Looking at the Paint Layers of the Grand Entrance Hall, Brunel Museum, Using Block Sectioning, Optical Microscopy, and Scanning Electron Microscopy

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1. Introduction

Understanding the painted layers of a historic structure is very important for a number of reasons. Beneath the topmost layer of paint, there may be designs, repairs, and alterations, with no visible evidence (Oestreicher, 2011). To better understand the chronology of a painted surface, various methods have become increasingly important in the field of material culture studies and conservation. In fact, instrumental analysis has become a strategic tool in determining the materials history and layers within material cultural heritage. One such method is to take a sample of the painted surface and create block sections of the samples, and perform instrumental analysis, including optical microscopy and scanning electron microscopy.

Analyses were undertaken on two paint samples taken from the Grand Entrance Hall at the Brunel Museum as part of a larger investigative project. Although initially analysed in an attempt to locate frescos within the space, this project has revealed the need for a more comprehensive sampling strategy in order to confirm or deny the presence of frescos. However, this paper will focus on the instrumental analysis of the paint layers, thus providing an understanding of the application of microscopy and scanning electron microscopy within the context of better understanding of the painted layers of the Grand Entrance Hall of the Brunel Museum.

Although the investigations of the frescos did not prove fruitful, the investigation of the painted layers did provide valuable information. Most notably, sampling provided a better understanding of what surface coatings existed underneath the topmost layer, which was black and unable to be penetrated by other methods used during the investigation, such as infrared photography. However, to understand the results, an introduction to the history of the space as well as investigations will be provided. Following this, a discussion on the sample preparation, instrumentation, and materials used will be given. A discussion of analysis and recommendations for further work, as well as limitations and potential damaging effects of the project and continuation of the project will follow.

2. Background Information: History of the Space

The Brunel Shaft, the Rotherhithe Shaft, or the Grand Entrance Hall are all terms used to describe the sunken shaft space situated at Rotherhithe. It was originally built as the first phase of construction for the underwater tunnel built beneath the River Thames in London to connect Rotherhithe and Wapping. Currently under care of the Brunel Museum, the historic significance of the Grand Entrance Hall has been recognised by English Heritage, and it is a Grade II listed structure (English Heritage, 2012).

One significant factor of the Grand Entrance Hall lies in that it had provided a direct passage to the underground tunnels, called the Thames Tunnel or the Brunel Tunnel (see **Figure 1**). This tunnel represents the first to have successfully been constructed underneath a navigable river as well as the first that was tunnelled in soft ground (Mathewson *et. al*., 2006). It is seen as a "wonder of the modern world" (Cruickshank 1995) and the "birthplace of the tube" (Time Out, 2012), now a vital part of everyday London life.

Figure 1: A poster created from an enlarged print on a handkerchief

The current floor of the Grand Entrance Hall was placed halfway up the original height of the shaft, slightly below the upper level staircase, as seen in this figure. Written accounts and depictions of the Grand Entrance Hall and tunnel space, as well as the active descent into the tunnel can still be seen today (Drew, 1852; Mathewson *et. al.*, 2006; Brunel Museum Web 2012). Image courtesy of the Brunel Museum.

The Thames Tunnel was an ambitious and dangerous project that took 18 years to complete and was engineered by Marc Isambard Brunel and his son Isambard Kingdom Brunel. The shaft itself is a significant engineering innovation, being the first "caisson" shaft constructed by means of allowing the brick tower to sink into place under its own weight, rather than being dug into the ground (see **Figure 2**) (Mathewson *et. al.*, 2006; Timpson, 2007). It was constructed above ground, using brick, cement, rubble, and iron, and was allowed to sink under its own 1000-tonne weight (Matthewson *et. al.*, 2006). The pioneering feats of engineering used in creating both the shaft and the tunnel epitomise the innovation of the Brunels, the advances seen in engineering during the Victorian era, and has had a significant impact on modern engineering concepts (Cruickshank, 1995).

