



Microscopy for the built environment

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Geomaterials such as concrete, stone, brick and asphalt are of huge economic importance to the global construction industry. The performance of built infrastructure is dependent on the availability of high quality construction materials, good construction workmanship and adequate maintenance throughout service life. Microscopy has been used to investigate building stone for over 150 years by geologists as part of the discipline of 'petrography'. Microscopical and petrographic techniques have since been applied by materials engineers to the investigation of a wide range of other geomaterials, to become an indispensable element of the construction and infrastructure maintenance industries. This article reviews the application of petrography to various stages of the construction material life cycle starting with resource assessment and quality assurance, through to in-service condition surveys, forensic engineering of failures and the conservation of historic buildings.

Geomaterials and the materials engineer

Geomaterials are geologically-derived materials that are used in the construction of buildings or other structures. They may include processed or unprocessed soils, rocks or minerals, including man-made materials such as bricks or cement. However, construction materials that require more extensive manufacturing, such as steel and synthetic paints, are not considered to be geomaterials. Common types of geomaterials include building stone, armourstone, adobe, fired clay bricks, terracotta and tiles, asphalt and roadstone, construction fill, cement, concrete, mortar and plaster. The value of geomaterials-based construction products to the construction sector, and hence the global economy, is considerable.

Materials engineers are an essential part of construction and civil engineering project teams. They use their knowledge of geology, civil engineering and materials science to improve the quality of our built environment by ensuring that construction materials are fit for purpose. They also apply their knowledge of the properties and performance of geomaterials to investigate the condition of structures during service, determine the cause of failures, specify repairs and conserve historic buildings.

Microscopy of geomaterials

Petrographic examination is one of the most powerful investigative tools available to the materials engineer. It involves using the polarising microscope to examine thin-sections or polished surfaces of samples in the laboratory, in the same way that geologists examine rock samples. This may be supplemented by various other microscopical techniques, chemical analysis and simple physical tests. Through the microscope, the materials engineer can determine the composition of geomaterials, assess their quality, and investigate the causes and extent of deterioration. Around the world, tens of thousands of petrographic examinations are performed on construction materials each year in commercial, research and academic environments.

Commercial petrographic examinations must be conducted by suitably qualified and experienced microscopists. The Applied Petrography Group (APG) of the Geological Society maintains a list of petrographers with appropriate experience (www.appliedpetrographygroup.com). Petrographic examinations are conducted in accordance with published standards according to material type and geographic region. In the European Union, standard EN 12407 is used for building stone (British Standards Institution, 2007), EN 12326-2 for roofing slate



Fig. 1. Obtaining a concrete core sample from a concrete structure by diamond drilling.



Fig. 2. Microscopical examination using the low-power stereo microscope (right) and high-power petrological microscope (left). Courtesy of IBIS Limited.

(British Standards Institution, 2000) and EN12407 for aggregates (British Standards Institution, 1997). No European standards currently exist for concrete and mortar, so codes of practice for the petrographic examination of concrete and mortar have been developed by the APG (Applied Petrography Group, 2008a and 2008b).

A considerable amount of geomaterials microscopy literature has been published including a number of handbooks, such as Ingham (2011), which offers the broadest coverage of geomaterials petrography currently available. St John *et al.* (1998) and Walker *et al.* (2006) have published handbooks specific to concrete petrography and Campbell (1999) has published an atlas for cement microscopy. A guide to concrete petrography aimed specifically at non-petrographers has been published by the Concrete Society (2010). The bi-annual Euroseminar on Microscopy Applied to Building Materials (EMABM) has been the leading

geomaterials microscopy conference worldwide for more than 20 years, and their various proceedings represent an invaluable repository of knowledge.

Petrographic techniques

Sampling

Samples of construction materials may be obtained shortly following their manufacture, or from structures during construction or while they are in service. The number of samples required to achieve the investigation objective will depend on the purpose of the investigation/testing, the size of the structure, the types of construction systems used and the number of construction phases. The actual sampling technique and the type of sample required for petrographic examination depends on the type of geomaterial being sampled, the objectives of the investigation and if any other types of tests are to be carried out on the sample. Bulk samples can be taken from the production run but for sampling of materials

