Chapter 1

INTRODUCTION

1.1 BACKGROUND

Concrete is a heterogeneous, multiphase material. On a macroscopic scale, it is a mixture of cement paste, fine aggregates in a range of sizes and shapes, large aggregates in a range of gradations, and various types of void spaces. At the microscopic and submicroscopic levels, concrete is heterogeneous when the paste is observed to be a mixture of different types of crystalline structures, at varying degrees of hydration, which form an amorphous gel. Loss of water to the hydration of cement particles and evaporation, as well as entrained and entrapped air, cause voids to form in the heterogeneous mass. Examination of a cross section of concrete (Figure 1.1) reveals two phases that can be easily distinguished: aggregate particles of varying size and shape, and the binding medium which is composed of an incoherent mass of hydrated cement paste. At the macroscopic level, therefore, concrete may be considered to be a two-phase material, consisting of aggregate particles dispersed in a matrix of cement paste.

At the microscopic level, the complexities of the concrete structure begin to appear. It becomes obvious that the two phases of the structure are neither homogeneously distributed with respect to each other, nor are they themselves homogeneous. The structure of hydrated cement paste in the vicinity of large aggregate particles is usually very different from the structure of bulk paste or mortar in the system. Many aspects of concrete behavior under stress can be explained only when the cement paste-aggregate interface is treated as a third phase of the concrete structure. Therefore, in order to study the structural behavior of concrete, it is most helpful to view this complex mass as a three-phase composite structure: a coherent mortar phase and fine aggregate, bonded to an aggregate phase which is the coarse or large aggregate itself; and the transition zone which represents the interfacial region between the particles of coarse aggregate and the hydrated cement paste. The transition zone which exists as a thin shell, typically 10 to 50 μm thick around large aggregate, is generally weaker than either of the two main components of concrete, and it therefore imposes a far greater influence on the mechanical behavior of concrete than is reflected by its size.

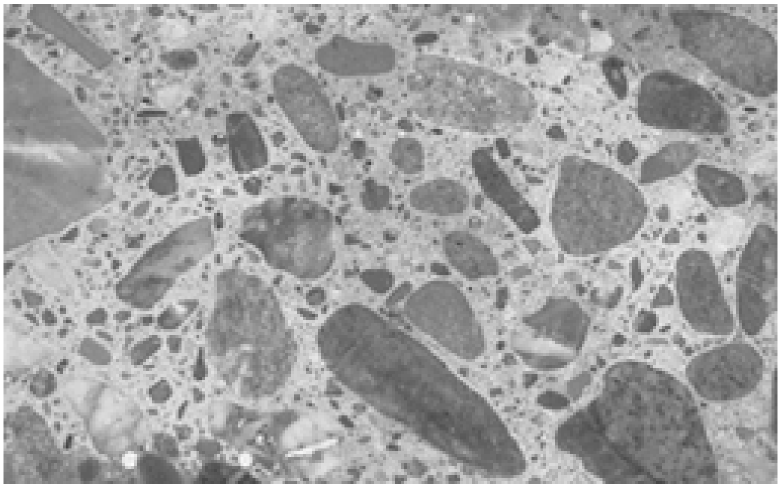

Figure 1.1 Polished section from a concrete specimen

In order to understand the structural characteristics of the transition zone, the sequence of its development is presented here as described by Maso (1980). In freshly compacted concrete, water films form around the large aggregate particles. This would account for the higher water/cement ratio that exists closer to larger aggregate than in the bulk mortar. Next, as in the bulk paste, calcium, sulfate, hydroxyl, and aluminate ions, produced by the dissolution of calcium sulfate and calcium aluminate compounds, combine to form ettringite (C-A-$\overline{\mathrm{S}}$-H) and calcium hydroxide (CH). Owing to a high water/cement ratio, these crystalline products in the vicinity of coarse aggregate consist of relatively large crystals, and therefore form a more porous framework than in bulk cement paste or mortar matrix. The platelike calcium hydroxide crystals tend to form in oriented layers, for instance, with the c-axis perpendicular to the aggregate surface. Finally, with the progress of hydration, poorly crystalline C-S-H and a second generation of smaller crystals of ettringite and calcium hydroxide start filling the empty space that exists between the framework created by the large ettringite and calcium hydroxide crystals. This helps to improve the density and hence the strength of the transition zone. Figure 1.2 is a diagrammatic representation of the transition zone in concrete.

