Industrial Characterization Challenges for Neutron Diffraction

With emphasis on "transformation"

Yan Gao
GE Global Research, Niskayuna, NY 12309

A presentation at the workshop of "Neutron Stress, Texture, Transformation – NST^2"
April 19. 2007, Spallation Neutron Source, Oak Ridge National Laboratory

GE Global Research is an active user of national facility

- Neutron (NCNR, IPNS, FRM-II): powder diffraction, single-crystal diffraction, residual stress, prompt γ-ray activation analysis (PGAA).
- Synchrotron (NSLS, APS): powder diffraction (high-resolution, high-energy, in-situ, grazing incidence), small-angle scattering (SAXS, USAXS, SAXS/WAXD), x-ray absorption fine structure (XANES and EXAFS), microdiffraction, residual stress, plastic deformation (peak broadening) radiography, tomography, and topography.
- Materials/systems: alloys for turbines, catalysis, hydrogen storage materials, solid oxide fuel cells (SOFC), thermal barrier coatings (TBC), nanoceramics, etc.

Our approach to materials characterization

No matter if it is a white cat or a black cat; as long as it can catch mice, it is a good cat.

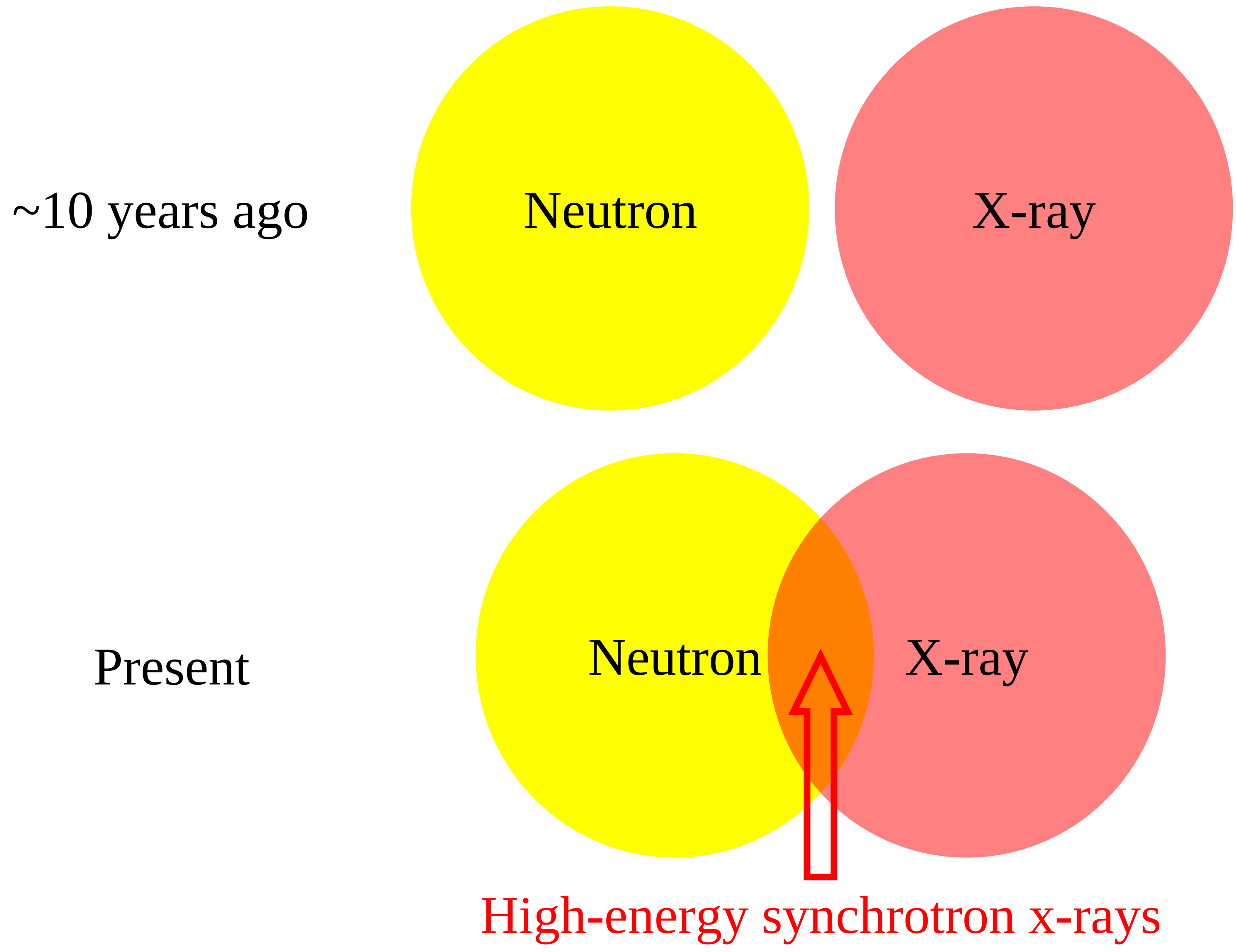
- Chinese proverb

- We focus on problem-solving and use any techniques that we see fit.
- We deal with a variety of materials characterization issues; x-ray and neutron are primary tools for understanding microstructures.
- We expect novel experimental opportunities from the *Vulcan Engineering Diffractometer*: high spatial resolution, high-throughput, and various *in-situ* and time-resolved measurement capabilities.

