Chapter 2
EXPERIMENTAL TECHNIQUE

2.1 INTRODUCTION

The experimental technique described here makes it possible to preserve the compressive stress-induced microcracks in concrete as they exist under applied loads. The results of these experiments can be used to better understand and quantify the general relationship between stress level and crack development, as well as the effects of confinement on crack behavior: crack orientation, crack density, crack length, crack localization, crack branching, interfacial cracks, etc. They will also facilitate investigations into the way small cracks form and then propagate in concrete, thus making the application of fracture mechanics to cement paste and concrete more realistic.

Special test equipment was created to preserve the cracks under applied load. This was accomplished by injecting a molten metal into the induced cracks and solidifying it before unloading. The experiments carried out involved three procedures:

a) concrete casting and preparation,

b) crack induction, and

c) molten metal injection and solidification.

The second and third procedures were carried out simultaneously.

2.2 CONCRETE SPECIMENS

Five normal-strength and three high-strength concrete cylinders, 8 inches (203 mm) long by 4 inches (102 mm) in diameter, were cast using the mix designs shown in Tables 2.1 and 2.2. Both the normal-strength and high-strength concrete cylinders were cast at the Civil Engineering Materials Laboratory of the University of California at Berkeley.

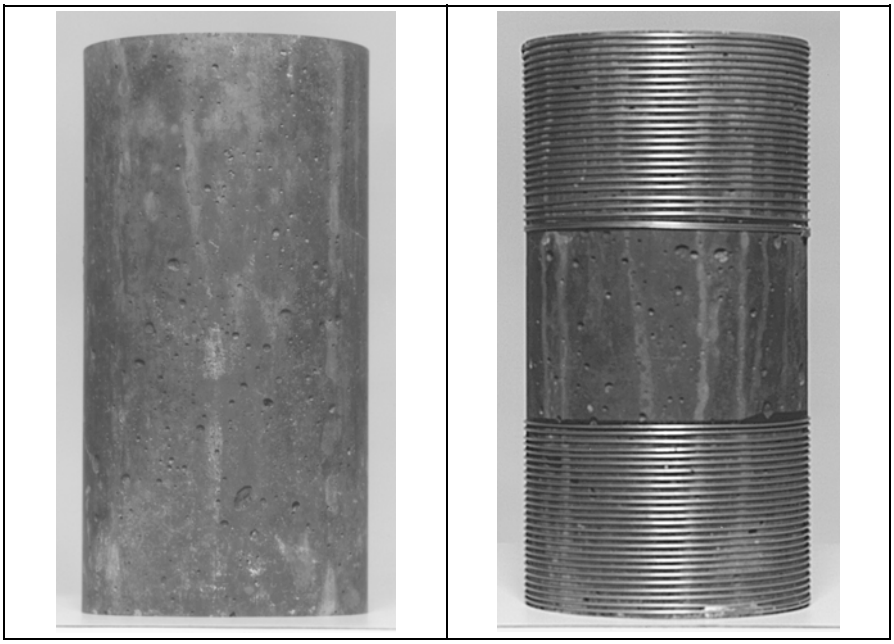
Table 2.1 Normal-strength concrete mix design

NORMAL-STRENGTH CONCRETE	
MATERIAL	QUANTITY/TYPE
Cement	583 pcy (346 Kg/m³)
Water	308 pcy (183 Kg/m³)
Coarse Aggregate (Pea Gravel)	1,650 pcy (979 Kg/m³)
Sand	1,448 pcy (859 Kg/m³)
HRWR Admixture	15 oz/100 lb. Cement Weight
W/C	0.528
Slump	1.5 inches (38 mm)
Date of Cast	November 16, 1989
Strength (on March 4, 1992)	7,500 psi (51.7 MPa)

Table 2.2 High-strength concrete mix design

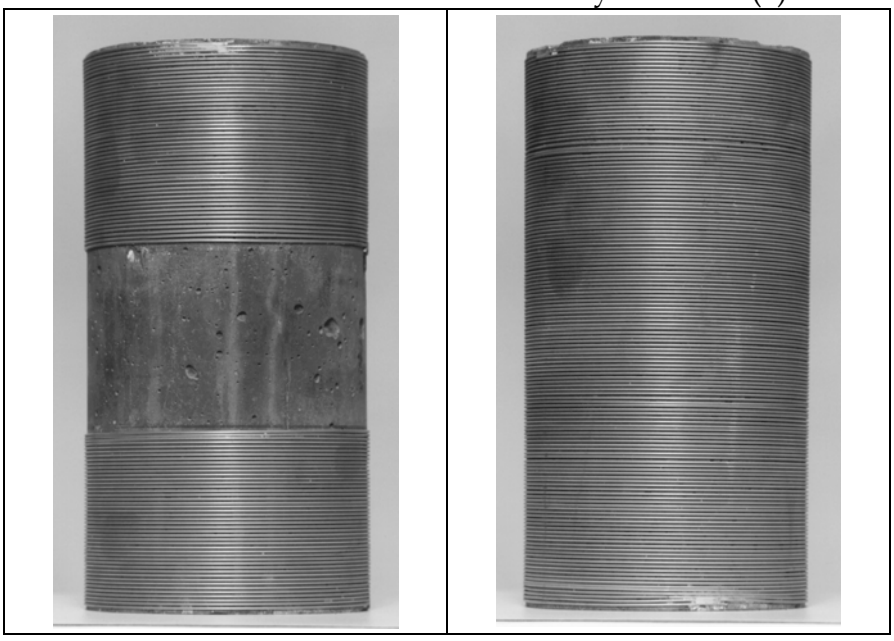
HIGH-STRENGTH CONCRETE	
MATERIAL	QUANTITY/TYPE
Cement Type I/II	600 pcy (356 Kg/m³)
Rice Husk Ash (RHA)	90 pcy (53 Kg/m³)
Crushed Limestone (3/8″ MSA)	1,760 pcy (1,044 Kg/m³)
Top Sand (FM=3.0)	1,325 pcy (768 Kg/m³)
Water	215 pcy (128 Kg/m³)
Superplasticizer Admixture	5.7 Liters/m³
W/C	0.358
Slump Unit Weight	1 inch (25 mm) 154 pcf (91 Kg/m³)
Date of Cast	October 30, 1991
Strength (28-day f_c')	11,000 psi (75.8 MPa)

The concrete cylinder ends were ground parallel to one another. Water was used as the cooling fluid during cutting and grinding. This procedure was performed at the Lawrence Berkeley Laboratory.



