Anderson Materials Evaluation, Inc.

Materials Characterization & Failure Analysis Laboratory

XPS, SEM/EDX, FTIR, Optical Microscopy, Thermal Analysis, Electrochemistry

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SUBJ XPS, FTIR, and Optical Microscopy Analysis of a White Material on a Tile Plank

Summary

The white material from the back of the tile and the brown front surface of the tile was analyzed with FTIR, XPS, and optical microscopy and the principal results are:

- The far more plentiful white material on the front of the tile looks upon microscopic examination to be the same powdered material as was found on the front surface of the tile.

- Infra-red spectroscopy analysis (FTIR) of the material showed that it had no significant organic material in it.

- White material from the back of the tile was analyzed with XPS and found to consist mostly of magnesium with lesser atomic concentrations of calcium, aluminum, and silicon in that order.

- A thin layer of the white material still on the front surface was analyzed, though it did not completely cover the brown front surface of the tile. The brown front tile surface where no white material appeared was also analyzed for comparison. The brown front surface material was primarily silicon, with some aluminum and calcium in order of atomic concentrations, but no magnesium. The thin white area, with some brown surface appearing through it, was found to be mostly magnesium with considerable silicon and lesser concentrations of calcium and aluminum in that order. It was deduced that this white material was also mostly the magnesium material found on the back side of the tile.

- It is conjectured that the white material may be a magnesium oxide powder used to cover the tile forming die in a thin layer on the bottom or abraded from refractory bricks which are not very abrasion resistant. MgO has a very high melting point, it has a very low thermal conductivity reducing heat losses, and it may make the removal of the tile from the forming die easier.

- The white powder on the back of the tiles will degrade the adhesive bonding of the tiles, so it should also be removed. The best way to remove it is probably with a pressure washer and some surfactant so it is less inclined to reattach itself to other areas of the tile upon being dislodged.

Samples and Background

The tile plank sample was delivered on 6 June 2016. The tile plank is shown below:

Fig. 1. The top surface of the tile is shown on the left and the bottom surface is shown on the right. The white material on the top surface of the tile has proven difficult to remove upon installation of the tiles. Larger quantities of what appeared to be the same white material on the bottom side along the edges of the square grid raised pattern were removed for FTIR and then for XPS analysis. This material was also examined with an optical microscope. Customer identification on the back of the tile has been blacked out.

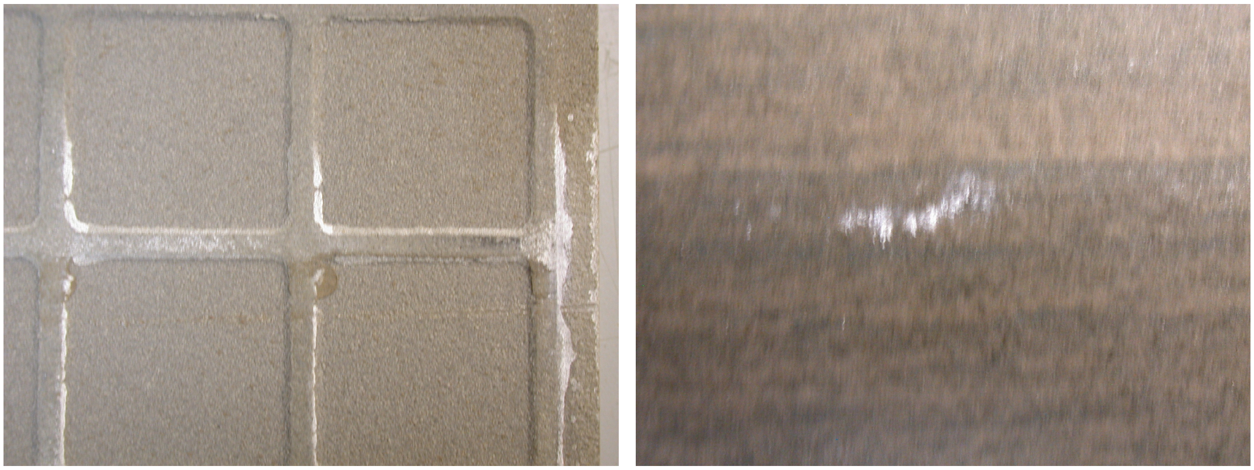

Fig. 2. Macro photographs showing the caked white material on the backside in the left image especially to the right of the raised square grid and on the brown and finished front surface in the right image.