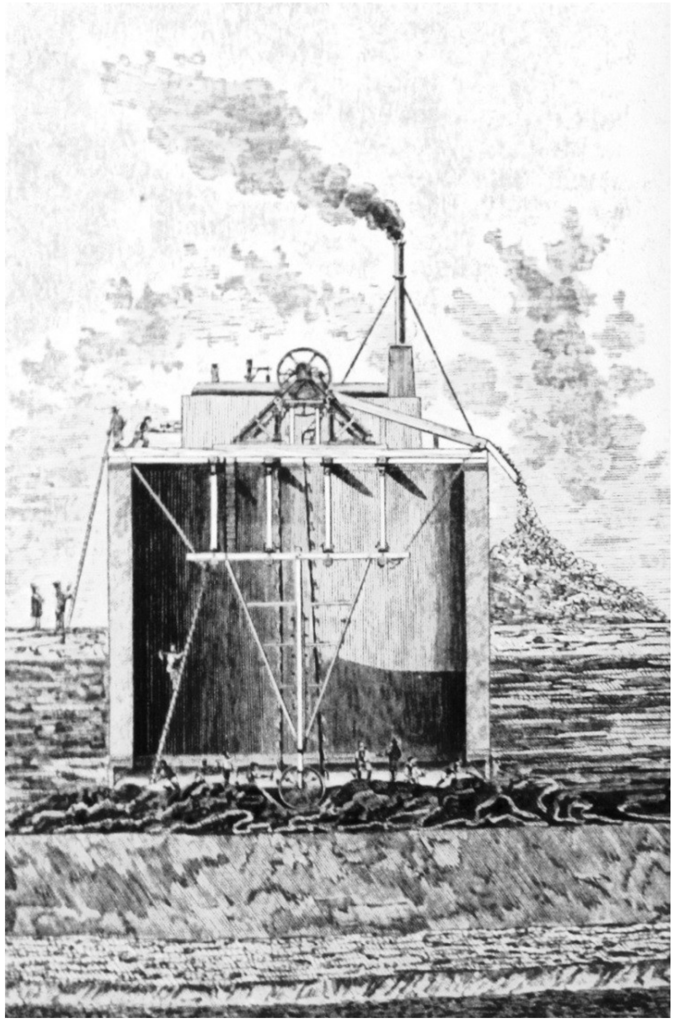

Figure 2: A cross-section of the sinking shaft showing the engine and chain buckets
The sinking of the ambitious and innovative caisson shaft process took 8 months to complete, and in November 1825 the tunnelling shield, a technology that had been developed by Thomas Cochrane and Marc Isambard Brunel, was lowered to a depth of 63 feet down the shaft, after which the excavation of the tunnel began (Mathewson *et. al*., 2006: 33; Timbs, 1860: 287; Smith, 2001: 17;). Image courtesy of the Brunel Museum (Mathewson *et. al*., 2006).

The Brunel Museum, open since 2005, is located in the old Engine House next to the Grand Entrance Hall. Although it had once housed the pumps that drained the tunnel, this function was discontinued in 1913, and the building was let out as a storage space until it was transferred to a charitable trust by the London Borough of Southwark, who obtained it from London transport in 1970 (Matthewson *et. al.,* 2006). At some point, the staircase had been removed, though its outline is still visible.

The Grand Entrance Hall stood open to the skies until it was capped in the 1940s, for fear that tunnel lights would attract an enemy aircraft (Matthewson *et. al.*, 2006). Currently, there is an aluminium roof that has been placed atop the cap. While tours into the Grand Entrance Hall continue today, tours into the tunnel itself were discontinued permanently in 2010 and a reinforced cement floor installed to divide this space from the tunnel itself. Currently, the London Overground line continues to run through the Thames tunnel beneath the concrete slab at the base of the Grand Entrance Hall.

Recently, the Brunel Museum has decided to undertake investigations of the Grand Entrance Hall as part of their bid for Heritage Lottery Funding. Heritage Lottery Funding can be used to repair and transform a wide variety of buildings that people want to keep and hand on to future generations (Heritage Lottery Fund, 2013). Their plan includes improving accessibility to the Grand Entrance Hall, and the eventual connecting of the space to the main museum, as well as the possible refurbishment of the space. However, before any of this can be undertaken, the space muse be investigated thoroughly.

3. Investigation of the Grand Entrance Hall, Brunel Museum

Before the results, interpretations, and recommendations for further work can be discussed, it is important to note that many stages have been involved in addition to the paint analysis of the Grand Entrance Hall. Beginning in 2012, students from the University College London's Institute of Archaeology began working with the Brunel Museum in a collaborative effort to document and understand the history, material, and condition of the Grand Entrance Hall. This research project started with a visit to the site in order to gain an appreciation of the space. High-resolution panoramic photographs were taken to document the space (see **Figure 3**). Documentary research had also been undertaken to better understand the history and significance of the site.