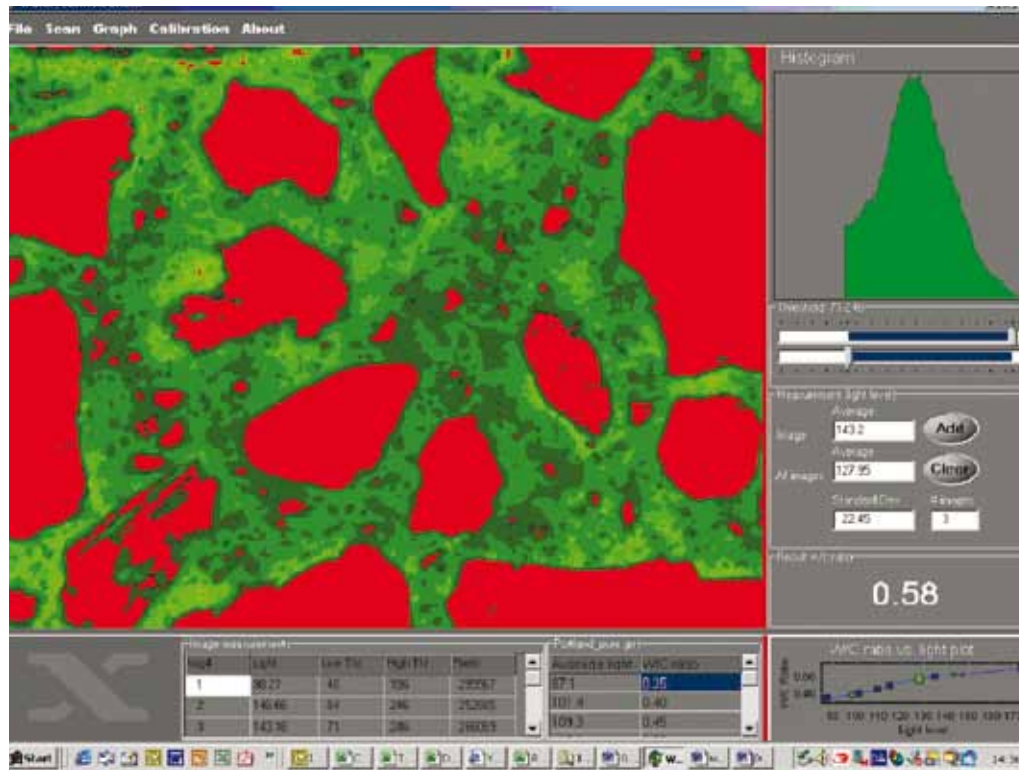


Fig. 3. A user interface of WIC-CHECK software for automatic determination of water/cement ratio of concrete thin-section specimens. Showing a colour segmented image, intensity histogram, calibration curve and analysis data (courtesy of Concrete Experts International).



Fig. 4. The RapidAir 457, Automated Air Void Analyzer system for concrete (courtesy of Concrete Experts International).



Fig. 5. The unexpected appearance of the new portico of a historic building, following construction with the wrong stone. The insets show the microscopical appearance of Anstrude limestone (upper left, field of view: 7 mm) and the Portland limestone (upper right, field of view: 5 mm).

from structures during service, samples are typically removed using hand tools or by diamond drilling techniques (see Figure 1).

Specimen preparation and examination

Following arrival in the laboratory, samples are first examined using a low-power stereo-zoom microscope at magnifications of up to $\times 100$ (see Figure 2). The initial examination is used to determine the most appropriate location for a thin-section/s (typically 75×50 mm area) to be taken for further, more detailed high-power optical microscopical examination.

Preparation of thin-section specimens involves production of 0.03 mm thick ground slices of the sample mounted on glass slides through which light will pass to allow microscopical observation. The thin-section preparation of many geomaterials presents considerable challenges for the technician as many are relatively soft and friable and heat and/or water sensitive. It is often necessary to dry the sample slowly at low temperatures ($<60^{\circ}\text{C}$), consolidate by impregnation with resin and conduct cutting and grinding operations using oil/alcohol as a coolant

rather than water. Further details of specimen preparation techniques for construction materials are provided by Jana (2006).

Thin-sections are examined in plane-polarised or cross-polarised transmitted light using a medium to high-power petrological microscope at magnifications typically up to $\times 600$ (see Figure 2). Fluorescent dyes added to the consolidating resin during sample preparation may be used to aid the examination of cracks and pore structures when the specimen is viewed in combination with a strong light source and an excitation filter. Sometimes highly polished specimens are prepared for examination in reflected light. Reflected light is particularly useful for examination of opaque minerals and cements.

