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CONCRETE-POLYMER MATERIALS

THIRD TOPICAL REPORT

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1. SUMMARY

Three distinct types of materials are being investigated in the Concrete-polymer Materials Program. These are (1) polymer-impregnated concrete (PIC), precast portland cement concrete impregnated by a monomer system which is subsequently polymerized in situ; (2) polymer-cement concrete (PCC), water, and aggregate followed by polymerization, monomer is added during mixing of portland cement, and (3) polymer-concrete (PC), a composite material formed by polymerizing a monomer and aggregate mixture.

Concrete-polymer materials are being developed as a new construction material which offers potential advantages of high strength and improved durability, as compared with conventional concrete. Investigations to date indicate that the most successful concrete-polymer material for construction is PIC, which is now receiving the major emphasis in the program. Investigations are continuing with PCC and PC because these materials will have many potential applications if feasible fabrication methods can be developed.

A growing interest in concrete-polymer materials is being shown, not only by Federal agencies, but also by industry, as evidenced by the number of commercial products now available for specialized applications, such as patching compounds and various composite products.

Survey experiments on five new monomer systems for normal temperature applications in PIC have been performed. Of the five monomer systems studied, *t*-butyl styrene and polyester-styrene gave the highest tensile and compressive strengths in 3-inch-diameter by 6-inch-long cylinders. Vinyl chloride, a gaseous monomer, was very difficult to handle during polymerization and gave erratic results.

Two of the elevated temperature monomer systems, 60 percent styrene plus 40 percent trimethylolpropane trimethacrylate (TMPTMA) and 90 percent diallyl phthalate (DAP) plus 10 percent methyl methacrylate (MMA), were selected for additional study. Tests were performed to determine the glass transition temperature (T_g), softening point temperature, and temperature for onset of decomposition of their respective polymers. Tests to determine the activation energy for decomposition and the coefficient of thermal expansion were also performed.

The investigation of techniques of preparing premixed-type concrete-polymer materials was expanded during this report period. Two types of

materials are being investigated: PCC was prepared using integrally mixed monomer, portland cement, water, and aggregate. Polymerization was accomplished after mixing by radiation or by thermal catalytic means. PC, consisting of a polymer binder and aggregate, was prepared by integrally mixing monomer and aggregate followed by thermal catalytic or radiation polymerization.

Only limited success has been achieved with PCC because of several problems that are unique to these studies:

(1) The highly alkaline environment of fresh portland cement concrete causes a reaction with organic monomers that forms acids and other products. The acids interfere with portland cement hydration and generally produce weak concrete.

(2) Most organic monomers are not soluble in water and subsequently produce nonuniform PCC specimens.

(3) The presence of water in PCC interferes with the polymer-aggregate bond.

One series of tests, in which an epoxy resin was added to fresh concrete, developed compressive strengths 60 percent greater than the corresponding control concrete.

Studies with polymer concretes have been more successful. A number of various monomer systems have been used to prepare PC specimens including MMA, styrene, acrylonitrile, MMA plus TMPTMA, polyester-styrene, styrene plus TMPTMA, and DAP plus TMPTMA. These monomers were mixed with Ottawa sand and the specimens were polymerized by radiation and by thermal-catalytic methods. Normally 10 to 25 percent of monomer by weight of concrete was required to wet the aggregate and produce a uniform specimen. The compressive strength of the PC specimens prepared in this manner varied from 10,000 to 17,000 psi. When a mixture of Type II portland cement and No. 30-mesh Ottawa sand was used as aggregate, 10 weight percent polyester-styrene provided specimens with a compressive strength of 23,500 psi.

Investigations of premixed concrete-polymer materials are continuing with emphasis being placed on the identification of improved monomer systems, aggregate selection and processing, and the development of practical polymerization techniques.

The basic procedure for fully impregnating precast concrete described in more detail in the First and Second Topical Reports,^{*1, 2} consists of oven-drying the concrete, evacuating the dried sample, soaking in monomer, wrapping the specimen, and polymerization. Work is continuing on the evaluation of variables, such as drying temperature, age of concrete at impregnation, and method and duration of concrete curing. A series of tests on drying concrete at temperatures ranging up to 482° F (250° C) indicated the compressive strength of MMA-impregnated concrete oven-dried at 482° F was approximately the same as oven-dried at 302° F (150° C). Concrete curing conditions investigated were curing in lime saturated water for periods of 2, 7, and 28 days. After completion of the scheduled limewater cure, specimens were oven-dried, impregnated with MMA, and radiation polymerized. Compressive strengths of the MMA-impregnated specimens averaged between 16,200 to 16,900 psi for the three periods of curing. In a study of the effect of aging prior to impregnation, specimens were impregnated with MMA at ages of 1, 28, and 63 days after the 28-day limewater cure, and polymerized by radiation. Compressive and tensile strengths of these specimens were lower than anticipated, averaging 13,740 psi in compression and 1,060 psi in tension. However, results indicate the age of concrete at impregnation or its initial strength does not significantly affect the final strength.

The testing program is continuing on polymer-impregnated concrete for normal temperature applications. Structural properties of MMA, styrene, acrylonitrile, and MMA plus 10 percent TMPTMA impregnated concrete are given in the First and Second Topical Reports. Structural properties of chlorostyrene-impregnated concrete are described in this report. Chlorostyrene-impregnated concrete specimens developed strength comparable with the previously tested polymer-impregnated concrete. The trend for radiation-polymerized specimens having generally 12 to 17 percent higher strengths than comparable thermal-catalytic polymerized specimens, as reported in the Second Topical Report, was evident in compressive and flexural strengths of chlorostyrene-impregnated concrete, but was not evident for tensile strength.

Compressive strength averaged 16,090 psi for radiation-polymerized chlorostyrene-impregnated concrete and 14,390 psi for thermal-catalytic polymerized chlorostyrene-impregnated concrete, improvements of 205 and 173 percent over unimpregnated control concrete. Modulus of elasticity was 5.6×10^6 psi for both radiation and thermal-catalytically polymerized chlorostyrene-impregnated concrete. Tensile strength

averaged 1,120 psi for radiation-polymerized and 1,200 psi for thermal-catalytic polymerized chlorostyrene-impregnated concrete, which are increases of 167 and 190 percent over control concrete. Flexural modulus of rupture was 2,380 psi for radiation-polymerized chlorostyrene specimens and 1,580 psi for thermal-catalytic polymerized specimens.

The trends observed in creep tests of PIC under 800 psi compressive load, discussed in the Second Topical Report, continued to be evident. Subsequent tests at higher loads at normal and elevated temperatures show substantially less creep than unimpregnated concrete.

The remarkable improvements in durability are evident in continuing tests on MMA, styrene, acrylonitrile, and MMA plus 10 percent TMPTMA impregnated concrete, and in tests of chlorostyrene-impregnated concrete started during this year. Durability properties appear to be related to polymer content and effectiveness in sealing the surface; the method of polymerization does not appear to make a great difference but shows in general slightly better durability for thermal-catalytic polymerization. All PIC specimens show improvements in freeze-thaw, sulfate, HCl, and H₂SO₄ resistance as compared with unimpregnated control concrete.

In the freeze-thaw test, the best durability results are being given by radiation-polymerized MMA specimens and thermal-catalytic polymerized MMA plus 10 percent TMPTMA specimens, which have exhibited 0.5 and 0 percent weight losses at 7,220 and 4,660 cycles, respectively. Unimpregnated control concrete failed the test at 740 cycles, with a weight loss of 25 percent. Sulfate attack, as determined by the accelerated sulfate test, shows small expansions for all PIC at ages ranging from 300 to 720 days, except for radiation-polymerized acrylonitrile and radiation-polymerized chlorostyrene specimens, which have expansion of 0.462 percent at 540 days and 0.168 percent at 300 days, respectively. Unimpregnated control specimens have expanded 0.467 percent at 480 days. All PIC specimens have shown excellent durability in 15 percent HCl ranging from more than 178 to over 667 percent improvement over control concrete. Exposure tests to 15 percent H₂SO₄, which is much more severe than 15 percent HCl for concrete, have been concluded and show improvements for polymer-impregnated specimens ranging from 43 to 157 percent over unimpregnated control concrete.

Experiments to partially impregnate concrete with selected monomer systems have indicated potential uses for improving the durability of ordinary concrete. Partial impregnation to controlled penetration depths reduces the amount of monomer required as compared

*Footnote indicators refer to references listed at end of report.

with full impregnation. Durability test results indicate a generally beneficial but somewhat erratic improvement in durability. Additional studies are scheduled to determine the required depth of penetration to obtain a reliable improvement in durability.

Development of PIC is continuing for high-temperature applications, as would be encountered in a saline water distillation plant. Screening test results on four monomer systems for PIC were reported in the Second Topical Report. Two of these monomer systems were selected for continued evaluation: 60 percent styrene plus 40 percent TMPTMA and 90 percent DAP plus 10 percent MMA. Additional strength and elasticity tests were made to confirm earlier results, and environmental testing in the brine loop system has been started. The brine loop tests consist of exposure to synthetic sea water brines at temperatures of 250° F (121° C) and 290° F (143° C). These tests have not been running long enough to report significant results.

A 28-inch-diameter by 12-inch-long CP-type concrete environmental test chamber specimen (weight 720 pounds including 110 pounds of reinforcing steel), was fully impregnated with 60 percent styrene plus 40 percent TMPTMA. This is the largest specimen that has been impregnated to date. Results indicate that walls of the thickness required for large desalting vessels can be successfully impregnated.

Fundamental studies conducted included determining the compressive stress-strain curve for MMA-impregnated concrete, development of PIC from low-strength concrete and from insulating-type lightweight concrete, studies on increasing polymer loading in hardened portland cement paste, and scanning electron microscopy for observing the polymer in PIC.

The compressive stress-strain curve for MMA-impregnated concrete, disclosed the highly significant fact that deformation is linear up to 75 percent of ultimate strength and departs less than 7.5 percent from linearity at failure, whereas the deformation of unimpregnated concrete increasingly departs from linearity as the loading increases. PIC appears to show elastic behavior which will have an important bearing on structural design.

Four methods of nondestructive testing were evaluated for use with concrete-polymer materials.

Preliminary tests indicated gamma radiography could not distinguish between monomer and polymer, and

could not detect the small amounts of polymer present in impregnated concrete.

Neutron radiography did not give meaningful density measurements of impregnated concrete and could not distinguish between monomer and polymer or MMA and water.

Modulus of elasticity appears to satisfy some of the requirements for nondestructive testing of concrete-polymer materials, but under actual test extensive scatter in plots of strength versus modulus were encountered.

The Schmidt Impact Hammer has been used in nondestructive testing of conventional concrete. When this device was applied to PIC, a correlation was found between polymer loading and rebound number. An extensive series of tests with the hammer indicated that within limitations it may be used to evaluate PIC.

The use of PIC as a construction material is being investigated for use in many areas. One area is in desalting plants where preliminary design of several vessels has been completed. Other areas being investigated are pipe, housing construction, marine applications, luminaires, and bridge decks.

Concrete drain tile applications are under study in the laboratory and in field tests conducted at the Westlands Experimental Drainage Plot, Tranquillity, California. The laboratory test program included three-edge bearing tests and tests of water absorption and resistance to sulfate attack. The field test plot is in an area in which the soil and the water have a high-sulfate content. The examination of test plot drainpipe is scheduled to begin next year.

A joint program with the American Concrete Pipe Association (ACPA) for PIC sewer and culvert pipe has been completed. Test results show a sufficient improvement in strength for both three-edge bearing and hydrostatic tests for the PIC to warrant further investigation.

PIC sewer pipe has been installed in the city of San Diego sewer system. The pipe will be examined periodically.

The cooperative program with the Naval Civil Engineering Laboratory (NCEL) to determine the suitability of PIC for underwater applications is continuing. A series of concrete spheres and test cylinders were impregnated and returned to NCEL for evaluation. Testing is currently underway.

2. INTRODUCTION

2.1. BACKGROUND AND PROGRAM DESCRIPTION

The Concrete-polymer Materials Program was initiated by the U.S. Atomic Energy Commission's (AEC) Division of Isotopes Development in 1967 as a cooperative effort between Brookhaven National Laboratory (BNL) and the Bureau of Reclamation (USBR) of the U.S. Department of the Interior. The Office of Saline Water (OSW) of the Department of the Interior also began support and contributed to the planning of the program in the same year. The OSW was particularly interested in the possible use of concrete-polymer in the construction of vessels for multistage distillation units in water desalination plants. To date, two topical reports have been issued. The First Topical Report,¹ issued in January 1969, covered the work to the end of June 1968. Work performed through the end of June 1969 was described in The Second Topical Report.² The current Third Topical Report describes the work accomplished through the end of June 1970. During this period the work at BNL and the USBR has continued to be sponsored by the AEC, the OSW, and the USBR. In August 1969, the Federal Highway Administration, U.S. Department of Transportation, initiated support for work at BNL on the use of concrete-polymer materials in highway applications. A progress report on this work has been written.³ Because of the possible value of concrete-polymer materials for applications of concern to other governmental agencies, arrangements were made for each of these agencies to cooperate in the program as described in Section 2.3.

The long-range objectives of the joint program are the investigation and development of concrete-polymer composite materials for use as new and improved materials of construction. The main tasks of the program include (1) evaluation and selection of monomers for incorporation in concrete-polymer composites, (2) development of process techniques for the preparation of the composites, (3) testing and measurement of pertinent physical, mechanical, and chemical properties, (4) development of applications, including preparation and testing of full-scale specimens, and (5) development of preliminary cost estimates for concrete-polymer products.

The general procedure followed in the preparation and testing of PIC specimens consists of fabricating the specimens at USBR and shipping them to BNL for impregnation and polymerization. The completed PIC specimens are returned to the USBR for measurements

of structural and durability properties. Both partial and complete penetration of the concrete with monomer are being evaluated.

In addition to the work on PIC, studies are being made at both laboratories on the incorporation of monomers in premixed materials. Two materials, PCC and PC are being studied. The PCC consists of a monomer added to a water portland cement aggregate premix which is subsequently polymerized in place. PC consists of an aggregate bound with a monomer which is premixed and subsequently polymerized.

Studies to determine the fundamental structure of the concrete-polymer composites are also underway at both laboratories. The studies include microscopic and petrographic investigations and the formulation of theories and mathematical models for predicting the properties of the composite.

2.2. SUMMARY OF PREVIOUS WORK

The principal findings published in the First and Second Topical Reports may be summarized as follows. Liquid and gaseous monomers can fully penetrate concrete and can be polymerized in situ by both radiation and thermal-catalytic techniques. To obtain a maximum polymer loading in the concrete, it was necessary to dry the concrete to constant weight, evacuate the specimen to remove air, soak it in a low-viscosity liquid monomer, pressurize with nitrogen, and wrap the specimen in polyethylene sheet to reduce evaporation prior to polymerization. Cylindrical specimens with dimensions of up to 6 inches in diameter and 12 inches long were fully impregnated, and weight loadings of the order of 6 to 7 percent of that of the dried unimpregnated concrete were measured.

The structural and durability properties of PIC being considered for normal temperature applications were measured. Five monomer systems under test were MMA, styrene, MMA plus 10 weight percent TMPTMA, acrylonitrile and chlorostyrene.

Significant improvements in structural properties were obtained. Concrete impregnated with MMA plus 10 percent TMPTMA had the highest compressive strength observed to date. Compare with a control strength of 5,270 psi for unimpregnated concrete, a maximum strength of 22,490 psi was obtained for a specimen containing 6.3 weight percent monomer polymerized by radiation. This is an increase over the controls of 327 percent.

A comparison of the compressive and tensile strengths obtained for radiation and thermal-catalytic polymerization indicated that with the five monomer systems studied, polymerization by radiation resulted in greater strengths than were obtained with the thermal-catalytic method. Differences, generally about 12 to 17 percent, were observed. With styrene-impregnated specimens, differences of about 35 percent were obtained.

Remarkable improvements in durability properties were obtained. In general, MMA-impregnated specimens showed the greatest improvement. Acrylonitrile showed the least. The durability properties of MMA-impregnated specimens are compared with those of the control specimens below.

a. Freeze-thaw durability improved by >366 percent (760 percent for one survey series specimen).

b. Chemical attack by sulfate brines reduced to negligible values compared with the severe attack observed on the control specimens.

c. Resistance to chemical attack by 15 percent hydrochloric acid increased by >318 percent.

d. Water permeability decreased to negligible values.

e. Water absorption decreased by as much as 95 percent.

The durability properties appeared to be dependent on the polymer content.

The mechanical properties of PIC at elevated temperatures were investigated. Four monomer systems were screened for use in composites having potential as a material of construction for distillation units in water desalting plants. High-strength OSW-type concrete* impregnated with a 60 weight percent styrene plus 40 weight percent TMPTMA mixture showed the greatest strength (19,720 psi at 76° F (24° C)) and the smallest decrease in strength at elevated temperatures (18,600 psi at 290° F (143° C)). Based upon the results of the screening tests, the 60 weight percent styrene plus 40 weight percent TMPTMA mixture and a 90 weight percent DAP plus 10 weight percent MMA mixture were chosen for continued evaluation.

*Maximum aggregate size, 1.5 inches, Type V cement and fly ash; compressive strength, 8,743 psi at age 120 days.

Tests to determine the structural properties of steel-reinforced PIC were started. Pullout tests of steel reinforcing bars indicated bond strengths up to 700 psi for MMA-impregnated specimens, compared with 238 psi for the controls. Concrete specimens containing 2 volume percent of randomly oriented-steel fibers and impregnated with 9.7 weight percent MMA exhibited compressive strengths up to 23,828 psi, an increase of 383 percent over the value for the unimpregnated reinforced control and of 477 percent over that for the nonreinforced control. The impregnation of fiber-reinforced slabs increased the modulus of rupture to as much as 3,000 psi, which corresponds to improvements of 418 and 173 percent over those for the nonreinforced and reinforced controls, respectively. Tests conducted on impregnated mortar containing 8 volume percent steel fiber indicated that the ultimate flexural strength of plain mortar was increased by a factor of 15 by the addition of fiber and impregnation with MMA.

The potential application of PIC pipe is being evaluated. Sixty sections of PIC drain tile are being field-tested in a highly saline soil. A program was initiated in cooperation with the ACPA for the evaluation of 12- and 24-inch-diameter unreinforced and reinforced pipe.

In this, the Third Topical Report, data are presented on the structural properties of specimens impregnated with chlorostyrene. Extended data are given on the durability properties, such as freeze-thaw durability, acid and sulfate resistance, as well as on the longer-term mechanical properties such as creep. Measurements of the mechanical and physical properties, and resistance to chemical attack by brine, at elevated temperatures (up to 290° F (143° C)) are also presented.

The investigations on the incorporation of monomers in premixed concrete were continued and a study of PC composites was initiated.

In addition, programs to evaluate the potential application of PIC pipe were continued.

2.3. COOPERATIVE PROGRAMS WITH GOVERNMENTAL AGENCIES

Several governmental agencies have joined the Concrete-polymer Program in a cooperative effort to investigate these materials for applications of specific interest.

The OSW has a direct interest in improving the properties of concrete so that it can be used effectively as a material of construction in, for example,

multistage distillation units in water desalting plants. The chief characteristics being sought are higher strength at temperatures up to 290° F (143° C) and resistance to chemical attack by hot brine and distilled water. To date, surface coatings have been found unsatisfactory. The Materials Technology Division of the OSW is providing support for a portion of the Concrete-polymer Program and contributes to its planning.

The Federal Highway Administration of the U.S. Department of Transportation is interested in the potential use of concrete-polymer materials for a number of highway applications. Improvements in the strength and durability of concrete would be of great value in improving highway pavements, bridge decks, and culverts. The use of impregnated insulating-type concrete may also have applications in breakaway lampposts and for precast bridge decks. On the basis of preliminary tests performed early in 1969, the Federal Highway Administration, in a cooperative program with the AEC, initiated support for a research program

at BNL. A report describing the initial results of this effort has been written.³

The NCEL of the Department of the Navy is interested in concrete-polymer materials of high strength and low water permeability for underwater applications. Exploratory experiments on impregnated concrete hulls have been conducted in cooperation with BNL during the last 2 years. The results of these experiments are given in Section 9 of this report.

The Bureau of Mines, U.S. Department of the Interior is interested in applying the technology developed in the Concrete-polymer Program to the improvement of the strength properties of rock in mines. The purpose is to effect a chemical stabilization of underground structures and supports in mines. Early in 1969, several preliminary tests were made in which a porous dacite was impregnated with MMA. Based on the improved strength of these specimens, the Bureau of Mines, in a cooperative program with the AEC, initiated support for a research program at BNL. The first progress report on this work has been written.⁴

3. MONOMER SURVEY EXPERIMENTS

3.1 MONOMERS FOR THE IMPREGNA- TION OF CONCRETE FOR NORMAL TEMPERATURE APPLICATIONS

The basis for the selection of monomers for the impregnation of precast concrete for use under normal temperature conditions has been described in detail in a previous report.²

Five monomer systems were selected for study: Vinyl chloride, vinylidene chloride, t-butyl-styrene, chlorostyrene, and a mixture consisting of 10 percent polyester plus 90 percent styrene. The above monomers represent a cross-section of monomer properties as shown in Table 1.

A series of 3-inch-diameter by 6-inch-long concrete cylinders was impregnated with the above monomers and tested for compressive and tensile strengths. The results are shown in Table 2.

Except for the vinyl-chloride specimens, all other specimens were removed from the impregnator and wrapped with polyethylene film prior to polymerization by radiation. Vinyl chloride is a gas having a vapor pressure of about 34 psi at 70° F (21° C). Due to the high-monomer vapor pressure, the monomer-saturated specimen could not be handled in the usual manner. The specimens were polymerized in the sealed impregnation vessel by placing the entire unit in the radiation field. Prior to polymerization the excess condensed vinyl-chloride monomer in the vessel was removed by displacement with water. The vessel was then pressurized to about 40 psi to keep the monomer from diffusing out of the specimen. The excess vinyl chloride was removed from the vessel in order to minimize the extremely high pressures that could result from the rapid polymerization of bulk monomer.

Although reasonable precautions were taken during the impregnation and polymerization steps with vinyl chloride and the low-boiling point liquid, vinylidene chloride, a high degree of polymer loading as compared to those obtained with low-vapor pressure monomers could not be obtained. The data in Table 2 indicate that the highest compressive and tensile strengths were obtained with t-butyl-styrene and a mixture of 10 percent polyester plus 90 percent styrene.

3.2 MONOMERS FOR THE IMPREGNA- TION OF CONCRETE FOR ELEVATED TEMPERATURE APPLICATIONS

Four monomer systems have been investigated for potential use under elevated temperature conditions.² The selection of these systems was based on the chemical and physical properties of the polymers formed and on the ability of the monomers to polymerize by thermally initiated processes.

The four systems were:

- (1) A mixture of 60 percent styrene plus 40 percent TMPTMA,
- (2) A mixture consisting of 90 percent DAP plus 10 percent MMA,
- (3) Acrylonitrile, and
- (4) A mixture consisting of 90 percent MMA plus 10 percent TMPTMA.

Test series consisting of OSW-type concrete impregnated with each of the four monomer systems were prepared. The specimens were used in compressive strength tests at 76° F (24° C), 250° F (121° C) and 290° F (143° C) and in tensile splitting strength tests. On the basis of the results obtained, the 60 percent styrene plus 40 percent TMPTMA and the

Table 1

SUMMARY OF MONOMER AND POLYMER PROPERTIES

Monomer	Monomer vapor pressure mm Hg	Monomer boiling point		Glass temperature T _g		Polymer physical characteristics
		° F	° C	° F	° C	
Vinyl chloride	1,660 at 68° F (20° C)	7	-14	167	75	White powder
Vinylidene chloride	599 at 77° F (25° C)	90	32	212	100	White powder
Tert.-butyl styrene	1 at 115° F (46° C)	424	218	293	145	Hard, glasslike
Chlorostyrene	0.68 at 68° F (20° C)	356	180	194	90	Hard, glasslike
10 percent polyester 90 percent styrene	4.5 at 68° F (20° C)	293	135	200	93	Hard, glasslike

Table 2
MONOMER SURVEY TEST RESULTS

3- by 6-inch Impregnated Specimens

Sample	Monomer	Weight increase percent	Compressive strength, psi	Young's modulus 10 ⁶ psi	Tensile strength, psi
CP-1A-13D	Vinyl chloride	3.0	—	—	835
CP-1B-14F		5.5	—	—	565
CP-1B-15D		5.0	—	—	620
CP-1A-12A		4.8	9,050	4.28	—
CP-1A-14A		3.6	12,640	4.26	—
CP-1B-15F		5.0	9,020	3.96	—
CP-1A-12D	Vinylidene chloride	2.0	—	—	410
CP-1A-15A		2.8	—	—	420
CP-1A-12C		1.9	—	—	275
CP-1A-12E		1.5	6,140	2.50	—
CP-1A-13F		2.5	6,680	3.15	—
CP-1A-11D		2.5	7,130	3.47	—
CP-1A-14F	t-butyl styrene	5.8	—	—	1,475
CP-1A-13E		6.0	—	—	1,375
CP-1A-15D		5.4	—	—	1,490
CP-1A-11C		5.9	17,820	6.38	—
CP-1A-15E		5.3	18,880	6.10	—
CP-1A-11F		6.0	17,960	6.62	—
CP-1A-15F	10 percent polyester— 90 percent styrene	5.0	—	—	965
CP-1A-12F		5.5	—	—	1,090
CP-1A-14E		5.7	—	—	1,360
CP-1A-13C		5.4	16,830	5.37	—
CP-1A-15C		5.5	16,690	5.37	—
CP-1A-11E		5.5	15,200	5.04	—

All specimens dried at 302° F (150° C) prior to monomer soak.
All monomers polymerized by radiation.

90 percent DAP plus 10 percent MMA systems were selected for further study.

Both of these mixtures were formulated in the laboratory as part of a monomer survey study and are at present not available as commercially prepared systems. High-temperature monomer systems, except for possibly acrylonitrile, have high viscosities and therefore cannot diffuse readily through concrete. In order to use these monomers, the viscosity is reduced by addition of a lower viscosity monomer. A typical example is the 60 percent styrene plus 40 percent TMPTMA system. TMPTMA is a high-temperature cross-linking monomer having a viscosity of about 46 centipoises at room temperature. It cannot penetrate a 6-inch-diameter OSW-type concrete test specimen. Styrene, on the other hand, has a viscosity of about 0.5 centipoise but produces a polymer with a low-softening point. The 60 percent styrene plus 40 percent TMPTMA mixture has a viscosity of 1.7 centipoises, low enough to penetrate the OSW-type concrete specimens.

In addition to the problem of proper viscosity, the chemical union between the two monomer species must result in a copolymer having the desired properties. Not all combinations of monomers react to form a copolymer. At times a given monomer will not react in the presence of another monomer, and the result is a homopolymerization reaction.