Experiments 1 & 2
No-load test

Experiment #3
Partially confined (1) test



Experiment #4
Partially confined (2) test

Experiment #5
Fully confined test

Figure 2.1 Normal-strength concrete specimens used in the experiments

Figure 2.1 represents the normal-strength concrete specimens used in Experiments 1 through 5. The high-strength concrete specimens used in Experiments 6, 7, and 8 resemble specimens used in Experiments 1, 2, and 3, respectively.

2.3 CONFINEMENT

The confining stress used to generate triaxial compression was supplied by stainless steel wires, 0.041 inch (0.3 mm) in diameter, that were wound around the concrete cylinders, both the entire length and one-third of the way from each end, at a pre-tension of 150 pounds (670 N). The purpose of the triaxial compression test was to observe the extensile microcracks generated under uniaxial compression in the middle of the concrete cylinder without causing unstable failure of the entire cylinder. The triaxial-compression test also eliminated the end effect caused by friction between the loading plates and the ends of the concrete cylinder because no failure occurred within the confined ends.

In a specimen subjected to uniaxial compression, most microcracks propagate to a certain length and stop. With the application of confining stress, the average length of the microcracks decreases. Coefficient α is the ratio of the incremental confining stress to the incremental axial stress and depends on the specimen diameter, elastic modulus, Poisson's ratio, and the pitch of the winding wire (Zheng 1989). Equation (2.1) is the expression for the coefficient α,

$$\alpha=\frac{\Delta\sigma_r}{\Delta\sigma_a}=\frac{\Delta\varepsilon_r n\pi R_W^2 E_W}{R_s\Delta\sigma_a} \tag{2.1}$$

and the confining stress σ_r itself can be expressed as:

$$\sigma_r=\frac{\varepsilon_r n\pi R_W^2 E_W + nT_W}{R_s} \tag{2.2}$$

where: ε_r = radial strain of the specimen

R_s = radius of specimen

σ_a = axial stress on the specimen

n = number of turns of wire per unit specimen length (pitch^{-1})

R_W = radius of wire

E_W = Young's modulus of wire

T_W = pre-tension on the wire.

Within the elastic range of the concrete the Poisson's ratio is constant. The ratio of $\frac{\Delta\varepsilon_r}{\Delta\sigma_a}$ can be shown as:

$$\frac{\Delta\varepsilon_r}{\Delta\sigma_a} = \frac{\Delta\varepsilon_r}{E_s\Delta\varepsilon_a} = \frac{\nu_s}{E_s} \tag{2.3}$$

Substituting Equation 2.3 in Equation 2.1, α can be expressed as:

$$\alpha = \frac{\nu_s n\pi R_W^2 E_W}{R_s E_s} \tag{2.4}$$

Multiplying the ratio in Equation 2.2 by E_s, it yields to:

$$\sigma_r = \frac{E_s\varepsilon_r n\pi R_W^2 E_W + nT_W E_s}{R_s E_s} \tag{2.5}$$

The Poisson's ratio of the specimen is:

$$\nu_s = \frac{\varepsilon_r}{\varepsilon_a} = \frac{\varepsilon_r}{\frac{\sigma_a}{E_s}} = \frac{E_s\varepsilon_r}{\sigma_a} \quad \text{or} \quad E_s\varepsilon_r = \nu_s\sigma_a \tag{2.6}$$

substituting Equation 2.6 in Equation 2.5, the confining stress can be shown as:

$$\sigma_r = \frac{\nu_s n\pi R_W^2 E_W\sigma_a + nT_W E_s}{R_s E_s} \tag{2.7}$$

where: ν_s = Poisson's ratio of the specimen

E_s = Young's modulus of the specimen (MPa or psi).

For the normal-strength concrete specimens:

R_s = 2 inches (51 mm)

σ_a = 6,375 psi (44.63 MPa)

n = 20 pitches/inch (8 pitches/cm)

$R_w = \frac{0.041 \text{ inches}}{2}$ = 0.0205 inches (0.5 mm)

E_w = 28×10⁶ psi (196 GPa)

T_w = 150 pounds (670 N)

ν_s = 0.15-0.20 (use 0.175)

E_s = 3.5×10⁶ psi (24,500 MPa)

Substituting the above values into Equation 2.7, σ_r can be computed as:

$$\sigma_r = \frac{(0.175)(20)\pi(0.0205 \text{ inch})^2(28\times10^6 \text{ psi})(6375\text{psi}) + (20)(150 \text{ lbs})(3.5\times10^6 \text{ psi})}{(2 \text{ inches})(3.5\times10^6 \text{ psi})}$$

or

$$\sigma_r = \frac{82.5\times10^7 + 1050\times10^7}{7\times10^6} \approx 1,620 \text{ psi (10.8 MPa)}$$

The maximum compressive stress, σ_p, under biaxial loading, is a function of the principal stress ratio $\alpha = \sigma_r / \sigma_a$ and the uniaxial compressive strength f_c' (Chen 1982). The values of maximum stresses in the two principal directions σ_{rp} and σ_{ap} are obtained from the modified-biaxial-strength envelope of Kupfer and Gerstle (1973). In the compression-compression region (σ_a=compression, σ_r=compression, $0 \le \alpha \le 1$):

$$\sigma_{ap} = \frac{1+3.65\alpha}{(1+\alpha)^2} f_c' \tag{2.8}$$

and

$$\sigma_{rp} = \alpha\sigma_{ap} \tag{2.9}$$

Therefore:

$$\alpha = \frac{\sigma_r}{\sigma_a} = \frac{1,620\,(\text{psi})}{6,375\,(\text{psi})} = 0.25$$

substituting for α and f_c' in Equation 2.8:

$$\sigma_{ap} = \frac{1 + 3.65(0.25)}{(1 + 0.25)^2}(7{,}500\text{psi}) = 9{,}180\text{psi (61.6 MPa)}$$

(20% increase) and from Equation 2.9,

$$\sigma_{rp} = (0.25)(9{,}180\text{ psi}) \approx 2{,}295\text{psi (15.4 MPa)}$$

2.4 TEST EQUIPMENT

The equipment used for this research was specially designed and developed at the University of California at Berkeley. It consists of five pieces: pedestal, vessel, piston, top cap, and heater. Each piece is described and illustrated below. For detailed design drawings refer to Appendix 3.

2.4.1 Pedestal

Made of graphatized steel, the pedestal is a monotonic, solid cylinder with a circular plate, the diameter of which is greater at the middle, as shown in Figure 2.2. The cylinder is 5 inches (127 mm) long and 4.25 inches (108 mm) in diameter, except for the upper 1 inch (25 mm) which has a diameter of 4 inches (102 mm). The circular plate has a thickness of 1 inch (25 mm) and a diameter of 7.25 inches (184 mm). The vessel is placed on the circular plate. There is an o-ring groove on the cylinder above the circular plate to provide an airtight system after assembly. There are six equally spaced ½-inch (12.7 mm) through holes on the circular plate to accommodate bolts that secure the vessel to the pedestal.

