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A Hard X-ray Micro-analytical Beamline at the CAMD Synchrotron*

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ABSTRACT

Argonne National Laboratory (ANL) is collaborating with Louisiana State University (LSU) in constructing a synchrotron x-ray micro-analytical beamline at the Center for Advanced Microstructures and Devices (CAMD) in Baton Rouge. This project grew from earlier work at the National Synchrotron Light Source (NSLS), where a team of ANL researchers developed techniques to examine small-scale structures in diffusion zones of a variety of materials. The ANL/CAMD beamline will use x-ray fluorescence, diffraction, and absorption spectroscopy techniques to reveal both compositional and structural information on a microscopic scale.

Keywords: synchrotron x-ray micro-analytical beamline, scanning electron microscopy, energy-dispersive x-ray, x-ray fluorescence spectroscopy, diffraction spectroscopy, absorption spectroscopy

1. NSLS STUDIES

Understanding chemical interactions at the interface between dissimilar materials is a major concern for many scientific and industrial applications and has been an important focus of research for ANL. For example, to better understand compatibility of metallic nuclear fuel and stainless steel cladding, detailed multi-component, isothermal interdiffusion experiments have been performed. In those studies, scanning electron microscopy (SEM) using energy-dispersive x-ray (EDX) analysis was used to measure composition profiles across the diffusion zones from which kinetic information was calculated. Intermetallic phase identification was not possible with these techniques, however. Moreover, conventional crystallographic techniques (such as standard x-ray diffractometry) could not resolve the micron-sized phase layers in the diffusion zones. Similarly, transmission electron microscopy would have involved formidable sample preparation difficulties.

The high x-ray fluxes associated with synchrotron radiation sources, on the other hand, make them uniquely suited for studying small phases because x-ray intensities are still significant even after substantial beam collimation. Synchrotron radiation can be used to reveal both composition (by fluorescence) and structure (by diffraction). To assess the feasibility of examining material interfaces using synchrotron radiation sources, x-ray diffractometry of diffusion couples was performed at the NSLS. Beam collimation allowed narrow regions of the interdiffusion zone to be isolated so that diffraction patterns from only a few phases were collected simultaneously.

As an example, Ref. 1 reported on NSLS diffractometry studies using a diffusion couple between a binary U-Zr fuel alloy and a Ni-Cr alloy. Figure 1 shows the SEM/EDX results. The diffusion zone was approximately 100 μm thick with nine distinct layers. Five of those layers consisted of a mixture of two phases; four of the regions were less than 10 μm wide.

X-ray diffraction scans were performed on synchrotron lines X-7A and X-14 at the NSLS. The x-rays were collimated by slits to produce an incident beam as small as 1 mm by 18 μm, as shown in Fig. 2. Motorized stages were used to translate the position of the slits with respect to the sample. In this way the intermetallic phases could be studied by repositioning the slits to move the incident beam across the diffusion zone. By collimating the x-rays, only a few phases were simultaneously within the beam, easing identification of the phases. Strains in the lattice due to solid solution were also observed.

In a separate set of studies at NSLS beamline X-18B, diffusion couples were examined using x-ray fluorescence.[2] Phase zone plates were used to focus the synchrotron x-rays to spots as small as 3 μm by 5 μm. Fluorescence x-ray signals were collected using a Si(Li) detector and a multi-channel analyzer. By stepping the sample through the x-ray spot, changes in the fluorescence signals could be monitored as a function of position. Furthermore, an image of the diffusion zone microstructure could be generated by scanning the sample in two dimensions.