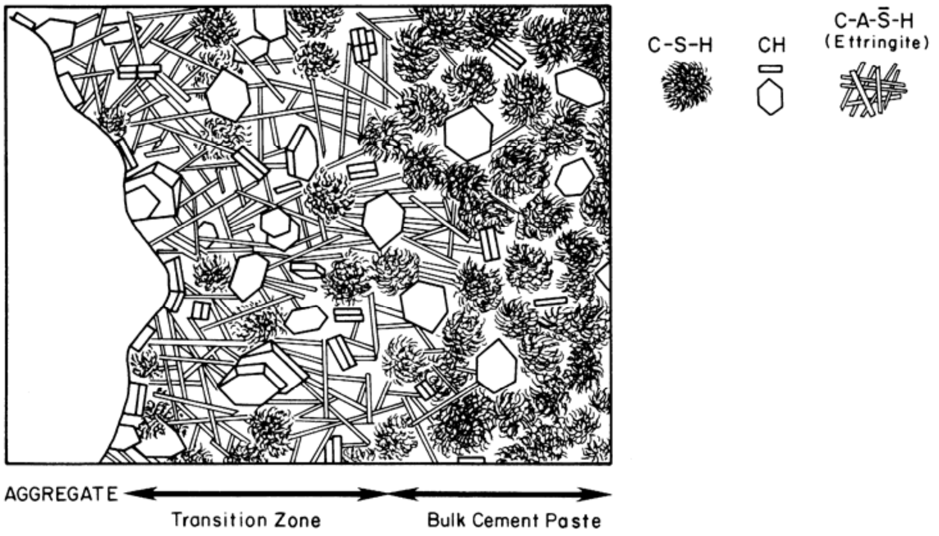

Figure 1.2 Diagrammatic representation of the transition zone and bulk cement paste in concrete (Mehta and Monteiro 1993)

In such a model, defects known as *microcracks* are imposed upon the three-phase composite. Bond cracks known as transition zone microcracks, may occur at mortar-aggregate interfaces, while mortar and aggregate cracks occur in the mortar and aggregate, respectively.

A quantitative connection between fracture stress and flaw size came from the work of Griffith (1920, 1924). He used the results obtained by Inglis (1913), and applied a stress analysis of an elliptical hole to the unstable propagation of a crack. According to the first law of thermodynamics, when a system goes from a nonequilibrium state to equilibrium, there will be a net decrease in energy. A crack can form (or an existing crack can grow) only if such a process causes the total energy to decrease or remain constant. Thus the critical conditions for fracture can be defined as the point where crack growth occurs under equilibrium conditions, with no net change in total energy (Anderson 1991). Griffith invoked the first law of thermodynamics to formulate a fracture theory based on simple energy balance. According to his theory, a flaw becomes unstable, and thus fracture occurs, when the strain energy change that results from an increment of crack growth is sufficient to overcome the surface energy of the material. Griffith's model correctly predicted the relationship between strength and flaw size in glass specimens. Since this model assumes that the work of fracture comes exclusively from the surface energy of the material, the Griffith approach only applies to ideally brittle solids.

Although the general relationships between stress level and crack development are reasonably well understood in concrete, the origin and development of cracks in concrete composite systems is still uncertain. In an effort to explore these issues, the theory of linear elastic fracture mechanics (LEFM) has been extended and applied to cement and concrete. These studies, however, have not been very successful; this may be because the observations they made about the way small cracks form and propagate in these materials were very limited.

Since the 1920s, researchers have suggested and assumed the existence of different kinds of defects that occur in concrete called microcracks (Richart 1929; Jones 1952; L'Hermite 1954; Hognestad et al. 1955; Czernin 1962; Hsu et al. 1963). However, only since the early 1960s have such cracks been carefully observed, measured, and characterized in interior portions of the system (Hsu 1963; Hsu and Slate 1963; Hsu et al. 1963; Slate and Olsefski 1963). These microcracks were verified and, to some extent, defined by microscopic study. This trend continued up until the middle 1970s when, finally, major advances were made. The contributions were based on the development of nonlinear fracture mechanics models, where the structure and behavior of concrete could be taken into account. In the 1980s and 1990s, intensive research has been performed. Applications of fracture mechanics in the design of beams, anchorage, and large dams are now becoming more common. In spite of this, the theory of fracture mechanics in concrete is not yet as mature as other continuum theories, such as elasticity, viscoelasticity, and thermal problems.