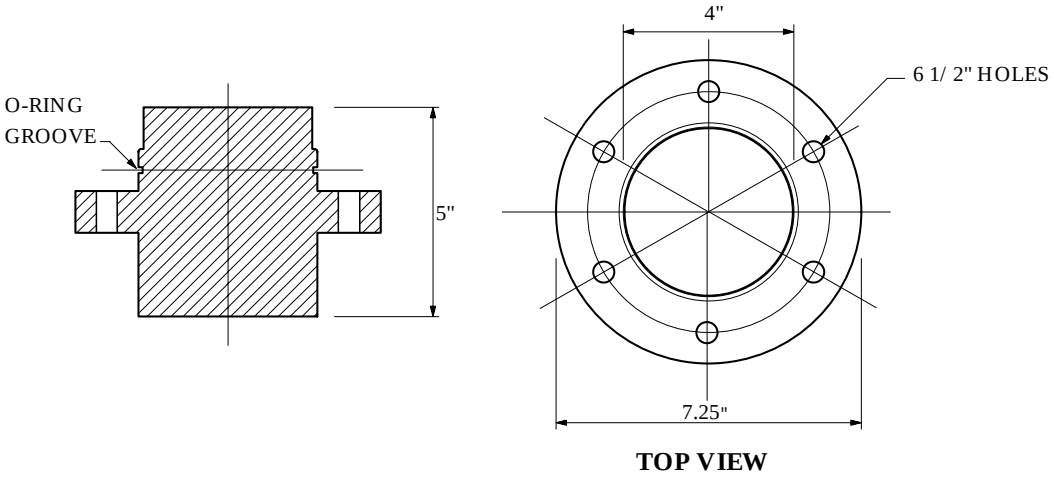


Figure 2.2 Pedestal

2.4.2 Vessel

Made of high-strength steel, the vessel is a hollow cylinder with an outside diameter of 7.25 inches (184 mm) and an inside diameter of 4.25 inches (108 mm). The diameter was enlarged to 5.25 inches (133 mm) at the top to provide a reservoir for the molten metal alloy at the time of testing. It is 11.75-inches (298 mm) tall. There are six 1½-inch (38-mm)-deep bolt holes with a diameter of ½ inch (12.7 mm) on top and bottom of the vessel in order to secure the pedestal and the top cap to the vessel. Close to the top of the vessel, there is a 45-degree, 1/8-inch (3 mm)-diameter hole with a plug at the end of it in order to facilitate vacuum and nitrogen connection to the airtight assembly (Figure 2.3). The inclination of the hole facilitates the drainage of Wood's metal into the reservoir should the molten metal freeze due to a premature drop in temperature or some other problem occur while the experiment is in progress. This prevents clogging.

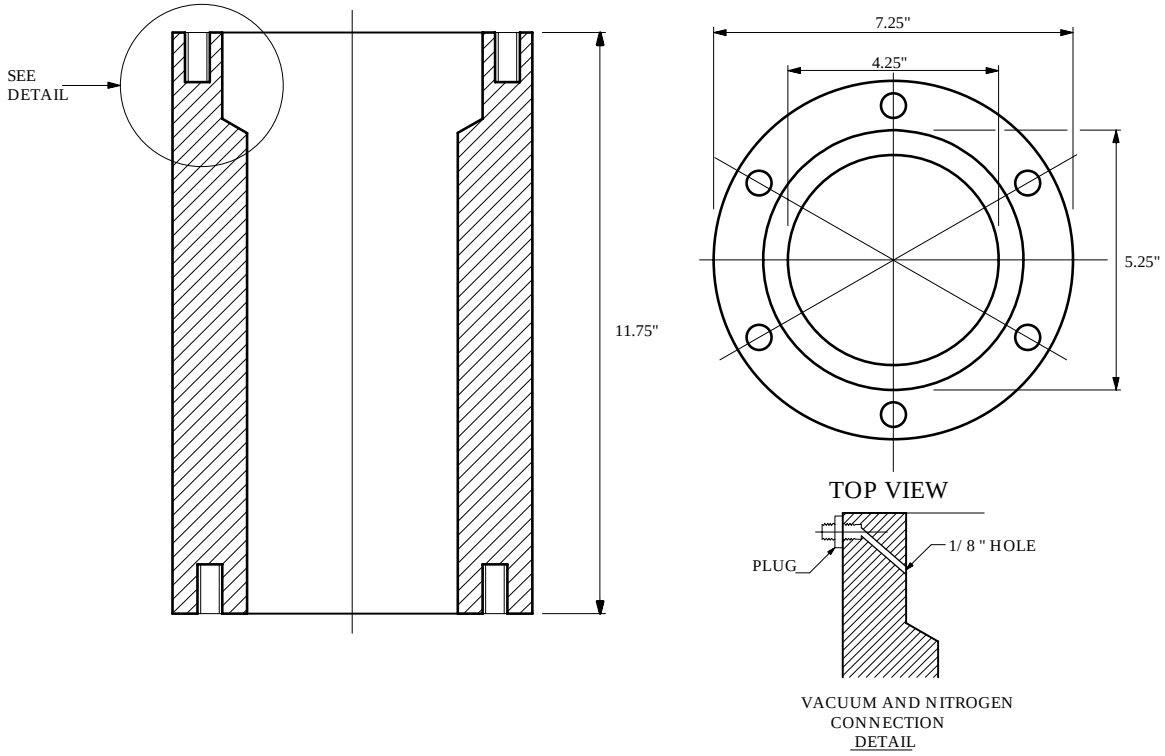


Figure 2.3 Vessel

2.4.3 Piston

Made of high-strength steel, the piston is a 4-inch (102 mm)-diameter, 5-inch (127 mm)-tall solid cylinder which is placed on top of the concrete specimen and

surrounded by the top cap. The piston transfers the applied compressive stresses from the loading machine to the concrete specimen (Figure 2.4).

