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MECHANICAL PROPERTIES OF VARIOUS POLYMER
IMPREGNATED CONCRETE MATERIALS

by

Einar Dahl-Jørgensen

A Thesis

Presented to the Graduate Committee

of Lehigh University

in Candidacy for the Degree of

Master of Science

in

Civil Engineering

Lehigh University

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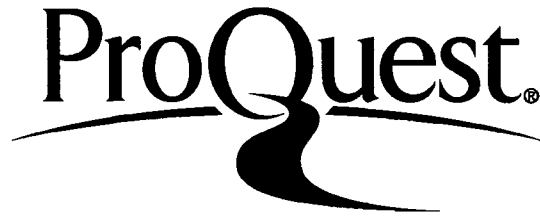
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Sept. 29, 1975
(date)

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Professor in Charge

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Chairman of Department

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preparation of the photographs and to Shirley M. Matlock for her care

in typing the manuscript.

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ABSTRACT

Polymer impregnation of concrete with for example, MMA (methyl methacrylate) will increase the strength up to 300 percent. Better polymerization will be achieved with azobisisobutyronitrile than with benzoyl peroxide which left a central core of monomer unpolymerized.

It was also shown that stress-strain behavior could be varied over a wide range, from ductile to brittle, by using combinations of plasticizing (n-butyl acrylate) or cross linking (TMPTMA) monomers with MMA, or both.

The pressure of a realistic level of salt (up to 1 percent) in concrete has little effect on polymer loading and mechanical properties, but requires more rigorous drying.

While high temperatures accelerate drying but decrease strength, subsequent polymer impregnation essentially yields a PIC with properties similar to a conventionally dried material.

Sulfur impregnated concrete (SIC) offers an inexpensive substitute for PIC, but requires higher pressure and a controlled, high temperature level for successful impregnation. On the other hand the polymerization step can be eliminated. Impregnation, through cast-in perforated pipes offers an enclosed, simple system where the fire hazard is reduced to a minimum. It is well suited for large size, cast-in-place concrete structures, such as bridge decks, although the strength and polymer loading might be less than normally

encountered with laboratory impregnation.

1. INTRODUCTION

Of the many new composites, one of the most interesting and potentially useful in civil engineering applications is polymer-impregnated concrete (PIC). The concrete is prepared and cured as usual, followed by drying to remove the residual water in the pore system. The next steps include impregnation with a monomer followed by polymerization to form within the pores a composite comprised of two interpenetrating networks, polymer and cement.

Brookhaven National Laboratories, Upton, New York, have demonstrated that PIC laboratory specimens show remarkable mechanical properties and corrosion resistance (1,2). These results have been confirmed by studies of PIC in other laboratories (3,4,5,6,7).

A number of questions, however, appeared after experimentation continued in various laboratories. Those included inadequate polymerization of the monomer in the pore system, the brittle behavior of PIC, the expense of impregnation with MMA (methyl methacrylate) and the problems of impregnating contaminated, existing structures such as bridge decks.

Some of these problems have been investigated in this thesis and are described in the following chapters.

2. PREPARATION AND TESTING PROCEDURES

Since for most of the experiments the fabrication of the specimens, the impregnation equipment, and the testing machines used were the same or only slightly modified, they will be described separately as an introduction to the actual experiments.

2.1 Preparation of Concrete Specimens

Most of the tests were carried out using 3x6 in. (76x152 mm) concrete cylinders. After compaction in cardboard molds and standing for 24 hours, the cardboard was removed and the specimens were cured for 28 days in a moisture room at 90 to 100 percent relative humidity (RH). After drying by various means which will be described later, the concrete cylinders were, after having cooled down, ready for impregnation.

2.2 Impregnation Vessel

The vessel (Fig. 1) used for impregnation was constructed from an 8x15 in. (203x381 mm) steel pipe, a top and a bottom flange with a lid bolted to the top flange. Two gages, one measuring the vacuum and the other measuring pressure, a safety valve, and a valve connected to a hose were attached to the lid. The vessel was capable of impregnating eight 3x6 in. (76x152 mm) concrete cylinders simultaneously. After vacuum had been applied through a pump to evacuate the air from the specimens, the monomer was forced into the impregnation vessel through the hose by opening the valve to the vacuum chamber. The vessel was then closed, and pressure from a nitrogen

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tube was applied to complete the penetration of the monomer into the concrete specimens. Nitrogen was used to eliminate the fire hazard involved in the handling of these flammable monomers. Polymerization was finally completed by placing the concrete cylinders in hot water.

2.3 Test Set-Up

A modification of the standard splitting tensile test (ASTM C496-66) was developed to be able to record the load-strain relationship automatically. The set-up, shown in Fig. 2, consisted of a strain gage glued to the bottom plane surface of each specimen, horizontally in the center of the concrete cylinder. This allowed the lateral strain to be recorded as a function of the load. Only one gage was used per specimen.

The specimens used in the compression tests (ASTM C39-66) were capped with high-strength gypsum plaster to assure that the two ends were plane and parallel with respect to each other. The compression test set-up is shown in Fig. 3. A frame that easily could be mounted on the specimen, was designed for the experiment. To measure strain, two clip-type extensometers were fixed on either side of the specimen between the two rings. The average strain for both sides was then recorded automatically as a function of the load. The set-up was designed with the explosive, brittle failure of PIC in mind. At failure, the clip gages would be detached from the rings, undamaged, and could be used repeatedly and satisfactorily even after several explosive failures of the PIC specimens had taken place. A close-up of the set-up is shown in Fig. 4.

An X-Y plotter was used to record the load-strain relation-
ship for both the tension and compression tests.

2.4 Testing Machines

Some of the first splitting tensile tests were carried out
in a 100 kip (445 kN) strain-controlled testing machine. This machine
was used to see whether a correct downward portion of the load-strain
curve could be obtained. Normally the energy stored in most testing
machines would be released suddenly once the specimen starts to fail
and any downward portion of the load-strain curve would be distorted.
The results were compared with load-strain curves obtained by using
a 300 kip (1.334 MN) hydraulic testing machine.

The comparison showed only an insignificant difference
between the results. The failure of the PIC specimens appeared to
be almost explosive in both machines. No conclusion could be made
whether the lack of ductility was due to the sudden release of energy
stored in both machines or to an inherent characteristic for the PIC
material (5). However, the failure was observed to be much more
brittle and explosive for the PIC specimens than for the control
specimens. All the compression tests and the major part of the
splitting tensile tests were therefore performed using the 300 kip
(1.334 MN) hydraulic testing machine.

3. SCOPE

The investigation was further divided into seven different parts and the scope of each one is outlined below.

3.1 Monomer Penetration

The concrete mix as well as curing conditions were the same for the specimens here named MMA1, MMA2, MMA3, and MMA4.

To simulate a possible field application the specimens named MMA1 were dried over an open gas flame and then impregnated with the monomer methyl methacrylate (MMA) using both vacuum and pressure.

After testing the first set of specimens (MMA1), it was found that the polymerized area only penetrated 1 in. (25 mm) into the specimens, leaving a 1 in. (25 mm) diameter inner core without polymer. The impregnation procedures were therefore changed for the next sets of specimens in order to achieve complete impregnation of the concrete cylinders.

To investigate the importance of the application of vacuum on the penetration of polymer into the concrete, the specimens named MMA2 were impregnated using both vacuum and pressure and the specimens named MMA3 were impregnated using pressure only.

For the first three sets of PIC specimens, (MMA1, MMA2, and MMA3), a benzoyl peroxide concentration of 3 percent by weight of the MMA (methyl methacrylate) was used as catalyst. For the last set

(MMA4), 0.5 percent of azobisisobutyronitrile (AZN) by weight was used as catalyst to examine whether better polymerization could be achieved.

3.2 Use of Co-Polymers

PIC impregnated with MMA is brittle and shows little ductility. It can therefore in its present form only be expected to find limited application as a structural material in building construction. Even if the ultimate strength is high, no plastic yielding or ductility has been observed before sudden and explosive failure occurs.

Various monomer combinations of methyl methacrylate and n-butyl acrylate (BA) was therefore used to impregnate the concrete specimens here named 100 MMA, 90 MMA 10 BA, 70 MMA 30 BA, 50 MMA 50 BA, which also indicates the percentage of each monomer in the mix. This investigation was designed to find ways of increasing not only the strength but also the ductility of the PIC.

Again the concrete mix and curing conditions were standar-dized for the four types of specimens. Also the same impregnation procedures were used for all the concrete cylinders. By varying the percentage of monomer combinations of MMA and BA, various strength and ductility properties of the composite material were obtained. To initiate the polymerization, an azobisisobutyronitrile (AZN) concentration of 0.5 percent by weight of the two monomers was used as catalyst. Polymerization was achieved by keeping the specimens

submerged in hot water at 70-80°C for 3 hours.

3.3 Optimization of Co-Polymer Composition

Further work was carried out with the co-polymer with the purpose to optimize the polymer composition in terms of stress-strain behavior (8).

For comparison with the co-polymer specimens, the first concrete cylinders, here named AI were impregnated using MMA only, and AII were impregnated using 60 percent methyl methacrylate and 40 percent n-butyl acrylate.

In an attempt to increase both the strength and the ductility of the PIC simultaneously, a cross-linking agent, trimethylolpropane trimethacrylate (TMPTMA) was added first to the MMA alone and then to various concentrations of the co-polymer mix MMA plus BA. The specimens BI contained therefore 90 percent MMA, BII 70 percent MMA and 20 percent BA, BIII 60 percent MMA and 30 percent BA, and BIV 50 percent MMA with 40 percent BA. In addition all of them contained 10 percent TMPTMA. The A and B series also included unimpregnated specimens for control. They were all made from the same concrete and impregnated and polymerized using the same procedures. Again 0.5 percent of AZN was used as catalyst.

3.4 Effect of Salt Contamination

The study described herein was also part of a larger program with the purpose to prevent deterioration and prolong the service life

of concrete bridge decks by means of polymer impregnation. It was therefore natural to look at the effects of chloride contamination encountered in normal bridge decks. Deicing salts are frequently used in the northern states during the winter season and the chloride concentration will reach a point in a bridge deck where corrosion starts in the first layer of reinforcing bars.

It was expected that both the impregnation rate and final loading would be reduced in such concrete since the salt would clog the capillary pores and restrict both the movement and the space available to the monomer.

A number of salt contaminated specimens were therefore prepared. Concrete cylinders, here referred to as CI and CII were immersed in a 3 percent aqueous salt solution for 24 hours. This was done twice for the CII and once for the CI specimens, after subsequent drying, but before impregnation with MMA.

In addition, another set of specimens here named D, DI, and DII were prepared for salt contamination because it appeared from experience with the CI and CII cylinders that longer drying periods were required to reach constant weight when salt was present. All specimens were impregnated using MMA, and unimpregnated control samples were also prepared for the C and D series.

3.5 Effect of High Temperature Drying

The major obstacle to monomer penetration is naturally the residual water occupying the pores in the concrete. Adequate drying

is therefore one of the most important factors in PIC production (4,10). To effectively dry a large concrete object such as a bridge deck, temperatures above $212^{\circ}F$ ($100^{\circ}C$) is required in order to remove the water within a practical time (9,11).

Two possible drying methods were tried to investigate the effect of high temperature drying on the final strength of both un-impregnated and polymer impregnated concrete cylinders.

The drying equipment which was developed for a related investigation (9,12) consisted of a propane burner constructed with a hood directing the flame towards the concrete surface (Fig. 5). An electric motor moved the hood back and forth over an approximate 3 ft. range. The concrete cylinders referred to as EI were dried under a sand cover using this burner. The temperature in the sand reached approximately $750^{\circ}F$ ($400^{\circ}C$). It was expected that such drying might induce cracks and microcracks that would lower the strength of the concrete.

EI specimens, with and without polymer impregnation were tested together with specimens dried at a lower temperature. This was done to determine whether a strength reduction could be expected and to what degree the strength was restored by polymer impregnation.

A kerosene burner was also tried as a possible drying method for bridge decks. The set-up as shown in Fig. 6 consisted of a burner which forced air at high velocity into a built-up enclosure. The specimens here named EII were placed inside and dried in the hot

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airflow from the burner. A thermostat shut the burner off when the air temperature inside reached 250°F (121°C) and turned it on again at 225°F (107°C). Both the EI and EII specimens, were finally impregnated with MMA and tested together with unimpregnated control samples.