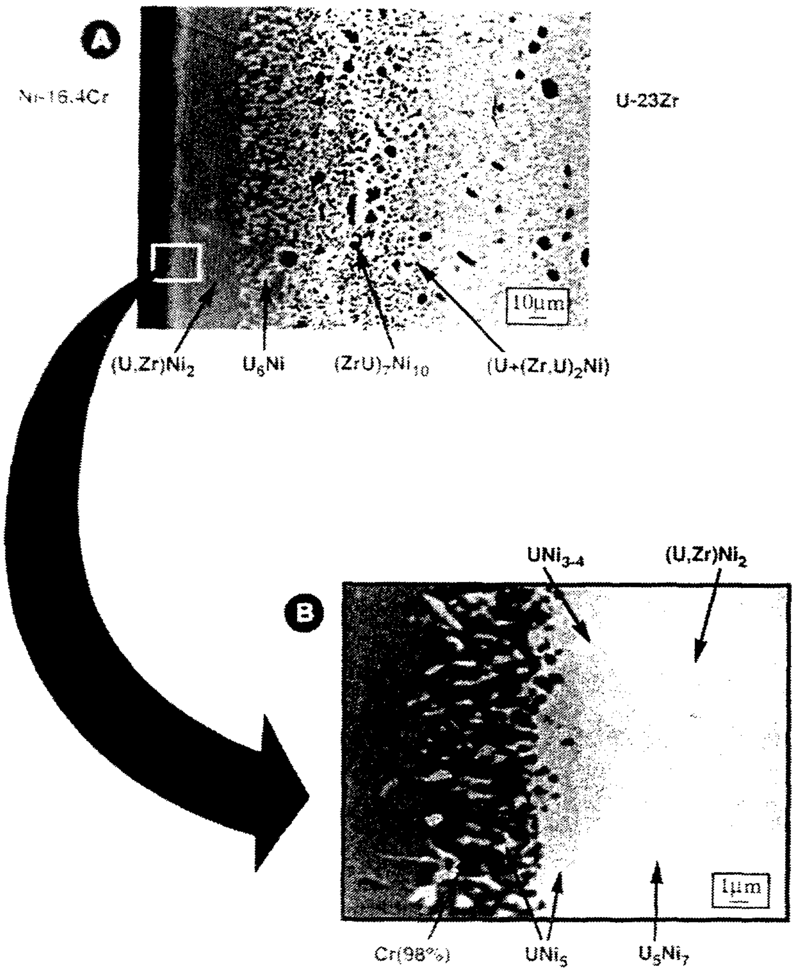

Fig. 1 SEM micrographs of Ni-16.4Cr/U-23Zr diffusion couple after four days at 700°C. (a) Overall view of diffusion zone, (b) high-magnification view of diffusion zone edge near Ni-16.4Cr alloy. *Adapted from the paper, "Interdiffusion between U-Zr fuel and selected Fe-Ni-Cr alloys," by D. D. Keiser and M. A. Dayananda, J. Nucl. Mat., Vol. 200, 1993, pp. 229-243, with kind permission from Elsevier Science B. V, Amsterdam, The Netherlands.*

For example, Fig. 3 gives fluorescence maps of Cr and Ni for the U-Zr/Ni-Cr diffusion couple, which used a 2 μm step size. The Cr map shows a build-up of Cr near the leading edge of the diffusion zone, consistent with the SEM/EDX results (Fig. 1). In contrast, the Ni map shows a depletion of Ni in the same region. Two-phase regions are also visible in the Ni map.

The studies at the NSLS demonstrated the feasibility of examining small-scale structures using x-ray diffraction and fluorescence techniques at a synchrotron light source. Three NSLS beamlines were used since no single beamline allowed simultaneous diffraction and fluorescence experiments. One objective, then, of the ANL/CAMD

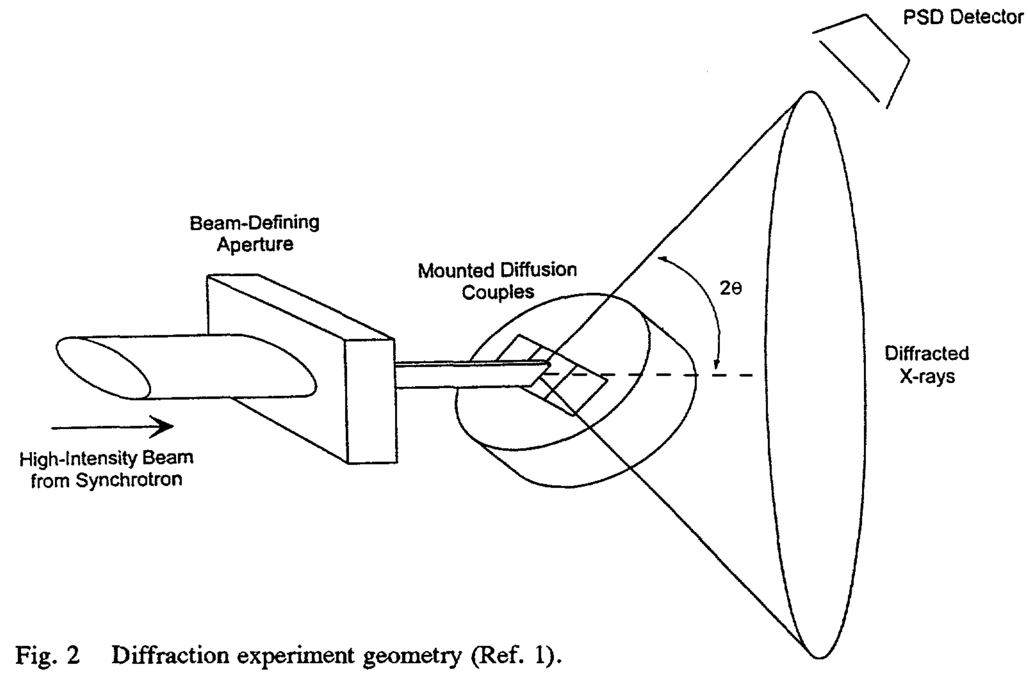

Fig. 2 Diffraction experiment geometry (Ref. 1).

micro-analytical beamline is to provide a single facility with small-beam capabilities for fluorescence (for chemical analysis), diffraction (for structure determination), and x-ray absorption spectroscopy (XAS) (for local element-specific coordination information).

2. THE CAMD FACILITY

CAMD, a state-owned synchrotron radiation facility that began operations in 1992, currently operates at 1.5 GeV with a critical energy of 2.5 keV. The electron storage ring includes eight bending magnets, allowing sixteen beam-line ports. Up to three insertion devices such as wigglers and undulators can be installed in straight sections between the magnets to produce higher-energy x-rays.