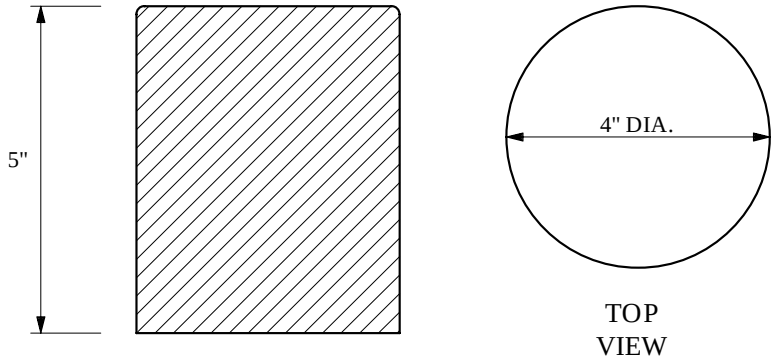


Figure 2.4 Piston

2.4.4 Top Cap

Made of galvanized steel, the top cap is a hollow donut-shaped cylinder with two tiers and an inner radius of 4 inches (102 mm). The top tier is 1-inch (25 mm) thick and has an outer radius of 7.25 inches (184 mm). The bottom tier is ½-inch (12.7 mm) thick and has an outer radius of 5.25 inches (133 mm). As with the pedestal, there are six equally spaced ½-inch (12.7 mm) through holes on the upper tier of the top cap to accommodate the bolts which secure it to the vessel. There are also two o-ring grooves on the inner and outer faces of the bottom tier (Figure 2.5).

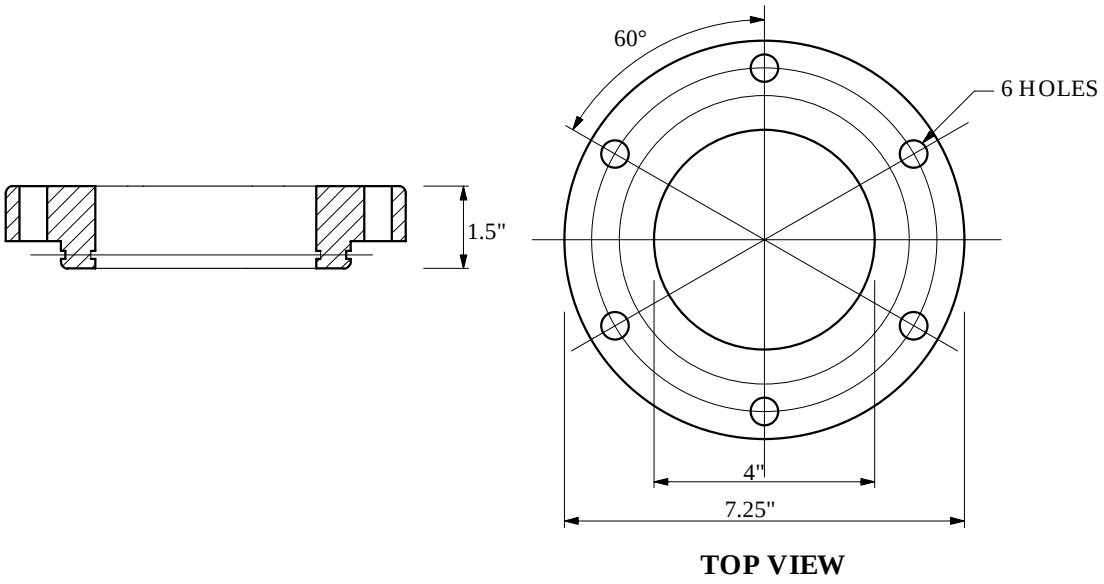


Figure 2.5 Top Cap

Once the entire system is assembled, with the concrete cylinder resting on the pedestal, the top cap closed, and the plug connected to either vacuum or nitrogen, the system is completely airtight.

2.4.5 Heater

The heat was supplied by a ceramic heater operated by a microprocessor-based ramping temperature control unit (Figures 2.11 and 2.13). The ceramic heater was assembled inside two stainless steel half-cylinders with an 18-inch (457 mm) outside diameter and 12-inch (305 mm) inside diameter. The half-cylinders were hinged together on one side and could be made into a monotonic unit by nuts and bolts on the other side of it.

2.5 WOOD'S METAL

Wood's metal, whose commercial name is Cerrosafe®, is a fusible alloy. In the liquid phase it is nonwetting, with an effective surface tension of about 400 mN/m (Yadev et al. 1984). It consists of 42.5% bismuth (Bi), 37.7% lead (Pb), 11.3% tin (Sn), and 8.5% cadmium (Cd). It has a melting point range from 160°F to 190°F (71.1°C to 87.8°C) below the boiling point of water, and is solid at room temperature. Wood's metal has a Young's modulus of 9.7 GPa and a density of 9.4 g/cm^3. The advantage of such an alloy is that it can be injected into voids and stress-induced microcracks at the desired stress level, then solidified at any stage of the experiment to preserve, in three-dimensional form, the geometry of the microcracks induced at any given stage of the experiment. Figure 2.6 shows Wood's metal in solid and molten modes.

Figure 2.6 Wood's Metal

2.6 EXPERIMENTAL PROCEDURE

Two different loading conditions, uniaxial and triaxial, were used to induce cracks in the concrete cylinders. Triaxiality was provided by the wire wound around the concrete cylinders.

After preparation for testing, each concrete cylinder was first dried in an oven at a temperature of 110°F (43.3°C). This removed the moisture in the concrete and preheated the cylinder, ensuring that the molten metal alloy could penetrate into pores and cracks deep within its core without solidifying prematurely. The concrete cylinder was then placed on the pedestal inside the vessel and the piston was placed on top of it. The top cap was left open resting on three wedges a short distance from the top of the vessel so that the molten metal could be poured through the gap using a funnel (Figure 2.10). At this point, a minimum load was applied to the piston to prevent the cylinder from floating after the Wood's metal was poured in. Once the concrete cylinder was submerged in the molten metal the top cap was dropped by removing the wedges and then bolted tightly to the vessel. Figure 2.7 shows a schematic diagram of the test apparatus. To monitor the temperature, a thermocouple was inserted into a predrilled hole on the top cap. The heater was then placed around the assembled system with a special noncombustible board placed on top to prevent heat convection and thus uniform heating of the test assembly (Figure 2.12).

The heat was supplied in three stages. Starting at room temperature, the heat was ramped up to 122°F (50°C) and held at that temperature for 10 minutes. Then the temperature was ramped up to 167°F (75°C) and held there for an additional 10 minutes. The final stage involved ramping the temperature up to a target of 205°F (96°C) for a period of 15 minutes and holding it at that temperature until the heat was no longer needed. Figure 2.8 shows the heating scheme diagram.

When the test assembly was thus heated, Wood's metal was poured into the vessel to a level above the top end of the concrete cylinder to form a metal reservoir with the concrete cylinder totally submerged inside. The top cap was then immediately closed and the thermocouple reinserted to the side of the top cap. A LVDT (linear variable displacement transducer) for the axial displacement measurement was attached to the loading frame. The ceramic heater was placed

around the vessel to liquefy the Wood's metal inside and to maintain a constant temperature throughout the experiment. This temperature was, in turn, monitored by a thermocouple that was attached to the side of the top cap. Figure 2.9 shows a schematic setup for the testing.

