rc-Mirrors-Tests to do

3.3 X-radiography

X-radiography was used to help assess the condition of the mirrors and to see if any information could be

gained about their manufacture. A LORAD mobile x-ray unit was used with a 3mm aluminum filter.

The X-rays passed through the mirrors and were recorded on Kodak MX125 14” x 17” film in a cassette

sandwiched between a 0.005 (top) and 0.010 (bottom) mil lead filters. The operating parameters were

160kV and 5mA for 3-8 minutes depending on the thickness of the mirror. The film was placed

approximately 36 inches from the radiography unit.

3.4 Energy Dispersive X-ray Fluorescence (XRF)

XRF was used to characterize the elements present on the surface of the mirrors and restoration materials

including solder and false corrosion products. An ArtTAX spectrometer with a molybdenum x-ray tube

was used, and the collected data was analyzed with Roëntec software. The mirrors were placed

approximately 1mm from the source and irradiated for 150s at 50kV and 600μA. The size of the area

irradiated was approximately 70 microns.

3.5 Scanning Electron Microscopy (SEM)

SEM was used to examine metal cross-sections from two mirrors to obtain detailed information on the

distribution of the metal phases and the extent of corrosion. Samples were run at The Museum of Fine

Arts, Boston on a JEOL JSM-6460LV SEM instrument at 20kV in low vacuum mode at 35 Pascal.

3.6 Fourier Transform Infrared Analysis (FTIR)

FTIR was used to analyze various adhesives, both original and restoration, and some false corrosion

products. A Nicolet 510 instrument coupled to a Spectra-tech IR-plan infrared microscope with a 32x

objective was used for the FTIR analysis. The sample was compressed onto a diamond cell (2mm x 2mm)

with a stainless steel roller and the sample area defined by double apertures contained in the microscope.

An absorbance spectrum (4000-500 wavenumbers) was measured (resolution setting 8cm-1) and

subtracted against a blank background. The spectrum was compared with a database of artist’s materials

at the Straus Center for Conservation.

3.7 Raman Spectroscopy

Raman spectroscopy was used to identify the pigments used in false corrosion products. Samples were

run at both the Massachusetts Institute of Technology and at Harvard University. A Kaiser Optical

Systems Hololab 5000R Modular Research Raman Spectrometer with Microprobe was used at MIT at

514.5nm and 785nm while samples at Harvard University were run with a LabRam system at 632.8nm.

Red lead or white lead standards were run during each session to verify the peaks positions. For each

unknown, an initial 10-20s run was used to check for fluorescence, and then the samples were run for

100-500s depending on the strength of the Raman signal.

4.5 Metallographic Samples

Metallographic samples were taken from two black surface mirrors and examined by SEM. The first was

a corroded surface sample with no bulk metal remaining (Fig. 11). The dark area at the top of the image

was a layer of false corrosion products. Underneath this, the top edge of the metal was severely corroded

and had only 18% copper left, with 67% tin, 7% lead and small amounts of iron, silicon, aluminum,

phosphorus and arsenic. This agrees well with Shoukang and Tangkun

(1993) findings of the top film of black mirrors containing 13% copper,

69% tin and small amounts of minerals. Shoukang and Tangkun did not

report finding any lead, and much of the 7% found in 1943.52.171 may be

present as corrosion products. Underneath the top layer, the altered zone

was mineralized and contained numerous voids, which originally would

have been filled with lead globules. The small amount of remaining lead

appears bright white in the image with most of it located towards the

bottom of the sample farther away from the surface. The altered zone was

composed of 43% copper, 43% tin and 8% lead. Chase and Franklin

(1979) report finding an average of 40% copper and 40% tin in the altered

zone of black mirrors.

In contrast, 1959.77 was a complete cross-section showing a corroded

zone at the top followed by uncorroded core metal at the bottom (Fig. 12).

In the uncorroded metal core, the bright white lead globules sit in the twophase

metallic structure with the lighter areas being the tin rich phase and

the darker areas the copper rich phase. The core metal averaged 68%

copper, 20% tin and 11% lead. In the corroded zone, almost all of the lead

has been leached out leaving behind black voids. The copper rich phase

has been mineralized to a darker, lower molecular weight product believed

by most researchers to be tin oxide, which is consistent with the high tin

content shown by SEM.