The relative proportions of various constituents of geomaterials can be approximately estimated visually by comparison with standards charts or reference samples. More accurate determinations of composition can be made by modal analysis, using petrographic techniques such as point counting or linear traverse. Modal analysis is most commonly applied to hardened

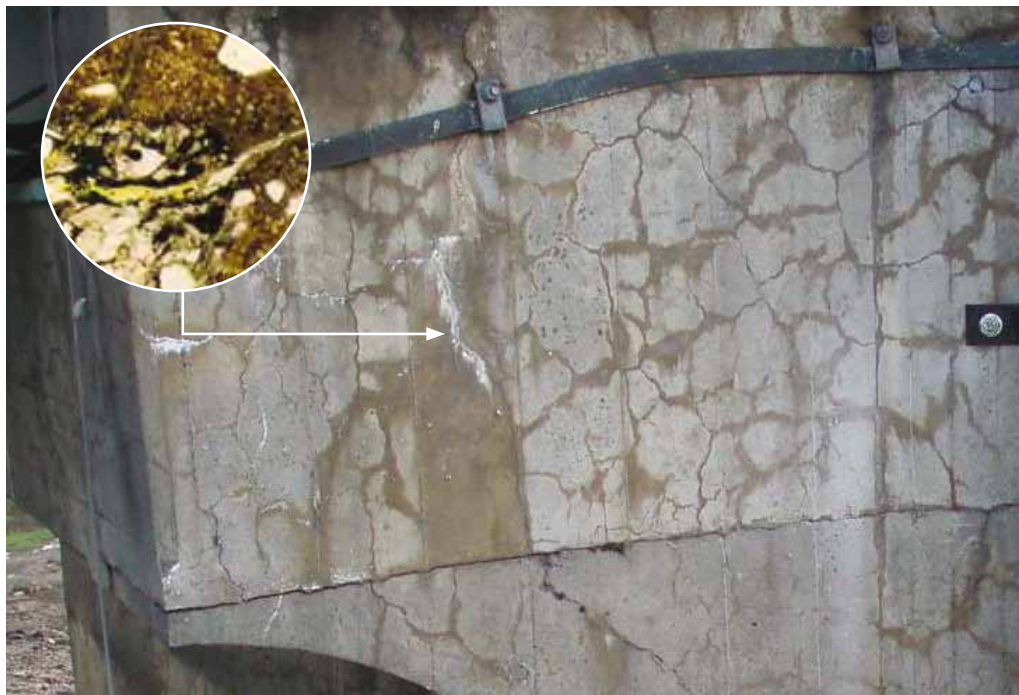


Fig. 6. View of a concrete reservoir intake structure showing pattern cracking caused by alkali-silica reaction. The inset shows the microscopical appearance of the deteriorated concrete (field of view: 5 mm).

concrete to determine cement content, aggregate content, aggregate grading and/or air void content. This is performed on finely ground concrete slice specimens (typically 100 x 100 mm minimum area). Manual point counting is labour intensive, so consequently digital image analysis techniques have been developed in an effort to make modal analysis more efficient. For example, image analysis software is available to determine the proportions of different minerals in thin-sections of rock. Also, specialist software is available to determine the water/cement ratio of hardened concrete from camera images of fluorescent impregnated thin-sections (see Figure 3). An automated apparatus has been developed to perform EN 480-11 (British Standards Institution, 2005) air void analysis of hardened concrete in less than 15 minutes (Jakobsen *et al.*, 2005). The apparatus comprises a computerised control unit (and monitor) with image analysis software, a video camera and a microscope objective mounted on a moving stage (see Figure 4).

Complimentary techniques

Sometimes there are benefits in supplementing optical microscopy with scanning electron microscopy (SEM)

or chemical analysis. These techniques are particularly useful where very high magnification/resolution imaging is required and when the identity of a particular mineral or compound is in doubt. SEM and associated X-ray microanalysis is the most commonly used complimentary technique. Mineralogical analysis by X-ray diffraction (XRD) can be helpful for identification of crystalline minerals and decay reaction products, when the optical properties do not allow definitive microscopical identification. Infrared spectroscopy is the method of choice for identifying organic materials.