Another important factor is the relative rate at which each of the monomeric species is consumed in the reaction. If the ratios of these rate constants are not known, monomer mixtures of the proper proportions cannot be readily formulated to give only copolymer. One of the monomers will become depleted during the reaction, with the result that a copolymer and a homopolymer are formed. If the depleted monomer is styrene as in the case of the styrene-TMPTMA mixture, the excess TMPTMA will continue to react by forming homopolymer. Since both the styrene-TMPTMA copolymer and the TMPTMA have desirable temperature properties the situation is not as critical as it might be if the TMPTMA became depleted and styrene homopolymer was formed. Polystyrene with a softening point of approximately 230° F (110° C) would be a very weak link in the system.

The same considerations apply to the DAP plus MMA system.

Except for the basic monomers comprising the above mixtures, information regarding the comonomer or copolymer properties is not available.

Two very important properties for high-temperature applications are the thermal stability and the thermal-mechanical behavior of the polymers.

A study on the thermal decomposition of the 90 percent DAP plus 10 percent MMA system and the 60 percent styrene plus 40 percent TMPTMA system was made isothermally with a Perkin-Elmer Differential Scanning Calorimeter. Specimens were subjected to temperatures ranging from about 257° F (125° C) to 617° F (325° C). The results are shown in Figure 1.

Figure 1. Thermal decomposition of OSW polymers.

It was determined that the onset of decomposition for the DAP plus MMA occurs at about 392° F (200° C). For the styrene plus TMPTMA mixture, decomposition starts at about 437° F (225° C). As the temperature is increased above the onset of decomposition an acceleration occurs.

An Arrhenius plot of the data from each of the above systems is given in Figure 2. Activation energies for decomposition of 22.0 and 17.0 kcal/mole were calculated for the DAP plus MMA and the styrene plus TMPTMA systems, respectively.

Thermal-mechanical properties such as the glass transition temperature, the coefficient of linear expansion, and the softening point were also measured for the above systems. These are shown in Table 3. The data show that the softening points for both systems are above the onset of decomposition temperature, indicating that failures due to softening of the polymer cannot occur in the ranges of temperature specified by OSW.

Figure 2. Arrhenius plots for thermal decomposition of OSW polymers.

The styrene plus TMPTMA monomer mixture is easily polymerized by the thermal-catalytic method. Approximately 1 percent by weight of benzoyl peroxide catalyst is dissolved in the monomer mixture prior to the impregnation step. Polymerization takes place after several hours at an oven temperature of about 167° F (75° C).

The DAP plus MMA system involves more severe polymerization requirements. This reaction takes place in two stages. During the first stage a temperature of about 250° F (121° C) is required until the gel phase is formed. The temperature is then increased to about

285° F (141° C) for a final cure. Approximately 5 percent by weight of t-butyl perbenzoate catalyst is used for this system. The amount of time required at each temperature phase must be determined experimentally and depends on the size of the specimen and also on the oven heat capacity. Rigid controls must be upheld in order to ensure complete polymerization.

3.3 POLYMER-CEMENT CONCRETE STUDIES

The number of potential applications of PCC and PC is believed to exceed that of PIC. Very little effort has been devoted to this area in the past. During the second half of this report period, investigations of integrally mixed polymer modified concrete were expanded at both the USBR and BNL laboratories. These investigations are proceeding in two major areas: PCC and PC.

3.3.1 BNL Studies

A series of experiments has been conducted to supplement previous reported work¹ on the addition of monomers to fresh concrete mixes. This series has been directed toward observing the problems involved in producing a homogeneous mixture of monomer in premix.

The basic premix components for this study consisted of three parts sand, one part Type II air-entrained cement, and one-half part water. To this mixture was added 10 percent by weight of a monomer listed in Table 4. Each of the monomers listed was used. After

Table 3

THERMAL AND THERMAL-MECHANICAL PROPERTIES OF OSW POLYMERS

Monomer mixtures	Polymer Properties					
	Glass transition temperature (T _g), °F	Softening point ¹ , °F	Onset of decomposition, °F	Activation energy ² , kcal/mole	Coefficient of thermal expansion 10 ⁻⁵ /°C	
					Below T _g	Above T _g
90 percent diallyl phthalate- 10 percent methyl methacrylate	367	658	392	22	4.7	13.3
60 percent styrene- 40 percent trimethylol propane trimethacrylate	417	705	437	17	3.8	12.8

¹ Temperature at which a probe 0.030-inch diameter penetrates 0.025 inch deep at 625 psi.

² Activation energy for decomposition.

Table 4

MONOMER SURVEY OF POLYMER-CEMENT CONCRETE

(7-day cure in H₂O)

Sample No.	Monomer 10 percent by weight	Polymerization technique	Radiation dose 10 ⁶ rads	Compressive strength, psi
C1	Control	—	—	3,450
C2	Control	—	—	3,625
C3	Control	—	—	3,500
A1	Acrylamide	Radiation	2.0	—
A2	Acrylamide	Radiation	2.0	—
A3	Acrylamide	Radiation	2.0	—
ZD1	Zinc diacrylate	Radiation	4.0	1,875
ZD2	Zinc diacrylate	Radiation	4.0	1,500
CX37-1	65 percent polyester— 35 percent styrene	Accelerator	—	3,290
CX37-2	65 percent polyester— 35 percent styrene	(0.5 percent cobalt naphthanate catalyst)	—	3,160
CX37-3	65 percent polyester— 35 percent styrene	(10 percent MEKP)	—	3,300
ACN-1	Acrylonitrile	Radiation	3.0	2,920
ACN-2	Acrylonitrile	Radiation	3.0	2,690
ACN-3	Acrylonitrile	Radiation	3.0	3,630
TS-1	40 percent TMPTMA— 60 percent styrene	Accelerator	—	—
TS-2	40 percent TMPTMA— 60 percent styrene	(0.5 percent dimethy- laniline catalyst)	—	—
TS-3	40 percent TMPTMA— 60 percent styrene	(1.0 percent benzoyl peroxide)	—	—
VA-1	Vinyl acetate	Radiation	3.0	—
VA-2	Vinyl acetate	Radiation	—	—
VA-3	Vinyl acetate	Radiation	—	—

combining the premix with the monomer, the mixture was cast into 1.5-inch-diameter by 3-inch-long wax-coated cardboard cylinders. The specimens were immediately placed in water at room temperature for a 7-day cure.

The monomers were selected to represent a cross section of monomer properties. A description of these properties and of the results obtained is given below.

Acrylamide.—Acrylamide is a water-soluble monomer that polymerizes under normal conditions to form a polymer that is also water-soluble. In order to obtain a uniform monomer distribution, the acrylamide was dissolved in a measured amount of water which was added to the sand and cement to form the premix.

The cured specimens were placed in a ^{60}Co gamma radiation cell to polymerize the monomer. When removed from the molds for inspection, after irradiation, the specimens fell apart and strong ammonia vapors were liberated. A marked acrylic acid odor was also detected. These results indicate that the acrylamide decomposed or hydrolyzed in the presence of the highly alkaline premix media.

Zinc Diacrylate.—Zinc diacrylate, another water-soluble monomer, was added to the premix in the same manner as acrylamide. This monomer, however, is more resistant to hydrolysis and did not crumble after removal from the molds. The compressive strength obtained with these specimens was 1,700 psi, which is approximately one-half the value obtained for the control specimens (Table 4).

Polyester-styrene.—A 65 percent polyester plus 35 percent styrene mixture was added to the premix. This monomer mixture is viscous (700 centipoises) and can be uniformly distributed throughout the wet mix. It contained an accelerator (0.5 percent cobalt naphthenate) and a chemical catalyst (1 percent methylethyl ketone peroxide). After curing, the specimens were firm and gave essentially the same compressive strengths as the controls (Table 4).

Acrylonitrile.—In previous work,¹ acrylonitrile monomer gave compressive strength values in excess of those obtained for the controls. The strength results obtained in this series were approximately the same or lower than those obtained for the controls.

60 percent styrene plus 40 percent TMPTMA.—This mixture has been used for preparing high-temperature test specimens with precast concrete. It also has a density approximating that of water, which enables it

to be more easily dispersed in the wet mix. The mixture contained an accelerator (0.5 percent dimethyl aniline) and a chemical catalyst (1 percent benzoyl peroxide). After curing, all specimens crumbled during removal from the molds. Tests indicated that the TMPTMA plus styrene mixture had a pH of 3. The high acidity probably had a strong effect on the hydration reaction, which requires a high degree of alkalinity. The neutralizing action affected both the cement and the monomer cure.

Vinyl Acetate.—In the presence of the high alkaline premix media, vinyl acetate hydrolyzed to form acetic acid and alcohol. The specimens were yellowish and crumbled under slight pressure.

From the results obtained several conclusions and observations can be made regarding the feasibility of incorporating monomers into fresh concrete premix.

(1) Most monomers are not soluble in water. This results in a nonuniform specimen. More uniform mixtures are obtained with viscous monomers such as polyester-styrene.

(2) The cement should be saturated with water prior to monomer addition in order for satisfactory hydration to take place. Monomer soaked cement cannot readily hydrate in the presence of water. The addition of monomer, without a reduction in the normal water content of the premix results in a mixture having poor workability. However, all of the water cannot be replaced by monomer if satisfactory cement hydration is to occur.

(3) Most organic monomers react to varying degrees in the presence of a strong alkali medium to form acids and other products. The acids in turn interfere with the cement hydration reaction, and the result is weak concrete.

(4) The results obtained in this work and in the previous work indicate that the addition of monomers to wet premix concretes does not appreciably improve the strength properties. However, reductions in the water absorption properties of concrete were obtained by the addition of monomer to premix concrete.

3.3.2 USBR Studies

Initial tests were performed in attempts to mix and polymerize monomers in fresh portland cement paste. Several monomer systems were tried including styrene, vinyl acetate, MMA, and acrylonitrile. Several types of

mixing were used in an effort to produce a homogenous mix. Polymerization was chemically initiated with benzoyl peroxide at 167° F (75° C) after varying periods of cement hydration. Some specimens were also polymerized under water.

The results of these tests were generally disappointing, the best specimens developed only about one-half the compressive strength of the control specimens. These low strengths are thought to be the result of one or a combination of three causes: (1) monomer interference with cement hydration; (2) reduction of polymer bond by the water in fresh cement paste; and (3) high monomer loss through evaporation during the polymerization process. Differential thermal analysis of selected specimens subsequently confirmed that very poor cement hydration occurred in the specimens containing monomer.

A series of experiments was performed in which epoxy resin was mixed with fresh cement mortar. The control cement mortar had a 1:2.75 cement-to-sand ratio and a 0.51 water-cement ratio; 14 percent metaphenylenediamine by weight of the resin was used as a catalyst or curing agent for the epoxy. The catalyzed epoxy resin was added to the fresh mortar mix as a partial replacement for portland cement with the water-cement ratio held constant at 0.51. After addition of the epoxy resin to the water, portland cement, and sand, mixing was continued until a uniform PCC mortar was obtained. The epoxy had a relatively high viscosity and entered the mortar mix uniformly. This uniformity was retained in the cured specimen. Two sets of 2-inch-diameter by 4-inch-long test specimens were prepared. In one, 50 weight percent of the cement was replaced with epoxy, in the other 25 weight percent of the cement was replaced with epoxy. These specimens were cured in sealed containers at laboratory air temperature for 6 days followed by a 7-hour oven cure at 212° F (100° C). The 7-day average compressive strengths developed are: control cement mortar, 3,700 psi; 50 weight percent replacement, 6,100 psi; 25 weight percent replacement, 3,800 psi. Additional tests with epoxies are underway to determine optimum performance and to identify additional beneficial systems.

3.4 POLYMER-CONCRETE STUDIES

Composite materials consisting of various aggregates bound by polymers are being studied. These materials are formed by curing aggregate and monomer premixes of varying proportions and aggregate size. The aggregate can be similar to that used in the

conventional portland cement concrete, but, other types of aggregate or filler can also be used. PC composites do not contain a hydrated cement phase although portland cement can be used as an aggregate or filler.

Prior to curing, the monomer is allowed to wet all aggregate surfaces in order to obtain a stronger bond at the aggregate-polymer interface. The mixtures usually are in the form of slurries that can be easily cast or molded in a manner similar to fresh portland cement concrete. Mixtures of monomer and aggregate can be polymerized by radiation, by chemical catalyst and temperature, and by the catalyst-accelerator methods described in a previous report.²

3.4.1 BNL Studies

Initial experiments were conducted by substituting monomer for portland cement in a typical CP-type concrete mix. The results indicated compressive strengths several times that of the conventional CP-type concrete; however, the monomer loadings required were about 25 percent by weight of the total materials. This was primarily due to the large void volume in the aggregate mix. In addition, the high monomer loadings produced large exotherms during polymerization which resulted in polymer foam and separation at the polymer aggregate interface.

The effect of sand size on the compressive strength of PC composites was studied (Table 5). Specimens

Table 5

COMPRESSIVE STRENGTH VERSUS SAND MESH SIZE Polymer-Concrete

Sand characteristics		Compressive strength, psi (average three samples)
Mesh size	Diameter-inch	
20-30	.0331-.0234	10,760
30-50	.0234-.0117	12,440
40-60	.0165-.0098	12,990
60-80	.0098-.0070	11,600
80-100	.0070-.0059	12,500
100-140	.0059-.0041	10,880
140-200	.0041-.0029	11,650
170-200	.0035-.0029	10,380

Monomer, polyester-styrene
Monomer loading 20 percent by weight
Radiation-induced polymerization

consisting of sand and a polyester-styrene mixture were prepared and polymerized by radiation. Each of the sand sizes listed in Table 5 were tested. The compressive strengths obtained, based on the average values for three specimens, were approximately the same. Since the polymer loading in each case was 20 percent by weight, a complete filling of the voids in each specimen was indicated.

In a subsequent experiment, Ottawa sand of 20 to 30 mesh size was placed in a die and crushed in a press at 50,000 psi in an attempt to minimize the void volume by crushing the sand. The size distribution obtained is shown in Table 6. Portions of the crushed sand were added to a polyester-styrene mixture and polymerized in sealed containers by radiation. The results showed a polymer loading of 15 percent and a compressive strength of 16,200 psi. The distribution obtained by crushing the sand reduced the monomer requirements by 25 percent and resulted in a compressive strength increase of 25 percent compared with the specimens containing 20 to 30 mesh size sand.

Compressive strength results obtained with monomers plus aggregates other than sand alone are given in Table 7. A portland cement plus 20 weight percent polyester-styrene mixture gave compressive strengths of 21,000 psi. This is twice the strength value obtained with sand containing the same amount of polymer (Table 5). When 30 weight percent of sand was added to portland cement, the monomer requirement decreased by half and the average compressive strength increased to 23,580 psi. This indicates that aggregate size distribution plays an important role with respect to both the monomer requirements and the strength values obtained. When another monomer system, 60 percent styrene plus 40 percent TMPTMA, was added

Table 6
SAND CHARACTERISTICS

Polymer-concrete		
Sand characteristics		Mass fraction percent
Mesh size	Diameter-inch	
140 and finer	.0041 maximum	27.6
100-140	.0059-.0041	5.8
80-100	.0070-.0059	4.3
60- 80	.0098-.0070	7.4
50- 60	.0017-.0098	2.6
40- 50	.0165-.0117	10.8
30- 40	.0234-.0165	22.3
20- 30	.0331-.0234	19.1

Initial sand size 20-30 mesh
Monomer, polyester-styrene
Average polymer content, $\approx$ 15 weight percent
Radiation-induced polymerization
Compressive strength, 16,200 psi

to portland cement (Table 7), the average strength values obtained were lower, although the monomer requirements were the same as for polyester-styrene. The differences may be due to the physical properties of the polymers.

Talc powder, when used as aggregate, absorbs a great amount of monomer (about 50 percent by weight) but has low compressive strength as compared to cement and sand aggregates (Table 7). Further work is being done to develop structurally strong and durable PC containing a minimum of polymer.

Table 7
COMPRESSIVE STRENGTH OF POLYMER-CONCRETE

Specimen size, 1-3/4-inch diameter by 3 inches long
Polymerization, catalyst-accelerator

Monomer	Aggregates	Average compressive strength, psi
Polyester-styrene	Type II cement plus 20 percent by weight monomer	21,000
	Type II cement, 70 percent; 30-mesh sand, 30-percent; monomer, 10 percent by weight	23,580
60 percent styrene- 40 percent TMPTMA	Type II cement plus 20 percent by weight monomer	17,100
75 percent polyester-styrene 25 percent styrene	TALC plus 50 percent, by weight, monomer	6,400

3.4.2 USBR Studies

The preparation of PC was initiated during this report period. To date, survey-type studies have been made using several monomer systems polymerized by thermal-catalytic methods. The most common aggregate used in these tests was graded Ottawa sand (ASTM Designation: C 109). Unless otherwise noted, the use of this material is assumed in the following discussion.

Two techniques are used to mix monomer with aggregate. The first is the conventional method of adding monomer to dry aggregate and stirring until a uniform blend is achieved. This method has been successful in some cases, but usually results in formation of excessive air bubbles and increased porosity. The second technique consists of placing monomer in the specimen mold and then gradually adding aggregate to the monomer. The mixture is consolidated by mechanical vibration. This method generally produces the most uniform product with minimum air entrapment and porosity.

Polymerization was accomplished in these studies by chemical initiation with 1 percent benzoyl peroxide at 140° F (60° C). All polymerizations were conducted in sealed containers to prevent monomer loss through evaporation. Additional tests were made at temperatures up to 212° F (100° C), but the resulting increased rate of polymerization resulted in reduced strengths.

PC specimens were made with MMA, MMA plus TMPTMA, and MMA-acrylonitrile. Polymerization time-temperature curves were obtained for these materials, and the specimens were tested for compressive strength.

MMA

MMA was added to Ottawa sand and to Clear Creek aggregate. With Ottawa sand, 15.7 weight percent MMA produced an average compressive strength in 2-inch-diameter by 4-inch-long cylinders of 14,000 psi. When graded Clear Creek aggregate was used, the monomer content could be reduced to 10 weight percent. The average compressive strength of 2-by-4-inch cylinders prepared in this manner was 13,600 psi. The specimens prepared with MMA showed substantial polymer shrinkage during polymerization. Generally the top fourth of the specimen was not completely filled with polymer. This monomer system also produced specimens showing bubbly or "popcorn" polymerization, a condition that increased as the polymerization initiation temperature was increased. Specimens prepared with 3/8-inch Clear Creek

aggregate displayed the most serious instance of polymer bubbling, while in the specimens containing Ottawa sand and well-graded Clear Creek aggregate (which both have higher heat capacities and less void space to be filled by monomer than the 3/8-inch aggregate), this type of polymerization occurred less often.

MMA plus TMPTMA

The cross-linking monomer TMPTMA has been added to several monomer systems to increase the high-temperature performance of PIC. Because of the promising results, a PC test series was performed using varying amounts of TMPTMA with MMA. The compressive strengths of specimens prepared with this system are shown in Figure 3. These very uniform

Figure 3. Compressive strength of MMA-TMPTMA monomer system in polymer-concrete containing 15 percent polymer.

specimens displayed little polymer shrinkage and no popcorn polymerization was evident. The rate of polymerization, however, was faster than that for PC specimens prepared with MMA and increased with increasing amounts of TMPTMA. The increase in specimen temperature due to the exothermic polymerization reaction was also higher for the MMA-TMPTMA specimens than for those containing only MMA. These conditions can be seen graphically in Figures 4 and 5, in which polymerization

Figure 4. Polymerization time-temperature curve for 100 percent MMA polymer-concrete containing 15.6 percent polymer.

time-temperature curves are plotted for PC specimens prepared with MMA and with 90 percent MMA plus 10 percent TMPTMA, respectively. The increased polymerization rates are thought to be at least partially responsible for the reduced compressive strengths of PC specimens containing TMPTMA.

MMA plus Acrylonitrile

Polyacrylonitrile has also been shown to improve the high-temperature performance of impregnated concrete. The polymer, however, is not soluble in its monomer and polymerizes as a white powder. When used to prepare PC specimens, no cohesive strength was developed and the specimens crumbled when removed from their molds.

Since polyacrylonitrile is soluble in MMA, it was thought that mixtures of MMA and acrylonitrile might copolymerize even though their respective reactivity ratios are widely different. Figure 6 shows the compressive strengths developed by PC specimens

Figure 5. Polymerization time-temperature curve for 90 percent MMA plus 10 percent TMPTMA polymer-concrete containing 15.6 percent polymer.

Figure 6. Compressive strength of MMA-acrylonitrile monomer system in polymer-concrete containing 15 percent polymer.

containing 15 weight percent of varying MMA plus acrylonitrile mixtures. The highest compressive strength, 16,900 psi, was achieved with a 60 percent MMA plus 40 percent acrylonitrile mixture.

These PC specimens showed polymer shrinkage and bubbling similar to that of the MMA specimens. As

before, the bubbling was most pronounced in specimens prepared with only coarse aggregate. With well-graded Clear Creek aggregate and Ottawa sand, the polymerization rate was relatively slow and the exothermic temperature rise during polymerization was significantly less than with any other monomer system. Since these results were unexpected, additional tests with this system will be performed. Figure 7 shows the polymerization time-temperature curve for a PC specimen prepared with the 60 percent MMA plus 40 percent acrylonitrile mixture. By comparing this curve with the polymerization time-temperature curve for

MMA (Figure 4), the polymerization rate differences of the two systems becomes obvious.

Research on PCC and PC is continuing. Additional monomer, epoxy, and polyester systems will be screened.

The use of promoters will be studied to eliminate the need of heat and radiation for polymerization. Studies of aggregate processing techniques and selection will be continued in an effort to improve the polymer-aggregate bond and reduce polymer requirements.

Figure 7. Polymerization time-temperature curve for 60 percent MMA plus 40 percent acrylonitrile polymer-concrete containing 16 percent polymer.

4. CONCRETE-POLYMER PREPARATION TECHNIQUES

4.1 FULL IMPREGNATION

4.1.1 Summary of Earlier Work

A number of procedures must be performed before a concrete specimen is impregnated with monomer. Certain conditions are required for full or maximum monomer loading. A generalized procedure for obtaining full impregnation is:

- (1) Dry the specimen.
- (2) Evacuate the dried sample.
- (3) Soak in monomer.
- (4) Drain monomer.
- (5) Wrap the specimen in polyethylene sheet or aluminum foil to reduce the evaporation loss of monomer from the sample.
- (6) Polymerize to completion by radiation or thermal-catalytic means.

Each parameter has been studied as a function of the polymer loading and the results are presented in the First and Second Topical Reports.^{1 2}

Experiments are being conducted to determine the effects of several other concrete-polymer preparation techniques on the final strength of the composite. Variables under study include drying temperature, method and duration of concrete curing, age of concrete at impregnation, and the effect of aging on strength. Initial work on these parameters is described in subsequent sections of this report.

4.1.2 Drying Temperature

A program was initiated to study the effect of drying temperature on the strength of PIC. Earlier work² had indicated that drying temperatures up to 392° F (200° C) did not have detrimental effects on the compressive strength.

A minimum of three MMA-impregnated specimens and three controls were dried at temperatures of 302° F (150° C), 347° F (175° C), 392° F (200° C), 437° F (225° C), and 482° F (250° C). The results for this series (Table 8) indicate a small, gradual reduction in the strength of the unimpregnated samples with increased drying temperature. Compared with an average strength of 5,660 psi for the undried controls,

samples dried at 482° F exhibited a reduction in strength of about 13 percent.

The results for the MMA-impregnated specimens exhibited greater scatter than those for the controls. Between 302° F and 392° F, the average strengths decreased with increased drying temperature; above 392° F, strength increased with drying temperature. The average strength for samples dried at 482° F was 18,350 psi, about equal to that for samples dried at 302° F (18,200 psi). The average strength for all impregnated specimens was 17,060 psi, with a coefficient of variation of 9.3 percent. Corresponding results for the dried controls were 5,230 psi and 5.6 percent. These results appear to be within the coefficient of variation normally obtained for impregnated and unimpregnated specimens. Therefore the apparent changes in strength with temperature may not be significant. A greater number of specimens will be needed to verify these observations.

4.1.3 Cure Time

Experiments were performed to evaluate the effect of the concrete cure time on the compressive and tensile strengths of PIC. Cure times of 2, 7, and 28 days in saturated lime water were used. The following experimental procedure was used for each cure time:

- (1) Eighteen specimens were fabricated and cured for the desired time in lime-saturated water.
- (2) On completion of curing, 12 specimens were dried immediately at 302° F (150° C) to constant weight, and 6 specimens were retained as undried controls.
- (3) At the completion of drying, six specimens were impregnated with MMA and radiation polymerized, and six specimens were retained as dried controls.

The experimental data for the series (Tables 9 to 11) indicate a reduction in the average polymer loading with increased cure time. Average polymer loadings of 5.2, 4.4, and 4.3 percent were obtained for cure times of 2, 7, and 28 days, respectively. This may be explained by the decrease in porosity that can occur with an increase in the hydration period.

Results of compressive and tensile strength measurements made at an age of 120 days (summarized in Tables 9 to 11) indicate that strengths of unimpregnated specimens increased with cure time. Specimens dried at 302° F immediately after completion of the water cure exhibited strengths about 8 percent lower than those of the specimens that were

Table 8

MMA-IMPREGNATED CONCRETE

Effect of Drying Temperature on Compressive Strength
and Modulus of Elasticity

Specimen	Drying temperature		Polymer loading, percent of unimpreg- nated weight	Compressive strength, psi	Modulus of elasticity 10 ⁶ psi
	°F	°C			
CP-3A-12B ^(a)	—	—	0	5,330	3.34
CP-3A-12C ^(a)	—	—	0	5,690	3.47
CP-3A-13E	—	—	0	5,970	3.53
CP-3B-13B	302	150	5.0	18,780	6.21
CP-3B-13C	302	150	6.1	16,970	6.26
CP-3B-13D	302	150	6.1	17,820	5.97
CP-3B-13A	302	150	5.4	19,260	5.68
CP-3B-13E	302	150	0	5,660	2.62
CP-3B-13F	302	150	0	5,570	2.51
CP-3B-11E	302	150	0	5,260	2.47
CP-3B-11F	347	175	5.3	17,990	5.66
CP-3B-12C	347	175	6.1	15,160	5.84
CP-3B-11B	347	175	6.5	15,470	6.18
CP-3A-14A	347	175	5.7	14,680	6.41
CP-3B-11A	347	175	0	5,050	2.43
CP-3B-14D	347	175	0	5,600	2.42
CP-3B-14C	347	175	0	5,500	2.39
CP-3B-15C	392	200	5.8	15,840	6.50
CP-3B-15D	392	200	6.4	17,430	6.21
CP-3A-12D	392	200	4.9	15,110	5.10
CP-3A-12A	392	200	6.3	14,510	6.47
CP-3B-11C	392	200	0	4,990	2.42
CP-3B-15E	392	200	0	5,590	2.35
CP-3B-12A	392	200	0	5,230	2.31
CP-3B-15B	437	225	5.8	19,290	5.31
CP-3B-12B	437	225	5.3	17,200	4.92
CP-3B-12E	437	225	5.7	16,440	5.72
CP-3B-11D	437	225	0	5,040	2.21
CP-3B-15F	437	225	0	5,230	2.23
CP-3B-12F	437	225	0	4,870	2.05
CP-3B-14E	482	250	6.5	17,960	6.10
CP-3B-14F	482	250	6.1	17,600	5.87
CP-3B-15A	482	250	6.0	19,490	6.44
CP-3B-14B	482	250	0	5,020	2.17
CP-3A-11A	482	250	0	4,640	2.22
CP-3B-14A	482	250	0	5,160	2.09
	Mean compressive strength, psi		Deviation	Coefficient of variation, percent	
Impregnated samples	17,060		±1,590	9.3	
Dried controls	5,230		± 293	5.6	

^(a)Undried control

MMA = methyl methacrylate

Radiation-induced polymerization

Drying time, 96 hours

Specimen size, 3-inch diameter by 6 inches long

Table 9

MMA-IMPREGNATED CONCRETE

Effect of 2 Days' Cure Time on Strength

Specimen	Cure time days	Polymer loading percent of unimpregnated weight	Compressive strength, psi	Modulus of elasticity 10^6 psi	Tensile strength psi	Difference ² percent
1	2	5.1	—	—	940	288
2	2	5.4	17,550	7.98	—	345
3	2	5.0	19,100	8.31	—	384
4	2	5.1	—	—	680	181
5	2	5.3	—	—	1,120	363
6	2	5.2	13,000	7.55	—	230
7	2	0	4,200	3.68	—	—
8	2	0	4,000	4.14	—	—
9	2	0	3,650	3.84	—	—
10	2	0	—	—	245	—
11	2	0	—	—	260	—
12	2	0	—	—	220	—
¹ 13	2	0	4,290	4.46	—	—
14	2	0	4,200	4.43	—	—
15	2	0	4,200	4.59	—	—
16	2	0	—	—	285	—
17	2	0	—	—	260	—
18	2	0	—	—	270	—

¹Samples 13-18 undried controls. Stored under laboratory air conditions after removal from curing water until time of test.