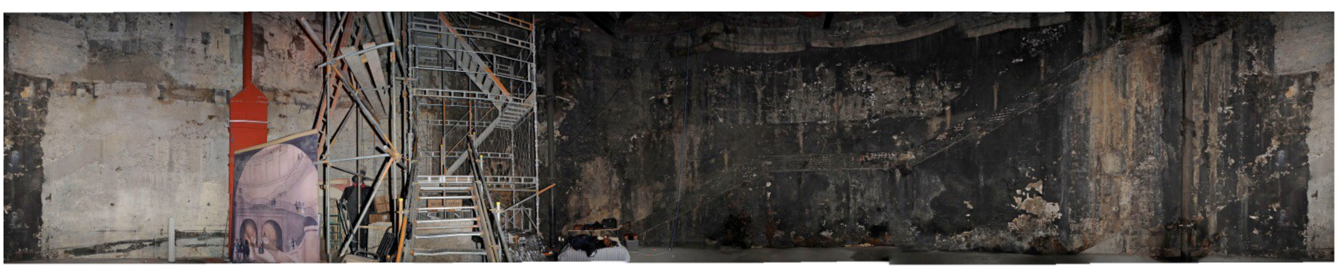

Figure 3: Panoramic photograph of the Grand Entrance Hall, Brunel Museum
As part of the documentation phase of the Grand Entrance Hall at the Brunel Museum, a panoramic photograph was created using a Nikon D700 (using 3.5 f-stop; 1/60 second exposure time; 200 ISO speed). Photographs were taken at 10° intervals to ensure optimal overlap and subsequently rectified using Photoshop CS5. Image courtesy of Stuart Laidlaw. Larger image available in **Appendix A**.

These investigations intend to provide a better understanding of the space ahead of a transformative project intended to increase accessibility into the Grand Entrance Hall.

However, the more specific initial aim of this particular investigation had been to explore the potential presence of painted frescos, a type of mural painting in which water-based paints are applied to fresh (usually still damp) plaster, on the walls of the Brunel tunnel Grand Entrance Hall (see **Figure 4**) (Britton, 1998). This preliminary work was also intended to have the potential to build and expand on the initial work should initial sampling indicate the presence of frescos.

Figure 4: Poster created from a 4 April 1857 advertisement the painted frescos within the Brunel Grand Entrance Hall
There are 19 different fresco paintings listed, which offer a number of scenic views. It is indicated that the artist is I. B. Henkin. Image courtesy of the Brunel Museum.

However, further research into historical accounts and preliminary site visits suggest that frescos may no longer be present on site, if they had been present at all. For example, one historical accounts notes that the walls were “finished in stucco, and hung with paintings and other curious objects” (Drew, 1851), which may suggest that any paintings that had been present were moveable, rather than having been applied directly to the wall, as is the case with traditional frescos.

One initial sampling strategy explored had been the option of inspecting “no less than five 50 cm x 50 cm squares, located in various positions around the shaft” (Jakobi *et.al.,* 2012). Since then, the entire space has been visually investigated and photographed using visible light, infrared light, with select sections having also been investigated using ultraviolet light. These methods, in addition to documentary research, yielded no results. Further survey work of the surface was also unsuccessful.

It was determined that a deeper examination of the painted surface was needed to understand the materials present underneath the topmost surface layer within the space, which was black and impenetrable (especially as there was nothing revealed through infrared photography of the space). Analysis of paint samples may inform decisions that will be made by the Brunel Museum regarding the establishment of a more comprehensive sampling strategy, should further sampling be desired. In addition, it is hoped that the analysis of the paint samples will provide options as to how to renovate the space. This will especially inform decisions regarding the issue of future surface coatings, should a more uniform coating be applied to the walls of the space in the future.

4. Methodology

Before the results can be discussed, the sampling strategy, the method of sample preparation and the instruments and materials used must be briefly discussed. Only following this can the results of the analyses be interpreted.

4.1. Sampling Strategy

Samples were taken from the Grand Entrance Hall during the survey process. As mentioned previously, the collecting of the paint samples occurred concurrently with the materials survey and condition assessment, which took place over two days. Prior to this, a visual analysis of the site was undertaken, as was documentary research. During the survey, two samples were taken. Sample A was taken from within the parameters of the initial sample survey area, while Sample NN was taken from outside the initial sample survey area (see **Figure 5**).

