Upgrade of the X-ray Deffraction Facilities at the Detroit Institute of Arts | 2002-25

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The Detroit Institute of Arts

Upgrade of the X-ray Diffraction Facilities at the Detroit Institute of Arts

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Executive Summary

The upgrade of the Detroit Institute of Arts' x-ray diffraction (XRD) facilities supported by this grant, together with matching funding provided by the Detroit Institute of Arts, has continued to develop the Midwest's only fully equipped and staffed conservation science laboratory. The improvements made to the x-ray diffraction, x-ray fluorescence and microscope systems in our laboratory have enabled us to carry out analyses more efficiently, accurately and reliably, and our productivity has increased correspondingly (by at least 25%). Working in collaboration with conservators and curators, studies carried out by the scientific staff helped resolve questions of condition, technique and authenticity; supported the acquisition of works of art; determined the suitability of storage and display materials and guided the selection of conservation treatments.

The equipment provided for under this grant included a Nikon zoom stereomicroscope, which has greatly facilitated the handling and preparation of small samples for analysis. A film scanner and Jade™ computer-assisted data processing and pattern matching software has greatly improved the ease with which x-ray diffraction patterns acquired using our Gandolfi film camera system can be analyzed. Additionally, the installation of a computer interface for the Philips/Norelco vertical goniometer has allowed diffraction patterns obtained on this system to also be analyzed using the Jade™ software. Using these new capabilities, we have begun building a searchable, digitized database of x-ray powder diffraction patterns of pigments, minerals and corrosion products.

As a result of these upgrades and improvements, we are better able to conduct materials analysis and scientific research on the DIA's collections and well as those of the museums, historical societies, universities, churches, and private individuals we serve. During the grant period, collaborative research was conducted with colleagues at a number of other cultural institutions, including the Asian Art Museum of San Francisco, the Royal Ontario Museum, the Getty Conservation Institute, the Van Gogh Museum, the Toledo Museum of Art, the Indianapolis Museum of Art, the Winterthur Museum and the Henry Ford Museum & Greenfield Village. These collaborative efforts contributed to the fields of conservation and historic preservation by generating information that enhanced our understanding of the materials and methods in works of art. This information was disseminated through the production of two peer-reviewed publications and a number of presentations at professional meetings and conferences. Furthermore, the scientific staff continually works to educate the general public about the efforts of conservation and conservation science through public presentations and tours of the DIA's conservation laboratories.

I. Introduction

The Detroit Institute of Arts is one of only a few art museums in the country that have integrated scientific research into their conservation program. The scientific research laboratory performs research and analysis of works of art for the Detroit Institute of Arts and other cultural and artistic institutions throughout the country. Working in collaboration with conservators and curators, the scientific staff works to help resolve questions of condition, technique and authenticity; support the acquisition of works of art; determine appropriate storage and display materials; and guide the selection of conservation treatments.

The DIA's Conservation Department is organized into eight sections according to specialty: paintings, works of art on paper, objects, frames, textiles, scientific research, mount fabrication and technical photography. A two-year renovation of the conservation laboratories was completed in June 2001. The newly renovated laboratories are contiguous, a design which facilitates and encourages interaction between the various specialties. The scientific research laboratory has a large lead-lined room that houses the x-ray fluorescence (XRF) and x-ray diffraction (XRD) units (Figure 1a), ample counter space for sample preparation, microscopy and image processing (Figure 1b), and adequate ventilation provided for by a large hood and movable fume exhaust trunk.

The present technical facilities of the laboratory include: energy dispersive x-ray fluorescence spectroscopy, x-ray powder diffraction, cross-section analysis (ultramicrotomy, polarized and ultraviolet fluorescence microscopy, photomicrography), x-radiography, infrared reflectography and digital image processing. Collaboration with local academic and industrial laboratories provides access to additional analytical techniques, including scanning electron microscopy with energy dispersive x-ray analysis (SEM-EDS), Fourier transform infrared (FTIR) and Raman spectroscopies, electron microprobe, transmission electron microscopy and x-ray photoelectron spectroscopy (XPS).