1.2 METHODS OF STUDY OF MICROCRACKING

The investigation of *microcracking* ranges from a macroscopic study of the behavior of cracked specimens to a microscopic study of the cracks themselves. The presence of microcracks was predicted on the basis of macrobehavior and verified by microscopic study.

1.2.1 Early Methods

Several methods have been used to study the microcracking of concrete. Some of those methods are as follows:

1.2.1.1 ***Acoustic emission.*** This method involves an investigator "listening" to the test specimen to detect the occurrence of internal cracking. The sophistication of the test depends on the type and sensitivity of the acoustic emission amplification, collecting, and recording equipment (Jones 1952; L'Hermite 1954; Hamstad 1986). An acoustic emission (AE) is a localized, rapid release of strain energy in a stressed material. AE is a microseismic wave generated by microcracking, dislocation movement, phase transformation, and other irreversible changes in a stressed material. These waves can be detected on the surface of the material by transducers which convert the mechanical acoustic vibrations to electric signals which are digitized, stored, and analyzed to obtain useful information about the AE events (Maji et al. 1990). Acoustic emission differs from most nondestructive methods in that the energy detected is released from within the test object rather than being supplied by an external source such as ultrasonic or radiography. Acoustic emission techniques are also capable of detecting the dynamic processes associated with the degradation of structural integrity (Ouyang et al. 1991).

1.2.1.2 ***Sonic testing.*** This method involves using resonant frequency to measure the apparent velocity of sound waves traveling through an elastic continuum. The velocity at which sound waves pass through an uncracked concrete specimen depends on the geometry and density of the concrete specimen. A crack in the specimen represents a discontinuity across which a sound wave cannot pass efficiently. Sound waves in a cracked specimen must pass around microcracks, thus traveling a further distance than in an uncracked specimen. The travel time for the cracked specimen is therefore greater than for the uncracked specimen (Whitehurst 1966). This method detects the presence of cracking by recording the increase in sound wave travel time in proportion to the increase in discontinuities in the elastic medium as the compressive load on the concrete specimen is increased. It has also been shown that the measurements of ultrasonic waves in mortar can be a powerful and reliable method of analyzing the elastic properties (Monteiro et al. 1988).

1.2.1.3 ***Microscope and X-ray techniques.*** These have been used at Cornell University since the 1960s to intensively study the interior cracks, as opposed to the surfaces, of concrete specimens. The following three procedures are representative of several methods studied at Cornell University involving radiant energy.

1.2.1.3.1 ***Hydrophilic tracer liquid technique.*** A hydrophilic tracer liquid, such as a fluorescent aqueous system, is allowed to penetrate cracks and voids in a slightly dried, cut, and polished surface. Afterwards the surface is lightly ground to remove the surface film of the tracer liquid. The cracks and voids shown by the tracer liquid which penetrated them can then be directly observed or photographed in a darkroom under ultraviolet radiation. The fluorescence method is useful, but cumbersome; it is not conducive to detailed, prolonged study such as can be done readily with the optical microscope (Wittmann 1983).

1.2.1.3.2 ***Dye technique.*** The bottom face of a slightly dried slice of ½-to 1-centimeter-thick concrete is placed in a container of an aqueous dye without being submerged. The capillary rise of the dye brings it to the top surface through the cracks. The colored cracks are then observed or photographed. This method does not work well because the internal surfaces of the cracks absorb most of the dye during the capillary rise. Additionally, many or most of the cracks may not be continuous for an appreciable distance (Wittmann 1983).

1.2.1.3.3 ***Lead salt.*** A partially dried, thin slice of concrete is subjected to the capillary rise of a saturated water solution of a lead salt. X-ray is then used to identify cracks and voids that were once relatively opaque (Wittmann 1983).