Figure 5: Samples taken from section of the Grand Entrance Hall, Brunel Museum
Pictured above is a section of the panoramic photograph of the Grand Entrance Hall, Brunel Museum. This section of the panoramic photograph depicts a section of the wall with easily removable surface layers. The red box depicts an enlargement where the red x indicates the origin point of Sample A. The blue box depicts an enlargement where the blue x indicates the origin point of Sample NN.

Small samples (approximately 1 cm x 1 cm) were collected from surface that was easily removable. This easily removable surface indicates a painted layer atop the stonework. Both samples were taken from roughly 5 feet above ground. They were removed from the edges of pre-existing damaged regions which exposed the stone substrate so as to minimise visual loss. They were removed using a scalpel blade.

4.2. Sample Preparation, Instruments, and Materials Used

After Sample A and Sample NN were collected from the Grand Entrance Hall, they were prepared separately preceding instrumental analysis. For this, they were vacuum impregnated using HXTAL NYL-1, a two part epoxy resin. Epoxy resin was chosen for several reasons:

- epoxy resin will physically adhere to the sample with good bond and eliminate shrinkage gap;
- epoxy will not react with the sample and with other solvents, etchants, chemicals, and oil lubricants used in sample preparation;
- epoxy is hard enough to produce flat surface during grinding and polishing;
- epoxy has a very low setting shrinkage, and can therefore provide excellent edge retention of samples due to better adherence to sample and low shrinkage; and
- due to low viscosity, epoxy can be drawn into pores and cracks by vacuum impregnation (Jana, 2006).

Vacuum impregnation and encapsulation were techniques employed to fill the voids, pores, and cracks of the paint sample; improve the overall integrity and preserve the original microstructure; allow for ease of handling; and to allow the application of electrically conductive materials to provide better analysis (Jana, 2006). The epoxy resin containing the sample (laid horizontally) was cold set in 30 mm diameter silicone release moulds. This was left to set for a week due to the curing time of HXTAL NYL-1.

HXTAL NYL-1 was chosen because it has good physical properties. Most notably, it has very good aging properties (with minimal yellowing), low viscosity, and good strength characteristics

(Restoration Supplies, 2013). This is important as it can be given to the Brunel Museum and be retained for future analyses, should they be needed.

Once set, a diamond wheel set on a Buehler Isomet 11-1180 Low Speed Saw was used to cut the sample in half to expose the cross section (and therefore the various paint layers) of the sample. Various weights were used to assist the process. Once cut, the impregnated and encased samples were once again cold set in epoxy resin, with the cross section placed face down in the silicone release moulds.

After the HXTAL NYL-1 cured, sanding was done using a Buehler MetaServ 3000 variable speed grinder-polisher loaded with sandpaper to expose the cross section. First, 2500 sandpaper was used, then 1200, 800, 400, and 250. During the sanding, the sample was checked using polarising light optical microscopy to ensure that scratches in the surface layer were minimal and did not interfere with the sample surface, and to ensure that the sample block was level.

Once sanding was complete, any excess debris was removed in an ultrasonic bath using industrial methylated spirits (IMS) as a solvent. IMS was chosen, as the relatively-newly dried epoxy resin would be less soluble in this than other solvents (such as acetone). Once cleaned, the samples were set to dry on a heater rack for a short period of time (less than one minute).

The samples were coated on the top and sides with a thin coating of carbon using an Edwards Auto 306 coating system. This carbon coating produces an electrically conductive material that will prevent accumulation of electrical charge on the surface (Jana, 2006). Once coated, the samples were kept in a Stuart box, which provides the samples with a dust-free environment.

Prior to scanning, a thin strip of electrically conductive adhesive transfer tape, which is conductive through the z-axis (thickness) of the adhesive and electrically connects and mechanically bonds circuits, was placed on the underside, which was uncoated (3M, 2013).

Scanning electron microscopy (SEM) was accomplished with a Philips XL30ESEM with an attached Oxford instruments environmental detector. The Philips XL-30 ESEM is a flexible scanning electron microscope with a large chamber that can be used for conventional high vacuum imaging, or in the environmental mode, can be used to examine wet, oily, gassy, or non-conducting samples (MEMS & Nanotechnology Exchange, 2013). The software used was XL microscope control software on the primary computer (imaging) and INCA point and ID software on the secondary computer (qualitative analysis). The readings were done using stoichiometry analysis with reading normalised to oxygen.

Optical microscopy was accomplished with Leica DM LM with 10x magnification eyepieces. Pictures were taken using a camera attachment.