Optical Microscopy Examination

The white material found on the tile was examined under an inspection microscope after being collected on clear tape. The as-printed magnification is 58 times.

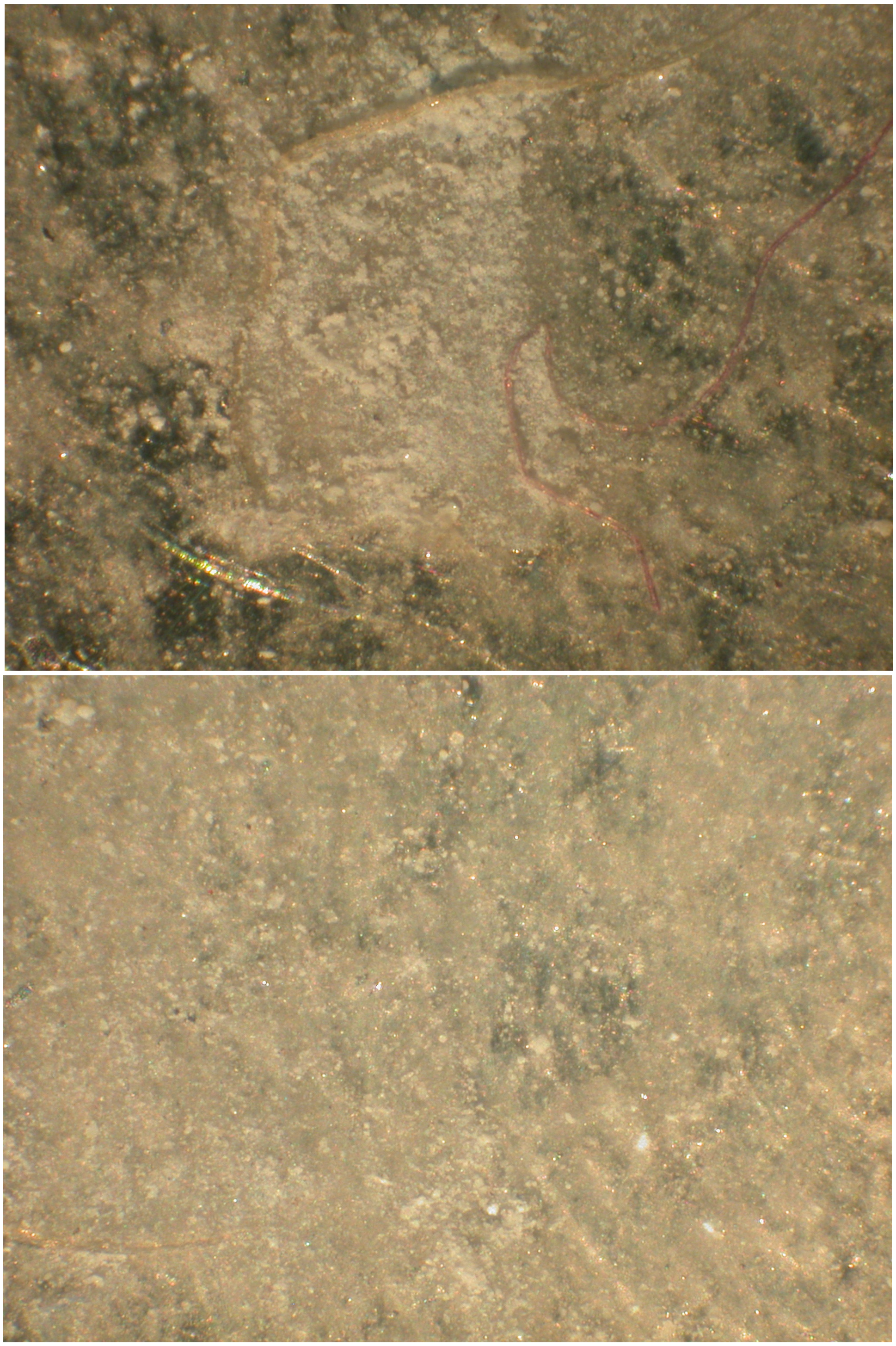

Fig. 3. The white material collected on 3M Clear Double-Stick adhesive tape is shown at an as-printed magnification of 58 times.

FTIR Spectroscopy Analysis

All FTIR data were collected using a JASCO 6100 infra-red spectrometer equipped with a Golden Gate GS-10515 Attenuated Total Reflectance (ATR) Cell with KRS-5 lenses. The FTIR spectrometer has a ceramic mid-infrared source and a temperature controlled DLATGS detector. The Michelson interferometer has KRS-5 lenses and the