1.2.1.4 ***Microscope technique with dye.*** The clean, surface-dried face of a specimen 0.15 to 3 inches (4 to 75 mm) thick is painted with carmine drawing ink, which then penetrates the voids and cracks. After the ink becomes surface dry on all the paste portions, the inked surface is ground wet until only a faint pink color can be seen by the naked eye. Cracks and voids thus dyed contrast visibly with the rest of the surface of the specimen. A stereomicroscope is used to observe the cracks, which are then sketched or photographed as observed (Slate and Olsefski 1963; Hsu et al. 1963).

1.2.1.5 ***Mercury intrusion porosimetry.*** This entails using high pressure to infuse mercury into the concrete structure. This technique makes it possible to determine the size and quantity of void spaces and pores as well as particle size distribution. Information regarding the shape and structure of pores can also be obtained from the volume of mercury expelled from the pores as a function of decreasing pressure (Orr 1969).

1.2.1.6 ***X-ray technique.*** Since meaningful observations and studies of cracks must focus on cracks on the interior of the specimen rather than on the exterior surface, the method of using X-ray was developed for observation at depth within concrete. The first use of X-radiography to study cracks and other internal structural features of concrete was that developed and used by Slate and Olsefski (1963). They used an industrial X-ray unit, with a rating of 150 kilovolts (KV), and directly X-rayed thin specimens of concrete after cracks were induced, or after simply curing. Even relatively small variations in the thickness of specimens caused significant differences in the darkness of the X-ray film. Specimens that were too thick yielded too much detail, causing confusion in the interpretation of the X-ray plates, or loss of definition of very small cracks which extended only a small part of the way through the specimen. Specimens that were too thin sometimes had cracks induced by the sawing process. Later, thin (½ inch or 13 mm) plates of models of concrete were loaded under uniaxial and biaxial compression and in tension, X-ray film was placed under the specimens, and radiation was passed through the specimens to expose film after film during loading. This resulted in a nondestructive test which showed progressive cracking and failure, as described by Buyukozturk et al. (1971, 1972), by Liu et al. (1972), and by Carino and Slate (1976).

1.2.1.7 ***Computerized tomography analysis.*** γ-ray computerized tomography (CT) has been used as a nondestructive method to assess the degree of distress existing in reinforced concrete members (Martz et al. 1993). Different specimen configurations were employed to study the determination of voids and location of reinforcing bars. The objective of γ-ray CT is to reconstruct object absorption cross sections from projections through the object by using CT scanners. When the scanner cannot fully surround the reinforced concrete structure, the problem of limited-angle tomography can occur.

1.2.1.8 ***Fiber optics.*** Using a special device, optical fibers are used to detect the formation and propagation of one or more cracks in a material containing a hydraulic binder, such as concrete. The operation of the device is based on the finding that an optical fiber embedded in a piece of concrete breaks as soon as a crack, propagating in the material surrounding, it reaches it; and that this break causes an almost complete disappearance of the luminous signal transmitted by the fiber (Rossi and Le Maou 1989).

1.2.1.9 ***Holographic interferometry.*** In this method, reflection holographic interferometry is used to characterize the initiation and propagation of microcracks in cement-based composites (Mobasher et al. 1990). Holographic interferometry makes it possible to observe a large area of the specimen with no surface preparation required. The whole-field capacity of holographic interferometry can allow real-time observations of gradual curvature of the propagating crack with high accuracy (Maji and Shah 1990). Unlike optical microscopy, which measures the total number of cracks developed, holographic interferometry is an incremental-displacement measurement technique which makes it possible to differentiate between previously existing and active cracks, cracks that open or propagate, at any load level. Holographic interferometry technique has been used to determine the fracture process zone of mortar and concrete (Regnault et al. 1990).

Almost all of the above-mentioned techniques failed to render accurate representations of the geometry and state of microcracks as they exist under load. Some of the techniques are limited in their resolution, sensitivity in detecting cracks, and ability to make full-field observations. Other methods are incapable of examining the specimen while under load; or they require special preparation of the specimen, which alters its behavior.