5. Results and Interpretations

Once the Sample A and Sample NN were collected and prepared, the exposed cross section was first examined with optical microscopy then scanning electron microscopy (SEM). In this section, a brief introduction will be given to both optical microscopy as well as SEM, the results will be presented, and interpretations of the data collected will be given. Since the analysis of the painted layers of the Grand Entrance Hall was completed as a preliminary investigation into the sub-surface, suggestions for further work and analyses will be provided, as will limitations and potential damaging effects.

5.1. Optical Microscopy: Results and Interpretations

Optical microscopes are used to magnify objects and can provide information about the structure and characteristics of the sample (Stuart, 2007). Where paint may appear similar to the naked eye, they show very different crystal properties at a microscopic level (Stuart, 2007). Both Sample A and Sample NN were examined under the microscope using 50x, 100x, and 200x magnification (see **Figure 6** and **Figure 7**).

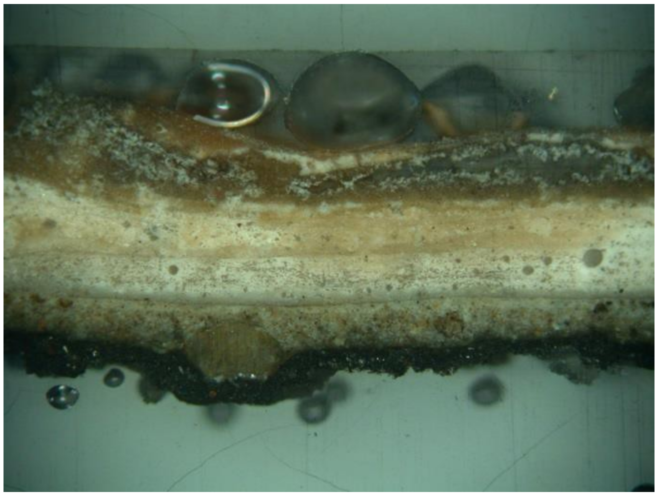

Figure 6: Central portion of Sample A under 50x magnification
The black layer orientated at the top of the photograph is the topmost layer, while the bottom layer is the layer closest to the stone substrate of the Grand Entrance Hall, Brunel Museum.

Figure 7: Central portion of Sample NN under 50x magnification

The black layer orientated at the top of the photograph is the topmost layer, while the bottom layer is the layer closest to the stone substrate of the Grand Entrance Hall, Brunel Museum.

Optical microscopy has provided further understanding into the irregularity of the easily removable surface of the Grand Entrance Hall. Magnification has provided an understanding into the differentiation of grain sizes present within the previous surface coatings. The results reveal at least four previous surface coatings underneath the black layer, visible from **Figure 5** and **Figure 6**. This can be discerned not only by differences in grain size, but by differences in general colouration as well.

Optical microscopy has revealed that in both samples, wet paint had been applied over dry paint throughout the history of the reapplication of surface coatings. This can be seen by the separation at the interface between layers.

Typically, the first paint layer identified on the altered element might provide a useful benchmark for dating other elements (Oestreicher, 2011). Unfortunately, because the lowest painted layer originated at an unknown date, with further maintenance cycles unknown, specific dating is actually an impossibility. Stratigraphic analysis therefore reveals only a relative chronological notion of the painted layers.

Under optical microscopy, specks of red were found within both samples (see **Figure 8**). Because they were within and adjacent to the black topmost layer, it was determined that further investigations would be needed to characterise this colourant. Additionally, grains can be characterised, should there be a desire for the historical reproduction of the coating to be reproduced to create a uniform surface layer within the Grand Entrance Hall in future renovations.