detector has DTGS windows. Spectra were analyzed using BioRad's KnowItAll Informatics System, Jasco IR Edition. Spectra were matched using the libraries contained in Fiveash Data Management's ATR Polymers and Adhesives Database. Each sample was scanned 128 times to obtain a high-signal-to-noise ratio at a resolution of 4 cm^{-1}.

FTIR infra-red spectroscopy analysis was performed on a compacted slab of the white material removed from the back of the tile. The white material was placed directly on the diamond window of the ATR.

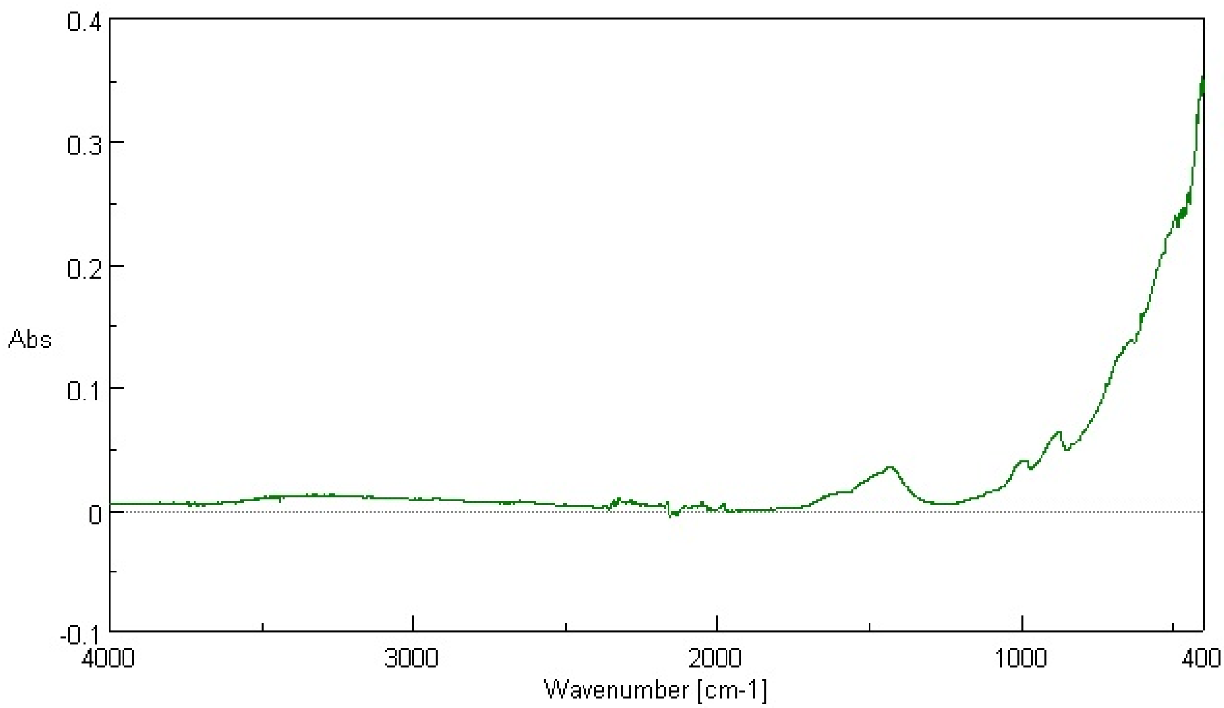

Fig. 2. The white material from the tile is clearly not an organic material. The spectrum shown is characteristic of a silicate type of material.