With funding provided by the NCPTT through this grant, together with matching funding provided by the DIA, the laboratory's XRF, XRD and microscope facilities were improved and upgraded. A description of the equipment, software and other upgrades that were made under this grant will be presented in the Section II. The improvements have enabled us to carry out analyses more efficiently, accurately and reliably, thus improving our overall productivity.

In addition to carrying out analysis and research on objects from the DIA's collection, the scientific research laboratory also provides services for and collaborates with other institutions throughout Michigan, the U.S., Canada and Europe. During the grant period, collaborative research was conducted with colleagues at a number of other cultural institutions, including the Asian Art Museum of San Francisco, the Royal Ontario Museum, the Getty Conservation Institute, the Van Gogh Museum, the Toledo Museum of Art, the Indianapolis Museum of Art, and the Henry Ford Museum & Greenfield Village. The results of many of these collaborations were published in scholarly journals

and presented at professional meetings and conferences, which will be discussed in more detail in Section III.

II. Equipment Upgrades

a. Zoom stereomicroscope

A Nikon SMZ-800 zoom stereomicroscope (Figure 2) was purchased in January 2000. This microscope system included: an optical body with 1x-6.25x zoom range, weighted boom stand, focus mount, tilting binocular eyepiece, 10x widefield eyepiece, 1x plan achromat objective, beam splitter, photo adaptor and a Fostec illumination system with both gooseneck fiber optic and ring light (with polarizer) illuminators.

The superior optics of this microscope has greatly assisted in the examination of art objects, selection of sample sites, sampling of objects and preparation of samples for analysis. This microscope has proven particularly useful for the preparation of cross-sections: sections being prepared on the ultramicrotome can be easily examined to judge the progress of the sectioning, and furthermore, the polarizer can be used to reveal whether the layers have been completely exposed or whether areas still remain under the mounting medium. In order to perform elemental analysis on mounted cross-sections using an SEM-EDS, it is imperative that the areas of interest are completely exposed at the surface. The large magnification range of this microscope is also routinely used in the manipulation and mounting of very small powder samples for analysis using the Gandolfi camera system on the XRD. Another benefit of this microscope is that it accepts the photographic system from our Nikon Optiphot-2 ultraviolet fluorescence microscope, which enables us to obtain sharp and clear photomicrographs.

b. Digitization of film patterns

Our Philips Cu anode x-ray diffractometer is equipped with a Gandolfi camera for small powder samples. In April of 2000 the Jade™ computer-assisted data processing and pattern matching software system (Figure 3) was purchased from Materials Data Incorporated (MDI). At the same time, we purchased the current ICDD CD-ROM database, the printed index for organic and organometallic phases, and the Hanawalt search manual and alphabetical index for inorganic phases. The DIA has committed to purchasing annual renewals of the ICDD database to ensure that we have the most current information. To run the software, a computer system, flatbed transparency scanner and printer were also acquired.

Films obtained with the Gandolfi camera are scanned using Photoshop™ and the brightness and contrast adjusted, if necessary. The scanned image is read by the FilmScan™ software, which is used to convert the scanned image into a trace pattern (d-spacing versus intensity). The resulting trace pattern is imported into Jade™ 5.0 for pattern processing (background correction, smoothing, profile fitting, peak finding) and analysis using the search/match routines. The pattern matching software can be used either with the ICDD database or a user-generated database.

c. Upgrade XRD x-ray generator

Due to space and safety constraints, the XRF and XRD systems were placed in storage during the renovation of the conservation laboratories (October 1999 to June 2001). Following the reinstallation of the XRD unit it was discovered that the high-voltage generator no longer functioned properly. The very old wiring in the system may have been unable to stand up to having been moved. Several attempts were made to repair the unit, but unfortunately all were unsuccessful. The DIA provided funding to purchase a new generator (Philips Analytical X-Ray Generator PW3830), which was installed in May 2002 (Figure 4). The new generator provides a stable, reliable x-ray output and should last for many years.