Microscopy for quality assurance of construction products

The construction of strong and durable structures is dependent on the use of high quality construction materials. The raw materials for geomaterials-based construction products are extracted from natural resources such as quarries. They are inherently variable in character and it is common to find the good soil/rock/aggregate of a certain part of a natural resource passing into less suitable material within a short distance. Consequently,

there is a need to ensure that geomaterials are fit for purpose by undertaking a programme of initial type testing for every new source. Once it has been established that the material is suitable for the proposed construction application, further testing of the factory product is required at regular intervals for quality assurance. Within the European Union, petrographic examination is undertaken as part of initial and production stage quality assurance testing for stone, slate and aggregate products, to enable their compliance with the Construction Products Directive (CPD). Although petrographic examination is not a required test for the routine quality assurance of concrete and other cementitious products, it is commonly used to determine the causes of apparent deficiencies observed during construction. For example, microscopical examination is often used to investigate the causes of low strength concrete supplied to site.

Microscopy can also be employed to check that the construction product supplied to a project is the one that was specified, rather than cheaper and/or inferior material. Microscopy can be used to provide a petrographic fingerprint for geomaterials that (in

favourable circumstances) can be used to identify their source. For example, during construction of a new masonry portico to a historic building, the architect was unhappy with the appearance of the natural stone that had been used. The architect had intended that Portland limestone be used to match the appearance of the rest of the building. The stone that the masons were using was of unexpectedly light colour and of visibly different texture (see Figure 5). This was investigated by microscopically examining core samples taken from a number of locations on the new portico. Point-counting of thin-section specimens (and references) was undertaken to obtain detailed proportions of the various limestone constituents. The results proved conclusively that the wrong stone had been used. Instead of Portland stone, the data indicated the use of French Anstrude limestone.

Microscopy for routine condition assessment of structures

The management of buildings and other structures is undertaken by a variety of authorities and owners. As part of asset management planning regular inspections are conducted to determine the

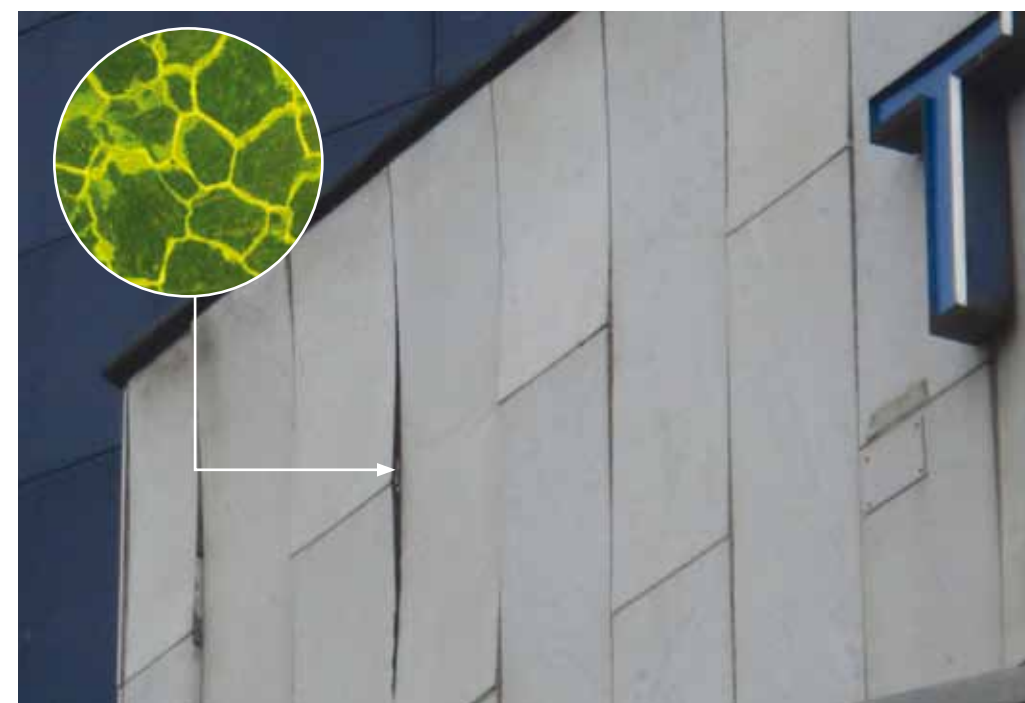


Fig. 7. View of warping marble cladding panels on a building (Courtesy of IBIS Limited). The inset shows the microscopical appearance of the marble exhibiting granoblastic texture viewed in fluorescent light (field of view: 1 mm).



