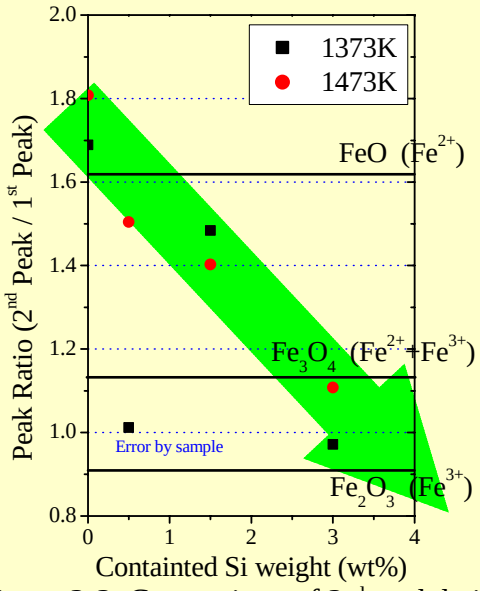


Figure 2-2 Comparison of 2nd peak height changed by contained Si weight and annealed temperatures.

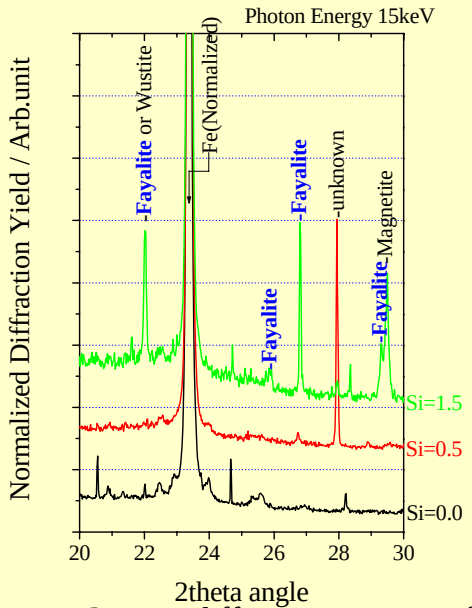
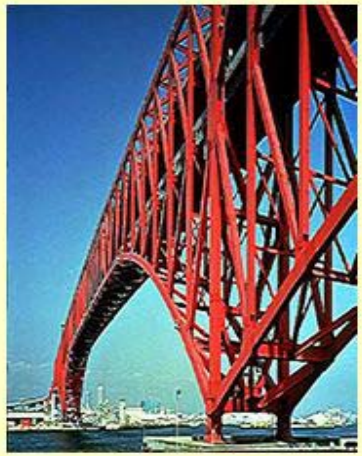


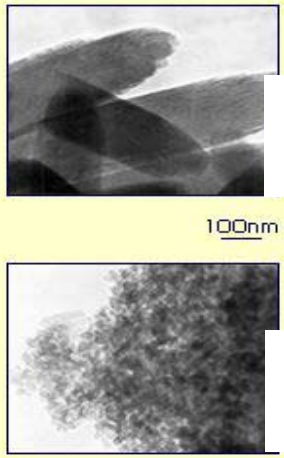
Fig.2-3 X-ray diffraction patterns of scale samples annealed at 1473K

Study of the microstructure in rust layer on steel surface

- Task
  - Improving atmospheric corrosion resistance of steel by making rust layer dense.



Pic.2 example of using high-corrosion resistant steel



Pic.3 Comparison of rust grain size

- Problem
  - Information of rust layer on steel surface obtained from conventional analytical tools is limited.

- Aim
  - To establish XAFS technique for rust study
  - To elucidate microstructures and formation processes of rust on steel.

Analysis of rust -part1

- Microstructure analysis of Zn oxide rusts contained a metal element (Fe, Co, Ni, Al) :XAFS (BL16B2)
  - Results
    - Zn oxide rusts transform from ZnO to ZHC ($Zn_5(OH)_8Cl_2 \cdot$ 5w:Simonkolleite) according to element content.
    - Zn oxide become amorphous by Zn-Zn network breaking in ZnO structure.
    - Zinc oxide structure was affected by alloying element species and content