d. Computer interface to Philips/Norelco vertical goniometer

X-ray diffraction patterns of larger samples can be obtained using the Philips/Norelco vertical goniometer attachment to our Philips XRD unit. This grant provided funding to obtain the necessary hardware and software to direct the output of this unit to a computer (Figure 5). In August 2002, we received and installed a stepper motor controller, data logger and DataScan^{TM} software package from MDI that enables us to control the goniometer and collect data via computer. The diffraction data thus obtained can be processed and analyzed using the Jade^{TM} software in the same manner as discussed above for the film data. Another benefit of automating the goniometer output is that all our XRD patterns are now produced in the same format. This will enable us to make direct comparisons between diffraction patterns obtained using either the Gandolfi camera or the goniometer, and incorporate data obtained from both systems into an in-house database. Additionally, the digitization of our XRD data enables us to more easily share information with colleagues at other institutions.

III. Products

The DIA's upgraded equipment was used to produce the following products during the grant period:

a. Characterization of aluminum corrosion products

The Dymaxion House is a unique aluminum housing structure designed by Buckminster Fuller and built in the 1940s. The house was donated to the Henry Ford Museum & Greenfield Village where it underwent extensive conservation, and the reconstructed house went on exhibit in October 2001. Working in collaboration with conservators from the Henry Ford Museum & Greenfield Village, we carried out studies into the characteristics of the aged aluminum alloys, in particular their composition, corrosion products, and metallographic structure.

The DIA's upgraded x-ray diffractometer was used to analyze white crystalline corrosion products found on many of the aluminum pieces of the house. The data revealed that the corrosion products were aluminum hydroxide, $Al(OH)_3$ (bayerite and possibly also small amounts of gibbsite and/or nordstrandite) together with pseudoboehmite, the immediate precursor to aluminum trihydroxides in the oxidation/hydration of aluminum. Other

studies carried out on the aluminum components of the house included: examination of the microstructure and age-hardening precipitates by transmission electron microscopy, and analysis of the cladding layers and grain structure via metallographic cross-section analysis.

The results of this study were presented (as an invited paper) at the Materials Issues in Art and Archaeology symposium held at the Material Research Society meeting, November 26-30, 2001. A paper on this study has also been published in the meeting's proceedings (Materials Research Society Symposium Proceedings, Vol. 712, p. II2.1-12, 2002). A copy of the paper is attached to this report.

b. Characterization of bronze corrosion products

An unusual blue corrosion product found on copper alloy artifacts at a number of institutions was characterized using x-ray diffraction, x-ray photoelectron spectroscopy (XPS), wavelength dispersive spectroscopy and Raman microspectroscopy. This work was carried out in collaboration with conservators and scientists from a number of institutions, including the Asian Art Museum of San Francisco, the Royal Ontario Museum, the Getty Conservation Institution, Ford Research Laboratories and Michigan State University.

The DIA's XRD was unavailable for use at the time of this study due to the laboratory renovation, and consequently the XRD studies were carried out at Ford Research Laboratories. However, the Jade™ software provided for by this grant was used to analyze the data and search the ICDD database for matches or related compounds. The data from the various studies indicate that the blue corrosion product is a copper (II) compound, containing copper and sodium in a ratio of approximately 1:1 together with bidentate formate and acetate groups bridging adjacent copper atoms. Although the XPS and Raman data were useful for elucidating the structural components of this compound, the XRD pattern provided a unique fingerprint by which the compound could be conclusively identified.