Fig. 8. The Great Wall of China at Panjiakou Reservoir, Hubei. The insets show the microscopical appearance of the brick (upper left, field of view: 1.8 mm) and lime mortar (upper right, field of view: 4.5 mm).

condition of physical assets. Special inspections may be requested to investigate particular problems, or when a change of use is proposed for the structure. Petrographic examination of core samples is often included in detailed condition investigations and special inspections. The utilisation of microscopical techniques enables the buildings to be surveyed on a microscopic scale, screening the structural fabric for evidence of distress. Concrete in particular is known to undergo a number of deleterious reactions that are readily diagnosed through the microscope, such as alkali-silica reaction (ASR) or delayed ettringite formation (DEF), which cause concrete structures to deteriorate at an increased rate. Figure 6 shows a concrete intake structure from a reservoir that was found to be suffering from alkali-silica reaction. Microscopical examination indicated that the reaction was linked to the use of highly alkali-reactive greywacke aggregate, which had reacted to cause expansion and pattern cracking of concrete elements.

When buildings change ownership a deleterious materials survey may be requested. Microscopical examination is used to identify the presence of undesirable materials such as asbestos and high-alumina cement concrete.

Microscopy for forensic engineering assessment of structures

Forensic engineering involves the investigation of failures in structural integrity, serviceability or performance of constructed facilities, both during and after construction. Microscopy has been successfully applied to determine the causes and significance of a wide variety of failures in the built environment, enabling lessons to be learnt for the future. Examples include determining the degree of damage caused during fire or blast incidents. Features such as cracking, porosity and mineralogical changes caused by heating can be observed microscopically in concrete, stone and mortar enabling accurate determination of the extent of damage to structural members (Ingham, 2008). Other failure scenarios where microscopy has been applied include defects with flooring and roofing, weakness in mortar, debonding or plaster/render, cracking in concrete, failures of concrete repairs and waterproofing systems.

A problem particular to certain marbles is non-reversible expansion caused by thermal cycling. For thin cladding on building exteriors, this can cause

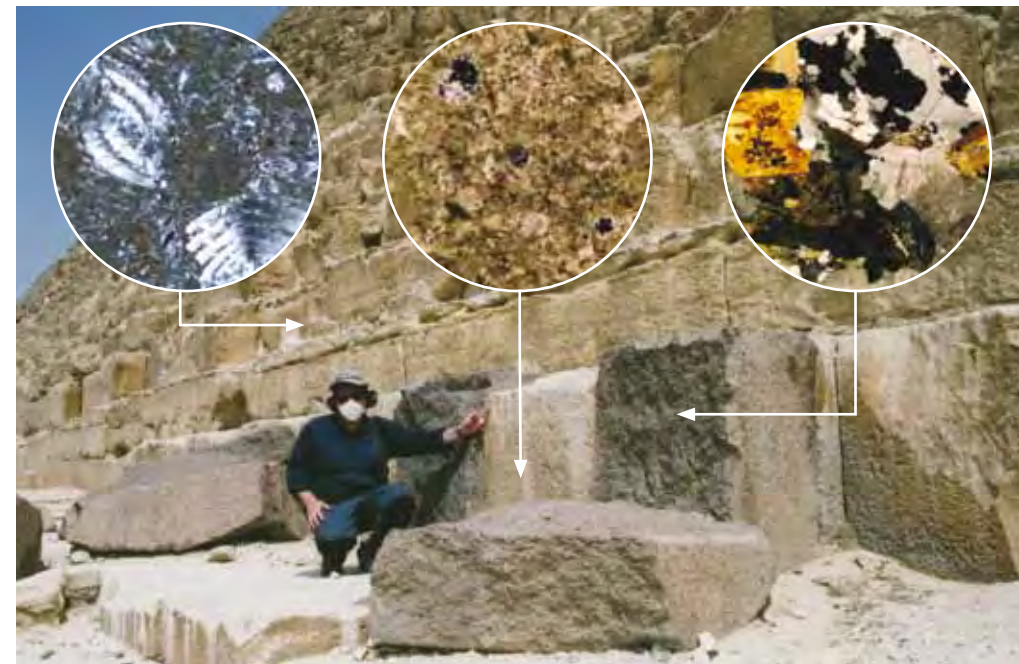


Fig. 9. Khafre's Pyramid at Giza, Egypt. The insets show microscopical appearance of the Mokattam limestone core of the pyramid (upper left, field of view: 7 mm), the Tura limestone casing (upper middle, field of view: 2.5 mm) and Aswan granite casing (upper right, field of view: 4.5 mm).

bowing or warping of the individual panels, which could potentially lead to panel/fixing failure (see Figure 7). The problem seems to be closely related to the microstructure of the marble. Marble with xenoblastic texture seems to be less prone to bowing than marble with the granoblastic texture. Xenoblastic marble has irregular grain boundaries that interlock with each other. Granoblastic marble has crystals that are almost equal width in all directions. The grains are typically polygonal with almost straight boundaries that often meet at triple points. The microtexture of marble can be easily studied using the polarising microscope. The definition of the crystal boundaries can be enhanced by using fluorescence microscopy or alternatively by etching.