3.6 Impregnation with Sulfur

The price of monomers has, however, followed the increase in oil prices during the last two years and a search for a cheaper substitute was therefore a natural next step. Sulfur was found to be such a material, and when molten could be used for concrete impregnation much in the same manner as a monomer (13,14). A monomer such as MMA is now approximately ten times the price of sulfur per lb.

Using the same impregnator as shown in Fig. 1, with some modifications added to the vessel, eight concrete cylinders (3x6 in.) were impregnated using molten sulfur. A propane burner was placed under the vessel to keep the sulfur molten during the impregnation time. A thermocouple was placed inside in the molten sulfur and extended through a pipe at the bottom of the vessel. By regulating the burner, the temperature could be kept between 230-300°F (110-148.89°C), or in the range where molten sulfur has the desired viscosity. The impregnator was also in this case pressurized to force the molten sulfur into the concrete. The set-up is shown in Fig. 7.

No polymerization step is required in this case since the sulfur naturally solidifies when the temperature drops below the melting point.

3.7 Impregnation Through Cast-In Perforated Pipes

Impregnation with monomer over a large concrete area under field conditions is often a difficult and cumbersome task and must be carried out with special care.

First the concrete must be dried to about $250^{\circ}F$ ($121^{\circ}C$) at the depth of desired impregnation. This again requires a considerable amount of energy, in this case by burning propane gas. The concrete must then cool down before the monomer can be applied. On a bridge deck, the monomer can be ponded on the surface or applied through a chamber bolted down to the concrete slab (9).

During the ponding procedure a large quantity of monomer will for some time be exposed to the open and extra care must be taken because of the great fire hazard. MMA is for example highly flammable and the effects of exposure to the liquid and the toxic vapors are not yet fully established.

Using a chamber bolted down to the concrete and forcing the monomer in under pressure offers an advantage in terms of safety since the system is enclosed. Pressure will speed up the time for impregnation considerably (9), but the area and pressure will be limited because of the load exerted on the slab. For a 48 sq. ft. area the total load will exceed a 100 kips at 15 psi pressure. Leaks from the

gasket sealing the impregnator to the concrete can also be expected, so a working pressure of about 5 psi is more realistic. Since the time for impregnation is proportional to the square of the pressure (9), the time requirement is therefore increased accordingly.

When polymer impregnation of a bridge deck can be planned in advance, a network of perforated pipes could be permanently cast in concrete inside the deck. Impregnation can then be carried out through these pipes in an enclosed system using high pressure where both the fire hazard and the exposure of monomer is reduced to a minimum. Such a system of pipes could be placed together with the upper layer of reinforcing steel and even be utilized as part of the reinforcement.

To demonstrate such a method, 1/4 in. perforated steel pipes were cast inside 6x12 in. (152x305 mm) concrete cylinders. To prevent the cement grout from clogging the pipe through the perforation, a steel rod was placed inside the pipe and pulled out when the concrete had started to harden. After curing, the concrete cylinders were dried for different length of time. This was done to establish to what degree such concrete should be dried to achieve sufficient impregnation.

Using the same impregnator as shown in Fig. 1 as a reservoir for the monomer and with a hose connected through the pipe at the bottom of the vessel, three specimens could be impregnated simultaneously with MMA as shown in Fig. 8. After polymerization in hot water, the specimens were tested in the splitting tensile test to

determine the tensile strength and the extent of successful impregnation.

4. MATERIALS AND IMPREGNATION

A general outline of the preparation of concrete specimens was described in Chap. 2. Except for the experiment using perforated pipes cast inside 6x12 in. (152x305 mm) concrete cylinders, all the other specimens described herein was made using 3x6 in. (76x152 mm) molds.

4.1 MMA1, MMA2, MMA3, and MMA4 Specimens

All of these specimens were made from Type 1 Portland cement. The fine aggregate was natural siliceous sand with a fineness modulus = 2.90 and the coarse aggregate was mixed gravel 3/8 in. (9.5 mm) in size, both according to ASTM C33-67. The water/cement ratio was 0.45 and coarse aggregate in proportion 1:2.2:3.7:3.0 by weight. No air-entraining admixture was used in the preparation of this concrete. The measured slump was 2-1/2 in. (64 mm) using the ASTM C143-66 procedure and the measured entrained air was 5-1/2 percent using an AE-55 air indicator. After 28 days in the moisture room, the specimens were stored for 14 days in air.

The MMA1 specimens were dried over an open gas flame for 4 hours, with the temperature at the concrete surface exceeding 600°F $(316^{\circ}$C). The MMA2, MMA3, and MMA4 specimens were dried in an oven where hot air was circulated at high velocity. The same oven was used for drying most of the other specimens except when they were dried using a propane burner.

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After drying for 24 hours at $260^\circ F$ ($127^\circ C$), constant weights were achieved and the concrete cylinders were cooled down before impregnation.

Methyl methacrylate with 3 percent by weight of benzoyl peroxide as catalyst was used for specimens MMA1, MMA2, and MMA3. The same monomer was used for MMA4 except that 0.5 percent azobisisobutyronitrile (AZN) was used as catalyst. All impregnation and polymerization details can be found in Table 1.

4.2 Specimens for Co-Polymer Impregnation

For these specimens, previously named 100 MMA, 90 MMA 10 BA, 70 MMA 30 BA, and 50 MMA 50 BA, Portland cement Type 1 was used. The fine aggregate was made from liliceous stone, crushed to a fineness modulus of 2.83, and the coarse aggregate was crushed limestone of 1/2 in. (13 mm) maximum size both according to ASTM C33-67. A water/cement ratio of 0.5 was used and the mix consisted of water, cement, fine and coarse aggregate in the proportions 1:2:4.3:4.7 by weight. Ten cubic centimeters of the air-entraining admixture "Darex" was added to the water and mixed into the fresh concrete.

The slump was recorded as 5 in. (127 mm) and the entrained air was 7 percent. After the usual moist curing, the specimens were stored for 14 days in air in the laboratory before drying and impregnation. Drying was completed in 10 hours at $260^\circ F$ ($127^\circ C$).

All the specimens were thereafter impregnated using the same procedures. The difference was only in the composition of MMA to BA as shown in Table 2.

Inside the vessel, the specimens were subjected to a vacuum of 20 in. (508 mm) of mercury for 1 hour, before the monomer or mixture of the two monomers containing 0.5 percent of AZN was induced into the vessel. A pressure of 60 psi (0.41 N/mm^2) was kept for 1 hour before the specimens were removed and polymerized under hot water at 70-80°C for 3 hours. Table 2 gives details of the polymerization.

4.3 Specimens for Optimization of Co-Polymers

The concrete used in the specimens referred to as AI, AII, BI, BII, BIII, and BIV had the same composition and was made from the same materials as those mentioned in Sec. 4.2. The only difference was that no air-entraining admixture was used and the result was a smaller slump, 2 in. (51 mm) and 5 percent entrained air. Drying was completed in the oven at 260°F (127°C) for 24 hours before impregnation.

The impregnation included vacuum at 29 in. (737 mm) of mercury for 1-1/2 hours before the various monomer combinations containing 0.5 percent of AZN were induced into the vessel. The vessel was then pressurized at 60 psi (0.41 N/mm^2) for 1-1/2 hours to complete the penetration of monomer. After removal from the vessel, the specimens were polymerized under hot water at 70-80°C for 4 hours. Table 3 contains details of the polymer combinations and polymer

loading.

4.4 Salt Contaminated Specimens

The same concrete mix as described in Sec. 4.2 and 4.3 was used here. CI, CII and their control specimens contained no air-entraining admixture while 10 cubic centimeters of "Darex" was added to the concrete mix for D, DI, DII and their control specimens. This resulted in an air content of 4 percent and a 2-in. (51 mm) slump for the C series and 7 percent air and a 2-3/4 in. (70 mm) slump for the D series. After moist curing, all specimens in the C and D series were initially dried at 260°F (127°C) for 24 hours before salt contamination.

CI and CII specimens were immersed in a 3 percent salt (sodium shloride) solution for 24 hours and then redried for 24 hours at the same temperature. This process was repeated for the CI specimens before both CI and CII were impregnated.

When it was found that the ultimate strength of the CI and CII PIC specimens were somewhat lower than expected, the D series was prepared for salt contamination, and constancy of weight after drying was checked carefully. After the initial drying, the D specimens were submerged in plain water, whereas the DI and DII specimens were kept in the 3 percent salt solution for 48 hours before drying. The weight was recorded at the beginning of the drying period, and at 17 hours, 49 hours, and 66 hours at 260°F (127°C). After an average loss of 90 g at 17 hours, constant weight

was obtained for the D series. For the DI and DII specimens the drying was continued for an additional 49 hours.

After the first 17 hours of drying for the DI and DII specimens, the average weight loss was 78 g. During the following 49 hours they lost an additional average of 10 g, giving a total average loss of 88 g, close to the value noted for the D specimens. The DII cylinders were once more immersed in the salt solution for 48 hours and redried at $260^{\circ}F$ ($127^{\circ}C$) for 60 hours. Table 4 shows the final weight percentages of salt in the CI, CII, DI and DII specimens.

Again using 1-1/2 hours of vacuum at 29 in. (737 mm) of mercury, 60 psi (0.41 N/mm^2) for 1-1/2 hours of pressure, the C and D series were impregnated using MMA with 0.5 percent AZN. Polymerization was completed after 4 hours in 70-$80^{\circ}C$ hot water.

4.5 Specimens for High Temperature Drying

The composition of the concrete was described in Sec. 4.2. For these specimens here referred to as EI and EII, 7.5 cubic centimeters of "Darex" was used. The air content was 6 percent and the slump 2-1/2 in. (64 mm).

Directly after moist curing, the EI specimens were dried using the propane burner shown in Fig. 5 and described in Sec. 3.5. The specimens were dried for 8 hours under 1 to 2 in. of sant at $750^{\circ}F$ ($400^{\circ}C$). The weight loss after drying showed that the specimens had been dried thoroughly when compared to the oven dried specimens.

No visible cracks were noted after the drying had been completed.

The EII specimens were first dried using the kerosene burner shown in Fig. 6 and described in Sec. 3.5. The drying was conducted outside during and after a light rainfall for 12 hours. However, since this procedure removed less than 50 percent of the estimated free water in the specimens, these cylinders were removed from the enclosure and oven dried at $260^{o}F$ ($127^{o}C$) for an additional 12 hours before impregnation. The EII specimens were then used for comparison with the EI specimens to determine the effect that high temperature drying might have had on the strength of these specimens. Both series were impregnated with MMA and 0.5 percent AZN and using the procedure described in Sec. 4.2.

4.6 Specimens Impregnated with Sulfur

The composition of the concrete was again 1:2:4.3:4.7 by weight of water, cement, fine and coarse aggregate respectively. The fine aggregate was natural siliceous sand with a fineness modulus of 2.67 and the coarse aggregate was crushed limestone with a 1/2 in. (13 mm) maximum size. Five cubic centimeters of "Darex" was used for this batch, and the air content was measured to 5 percent with a slump of 3 in. (76 mm). After moist curing the specimens were stored in air for 21 days.

Drying was carried out at $260^{o}F$ ($127^{o}C$) for 24 hours before impregnation. The specimens were taken directly from the oven to the impregnator, sulfur was added and at the same time heated using the

propane burner as shown in Fig. 7. When molten sulfur covered all the specimens, the vessel was closed and the pressure was kept at 30 psi (0.21 N/mm^2) for 24 hours. In the meantime the temperature in the molt was kept between 230-300°F (110-150°C) by reading the temperature recorded by the thermocouple and regulating the temperature. In this temperature range the sulfur has the lowest viscosity and can therefore more easily penetrate the concrete. After removal and cooling down, the SIC specimens (Sulfur Impregnated Concrete) were ready for testing.

4.7 Specimens with Cast-In Perforated Pipes

The specimens for this experiment were the only ones of the 6x12 in. (152x305 mm) size used in this investigation.

A 14 in. (356 mm) long, 1/4 in. (6 mm) galvanized steel pipe was perforated by drilling 1/8 in. (3 mm) holes, 4 holes on the circumference and spaced 1/2 in. (13 mm) apart on a 6 in. (152 mm) length on the pipe. A cap was put on the end that was cast inside the specimen. A collar held the pipe in place in the center of the cardboard mold during the casting. The pipe was located in such a position that the holes were placed 3 in. (76 mm) from the ends and from the circumference of the concrete cylinder.