Characterization challenges for the future Vulcan

- ❑ Selected characterization cases for Vulcan applications
  - Phase transformation in Ni-base superalloy
  - Dislocation density analysis for alloys
  - Thermal barrier coatings (TBCs)
  - High-performance titanium alloys
  - Chemistry of Na-$NiCl_2$ battery
- ❑ Thoughts on future industrial uses of Vulcan

Comparison of neutron and x-rays (from a user's perspective)



Comparison of neutron and high-energy synchrotron x-rays (HEX)

- Neutron has greater penetration depth, is good for bulk measurement, and better suited for large-grain alloys
- HEX has smaller beam size, is good at spatial resolution, and better suited for surface and sub-surface (such as shot-peened) residual stress measurement.
- Neutron and HEX have comparable angular resolution for peak shape analysis and residual stress measurements
- HEX has greater flux/intensity and thus higher data collection speed
- Neutron can accommodate larger apparatus and is ideal for industrial type real-world samples measured under various external conditions
- Neutron is more sensitive to lighter elements (such as O and H), can better differentiate similar elements (such as Fe and Co), and can measure both nuclear and magnetic scatterings; but isotope substitution (e.g. for H and B) may be necessary for some materials.
- HEX is better suited for minute sample material, detecting trace phases, and following fast chemical processes and crystal structure changes
- While x-ray has no impact on the samples, ueutron can leave some sample (such as Co-containing materials) “hot”.

Analysis of dislocation density for alloys

Why Vulcan?

- In-house x-ray analysis is limited to the surface.
- High-energy synchrotron x-rays (in transmission mode) is still limited to samples of a few mm in thickness; not able to measure thicker samples with complex geometry, whereas neutron has greater penetration depth.
- X-ray is unable to handle alloys of large grain size, whereas neutron with larger beam size can handle those samples better.
- Vulcan appears to have adequate angular resolution for obtaining accurate peak-shape profiles.
- In-situ measurements at elevated temperatures and under load become feasible.

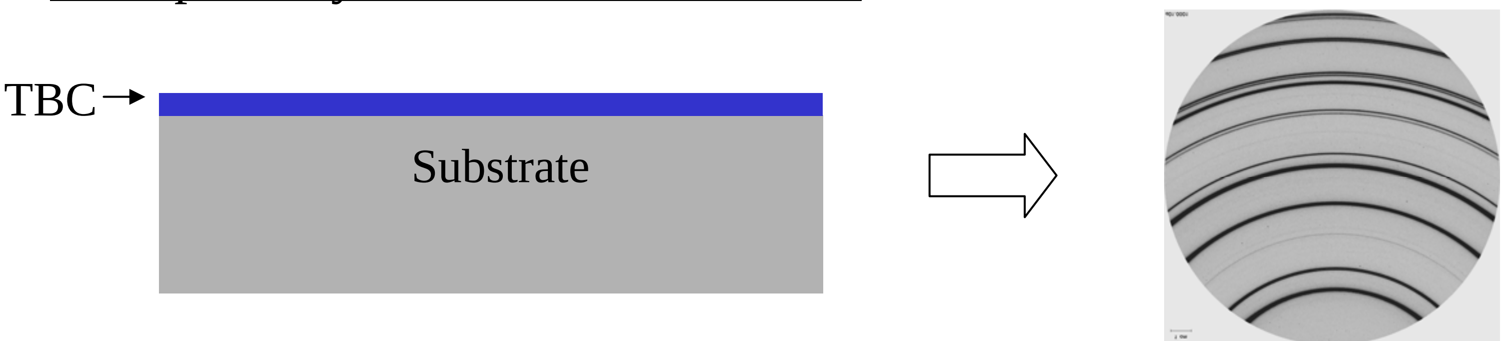
Ni-base superalloy: other issues/questions

- The most appealing aspect at Vulcan seems to be its *in-situ* capability under high-temperature and loading conditions. How do we deal with some testings, such as creep, that requires longer testing time than being practical at a beamline?
- What if the samples become "hot" after neutron irradiation that may prevent continued testing on the same samples, since most laboratory-based testing equipment is not setup to handle radioactive materials? Will Vulcan have some "on-site" mechanical testing equipment that can handle radioactive samples?
- Will 3-D mapping feasible for actual component (like turbine blade) for which location-specific microstructure evolutions (e.g. from tip and root) during processing or service may be of interest?