Petrography applied to conservation of historic masonry structures

Over time, exposure to natural weathering processes and in-service 'wear and tear' causes the deterioration of historic buildings. Prior to specifying repairs or cleaning programmes for historic buildings, it is necessary to undertake inspections and investigations to determine the types of construction materials

present, the causes/extent of deterioration and types of surface soiling.

Petrographic examination is a useful technique for determining the historical source of materials such as building stone and the ingredients for mortar, brick and terracotta (Ingham, 2005). For stone masonry, the most compatible building stone for repair and replacement is usually one obtained from the original source quarry. Historically building stones were obtained from locally occurring sources but as communications developed stone started to be traded over considerable distances to suit particular aesthetic or durability criteria. Many historic quarries are no longer working, in which case petrographical information is used to help locate an appropriate substitute stone.

Burnt bricks and terracotta are manufactured from clays with an added temper of sand to reduce shrinkage, and various additives. On firing, the clays are sintered and lower melting point ingredients such as metallic oxides and lime undergo mineralogical changes with a resulting increase in strength. By examining relict materials, microscopical examination can be used to

determine the source of the raw materials and the manufacturing process. Figure 8 shows an unrestored section of the Great Wall of China (Ming Dynasty, circa 1368-1644 AD) where petrography has been used to investigate both the historic brick and lime mortar. The bricks were found to contain a temper of crushed granite and remarkably, the lime mortar consisted wholly of carbonated lime with no aggregate.

Petrographic examination is also useful for determining the cause and extent of deterioration. For example in the case of masonry structures, evidence of the common decay mechanisms (salt crystallisation, air pollution, frost action) are readily identified through the microscope and the depth of damage can be accurately determined. Types of surface soiling (e.g. salt crusts, particulates, biological growth) are also observed microscopically to enable soiling/substrate specific cleaning techniques to be selected.

Microscopy has been successfully applied to the archaeological investigation of built cultural heritage (Reedy, 2008). For example, in 1974, a radical theory was proposed that involved the casing stones of the Pyramids of Giza in Egypt being cast *in-situ* as geopolymer 'concrete', rather than being natural stone. A petrographic study (Jana, 2007) has recently disproved this theory. Microscopy indicated that cores of the Giza Pyramids were built from blocks of the local Eocene limestone (Mokattam Formation), while the majority of the casing stones were quarried at Tura on the opposite (East) side of the River Nile. Here, the same rock (Mokattam Formation) is finer-grained and it was for this quality (and its ability to form tighter joints) that it was selected for use as casing blocks. Some of the lower courses of casing blocks comprise granite from much further up the Nile at Aswan. Figure 9 illustrates examples of microscopical appearance of these stones with the core stone comprising fine-grained, buff/cream coloured biomicrite that consisted of 30-50% foraminifera microfossils (mainly Nummulites) and 50-70% micrite matrix. In contrast, the lighter coloured Tura limestone casing consists of sparse biomicrite that is finer grained with a much lower microfossil content. The stone from Aswan is red hornblende granite consisting chiefly of plagioclase feldspar, orthoclase feldspar and quartz with minor hornblende and biotite mica.

Conclusions

The application of microscopy to the construction industry is much more widespread than is commonly recognised. Having started as a fundamental tool for the investigation of rocks, microscopical techniques are now applied to the examination of a wide range of geomaterials. Petrographic examination utilising optical microscopy is a requirement for quality assurance of certain geomaterials-based construction products. A plethora of further applications exist within the realm of building and infrastructure maintenance, where petrographic examination is used for condition assessment and forensic engineering for structures. Microscopy also makes a valuable contribution to the conservation of historic buildings.

Acknowledgements

The author wishes to thank Barry Hunt of IBIS Ltd and Manson Publishing Ltd for generously contributing photographs for inclusion in this article and also Concrete Experts International ApS who gave permission to include photographs of their products.

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