A paper describing the results of this study was submitted to *Studies in Conservation*, and in May 2002 was accepted for publication. The paper is presently in press, but a preprint is attached with this report. A reprint will be provided to NCPTT once the paper has been published.

c. Creation of XRD database

We are building a searchable, digitized database of XRD patterns of artists' pigments and minerals. The Detroit Institute of Arts is a repository of over 800 specimens from the Forbes pigment collection (Figure 6). We have already scanned over 500 film patterns into the database using the FilmScan™ and Jade™ software. With the recent addition of the computer interface for the goniometer and DataScan™ software, we will be able to add data collected on the goniometer to the database. When new XRD patterns are acquired, they can be searched against the ICDD database as well as the specialty database we are building. We will share this database with other institutions, and would

like to collaborate with them to incorporate data from their pigment collections as well in order to build as complete a database as possible.

IV. Conclusions

The funding provided by the NCPTT with this grant, together with matching funding provided by the Detroit Institute of Arts, has continued to develop the Midwest's only fully equipped and staffed conservation science laboratory. The laboratory uses a variety of analytical techniques to study works of art, the core techniques being microscopy, x-ray fluorescence and x-ray diffraction. Among these, XRD is unique in that it can provide a definitive identification of crystalline materials. Its usefulness as a fingerprint technique was demonstrated in our study of a blue corrosion product found on copper alloy artifacts. It was also the only technique by which we were able to determine that a mixture of corrosion products was present on the aging aluminum components of the Dymaxion House. The upgrades to the XRD system have enabled us to utilize this important technique more efficiently and fully. With the digitization of the diffraction patterns and creation of an in-house database, we are more easily able to analyze the data and share information with colleagues at other intuitions.

The scientific staff at the Detroit Institute of Arts is committed to contributing to the fields of conservation and historic preservation. We conduct educational and outreach activities, pursue in-depth research projects, present our findings at professional conferences and publish them in scholarly peer-reviewed journals. To supplement the analytical resources available in-house, we have established long-term collaborative research programs with local academic (Wayne State University, Michigan State University, University of Michigan, University of Detroit-Mercy) and industrial (General Motors Research and Development Center, BASF, Ford Motor Company Research Laboratory and Detroit Edison) scientists. We also continue to collaborate with colleagues at other cultural institutions including the Asian Art Museum of San Francisco, the Getty Conservation Institute, the Henry Ford Museum & Greenfield Village, the Indianapolis Museum of Art, the Royal Ontario Museum, the Toledo Museum of Art, the Van Gogh Museum, and the Winterthur Museum. These efforts towards understanding the materials and methods used in the creation of works of art will further enhance the abilities of conservators and other stewards of artistic and historic collections to preserve them.

Figure 1a: Lead-lined x-ray equipment room. Philips x-ray diffractometer (in back) and Kevex 0750A x-ray fluorescence spectrometer (in front).

Figure 1b: Scientific Research Laboratory (main room) showing digital image processing and cross-section preparation areas.

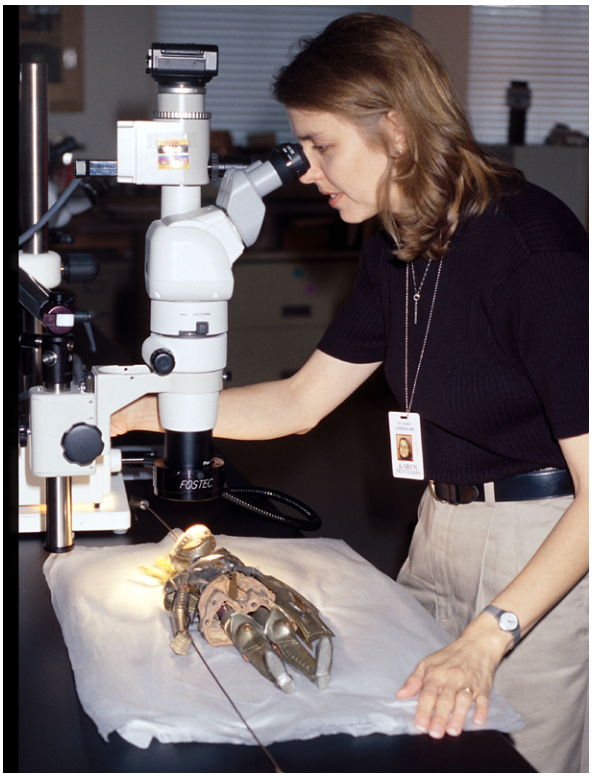

Figure 2: Microscopy. Top: Nikon SMZ-800 zoom stereomicroscope. Bottom: Microscope bench with (from left to right) Nikon SMZ-800, Zeiss Universal polarizing microscope and Nikon Optiphot-2 ultraviolet fluorescence microscope.