²Difference compared with dried controls cured for an equal time; difference, percent = [(test - av control)/av control] 100.

Specimen size, 3-inch diameter by 6 inches long

MMA methyl methacrylate

Radiation—induced polymerization

Specimens cured in lime-saturated water for 2 days

All specimens, except undried controls dried at 302° F (150° C) to constant weight immediately after curing

Age at test, 120 days

Table 10
MMA-IMPREGNATED CONCRETE
Effect of 7 Days' Cure Time on Strength

Specimen	Cure time, days	Polymer loading, percent of unimpregnated weight	Compressive strength, psi	Modulus of elasticity 10 ⁶ psi	Tensile strength, psi	Difference ² percent
1A	7	4.4	18,000	8.42	—	306
2A	7	4.4	—	—	1,320	304
3A	7	4.5	—	—	595	82
4A	7	4.3	12,750	7.80	—	188
5A	7	4.2	—	—	1,160	254
6A	7	4.4	17,950	8.22	—	306
7A	7	0	4,050	4.51	—	—
8A	7	0	4,700	4.58	—	—
9A	7	0	4,525	4.67	—	—
10A	7	0	—	—	365	—
11A	7	0	—	—	290	—
12A	7	0	—	—	—	—
¹ 13A	7	0	4,625	4.72	—	—
14A	7	0	4,575	5.37	—	—
15A	7	0	4,625	4.73	—	—
16A	7	0	—	—	320	—
17A	7	0	—	—	385	—
18A	7	0	—	—	320	—

¹ Samples 13A-18A undried controls. Stored under laboratory air conditions after removal from curing water until time of test.

² Difference compared with dried controls cured for an equal time; difference, percent, = [(test — av control)/av control] 100.

Radiation-induced polymerization

MMA = methyl methacrylate

Specimen size, 3-inch diameter by 6 inches long

Specimens cured in lime-saturated water for 7 days

All specimens except undried controls dried at 302° F (150° C) to constant weight immediately after curing.

Age at test, 120 days

Table 11

MMA-IMPREGNATED CONCRETE

Effect of 28 Days' Cure Time on Strength

Specimen	Cure time days	Polymer loading, percent of unimpreg- nated weight	Compressive strength, psi	Modulus of elasticity 10 ⁶ psi	Tensile strength, psi	Difference ² percent
1B	28	4.1	15,500	7.18	—	172
2B	28	4.3	18,750	7.76	—	229
3B	28	4.7	16,500	7.76	—	190
4B	28	4.2	—	—	955	158
5B	28	4.4	—	—	940	154
6B	28	4.4	—	—	1,250	237
7B	28	0	5,500	4.36	—	—
8B	28	0	6,100	4.14	—	—
9B	28	0	5,500	4.99	—	—
10B	28	0	—	—	380	—
11B	28	0	—	—	362	—
12B	28	0	—	—	371	—
¹ 13B	28	0	6,125	5.27	—	—
14B	28	0	6,450	5.15	—	—
15B	28	0	5,980	5.06	—	—
16B	28	0	—	—	283	—
17B	28	0	—	—	433	—
18B	28	0	—	—	358	—

¹ Samples 13B-18B undried controls. Stored under laboratory air conditions after removal from curing water until time of test.

² Difference compared with dried controls cured for an equal time; difference, percent = $[(\text{test} - \text{av control})/\text{av control}] 100$.

Radiation-induced polymerization

MMA, methyl methacrylate

Specimen size, 3-inch diameter by 6 inches long

Specimens cured in lime-saturated water for 28 days

All specimens except undried controls dried at 302° F (150° C) to constant weight immediately after curing.

Age at test, 120 days

stored under laboratory air conditions until the test date. The samples dried and impregnated immediately after curing in water for 2 days exhibited essentially the same strengths as those of impregnated specimens that were cured for 7 and 28 days. Average compressive strengths of 16,500, 16,200, and 16,900 psi were obtained for 2-, 7-, and 28-day cured specimens, respectively. These correspond to increases of 290, 253, and 174 percent compared with those for undried controls cured for the same time. The average compressive strength for all the impregnated specimens was 16,570 psi, with a coefficient of variation of 13.4 percent. Results from the tensile strength measurements show similar trends, although the data exhibit a greater amount of scatter, with an average of 996 psi and a coefficient of variation of 23 percent.

The results from this study are in agreement with previous observations that the initial strength of the concrete has little effect on the final strength of the PIC. However, the polymer loadings of low-strength concretes are greater than those of high-strength materials.

4.1.4 Impregnation Age

Experiments similar to those described in Section 4.1.3 were conducted to determine the effect of the concrete age at impregnation on the strength properties of the composite.

Twenty-four 3-inch-diameter by 6-inch-long specimens were fabricated and cured in water for 28 days. Eighteen specimens were dried, impregnated with MMA, and radiation polymerized at ages of 1, 28, and 63 days after the water cure was completed. Polymer loadings averaged 4.2 percent in these specimens. The remaining specimens were used as controls. All samples were tested at a total age of 120 days and the results (Table 12) indicate little change in compressive strength (<4 percent) with age. Compared with a control strength of 5,045 psi, average compressive strengths of 13,527 and 14,000 psi were obtained for materials aged 29 and 91 days, respectively. The average compressive strength for all the specimens was 12,740 psi, with a 3.6 percent coefficient of variation.

Similar results were obtained for tensile strength measurements (Table 12). Tensile strengths of 1,120 and 1,038 psi were obtained for materials aged 29 and 91 days, respectively. The average tensile strength was 1,060 psi, with a 10.3 percent coefficient of variation.

Compared with previous work on CP-type concrete, the compressive and tensile strengths obtained in this series were considerably lower than expected. This is probably due to the relatively low-polymer loadings,

which may have resulted from faulty impregnation or excessive evaporation losses during polymerization. However, on the basis of the above results, impregnation age does not appear to greatly affect the final strength of PIC.

4.2 PARTIAL IMPREGNATION AND COATING TECHNIQUES

Earlier experiments² have shown that various depths of penetration of monomer in concrete can easily be obtained. This technique can be of great economic value when, for example, impermeability to water and resistance to chemical attack are of more importance than maximum strength properties. The depth of penetration required for a specific application can best be determined by testing specimens impregnated with monomer to various depths.

There are several basic parameters in this technique. To obtain maximum protection with a minimum of monomer, the viscosity and the vapor pressure of the selected monomer is of prime importance. Low-viscosity monomers such as styrene and MMA have very high-initial rates of diffusion through concrete. As a result, penetrations up to one-half inch or less are extremely difficult to control. The combined action of low-surface tension, capillary action, and sensitivity to pressure equalization results in monomer permeation beyond the desired depth after the specimen has been removed from the soak bath. This effect is due to drainage or siphoning action of monomer in the initially saturated voids in the concrete. As a result, the monomer loadings obtained on a per unit volume basis are far lower than the loadings obtained at full penetration with the same monomer and concrete. The monomer depleted or partially filled voids increase the possibility of attack or permeation, even beyond the polymer barrier, by chemicals, water, etc. Viscous monomer systems such as polyester-styrenes, epoxy-styrenes and monomer-polymer mixtures have been successfully used to obtain controlled depths of penetration. Capillary action is greatly reduced with viscous monomers, and cross-sectional examinations have shown that drainage does not occur and therefore the voids remain filled with monomer.

In almost all cases low-viscosity monomers have higher vapor pressures than higher viscosity monomers, and are therefore more volatile. Unless special precautions are taken, the more volatile monomers tend to vaporize from the outer surface of the concrete specimen during the time required to cure the monomer. The result is at best a noncontinuous coating on the surface which is very vulnerable to attack.

Table 12
MMA-IMPREGNATED CONCRETE

Impregnation Age Test Series

Specimen	Total age at impregnation days	Polymer loading percent of unimpregnated weight	Compressive strength, psi	Difference percent ²	Modulus of elasticity 10 ⁶ psi	Difference percent ²	Tensile strength psi	Difference percent ²
1C	29	4.0	—	—	—	—	935	164
2C	29	4.0	—	—	—	—	1,305	260
3C	29	3.9	—	—	—	—	1,120	216
4C	29	3.7	13,800	174	5.74	26	—	—
5C	29	4.2	13,895	176	6.95	63	—	—
6C	29	4.0	12,895	156	6.65	46	—	—
7C	56	4.2	—	—	—	—	965	172
8C	56	4.1	13,770	173	6.78	40	—	—
9C	56	4.1	13,350	165	6.07	33	—	—
10C	56	4.4	—	—	—	—	970	174
11C	56	4.4	13,600	174	7.02	54	—	—
12C	56	4.1	—	—	—	—	1,130	219
13C	91	4.3	—	—	—	—	990	179
14C	91	4.4	14,675	195	6.85	51	—	—
15C	91	4.3	—	—	—	—	1,095	209
16C	91	4.2	—	—	—	—	1,030	190
17C	91	4.2	14,255	183	7.50	67	—	—
18C	91	4.2	12,980	158	7.35	61	—	—
19C	1	0	—	—	—	—	370	—
20C	1	0	—	—	—	—	320	—
21C	1	0	—	—	—	—	375	—
22C	1	0	5,150	—	4.81	—	—	—
23C	1	0	5,020	—	4.38	—	—	—
24C	1	0	4,965	—	4.46	—	—	—

¹ Undried controls tested at 120 days' age.

² Difference, percent = [(test - average control)/average control] 100

Radiation-induced polymerization

MMA = methyl methacrylate

Specimen size, 3-inch diameter by 6 inches long

Specimens oven dried at 302° F (150° C) to constant weight

Specimens cured in lime-saturated water for 28 days

Age at test, 120 days

When viscous monomer systems are used for partial impregnation, clear demarcation lines can be seen at the penetration depth (Figure 8). The irregularity observed in the depths of impregnation is due to the nonhomogeneous nature of concrete specimens. These specimens were prepared by total immersion in a polyester-styrene bath. Irregularities in penetration depths are more pronounced when one or more surfaces are specially treated during fabrication of the specimen.

A considerable effort was made to obtain partial penetration of monomer in concrete drain tile of the type described in Section 9.1.1. Since the same depth was desired from the inner and outer surfaces of the pipe, the specimens were totally immersed in monomer for varying times. The rate of monomer diffusion from the outer surface of the pipe toward the center was much greater than from the inside surface. A dye was used in the monomer to make visual observations (Figure 9).

Examination of sections impregnated with monomer-dye mixtures indicated little or no diffusion of monomer from the inside of the pipe toward the center. Attempts to obtain an appreciable depth of penetration from the inside always resulted in complete penetration by the diffusion of monomer from the outside surface.

Conditions such as viscosity, pressure, and soaking time were varied without effectively increasing the inside penetration rate in relation to the rate of diffusion from the outside. Figure 9 shows depths of penetrations as a function of time.

The problem of penetrating the pipe from the inside was encountered with three different qualities of pipe (described in Section 9.1.1).

A microscopic examination of the cross sections of the specimens showed the inside surfaces of the pipes to be very smooth, with low porosity and a high cement to aggregate ratio. The outer surface, however, contained many large voids and a much lower cement to

aggregate ratio. Different fabrication methods must be adopted if the inner and outer surfaces of concrete pipe are to be uniformly penetrated to a desired depth.

Two test series, each consisting of eighteen 3-inch-diameter by 6-inch-long specimens, have been impregnated to varying depths and tested for resistance to chemical attack and water permeation. The monomer systems selected were polyester-styrene and an epoxy-styrene mixture. Both systems are viscous, with fairly low vapor pressures. Table 18 lists the various mixtures and viscosities used to obtain depths of penetrations ranging from about 1/32 to 1/4 inch. The results of these tests are discussed in Section 5.

In general, the depth of penetration obtainable under a given set of conditions such as monomer viscosity, soaking time, and pressure is dependent on the type of concrete, fabrication methods, and surface treatments. With experimentation, a set of conditions required for various desired depths of penetration can be readily obtained if the same concrete is used throughout. A detailed procedure describing the various experimental parameters and the equipment used for performing depth of penetration studies is given in a separate report.¹

Another effective method for sealing concrete surfaces is the "paint-on" technique. Polyester-styrene mixtures are diluted with styrene monomer to reduce the viscosity. The mixture is applied to the surface of concrete with a paint brush or roller. The depth of penetration obtained by this method depends on the viscosity of the mixture and the number of "coats" applied. Figure 10 shows a cross section of a conventional building block treated with six brushed-on coats of a polyester-styrene mixture. A depth of penetration of about three-fourth inch was obtained, as indicated by the dye mark. The resin can be cured by introducing a catalyst-accelerator system to the resin prior to application, or by the thermal-catalytic or radiation method.² Specimens treated as above showed water absorption of 0.3 percent by weight when subjected to the boiling water test.¹

Figure 8. Cross sections of partially impregnated concrete specimens.

Figure 9. Cross sections of drain tile specimens impregnated by total immersion in monomer for varying periods of time. Soaking time increases from left to right. Note lack of penetration from inner surface. The monomer was unable to diffuse through inner surface of tile because surface treatment during manufacture.

Figure 10. Cross section of a conventional building block impregnated with a polyester-styrene mixture containing a chemical catalyst and an accelerator, showing the depth of penetration obtained when the monomer was applied with a paint brush.

5. TEST RESULTS ON POLYMER-IMPREGNATED CONCRETE

The testing program for determining the structural and durability properties of PIC consists of a series of tests on two basic types of concrete, one designed for normal temperature applications and the other for high-temperature applications. The concrete is prepared in the USBR laboratories by conventional mixing and casting methods, fog cured for 28 days, and then shipped to BNL for impregnation and polymerization. The hardened concrete is impregnated with a monomer and polymerized either by gamma radiation from a ^{60}Co source or by thermal-catalytic means. Details of concrete fabrication are given in the First Topical Report.¹ Tests are usually made on concrete at 120 days' age. In monomer survey tests, monomer impregnation is made on both undried concrete (exposed to the air during the period following removal from the 28 day fog cure) and concrete oven-dried at $>221^{\circ}\text{F}$ (105°C). Experience with the monomer systems tested has shown oven-dried concrete is more fully impregnated by monomer and has greater improvement in properties than the undried concrete. Unless designated by "U" for undried, test results on PIC are for concrete oven-dried at $>221^{\circ}\text{F}$ prior to impregnation.

The test results are obtained from relatively few specimens and are to be considered indicative of the properties of PIC; they are not intended to be used as a basis for final design of engineering structures. Most of the values are averages of two or three specimens, and in the concrete test series for OSW-type concrete, averages are given for up to 10 specimens. Work on testing sufficient numbers of specimens to obtain statistically valid data for design purposes is scheduled.

Concrete for normal temperature applications, designated as CP-type concrete, is a standard mix concrete made with 5.5 sacks of a Type II laboratory blend cement, Clear Creek aggregate graded to 3/4-inch maximum size, 0.51 water/cement ratio, 3-inch slump, and 6-percent entrained air. The CP test series concrete is fully impregnated with monomer. In addition, tests are also made on partially impregnated CP-type concrete, designated as CP-DP to develop a technique for impregnating the surfaces of concrete to controlled depths in order to achieve a savings in the amount of monomer while still imparting the beneficial durability qualities to the concrete. Monomers for partial impregnation are formulated to specific viscosities to give a controlled depth of penetration.

Concrete for high temperature applications, designated as OSW-type concrete, was initially designed as a special high-strength mix for use in desalting plants. The high-strength concrete is made with 6 sacks/cubic yard of Type V cement, 1.8 sacks/cubic yard of fly ash pozzolan, 0.35 water/cement plus pozzolan ratio, a water-reducing agent, air entrainment, and an aggregate from California graded to 1-1/2-inch maximum size. More recent tests have shown that CP-type concrete, impregnated with the same monomers for high temperature applications as those used with OSW-type concrete has strength properties as good as OSW-type PIC and is easier to fully impregnate. CP-type concrete is now being used in all tests.

5.1 POLYMER-IMPREGNATED CONCRETE FOR NORMAL TEMPERATURE APPLICATIONS

5.1.1 CP Test Series

The testing program consists of a series of structural and durability tests on CP-type concrete impregnated with various monomers. In addition, special tests have been made to determine the effects of time on the compressive strength of MMA-impregnated concrete.

Each test series consists of 38 specimens. Descriptions of specimen preparation and test procedures are given in the First Topical Report.¹

The monomer systems in the concrete-polymer test series are designated as follows:

- CP-0 Control (unimpregnated concrete)
- CP-1A MMA, radiation polymerized
- CP-1B MMA, thermal-catalytically polymerized
- CP-2A Styrene, radiation polymerized
- CP-2B Styrene, thermal-catalytically polymerized
- CP-3A Unimpregnated, radiation treated
- CP-3B Unimpregnated, thermally treated
- CP-4A MMA plus 10 percent TMPTMA, radiation polymerized
- CP-4B MMA plus 10 percent TMPTMA, thermal-catalytically polymerized
- CP-5A Acrylonitrile, radiation polymerized
- CP-5B Acrylonitrile, thermal-catalytically polymerized
- CP-6A Chlorostyrene, radiation polymerized
- CP-6B Chlorostyrene, thermal-catalytically polymerized

5.1.1.1. Structural Properties

A summary of results to date, including data reported in the First and Second Topical Reports, is given in Table 13. This table gives average values of from one to three specimens tested for each property. Test results on chlorostyrene-impregnated concrete, CP-6 series, obtained since the Second Topical Report are given in Tables 14 and 15. Creep tests are continuing on CP series concrete under 800 psi sustained compressive load; the results are shown in Figure 11. A new series of creep tests has been started on MMA- and chlorostyrene-impregnated concrete under high stress in tension and compression; these test results are shown in Figure 12. Special tests to determine the effect of time on strength of MMA-impregnated concrete are given in Table 16. Results on the test series CP-1 to CP-5 were discussed in the First and Second Topical Reports. Tests on the CP-6 series, chlorostyrene-impregnated concrete, are now complete. Generally speaking, chlorostyrene-impregnated concrete performed favorably and produced results intermediate between the highest values, generally obtained with MMA- or MMA plus 10 percent TMPTMA-impregnated concrete, and the lowest values generally obtained with acrylonitrile- or styrene-impregnated concrete.

5.1.1.1.1. Structural properties of chlorostyrene-impregnated concrete

a. Compressive Strength and Elasticity.—Radiation-polymerized specimens had an average compressive strength of 16,090 psi, an improvement of 205 percent over control concrete of 5,270 psi. The compressive strength of thermal-catalytically polymerized specimens was a little lower at 14,390 psi. The modulus of elasticity was 5.6×10^6 psi for both radiation and thermal catalytically polymerized chlorostyrene impregnated concrete, an improvement of 60 percent over that of control concrete which had a modulus of elasticity of 3.5×10^6 psi.

b. Tensile Strength.—Thermal catalytically polymerized specimens had a tensile strength of 1,200 psi. Comparable strengths were obtained with radiation-polymerized specimens (1,120 psi). These strengths are 190 and 167 percent greater, respectively, than that for control concrete (420 psi).

c. Flexural Strength and Elasticity.—The flexural modulus of rupture for radiation-polymerized specimens was 2,380 psi, an increase of 223 percent over that of control concrete (740 psi). Thermal catalytically polymerized specimens were somewhat lower at 1,580 psi. Flexural modulus of elasticity was 6.3×10^6 psi for the radiation-polymerized concrete and 5.0×10^6 psi for thermal-catalytically polymerized concrete, both of which showed good improvement over the control concrete (4.3×10^6 psi).

d. Cavitation Resistance. The radiation-polymerized specimen produced fairly good cavitation-resistant concrete, with cavitation to a depth of 0.072 inch in a 2 hour exposure an improvement of 77 percent over control concrete. The thermal-catalytically polymerized specimen had 0.115-inch cavitation, which is the highest so far obtained with the CP test series concrete, but is still an improvement of 64 percent over control concrete.

e. Thermal Properties.—Thermal properties of concrete show little change by impregnation. The greatest effect observed was on coefficient of expansion. Thermal-catalytically polymerized specimens showed an increase of 19 percent in coefficient of expansion, and radiation-polymerized concrete showed an increase of 10 percent. Thermal conductivity remained essentially unchanged for both types of polymerization. Diffusivity values were increased by 13 percent for radiation-polymerized, and 3 percent for thermal-catalytically polymerized specimens. Specific heat decreased 17 percent for radiation-polymerized and 6 percent for thermal-catalytically polymerized specimens.

5.1.1.1.2. Creep tests

Creep tests are continuing on PIC under a sustained compressive load of 800 psi, and results are available for periods of 275 to 799 days under load. The creep values in Figure 11 are averages of two specimens, except that the radiation polymerized styrene specimens are plotted individually. Most of the PIC continues to show less creep than unimpregnated concrete. Low positive creep (a normal contraction in length under compression) was obtained with chlorostyrene, acrylonitrile, and MMA plus 10 percent TMPTMA impregnated concrete. Negative creep (an expansion in length under compression), which was

Table 13

SUMMARY OF MECHANICAL AND PHYSICAL PROPERTIES OF POLYMER IMPREGNATED CARBONS
OF Test Series

Property	Unimpregnated (control)			NMP impregnated			NMP impregnated			NMP impregnated			NMP impregnated			NMP impregnated		
	Control	CP-1	CP-2	CP-1	CP-2	CP-3	CP-1	CP-2	CP-3	CP-1	CP-2	CP-3	CP-1	CP-2	CP-3	CP-1	CP-2	CP-3
Compressive strength, psi	1,277	4,112	1,242	2,260	14,16	40	4,197	11,977	11,977	11,977	11,977	11,977	11,977	11,977	11,977	11,977	11,977	11,977
Percent improvement		4	4	240	1400	40	40	40	40	40	40	40	40	40	40	40	40	40
Modulus of elasticity, 10 ⁶ psi	3.3	2.1	2.5	4.3	4.3	4.3	3.2	4	4	4	4	4	4	4	4	4	4	4
Percent improvement		23	17	31	31	31	43	21	21	21	21	21	21	21	21	21	21	21
Tensile strength, psi	424	331	360	1,477	1,370	1,370	2	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370
Percent improvement		19	7	247	212	212	73	247	247	247	247	247	247	247	247	247	247	247
Modulus of rupture, psi	67	132	444	2,447	1,194	1,194	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370	1,370
Percent improvement		11	107	214	212	212	201	201	201	201	201	201	201	201	201	201	201	201
Flexural elongation, modulus, 10 ⁶ psi	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3
Percent improvement		11	11	44	44	44	47	47	47	47	47	47	47	47	47	47	47	47
Mooney (100 psi)	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Percent improvement		1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Hardness, 10 ³ psi	24	21	27	11	12	12	17	14	14	14	14	14	14	14	14	14	14	14
Percent improvement		11	11	73	62	62	57	56	56	56	56	56	56	56	56	56	56	56
Alkaline loss, percent	0.1487	0.0312	0.0312	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167	0.0167
Percent improvement		31	31	42	42	42	42	42	42	42	42	42	42	42	42	42	42	42
Loss in weight, percent	14	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4
Percent improvement		17	17	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71
Contraction, 10 ³ psi	0.123	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144	0.144
Percent improvement		1	1	97	97	97	97	97	97	97	97	97	97	97	97	97	97	97
Water absorption, percent	0.40	4.1	4.2	1.19	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.34
Percent decrease		2	1	63	61	61	61	61	61	61	61	61	61	61	61	61	61	61
Water uptake, 10 ³ psi	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Percent decrease		100	100	63	63	63	63	63	63	63	63	63	63	63	63	63	63	63
Thermal conductivity, 10 ³ psi	1.132	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144	1.144
Percent decrease		21	11	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Dilatometer at 10 ³ psi	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132	1.132
Percent increase		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Coefficient of expansion, 10 ³ psi	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12	4.12
Percent increase		1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Specific heat, 10 ³ psi	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245	0.245
Percent reduction		1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Specific gravity	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317	2.317

Results are averages of two to three specimens tested for each property.
Percentages calculated as 100% of control specimen.