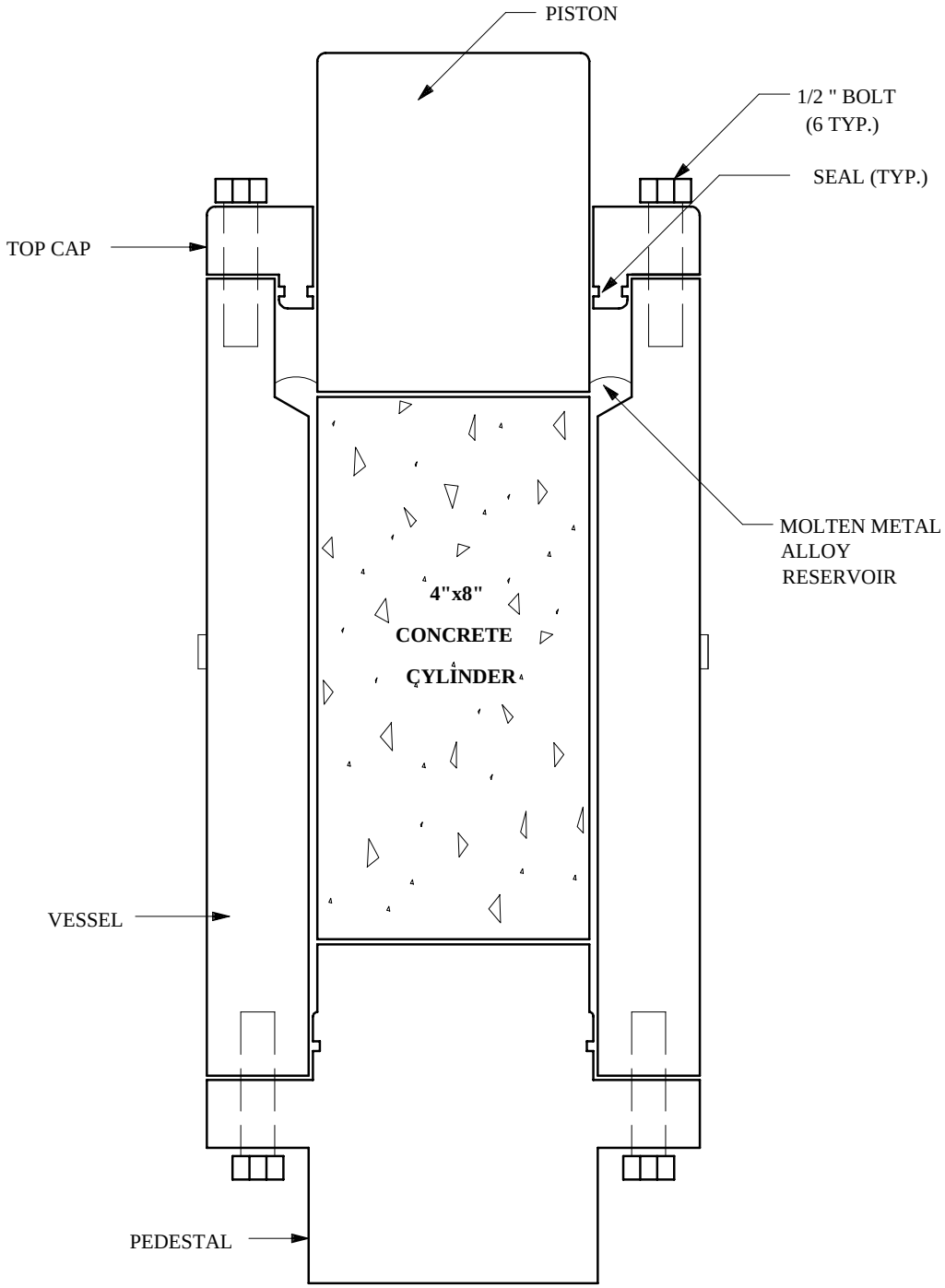


Figure 2.7 Diagram of the test apparatus

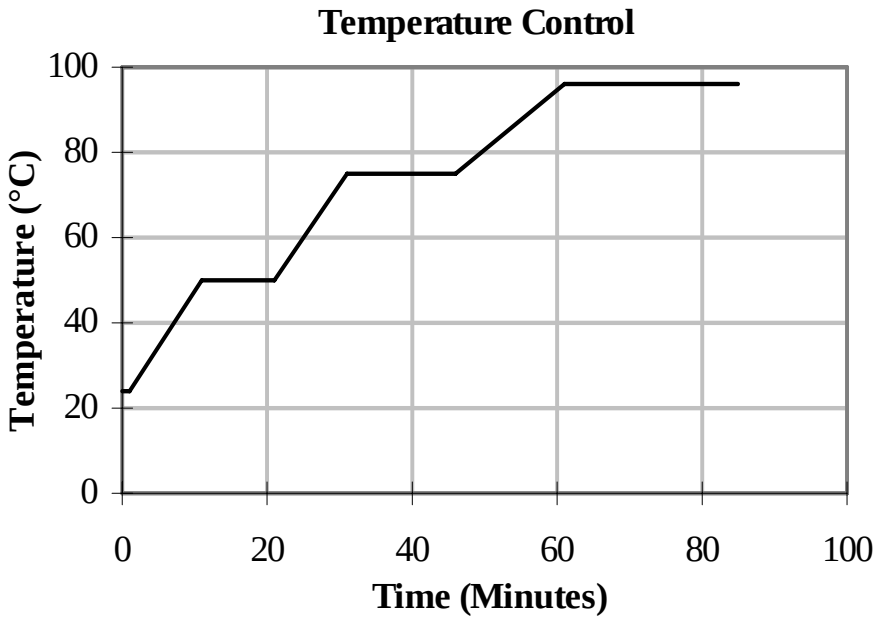


Figure 2.8 Heating scheme diagram

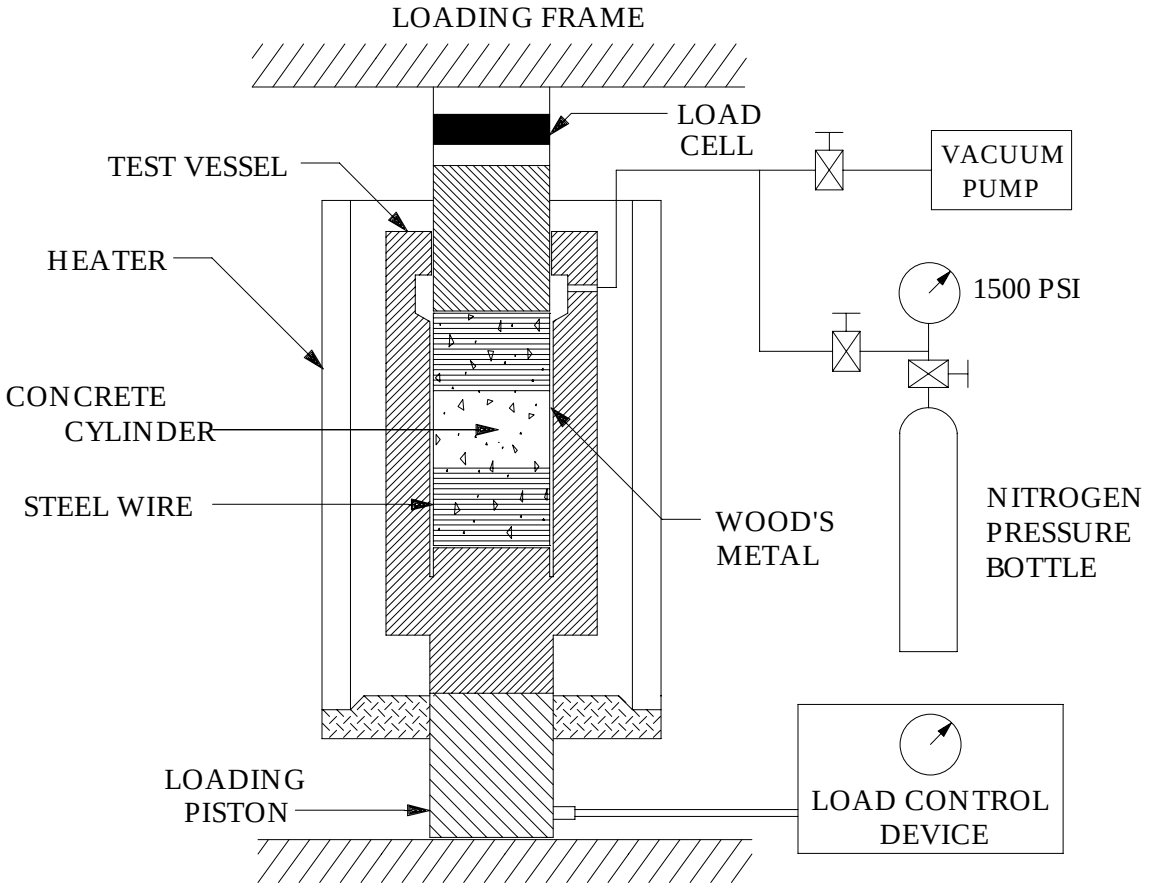


Figure 2.9 Schematic diagram of the test assembly

With the internal temperature thus established and maintained at 96°C (205°F), a vacuum was applied to the vessel and kept constant for at least 30 minutes. The vacuum removed any air that had become trapped in the concrete cylinder when it was assembled inside the vessel. An axial stress of up to 90% of the ultimate strength was applied to the concrete cylinder, at which point the vacuum was removed.

Finally, in order to saturate the induced microcracks with the molten metal, nitrogen pressure was applied to the top of the vessel. It was controlled by a high-pressure regulator on a bottle of nitrogen (Figure 2.14). A nitrogen pressure of 1500 psi (10.3 MPa) was applied to the molten metal as the pore pressure, which was kept constant throughout the tests and which did not alter the effective stresses on the concrete cylinder. With a surface tension of 400 mN/m, the alloy could penetrate into flat cracks with apertures as fine as 0.08 microns.

Throughout the period of loading and unloading, the axial load and axial displacement were recorded on a data acquisition system and monitored on an X-Y plotter. The axial stress of interest was kept constant for 2 hours to allow the liquid metal to penetrate into