Figure 3: Scanning XRD film patterns (top) and analyzing XRD patterns using Jade™ computer-assisted data processing and pattern matching software (bottom).

Figure 4: Philips Analytical PW3830 X-ray generator

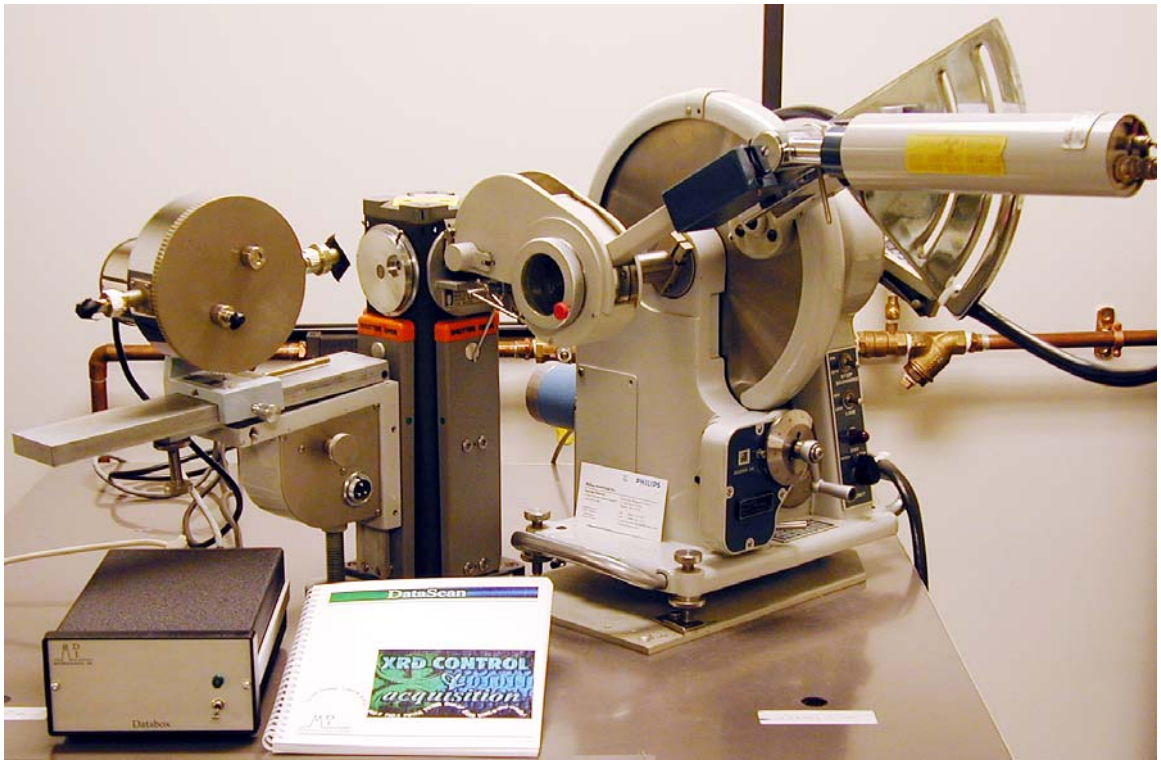

Figure 5: Philips/Norelco vertical goniometer with automated scanning hardware (Databox) and data acquisition software (DataScan^{TM}).

Figure 6: A portion of the Forbes pigment collection housed at the Detroit Institute of Arts. The XRD patterns from over 500 samples from this collection form the foundation of a database of XRD patterns of artistically important materials.