The cement used in the cylinders was a Portland Type 1 cement. The fine aggregate was a natural siliceous sand with a fineness modulus of 2.67 and the coarse aggregate was crushed limestone of 1 in. (25 mm) maximum size, both according to ASTM. The concrete

mix consisted of water, cement, fine and coarse aggregate in the proportion 1:2:3.5:4.2 by weight. Three cubic centimeters of "Darex" was added to the water and mixed into the concrete. The air content was measured to 4.5 percent and the slump was 2-1/2 in. (64 mm).

To prevent the cement paste from filling up the pipe by leaking in through the holes, a rod was placed inside the pipe and removed when the concrete started to harden.

After 24 hours, the specimens were removed from the mold and cured at 90-100 percent relative humidity for 28 days. To investigate the necessary degree of drying needed for successful impregnation, 9 specimens were dried for different lengths of time before impregnation. The drying time and the approximate degree of free water removed by drying can be found in Table 6.

Impregnation was carried out using the impregnation vessel as a reservoir for the MMA with 0.5 AZN as catalyst. The set-up was described in Sec. 3.7 and shown in Fig. 8. The pressure was kept at 80 psi for 5 hours, before polymerization in 65-70°C for 6 hours.

Since only a limited number of specimens were available it was chosen to use only the standard splitting tensile test (ASTM C496-66). This was done not only to record the tensile strength of the concrete, but also to be able to observe more easily the polymer penetration on the two halves.

5. TEST RESULTS

All the 3x6 in. (77x152 mm) PIC of SIX specimens together
with the respective control samples were tested in both splitting
tensile and compression tests. For the PIC specimens the load-strain
relationship was also recorded.

5.1 Results from Specimens MMA1, MMA2, MMA3 and MMA4

As mentioned in Sec. 3.1, it was found after testing the
MMA1 that complete penetration of polymer was not achieved. A 1 in.
(25 mm) core was left without polymer.

The vacuum, pressure and polymerization time for the MMA2
and MMA3 specimens were subsequently increased. The results, however,
were found to be similar to that of the MMA1 specimens. This again
confirmed the assumption that the difference in drying method had
little influence on the penetration of polymer.

The lack of complete polymerization of the polymer through-
out the specimens in MMA2 and MMA3 was evident when the specimens
were broken and a strong smell of monomer was released. This suggested
that full penetration of the monomer was achieved, but without com-
plete polymerization.

One investigation (15) suggested that the benzoyl peroxide
has a greater affinity to the cement than does the monomer molecules.
The catalyst was believed to be absorbed by the cement and thus
reduces the concentration of catalyst as the monomer penetrates into

the specimen. A central core is formed inside the specimen without sufficient catalyst to promote polymerization. By changing the catalyst and using 0.5 percent of AZN a more complete polymerization was achieved in specimens MMA4. The specimens appeared to have a uniform distribution of polymer even in the center. No smell of monomer could be detected after the specimens were broken.

When comparing specimens MMA2 and MMA4, where the specimens had approximately the same polymer loading, it was evident that the catalyst used in MMA4 gave a more efficient polymerization.

The following all-around penetration of polymer into the concrete is shown in Table 1.

Figure 9 shows the average tensile load-strain curves for MMA1, MMA4, and control specimens, and the corresponding compressive stress-strain curves are shown in Fig. 10.

The load-strain curves for the PIC samples show the same linear relationship up to approximately 75 percent of the ultimate load. This has already been observed by other investigators. Table 7 lists the average ultimate strengths and Young's modulus for the specimens tested.

5.2 Results of Co-Polymer Impregnation

Since AZN was used as catalyst, impregnation was achieved throughout the specimens. Only a slight smell of monomer was released when the specimens were broken, indicating that almost complete

polymerization of the monomer had taken place.

Figure 11 shows the average load-strain curves for the splitting tensile tests and the corresponding compressive stress-strain curves are shown in Fig. 12. Clearly, the modulus of elasticity, ultimate strength, and energy to break of the concrete specimens are dramatically increased by incorporation of MMA in comparison with the control specimens. Further, the incorporation of BA results in less increase in strength and modulus but with a considerable gain in ductility as implied by plastic yielding, at least for the specimens with 50 percent MMA and 50 percent BA. The tensile and compressive properties all tend to be decreased in direct proportion to the amount of BA used.

The load-strain or stress-strain curves for the specimens with 100, 90, and 70 percent MMA show an almost linear relationship up to approximately 75 percent of the ultimate load. For the specimens with 70 percent MMA and 30 percent BA, the load-strain or the stress-strain curves show some yielding (unloading) after the ultimate load was reached.

For 50 percent MMA and 50 percent BA, the specimens show a remarkable ductile behavior and specimens still carried a higher load than the control concrete's ultimate strength with a strain of 9000×10^{-6} in compression (Fig. 12). This is a strain three times larger than that reached by the control concrete at failure. Table 8 lists some average ultimate strengths and values of Young's modulus for the specimens tested.

5.3 Results of Co-Polymer Optimization

Essentially uniform impregnation and complete conversion to polymer were achieved for all the PIC specimens. This was shown by visual and microscopic examination and by the observation of at most a faint odor due to unreacted monomer in only a few specimens on freshly broken surfaces.

Table 9 lists the ultimate strengths and values of Young's modulus for the A and B series with control specimens, and Figs. 13-16 give the stress-strain and load-strain curves themselves.

As shown in Figs. 13 and 14, the AI specimens, impregnated with 100 percent MMA, display the now familiar linear load or stress-strain relationship almost up to the point of ultimate brittle failure. In comparison, the AII specimens, impregnated with a 60/40 MMA/BA mixture, show a significant increase in ductility in the sense of yielding followed by continuous increase in deformation without significant decrease in load. Only a 20 percent reduction in strength was observed compared to the AI specimens. These results agree well with the trend observed for the specimens described in Sec. 5.2.

Effects of adding the crosslinking agent, TMPTMA, are shown also in Table 9 and Figs. 15 and 16. For specimens BI to BIV, the compositions are given in Table 3. It is seen that the addition of 10 percent TMPTMA as crosslinking agent of various combinations of MMA and BA increases the Young's modulus of PIC, but has a complex effect on strength. While 10 percent TMPTMA causes a decrease in both tensile and compressive strengths for the case of 100 percent MMA,

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the compressive strengths for the MMA/BA combinations with TMPTMA pass

through a maximum for a 60:30:10 mixture, the maximum value being

similar to the value for 100 percent MMA. However, in all cases the

use of TMPTMA tends to reduce ultimate strain and ductility.

5.4 Results with Salt Contaminated Specimens

Figures 17 and 18 show the load-strain and the stress-strain

behavior of the salt contaminated specimens CI and CII, containing

0.8 and 0.4 percent salt, respectively. In this case 100 percent MMA

had been used for impregnation. Clearly, at least in these specimens,

salt contamination prior to impregnation reduces the ultimate strength

somewhat (15-20 percent in compression and 30-35 percent in tension)

though the reduction is small in comparison with the relative increase

due to impregnation.

Results for DI and DII specimens, containing 0.5 and 1.0

percent salt, respectively, and which had undergone extended drying

prior to impregnation, are given in Figs. 19 and 20 along with data

for the uncontaminated specimens D. Since the DI and DII specimens

all show higher loading (Table 4) and higher strength than the CI

and CII series, the additional drying and air voids in the DI and

DII series ultimately appear to be responsible for the improved

behavior. The ultimate strengths and Young's modulus for both series

can also be found in Table 10.

5.5 Results of High Temperature Drying

As shown by Figs. 21 and 22 and Table 11, high temperature drying with the propane burner at $750^{\circ}F$ ($400^{\circ}C$) and subsequent polymer impregnation results in a 15 percent decrease in the tensile strength of the high temperature dried specimens EI, as compared to the much less rigorously dried specimens EII.

On the other hand, in compression, the impregnated specimens EI exhibits levels of strength as high as any observed in the study. However, a larger deviation from the linear stress-strain relationship commonly found for PIC could be noted.

5.6 Results with Sulfur Impregnation

The sulfur impregnated specimens were tested in both the splitting tensile test and compression together with control from the same concrete mix.

The load-strain relationship was not recorded for the SIC specimens, only the ultimate load in the tensile test and compression.

The average loading with sulfur in the specimens were calculated to 9.7 percent by weight. The apparently high loading was due to the higher specific weight of sulfur compared to MMA. The sulfur had only penetrated 3/4 to 1 in. into the specimens leaving at least a 1 in. central core without impregnation.

In compression the strength was calculated to 5.2 ksi (35.5 N/mm^2) for the controls and 9.8 ksi (67.9 N/mm^2) for the SIC

specimen. This gives an increase in strength of about 90 percent.

In the splitting tensile test the strength was calculated to 0.61 ksi (4.23 N/mm^2) for controls and 0.88 ksi (6.04 N/mm^2) for the SIC or only 42 percent increase in strength.

5.7 Results for Specimens with Cast-In Perforated Pipes

As mentioned in Sec. 3.5, the major obstacle to monomer penetration is the presence of water in the pore system. Table 6 shows the approximate degree of drying and Table 12 show the polymer penetration radially from the pipe and the ultimate tensile strength of each specimen.

It is apparent that the obtained values are approximate due to the limited number of specimens.

Detectable impregnation was not achieved after about 9 hours of drying at 260°F (127°C). The penetration increased from only 1/2 in. (13 mm) after 9 hours of drying where about 70 percent of the water was removed, up to almost complete penetration after 21 hours where more than 90 percent of the water was removed. Although both specimens 3 and 4 showed some degree of penetration, specimen 5 was left without any detectable impregnation.

Specimen 6 gives the highest strength of all the specimens after 18 hours of drying. Complete penetration was not achieved for this particular specimen, although the weight indicated that 100 percent drying was achieved.

For the last three specimens, both the strength and the polymer penetration appeared to be similar, but even for these specimens the impregnation was not uniform. The edges and about 1/4 in. from the circumference were left with little or no detectable impregnation.

The specimens with their respective numbers can be found in Figs. 23 to 25. The degree of penetration is somewhat difficult to observe in the pictures. It can most easily be detected where the fracture runs through the aggregate instead of the aggregate being pulled out of the cement matrix. This is most apparent for the four last specimens.

6. CONCLUSION

6.1

The effect of the applied vacuum and pressure appears to be less important than choosing the proper catalyst in the PIC specimens. Complete penetration was apparently achieved at least for the MMA2 and MMA4 specimens but only in the MMA4 specimens had the monomer polymerized throughout the concrete cylinders. This suggests that the azobisisobutyronitrile (AZN) is a more effective catalyst than benzoyl peroxide in this case. Also the strength obtained for the impregnated MMA1 specimens suggests that a high temperature drying method is feasible for preparing PIC.

6.2

Modification of the brittle behavior of PIC impregnated with methyl methacrylate is possible by combining the MMA with n-butyl acrylate (BA). The higher the percentage of BA, the greater the ductility, corollary decrease in strength and modulus of elasticity. These results show that PIC material can be prepared to fit any specified relationship between strength and ductility, thus potentially providing tailored material properties to fit a particular service requirement as a structural material.

The investigation also showed that concrete can just as easily be impregnated with the co-polymer system than with a 100 percent MMA.

6.3

A 60/40 MMA/BA combination shows a pronounced increase in ductility, or the ability to yield and deform at stresses close to the maximum observed before failure. The strain is 60-70 percent larger than for the control specimens and at the same time the strength is more than doubled in both compression and the tensile test. Despite the larger strain, it has a larger Young's modulus than the controls. It also has more ductility than the 70/30 MMA/BA combination and less than the 50/50 combinations, and appears well suited for structural purposes.

Incorporation of a cross linking agent, TMPTMA, in the monomer mixture has several effects: (a) a slight decrease in tensile and compressive strengths in the case of MMA alone; (b) a slight decrease in tensile strengths for all MMA/BA composition studied (up to 40 percent BA); (c) a slight decrease to a negligible change in compressive strength, depending on the concentration of BA; and (d) a general reduction in ultimate strain and ductility, along with an increase in Young's modulus. Thus, a wide variety of stress-strain and ductility characteristics can be obtained by suitable variation in monomer composition.

It could also be noted that the polymer loading is increased when air-entraining admixture is added. This is presumably due to a consequent increase in porosity.

6.4

Although larger concentration of salt are known to reduce
polymer loading (9), the presence of up to 1 percent sodium chloride
in a concrete matrix has little practical effect on the amount of
polymer loading attainable, as long as specimens are dried adequately.
Adequate drying does appear to take longer with salt-contaminated than
with uncontaminated concrete. Well-dried salt-contaminated and im-
pregnated specimens (DI, DII) show strengths and moduli at least as
high as untreated ones, if comparison is made at equal polymer load-
ings. This was not the case for the impregnated specimens CI and
CII, where the drying apparently was inadequate.