Thermal barrier coatings: diffraction analysis

- In-house XRD: limited to simple geometry and surface layer
- High-energy synchrotron: transmission measurement is able to obtain diffraction patterns with great spatial resolution and depth-dependence, but it’s destructive.

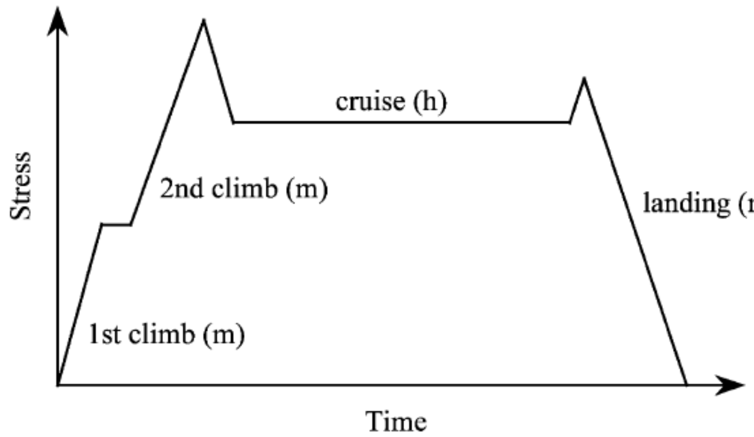
Example of synchrotron measurement



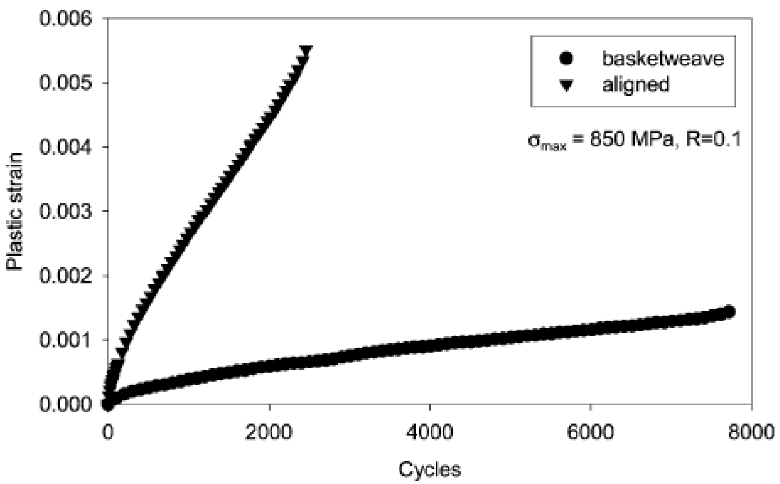
Why Vulcan?

- Non-destructive: able to measure TBC on turbine blade.
- In-situ: apparatus similar to the lab test jig may be built to study the YSZ crystal structures and phase transformation under similar to operation conditions (with temperature gradient and heating/cooling cycles)
- Neutron is more sensitive to oxygen and may be more accurate in phase composition analysis than x-rays.

Dwell fatigue vs. microstructure for titanium alloys



(M.R. Bache, 2003)



(M.R. Bache, 2003)

This complex material problem involves texture (orientation of HCP basal planes), dislocation, fatigue (esp. dwell fatigue), stress and strain life and external loading may constitute an interesting, challenging, and important material systems for Vulcan.

Thoughts on future industrial uses of Vulcan

- Rapid access: since industrial R&D runs on annual budge, rapid access (not 6-month), from submitting proposal to completion of measurement, is very important for industrial type problem-solving applications.
- Productivity: being able to measure multiple samples in a single trip (a few days) is essential for many industrial problems – can Vulcan provide a high throughput mode for "routine" measurements?
- Science vs. technology oriented proposals: as industrial problems are technologically important (real parts, more complex), they should not be rated just based on their scientific merit; can we have "double standard"or a kind of "affirmative action" for "minority" industrial uses?
- Will Vulcan provide "mail-in" service for simple measurements, like what APS (11-BM) does for high-resolution powder diffraction?
- The pros and cons of a "compound" instrument: does Vulcan want to do "everything", or do a few things superb unmatched by other neutron and synchrotron facilities? Frequent change of configuration has always been a concern for user facilities.

Acknowledgement

I'd like to thank my colleagues at GE for sharing their valuable thoughts and contributing examples of their work to make this presentation possible.

Global Research: Liang Jiang, Curt Johnson, Wayne Hasz, Job Rijssenbeek, Andy Deal, Ernie Hall

GE Energy: Ling Yang, Jeff Hawk, Warren Nelson

GE Aviation: Andy Woodfield, Rob Fecke, Ben Nagaraj