NMP = n-methyl pyrrolidone
CP-1 = 100% n-methyl pyrrolidone
CP-2 = 50% n-methyl pyrrolidone
CP-3 = 25% n-methyl pyrrolidone

1 = 100% n-methyl pyrrolidone
2 = 50% n-methyl pyrrolidone
3 = 25% n-methyl pyrrolidone

Mooney tests - Mooney under constant compressive load of 100 psi

1 - Loaded for 100 days

2 - Loaded for 200 days

3 - Loaded for 300 days

4 - Loaded for 400 days

5 - Loaded for 500 days

6 - Loaded for 600 days

7 - Loaded for 700 days

8 - Loaded for 800 days

9 - Loaded for 900 days

10 - Loaded for 1000 days

11 - Loaded for 1100 days

12 - Loaded for 1200 days

13 - Loaded for 1300 days

14 - Loaded for 1400 days

15 - Loaded for 1500 days

16 - Loaded for 1600 days

17 - Loaded for 1700 days

18 - Loaded for 1800 days

19 - Loaded for 1900 days

20 - Loaded for 2000 days

21 - Loaded for 2100 days

22 - Loaded for 2200 days

23 - Loaded for 2300 days

24 - Loaded for 2400 days

25 - Loaded for 2500 days

26 - Loaded for 2600 days

27 - Loaded for 2700 days

28 - Loaded for 2800 days

29 - Loaded for 2900 days

30 - Loaded for 3000 days

31 - Loaded for 3100 days

32 - Loaded for 3200 days

33 - Loaded for 3300 days

34 - Loaded for 3400 days

35 - Loaded for 3500 days

36 - Loaded for 3600 days

37 - Loaded for 3700 days

38 - Loaded for 3800 days

39 - Loaded for 3900 days

40 - Loaded for 4000 days

41 - Loaded for 4100 days

42 - Loaded for 4200 days

43 - Loaded for 4300 days

44 - Loaded for 4400 days

45 - Loaded for 4500 days

46 - Loaded for 4600 days

47 - Loaded for 4700 days

48 - Loaded for 4800 days

49 - Loaded for 4900 days

50 - Loaded for 5000 days

Table 14

MECHANICAL PROPERTIES OF CHLOROSTYRENE IMPREGNATED CONCRETE

CP Test Series

Compressive Strength and Modulus of Elasticity

Specimen number	Polymerization and preparation	Polymer loading percent	Compressive strength psi	Modulus of elasticity 10^6 psi
Control			5,270	3.50
CP-CA-1A	R, D	6.2	14,950	5.71
CP-CA-1B	R, D	6.6	15,840	5.50
CP-CA-1C	R, D	6.2	17,470	5.53
CP-CA-1D	R, U	3.0	8,660	5.07
CP-CA-1E	R, U	4.3	7,160	5.39
CP-CA-1F	R, U	3.5	9,160	4.72
CP-CB-1A	T, D	6.3	12,030	5.68
CP-CB-1B	T, D	6.0	14,570	5.64
CP-CB-1C	T, D	6.4	15,760	5.54
CP-CB-1D	T, U	3.1	9,650	4.64
CP-CB-1E	T, U	3.4	9,200	4.18
CP-CB-1F	T, U	3.4	9,010	4.68

Tensile Strength

Specimen number	Polymerization and preparation	Polymer loading, percent	Tensile strength, psi
Control			420
CP-CA-2A	R, D	6.2	1,140
CP-CA-2B	R, D	6.2	1,130
CP-CA-2C	R, D	6.5	1,060
CP-CA-2D	R, U	3.9	600
CP-CA-2E	R, U	4.6	710
CP-CA-2F	R, U	4.1	660
CP-CB-2A	T, D	6.5	1,100
CP-CB-2B	T, D	6.5	1,230
CP-CB-2C	T, D	6.4	
CP-CB-2D	T, U	2.9	640
CP-CB-2E	T, U	3.6	660
CP-CB-2F	T, U	3.9	600

Modulus of Rupture and Flexural Modulus of Elasticity

Specimen number	Polymerization and preparation	Polymer loading percent	Modulus of rupture, psi	Flexural modulus of elasticity 10^6 psi
Control			740	4.30
CP-CA-5A	R, D	6.6	2,520	6.91
CP-CA-5B	R, D	6.8	2,320	6.15
CP-CA-5C	R, D	5.9	2,320	6.15
CP-CA-5D	R, U	2.6	1,010	5.73
CP-CA-5E	R, U	2.6	1,110	5.06
CP-CA-5F	R, U	2.6	700	6.14
CP-CB-5A	T, D	5.4	1,360	5.14
CP-CB-5B	T, D	5.4	1,660	4.65
CP-CB-5C	T, D	5.9	1,680	5.10
CP-CB-5D	T, U	2.5	1,120	4.72
CP-CB-5E	T, U	3.0	1,190	4.91
CP-CB-5F	T, U	2.7	1,050	4.79

Test Results on 6 by 12 inch cylinders

R - radiation polymerization

T - thermal catalytic polymerization

D - oven-dried at 302°F (150°C) prior to impregnation

U - undried, specimens stored in air prior to impregnation

Table 15

PHYSICAL PROPERTIES OF CHLOROSTYRENE-IMPREGNATED CONCRETE

CP Test Series

Property	Specimen		Polymer loading, weight percent	Test results	
				Percent absorption	
Water absorption	CP-6A-9A	R	5.9		0.88
	CP-6B-9A	T	4.9		1.97
Abrasion resistance	CP-6A-10A	R	6.2	depth, in.	wt. loss, grams
	CP-6A-10B	R	5.6	0.045	9
	CP-6A-10C	R	5.4	0.046	8
				0.050	8
	CP-6B-10A	T	5.5	0.050	10
	CP-6B-10B	T	5.4	0.052	9
	CP-6B-10C	T	5.6	0.064	10
Hardness	—	R	—	"L" impact hammer	
	—	T	—		30
					23
Cavitation resistance	CP-6A-16A	R	6.7	depth, in., (2-hr exposure)	
	CP-6B-16A	T	6.0		0.072
					0.115
Thermal properties coefficient of expansion	CP-6A-9A	R	5.9	in./in./°F	
	CP-6B-9A	T	4.9		4.67
					4.77
Specific heat	CP-6A-8A	R	6.7	btu/lb/°F at 73° F	
	CP-6B-8A	T	6.3		0.201
					0.225
Conductivity	CP-6A-8A	R	6.7	btu/ft/hr/°F at 73° F	
	CP-6B-8A	T	6.3		1.300
					1.330
Diffusivity	CP-6A-8A	R	6.7	ft²/hr at 73° F	
	CP-6B-8A	T	6.3		0.044
					0.040
Specific gravity	CP-6A-8A	R	6.7		2.360
	CP-6B-8A	T	6.3		2.373

CP Concrete

Ovendried at 221° F (105° C) prior to impregnation

R = radiation polymerization

T = thermal-catalytic polymerization

Plotted values are averages of two specimens, except for results on single specimens plotted for radiation polymerized styrene-impregnated concrete.

6x12-inch specimens, oven dried at 221° F (105° C) prior to impregnation.

R radiation polymerization

T thermal-catalytic polymerization

Creep values above the zero line indicate a contraction in length of specimen.

Creep values below the zero line indicate an expansion in length of specimen (negative creep).

Figure 11. Creep tests on polymer-impregnated concrete under 800 psi compressive load.

Plotted values are averages of two specimens.

4-1/2x12-inch specimens, oven dried at 302° F (150° C) prior to impregnation.
Radiation polymerization.

Points below the zero line indicate negative creep; i.e., a contraction of specimens under tensile stress, or an expansion of specimens under compressive stress.

Figure 12. High-stress creep tests on polymer-impregnated concrete under tensile and compressive loads.

Table 16
EFFECT OF TIME ON COMPRESSIVE STRENGTH AND ELASTICITY

MMA-impregnated Concrete

Specimen number	MMA weight percent	Age after impregnation, days	Compressive strength, psi	Modulus of elasticity 10 ⁶ psi
1D	4.3	7	21,780	7.98
2D	4.3	7	18,310	7.59
3D	4.3	7	21,350	7.12
Average	4.3		20,480	7.6
19D	0	7	5,740	4.46
20D	0	7	5,980	4.33
21D	0	7	6,250	4.42
Average			5,990	4.4
4D	4.0	21	17,890	7.51
5D	4.0	21	18,030	7.80
6D	3.8	21	14,500	6.80
Average	3.9		16,810	7.4
22D	0	21	5,770	4.05
23D	0	21	5,430	3.79
24D	0	21	5,370	3.75
Average			5,520	3.9
7D	4.1	60	16,970	6.95
8D	4.2	60	17,390	7.14
9D	4.2	60	18,810	7.02
Average	4.2		17,720	7.0
25D	0	60	5,420	3.98
26D	0	60	5,880	3.86
27D	0	60	5,420	3.73
Average			5,570	3.9
10D	4.1	90	17,300	7.86
11D	3.9	90	19,500	7.24
12D	4.3	90	20,800	7.64
Average	4.1		19,200	7.6
23D	0	90	5,500	4.45
29D	0	90	5,450	4.64
30D	0	90	5,340	4.18
Average			5,430	4.4

MMA = Methyl methacrylate

3- by 6-inch specimens

Ovendried at 302° F (150° C) prior to impregnation

Radiation polymerization

Figure 13. Test facility for high-stress creep tests. Room is kept at 50 percent relative humidity and a constant temperature of 73° F (23° C).

Figure 14. Measuring creep under sustained compressive loading.

reported in the Second Topical Report at earlier loading times, continued to be found in most MMA specimens. One radiation-polymerized MMA specimen has a slight positive creep of 8 millionths and the other, a small negative creep of 26 millionths. The thermal-catalytically polymerized MMA specimens both show consistent negative creep, averaging 62 millionths. The styrene-impregnated specimens show erratic creep. Thermal-catalytically polymerized styrene specimens show high-positive creep, which now exceeds the creep of control concrete; the

radiation-polymerized specimens now show a positive creep of 4 millionths for one specimen and a negative creep of 158 millionths for the companion specimen.

To further investigate creep behavior, MMA- and chlorostyrene-impregnated specimens are being tested under tensile loads and under higher compressive loads. Results for 31 days under sustained load are available (Figure 12), and show both materials have significantly less creep in tension and compression than unimpregnated concrete. Creep test facilities are shown in Figures 13 and 14.

The high-stress compressive creep tests on MMA- and chlorostyrene-impregnated concrete show low-positive creep under sustained compressive loads of 7,000, 2,313, and 690 psi. Unit deformation (millionths per pounds per square inch) is essentially the same for all three loads. Both materials showed low-negative creep on initial readings at 1 day, but since then have shown low-positive creep.

The high-stress tensile creep tests show negative creep (in this case, a decrease in length under tension) for MMA-impregnated concrete under 178- and 345-psi tensile load, and chlorostyrene-impregnated concrete under 178-psi tensile load. Chlorostyrene-impregnated concrete shows essentially zero creep under 345-psi tensile load.

5.1.1.1.3. Effect of time on strength of MMA-impregnated concrete

A series of tests is in progress to determine the effects of time on the compressive strength and elasticity of MMA-impregnated concrete. Test specimens (3- by 6-inch cylinders) are impregnated, polymerized, and stored in the 50-percent relative humidity room at 73° F (23° C). Testing is scheduled at ages of 7, 21, 60, 90, 180, and 360 days after impregnation and polymerization. Test results to date are available for up to 90 days' aging (Table 16). Modulus of elasticity is generally in line with compressive strength. The data show some decrease in strength for the 21- and 60-day MMA specimens, but fairly high strength for 90-day specimens (19,200-psi average, as compared with 20,480-psi average for the initial strengths at 7 days). This appears to be due to the normal variations encountered in test specimens and is not considered indicative of a trend toward lower strengths in this time interval.

5.1.1.2. Durability Properties

Durability tests on impregnated CP-type concrete include freeze-thaw, sulfate attack, and resistance to 15

percent HCl and 15 percent H_2SO_4 . Test results on single specimens for each test are plotted in Figures 15, 16, 17, and 18; a summary of averaged results is given in Table 17.

Freeze-thaw Durability.—All PIC specimens show a greatly improved resistance to freezing and thawing as compared with unimpregnated concrete.

The greatest durability obtained so far was with an earlier survey series specimen impregnated with MMA and radiation polymerized. This specimen had a weight loss of 0.5 percent after 7,220 cycles, an improvement of about 900 percent over control concrete. The control specimens failed at 740 cycles (25 percent weight loss is the criterion used for failure), which is normally considered good durability. The unimpregnated radiation and thermally treated CP-3 series concrete failed at 440 cycles. Radiation and thermal-catalytically polymerized MMA and MMA plus 10 percent TMPTMA, and thermal-catalytically polymerized acrylonitrile had little weight loss at from 3,650 to 5,550 cycles of freeze-thaw. Such specimens show improvements in durability ranging from more than 390 percent to over 650 percent as compared with control concrete. Radiation-polymerized acrylonitrile specimens which showed the lowest durability of PIC in this test series had 128 percent improvement in durability over control concrete. The thermal-catalytically polymerized styrene concrete is still in test, with a weight loss of 21 percent at 5,400 cycles; the radiation-polymerized styrene specimens failed at 2,635 cycles. The radiation and thermal-catalytically polymerized chlorostyrene specimens are showing weight losses of 22 and 10 percent, respectively, at 1,800 cycles.

Resistance to Chemical Attack by Soluble Sulfates.—The accelerated sulfate resistance test, consisting of 1 cycle per day of alternately soaking the specimens in a 2.1-percent solution of Na_2SO_4 at 73° F (22° C) and drying at 130° F (54° C), is continuing on the CP series concrete specimens. MMA, styrene, and MMA plus 10 percent TMPTMA specimens, both radiation and thermal-catalytically polymerized, are showing very little expansion at exposures of from 540 to 720 days. The thermal-catalytically polymerized acrylonitrile is showing good sulfate resistance; however, one radiation-polymerized acrylonitrile specimen was removed at 180 days because of excessive deterioration, and the companion specimen is showing 0.462-percent expansion at 540 days. Thermal-catalytically polymerized chlorostyrene has a

very low expansion of 0.009 percent at 300 days, and radiation-polymerized chlorostyrene has a much higher expansion of 0.168 percent. The unimpregnated CP-3 series concrete specimens have high expansion; the radiation-treated specimens expanded 0.395 percent at 690 days, and thermally treated specimens expanded 0.522 percent at 605 days. The control concrete showed 0.467 percent expansion at 480 days.

Resistance to Chemical Attack by 15 Percent Hydrochloric Acid.—This test consists of continuous immersion of 3- by 6-inch cylindrical specimens in 15 percent HCl at room temperature. The unimpregnated controls and the thermally and radiation treated CP-3 series unimpregnated concrete show low resistance to 15 percent HCl; these specimens have failed at about 105 days' exposure. All polymer-impregnated specimens show significantly improved durability in exposure to 15 percent HCl. Improvements in HCl resistance of impregnated-concrete range from more than 178 percent to over 667 percent for exposures ranging from 292 to 805 days.

Resistance to Chemical Attack by 15 Percent Sulfuric Acid.—This test consists of continuous immersion of 3- by 3- by 5-inch specimens in 15 percent H_2SO_4 at room temperature. Several MMA-impregnated 3- by 6-inch cylindrical specimens have also been tested. The test series has now been concluded. Exposure to 15 percent H_2SO_4 is more severe for concrete than exposure to 15 percent HCl. The control concrete failed at 49 days. In the CP-3 series the unimpregnated radiation-treated concrete failed at 63 days, and thermally treated concrete failed at 77 days. The MMA and MMA plus 10 percent TMPTMA-impregnated concrete produced the highest resistance to 15 percent H_2SO_4 with failures at 119 to 133 days, which is about a 143- to 157-percent increase in durability as compared with control concrete. The 3- by 6-inch MMA-impregnated specimens showed comparable performance, with failures at 91 and 119 days for radiation polymerization and over 133 days for thermal-catalytic polymerization. Styrene impregnation showed an average of 86-percent improvement over the controls for radiation polymerization and 57 percent for thermal-catalytic polymerization. Chlorostyrene showed a comparable performance, averaging 71-percent improvement for radiation polymerization and 57-percent improvement for thermal-catalytic polymerization. Acrylonitrile results were slightly lower, with 57-percent improvement for radiation polymerization and 43 percent for thermal-catalytic polymerization.

Plotted values are results on single specimens.

3x6-Inch specimens, oven dried at 221° F (105° C) prior to impregnation.

R_a, R_b radiation polymerized specimens

T_a, T_b thermal-catalytically polymerized specimens

Figure 15. Freeze-thaw durability of polymer-impregnated concrete.

Plotted values are results on single specimens, except as noted. Average values of two companion specimens are plotted when results closely coincide.

Specimens oven dried at 221° F (105° C) prior to impregnation.

R_a , R_b radiation polymerized specimens
 T_a , T_b thermal-catalytically polymerized specimens

Figure 16. Resistance of polymer-impregnated concrete to chemical attack by sulfates.

Plotted values are results on single specimens.

3x6-inch specimens, oven dried at 221° F (105° C) prior to impregnation.

R_a , R_b radiation polymerized specimens
 T_a , T_b thermal-catalytically polymerized specimens

Figure 17. Resistance of polymer-impregnated concrete to chemical attack by 15 percent HCl.

Figure 18. Resistance of polymer-impregnated concrete to chemical attack by 15 percent H_2SO_4 .

ACRYLONITRILE IMPREGNATED CONCRETE, CP-5 SERIES

CHLOROSTYRENE IMPREGNATED CONCRETE, CP-6 SERIES

METHYL METHACRYLATE IMPREGNATED CONCRETE, CP-1 SERIES

Plotted values are results on single specimens

Specimens oven dried at 221° F (105° C) prior to impregnation

R_a, R_b radiation polymerized specimens

T_a, T_b thermal-catalytically polymerized specimens

Figure 18 (Continued). Resistance of polymer-impregnated concrete to chemical attack by 15 percent H_2SO_4 .

Table 17

SUMMARY OF DURABILITY PROPERTIES OF POLYMER-IMPREGNATED CONCRETE
CP Test Series

Property	Unimpregnated concrete			MMA impregnated		S impregnated		MMA+TMPTMA impregnated		AN impregnated		CS impregnated	
	CP-0 series Controls	CP-3 test series		CP-1 test series		CP-2 test series		CP-4 test series		CP-5 test series		CP-6 test series	
		R	T	R	T	R	T	R	T	R	T	R	T
Freeze-thaw durability													
Polymer loading, %	0	0	0	5.9	6.8	4.9	4.4	1/ 6.2	2/ 7.2	5.1	5.5	4.2	6.4
Number of cycles	740	440	440	5,550	3,650	2,635	5,440	4,560	4,660	1,690	4,120	1,800	1,800
Percent weight loss	25	28	35	7	2	25	21	8.5	0	25	6	22	10
Percent improvement		-	-	>650	>393	>256	>635	>530	>530	128	>457	>143	>143
Sulfate attack													
Polymer loading, %	0	0	0	2/ 6.6	6.4	5.4	2/ 4.5	2/ 6.5	7.3	3/ 5.1	4/ 6.0	6.0	6.4
Test cycle days	480	690	605	720	720	690	690	690	630	540	540	300	300
Percent expansion	0.467	0.396	0.522	0.018	0.006	0.023	0.030	0.010	0.003	0.462	0.032	0.148	0.009
Acid resistance, 15% HCl													
Polymer loading, %	0	0	0	6.1	2/ 6.4	4.9	4.6	6.1	2/ 7.1	5.2	5.4	6.0	6.4
Exposure days	105	103	106	805	805	805	805	709	709	623	623	292	292
Percent weight loss	27	26	26	12	9	16	12	13	7	16	12	9	8
Percent improvement		0	0	>667	>667	>667	>667	>575	>575	>493	>493	>178	>178
Acid resistance, 15% H ₂ SO ₄													
Polymer loading, %	0	0	0	5.7	2/ 5.7	5.4	4.0	6.1	-	2/ 4.4	3.8	6.4	5.7
Exposure days	49	63	77	126	119	91	77	126	-	77	70	84	77
Percent weight loss	35	30	30	29	26	27	29	27	-	28	33	28	26
Percent improvement	-	29	17	157	143	86	57	157	-	57	43	71	57
Distilled water corrosion, 120-day exposure at 206° F (97° C)	Severe	-	-	No attack	-	No attack	-	-	-	-	-	-	-

Results are averages of two specimens, except as noted.

1/ Single specimen, companion failed (25 percent weight loss) at 3,350 cycles.

2/ Single specimen, companion removed for display.

3/ Single specimen, companion removed at 180 days due to excessive deterioration.

4/ Single specimen, companion removed at 390 days due to excessive deterioration.

Specimens oven-dried at 221° ± (105° C) prior to impregnation.

MMA = methyl methacrylate

S = styrene

TMPTMA = trimethylolpropane trimethacrylate

AN = acrylonitrile

CS = chlorostyrene

R = Radiation polymerization

T = Thermal-catalytic polymerization

5.1.2. Partially Impregnated Concrete

Partially impregnated concrete is designed for durability rather than high strength, which permits a saving in amount of monomer as compared with fully impregnated concrete. The concrete is impregnated with specially formulated monomer systems to give controlled depths of penetration. The monomers should effectively seal off the surface and penetrate to a sufficient depth so that separation of the impregnated layer does not cause premature failure. Viscous monomer systems are used because penetration depths are more easily controlled, voids in the penetrated portion remain filled, and monomer losses due to evaporation and drainage during the time between impregnation and complete polymerization are lower.

Epoxy- and polyester-styrene monomer mixtures have been investigated for partial impregnation of CP-type concrete. The compositions of the monomer mixtures, viscosity, and depth of penetration are given in Table 18. Tests showed a general improvement in strength ranging from about 5 to 50 percent as compared with

unimpregnated concrete, and a generally beneficial but erratic improvement in durability. Depth of penetration in the ranges investigated does not appear to have a consistent measurable effect on durability. Generally speaking, polyester-styrene improved concrete properties a little better than epoxy-styrene.

The difficulties in attempting to obtain uniform penetration of concrete to controlled depths within a very narrow zone are reflected in the data on polymer loadings for the various penetration depths, which do not show a consistent increase in polymer loading with the penetration depth. The surface irregularities of concrete and the distribution of aggregate particles and voids within the concrete contribute to variations in the permeability of the concrete. The problem is probably complicated by shrinkage during polymerization. The determination of an optimum penetration depth, which may be alternatively viewed as determining the proper viscosity and the immersion times required to achieve an effective sealing of the concrete, is essentially a trial and error process.

Table 18
COMPOSITION AND VISCOSITY OF MONOMER MIXTURES
Partially Impregnated Concrete

Depth of penetration				Monomer mixtures	
Sulfate, HCl, and freeze-thaw specimens		Strength and elasticity specimens			
Penetration depth, inches	Nominal penetra- tion, inches	Penetration depth, inches	Nominal penetra- tion, inches	Composition weight, percent	Viscosity cps
Epoxy-styrene					
1/32 to 1/16	1/16	1/16 to 1/8	1/8	65E + 35S	250
1/16 to 1/8	1/8	1/8 to 3/16	3/16	60E + 40S	125
1/8 to 1/4	1/4	3/16 to 1/4	1/4	58E + 32S	90
Polyester-styrene					
1/32 to 1/16	1/16	1/16 to 1/8	1/8	58P + 42S	860
1/16 to 1/8	1/8	1/8 to 3/16	3/16	52P + 48S	420
1/8 to 1/4	1/4	3/16 to 1/4	1/4	44P + 56S	126

E = Epoxy
P = Polyester
S = Styrene

Specimens are immersed in monomer solutions of selected viscosities for varying periods of time to give the different penetration depths. Immersion times and ranges in penetration depths were determined from previous trial experiments.

Strength and elasticity test results on partially impregnated CP concrete are given in Table 19. Polymer loadings range from 1.60 to 2.25 percent by weight for epoxy-styrene and from 2.02 to 2.59 percent for polyester-styrene. Compressive strengths of specimens with these relatively small amounts of polymer all showed an increase over those of unimpregnated concrete, ranging from 5,540 to 7,380 psi for epoxy-styrene and 6,790 to 8,220 psi for polyester-styrene.

Durability test results were erratic, but generally showed that partial impregnation improves durability as compared with unimpregnated concrete. The tests do not show a consistent improvement in durability with increased penetration depths.

Concrete in the freeze-thaw test, Table 20 showed excellent durability. A problem of interpretation presented by this test series was the very high durability of the control specimens. One control

specimen did not fail until 2,060 cycles, and the other is still in test at 2,310 cycles with a weight loss of 13 percent. Usually, this type of concrete fails the test at about 800 or 900 cycles, which is considered good durability. Concrete exceeding 1,000 cycles is generally considered to have excellent durability. Concrete test specimens for the freeze-thaw test are sensitive to curing conditions and subsequent drying. Past tests have shown that concrete specimens similar to these control specimens, given a 28-day fog cure and stored 90 days or more in a controlled-temperature atmosphere at 50 percent relative humidity, may develop durabilities of over 3,000 cycles. In other cases, drying may decrease durability. When specimens such as these are exposed to a variety of conditions during preparation, great care must be exercised in interpreting results from the freeze-thaw test.

In resistance to 15 percent HCl (Table 21) both polyester-styrene and epoxy-styrene show improved durability. Unimpregnated control specimens failed the

Table 19

MECHANICAL PROPERTIES OF PARTIALLY IMPREGNATED CONCRETE

Monomer	Specimen number	Nominal penetration inches	Polymer loading, weight percent	Compressive strength, psi	Modulus of elasticity, 10 ³ psi
None	Control	—	—	5,270	3.50
Epoxy-styrene	CP-6-24	1/8	1.75	6,580	3.71
	CP-6-25	1/8	1.60	6,360	3.22
	CP-6-22	3/16	1.74	5,540	3.13
	CP-6-23	3/16	1.65	5,800	3.04
	CP-6-20	1/4	2.05	6,440	3.65
	CP-6-21	1/4	2.25	7,380	3.88
Polyester-styrene	CP-6-26	1/8	2.02	6,900	3.42
	CP-6-27	1/8	2.19	6,790	3.64
	CP-6-28	3/16	2.21	7,880	3.94
	CP-6-29	3/16	2.23	7,600	3.86
	CP-6-30	1/4	2.56	8,220	3.88
	CP-6-31	1/4	2.59	7,920	4.10

CP concrete
3- by 6-inch cylinders
Ovendried at 302° F (150° C) prior to impregnation
Radiation polymerization

Table 20

FREEZE-THAW DURABILITY OF PARTIALLY IMPREGNATED CONCRETE

CP Concrete Impregnated to Various Depths by Epoxy-styrene and Polyester-styrene

Monomer	Specimen number	Nominal penetration depth, inches	Polymer loading weight percent	Percent weight loss from exposure to freeze-thaw cycles					
				510 cycles	800 cycles	1,000 cycles	1,480 cycles	1,860 cycles	2,310 cycles
None (controls)	CP-DP-51	—	—	2.5	6.0	8.5	17.0	23.0	F ¹
	CP-DP-52	—	—	2.5	3.5	4.0	6.5	10.5	18.0
Epoxy-styrene	CP-DP-5	1/16	1.96	*(2.0)	*(1.5)	*(1.0)	*15.0	F ²	—
	CP-DP-10	1/16	2.06	*(2.0)	*(1.5)	*(1.0)	3.0	5.0	11.5
	CP-DP-36	1/8	1.81	0.5	4.5	5.5	11.0	16.5	F ³
	CP-DP-16	1/8	1.99	*(3.5)	*(3.0)	*(2.5)	*(2.5)	*(2.0)	*(2.0)
	CP-DP-47	1/4	2.53	0	2.0	3.0	6.5	10.0	F ⁴
	CP-DP-13	1/4	1.38	*(3.5)	1.5	4.0	18.0	F27.0	—
Polyester-styrene	CP-DP-14	1/16	1.20	*(4.5)	*(5.0)	*(5.0)	*(5.0)	*(5.0)	1.0
	CP-DP-42	1/16	1.00	*(1.0)	3.0	9.0	F ⁵	—	—
	CP-DP-21	1/8	1.13	*(4.5)	*(5.0)	*(5.0)	F29.0	—	—
	CP-DP-41	1/8	1.72	*(4.0)	0.5	0.5	4.0	7.5	14.0
	CP-DP-32	1/4	1.92	*(4.0)	*(4.0)	*(4.0)	*(4.0)	6.5	15.5
	CP-DP-46	1/4	1.61	*(2.5)	*(1.5)	*(1.0)	2.0	2.5	3.0

CP concrete

3- by 6-inch cylinder specimens

Radiation polymerization

Specimens oven-dried at 302° F (150° C) prior to impregnation

*Gain in weight

F = Specimen failed, weight loss of 25 percent or more

¹ 25 percent at 2,060 cycles² 35 percent at 1,560 cycles³ 27 percent at 2,210 cycles⁴ 33 percent at 1,960 cycles⁵ 40 percent at 1,320 cycles

Table 21

RESISTANCE OF PARTIALLY IMPREGNATED CONCRETE TO CHEMICAL ATTACK BY
15 PERCENT HCL

Monomer	Specimen No.	Nominal penetration depth inches	Polymer loading wt %	Water absorption wt %	Percent weight loss from exposure to 15% HCl					
					70 days	105 days	161 days	231 days	294 days	350 days
None (controls)	CP-DP-53	-	-	6.84	5.03	8.80	13.41	17.60	F 1/	-
	CP-DP-43	-	-	6.91	4.12	7.90	12.33	17.01	F 2/	-
Epoxy-styrene	CP-DP-6	1/16	1.93	3.13	1.92	3.60	5.59	6.85	7.70	8.48
	CP-DP-45	1/16	1.98	3.22	1.51	2.93	4.79	5.88	6.70	7.56
	CP-DP-38	1/8	2.05	4.37	4.06	7.94	10.55	12.22	13.62	14.57
	CP-DP-44	1/8	2.13	4.49	4.54	7.73	10.44	11.97	13.88	14.95
	CP-DP-33	1/4	2.01	3.87	2.63	4.14	6.08	7.35	8.28	9.04
	CP-DP-40	1/4	2.00	4.80	5.21	7.99	10.76	12.79	15.79	17.47
Polyester-styrene	CP-DP-1	1/16	1.10	2.98	3.20	5.00	7.67	8.81	9.69	10.33
	CP-DP-28	1/16	1.46	3.70	2.10	3.82	5.91	7.88	8.72	10.01
	CP-DP-8	1/8	1.61	0.60	0.21	1.87	3.87	5.03	5.86	6.60
	CP-DP-48	1/8	1.72	1.65	0.92	2.78	4.92	6.08	6.70	7.49
	CP-DP-29	1/4	1.78	0.26	0.39	1.80	3.87	5.15	6.12	7.15
	CP-DP-35	1/4	1.92	1.10	*(1.44)	*(0.42)	1.20	2.44	3.41	4.57

CP concrete

3- by 6-inch cylinders

Ovendried at 302° F (150° C) prior to impregnation

Radiation polymerization

* Weight gain

F Failed, weight loss of 25 percent or more

1/ 27 percent at 259 days.