6.5

In a separate study (10), high-temperature drying (750°F)
has been shown to be useful in the field impregnation of a bridge
deck. In this study, it is seen that, although such drying may induce
cracks and microcracks that lower the strength of concrete, impregna-
tion restores or heals any cracks so that the final PIC is as strong
in compression, and almost as strong in tension, as conventionally
dried material.

6.6

It is considerably harder to impregnate with sulfur than
with a monomer. The viscosity is about 10 times that of MMA and the
temperature has to be maintained at the right level (230-300°F or
110-150°C) during impregnation. With a relatively low pressure,

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impregnation will take considerable time.

However, in a plant situation, where high pressure and temperature can be easily controlled, SIC offers considerable advantages because at high strength and low price of the raw material. Another advantage is than the polymerization step is eliminated.

6.7

Cast-in perforated pipes offer a great advantage in large size concrete structures where polymer impregnation can be planned in advance. As seen from Table 12, the splitting tensile strength is not considerably increased. Apparently the polymer loading is relatively low, but it might be more than adequate to prevent movements of chlorides in a bridge deck and present deterioration of the concrete. Since specimen 6 gave the highest strength, it is possible that some residual water will help the monomer from diffusing through too fast and evaporating from the surface without filling up the smaller pores. But at least a certain degree of drying is necessary to obtain any useful degree of impregnation.

Further research could include a pre-polymer coating on the surface before pressurizing from the inside to increase the polymer loading.

Table 1 Impregnation and Polymerization Details

Specimens	MMA1	MMA2	MMA3	MMA4
Number of Specimens	4	6	6	2
Drying Method	Gas Flame	Oven	Oven	Oven
Vacuum in. of Mercury (mm)	10 (254)	20 (508)	0	20 (508)
Time of Vacuum Hr.	2	2	0	2
Pressure psi (kN/m^3	20 (138)	40 (276)	40 (276)	40 (276)
Time of Pressure Hr.	2	2	2	2
Time for Polymerization 70-75°C in Water Hr.	2	4	4	4
Polymer Loading %	5.4	5.9	4.9	6.0
Penetration Depth in. (mm)	1 (25)	1-1 1/4 (25-32)	3/4-7/8 (19-22)	Full Penetration

Table 2 Specimens with Co-Polymers

Number of Specimens	Polymer as a Percentage	Polymer Loading (% by Weight)
8	100 MMA	6.8
8	90 MMA + 10 BA	7.2
8	70 MMA + 30 BA	7.2
8	50 MMA + 50 BA	6.9
8	Control	0

Table 3 Specimens for Optimization of Co-Polymers

Specimens	No. of Specimens	Polymer as a Percentage	Polymer Loading (% by Weight)
AI	4	100 MMA	4.8
AII	8	60 MMA + 40 BA	4.3
BI	4	90 MMA + 10 TMPTMA	5.2
BII	8	70 MMA + 20 BA + 10 TMPTMA	4.6
BIII	8	60 MMA + 30 BA + 10 TMPTMA	4.9
BIV	8	50 MMA + 40 BA + 10 TMPTMA	5.1

Table 4 Salt Contaminated Specimens

Specimens	No. of Specimens	Salt (% by Weight)	Polymer Loading (% by Weight)
CI	8	0.8	5.1
CII	8	0.4	4.9
D	8	0.0	7.7
DI	8	0.5	7.7
DII	8	1.0	7.1

Table 5 Polymer in High Temperature Dried Specimens

Specimens	No. of Specimens	Polymer Loading (% by Weight)
EI	8	7.4
EII	8	6.8

Table 6 Specimens with Perforated Pipes

Specimens No.	1	2	3	4	5	6	7	8	9
Drying Time Hours	3	6	9	12	15	18	21	24	27
Degree of Drying Percent	31	44	72	72	94	100	94	86	100

Table 7 Ultimate Strength, MMA Specimens

Specimens	Tensile Strength ksi (N/mm²)	Compressive Strength ksi (N/mm²)	Young's* Modulus ksi x 10³ (kN/mm²)
MMA1	1.27 (8.76)	14.4 (99.4)	5.4 (36.6)
MMA2	1.36 (9.38)	16.0 (110.4)	6.0 (41.4)
MMA3	1.19 (8.21)	15.2 (104.9)	5.3 (36.6)
MMA4	1.51 (10.42)	19.6 (135.2)	6.0 (41.4)
Control	0.62 (4.28)	6.6 (45.5)	3.9 (26.9)

Table 8 Ultimate Strengths of Co-Polymer Specimens

Specimens	Tensile Strength ksi (N/mm²)	Compressive Strength ksi (N/mm²)	Young's* Modulus ksi x 10³ (kN/mm²)
100 MMA	1.7 (11.8)	17.2 (118.6)	6.7 (46.2)
90 MMA, 10 BA	1.6 (11.2)	15.6 (107.6)	5.4 (37.2)
70 MMA, 30 BA	1.6 (11.0)	15.2 (104.8)	5.0 (34.5)
50 MMA, 50 BA	1.1 (7.4)	10.5 (72.4)	3.5* (24.1)
Control	0.4 (3.0)	4.4 (30.3)	3.8* (26.2)

Table 9   Strength of Optimal Co-Polymer Specimens

Specimen	Tensile Strength ksi (N/mm^2)	Compressive Strength ksi (N/mm^2)	Young's* Modulus ksi x 10^3 (kN/mm^2)
AI	1.38 (9.52)	18.6 (128.3)	7.5 (51.8)
AII	1.13 (7.80)	14.4 (99.4)	4.0* (27.6)
A Control	0.50 (3.45)	5.5 (38.0)	3.4* (23.5)
BI	1.10 (7.59)	16.1 (111.1)	7.8 (53.8)
BII	0.94 (6.49)	16.4 (113.2)	7.1 (49.0)
BIII	1.01 (6.97)	18.4 (127.0)	7.1 (49.0)
BIV	1.18 (8.14)	17.8 (122.8)	7.1 (49.0)
B Control	0.44 (3.04)	5.4 (37.3)	4.1* (28.3)

*Young's modulus for these specimens is the "secant modulus" measured at a stress one-half the ultimate value. The "tangent modulus" is given for the other specimens. Values are based on compressive tests.

Table 10 Strength of Specimens with Salt

Specimen	Tensile Strength ksi (N/mm²)	Compressive Strength ksi (N/mm²)	Young's* Modulus ksi x 10³ (kN/mm²)
CI	-0.89 (6.00)	15.9 (109.7)	5.7 (39.3)
CII	0.90 (6.2)	14.9 (102.8)	5.7 (39.3)
CI Control	0.48 (3.3)	5.0 (34.5)	4.4* (30.4)
CII Control	0.51 (3.5)	3.8 (26.2)	3.6* (24.8)
D	1.45 (10.0)	19.6 (135.2)	6.7 (46.2)
DI	1.40 (9.7)	18.8 (129.7)	5.8 (40.0)
DII	1.38 (9.5)	19.9 (137.3)	6.3 (43.5)
D Control	0.39 (2.7)	4.2 (29.0)	2.3* (15.9)
DI Control	0.42 (2.9)	4.5 (31.1)	2.9* (20.0)
DII Control	0.47 (3.2)	4.8 (33.1)	3.6* (24.8)

Table 11 Effect of High Temperature Drying

Specimen	Tensile Strength ksi (N/mm^2)	Compressive Strength ksi (N/mm^2)	Young's* Modulus ksi x 10^3 (kN/mm^2)
EI	1.18 (8.14)	19.4 (133.9)	6.4 (44.2)
EII	1.47 (10.14)	19.6 (135.2)	6.4 (44.2)
EI Control	0.42 (2.90)	4.7 (32.4)	3.4* (23.5)
EII Control	0.50 (3.45)	5.6 (38.6)	3.7* (25.5)