XPS Analysis

The XPS analysis was performed over an elliptical area irradiated by the low-energy (1487 eV) monochromatic aluminum Kα x-ray with a major axis of 1.0 mm and a minor axis of 0.5 mm. This is an area of approximately 0.4 mm^2. A wide-angle input lens, hemispherical analyzer, and a multi-channel detector make the spectrometer very efficient with respect to the input x-ray flux. The depth of the analyzed volume is about 8 nm, which is determined by the small mean-free path of the emitted photoelectrons. The elemental survey spectra cover the binding energy range from 0 to 1100 eV, with a step size of 0.5 eV. This step size, with the monochromator, the moderate analyzed area size, and a high signal-to-noise ratio, improves the quantitative accuracy and sensitivity beyond industry standards. The XPS system consists of a turbomolecular pumped introduction chamber, an ion pumped sample preparation chamber, and an analysis chamber which is also ion pumped. When samples are inserted into the Analysis Chamber, they pass through the Preparation Chamber, which decreases the exposure of the Analysis Chamber to water vapor and hydrocarbons from the Introduction Chamber. We also make a practice of segregating the samples of different customers to minimize cross contamination.

The quantitative elemental results obtained on the white material shown in the upper image of Fig. 3 from the back of the tile, from the white material on the front of the tile shown in the right image of Fig. 2, and from a brown area on the front of the tile are presented in Table 1. The brown tile surface is primarily composed of aluminum and calcium silicate. The white materials from the front and the back of the tile are fairly similar, though the white material on the front of the tile did not entirely cover the brown surface of the tile. This incomplete coverage has enhanced the silicon content of the data taken of the white material on the brown surface at the front of the tile. So, the white material itself appears to be mostly a magnesium oxide, whose particles are wrapped in a surface layer likely to be magnesium carbonate. There is also some calcium oxide/carbonate in the material and some alumino-silicate material in the white material as well, though these materials may be added to the MgO from the clay materials used to make the tile.

Magnesium oxide powder has a particularly low thermal conductivity (Modern Ceramic Engineering, David W. Richardson, 2nd Edition, Marcel Dekker, New York, 1992, p. 137) and a high melting point of 2620°C. MgO is used in refractory bricks in kilns, though such bricks commonly do not have good abrasion resistance. The fact that most of this white material was found on the bottom of the tile might be due to the tile scrapping this material from refractory bricks. It might also be consistent with the application of a thin layer of the magnesium oxide powder to line the bottom of a tile forming die and reduce heat flow out of the bottom of the die or to serve to ease the ready removal of the tile from the die. Subsequently, when the tiles are stacked, the MgO material is partially transferred from the back to the front surfaces of tiles. It is probably best removed using a pressure water wash prior to installation.

TABLE 1. Atomic concentrations in atomic percent of elements in the surface of samples of the white material from the back and the front of the tile and from a brown area of the front of the tile are given.			
Element	**White Material from Back of Tile**	**White Material from Front Tile Surface**	**Brown Area on the Front Tile Surface**
Carbon	56.24	35.29	33.40
Oxygen	27.53	43.85	42.87
Silicon	1.62	5.52	14.34
Aluminum	2.08	2.20	3.55
Magnesium	8.71	8.11	
Calcium	3.38	3.30	3.20
Sodium		0.30	0.54
Potassium			0.48
Nitrogen	0.44	1.16	1.18
Zinc		0.28	0.40
Iron			0.05