2/ 35 percent at 259 days.

test by 259 days with weight losses of over 25 percent. The polyester-styrene specimens showed better acid resistance and some indication of improvement with increased depth of penetration; weight losses at 350 days ranged from about 5 percent for 1/4-inch penetration to about 10 percent for 1/16-inch penetration. Weight losses for epoxy-styrene specimens did not show an improvement with penetration depth and varied between 7 and 15 percent at 350 days.

The accelerated sulfate resistance tests (Table 22) show improved durability for partially impregnated concrete, with epoxy- and polyester-styrene giving about the same improvement. Depth of penetration appears to have no effect. Epoxy- and polyester-styrene specimens have expanded about 0.1 percent at 240 days, which is about one-half the expansion of the unimpregnated control specimens.

5.2. POLYMER-IMPREGNATED CONCRETE FOR DESALTING PLANT APPLICATIONS AT TEMPERATURES UP TO 290° F

Construction materials for distillation units in a desalting plant have special requirements of high strength and high resistance to attack by brine and distilled water at temperatures up to 290° F (143° C). The polymers investigated for high-temperature applications are selected for stability and durability at higher temperatures, and for proper viscosity for full impregnation. These monomer systems generally contain a cross-linking monomer to provide stability at high temperatures. Many monomers suitable for normal temperature applications, such as MMA, cannot be used because they have low-softening points.

Table 22

RESISTANCE OF PARTIALLY IMPREGNATED CONCRETE TO CHEMICAL ATTACK BY SULFATES

Monomer	Specimen number	Nominal penetration depth inches	Polymer loading weight percent	Accelerated sulfate resistance test Percent expansion			
				65 days	156 days	174 days	240 days
None (controls)	CP-DP-49	—	—	0.074	0.140	0.156	0.192
	CP-DP-50	—	—	0.078	0.130	0.144	0.168
Epoxy-styrene	CP-DP-20	1/16	1.80	0.080	0.086	0.086	0.086
	CP-DP-11	1/16	2.27	0.096	0.106	0.112	0.118
	CP-DP-39	1/8	1.98	0.090	0.100	0.102	0.112
	CP-DP-12	1/8	2.30	0.078	0.082	0.086	0.092
	CP-DP-24	1/4	1.42	0.098	0.098	0.104	0.108
	CP-DP-43	1/4	2.51	0.088	0.102	0.110	0.108
Polyester-styrene	CP-DP-22	1/16	0.90	0.072	0.084	0.084	0.082
	CP-DP-15	1/16	0.90	0.086	0.090	0.090	0.086
	CP-DP-2	1/8	1.13	0.130	0.118	0.116	0.118
	CP-DP-34	1/8	1.66	0.082	0.068	0.078	0.088
	CP-DP-9	1/4	1.88	0.086	0.092	0.100	0.092
	CP-DP-26	1/4	1.66	0.080	0.082	0.078	0.086

CP concrete

3- by 6-inch cylinders

Ovendried at 302° F (150° C) prior to impregnation

Radiation polymerization

Acrylonitrile has a high-softening point and is potentially suitable for use without a cross-linking agent.

The concrete, designated as OSW-type, is a specially designed high-strength mix, made with Type V cement, fly ash pozzolan, a low water/cement plus pozzolan ratio, air entrainment, water-reducing agent, and California aggregate. The concrete is mixed and placed by conventional methods. The concrete is fog cured for 28 days and oven-dried at 302° F (150° C) prior to impregnation. Some of the earlier impregnated concrete was polymerized by radiation, but now the impregnated concrete is thermal-catalytically polymerized. The testing program consists of tests of structural properties and tests to determine effects of exposure to concentrated brine. Tests are conducted at 76° F (24° C), 250° F (121° C), and 290° F (143° C).

5.2.1. Structural Properties

A survey series of test experiments was made to obtain preliminary compressive and tensile strength data at temperatures of 76°, 250°, and 290° F on prospective monomer systems for high-temperature applications. So far, four monomer systems have been evaluated: MMA plus 10 percent TMPTMA, 60 percent styrene plus 40 percent TMPTMA, acrylonitrile with an epoxy-styrene coating, and 90 percent DAP plus 10 percent MMA. Tests were made on three specimens for each condition.

The test experiments in the survey series are identified as follows:

- OSW-1 MMA plus 10 percent TMPTMA, radiation polymerization
- OSW-3 60 percent styrene plus 40 percent TMPTMA, radiation polymerization
- OSW-4 Acrylonitrile with an epoxy-styrene coating, radiation polymerization
- OSW-5 90 percent DAP plus 10 percent MMA, thermal-catalytic polymerization
- OSW-6 Unimpregnated controls

Results of these tests were reported in the Second Topical Report. These results are summarized and included in Table 23. In addition, a few tests were made on CP-type concrete impregnated with 60 percent styrene plus 40 percent TMPTMA.

On the basis of the results of the survey series experiments, two monomer systems were selected for further investigation: 60 percent styrene plus 40

percent TMPTMA and 90 percent DAP plus 10 percent MMA. A test series of experiments was made on these two systems to confirm survey series experiments and to obtain more reliable data. In these tests 10 specimens were used for each condition for compressive strengths at 76°, 250°, and 290° F. A summary of averaged results is included in Table 23, and results on individual specimens are given in Tables 24 and 25. Results on CP-type concrete impregnated with 60 percent styrene plus 40 percent TMPTMA are given in Table 26.

In the test series, OSW concrete impregnated with 60 percent styrene plus 40 percent TMPTMA and radiation polymerized, showed some decrease in average compressive strength from 19,210 psi at 76° F to 16,130 psi at 250° F and 16,180 psi at 290° F. This decrease was a little more pronounced than in the survey series, which averaged 19,710 psi at 76° F, 18,750 psi at 250° F, and 18,600 psi at 290° F. The 90 percent DAP plus 10 percent MMA specimens ranged from 18,430 psi at 76° F, 16,130 psi at 250° F, to 15,260 psi at 290° F in the test series experiments. Tensile strengths from the survey experiments showed little or no decrease due to high temperature. Radiation polymerized 60 percent styrene plus 40 percent TMPTMA showed a slight increase in tensile strength, from 1,140 psi at 76° F, to 1,260 psi at 250° F, to 1,280 psi at 290° F. Thermal-catalytically polymerized 90 percent DAP plus 10 percent MMA showed a slight decrease with temperature, from 1,180 psi at 76° F to 1,060 psi at 250° F and 1,040 psi at 290° F.

The survey series on the standard mix CP-type concrete, impregnated with 60 percent styrene plus 40 percent TMPTMA and thermal-catalytically polymerized, showed in general, comparatively high compressive and tensile strengths with no real indication of lower strengths at higher temperatures. Compressive strengths were 17,140 psi at 76° F, 15,020 psi at 250° F, and 16,180 psi at 290° F; and tensile strengths were 910 psi at 76° F, 1,000 psi at 250° F, and 1,010 psi at 290° F. These test results indicate that for high-strength PIC, standard concrete mixes give results comparable to those for the more expensive high-strength concrete mixes.

Creep tests on OSW-type concrete impregnated with 60 percent styrene plus 40 percent TMPTMA and 90 percent DAP plus 10 percent MMA to investigate creep under high-compressive load at 290° F have been in progress for 54 days. Test facilities are shown in Figures 19 and 20. Additional tests are planned to study creep of CP-type concrete impregnated with

Table 21
SUMMARY OF MECHANICAL PROPERTIES
Polymer Impregnated Concrete for High-Temperature Applications

Concrete mix	Polymer	Polymerisation method	Compression tests				Tension tests		Improvement in properties, percent increase due to impregnation			
			Polymer loading wt percent	Compressive strength psi	Modulus of elasticity $\times 10^6$ psi	Poisson's ratio	Polymer loading wt percent	Tensile strength psi	Compressive strength	Modulus of elasticity	Poisson's ratio	Tensile strength
TESTS AT 76° F												
Survey Series 1/												
OSW	Control	-	0	8,740	5.2	0.16	0	560	-	-	-	-
OSW	MMA + THPTMA	R	4.3	16,321	4.9	0.24	4.2	1,000	87	-7	50	78
OSW	AN + E	R	3.4	16,640	5.8	0.17	3.3	1,090	90	11	6	95
OSW	DAP + MMA	T	4.9	18,120	6.9	0.19	4.8	1,180	107	32	16	112
OSW	S + THPTMA	R	5.4	19,710	7.2	0.23	5.3	1,140	125	38	44	103
OSW	S + THPTMA	T	4.4	19,920	7.7	0.21	-	-	128	48	31	-
CP	Control	-	0	4,570	3.6	0.17	0	390	-	-	-	-
CP	S + THPTMA	T	6.6	17,140	6.3	0.21	6.6	910	275	79	24	133
Test Series 2/												
OSW	Control	-	0	8,850	5.1	0.19	-	-	-	-	-	-
OSW	DAP + MMA	T	4.8	18,420	7.2	0.20	-	-	108	42	5	-
OSW	S + THPTMA	R	4.0	19,210	7.7	0.20	-	-	117	41	5	-
TESTS AT 250° F												
Survey Series 1/												
OSW	Control	-	0	7,080	4.5	0.22	0	450	-	-	-	-
OSW	MMA + THPTMA	R	4.5	11,905	3.6	0.20	4.2	780	68	-20	-9	71
OSW	AN + E	R	3.4	12,670	4.4	0.17	3.3	960	79	-4	-23	108
OSW	DAP + MMA	T	4.7	13,930	6.4	0.25	4.8	1,060	97	42	12	135
OSW	S + THPTMA	R	5.6	18,750	7.4	0.25	5.4	1,260	165	63	14	178
OSW	S + THPTMA	T	3.3	13,000	6.4	0.27	-	-	83	42	23	-
CP	Control	-	0	3,950	3.1	0.11	0	340	-	-	-	-
CP	S + THPTMA	T	6.7	15,070	6.4	0.18	6.1	1,050	282	102	64	194
Test Series 2/												
OSW	Control	-	0	6,300	4.6	0.21	-	-	-	-	-	-
OSW	DAP + THPTMA	T	4.8	16,420	6.6	0.22	-	-	161	47	5	-
OSW	S + THPTMA	R	3.9	16,130	7.4	0.20	-	-	156	60	-5	-
TESTS AT 250° F												
Survey Series 1/												
OSW	Control	-	0	7,500	5.1	0.20	0	480	-	-	-	-
OSW	MMA + THPTMA	R	4.0	9,619	2.6	0.17	4.2	790	28	-44	-15	67
OSW	AN + E	R	3.4	11,880	4.3	0.13	3.3	900	58	-14	-35	89
OSW	DAP + MMA	T	4.6	12,920	5.7	0.23	4.4	1,040	72	14	15	118
OSW	S + THPTMA	R	5.8	18,600	7.2	0.23	5.8	1,280	148	43	15	169
OSW	S + THPTMA	T	3.7	13,970	6.7	0.23	-	-	74	32	15	-
CP	Control	-	0	4,400	3.0	0.21	0	330	-	-	-	-
CP	S + THPTMA	T	6.6	16,180	6.1	0.24	6.5	1,010	259	107	14	206
Test Series 2/												
OSW	Control	-	0	6,670	4.7	0.19	-	-	-	-	-	-
OSW	DAP + MMA	T	4.9	15,260	6.2	0.17	-	-	124	31	-11	-
OSW	S + THPTMA	R	3.9	15,850	7.1	0.20	-	-	138	52	5	-

Average test results on 6- by 12-inch cylinders, fog cured 28 days, tested at 120 days age:

1. Survey series results are averages of three specimens.
2. Test series results are averages of 10 specimens.

OSW - High-strength concrete mix.

CP - Standard concrete mix.

Specimens oven-dried at 302° F (150° C) prior to impregnation.

R - Radiation polymerization

T - Thermal-catalytic polymerization

MMA + THPTMA - 90 percent methyl methacrylate + 10 percent trimethylolpropane trimethacrylate

AN + E - Acrylonitrile with an in-depth coating of epoxyl

DAP + MMA - 90 percent diallyl phthalate + 10 percent methyl methacrylate

S + THPTMA - 60 percent styrene + 40 percent trimethylolpropane trimethacrylate

Table 24

HIGH-TEMPERATURE MECHANICAL PROPERTIES

Styrene plus TMPTMA-impregnated OSW-type Concrete

Specimen number	Polymerization	Polymer loading, percent	Specimen temperature, ° F	Compressive strength, psi	Modulus of elasticity 10 ⁶ psi	Poisson's ratio
Control ¹	—	0	76	8,850	5.06	0.19
Control ¹	—	0	250	6,300	4.60	0.21
Control ¹	—	0	290	6,670	4.71	0.19
LA-1-1	R	4.0	76	21,140	7.77	0.21
LA-1-3	R	4.0	76	19,500	7.25	0.18
LA-1-4	R	3.7	76	17,710	7.63	0.20
LA-1-5	R	4.0	76	19,500	7.16	0.20
LA-1-7	R	4.2	76	20,570	8.19	0.23
LA-1-10	R	4.0	76	18,640	7.58	0.20
LA-1-11	R	3.9	76	17,710	8.41	0.23
LA-1-12	R	4.1	76	18,930	7.25	0.19
LA-1-2	R	3.3	249	13,860	6.05	0.23
LA-1-6	R	3.7	250	17,390	8.41	—
LA-1-15	R	4.0	250	16,000	6.69	0.19
LA-1-17	R	4.0	251	18,460	7.69	0.25
LA-1-19	R	4.2	250	16,800	7.98	0.18
LA-1-20	R	3.7	251	16,860	5.44	0.18
LA-1-21	R	4.0	249	14,430	7.60	0.19
LA-1-22	R	4.0	250	15,710	8.48	0.10
LA-1-25	R	4.2	252	16,710	7.66	0.23
LA-1-27	R	3.7	251	15,070	7.57	0.23
LA-1-8	R	3.5	291	15,430	7.33	0.23
LA-1-14	R	3.5	290	14,710	7.33	0.19
LA-1-16	R	3.8	290	15,710	7.69	0.25
LA-1-23	R	4.0	290	15,790	6.43	0.29
LA-1-24	R	4.0	290	15,890	—	—
LA-1-26	R	4.2	290	16,500	6.98	0.18
LA-1-28	R	4.0	290	14,860	7.17	0.18
LA-1-30	R	4.0	292	16,120	6.48	0.18
LA-1-31	R	4.2	290	16,790	7.69	0.08
LA-1-32	R	3.7	289	16,640	7.12	0.18
LA-2-15	T	4.2	76	19,210	7.51	0.20
LA-2-28	T	4.6	76	20,640	7.93	0.22
LA-2-16	T	3.3	256	12,570	6.37	0.27
LA-2-25	T	3.6	260	13,430	6.48	—
LA-2-2	T	4.0	292	14,070	6.60	0.27
LA-2-3	T	3.3	290	13,860	6.74	0.17

S + S+TMPTMA = 60 percent styrene + 40 percent TMPTMA

6- by 12-inch cylinders, tests at 120 days' age

Specimens dried at 302° F (150° C) prior to impregnation

R = Radiation polymerization

minimum radiation intensity 2.5 by 10⁵ rads/hrminimum radiation dose 3.3 by 10⁶ rads

T = Thermal-catalytic polymerization

catalyst 1 percent benzoyl peroxide

¹ Average of 10 specimens.

Table 25

HIGH-TEMPERATURE MECHANICAL PROPERTIES

DAP plus MMA-impregnated OSW-type Concrete

Specimen number	Polymer load- ing weight percent	Specimen temperature ° F	Compressive strength psi	Modulus of elasticity 10 ⁶ psi	Poisson's ratio	Tensile splitting strength, psi
Survey series						
Control ¹	0	76	8,740	5.22	0.16	559
Control ¹	0	250	7,080	4.54	0.22	451
Control ¹	0	290	7,500	5.06	0.20	475
OSW-5-7	4.9	76	17,610	6.94	0.20	—
OSW-5-4	4.9	76	18,640	6.79	0.18	—
² OSW-5-13	3.2	76	11,480	3.76	0.13	—
OSW-5-15	4.9	256	13,290	6.76	0.27	—
OSW-5-2	4.7	255	13,140	6.46	0.26	—
OSW-5-21	4.4	256	15,360	6.62	0.22	—
OSW-5-12	5.1	293	12,930	6.21	—	—
OSW-5-5	4.7	292	14,210	—	—	—
OSW-5-11	4.1	294	11,640	5.26	0.23	—
OSW-5-18	4.9	76	—	—	—	1,275
OSW-5-8	4.7	76	—	—	—	1,091
² OSW-5-1	3.4	76	—	—	—	626
OSW-5-16	4.9	248	—	—	—	1,043
OSW-5-10	4.7	247	—	—	—	1,079
² OSW-5-6	3.0	247	—	—	—	645
OSW-5-17	4.9	289	—	—	—	1,220
OSW-5-22	4.7	288	—	—	—	995
OSW-5-14	4.5	286	—	—	—	893
Concrete test series						
OSW-7-3	4.9	76	18,571	7.05	0.17	—
OSW-7-5	4.9	76	19,357	6.98	0.19	—
OSW-7-7	4.4	76	18,071	7.30	0.21	—
OSW-7-8	4.6	76	16,857	6.96	0.17	—
OSW-7-9	5.1	76	18,571	6.83	0.20	—
OSW-7-10	4.9	76	17,214	7.04	0.19	—
OSW-7-11	4.6	76	19,286	8.07	0.21	—
OSW-7-14	5.3	76	20,143	7.13	0.22	—
OSW-7-18	4.7	76	17,786	7.06	0.20	—
OSW-7-2	5.1	252	16,071	7.39	0.21	—
OSW-7-12	4.8	250	16,714	6.74	0.26	—
OSW-7-15	4.9	252	16,000	6.42	0.19	—
OSW-7-17	5.3	251	17,143	6.33	0.20	—
OSW-7-21	4.6	249	16,714	6.46	0.15	—
OSW-7-23	4.9	251	16,000	6.86	0.18	—
OSW-7-24	5.1	252	16,286	6.93	0.22	—
OSW-7-28	4.6	254	16,429	6.86	0.31	—
OSW-7-19	5.1	293	15,286	6.03	0.10	—
OSW-7-20	4.8	293	14,857	6.44	0.19	—
OSW-7-22	5.3	294	15,000	6.29	—	—
OSW-7-25	5.1	293	16,500	6.21	0.12	—
OSW-7-26	4.8	292	14,714	—	—	—
OSW-7-27	4.4	294	15,214	5.99	0.27	—

DAP + MMA = 90 percent diallyl phthalate + 10 percent methyl methacrylate

Thermal-chemical polymerization

Catalyst: 5 weight percent tertiary butyl perbenzoate

6- by 12-inch cylinders, tests at 120 days' age

Specimens dried at 302° F (150° C) prior to impregnation

¹ Average of three specimens.² Specimen incompletely impregnated.

Table 26
HIGH-TEMPERATURE MECHANICAL PROPERTIES
Styrene plus TMPTMA impregnated CP-type Concrete

Sample number	Polymer loading, weight percent	Compression tests			Tensile tests		
		Compressive strength, psi	Modulus of elasticity, 10 ⁶ psi	Poisson's ratio	Sample number	Polymer loading, weight percent	Tensile strength, psi
Tests at 70° F							
CP-28	6.5	16,000	6.31	0.22	CP-54	6.5	800
CP-25	5.9	17,750	6.39	0.20	CP-11	7.0	950
CP-14	7.3	17,680	6.33	0.21	CP-13	6.2	980
CP-64	0	4,610	3.56	0.13	CP-87	0	390
CP-92	0	4,500	3.66	0.21	CP-79	0	430
CP-68	0	4,590	3.44	0.17	CP-71	0	340
Tests at 250° F							
CP-30	6.8	14,780	6.47	0.16	CP-20	6.7	1,000
CP-35	6.9	13,930	7.14	0.14	CP-63	6.6	670
CP-53	6.3	16,500	5.45	0.24	CP-4	6.4	1,340
CP-90	0	4,070	3.42	0.09	CP-72	0	340
CP-70	0	3,860	3.04	0.16	CP-66	0	350
CP-77	0	3,930	2.98	0.08	CP-74	0	340
Tests at 290° F							
CP-10	6.4	16,790	6.22	0.28	CP-6	7.1	1,180
CP-15	6.5	15,540	6.14	—	CP-32	5.9	880
CP-3	6.9	16,210	6.05	0.21	CP-22	6.6	980
CP-69	0	3,930	2.90	—	CP-93	0	290
CP-98	0	4,790	3.13	0.06	CP-94	0	290
CP-99	0	4,710	2.84	0.21	CP-95	0	400

S + TMPTMA = 60 percent styrene + 40 percent TMPTMA
Thermal-catalytic polymerization
Specimens conditioned at 302° F (150° C) prior to impregnation
6- by 12-inch cylinders; tests at 120 days' age

Figure 19. Automatic constant-pressure system for high-temperature creep investigations.

Figure 20. Measuring deformations for high-temperature creep studies. Specimens are under load in ovens equipped with creep frames.

monomers for high-temperature applications, under a range of stress conditions at 250° and 290° F.

Compressive strength tests were performed on creep test specimens to aid in the evaluation of compressive stresses used in creep tests (Table 27). Tests were made on single specimens at 76° F and at 1-day and 1-week exposure to a test temperature of 290° F. Results show some scatter, but indicate the strengths are in the range of 15,000 psi, and persist during exposure to a temperature of 290° F for a period of 1 week.

The creep tests are conducted on PIC specimens loaded at 7,000 psi compressive stress. Unimpregnated control specimens are loaded at 2,313 psi. These loadings are about 37 percent of the ultimate strength for each type of concrete. Two loading temperatures are being investigated. In one condition, specimens are heated to 290° F prior to loading and kept at 290° F during the test. In the other condition, specimens are loaded at 76° and then brought up to 290° F during the test. Test results on single specimens are plotted in Figure 21.

Early results indicate a similar type of creep behavior for OSW-type concrete impregnated with 60 percent styrene plus 40 percent TMPTMA and 90 percent DAP plus 10 percent MMA. PIC specimens and unimpregnated concrete specimens loaded at 290° F show a creep of about one-half that of companion specimens loaded at 76° F and then brought up to 290° F. The higher creep of specimens loaded at 76° F and subsequently heated to 290° F is normal since changes in environmental conditions normally increase creep. Both types of PIC show comparable creep for specimens loaded at 76° F, which is about one-tenth the creep for the unimpregnated concrete loaded at 76° F. The creep of 60 percent styrene plus 40 percent TMPTMA specimens loaded at 290° F is about one-tenth that of unimpregnated concrete loaded at 290° F and about one-half that of the 90 percent DAP plus 10 percent MMA specimens loaded at 290° F.

5.2.2. Durability Properties

5.2.2.1. Resistance to Chemical Attack by Hot Brine and Distilled Water

Loop test specimens (6- by 12-inch cylinders and 4- by 4- by 30-inch bars) are exposed to concentrated synthetic sea water at high temperature in an enclosed recirculating system. Six- by twelve-inch cylinders and 4- by 4- by 30-inch bars are tested. Specific gravity, resonant frequency, and ultrasonic pulse velocity are determined on specimens prior to testing. During the loop test, specimens periodically are removed for determination of specific gravity, pulse velocity, compressive strength, Poissons ratio, modulus of elasticity, resonant frequency, and changes in weight and length, and for petrographic examination.

Table 27

HIGH-TEMPERATURE MECHANICAL PROPERTIES OF CREEP TEST SPECIMENS

OSW Concrete Impregnated with S + TMPTMA and DAP + MMA

Cylinder number	Polymer ¹	Polymer loading, weight percent	Test temperature, ° F	Temperature exposure time ²	Compressive strength, psi	Modulus of elasticity, 10 ⁶ psi	Poisson's ratio
28	None	0	76	1 day	6,030	4.27	0.32
29	None	0	281	1 day	6,380	4.09	0.33
30	None	0	287	1 week	6,190	3.51	—
31	None	0	286	1 week	6,930	3.33	—
4	S + TMPTMA	4.3	76	1 day	20,360	7.96	0.25
1	S + TMPTMA	5.1	283	1 day	14,240	6.74	0.22
11	S + TMPTMA	4.6	290	1 week	16,160	6.60	—
14	S + TMPTMA	5.1	290	1 week	15,260	6.74	—
13	DAP + MMA	5.4	76	1 day	17,190	6.78	0.29
15	DAP + MMA	4.9	282	1 day	13,020	6.00	0.25
17	DAP + MMA	5.0	287	1 week	11,420	6.00	—
18	DAP + MMA	5.0	288	1 week	14,910	6.24	—

4-1/2- by 12-inch specimens

¹ None = Unimpregnated control specimens.² Tests conducted after 1-day or 1-week exposure to test temperature.S + TMPTMA = 60 percent styrene + 40 percent TMPTMA
catalyst 1 weight percent benzoyl peroxideDAP + MMA = 90 percent diallyl phthalate + 10 percent methyl methacrylate
catalyst 5 weight percent tertiary butyl perbenzoate

Thermal-catalytic polymerization

60% STYRENE + 40% TMPTMA

90% DIALLYL PHTHALATE + 10% METHYL METHACRYLATE

Plotted values are results on single specimens.