pores and fractures. Afterwards, fans were used to cool the vessel down to room temperature and to expedite solidification (Figure 2.15). Approximately 3 hours elapsed between the time pore pressure was applied and the period during which the metal was allowed to solidify. Figures 2.10 through 2.13 illustrate some aspects of the experiments.

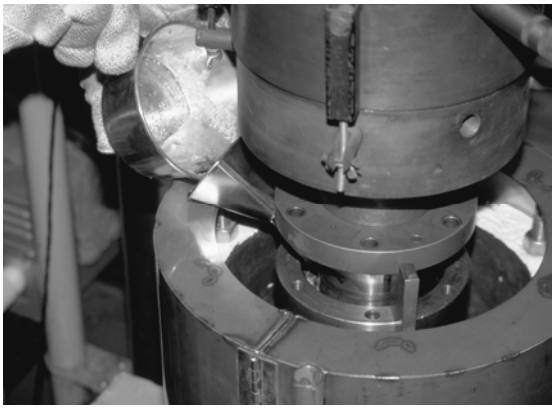


Figure 2.10 Pouring molten metal into the vessel

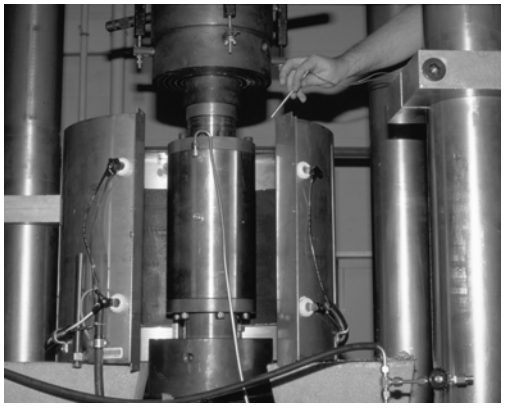


Figure 2.11 Inserting the thermocouple into the top cap

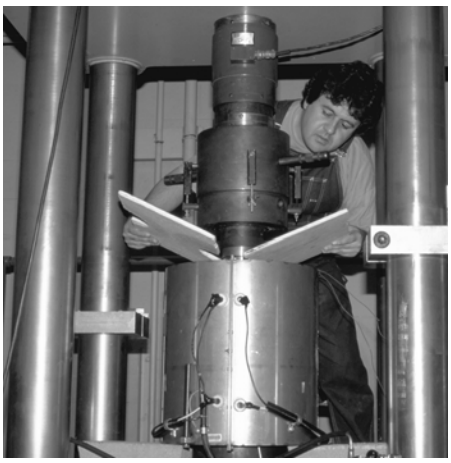


Figure 2.12 Placing the heater and the insulation

Figure 2.13 Temperature and load control devices

A step-by-step explanation of the experimental procedure is outlined below:

1. The hydraulics of the Universal Testing Machine are activated.
2. Piston of cell engages swivel head. Top cap and pedestal bolts are tightened. 100 psi (0.7 MPa) axial load is applied.
3. The head remains unlocked during heating.
4. Feedback thermocouple is positioned in top cap (Figure 2.11).
5. Vacuum line is attached with bleed valve open.
6. M-board is placed in position (Figure 2.12).
7. Heater is activated to 50°C, soak, 75°C, soak, 96°C, soak (Figure 2.13).
8. Vacuum is applied.
9. Heater is operated for 100-120 minutes.
10. Specimen is loaded to 85-90% of f_c', is held under load control.
11. Vacuum is changed to nitrogen at 1,500 psi (10.3 MPa) (Figure 2.14).
12. Heater is turned off and removed.