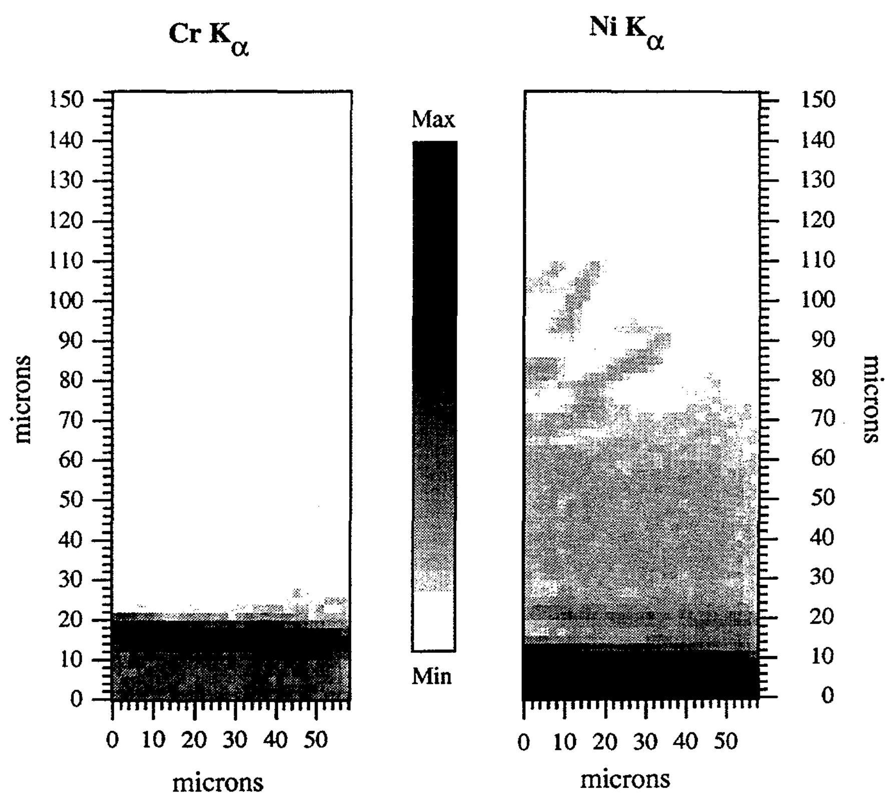

Fig. 3 Fluorescence mapping of Ni-16.4Cr/U-23Zr diffusion couple after four days at 700°C.

Currently, eleven beamlines are operating at CAMD or are near completion. The majority of these beamlines are devoted to micro-machining, lithography, and soft x-ray analysis of molecules, surfaces, and solids. Research programs at CAMD, unlike those at the national user facilities, are built upon long-term partnerships aimed at solving complex technological problems.

3. THE ANL/CAMD BEAMLINE

When completed, the ANL/CAMD beamline will provide an ideal tool for studying the composition and structure of materials on a microscopic scale using x-ray diffraction, fluorescence, and XAS techniques. The beamline includes a large x-ray isolation room and a kinematic vibration-control table. A unique four-circle goniometer with an X-Y-Z translation stage with 1-μm stepping motors allows samples to be manipulated and scanned in many orientations.

The micro-analytical beamline is currently available for x-ray fluorescence studies using a collimated beam of white x-rays. An intrinsic germanium detector is used to collect characteristic x-rays from the sample. This detector has a 12-inch-long by 1-inch-diameter snout that can be placed close to the sample to maximize collection efficiency. In addition, a video microscope with a long working distance monitors the orientation of the microstructure. Moreover, a pan-and-zoom video surveillance camera eases remote operation of the equipment.

During 1996 a fixed-exit, double-crystal monochromator will be added to tune the beam to a single x-ray wavelength. Focusing optics will also be added to intensify the x-ray flux. These upgrades will allow x-ray diffractometry and XAS for structural analyses. In a typical study, fluorescence at high energies can be used to locate specific microstructural features on a sample. The monochromator can then be tuned to a lower x-ray energy for diffraction and XAS of that feature.

The ANL/CAMD micro-analytical beamline will complement the capabilities of DOE user facilities such as Argonne's Intense Pulsed Neutron Source and Advanced Photon Source (APS). The power of APS, in particular, to produce high-energy, high-intensity x-rays is unmatched. Nevertheless, as a DOE user facility, APS provides only limited access to independent researchers. This is not the case with CAMD, which is owned by the State of Louisiana. The essentially unrestricted beam time available at CAMD will allow research that cannot be performed at APS. Examples would be screening large numbers of samples or providing fast turn-around times for analyses—features that are attractive to industrial users. Indeed, collaborations have already been established to perform non-destructive examinations of quality control samples for industry using the ANL/CAMD beamline.

Although certain studies require the higher energies and intensities of APS, preliminary work at CAMD (including testing optical components or verifying experimental techniques) would make a session at APS more productive. Moreover, the APS experiment review committee may look favorably on programs with prior synchrotron experience at CAMD.

4. ACKNOWLEDGMENTS

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5. REFERENCES

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