Table 12 Tensile Strength, Perforated Pipe Specimens

Specimen No.	Polymer Penetration Radially from Pipe in.	(mm)	Ultimate Tensile Strength ksi	(N/mm^2)
1	0	0	0.40	(2.78)
2	0	0	0.44	(3.01)
3	1/2	(13)	0.45	(3.13)
4	1	(25)	0.45	(3.13)
5	0	0	0.35	(2.40)
6	2	(51)	0.69	(4.74)
7	2-3/4	(70)	0.51	(3.53)
8	2-3/4	(70)	0.52	(3.59)
9	2-3/4	(70)	0.54	(3.73)
Control (Dried)	0	0	0.41	(2.86)
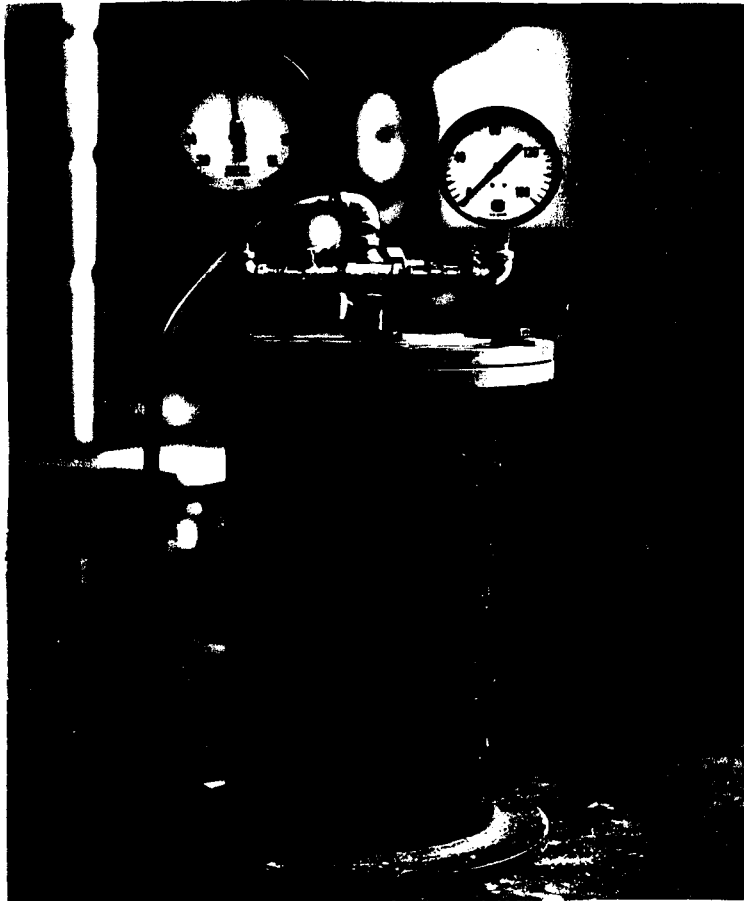
Fig. 1   Impregnation Vessel
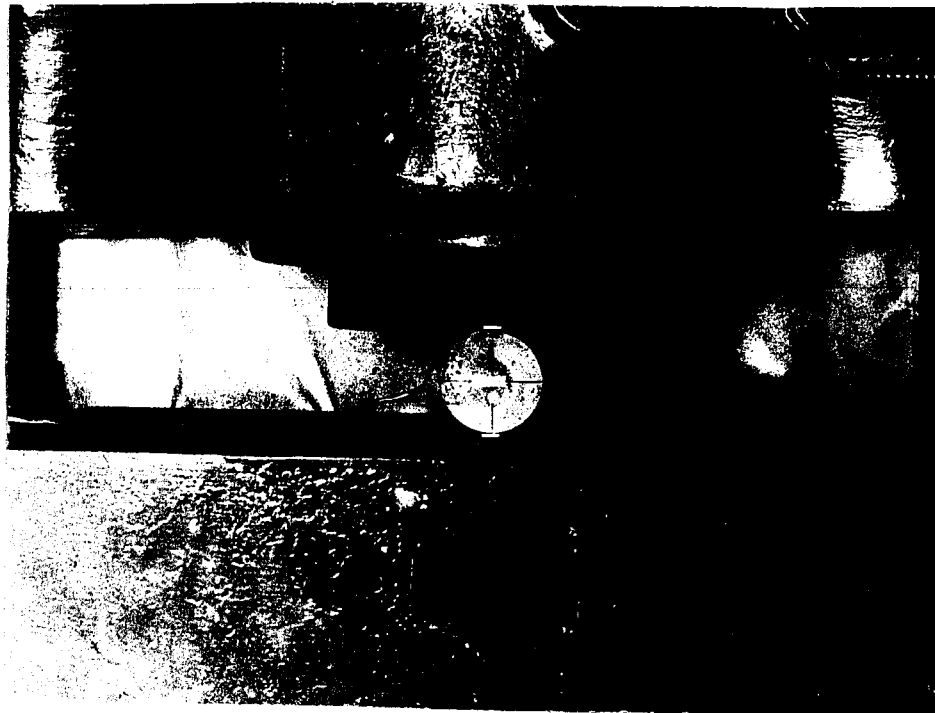
Fig. 2   Tensile Test Set-Up
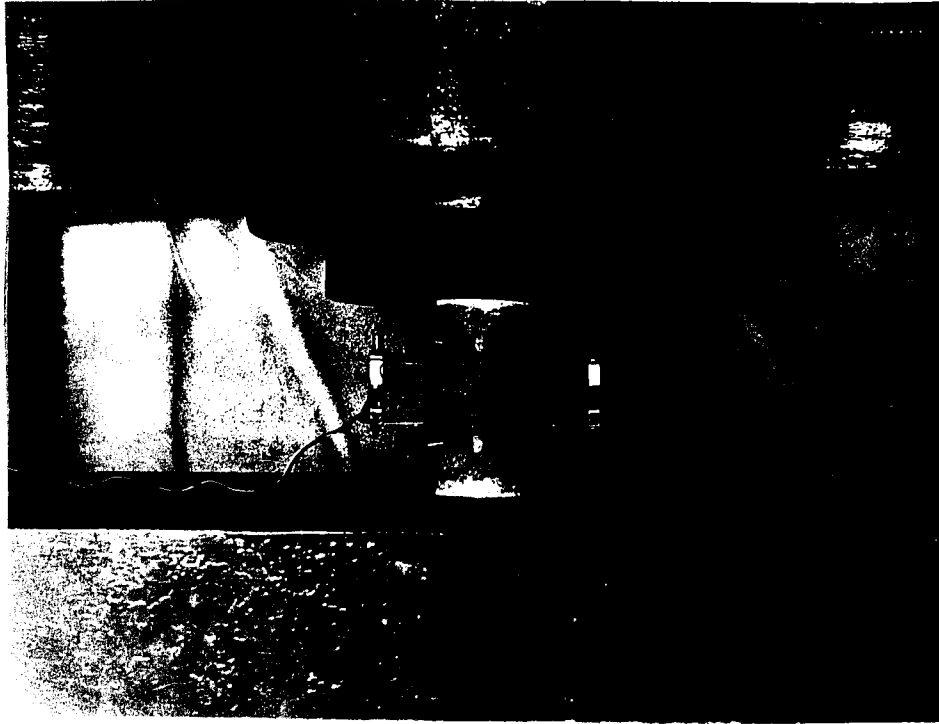
Fig. 3 Compression Test Set-Up
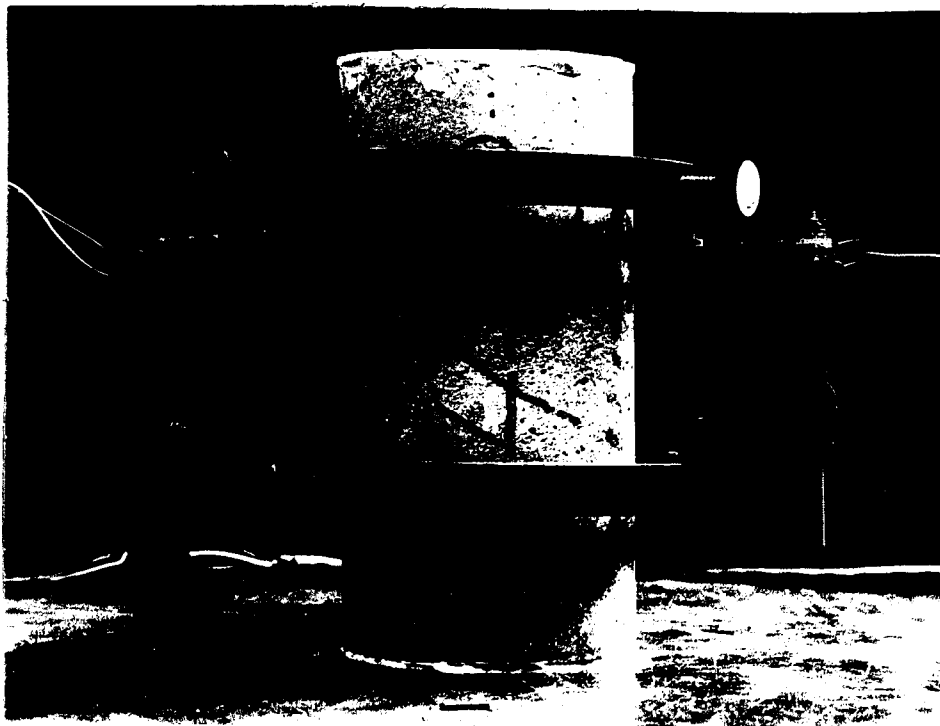
Fig. 4 Clip Gages for Compression Test
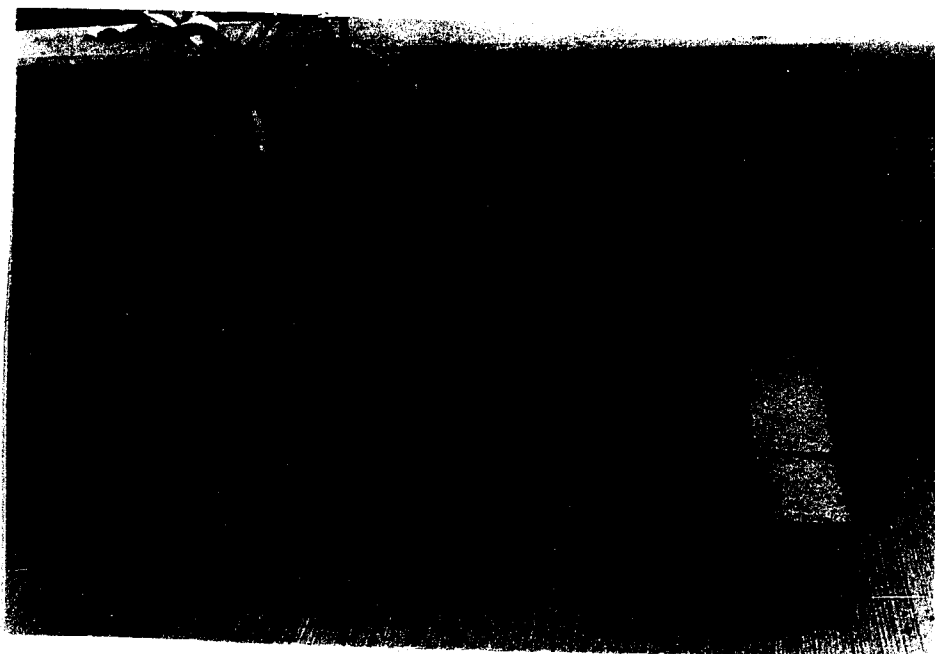
Fig. 5 Propane Burner for Drying
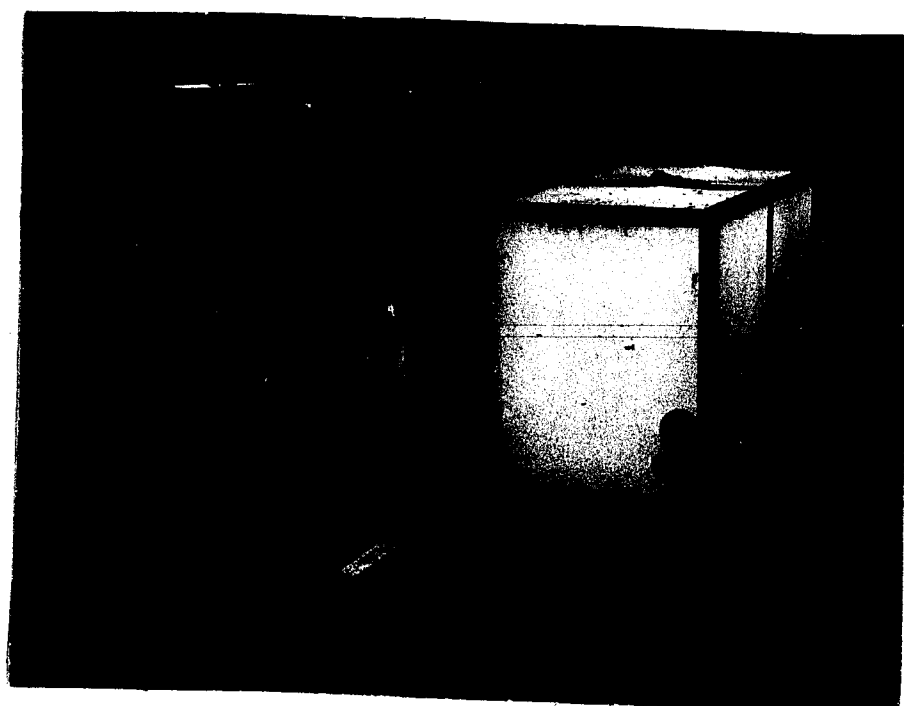
Fig. 6 Kerosene Burner for Drying
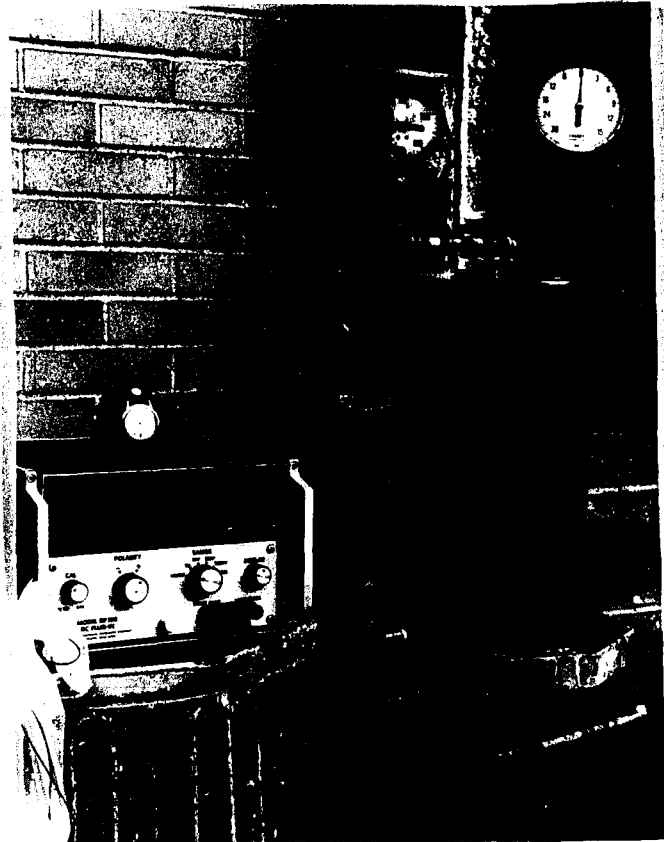
Fig. 7 Sulfur Impregnation
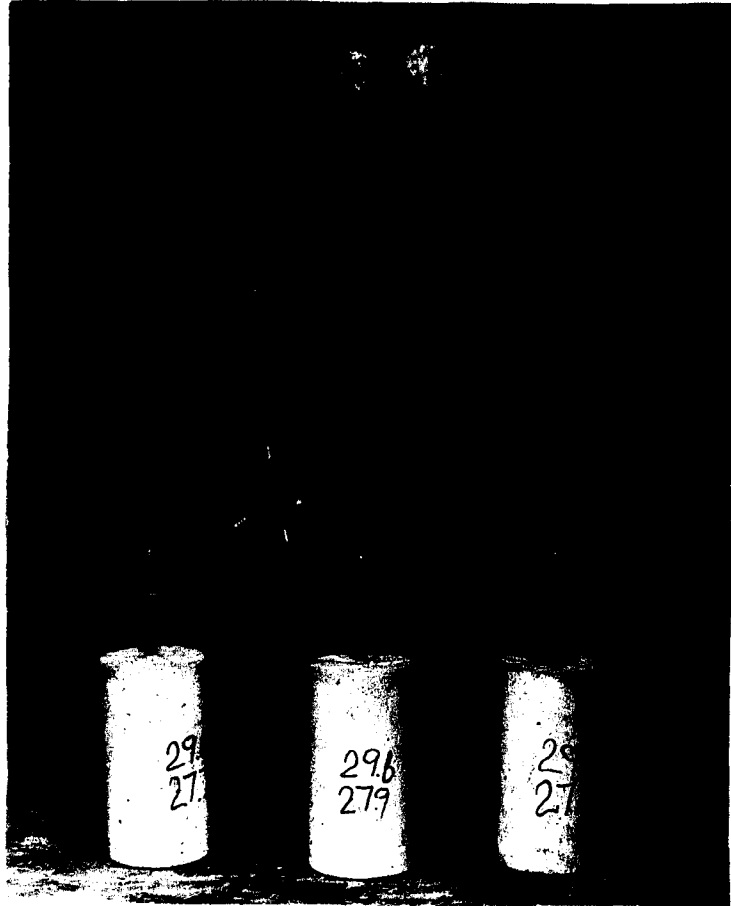
Fig. 8   Impregnation Through Perforated Pipes
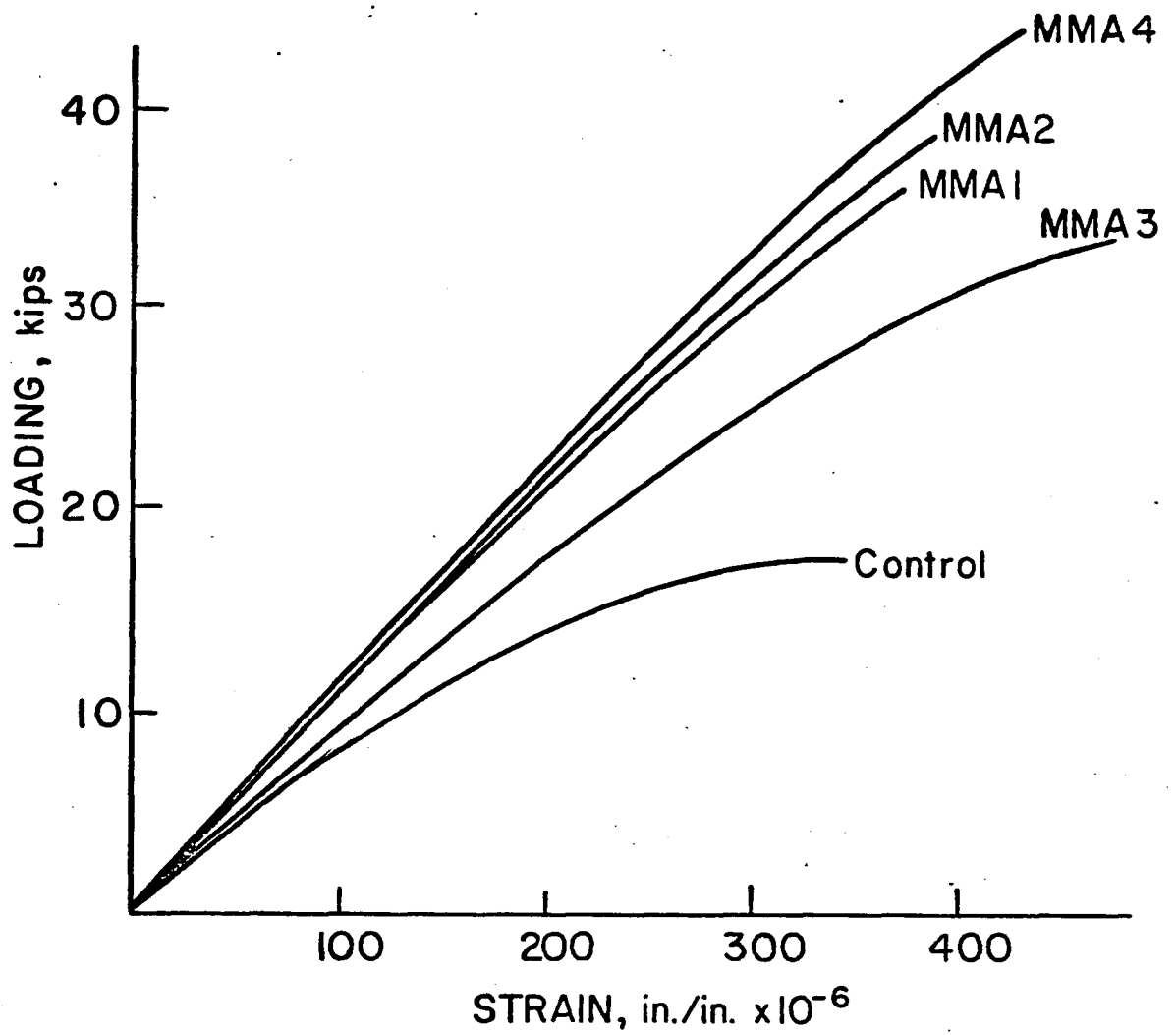
Fig. 9   Tensile Load-Strain Curves
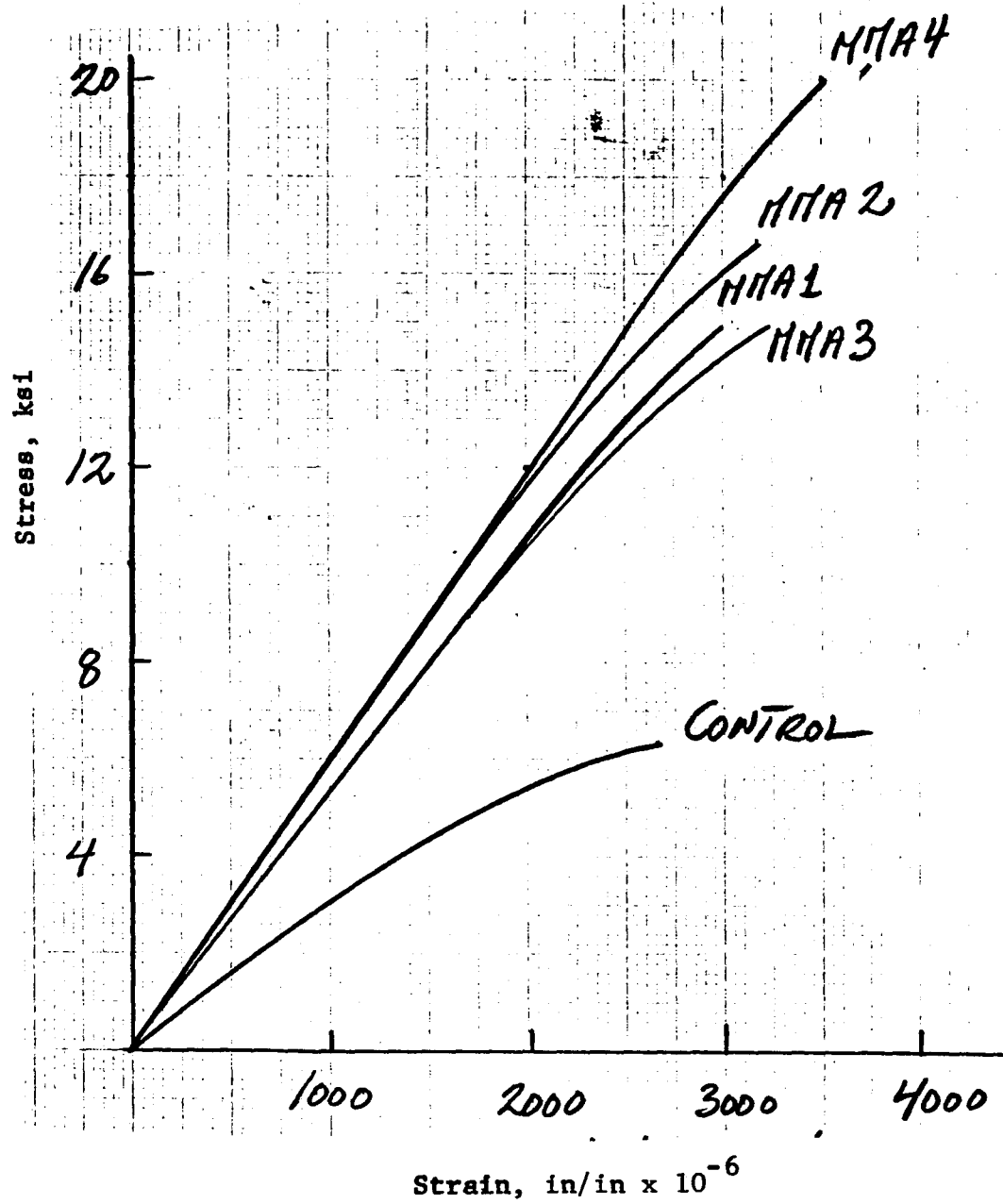
Fig. 10   Compressive Stress-Strain Curves
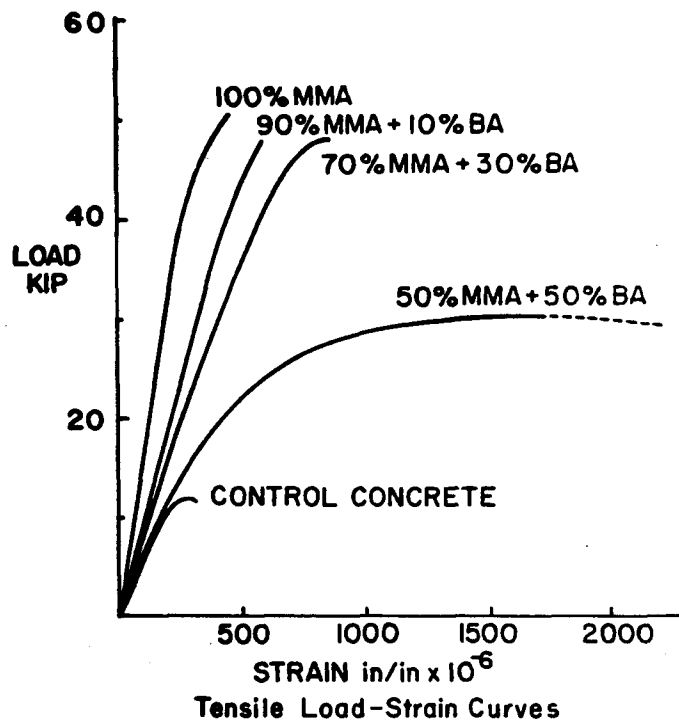
Fig. 11   Tensile Load-Strain Curves for Co-Polymer PIC