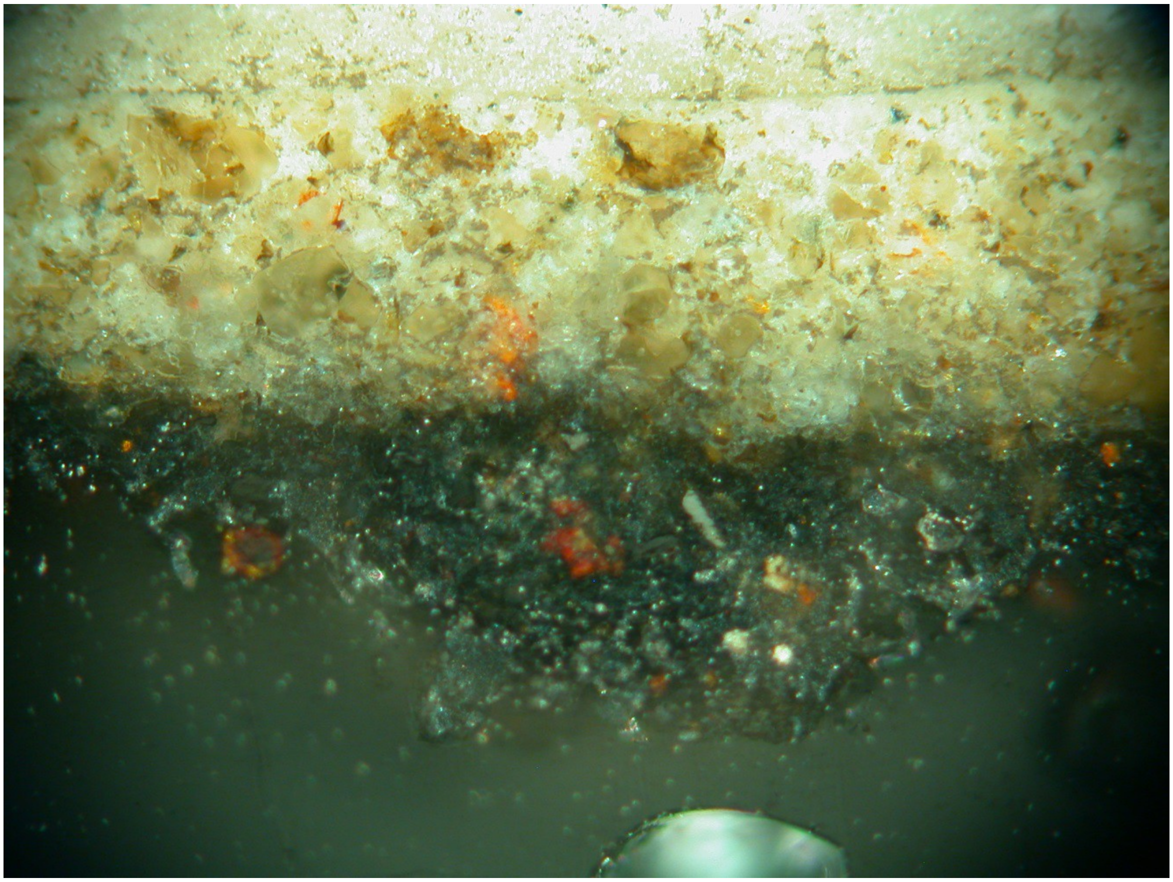

Figure 8: Central portion of Sample A under 200x magnification
Pictured above is a 200x magnification of the central portion of Sample A. Note the red specks throughout the sample, especially within and adjacent to the black topmost layer.

There are some limitations to optical microscopy. Due to the sample block's height, magnifications of larger than 200x were not obtainable. Despite this, optical microscopy has provided an effective preliminary look into the microstructure of the sample.

5.2. Scanning Electron Microscopy: Results and Interpretations

Scanning electron microscopy (SEM) was undertaken in an attempt to characterise the materials of the various layers within the sample. SEM can provide a high-magnification imaging and characterisation of the elements within a sample through scanning with a beam of electrons within a vacuum (Stuart, 2007). In addition to this, SEM images have great depth of field, yielding a characteristic three-dimensional appearance useful for understanding the structure of a sample (Stuart, 2007).

A number of readings were taken of each sample with no peaks omitted. Point analyses were taken along a line across the width of the centre of Sample A and the edge of Sample NN to ensure that each layer was analysed. Box analyses were taken to acquire averaged readings of larger areas. In addition to this, specific grains were also analysed using point analysis to better understand the nature of inclusions within the layers.

Various particles and grains throughout the sample were examined and characterised. Sodium, chloride, calcium, sulphur, magnesium, potassium, and silica have been found throughout the sample (see **Figure 9a**, **Figure 9b** and **Figure 10a**, **Figure 10b**). No traces of pigmenting agents were detected, aside from titanium oxide and zinc oxide, which is white in colour. All these elements are consistent with the composition of plaster.