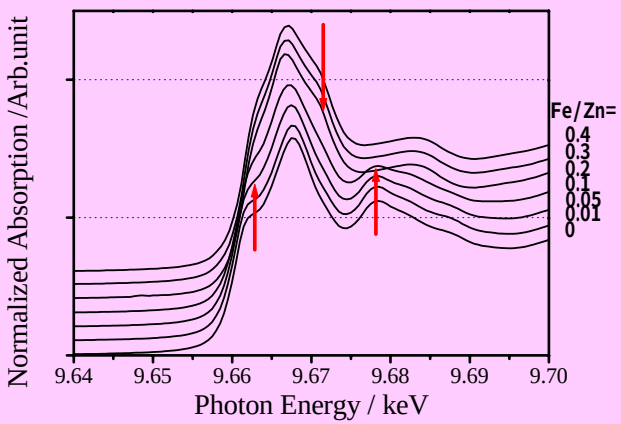


Fig.3-1 XAFS spectra of Fe-contained Zn oxides

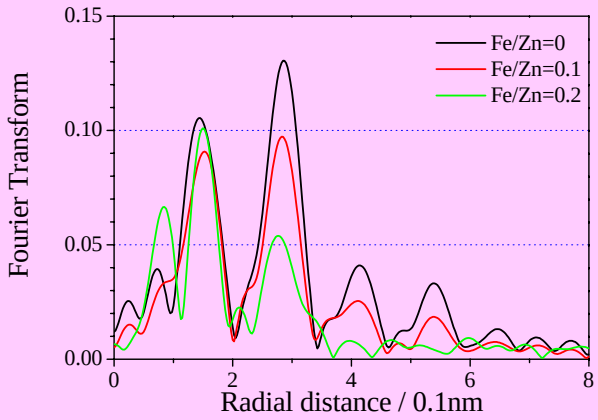


Fig.3-2 RDF of Fe-contained Zn oxides

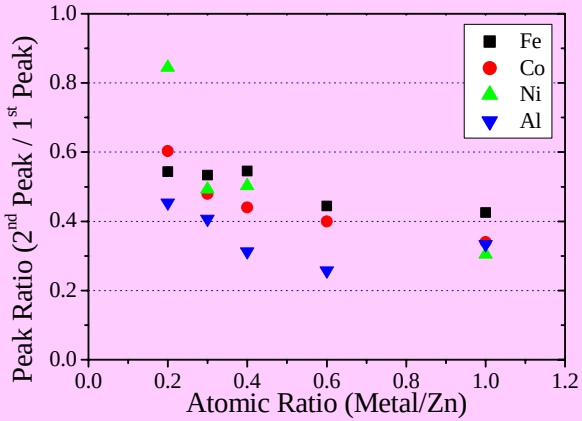


Fig.3-3 Comparison of peak ratio changed by added elements and quantities.

Analysis of rust -part2

- Microstructural analysis of Zn oxide rusts made from $ZnSO_4$ solution and iron compound species such as $FeCl_2$, $FeCl_3$* or $Fe_2(SO_4)_3$:XAFS (BL16B2)

  * To simulate environment in seaside area, Cl compounds were adopted as samples.

  – Results

    - Zinc oxide rusts consist of mixture of Zinc oxide compounds such as ZnO, $ZnFe_2O_4$ and Zn(OH)Cl
    - Mixture ratio of Zinc oxide compounds was changed with iron compound species and amounts of iron.

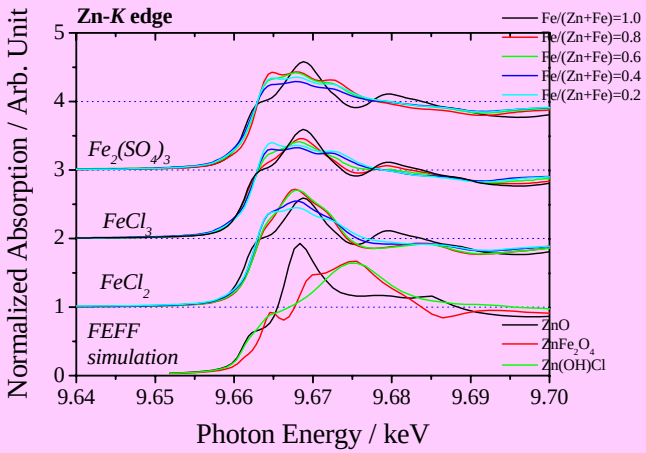


Fig.4-1 Zn K-edge XANES of Zinc oxide rust in response to iron compound addition

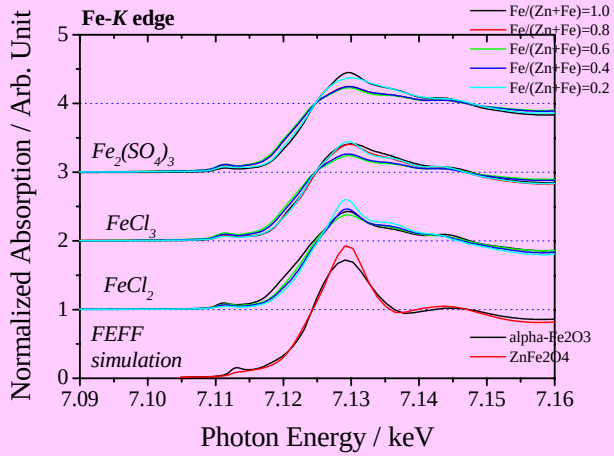


Fig.4-2 Fe K-edge XANES of Zinc oxide rust in response to iron compound addition