MMA = Methyl Methacrylate
BA = Butyl Acrylate
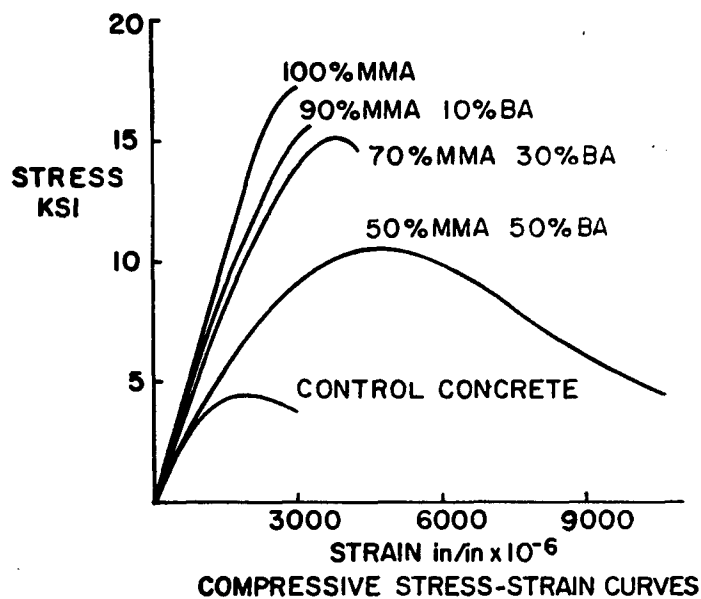
Fig. 12   Compressive Stress-Strain Curves for Co-Polymer PIC
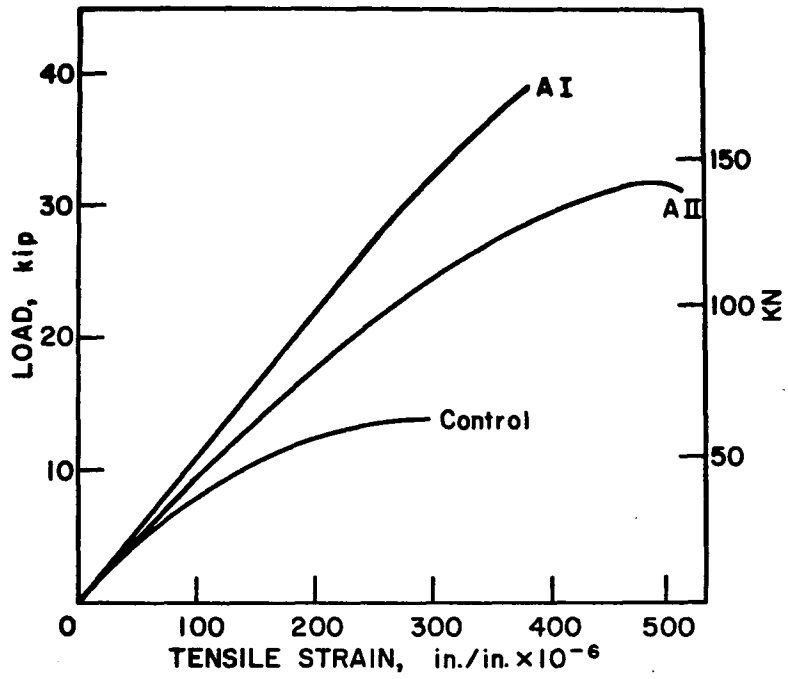
Fig. 13 Tensile Load-Strain Curves for 60/40-MMA/BA Combination
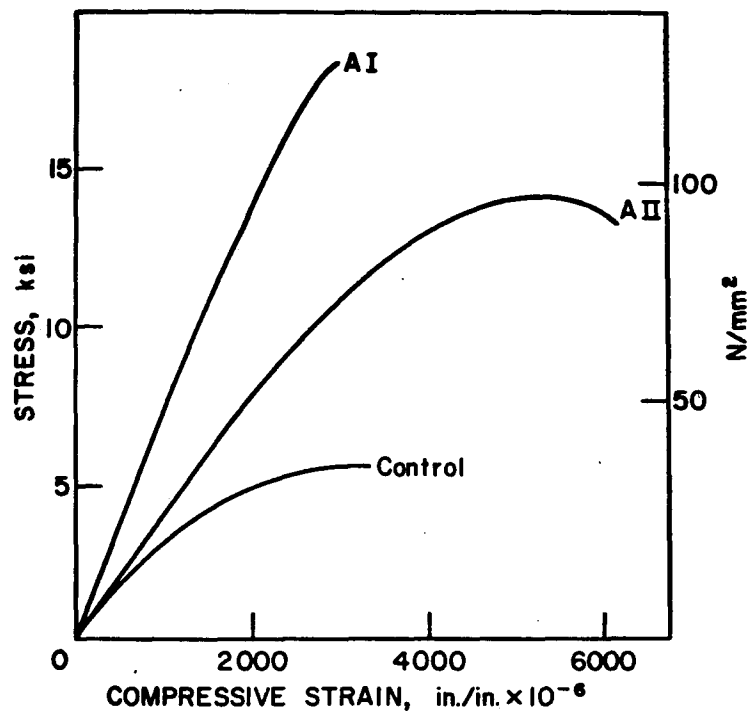
Fig. 14 Compressive Stress-Strain Curves for the 60/40-MMA/BA Combination
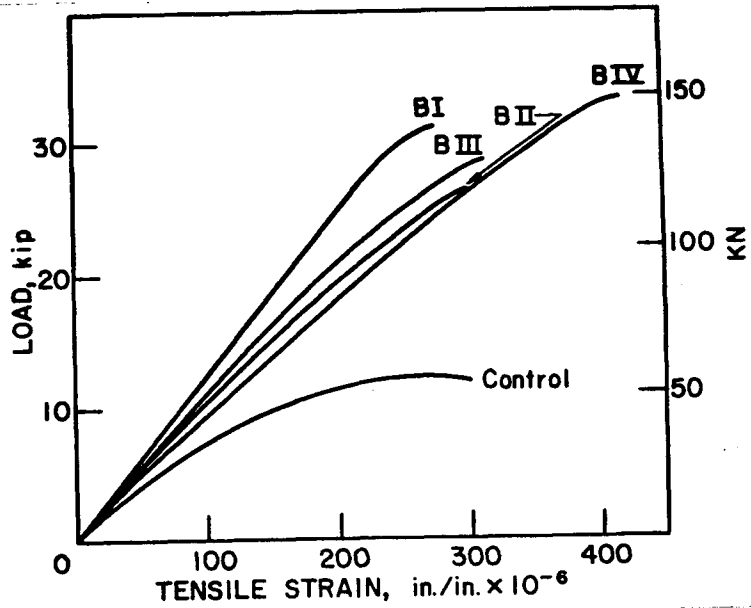
Fig. 15 Tensile Load-Strain Curves for MMA/BA/TMPTMA Combinations
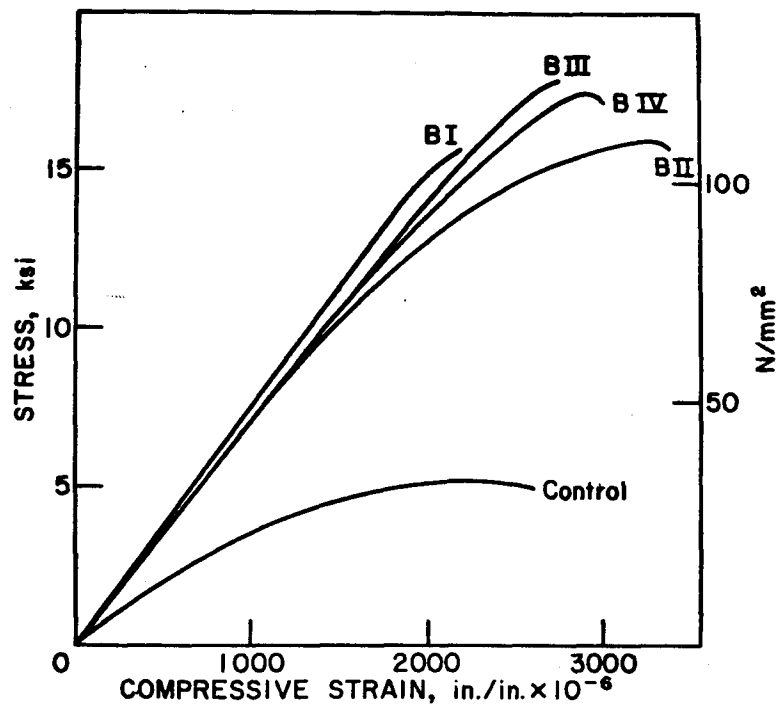
Fig. 16 Compressive Stress-Strain Curves for MMA/BA/TMPTMA
Combinations

