Chemical state mapping of barrier coating using newly-developed XAFS-CT

KEK-PF
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JFCC
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Overview

• Introduction
  • EBC material: Yb–Si–O
  • Our approach
• XRD investigation of
  • Yb–Si–O in fabrication process
  • Yb–Si–O after oxygen permeation experiment
• XAFS-CT investigation of
  • Yb–Si–O after oxygen permeation experiment
• Conclusion
Coating material for turbine engine blades

- Thermal barrier coating (TBC) to environmental barrier coating (EBC)
- Environment: both thermal and chemical
- $Yb_2Si_2O_7$ is a candidate material for top coat of EBCs.
- More information about chemical reactions both in processing and practical use is mandatory.

Microstructures of TBCs (YSZ/CoNiCrAlY/Ni superalloy)

Our approach: X-ray based measurements

X-ray absorption fine structure (XAFS)
X-ray diffraction (XRD)
X-ray semi-microprobe
Lab-source X-CT
Synchrotron-based CT
Fabrication process of Yb–Si–O

- Sample fabricated by JFCC
- Controlling formation of $\text{Yb}_2\text{Si}_2\text{O}_7$ and $\text{Yb}_2\text{SiO}_5$
- Synchrotron-based XRD investigation

1. Spray decomposition of $\text{Yb}_2\text{O}_3$ and $\text{SiO}_2$
2. Sintering @1300 °C
3. Jet mill grinding

Further sintering etc.

N. A. Toropov (1961).
Rietveld analysis of Yb–Si–O “cond. 2→3”

- Data was well fitted by using only Yb$_2$Si$_2$O$_7$ phase.
- Rwp = 1.852%, Rp = 1.353%
- Yb$_2$SiO$_5$ amount <0.01 wt% (estimation from background statistics)
Crystal structure of Yb$_2$Si$_2$O$_7$

- Phase 1: Yb$_2$Si$_2$O$_7$ (Space group: C12/m1 (12))

<table>
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<th></th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>α (deg.)</th>
<th>β (deg.)</th>
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<td>O1/O (最適化前)</td>
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<td>a 0.218261 b 0.000000 c 0.410652</td>
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<td>O2/O “条件2→3”</td>
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<td>Yb1/Yb（最適化前）</td>
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BL-15A1:
Semi-microbeam XAFS/XRF/XRD

- 20 µm, 2.1–15 keV X-ray probe + sample scanning stage
- Ion chamber (trans.), SDD (fluo.), PILATUS 100k (XRD)
- Macro-scale mapping of chemical/structural properties

NW2A has been operating as a “time-resolved XAFS” station.
We expanded the experimental hutch to introduce the CT system based on the design by Carl Zeiss X-ray Microscopy (former Xradia).
XAFS-CT @PF-AR NW2A

- Energy range: 5-11 keV, from an undulator source in 6.5 GeV SR
- Fresnel zone plate projects the image on the scintillator.
- Visible-light optics magnifies the image onto the CCD.
- Spatial resolution: <50 nm, FOV: 20 or 40 µm
“Spectromicroscopy” analysis of Yb–Si–O

- XANES analysis (background subtraction, normalize...) to all “voxels”
- Inspection to local XANES spectra
- ROI classification from edge jump and background
- Yb$_2$SiO$_5$ vs. Yb$_2$Si$_2$O$_7$
Conclusion

- We performed XRD experiments on Yb–Si–O EBC material
  - In fabrication process
  - After oxygen permeation experiments and succeeded to detect ~0.1 wt% Yb$_2$SiO$_5$ phase.

- We tried XAFS-CT investigation on Yb–Si–O.
- X-ray spectromicroscopy analysis in 3D, ~50 nm resolution was successfully performed.
- We found micropores, ~100 nm layer and sparse grain distribution of Yb$_2$SiO$_5$ in Yb$_2$Si$_2$O$_7$ specimen.