All specimens are under compressive load at 290°F (143°C):

Specimens No. 2, 6, and 8 were preheated to 290°F before loading.

Specimens No. 5, 7, and 19 were loaded at 76°F, and then heated to 290°F.

4-1/2x12-inch specimens, oven dried at 302°F (150°C) prior to impregnation.

Thermal-catalytic polymerization

MMA methyl methacrylate

TMPTMA trimethylolpropane trimethacrylate

DAP diallyl phthalate

Figure 21. High temperature high-stress creep tests on polymer-impregnated OSW-type concrete.

Loop tests include exposure to concentrated synthetic sea water containing 73,000 ppm dissolved salt at 250° F (121° C), and 38,400 ppm dissolved salt at 290° F (143° C). Additional brine loops are being modified and test specimens are being prepared for a further series of tests involving exposure to concentrated brine, exposure to the brine-vapor interface, and vapor at temperatures of 250° and 290° F. Test results will be reported in the Fourth Topical Report.

A facility has been constructed to rapidly evaluate the resistance of concrete and concrete-polymer to attack by hot brine and distilled water. The facility consists of 12 instrumented pressure vessels designed for continuous operation at pressures up to 350 psi. The pressure vessels are commercially available, completely self-contained autoclaves, modified to provide

temperature control rather than pressure control, high/low pressure cutoffs, and a high-temperature cutoff should the pressure shutdown system fail. The steel autoclaves are coated with a baked phenolic lining to eliminate corrosion from oxidation and chloride. A water-cooled condenser has been installed in the cover plates to provide a source of condensate for the condensate drip testing mode. Figure 22 is a photograph of the completed facility.

The inside dimensions of the pressure vessel are 6-1/8-inch diameter by 16 inches high. The system is designed to subject concrete to four environments: completely immersed in water, at the water-vapor interface, in the vapor phase, and subjected to dripping condensate. The test specimens are small concrete cylinders 1-1/2-inch diameter by 3 inches long. The

Figure 22. Autoclaves for measuring chemical resistance of polymer-impregnated concrete to hot brine and distilled water. Specimens can be tested completely immersed in water at the water-vapor interface in the vapor phase, or subjected to a dripping condensate.

specimens for condensate drip are 1-1/2-inch diameter by 5/8 inch high, with the surface exposed to condensate drip initially highly polished. A unique feature of the condensate drip mode is the condensate distribution technique: the entire autoclave is tilted by a series of air pistons and a sequential switching device so that all the condensate falls on one sample for a preselected time, and then is switched to another. A tilted autoclave, pistons, and timing device are shown in Figure 23. Also shown are the condensate coolant

Figure 23. Detail of autoclave for measurements of chemical resistance to hot brine and distilled water. Each autoclave is equipped with an automatic tilting system used for simultaneous testing of six samples in the dripping condensate mode of exposure. The vessels are sequentially tilted to six positions to provide a uniform exposure on each sample.

flowmeter and coolant inlet and outlet thermometers for determining condensate flow rate. This method of operation guarantees uniform exposure of the six drip samples to the condensate.

Pressure vessel loadings currently consist of an unimpregnated concrete series, a series with 60 percent styrene plus 40 percent TMPTMA polymer, and a series with 90 percent DAP plus 10 percent MMA polymer. There are duplicate specimens for each condition. The concrete for the study is based on the CP formulation using 3/8-inch local aggregate and a 28-day cure.

The present test program consists of six autoclaves, three for distilled water and three for brine, at temperatures of 250° F (121° C), 290° F (143° C), and 350° F (177° C). One autoclave is being modified for use to 420° F (216° C), another is being modified to permit operation with different gaseous atmospheres, and four are available for other materials. One of the four is being used to test a similar concrete series cured only 7 days. This test series is in brine at 350° F.

Measurements include weight change, length change, sonic velocity, and initial strengths (of control samples) and final strengths at shutdown.

Only very limited results are available for the 7-day cure series. The most sensitive change appears to have taken place in the condensate drip samples, where, after 1,010 hours (42 days), the weight loss was 2.3 percent for unimpregnated samples, 0.08 percent for the 60 percent styrene plus 40 percent TMPTMA-impregnated samples, and 3.4 percent for the 90 percent DAP plus 10 percent MMA-impregnated samples. The high weight loss from the 90 percent DAP plus 10 percent MMA-impregnated samples was unexpected. The test is continuing.

5.2.2.2. Environmental Tests

The environmental testing program includes several tests in addition to loop tests. Included in the program are an environmental chamber, a weatherometer, high-temperature distilled water exposure, and outdoor weathering. These investigations are in the early stages or are scheduled for testing in the coming year, and test results are not yet available.

An environmental test chamber (Figure 24) has been installed and is being checked out before testing begins.

Figure 24. Environmental test chamber for exposing polymer-impregnated concrete specimens to conditions encountered in a sea water desalting plant.

The chamber simulates the exposure of a reinforced concrete wall to the conditions encountered in a saline water distillation plant. The chamber on one side of the specimen will be half full of synthetic sea water at 290°F (143°C) and 135 psi; the other side will be air at 50 percent relative humidity, 73°F (23°C), and atmospheric pressure. The temperature gradient and moisture movement through the reinforced PIC and control specimens will be measured by thermocouples and humidity gages embedded in the specimen. To date, three reinforced concrete specimens with embedded thermocouples and humidity gage wells have been fabricated. Two of the specimens were used for trial impregnation tests and the third will be tested in the environmental chamber as an unimpregnated control. Testing of PIC specimens will follow completion of tests on the control cylinder. Prior to and immediately after testing, sonic velocity and Schmidt hammer readings will be made to determine the effects on concrete quality. Petrographic examination and comparisons of the compressive strengths of companion cylindrical specimens and the test specimen will also be made.

A 28-inch-diameter by 12-inch-long OSW-type environmental test specimen (weight 720 pounds, including 110 pounds of steel reinforcing rod) and three companion 6-inch-diameter by 12-inch-long cylinders were impregnated with 60 percent styrene plus 40 percent TMPTMA. Saturation of the

environmental specimen with monomer was accomplished within a soak time of 2 hours. A polymer loading of 5.3 percent was obtained. This compared well with the average polymer loading obtained in the cylinders (5.4 percent). Compressive strength measurements made on two 3-inch-diameter by 6-inch-long cores taken from the environmental specimen indicated an average strength of 18,750 psi. Previous work had indicated an average strength of approximately 17,000 psi at 70°F (21°C). Based upon these results, microscopic examination of the core samples, and Schmidt impact hammer readings, it has been concluded that complete and uniform penetration was obtained. This is the largest specimen that has been impregnated to date. The results indicate that walls of a thickness of the order of that required for large desalting vessels can be successfully impregnated.

Preliminary tests are being made of a weatherometer apparatus for accelerated testing of the weathering resistance of PIC. Specimens under test include three controls and three samples of CP-type concrete impregnated with 60 percent styrene plus 40 percent TMPTMA at 6.5 percent polymer loading. The apparatus is an Atlas twin-arc weatherometer (see Figure 25) used primarily for accelerated exposure testing of paints and protective coatings. Carbon arc

Figure 25. Atlas twin-arc weatherometer used for accelerated exposure tests on polymer-impregnated concrete. Specimens are exposed to repeated cycles of simulated sunlight and rain.

lamps and a cold demineralized water spray are used to simulate exposure to sunlight and rain. Samples are exposed to a daily cycle of 20 hours of light with a water spray for 12 minutes each hour. The carbon arc lamps are capable of heating a black sample to 150° F (66° C). The test is generally considered to be about 25 times as severe as outdoor weathering.

Plans are being made for a series of tests on exposure to distilled water at high temperature and exposure to outdoor weathering, with testing scheduled to begin next year.

6. FUNDAMENTAL STUDIES

6.1. STRESS-STRAIN CURVE FOR MMA-IMPREGNATED CONCRETE

The compressive stress-strain curve to failure for MMA-impregnated concrete has been measured, apparently for the first time. The curve and that of an unimpregnated concrete cylinder is shown in Figure 26. The strain reported is the average reading from two strain gages on each sample. Both samples were CP-type concrete, 3-inch diameter by 6 inches long, and the impregnated samples contained 5.4 weight percent MMA. Polymerization was initiated thermal-catalytically at 158° F (70° C) using 2 percent benzoyl peroxide as catalyst.

Figure 26. Compressive stress-strain curve for MMA-impregnated concrete.

The modulus of PIC has been shown to be significantly higher than that of unimpregnated concrete.¹ The most significant fact about these curves is that while the deformation of the unimpregnated concrete sample is continuously nonlinear and irreversible, the deformation of PIC is completely linear up to more than 75 percent of the load at failure, and at failure the

departure from linearity is less than 7.5 percent. It appears that PIC behaves essentially as an elastic solid. This can have an important bearing on structural designing with this material.

The inelasticity of concrete is thought to result from the relatively weak bond between the cement paste and aggregate phases. Under compressive load the failure along the mortar-aggregate interface behaves essentially plastically and produces a gradually curving stress-strain diagram.⁵ The implication for PIC is then that the presence of polymer significantly improves the bond between mortar and aggregate, or at least plays a contributing role. It has also been demonstrated that polymer improves the strength of the cement phase.² If this happens without improved bonding between phases, then an inelastic stress-strain curve should result. The fact that the stress-strain curve is linear for PIC may thus be considered proof of improved bonding between phases.

6.2. MMA-IMPREGNATED LOW-STRENGTH CONCRETE

It has been observed that upon polymer impregnation both CP-type concrete (nominal compressive strength 5,000 psi) and OSW-type concrete (nominal compressive strength 8,500 psi) show strength increases to about 20,000 psi.² Further, according to a model developed for predicting the improvement in the strength of concrete by polymer impregnation,^{6, 7} the incorporation of polymer may increase the strength of a weak concrete to very nearly that of a PIC made from a stronger concrete.

A series of tests was made to investigate the strength improvement from polymer impregnation of low-strength concrete. Three types of concrete were formulated with compressive strengths of 1,670, 2,200, and 4,300 psi. The concretes differed significantly in porosity (15 percent for the strongest and 27 percent for the weakest), and in polymer loading (6.6 weight percent MMA for the strongest and 10.8 weight percent MMA for the weakest). Tests indicated significant improvement in compressive strength for all the concretes. The weakest concrete increased in strength to 15,400 psi, an increase by a factor of nine, while the strongest concrete upon polymer loading increased by a factor of four to 16,700 psi.

Strength data and polymer loadings of the samples are tabulated in Table 28. Initially the strongest and weakest concretes differed in strength by a factor of 2.6. After polymer impregnation, the difference was less than 9 percent, suggesting that for some applications it may be economically advantageous to

Table 28

PROPERTIES OF MMA-IMPREGNATED LOW-STRENGTH CONCRETE

Specimen	W/C	Density	Porosity	Initial strength psi	Polymer loading, weight percent	Polymer loading, volume percent	Final strength psi	Strength increase
CI-5	0.5	2.24	15 percent	4,300	6.6	12.6	16,700	4 times
CI-7	0.7	2.14	21 percent	2,200	8.1	14.9	16,000	7 times
CI-7A	0.7	1.9	27 percent	1,670	10.6	18.3	15,400	9 times
CP concrete ¹	—	—	—	5,000	5 to 7	—	~20,000	4 times
OSW concrete ¹	—	—	—	8,500	4 to 6	—	~21,000	2.5 times

¹ Approximation of values reported in First Topical Report (Reference 1).

begin with a relatively low-strength concrete for making PIC.

The first approach gives:

$$S_c = S_m V_m$$

6.3. MMA-IMPREGNATED INSULATING-TYPE LIGHTWEIGHT CONCRETE

where:

A limited study was made of strength measurements of unimpregnated and MMA-impregnated lightweight concrete to see whether the models developed for the strength of normal concrete and PIC could be applied to this material.⁷

S_c = compressive strength of composite
 S_m = compressive strength of matrix
 V_m = volume of matrix

The study was based on results for perlite concrete, foamed-glass aggregate concrete, and mearlcrete (a foamed portland cement containing no aggregate phase).

The aggregate is assumed to have zero strength. The equation is for an ideal composite system and may give values twice as high as obtained from actual compressive strength tests.

The second approach gives:

$$S_c = S_o \exp (-6.9P)$$

where:

S_o = compressive strength of pore-free material
 P = porosity

The estimated porosities of the concretes varied from about 45 percent for a foamed-glass aggregate concrete to about 80 percent for some perlite concretes. Compressive strengths of unimpregnated specimens ranged from a low of 100 psi for a perlite sample to about 900 psi for a foamed-glass concrete. After impregnation with MMA, strengths increased to a maximum of 7,100 psi for a perlite sample with a polymer weight loading of 95 percent (45 percent by volume). At loadings this high the aggregate particles are as much as half filled with polymer. Estimates of the strengths of unimpregnated specimens were made (1) by assuming that a mixing type of rule applied where the strength of the system is given by the sum of the strengths of the individual phases multiplied by their volume fractions, or (2) by assuming that the strength is simply a function of the total porosity.

S_o is unknown and is given an assumed value. Values of 30,000 psi for S_o were assumed for a hypothetical pore-free perlite and foamed-glass aggregate. For mearlcrete, which is a pure cement, an S_o of 120,000 psi was used.⁶ This equation generally gives results lower than obtained by actual compressive strength tests.

The strengths of the MMA-impregnated lightweight concretes were estimated in the same manner as that described in earlier reports^{5, 6} for cements. A mixing rule type of model is assumed to give the strength of the pore-free system, and then, to account for partial pore filling, the effect of porosity is introduced by using the exponential porosity function described earlier. Estimates of the expected increases and observed increases are given in Table 29. The expectations are averages based on the two estimates of the unimpregnated strength. It should be noted that the actual compressive strength data are based on only one or two samples and may not be representative. The computations were based on estimates, not measurements, of the porosity, and relatively small errors in the porosity for highly porous samples may result in large errors due to the exponential extrapolation. In general the agreement was variable. The perlite results were low by an average factor of three, and the foamed-glass concrete was high by an

average factor of nearly four. The rest of the results were within these limits. Since neither the structural properties of the aggregates or any polymer-aggregate interactions that could result in significantly stronger aggregates were considered, the agreement in this limited study appears reasonable.

6.4. POLYMER-IMPREGNATED HARDENED PORTLAND CEMENT PASTE

The model developed for predicting the strength of concrete indicated that maximum strength would be achieved when the porosity was completely filled with polymer. As observed earlier for water-cured portland cements only 75 to 80 percent of the available pore volume (as measured by water absorption) could be monomer filled.² There was practically no difference between impregnation by vacuum soaking or by boiling in monomer. This observation is not unique for monomers in cement. For example, in measurements

Table 29

STRENGTH IMPROVEMENT FOR LIGHTWEIGHT CONCRETE

Aggregate	Initial porosity (estimate)	Loaded porosity (estimate)	Observed strength improvement factor	Expected strength improvement factor
Pearlyte				
1-4	80 percent	35 percent	17	10
1-6	82 percent	33 percent	25	15
1-8	83 percent	41 percent	35	10
Foamed Glass				
I	81 percent	32 percent	12	12
II	73 percent	28 percent	3	11
III	60 percent	20 percent	12	10
IV	46.5 percent	15 percent	4	9
Mearlcrete	78 percent	39 percent	>6	5

of the surface area of hardened portland cement pastes by various organic vapors it is concluded that while water measures the entire pore structure of the paste, cyclohexane and isopropanol measure larger pores, and nitrogen and methanol are partly admitted into the fine pore structure. Mikhail and Selim⁸ state that the ability to penetrate into pores depends mainly on the size of the molecule. It appears that the limitation in pore filling of hardened water-cured portland cement with monomers is not a permeability problem but is one due to the intrinsic pore structure of cement.

Two approaches have been made to obtain greater monomer and polymer loadings. The first is to use monomers with low shrinkage. For example, MMA monomer fills only 80 percent of the cement pores. On polymerization there is about a 20-percent shrinkage in volume so only about 65 percent of the pores in the cement are filled in the final product. Using a monomer like *t*-butyl styrene, which shrinks only 6 percent on polymerization, results in filling about 77 percent of the pores. Other monomers with low shrinkage include chlorostyrene (11 percent shrinkage) and cyclohexylmethacrylate (15 percent shrinkage). These monomers have been used in experiments with hardened cement pastes. The results show that greater polymer loadings are possible with such monomers, but the increase is less than expected and the strengths do not appear significantly different than those obtained with MMA.

The second approach to higher polymer loadings is to modify the pore structure of the hardened cement paste so that there will be greater pore filling by monomer, and therefore proportionately greater polymer loadings. This can be done quite easily by steam curing or by steam treating water-cured cement pastes. In the steam-curing procedure, samples are water cured for 1 day and then steam cured in a pressure vessel 48 hours at 150 psi at 360° F. The steam treatment consisted of subjecting 14- and 28-day water-cured specimens to 150 psi steam for 48 hours. In both treatments all the porosity as measured by water absorption becomes accessible to monomer, and the desired goal, 100 percent monomer filling, is achieved. With 100 percent monomer filling, polymer loadings of 80 to 90 percent of the total porosity are readily achieved.

The steam treatment does not change the density or total porosity of these samples. Since all the pores were filled with monomer it is concluded that the steam treatment changed the pore size distribution without changing the total porosity. The change in pore distribution may have resulted from either the elimination of small pores and growth of larger ones, or the coalescence of many small pores into fewer large ones. A series of mercury porosimeter measurements

was made on water-cured and steam-treated hardened pastes in an attempt to quantitatively evaluate the changes in pore structure. The results of these measurements indicated two pore structure changes: (1) The steam treatment resulted in a greater fraction of the pores (65 versus 41 percent) greater than 140Å diameter (the experimental limit) and (2) in the pore size range measured, the pores of the steam-treated cement were more cylindrical than those of the water cured (i.e., the pores of the water-cured cement are more "bottled-necked"). Some of the porosity and loading data for these samples are tabulated in Table 30.

Table 30
POROSITY AND POLYMER LOADING

MMA-impregnated Portland Cement Paste (w/c = .35)		
Sample number	5	9
Cure	28 days water	28 days water and steam, 150 psi, 48 hours
Density	1.73 gm/cc	1.74 gm/cc
Porosity	34.8 percent	34.8 percent
Porosity >140Å diameter	14.3 percent	22.6 percent
Monomer loading (volume)	27.4 percent	34.7 percent
Monomer loading (percent of pores filled)	78.7 percent	99 plus percent

The strength model for polymer-impregnated cement paste predicts fully impregnated samples could have compressive strengths from 30,000 to 70,000 psi depending on the size and distribution of stress concentrators. In actual practice, although the polymer loadings for steam-treated samples are appreciably higher than those for water-cured samples, the strengths observed so far were not consistently higher, nor have they exceeded the 30,000-psi level. The highest strengths obtained for steam-treated and steam-cured polymer-impregnated cement were 27,000 psi.

It should be noted that in those tests steam curing or steam treating weakened the cement by about a factor of two. This may be due to the larger average pore size which is more detrimental to strength than small pores. In general the strength improvement factors for steam-cured or steam-treated polymer-impregnated cement paste were about twice as great as those for

Figure 27. Scanning electron photomicrograph of MMA-impregnated portland cement showing fracture surface.

Figure 28. Scanning electron photomicrograph of MMA-impregnated portland cement after polymer has been removed from fracture surface by dissolution in acetone.

water-cured materials, although at higher polymer loadings, the absolute strengths were comparable.

6.5. MICROSCOPIC STUDIES

Samples of polymer-impregnated cement were examined by the scanning electron microscope in an attempt to detect the presence of polymer. Observations were made by first photographing a fractured surface of the specimen and then removing the polymer by dissolution in acetone and rephotographing the same general area. Two examples

are shown in Figures 27 and 28. Figure 27 is the fractured surface of a specimen containing 17 volume percent MMA (55 percent of the pore volume was filled). Figure 28 shows the same material at the same magnification after soaking in acetone in an ultrasonic bath for 66 minutes to dissolve the polymer. The surface without polymer was rougher than the polymer-filled surface but the polymer itself, under these conditions, was not uniquely distinguishable. What is most interesting is that the polymer appears to fill the more or less solid matrix of the cement, rather than just the large pores caused by air entrapment.

7. QUALITY CONTROL

Nondestructive testing of concrete is a well-developed area with a considerable body of literature. However, existing techniques cannot be directly transferred to the nondestructive evaluation of concrete-polymer materials without taking cognisance of the uniqueness of these materials and the objectives of the nondestructive test. In this connection, sonic velocity measurements were discussed in the Second Topical Report.² It was demonstrated there that the strength of PIC could not be estimated from the sonic velocity alone, although there appears to be a relationship between strength and the velocity ratio, that is, the ratio of velocity in PIC to that in unimpregnated concrete. The greater the ratio, the higher the strength. It was also pointed out that absolute velocity measurements may be misleading because of the sensitivity of the velocity to moisture in concrete. Furthermore, monomer could not be distinguished from polymer in concrete because both result in velocity increases.

During the past year, four additional nondestructive techniques have been evaluated.

(1) *Gamma radiography.*—Gamma radiation is attenuated by concrete, and has been used to determine the thickness and density of concrete to accuracies approaching 2 percent for thick slabs.⁹ However, the attenuation by the polymer is so slight that there is no significant decrease in radiation for fully impregnated concrete. In addition, the technique could not distinguish between monomer and polymer.

(2) *Neutron radiography.*—Neutron radiography appears promising because of the high-hydrogen content of the organic polymers. Computations for MMA-impregnated concrete indicate that the neutron attenuation is 50 to 70 percent greater than for unimpregnated concrete. Consequently samples of unimpregnated, partially impregnated, and fully impregnated concrete were examined by neutron radiography. Two neutron radiographs are shown in Figure 29. Polymer-impregnated samples should appear lighter. This was observed in the radiographs, but because of the overlapping and extensive neutron shadows of the stone aggregate, meaningful radiographic density measurements could not be obtained. This technique also could not distinguish monomer from polymer or distinguish water from MMA. However, the method appears to have potential usefulness for observing gross variations in polymer content within a single sample.

(3) *Modulus of elasticity.*—Modulus of elasticity appears promising because it is sensitive to the polymer in PIC and is an overall measurement of the sample. The modulus is nearly twice as large for PIC, is related to compressive strength, and is not sensitive to monomer. However, an evaluation of the reported modulus and strength measurements for PIC indicates extensive scatter in plots of strength versus modulus. The scatter is so great in some cases that samples with compressive strengths of around 20,000 psi have moduli only 20 percent higher than samples with compressive strength of 5,000 psi. The technique may also be somewhat difficult to apply in the field.

(4) *Schmidt hammer.*—The Schmidt impact hammer is a commonly used field instrument for nondestructive testing of concrete. The hammer is essentially a sclerometer, which measures hardness by means of impacting a steel rod against the test surface, and measuring rebound. The rebound number is considered indicative of the quality (i.e. strength, durability) of the concrete; the higher the rebound number, the higher the quality. An approximate correlation between rebound number and strength has been observed. For compressive strengths from 3,000 to 8,000 psi the rebound number varies from about 30 to 50, depending upon hammer orientation. In the First Topical Report¹ PIC gave a 70 percent higher rebound number than did unimpregnated concrete.

The advantages of the Schmidt hammer are: simple and easy operation, the rebound number changes significantly with polymer content, it should not respond to the presence of liquid in the pores, and while it cannot sense the whole volume of the piece under test, it is sensitive to a depth of a few inches.

Initial work with the Schmidt hammer (Type N) established a variation in rebound number with polymer loading. This is shown in Figure 30 for a series of 3-inch-diameter by 6-inch-long CP-type concrete cylinders. Each point is the average of five rebounds, and the results indicate a 65 percent increase in rebound number from unimpregnated to fully impregnated PIC. The increase in rebound number is not linear with loading, but is S-shaped, the function being least sensitive for low- and high-polymer loadings.

A series of OSW-type 6-inch-diameter by 12-inch-long concrete cylinders has been tested with the hammer. These results are shown in Table 31. Unimpregnated samples had rebound numbers around 40, and

Figure 29. Neutron radiograph (negative) of unimpregnated and MMA-impregnated concrete. Polymer produces a lighter image. Polymer content is indicated.

Table 31

SUMMARY OF SCHMIDT HAMMER TESTS

Polymer Impregnated OSW-type Concrete

Designation	Polymer	Number of samples	Polymer loading, weight percent	Rebound number	Deviation from the mean Ave.
OSW-9	—	9	Unloaded	38.9	1.2
OSW-LA 2	—	2	Unloaded	40.6	—
OSW-8	S + TMPTMA	18	4.6	53.4	1.8
OSW-8	DAP + MMA	17	4.55	53.0	1.3
OSW-9	S + TMPTMA	9	4.4	51.9	2.3
OSW-9	DAP + MMA	9	5.3	48.5	1.1

6 x 12-inch specimens

S + TMPTMA—60 percent styrene + 40 percent trimethylolpropane trimethacrylate

DAP + MMA—90 percent diallyl phthalate + 10 percent methyl methacrylate

Figure 30. Schmidt hammer measurements of MMA-impregnated concrete.

impregnated samples averaged between 48 and 53. The reported values are averages of 12 rebounds. The results for the OSW-9 DAP plus MMA group, which had the highest loading (5.3 weight percent) and lowest average rebound number (48.5), are an apparent inconsistency. A low loading with a high-rebound number might be an expected anomaly, indicative of a central core with no polymer.

An extensive series of Schmidt hammer measurements was made on 19 pieces of concrete pipe of various sizes. The increase in rebound number with polymer loading varied from 21 to 36 percent for unreinforced pipe and was independent of wall thickness. This is illustrated in Figure 31. The absolute value of the rebound number increased with increasing wall thickness and appeared to level off between wall

Figure 31. Schmidt hammer tests on polymer-impregnated concrete pipe, percent increase in rebound number versus wall thickness. Open circles indicate reinforced pipe.

thicknesses of 2-1/2 to 3 inches (Figure 32). This probably indicates the limits of the sensitive range of the Schmidt hammer and would suggest that sections greater than 5 to 6 inches can have central regions inaccessible to the Schmidt hammer.

Figure 32. Schmidt hammer tests on polymer-impregnated concrete pipe, rebound number versus wall thickness. Open circles indicate reinforced pipe.

One pipe section was soaked in monomer for only 19 of the total 30-inch length. Schmidt hammer measurements were made, and cores taken along the length of the pipe were analyzed for polymer. Rebound number and polymer content are plotted as a function of pipe length in Figure 33. In general with the increase in polymer loading there is a corresponding increase in rebound number, except the rebound number was not sensitive to very low- or high-polymer contents. This same behavior was noted for 3-inch diameter by 6-inch cylinders (Figure 30).

Figure 33. Schmidt hammer tests on polymer-impregnated concrete pipe, concrete pipe immersed in monomer for 19 of total 30-inch length.

At present no single nondestructive technique completely meets the unique requirements of PIC. However, two or more techniques in conjunction can give meaningful information.