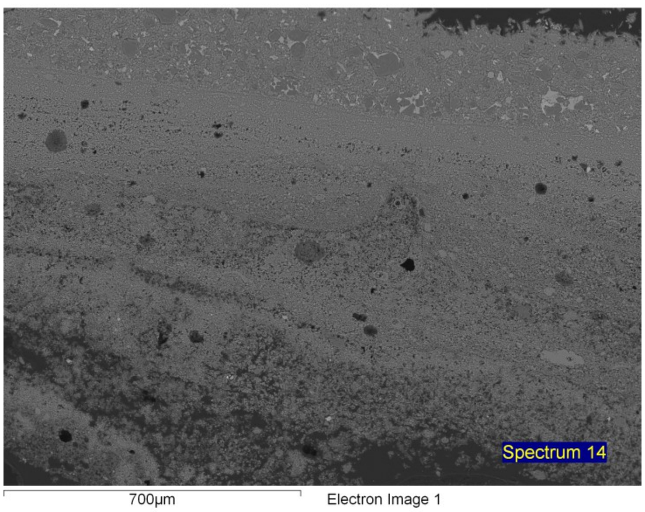

Figure 9a: Central portion of Sample A in SEM

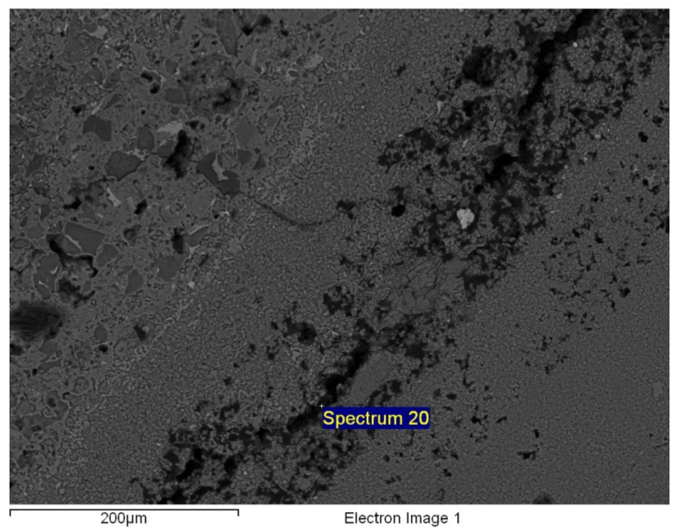

Figure 10a: Central portion of Sample NN in SEM

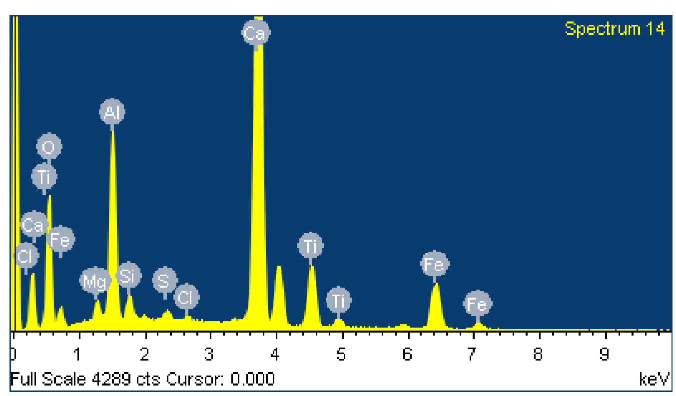

Figure 9b: SEM spectrum of central portion of Sample A

This is an example of one SEM spectrum resulting from a point reading from Sample A. As can be seen from this spectrum, a number of elements are present, including chloride, calcium, titanium, iron, magnesium, aluminium, silica, and sulphur.

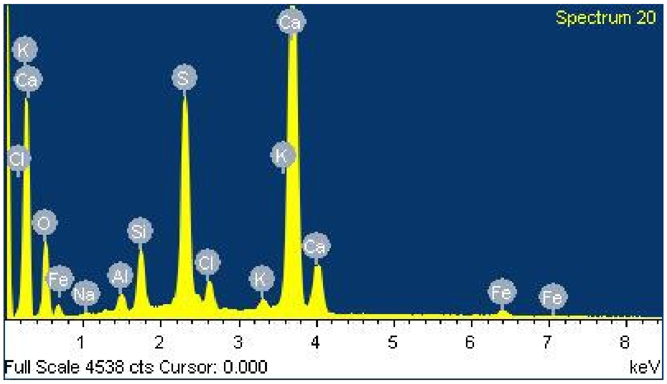

Figure 10b: SEM spectrum of central portion of Sample NN
This is an example of one SEM spectrum resulting from a point reading from Sample NN. As can be seen from this spectrum, a number of elements are present, including chloride, calcium, potassium, iron, magnesium, aluminium, silica, and sulphur.