13. Fans are turned on (Figure 2.15).

14. Specimen is cooled for at least 70 minutes.

15. Nitrogen pressure is dropped.

16. Specimen is unloaded.

17. Bolts are broken loose from top cap and pedestal.

18. Head of machine is positioned so as not to bang human head.

19. Top cap and piston are removed using T-handle allen wrench.

20. Cell is inverted and pedestal bolts removed. (Caution: nitrogen pressure remains high below sample until o-ring is fully exposed.)

21. Pedestal is removed.

22. Aircraft cable is used to lift cell (Figures 2.17 & 2.18).

23. Spacer is used to elevate cell (Figure 2.16).

24. Head is locked, specimen extruded (Figure 2.19).

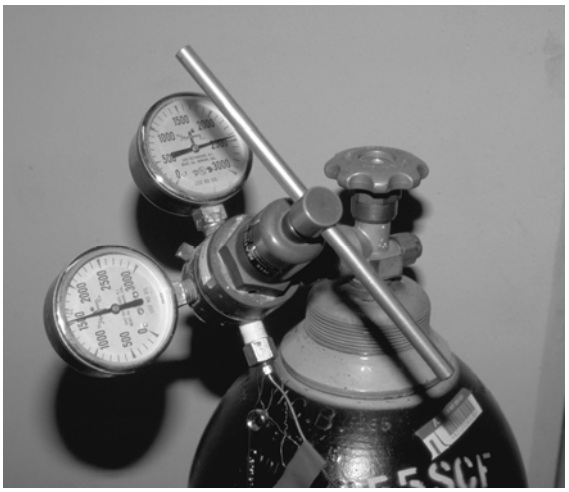


Figure 2.14 Nitrogen dispensed through a high pressure regulator

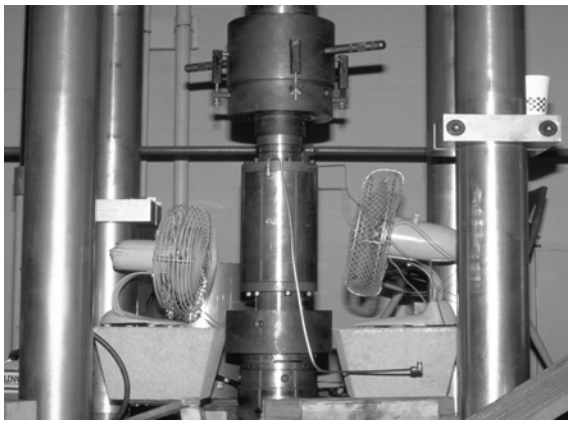


Figure 2.15 Post-experiment cooling of the cell

Figure 2.16 Extrusion of concrete specimen

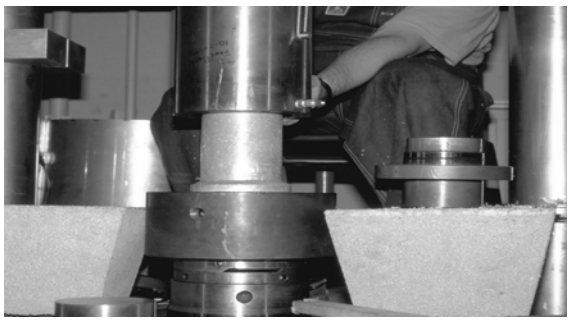


Figure 2.17 Raising the vessel

Figure 2.18 Raising the vessel by aircraft cable

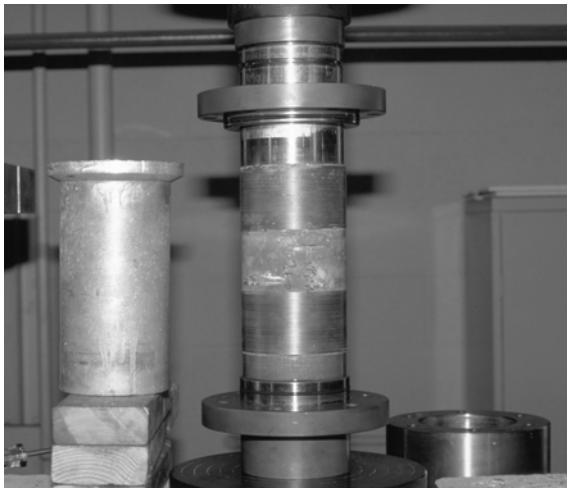


Figure 2.19 Specimen prior to and after Wood's metal injection

2.7 EXPERIMENTS CONDUCTED

A total of eight experiments, five on normal-strength and three on high-strength concrete cylinders, were conducted with conditions as defined in Table 2.3:

Table 2.3 Experiments conducted

Test	LOADING CONDITION	CONCRETE TYPE
Experiment 1	No-load	Normal-Strength
Experiment 2	Uniaxial	Normal-Strength
Experiment 3	Partially Confined	Normal-Strength
Experiment 4	Partially Confined	Normal-Strength
Experiment 5	Fully Confined	Normal-Strength
Experiment 6	No-load	High-strength
Experiment 7	Uniaxial	High-strength
Experiment 8	Partially Confined	High-strength

Complete information on concrete strength, confinement conditions, applied stresses, and drying data on each experiment is provided in Appendix 1.

2.8 SPECIMEN PREPARATION

2.8.1 Cutting

After each experiment, the 8-by-4-inch concrete cylinder was sectioned along its long axis, using oil to cool the cutting saw (Figure 2.20). Then one of the half-cylinders was sectioned at the middle, along its diameter (Figure 2.21). An axial slab, approximately 1/8-inch thick, was sliced parallel to the direction of the load. Two half-circle slabs of similar thickness, one from the top and one from the bottom of the quarter-cylinder, were sliced as well. Figure 2.23 shows a cross section of a concrete cylinder cut along its diameter.