For example, Schmidt hammer measurements on some 6-inch diameter by 12-inch-long cylinders indicated full polymer loading. This would be valid for a depth of about 2 inches. Upon sectioning the cylinders, it was observed that the cylinders were impregnated only to a depth of 2 inches and had an unimpregnated core 2 inches in diameter by 8 inches long. Subsequent sonic velocity measurements on the sectioned cylinder showed a velocity change of only one-third the change expected if the cylinder had been fully impregnated.

Another example, which has not been observed, would be the previous case, but with the central core filled with monomer. In this case the hammer test would indicate maximum loading for about 2 inches and the sonic velocity would be high, therefore reflecting only the difference in velocity between monomer and PIC. For this case, a velocity change of only 10 to 20 percent less than the expected change would be observed. This could suggest a very small unimpregnated core as well as the 2-inch diameter monomer-filled core.

8. DESIGN STUDIES: DESALTING VESSELS

A preliminary survey of vessels for multistage flash distillation units using concrete-polymer materials has been completed. A separate report has been prepared which covers this work in detail.¹⁰ A summary of the effort and of the pertinent results are given in this section.

8.1 DESCRIPTION OF DESIGN BASIS VESSELS

The design basis for the vessel was as follows:

Configuration:	Horizontal cylinder with hemispherical heads
Dimensions:	Inside diameter = 30 feet, length = 90 feet
Design pressure:	50 and 15 psi-g. All vessels to withstand full vacuum
Internal loads:	Distributed load corresponding to 2 feet of brine and supporting fill of light aggregate
Penetrations:	None
Support points:	Every 15 feet along cylinder

The simple configuration with no penetrations allowed relatively straightforward stress calculations, so that the two levels of design pressure and several types of construction could be examined in the study in a reasonable time. The design pressures correspond to the first and second effect pressure ratings for a typical plant. It was assumed that the stage baffles would carry the tube bundle and product tray weights and would transmit these loads to the supports without burdening the vessel shell.

The properties of PIC were assumed to be those currently reported from other areas of the development program. In particular, the compressive strength was taken as 18,000 psi and the tensile strength as 1,600 psi. Variations of up to 15 percent in the assumed compressive and tensile strengths would not affect the results of these design studies. Young's modulus values used were 7.5×10^6 psi in the first set of calculations and 6.5×10^6 psi in the final set. Reference should be made to the concrete-polymer topical reports for other properties.^{1 2}

8.2 TYPES OF VESSEL CONSTRUCTION

The types of vessel construction considered in the design studies were:

- (1) Standard reinforced construction.
- (2) Prestressed construction.
- (3) PIC lined (coating in depth) ordinary concrete construction.
- (4) Ferro-cement construction.
- (5) Premix ferro-concrete-polymer construction.

The first three types of construction were studied in detail using computer analyses. The lack of experience and background data on the last two types limited the present studies to an evaluation of potential performance.

In the design studies reported here, freedom from cracks is achieved by designing the prestressed vessels to have essentially zero tension in the vessel wall and by keeping the concrete tension below 700 psi, or about 45 percent of the tensile strength, in the reinforced vessels. Compressive stresses in the PIC were limited to 8,100 psi, or 45 percent of the compressive strength. For ordinary concrete, in layered vessels, compressive stresses were limited to 2,250 psi and tensile stresses to 113 psi, corresponding to 5,000 psi concrete and using ACI-318-63 allowable values.

Of the five types of construction studied, the reinforced and prestressed types have been used extensively in ordinary concrete tanks and vessels. In the PIC versions, the general layout of reinforcing steel or prestress tendons is similar to that used in conventional concrete vessels. The greater strength of the PIC allows substantially thinner wall sections to be used, and the durability under desalting plant operating conditions should give a long and maintenance-free service life for these vessels. Figure 34 illustrates the assembly of typical prestressed vessels.

The PIC lined vessel uses an interior layer of 1.5 to 2.5 inches of the composite on an ordinary concrete prestressed shell. The area of particular interest for this study was the stress state along the interface of the liner.

The ferro-cement types of construction involve a fine aggregate mortar reinforced by an extended layer of wire mesh. Two types of mortar were considered. The first is a portland cement mortar impregnated with monomer after the mortar has been cured and dried. The second is a mixture of graded fine aggregate and monomer. The second type is referred to as a "premix" mortar because the aggregate and monomer are mixed before being applied to the wire mesh.

Figure 34. Assembly layout for prestressed polymer-impregnated concrete desalting vessel.

8.3 STRESS ANALYSIS

Vessel stress determinations were made with the use of two computer codes, the R-Z code (BNL-ME-21) and the R- θ code (BNL-ME-23). The R-Z code is a finite element routine designed to evaluate the stresses in axially symmetric, heterogeneous structures. As such, it is used in these analyses to determine the variation of vessel cross section averaged stresses with length, as well as the Z components of the vessel stresses. The R- θ code, also a finite element routine, is designed to evaluate the stresses in any two-dimensional, heterogeneous structure. It was used in these analyses to determine the variations in the R and θ components of the vessel stresses at a typical vessel cross section. Figures 35 and 36 show typical nodal point grids used in conjunction with the codes. In the codes, stresses are evaluated at each nodal point.

Two limitations are imposed on the analyses by the nature of the computer codes. First, since only axially symmetric loadings are considered in the R-Z runs, the asymmetric effects of the vessel weight, vessel internal loads, and support reactions are not determined. The effects of weight, internal loads, and supports are included in the R- θ runs. This defect in the analysis is not serious at this preliminary design stage, in view of

the relatively close spacing of the supports (half the vessel diameter). Secondly, since the codes are not specifically designed to consider stability effects, the stability or buckling characteristics of each structure must be separately evaluated. A consideration of the buckling problem is included in informal report¹⁰ BNL-15138, along with a discussion of the effect of discrete supports on the axial profiles. In addition to the limitations imposed by the capability of the codes, there are several other limitations in this survey. The head configuration of the design basis vessel must be altered to suit the various types of construction. It was not considered worth while in this preliminary study to develop these approaches, since the head design will depend strongly on the penetration configurations for the tube bundles, product water pipes, and brine ducts. At a later stage of the design effort, when penetration details are considered, the head design can be corrected. Consequently, in the present studies, if overstressing occurred only in the head, it was not considered cause for rejection of a given design.

The effects of temperature gradients, penetrations, localized loadings, and tendon and/or concrete creep were not evaluated, since they lay outside the scope of the study. Nor was any serious effort devoted to the problems of physical arrangements; tendon layout,

Figure 35. R- θ code finite element grid, five nodes in radial direction.

optimum spacing, etc. In short, the results given here provide a good comparison of the basic vessel shells when continuously supported and free of localized effects.

8.4 SUMMARY OF CASES STUDIED

In all, 13 vessel designs were evaluated in the survey. For the higher pressure (50 psi-g) the designs evaluated included:

- (1) Wall thickness, 4 inches, prestressed construction, tendon stress $\approx 150,000$ and $\approx 180,000$ psi.

Figure 36. R-Z code finite element grid, five nodes in radial direction.

- (2) Wall thickness, 6 inches, prestressed construction, tendon stress $\approx 180,000$ psi.

- (3) Layered wall, 6 inches, prestressed construction, tendon stress $\approx 150,000$ and $\approx 180,000$ psi.

- (4) Wall thickness, 12 and 15 inches, reinforced construction.

- (5) Layered wall, 15 inches, reinforced construction.

For the lower pressure (15 psi-g) the designs evaluated were:

- (1) Wall thickness, 4 inches, prestressed construction, tendon stress $\approx 100,000$ psi.
- (2) Wall thickness 4, 5, and 6 inches, reinforced construction.
- (3) Layered wall, 6 inches, reinforced construction.

Initially, a high level of tendon prestress was utilized in the prestressed vessel designs. The resulting stress profiles were strongly compressive with little variation through the vessel walls. Accordingly a reduction of tendon prestress was investigated in the final runs, the reduction improving the stability characteristics of the vessels. Both 4- and 6-inch wall prestressed designs were carried through, the 4 inch being preferred if its stability and handling characteristics can be shown to be acceptable. A 12-inch wall rebar vessel initially investigated for the high-pressure design was found, as were the 4- and 5-inch wall rebar vessels for the low-pressure design, to be overstressed, which necessitated revisions to 15- and 6-inch walls, respectively. Each design found adequate was re-evaluated in a layered construction option.

Tables 32 and 33 summarize the results of the computer runs on the 13 vessels studied. The stress values given are all principal stresses. The columns marked "max" stress are the largest tensile or the

smallest compressive stress occurring in the cylinder section or head section of the vessel. For the prestressed vessels, there tends to be no positive (tensile) stress. Where two values are given in an entry, the vessel is of layered construction and the first value refers to the PIC lining and the second to the ordinary concrete wall. The columns marked "min" stress are the largest compressive stresses occurring anywhere in the vessel cylinder or head.

Figures 37 and 38 show typical spacial distribution of the principal stresses, in this case for the 4-inch wall, prestressed, 50 psi-g vessel. The maximum and minimum principal stresses at the inside and outside surfaces of the vessel are given at various points along the vessel shell. The principal stresses at nodal points within the vessel wall fall between the values shown for the inner and outer surfaces.

8.5 CONCLUSIONS

The conclusions drawn from these design studies are discussed in the following sections. Several general conclusions, applicable to all the types of construction, were noted previously.

- (1) *Standard reinforced construction.*—The reinforced construction vessels are feasible in both the 50 and 15 psi-g design pressure ranges. The

Figure 37. Desalting vessel design studies, typical spacial distribution of principal stresses for run 26.

Figure 38. Desalting vessel design studies, typical spacial distribution of principal stresses for run 27.

substantial wall thicknesses required, particularly at the higher design pressure impose an economic penalty on this type of construction, and prestressed construction is expected to be less expensive. The thicker walls may also offer some difficulties in obtaining complete impregnation. Reinforced construction appears to require an as yet undeveloped technique for in-place impregnation of the complete vessel.

Because the reinforced vessel wall is in tension at operating conditions for the 50 and 15 psi-g design pressures the necessary freedom from cracking is less assured than in the prestressed vessels. The tension forces make the reinforced vessel susceptible to flaw-induced stress concentrations. If PIC is to some degree elasto-plastic, the effect of flaws will be minimized by localized yielding and stress redistribution; if it is elasto-brittle, cracking may occur.

It is concluded that reinforced construction with PIC is unlikely to be as economical as prestressed

construction in the 50 and 15 psi-g design pressure vessels, but may offer an economic advantage for vacuum stage vessels.

Reinforced construction with a premixed PC has the potential of overcoming the difficulties noted above. Construction problems would be essentially eliminated with use of a premixed material. The economic disadvantage of the thicker shell required might also disappear if the modulus of elasticity in tension of the premixed material was sufficiently small. The large wall thickness of the present reinforced design results from the need to keep the PIC tension stress at an acceptably small fraction of the tensile strength. With a steel/concrete modulus ratio of 30:7, or about 4.5 for PIC, and an allowable PIC tension stress of 700 psi, the reinforcing steel stress is only 3,000 psi. Thus, the steel is inefficiently used. If the premixed material had a tension modulus in the range of the polymer material itself, say 500,000 psi, the modulus ratio would be 60 and the reinforcing steel strength could be fully utilized. The results would be a substantial reduction in the required wall thickness.

(2) *Prestressed construction.* The prestressed vessels are the favored choice for the 50 and 15 psi-g design pressures. Quite thin walls (4 inches) can be used, and in fact the lower limit on wall thickness is set by shell stability considerations rather than stress limitations. With the vessel walls in compression under the prestressing load, there is ample assurance of crack free operation.

The prestressed vessel can be constructed with use of existing impregnation techniques. As shown in Figure 34, the vessel can be made up of precast and impregnated cylindrical sections of a length compatible with the desired stage wall spacing. The cylindrical sections would be field assembled, with stage walls sandwiched between them, and the junctions sealed with a castable polymer-sand grout. Cylindrical sections of a prestressed vessel would have the radial tendons placed and tensioned prior to assembly. Axial tendons would be inserted and tensioned at the time of assembly. Each section would be supported on a footing designed to permit axial thermal expansion. The vessel can be of any desired length, depending on the appropriate number of cylindrical sections.

(3) *Concrete-polymer lined (coating in depth) ordinary concrete construction.* The PIC lined (layered) vessels were studied in both reinforced and prestressed versions. The reinforced layered vessels suffer from the low-tensile strength of the ordinary concrete portion of the vessel wall. The required

Table 32

DESALINATION VESSEL DESIGN STUDIES

Summary of Results of Computer Runs on 13 Vessels Studied (Runs 1 through 25)

Run no.	Type	Thick- ness in.	Impregnation full partial	Prestress			Reinforcement		Pressure		Vessel Principle Stresses			
				level	radial	long	in. ² /in.	in. ² /rad	internal psig	external psig	Cylinder stress		Head stress	
				equiv. psi	in. ² /in.	in. ² /rad					max psi	min psi	max psi	min psi
1	R-Z	4	X						50	125	-56	-3531	-67	-2555
2	R-Z	4	X						0	125	4	-5880	-28	-4175
3	R-Z	6	X						50	125	-45	-2438	-59	-1766
4	R-Z	6	X						0	125	8	-3978	-21	-2860
5	R-Z	6		2 in. i.d.					50	125	-46/-93	-3766/-1744	-58/-101	-2706/-1220
6	R-Z	6		2 in. i.d.					0	125	6/-71	-6156/-2643	-17/-79	-4388/-1971
7	R-Z	4	X		125	.1958	11.75		50	0	-57	-3876	2672	-6989
8	R-Z	4	X		125	.1958	11.75		0	0	2	-5865	2319	-7598
9	R-Z	6	X		125	.1958	11.75	.1958	50	0	13	-2497	2008	-5244
10	R-Z	6	X		125	.1958	11.75	.1958	0	0	33	-3744	1700	-5704
11	R-Z	6		2 in. i.d.	125	.1958	11.75	.1958	50	0	-56/10	-3467/-1608	2434/1227	-5075/-3437
12	R-Z	6		2 in. i.d.	125	.1958	11.75	.1958	0	0	-9/16	-5207/-2407	2008/1110	-5985/-3734
13	R-Z	4	X		90	.1958	11.75		50	0	-52	-2276	1441	-6388
14	R-θ	4	X		90	.1958	11.75		50	0	13	-2160		
15	R-θ	4	X		90	.1958	11.75		-15	0	5	-4651		
16	R-θ	6	X		125	.1958	11.75	.1958	50	0	76	-2446		
17	R-θ	6	X		90	.1958	11.75	.1958	50	0	106	-1439		
18	R-θ	6	X		90	.1958	11.75	.1958	-15	0	32	-2951		
19	R-θ	12	X					.750	50	0	1479	-231		
20	R-θ	15	X					1.000	50	0	1424	-287		
21	R-θ	15	X					1.000	-15	0	781	-621		
22	R-Z	15	X					1.000	50	0	506	-46	377	-45
23	R-θ	5	X					.375	15	0	1393	-446		
24	R-θ	5	X					.375	-15	0	972	-790		
25	R-Z	5	X					.375	15	0	437	-14	327	-28

Note: E (modulus of elasticity) and
 μ (Poisson's ratio) for all runs
 are 7.5×10^6 and .2, respectively.

Table 33

DESALINATION VESSEL DESIGN STUDIES

Summary of Results of Computer Runs on 13 Vessels Studied (Runs 26 through 46)

Run	Type	Thickness		Tendons				Reinforcement		Pressure	Vessel Principle Stresses			
		Total Impreg.		Hoop		Longitudinal		Hoop	Long.		Cylinder		Head	
		in.	in.	Area in. ² /ft	Stress psi×10 ³	Area in. ² /ft	Stress psi×10 ³	Area in. ² /ft	Area in. ² /ft		Maximum psi	Minimum psi	Maximum psi	Minimum psi
26	R-Z	4	4	1.175	150.3	0.526	151.3	-	-	50	-51	-1454	502	-970
27	R-0	4	4	1.175	150.4	-	-	-	-	50	142	-1478	-	-
28	R-Z	4	4	1.175	139.4	0.526	147.3	-	-	-15	4	-4182	202	-2338
29	R-0	4	4	1.175	138.8	-	-	-	-	-15	-10	-4085	-	-
30	R-0	4	4	1.175	97.8	-	-	-	-	15	105	-1743	-	-
31	R-Z	5	2	1.175	150.6	0.526	151.4	-	-	50	-56/-45*	-1404/-754*	474/240*	-970/-445*
32	R-0	6	2	1.175	150.6	-	-	-	-	50	46/-64	-1396/-1016	-	-
33	R-Z	6	2	1.175	140.1	0.526	147.4	-	-	-15	2/-38	-4052/-2163	143/37	-2279/-1141
34	R-0	6	2	1.175	139.7	-	-	-	-	-15	-8/-39	-3976/-3257	-	-
35	R-Z	15	15	-	-	-	-	12.0	6.0	50	491	-48	313	-47
36	R-0	15	15	-	-	-	-	12.0	-	50	609	-266	-	-
37	R-Z	15	15	-	-	-	-	12.0	6.0	-15	0	-160	-1	-107
38	R-0	15	15	-	-	-	-	12.0	-	-15	18	-273	-	-
39	R-Z	15	2.5	-	-	-	-	12.0	6.0	50	703/370	-48/-33	470/223	-45/-30
40	R-0	15	2.5	-	-	-	-	12.0	-	50	849/406	-249/-80	-	-
41	R-Z	6	6	-	-	-	-	3.95	2.4	15	376	-15	220	-14
42	R-0	6	6	-	-	-	-	3.95	-	15	461	-155	-	-
43	R-Z	6	6	-	-	-	-	3.95	2.4	-15	0	-389	-1	-234
44	R-0	6	6	-	-	-	-	3.95	-	-15	15	-431	-	-
45	R-Z	6	1.5	-	-	-	-	3.95	2.4	15	525/279	-16/-8	328/168	-13/-6
46	R-0	6	1.5	-	-	-	-	3.95	-	15	635/308	-28/-139	-	-

* Polymer concrete stress/ordinary concrete stress.

NOTE: Modulus of elasticity (psi), Poisson's ratio - polymer concrete, 6.5×10^6 , 0.2
 concrete, 3.5×10^6 , 0.2
 steel, 30.0×10^6 , 0.3

wall thickness is thus much greater than in the case of PIC alone, to keep the tensile strains at the liner interface low. The reinforced layered construction also presents the same difficulties as reinforced PIC vessels.

The possible utility of reinforced layered construction probably depends on new developments in techniques of in-place, partial-depth impregnation. If a simple, easy, and inexpensive method for in-place partial impregnation were available, then the large wall thickness required for ordinary concrete might be offset by the advantages of using well-understood, conventional reinforced concrete construction practices.

It is concluded that the reinforced layered type of construction should be dropped from the program for the time being.

The prestressed layered vessels have the advantage that the wall materials are in compression at all operating conditions. The tensile weakness of the ordinary concrete portion of the vessel shell is thus not an important disadvantage in prestressed layered construction. The ordinary concrete is also weaker in compression than PIC, and a greater wall thickness is required for layered construction than for PIC material alone. The increase in thickness is not as great as might be expected from the compressive strength ratio, since the minimum wall thickness of the PIC vessel is set by shell stability considerations.

For both the reinforced and prestressed layered construction types, the differences in the modulus of elasticity between the materials cause a sharp discontinuity in stress at the layer interface. Presumably, if the stresses in each material are below the allowable values, the structure is sound. But this area should be investigated experimentally to establish the shear, tensile, and compressive behavior of the interface region.

(4) Ferro-cement construction and (5) Premix ferro-concrete-polymer construction.—The ferro-cement constructions were thought to be of interest because of possible improvements in strength and resistance to cracking over the standard reinforced construction. However, remarkably little is known about this form of concrete, and, in particular, there are no accepted theories on the action of the distributed reinforcing. Some authors have commented on synergistic effects, either

postulated or thought to have been observed, but there is at present no substantive basis for assuming better properties for the ferro-cement material than for conventional reinforced material. It would be worth while to supplement the few measurements made thus far on samples of polymer-impregnated ferro-cement to establish whether or not such effects exist.

In view of the lack of basic information on ferro-cement, no design calculations have been made specifically for these construction types. The general characteristics of the standard reinforced construction vessel must be assumed to apply to the ferro-cement construction, for lack of better information.

As is the case for the standard reinforced construction vessels, the premixed material ferro-cement version is more interesting than the impregnated portland cement version. If there is any useful change in properties due to the finely distributed reinforcement, it is most likely to occur with the premix-type material. The material program for ferro-cement, then, should concentrate on the premix types of concrete-polymer.

8.6 ADDITIONAL MATERIALS DATA REQUIRED

One purpose of this survey was to delineate areas in which additional data on concrete-polymer materials are needed for detailed structural designs. Although a thorough knowledge of material properties is required for all types of construction, the study of each type emphasized the importance of certain properties or characteristics.

The basic list of material parameters that are needed for elastic analyses of desalting vessels is as follows:

- (1) Modulus of elasticity.
- (2) Poisson's ratio.
- (3) Density.
- (4) Thermal conductivity.
- (5) Coefficient of thermal expansion.
- (6) Specific heat.

The variation of these parameters with temperature, if appreciable, should be known. Any variation in

Young's modulus or Poisson's ratio with the sign (i.e., tension or compression) of the total stress state should also be known.

To carry out design details such as reinforcing arrangements, tendon anchorages, and section dimensions, a further set of material properties is required:

- (7) Tensile and compressive strengths.
- (8) Shear strength.
- (9) Bond strength.
- (10) Bearing strength.

Again, the variation with temperature should be known.

Finally, to assess the adequacy of a design, the following must be known:

- (11) Stress-strain characteristics in tension and compression as a function of temperature.
- (12) Creep characteristics in tension and compression at operating temperature.

(13) Fatigue behavior.

(14) Early life effects, such as shrinkage.

(15) Durability properties and compatibility with the operating condition environment.

At this point in the concrete-polymer materials development program some data are available on most of these items. The outstanding need in connection with structural design at present is for stress-strain data on both tension and compression as a function of temperature for the reference PIC materials and for PC and PCC materials. These items are now included in the program plan.

As the program goes on and the list of possible materials is narrowed, preparation procedures must be standardized and a statistically significant body of data developed for each of the items noted above. The nominal values of each of the parameters can then be set for the "standard preparation" material, and the expected range of variation in each parameter determined. The variation ranges are important to the structural designer so that he can account in advance for off-nominal material conditions.

9. APPLICATIONS DEVELOPMENT

9.1. PIPE APPLICATIONS

9.1.1. Concrete Draintile

The field and laboratory test program to evaluate PIC draintile continued during the year. Field tests are being conducted at the Westlands Experimental Drainage Plot, Tranquillity, California. The test program consists of three classes of draintile: (1) 30 lengths of poor quality (PQ), made with Type I cement, not acceptable for use in USBR projects; (2) 30 lengths of average quality (AQ), made with Type II cement, meeting the requirements of ASTM Designation: C 118; and (3) 30 lengths of high quality (HQ), made with Type V cement and fly ash, meeting USBR special requirements for exposure to high sulfate environments.

From each class of pipe, 10 lengths were fully impregnated with MMA, 10 were fully impregnated with epoxy styrene, and the remainder served as controls. Average polymer content of draintile for each experimental condition is as follows:

Class of pipe	Monomer	Polymer loading, percent
PQ	MMA	6.78
AQ	MMA	6.38
HQ	MMA	5.77
PQ	ES	9.08
AQ	ES	7.69
HQ	ES	4.97

PQ = Poor quality MMA = methyl
AQ = Average quality methacrylate
HQ = High quality ES = epoxy styrene

One length of draintile representative of each experimental condition, a total of nine sections, is continuing to undergo laboratory testing at the USBR in Denver. The 81 remaining sections are installed in drainage lines at Westlands. Sulfate concentrations in this area are extremely high; analyses indicate concentrations ranging up to 7.5 percent in the soil and 5.9 percent in water taken from the drainage lines. Pipe specimens will be removed periodically for evaluation of their relative resistance to attack by sulfates.

9.1.1.1. Laboratory Test Data

Tests performed include three-edge bearing strengths, water absorption, and resistance to sulfate attack in both the accelerated sulfate test and the continuous soaking test in 10-percent sodium sulfate solution.

Results of three-edge bearing tests are shown in Table 34. Increases in strength resulting from impregnation with MMA are 73 percent for PQ pipe, 121 percent for AQ pipe, and 90 percent for HQ pipe. With epoxy styrene, increases in strength are 281, 143, and 100 percent, with PQ, AQ, and HQ pipe, respectively.

Table 35 shows the results of the water absorption tests. The epoxy-styrene-impregnated PQ, AQ, and HQ samples exhibited less absorption than the corresponding MMA-impregnated samples, with the PQ epoxy-styrene specimens absorbing the least water of all specimens tested. This may be due to the greater porosity and correspondingly higher polymer loading achieved with the PQ epoxy-styrene specimens. Low-water absorption appears to be a function of increased polymer loading, except that with HQ specimens it appears to result from the combined effects of polymer impregnation and high initial concrete density. Because of the higher initial concrete density, the polymer loading of these specimens is predictably less than that of the PQ or AQ specimens.

Laboratory sulfate-resistance tests are reported in Table 36. Results are given for the 10-percent sodium sulfate immersion test and the accelerated (1-day wetting and drying cycle) 2.1-percent sodium sulfate test. Sulfate resistance of unimpregnated control specimens, in both the immersion test and the accelerated test, is related to the quality of the tile. The PQ tile is showing some minor disintegration and a few small cracks. The AQ tile is in better condition, but shows a few very small and tight cracks. The HQ tile is in excellent condition.

Impregnation with epoxy styrene appears to improve sulfate resistance of AQ and PQ tile (the test has not run long enough to evaluate the effect on HQ tile).

Draintile impregnated with MMA has shown some unexpected results, showing in general somewhat lesser sulfate resistance than either the epoxy-styrene-impregnated or the unimpregnated control specimens. However, in the accelerated test, MMA-impregnated AQ tile showed excellent sulfate resistance. The MMA impregnated PQ and HQ tiles in the accelerated test, and all the MMA-impregnated tiles in the immersion test all showed more expansion than either the epoxy-styrene-impregnated or the unimpregnated control specimens. Additional tests will be performed to confirm or provide an explanation for these results.

9.1.1.2. Field Test Data

The 81 sections of draintile at the Westlands Experimental Drainage Plot were separated into 27

Table 34

THREE-EDGE BEARING STRENGTH

Polymer-impregnated Concrete Draintile

Specimen number	Class of pipe	Monomer	Polymer loading, percent	Wall thickness, inches		Ultimate load, pounds	Difference, percent ¹	
				Minimum	Maximum			
A1	PQ	None	0	0.82	0.99	2,480)	2,380	—
B1	PQ	None	0	0.78	0.93	2,280)		—
1-5	PQ	MMA	7.4	0.84	0.99	4,120		73
1-12	PQ	ES	9.5	0.80	0.95	9,060		281
A2	AQ	None	0	0.80	1.01	2,900)	2,970	—
B2	AQ	None	0	0.85	1.02	3,040)		—
2-3	AQ	MMA	7.1	0.85	1.00	6,560		121
2-13	AQ	ES	8.6	0.79	0.94	7,220		143
A3	HQ	None	0	0.80	1.05	3,320)	3,160	—
B3	HQ	None	0	0.83	0.93	3,000)		—
3-2	HQ	MMA	6.6	0.91	0.99	6,000		90
3-19	HQ	ES	5.8	0.79	0.94	6,320		100

Specimen size: 4-inch inside diameter by 12 inches long

PQ = poor quality

AQ = average quality

HQ = high quality

MMA = methyl methacrylate

ES = epoxy-styrene

$$^1 \text{Difference, percent} = \frac{\text{test} - \text{control}}{\text{control}} \times 100$$

lengths each and placed in three separate drainage lines. Placement of draintile was based on a statistical random arrangement, with consideration given to different polymer contents of the pipes. The arrangement was such that three groups of pipe, each group representing all nine pipe treatments, were placed in each of the three drainage lines. This will enable statistically randomly arranged groups of pipe representing all treatments to be removed periodically for evaluation of their relative resistance to sulfate attack. The first such removal is scheduled for 1971, following 2 years in service.