-52-
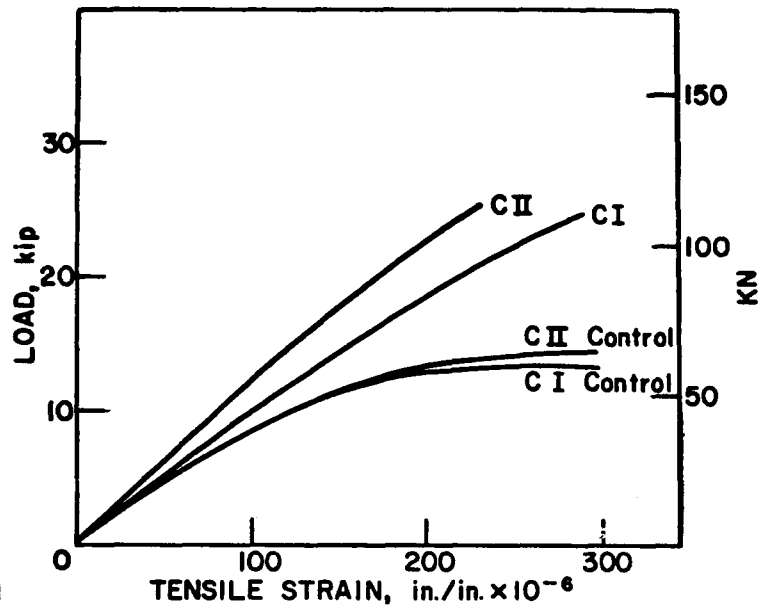
Fig. 17 Tensile Load-Strain Curves for Salt Contaminated
Specimens, C Series
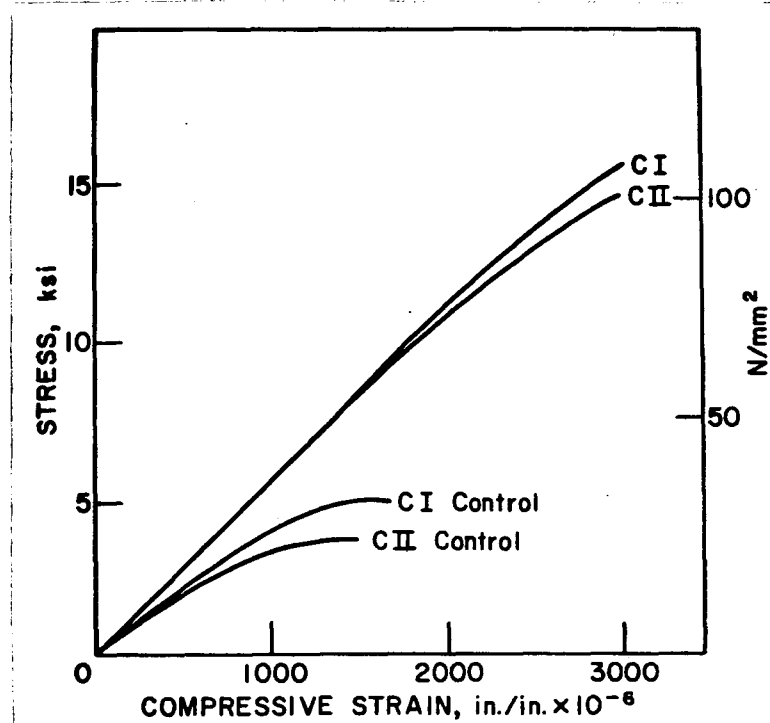
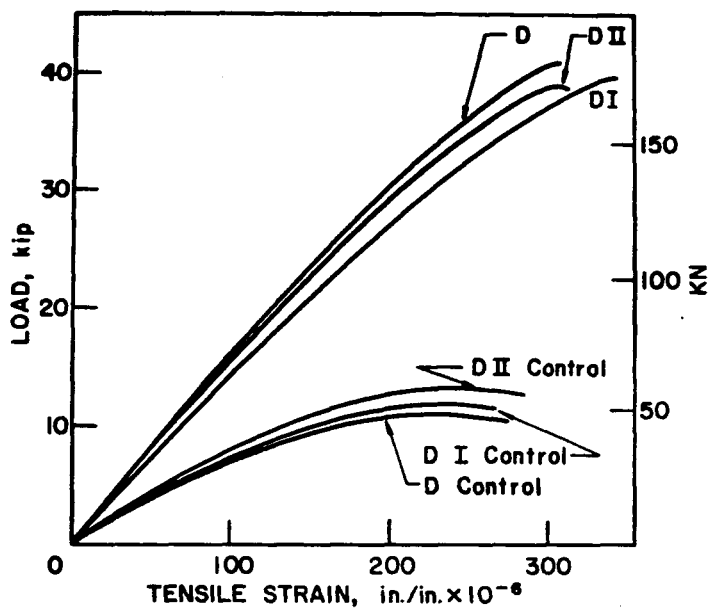**Fig. 18** Compressive Stress-Strain Curves for Salt Contaminated
Specimens, C Series

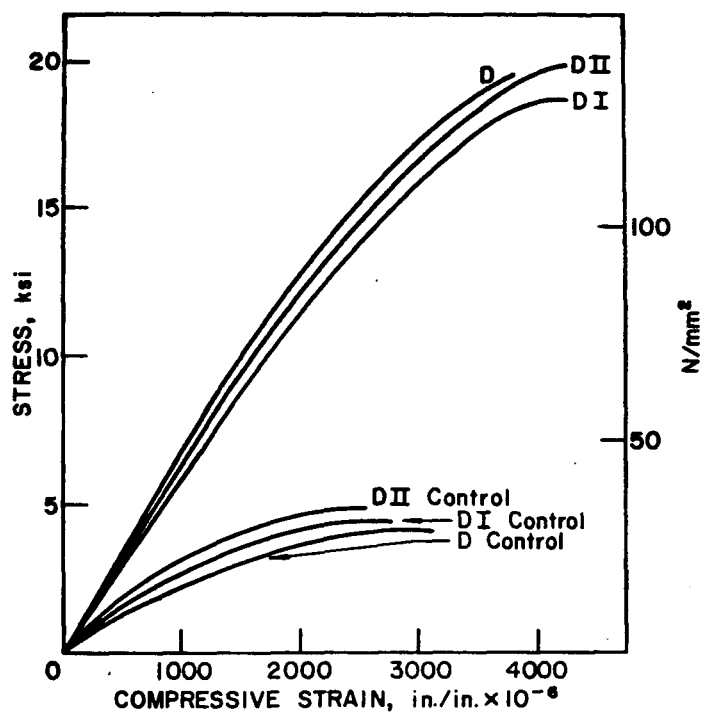**Fig. 19 Tensile Load-Strain Curves for Salt Contaminated Specimens, D Series**