Figure 2.20 Axial cut of concrete cylinder

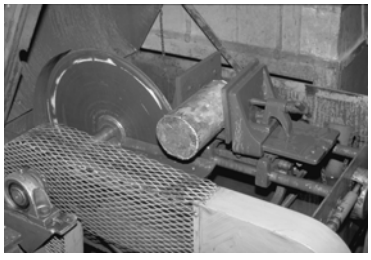


Figure 2.21 Lateral cut of concrete cylinder

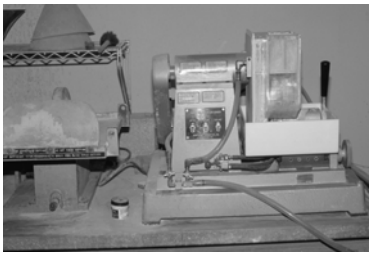


Figure 2.22 Specimen extraction

Four specimens were extracted from the axial slab (specimens 1, 2, 3, and 4). Specimens 5 and 6 were extracted from the top lateral slab, and specimens 7 and 8 were taken from the bottom lateral slab (Figure 2.25). The next step was to polish the specimens for the SEM study.

2.8.2 Polishing

The concrete specimens extracted from the axial and lateral slabs were 1-inch (25 mm) square and had an approximate thickness of 5 mm. First, one side of each specimen was polished with 120#, 220#, 320#, and 600# silicon carbide using a rotating grinder and mounting it against a 1-inch (25.4 mm)-diameter glass plate with epoxy. In order to make both sides of the specimen parallel to each other, the samples were cut 2-3-mm-thick by using a diamond slicing wheel with a nonaqueous lubricant (propylene glycol coolant) (Figure 2.22). The specimens were then lapped (grinding on a flat surface) with a wheel grinder and polished with 600# silicon carbide. Further polishing was performed with 100-, 50-, and 10-micron aluminum powder on a glass plate. The final stage involved was treating specimens with 5-, 3-, and 1/4-micron diamond paste using a special polishing equipment (ASTM 1993). After each stage of polishing, the specimens were immersed in acetone and placed in an ultrasonic machine in order to remove the residual silica film on their surfaces, thus preparing them for the next stage of polishing.

Figure 2.23 A lateral section showing macrocracks filled with metal alloy

Figure 2.24 Specimen placed on glass plate and aluminum base for SEM study

2.8.3 Specimen Identification

All the experiments conducted are identified by a number; for example, Experiment 5 is the triaxial-compression test on a normal-strength concrete cylinder, with continuous wire confinement over the entire length of the cylinder at a pitch of 20 threads per inch. The specimens are identified by two sets of numbers. The first number refers to the experiment number, i.e., 1 through 8, and the second number indicates the specimen location shown in Figure 2.25.

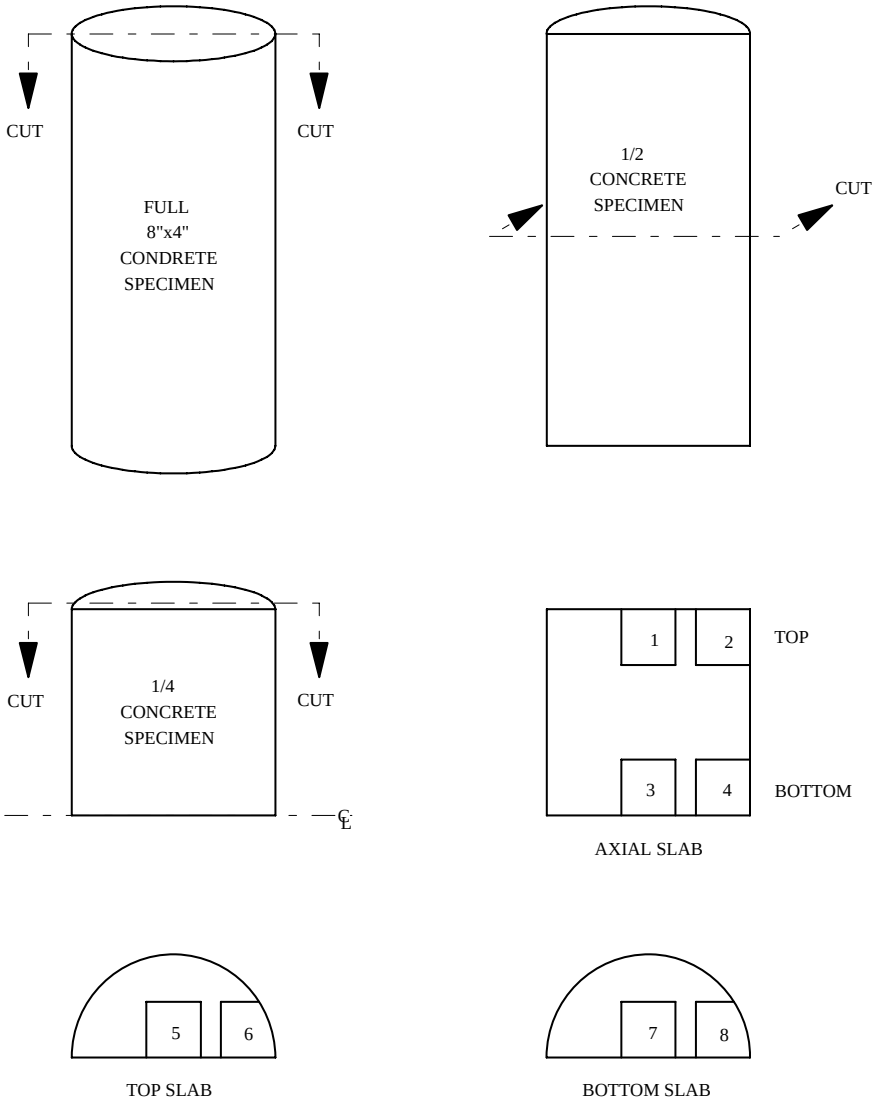


Figure 2.25 Specimen extraction and numbering scheme

2.8.4 Preparation for Scanning Electron Microscope (SEM) Analysis

After all the specimens were thus prepared, and prior to observation by SEM, they were gold coated. The single most important reason for coating or increasing the bulk conductivity is to increase the electrical conductivity of the sample. Materials of high resistivity, such as concrete, charge rapidly under the incident beam and might develop a potential sufficient to cause a dielectric breakdown in regions of the specimen. This could lead to variations in the surface potentials, giving rise to the complex and dynamic image artifacts commonly referred to as *charging*. A suitable conducting path may be established with a thin coating layer of gold which eliminates the problems associated with charging (Goldstein et al. 1992). Once the gold coat is in place, the specimen is then ready to be put into the scanning electron microscope (SEM).