Sodium and chloride may be leaching through the walls from the water of the Thames River. If this is the case, some of the elements may have leached out from the concrete as well. The presence of calcium sulphate is commonly found within plaster (Cassar and da Angelis, 2000). Magnesium oxide is a common additive to provide expansion in cement (Swanson, nd). Potassium chloride is added to cement slurries for application in water-sensitive clays (Halliburton, 2013). Silica (perhaps in the form of quartzite grains such as sand) may have been added as a bulking agent.

Therefore, the various layers can be characterised as plaster or plaster wash applications with various inclusions. In addition to this, the topmost black layer has been characterised as most likely soot. Because pigment particles appear denser than carbon particles in SEM, the imaging suggests soot rather than pigment (Stuart, 2007).

The bright red colouring has been characterised as iron. Since there is water damage which can be seen down the walls of the Grand Entrance Hall, the presence of this is consistent with metal or rust particles that have been carried down the wall and deposited along the topmost surface coating. This may have been originated from piping. Similarly, the abundance of aluminium within the sample may be runoff from the aluminium roof on the top of the Grand Entrance Hall.

There are limitations to SEM. It must be noted that one limitation of SEM is that many elements will display overlapping peaks. Additionally, sample preparation and analysis have been a very time-consuming process.

5.3. Suggestions for Further Work and Analyses

Colourants have not been found within this paint sample, which may exclude the possibility of frescos. However, despite the results of the initial sampling and analysis, it is also important to note that the lack of frescos or other decorations in these samples do not eliminate them from the wider space of the Grand Entrance Hall. Suggestions for future work include a wider sampling methodology (see **Figure 11**). This means that a more comprehensive sampling strategy which takes into account the entirety of the surface and potential designs will need to be utilised.

Figure 11: Potential sampling strategy taken from section of the Grand Entrance Hall, Brunel Museum
Pictured above is the section of the panoramic photograph of the Grand Entrance Hall, Brunel Museum with easily removable surface layers. The red x indicates the origin point of Sample A and the blue x indicates the origin point of Sample NN. Taking into account areas of poor condition (as noted by the condition survey), the above schematic is one possible strategy where the yellow xs denote potential areas for future sampling.

If the line of the original staircase is accepted as a feature original to the time period in which the Grand Entrance Hall was used as an attraction, then it may stand to reason that any frescos would be in the space above, and therefore easily viewable. Therefore, any potential frescos may be higher up than in the space sampled.

If a comprehensive sampling strategy is devised, then it is important to note the limitations and potential damaging effects which may arise from undertaking such a project. One limitation of this investigation is the visual consequences. Further sampling will create holes within the surface of the Grand Entrance Hall where there had previously been an uninterrupted layer. Additionally, because the entirety of the Grand Entrance Hall is not available for investigation (due to the cement floor and the active train line running beneath the space), any study cannot be considered complete. There is also damage which has resulted from the years that the Grand Entrance Hall laid in disuse and disrepair, with unrecorded maintenance. These unknown conditions, and suggestions from previously mentioned historical sources, may already preclude the presence of existing frescos.

6. Conclusions

In addition to providing an initial analysis of the painted layers, this project included high-resolution panoramic photographs of the space, a materials survey, and a condition assessment. It is important to note that an analysis of the paint layer alone will likely never yield informative results. Rather, site visits and historical documentation are also of great importance in understanding the chronology of a site, and should not be overlooked.

Paint analysis is a valuable tool that can be used to better understand the chronological notion of a painted structure. Visual analysis and the combination of the instrumental methods of optical microscopy and scanning electron microscopy enabled the characterisation of several surface coatings beneath the topmost layer. However, regarding questions pertaining to the presence of frescos within the Grand Entrance Hall, this initial examination has been less successful.

This initial investigation had been intended to be preliminary work that would be carried out with the goal of either confirming or denying the presence of frescos within the Grand Entrance Hall. While there was no indication of pigmentation within the samples, as mentioned previously, a more comprehensive sampling strategy may need to be employed in the future in order to firmly confirm or deny the presence of frescos within the Grand Entrance Hall. However, even if further sampling is not undertaken, the value of these readings lies within understanding previous surface coatings, should the Brunel Museum trust decide to further renovate the space and create a uniform surface coating.

List of Figures

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Appendices

Appendix A: Panoramic Photograph of the Grand Entrance Hall, Brunel Museum