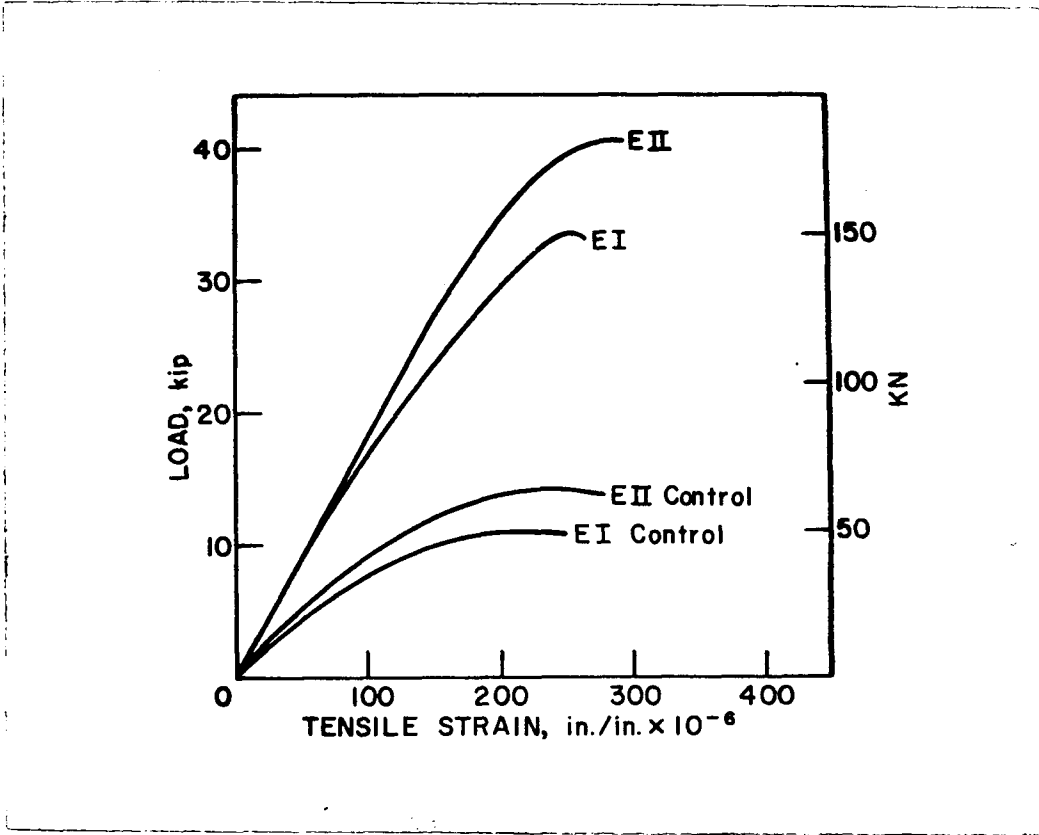**Fig. 20 Compressive Stress-Strain Curves for Salt Contaminated Specimens, D Series**

Fig. 21   Tensile Load-Strain Curves for High Temperature Dried Specimens
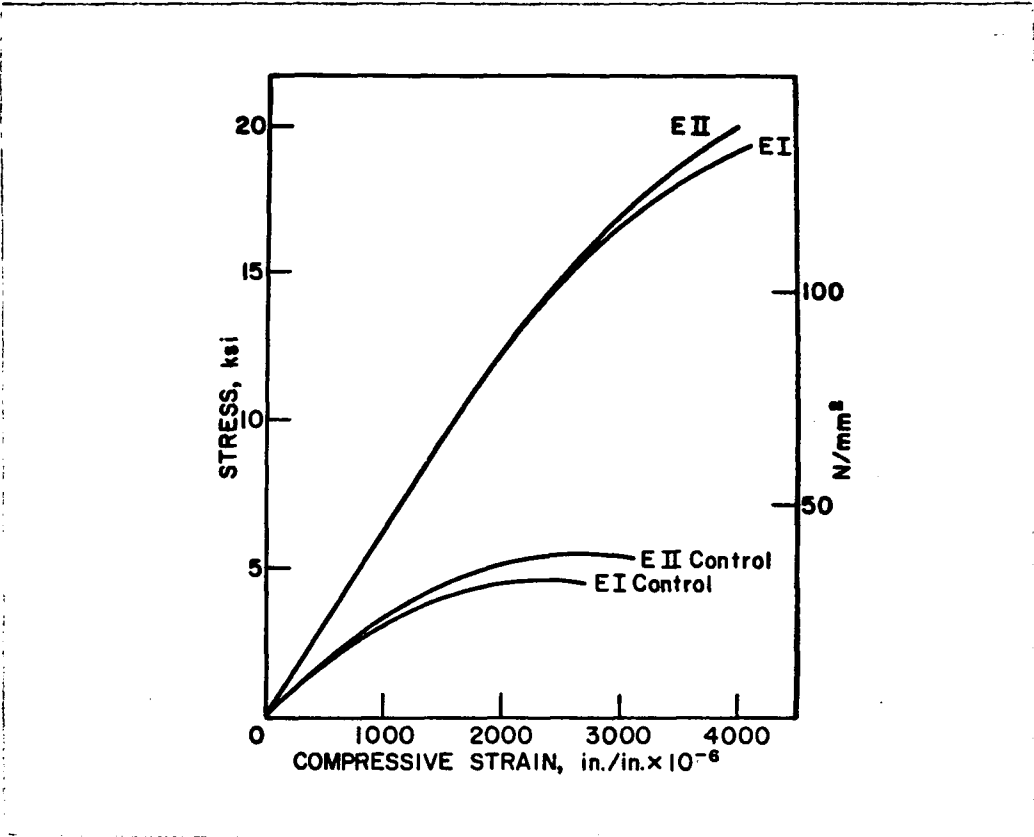
Fig. 22 Compressive Stress-Strain Curves for High Temperature Dried Specimens
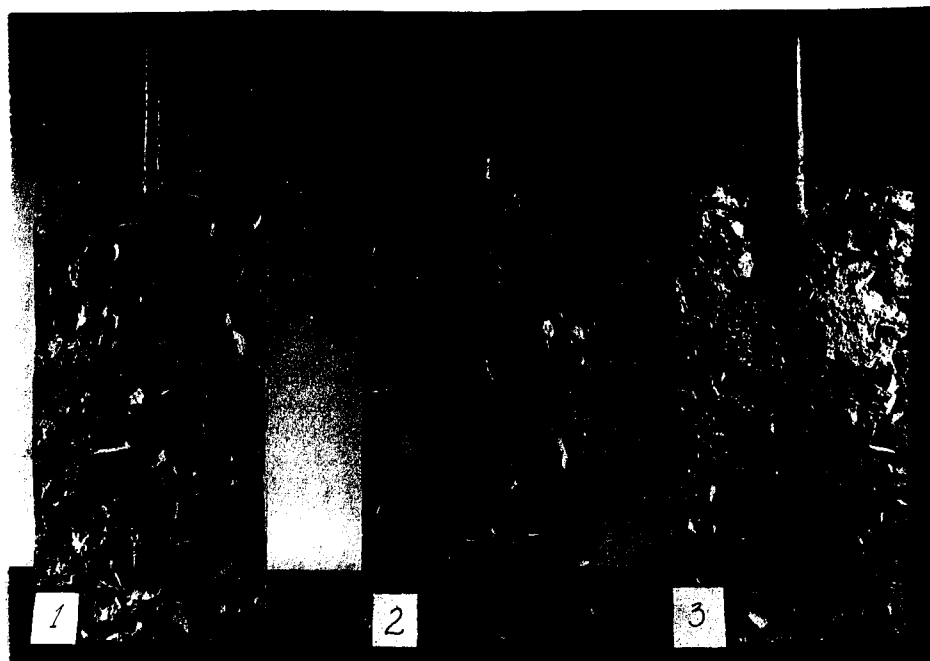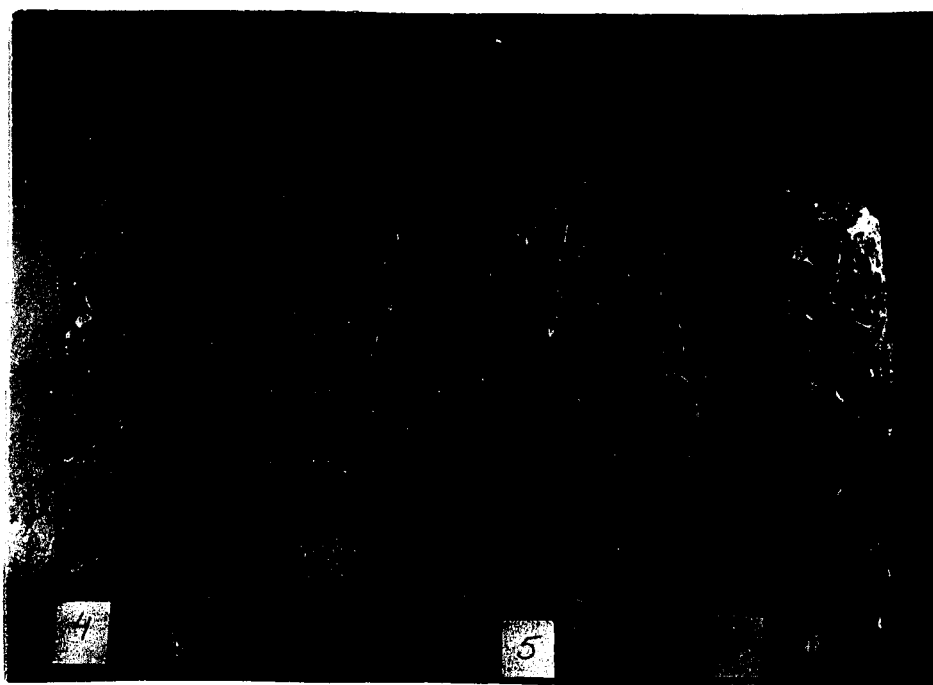
Fig. 23   Specimens 1, 2, 3 with Cast-In Perforated Pipes

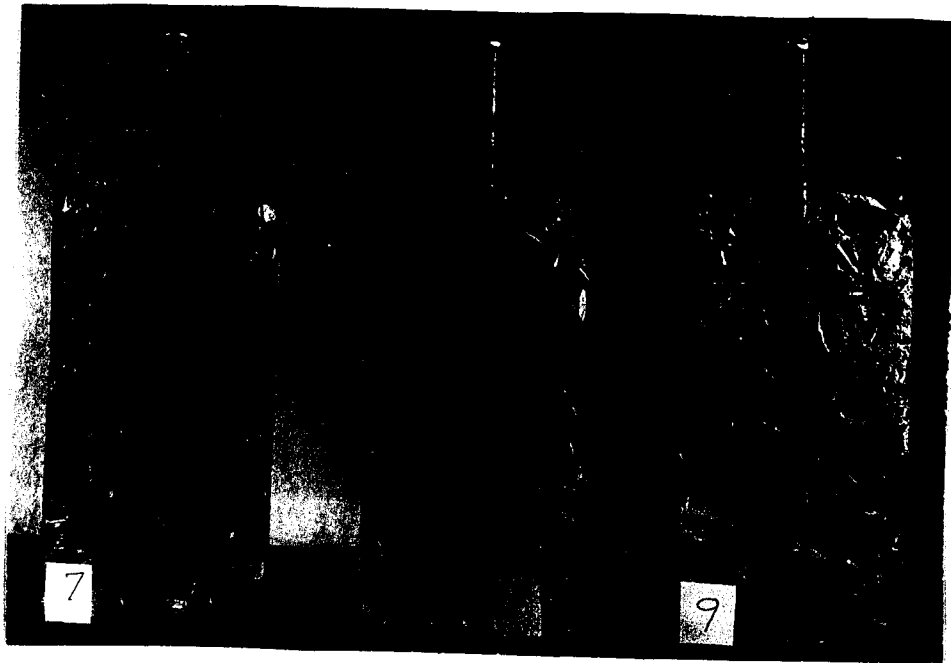Fig. 24   Specimens 4, 5, 6 with Cast-In Perforated Pipes

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Fig. 25   Specimens 7, 8, 9 with Cast-In Perforated Pipes

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