1.2.2 Proposed Method

The proposed method involves the application of a metal in liquid phase, *Wood's metal*, which has a melting point below the boiling point of water, to preserve the microstructure of stress-induced microcracks in concrete. Used in conjunction with scanning electron microscopy (SEM), it has made possible the detailed observation of microcracks in concrete as they exist under load. Named after the astronomer who used an alloy of bismuth, lead, tin, and cadmium to create a perfect parabolic surface for astronomical observations, Wood's metal has been used in the past few years to study the microstructure of different materials. Yadev (1984) used Wood's metal to study pore fluid porosimetry and to measure contact areas and voids between the surfaces of natural fractures. Pyrak (1988) used Wood's metal to study the fracture of rocks. Zheng (1989) used Wood's metal to fill voids and microcracks in clastic rock specimens during loading and solidified it before unloading to preserve the microstructure in specimens under load.

This research expands the application of Wood's metal as a preservative of the microstructure of concrete under applied compressive stresses. In this study microcracks induced in concrete by applied stresses were preserved and identified, employing the method introduced by Zheng (1989), in order to understand the mechanisms by which stress-induced microcracks generate and interact.

Eight concrete cylinders-five normal-strength and three high-strength-each 8 inches (203 mm) long by 4 inches (102 mm) in diameter, were tested at the structural engineering laboratory of the University of California at Berkeley. Wood's metal was used to preserve the cracks induced in concrete by various stresses. After the metal was solidified, each of the cylinders was sectioned into eight specimens. The specimens were observed at the materials department of the Imperial College of Science and Technology at the University of London, using JEOL JSM-35CF scanning electron microscopy (SEM). Fifty-five images were taken from each specimen. The SEM images were then analyzed using an image analyzing system.

1.3 OBJECTIVES

The objectives of this investigation are as follows:

1. Examine the mechanisms for the generation of stress-induced microcracks in concrete under compression, and establish the effect of microstructure on the generation of stress-induced microcracks.

2. Determine the real shapes and geometry of stress-induced microcracks as they exist under load.

3. Assess how the density, length, orientation, localization, and behavior of microcracks depend upon the confining stresses.

4. Evaluate the interactions between microcracks and provide experimental information for micromechanical models, such as crack growth, under sustained load.

5. Determine the shapes of the stress-induced microcracks in three dimensions, and the relationship of microcrack planes to the boundary of the specimen.

6. Ascertain whether the microstructure or the stress conditions are the major factors that control the propagation of microcracks once they are generated.

7. Compare the experimental results obtained in this research with some of the existing micromechanical models and determine whether a simple numerical model could be used to simulate the interactive effect of existing microcracks on the location of new microcracks to be generated.

1.4 SUMMARY OF THE CHAPTERS

The chapters describing the work performed in this study were divided in such a way that they are predominantly self-contained. In order to help the reader to obtain a better understanding of the topics covered, a summary of Chapters 2 to 6 is presented below:

CHAPTER 2

This chapter explains the experimental technique used to preserve the compressive stress-induced microcracks in concrete. The components of the special test equipment that was developed for this research, as well as the concrete specimens, the metal alloy-Wood's metal-used to fill the voids and compressive stress-induced cracks in concrete, and the experimental procedure, are described in detail. Confinement and its effect on microcrack behavior is discussed. The experiments conducted and specimen preparation for scanning electron microscopy (SEM) study are discussed.

CHAPTER 3

The use of scanning electron microscopy (SEM) to extract images from the concrete specimens for different testing conditions is explained. The images rendered were analyzed using an image analyzer. The image analyzer identifies Wood's metal, which represents the crack network in concrete specimens. Special computer programs were developed to perform stereological and two-dimensional measurements on the stress-induced cracks in concrete. The scanning electron microscope and the computer programs used are described. Stereology deals with the interpretation of three-dimensional structures from their two-dimensional sections. Stereology attempts to characterize numerically the geometrical aspects of features in the microstructure, such as microcracks in concrete represented by Wood's metal. The basic measurements and parameters used in stereological analysis and the application of stereology to concrete fracture mechanics are described in this chapter.

CHAPTER 4

The data obtained from the image analyzer and the micrographs from the scanning electron microscope (SEM) are analyzed and examined quantitatively and qualitatively in order to determine the characterization of microcrack initiation; orientation; density; length; crack branching; and interfacial cracks.

CHAPTER 5

The results of the analysis performed in Chapter 4 are compared to some of the existing models of fracture mechanics for composite materials.

CHAPTER 6

Summary, conclusion, and recommendations for future work.