Petrographic examination and SEM/EDX analysis of ultra-high performance concrete

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INTRODUCTION

Petrographic examination of concrete is routinely conducted to evaluate its constituents, assess uniformity and microstructure, and investigate causes of distress and unexpected behavior. Specimen preparation techniques that enable a petrographer to conduct a detailed microscopical investigation of concrete are well established. These techniques are often inadequate for preparing specimens of ultra-high performance concrete (UHPC), which has compressive strength greater than 117 MPa. UHPC is denser than most concrete and often contains metal fibers that complicate sectioning, grinding, and polishing. Specimen preparation methods capable of producing high-quality ultra-thin polished thin sections (less than 18 µm thick) without loss of aggregates and fibers are generally suitable for UHPC. Examination of these sections in reflected light, transmitted light, and with epi-fluorescence enables the petrographer to identify most constituents, study their distribution, and evaluate microstructure. Following petrographic study, the same specimen can be examined using scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDX) to identify remaining unknown components and analyze the micro-scale chemistry of the cementitious paste. The addition of SEM/EDX to the petrographic toolkit enables the petrographer to compare field samples with reference mixtures when these are available.

Petrographers are asked to examine UHPC for many of the same reasons they are instructed to examine any other concrete. Among these reasons are problems that develop during mixing and placement, concerns about constituents and proportions, and strength and durability concerns. In general, the tools and methods described in ASTM Practice C856 can be adapted for investigating UHPC. Stereomicroscope examination of lapped sections and polarized-light (petrographic) microscope examination of thin sections are essential elements of most petrographic studies. Modifications to sample preparation methods are usually needed if the concrete contains steel fibers. Care must be exercised to avoid damaging lapping wheels and grinding disks. Thin sections prepared to the normal thickness of 20 to 25 µm will likely be too thick and of limited use if the water-cementitious materials ratio (w/cm) is below 0.30. The presence of steel fibers can complicate the preparation of thin sections as the fibers can pull out during grinding and smear across the face of the

section. Surface impregnation with epoxy can improve the quality of the lapped section and the thin section. Colored or fluorescent epoxy can provide the petrographer with a limited view of the microstructure of the concrete. Many of the preparation techniques have been borrowed from ore microscopy and metallography.

BACKGROUND

UHPC, also known as reactive powder concrete, by definition exceeds the performance of HPC in these critical characteristics: freeze-thaw durability, scaling resistance, abrasion resistance, chloride penetration, compressive strength, modulus of elasticity, shrinkage, and creep. Due to its higher tensile strength, high abrasion resistance, low permeability, and other properties (Table 1), UHPC has been considered ideal for bridge decks, nuclear waste storage structures, and precast products such as beams, girders, pipes, and manhole covers. The principal obstacles to greater use of UHPC have been workability issues and its higher cost.

Table 1: Expected Properties of UHPC.

Property	UHPC
Compressive Strength	120 to > 150 MPa (17,000 to >20,000 psi)
Flexural Strength	15 to >25 MPa (2,200 to >3,600 psi)
Modulus of Elasticity	45 to 60 GPa (6,500 to 7,300 ksi)
Abrasion Resistance	1.7 (relative volume loss index)
Freeze-Thaw Durability	100% after 300 cycles
Salt Scaling	< 60 g/m^2 (<0.013 lb/ft^3)

Most UHPC mixtures contain some combination of portland cement, supplementary cementitious materials, reactive powders, limestone flour, quartz flour, fine sand, metal or polymer fibers, and HR high-range water reducers.

CHALLENGES AND SOLUTIONS

UHPC normally contains a large amount of fibers. Polymer fibers, metal fibers, and a combination of different types of fibers are common and the fiber content is generally substantial (Fig. 1). Careful handling during cutting, lapping, and other sample preparation is essential.

UHPC concrete is very dense. Thin sections prepared to the normal thickness of approximately 20 micrometers is usually too thick; overlapping of constituents obscures details. Various preparation methods have been explored by the authors. Ultra-thin polished thin sections have been found to be ideal for transmitted and reflected polarized-light analysis. Impregnation under vacuum with fluorescent epoxy allows the petrographer to examine microstructural details. Double polish, i.e. top and bottom polished surfaces, sharpens the details that can be seen with the petrographic microscope.

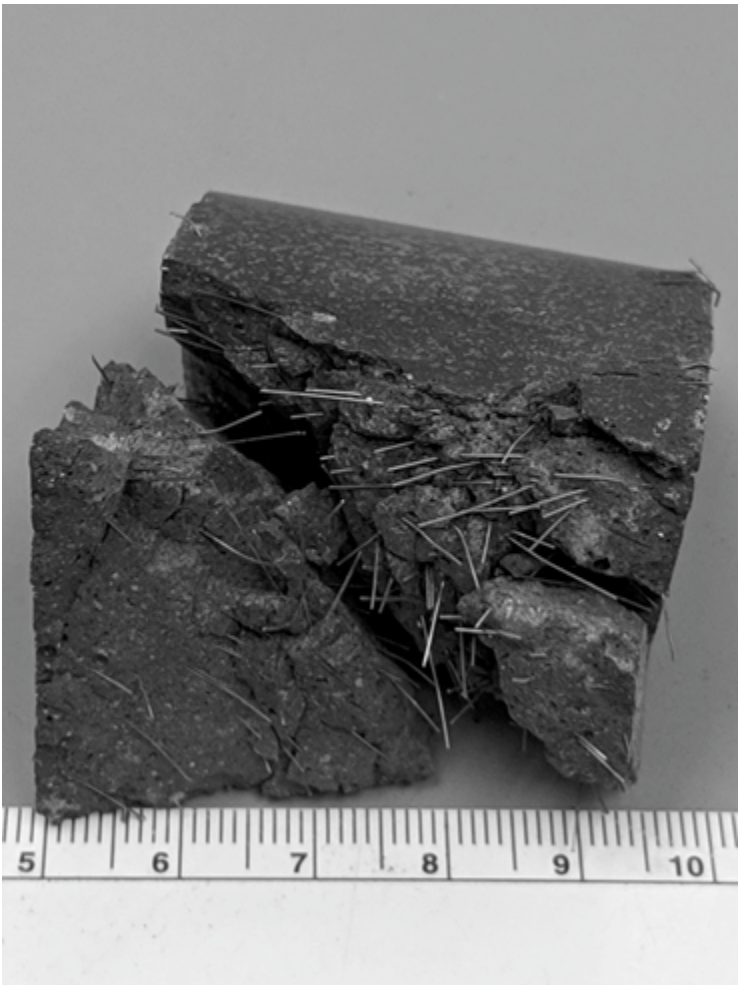

Figure 1: Metal fibers in UHPC

Some of the cementitious and inert constituents may be unfamiliar to the petrographer. Polished thin sections allow the petrographer to move seamlessly between the petrographic microscope and the scanning electron microscope, where EDX analysis can be performed to identify the constituents.

Well-made reference samples are essential when the petrographer is called upon to evaluate the composition of the field mixtures of UHPC. SEM viewing conditions and EDX acquisition parameters must be standardized for a specific mixture. Compositional analyses of the reference sample are averaged over a number of areas. Similar analysis is conducted on the field sample. Comparison of the results often

allows the petrographer to identify an excess or deficiency of some component of the field mixture.

Three case studies of UHPC investigations are presented and specimen preparation techniques are described.