9.1.2. Sewer and Culvert Pipe;
*Joint Program with American
Concrete Pipe Association*

The testing and evaluation of concrete and PIC pipe was a cooperative effort between the ACPA, AEC/DID, BNL, and USBR. Forty specimens were manufactured by the Gifford-Hill Pipe Company, Dallas, Texas, in accordance with ASTM Designations: C 14 and C 76. Twenty specimens of concrete pipe were supplied to

USBR for controls and an equal number to BNL for impregnation.

The nomenclature used for specimen designation was set up in the following manner for the pipe:

a. First character

(1) A—thin wall (1-3/8 and 2 inches)

(2) B—thick wall (2-1/2 and 3 inches)

(3) R—reinforced pipe (2 inches)

b. Second character (two digits)

(1) 12—12-inch internal diameter

(2) 24—24-inch internal diameter

c. Third character—specimen number in the group (1 through 8)

Table 35

WATER ABSORPTION TEST

Polymer-impregnated Concrete Drintile

Specimen number	Class of pipe	Monomer	Polymer loading percent	Absorption percent		Difference percent
1-12G	PQ	ES	9.5	0.58	0.47	-95
1-12H	PQ	ES	9.5	0.52		
1-12I	PQ	ES	9.5	0.30		
1-5G	PQ	MMA	7.4	2.02	2.46	-76
1-5H	PQ	MMA	7.4	3.68		
1-5I	PQ	MMA	7.4	1.68		
A1-G	PQ	None	0	10.32	10.10	0
A1-H	PQ	None	0	10.33		
A1-I	PQ	None	0	9.38		
B1-G	PQ	None	0	9.67		
B1-H	PQ	None	0	9.96		
B1-I	PQ	None	0	10.91		
2-13G	AQ	ES	8.6	1.69	1.35	-86
2-13H	AQ	ES	8.6	1.68		
2-13I	AQ	ES	8.6	0.68		
2-3G	AQ	MMA	7.1	1.50	1.42	-85
2-3H	AQ	MMA	7.1	1.60		
2-3I	AQ	MMA	7.1	1.17		
A2-G	AQ	None	0	9.04	9.63	0
A2-H	AQ	None	0	9.12		
A2-I	AQ	None	0	10.46		
B2-G	AQ	None	0	8.61		
B2-H	AQ	None	0	10.03		
B2-I	AQ	None	0	10.53		
3-19G	HQ	ES	5.8	0.42	0.60	-93
3-19H	HQ	ES	5.8	0.76		
3-19I	HQ	ES	5.8	0.63		
3-2G	HQ	MMA	6.6	1.63	2.24	-73
3-2H	HQ	MMA	6.6	3.60		
3-2I	HQ	MMA	6.6	1.49		
A3-G	HQ	None	0	5.59	8.24	0
A3-H	HQ	None	0	7.78		
A3-I	HQ	None	0	8.51		
B3-G	HQ	None	0	8.53		
B3-H	HQ	None	0	9.83		
B3-I	HQ	None	0	9.19		

PQ = Poor quality

AQ = Average quality

HQ = High quality

MMA = Methyl methacrylate

ES = Epoxy-styrene

$$\text{Difference, percent} = \frac{\text{Test-control}}{\text{control}} \times 100$$

Table 36

RESISTANCE TO CHEMICAL ATTACK BY SULFATES

Polymer-impregnated Concrete Draitile

Class of pipe	Monomer	Polymer loading percent	Sample number	Test days	Immersion test expansion percent ¹	Accelerated test	
						Expansion percent ¹	Specimen condition ⁴
PQ	None	--	1	120	0.063	0.139	Fair, 1
AQ	None	—	2	120	0.052	0.137	Good, 1
HQ	None	—	3	120	0.048	0.044	Excellent
PQ	MMA	7.4	1-5	120	0.271	² 0.264	Fair, 2
PQ	ES	9.5	1-12	120	0.049	0.064	Excellent
AQ	MMA	7.1	2-3	120	0.153	0.012	Excellent
AQ	ES	8.6	2-13	120	0.072	0.106	Excellent
HQ	MMA	6.6	3-2	120	0.145	³ 0.224	Fair, 2
HQ	ES	5.8	3-19	120	0.037	0.054	Excellent

PQ = Poor quality

AQ = Average quality

HQ = High quality

MMA = Methyl methacrylate

ES = Epoxy-styrene

Specimens dried in oven prior to impregnation

Radiation polymerization

¹ Values for control are the average net expansion for six specimens. Values for polymer-impregnated concrete are the average net expansion for three specimens.

² One of three specimens in this set developed a large crack at 28 days. Not included in average.

³ One of three specimens in this set has high expansion due to a single large crack (0.316 percent). Other two specimens show expansion, but are not cracked. All three included in average expansion.

⁴ 1 = Fine cracks

2 = Large crack.

Each specimen was measured for internal diameter, wall thickness, and length. These values are shown in Tables 37, 38, and 39, respectively. The positions

for measuring internal diameter and wall thickness are shown in Figure 39 and 40.

Table 37

MEASUREMENT OF INTERNAL DIAMETER
ACPA PIPE

Specimen	Inside diameter, inches				Average
	1	2	3	4	
A-12-1	11-15/16	11-15/16	11-15/16	11-15/16	11-15/16
A-12-3	12	12	12	12	12
A-12-4	12	12	12	12-1/16	12
A-12-5	12	11-15/16	11-15/15	11-15/16	11-15/16
A-12-6	12	12-1/16	12-1/16	12	12
A-12-7	12	11-15/16	12	12	12
A-12-8	12	11-15/16	11-15/16	11-15/16	11-15/16
B-12-2	12-1/16	12-1/16	12-1/16	12-1/16	12-1/16
B-12-3	12-1/16	12-1/16	12-1/16	12	12-1/16
B-12-4	12	12-1/16	12-1/16	12	12
B-12-5	12	12-1/16	12-1/16	12-1/16	12-1/16
B-12-6	12	12	12	12-1/16	12
B-12-7	12	12	12	11-15/16	12
B-12-8	12-1/16	12-1/16	12	12-1/8	12-1/16
A-24-1	24-3/16	24-1/4	24-1/16	24-1/8	24-1/8
A-24-2	23-15/16	23-7/8	23-7/8	23-13/16	23-7/8
A-24-3	23-15/16	23-7/8	23-7/8	23-13/16	23-7/8
A-24-4	23-13/16	23-7/8	24	23-15/16	23-7/8
A-24-5	24-1/4	24-3/8	24	24-3/16	24-3/16
A-24-6	24	24-5/16	24-5/16	24-1/4	24-3/16
A-24-7	24-3/16	24-3/16	24-1/8	24-1/4	24-3/16
A-24-8	24-1/8	24-3/16	24-1/4	24-5/16	24-1/4
B-24-2	24-1/8	24-3/8	24-1/4	24-1/8	24-1/4
B-24-3	24-1/8	24-3/16	24-3/16	24-3/8	24-3/16
B-24-4	24	24-7/16	24-3/8	24	24-3/16
B-24-5	24-1/8	24	24-1/8	24-1/4	24-1/8
B-24-6	24-1/8	24-3/16	24-1/8	24-3/8	24-1/16
B-24-7	24-1/8	24-1/8	24-1/8	24-1/8	24-1/8
B-24-8	24-1/4	24-1/8	24-3/16	24-1/8	24-3/16
R-24-1	24-3/8	24-3/16	24-1/16	24-1/4	24-1/4
R-24-2	24-5/16	24-1/4	24-3/16	24-3/16	24-1/4
R-24-3	24-1/16	24-3/16	24-1/4	24-5/16	24-3/16
R-24-4	24-1/8	24-1/4	24-1/4	24-1/4	24-1/4
R-24-5	24-1/16	24-3/16	24-1/4	24-1/8	24-1/8
R-24-6	24-5/16	24-5/16	24-3/16	24-1/4	24-1/4
R-24-7	24-1/8	24-3/16	24-3/16	24-5/16	24-3/16
R-24-8	24-5/16	24-3/16	24-1/8	24-3/16	24-3/16

Table 38

**MEASUREMENT OF WALL THICKNESS
ACPA PIPE**

Specimen	Wall thickness, inches				Average
	a	b	c	d	
A-12-1	1-1/2	1-1/2	1-9/16	1-5/8	1-1/2
A-12-3	1-1/2	1-1/2	1-9/16	1-1/2	1-1/2
A-12-4	1-1/2	1-3/8	1-3/8	1-1/2	1-7/16
A-12-5	1-1/2	1-9/16	1-9/16	1-1/2	1-1/2
A-12-6	1-3/8	1-7/16	1-9/16	1-7/16	1-7/16
A-12-7	1-7/16	1-1/2	1-1/2	1-1/2	1-1/2
A-12-8	1-1/2	1-9/16	1-9/16	1-9/16	1-9/16
B-12-2	2-1/2	2-9/16	2-1/2	2-7/16	2-1/2
B-12-3	2-1/2	2-5/8	2-1/2	2-7/16	2-1/2
B-12-4	2-1/2	2-9/16	2-1/2	2-1/2	2-1/2
B-12-5	2-1/2	2-1/2	2-9/16	2-1/2	2-1/2
B-12-6	2-1/2	2-1/2	2-9/16	2-9/16	2-1/2
B-12-7	2-1/2	2-9/16	2-9/16	2-9/16	2-9/16
B-12-8	2-7/16	2-9/16	2-1/2	2-9/16	2-1/2
A-24-1	2	1-15/16	2-1/16	1-15/16	2
A-24-2	2-1/4	2	2-3/16	2	2-1/8
A-24-3	2-1/4	2	2-1/8	2	2-1/8
A-24-4	2	2-3/16	2-1/8	2-1/8	2-1/8
A-24-5	1-7/8	1-15/16	2-1/4	1-15/16	2
A-24-6	1-7/8	2	1-15/16	2	1-15/16
A-24-7	1-7/8	1-15/16	1-7/8	2-1/16	1-15/16
A-24-8	1-7/8	2	1-15/16	2-1/16	2
B-24-2	2-7/8	3	3	2-7/8	2-15/16
B-24-3	2-7/8	2-7/8	2-13/16	2-15/16	2-7/8
B-24-4	3	2-7/8	3	2-15/16	2-15/16
B-24-5	2-13/16	2-15/16	3	2-15/16	2-15/16
B-24-6	2-13/16	2-15/16	2-15/16	2-7/8	2-7/8
B-24-7	2-3/4	3-1/16	2-7/8	2-13/16	2-7/8
B-24-8	2-13/16	2-15/16	2-15/16	2-7/8	2-7/8
R-24-1	2-3/16	2-1/16	2	1-15/16	2-1/16
R-24-2	1-15/16	2	2	1-15/16	2
R-24-3	2-1/16	1-15/16	2	1-13/16	1-15/16
R-24-4	2-3/16	2	1-15/16	1-7/8	2
R-24-5	1-7/8	2	1-15/16	1-15/16	1-15/16
R-24-6	1-7/8	1-7/8	2-3/16	2	2
R-24-7	1-7/8	2	1-15/16	2	1-15/16
R-24-8	1-7/8	2	1-7/8	2	1-15/16

Table 39

MEASUREMENT OF LENGTH
ACPA PIPE

Specimen	Length, inches	
	Maximum	Minimum
A-12-1	30-1/8	30
A-12-3	29-13/16	29-5/8
A-12-4	29-7/8	29-13/16
A-12-5	30-1/8	30
A-12-6	30	29-15/16
A-12-7	30-3/16	30-1/16
A-12-8	30-1/16	30-1/16
B-12-2	30	29-15/16
B-12-3	30	29-7/8
B-12-4	30	29-15/16
B-12-5	29-13/16	30-1/16
B-12-6	29-7/8	30-1/8
B-12-7	29-7/8	30-1/4
B-12-8	29-7/8	30
A-24-1	30-1/4	30-1/16
A-24-2	30-1/4	30-1/8
A-24-3	30-1/4	30-1/8
A-24-4	30-1/4	30-1/8
A-24-5	30-1/8	30-1/4
A-24-6	30-1/8	30-1/4
A-24-7	30-1/8	30-1/4
A-24-8	30-3/16	30-1/4
B-24-2	29-7/8	29-11/16
B-24-3	30	29-7/8
B-24-4	30-1/16	29-7/8
B-24-5	29-7/8	29-15/16
B-24-6	29-11/16	29-7/8
B-24-7	29-7/8	30
B-24-8	29-15/16	30
R-24-1	30-5/16	30-1/8
R-24-2	30-1/8	30-1/16
R-24-3	30-3/16	30-1/16
R-24-4	30-1/16	30
R-24-5	30-1/4	30-1/8
R-24-6	30-1/8	30-1/16
R-24-7	30-3/16	30-1/16
R-24-8	30-3/16	30-1/8

Thirty-seven specimens were either tested in three-edge bearing or hydrostatically at the USBR. Seventeen of these specimens were PIC pipe. Twenty specimens were untreated and used as controls.

Figure 39. Measurement of internal diameter, ACPA pipe.

Figure 40. Measurement of wall thickness, ACPA pipe.

9.1.2.1. Impregnation Procedures

Eighteen were impregnated with t-butyl-styrene and radiation polymerized. Preparation of test specimens is shown in Figures 41 to 45.

Figure 41. Electrical resistance heated oven of the walk-in type used for drying ACPA concrete pipe specimens.

Figure 43. Typical assembly of an ACPA concrete pipe specimen in the impregnator.

Figure 42. Impregnator and ancillary equipment used for impregnation of ACPA concrete pipe specimens.

Figure 44. Removal of an ACPA concrete pipe specimen from impregnator.

Figure 45. Wrapping a monomer saturated ACPA concrete pipe specimen with aluminum foil to reduce evaporation losses prior to polymerization.

The impregnated samples are described in Table 40. Four specimens of each size and type were impregnated. Three of each set were to be tested, the fourth retained as a spare. One spare specimen, B-12-1 (12-inch inside diameter by 2-1/2-inch wall) was used in a trial experiment to verify the handling and impregnation methods prior to preparing the test specimens. About 50 core samples (1-5/16-inch diameter by 2-1/2 inches long) were cut from Specimen B-24-1, for use in quality control procedures. Two core specimens accompanied a pipe specimen throughout the entire impregnation process. After irradiation the cores were destructively tested, to determine the compressive strength, and examined visually for evidence of unpolymerized monomer and to determine the depth of penetration.

Monomer Selection.—The selection of t-butyl styrene was based primarily on the processing conditions imposed by the existing impregnation equipment. As shown in Figures 44 and 45, it was necessary to remove the monomer saturated specimens from the impregnator and to wrap them prior to initiating polymerization. Because of the design of the impregnator, the weight of the pipe sections, and the distance between the impregnation and the radiation facilities, exposure of the pipe to the atmosphere for times up to 60 minutes was expected. Previous

experience with the same equipment and procedures for impregnation of 16-inch outside diameter by 1-inch wall hemispheres with MMA, which has a relatively high vapor pressure (40 mm at 77° F (25° C)) indicated that the evaporation losses from specimens having high surface/volume ratios would be considerable. This loss appreciably affected the final strengths of the composites. Vapor pressure was therefore the prime consideration in the selection of a monomer for the pipe impregnation.

T-butyl styrene (vapor pressure < 1 mm at 77° F (25° C)) has been studied as a potential candidate for general applications of PIC at ambient temperatures. The potential advantages of this monomer include its moderate cost, relatively high polymerization rate, and low volatility. Another advantage is its relatively low volume shrinkage (about 7 percent) on polymerization, which would minimize the void volume in polymer-filled concrete. Test results from survey-type experiments indicated that composites with compressive strengths of about 18,000 psi and tensile strengths of about 1,400 psi could be produced with t-butyl styrene. On the basis of these test results and the potential advantages, t-butyl styrene was selected.

Impregnation Method.—The method of impregnation used for the pipe sections was the standard vacuum-soak procedure, summarized below:

- (1) Dry concrete specimen to constant weight.
- (2) Evacuate the dried specimen at a pressure of < 3 inches Hg.
- (3) Soak in monomer.
- (4) Drain monomer.
- (5) Wrap the specimen to reduce evaporation of monomer from the sample.
- (6) Polymerize to completion by use of gamma radiation.

Specific details for each of these procedures as related to the pipe specimens are given below.

Drying.—The concrete pipe specimens and core specimens were dried to constant weight at 302° F (150° C) in an electrical resistance heated oven of the "walk-in" type (Figure 41). Six lengths of 12-inch diameter or two lengths of 24-inch-diameter pipe could be dried at the same time. Data were initially obtained on the rate of drying for one of the largest samples, 24-inch inside diameter by 3-inch-thick wall. These data indicated that approximately constant weight was

Table 40
OVEN DRYING DATA
ACPA PIPE

Identification	Size, inside diameter by wall thickness, inch	Initial weight pound	Dried weight pound	Weight loss during drying, percent
A-12-2	12 by 1-3/8	158	153	3.2
A-12-3	12 by 1-3/8	159	153	3.8
A-12-4	12 by 1-3/8	158	152	3.8
A-12-5	12 by 1-3/8	163	157	3.7
¹ B-12-1	12 by 2-1/2	304	294	3.3
B-12-2	12 by 2-1/2	306	296	3.3
B-12-3	12 by 2-1/2	302	296	2.0
B-12-4	12 by 2-1/2	307	297	3.3
A-24-1	24 by 2	436	425	2.5
A-24-2	24 by 2	436	422	3.2
A-24-3	24 by 2	435	422	3.0
A-24-4	24 by 2	434	421	3.0
² R-24-1	24 by 2	464	452	2.6
² R-24-2	24 by 2	437	425	2.8
² R-24-3	24 by 2	438	424	3.2
² R-24-4	24 by 2	420	413	1.7
³ B-24-1	24 by 3	—	—	—
B-24-2	24 by 3	638	616	3.5
B-24-3	24 by 3	640	627	2.0
B-24-4	24 by 3	650	628	3.4

All specimens dried at 302° F (150° C) to constant weight.

¹ Used in trial impregnation.

² Steel reinforced.

³ Not impregnated. Core samples cut from specimen.

attained in a maximum of 24 hours with the specimen showing a weight loss of 3.5 percent, and therefore, all specimens were dried for a minimum of 48 hours. These results are summarized in Table 40.

Monomer Impregnation.—The dried pipe sections and core specimens were weighed and allowed to cool in air to ambient temperature before being placed in the impregnator. The impregnations were performed in a 32-inch-diameter by 36-inch long upright type of impregnator (see Figure 42). To reduce the amount of monomer handled during an experiment, volume displacement sections were placed inside and, in the case of the 12-inch specimens, around the pipe. About 30 gallons of t-butyl styrene were required to fill the impregnator to a level 5 inches above the pipe. A typical loading of a pipe, cures, and volume displacement section in the impregnator is shown in Figure 43.

The impregnator was evacuated to a pressure of < 3 inches Hg by using a 5.6-cfm oil-sealed two-stage rotary-vacuum pump. The minimum pressure was attained within 15 minutes and the total evacuation time was 4 hours.

After evacuation, the t-butyl styrene was siphoned into the impregnator. Prior to each contacting, the monomer viscosity and inhibitor concentration were measured to decrease the probability of premature polymerization. No change in either property was noted during the entire 5-week period in which impregnations were performed. All unconsumed monomer was reused for impregnation.

The pipe sections were covered with monomer to a depth of 5 inches. This level was sufficient to cover the two core samples and to ensure complete immersion of the pipe after saturation with monomer. The

impregnator was then pressurized with air to 15 psi-g to increase the monomer impregnation rate.

Monomer liquid level measurements were made in order to estimate the rate at which the monomer diffused into the concrete. In all cases, saturation (observed as a constant monomer level) was attained within 45 minutes. However, soaking times of about 17 hours were used in order to coordinate the impregnation process with the slower polymerization process.

After soaking, the specimens were removed from the impregnator (Figure 44), wrapped in aluminum foil (Figure 45), and transported to the High Intensity Radiation Development Laboratory (HIRDL) at BNL for irradiation.

T-butyl styrene was then added to the monomer storage tank to replace the amount absorbed by the concrete specimen. Inhibitor concentrations and monomer viscosity were determined prior to reuse with the next specimen.

Polymerization.—Polymerization of the t-butyl styrene was initiated by gamma radiation from the 6×10^5 Ci ^{60}Co source at HIRDL. The concrete pipe was placed on a platform in the irradiation cell and rotated at 2 rpm throughout the irradiation period to impart as uniform a radiation dose as possible. The dosimetry measurements for each size of specimen are tabulated below.

Size	Dose rate, 10^6 rads/hr		
	Maximum	Minimum	Average
12-inch inside diameter by 1-3/8-inch wall	0.86	0.55	0.70
12-inch inside diameter by 2-1/2-inch wall	0.68	0.44	0.56
24-inch inside diameter by 2-inch wall	0.78	0.46	0.62
24-inch inside diameter by 3-inch wall	0.78	0.42	0.56

During each irradiation, two monomer saturated core specimens were attached to the inner surface of the pipe at points of minimum radiation intensity. On completion of the irradiation, the samples were visually examined for unpolymerized monomer. Two irradiations were also conducted in which small test tubes of monomer were attached to the inner surface of the pipe. These samples were periodically examined

visually to obtain an estimate of the required radiation dose for complete polymerization.

Based upon these observations, it was estimated that complete conversion could be obtained with a radiation dose of 12×10^6 rads at an intensity of about 0.6×10^6 rads per hour. As noted in Table 41 each specimen received a dose in excess of that amount.

9.1.2.2. Test Results

Nondestructive Testing.—Each section of the pipe was subjected to the Schmidt hammer test after drying and again after irradiation. The rebound readings were taken with the pipe in a vertical position. The hammer was held horizontally and two sets of 16 readings were taken from top to bottom along two diagonally opposite sides of the pipe. After impregnation and polymerization the procedure was repeated. The results from these tests are given in Table 41.

Discussion of Results.—The polymer loadings obtained in 18 pieces of pipe are summarized in Table 41. Polymer loadings ranging between 4.4 and 5.6 percent were obtained for pipe of wall thicknesses between 2 and 3 inches. There appears to be no correlation between polymer loading and wall thickness. The average polymer loading was 4.8 percent. Steel reinforced specimens exhibited polymer loadings similar to those of the nonreinforced specimens. Higher polymer loadings, averaging 6.3 percent were obtained in the 12-inch inside diameter by 1-3/8-inch-thick wall specimens. This increase in loading may have been due to a high porosity brought about by differences in aggregate size and degree of vibration during the manufacture of this pipe.

The potential reliability of the Schmidt hammer as a nondestructive testing device was demonstrated during the testing of Specimen B-12-1. This specimen was used in a trial impregnation to verify the handling and processing procedure. This was fortunate since, as the result of an error, the pipe was partially immersed in monomer. After polymerization, predictions were made of the distribution of polymer throughout the specimen by use of the Schmidt hammer. These predictions were substantiated by the results of compression tests performed on cure samples and a three-edge bearing test. For the latter, a bearing load of 9,100 pounds per linear foot was measured for a section of fully impregnated material. Compared with a bearing load for the control of 5,120 pounds per linear foot, this represents an increase of 89 percent and is in agreement with earlier results obtained with fully impregnated 4-inch-diameter specimens.

Table 41

SUMMARY OF EXPERIMENTAL DATA

ACPA Pipe

Identification	Size, inside diameter by wall thickness inches	Polymer loading percent of dried unimpregnated weight	Average radiation intensity 10^6 rads/hr	Average radiation dose 10^6 rads	Average rebound number ²		Increase in rebound number percent
					Unimpregnated	Impregnated	
A-12-2 ³	12 by 1-3/8	5.2	0.70	16.7	40.3 ± 1.3	53.2 ± 2.1	32.0
A-12-3	12 by 1-3/8	6.5	0.70	12.6	40.2 ± 1.6	52.8 ± 2.5	31.3
A-12-4	12 by 1-3/8	7.2	0.70	16.5	40.2 ± 2.0	52.4 ± 2.8	30.3
A-12-5	12 by 1-3/8	6.4	0.70	16.1	42.5 ± 2.7	51.4 ± 3.4	20.9
B-12-2	12 by 2-1/2	4.7	0.56	13.2	45.5 ± 1.8	55.8 ± 3.5	22.6
B-12-3	12 by 2-1/2	4.7	0.56	13.2	44.6 ± 2.7	54.7 ± 2.8	22.6
B-12-4	12 by 2-1/2	4.7	0.56	13.2	43.8 ± 2.7	56.1 ± 2.5	28.0
A-24-1	24 by 2	4.7	0.62	14.9	41.4 ± 2.2	52.0 ± 3.6	25.6
A-24-2	24 by 2	5.0	0.62	14.9	41.6 ± 3.6	55.1 ± 2.0	33.1
A-24-3	24 by 2	4.7	0.62	14.9	42.7 ± 2.7	52.7 ± 2.6	23.4
A-24-4	24 by 2	5.2	0.62	14.9	37.9 ± 4.4	51.7 ± 4.8	36.4
R-24-1 ¹	24 by 2	4.4	0.62	14.9	47.1 ± 2.2	53.8 ± 2.6	14.2
R-24-2 ¹	24 by 2	4.5	0.62	14.9	44.8 ± 2.5	51.8 ± 2.4	15.6
R-24-3 ¹	24 by 2	5.5	0.62	14.9	43.7 ± 1.3	54.1 ± 2.0	23.7
R-24-4 ¹	24 by 2	5.6	0.62	14.9	41.7 ± 1.6	54.6 ± 2.1	30.9
B-24-2	24 by 3	5.4	0.56	12.9	43.6 ± 1.1	56.1 ± 1.7	28.6
B-24-3	24 by 3	4.6	0.56	13.3	44.3 ± 1.8	57.1 ± 2.1	28.8
B-24-4	24 by 3	4.8	0.56	12.9	45.6 ± 2.4	56.4 ± 1.8	23.6

¹ Steel reinforced.² Schmidt impact hammer held in horizontal position.³ Retained at BNL.

Monomer, tertiary-butyl styrene.

Radiation induced polymerization.

Specimens dried at 302° F (150° C) to constant weight.

The results of the Schmidt hammer tests are included in Table 41. The average and mean deviation of 32 readings on each specimen taken before and after impregnation are listed. In all cases, substantial increases in the rebound readings were obtained after impregnation. Little variation along the pipe was noted. These results indicate that the specimens were fully impregnated.

Two core samples, 1-5/16-inch diameter by 2-1/2 inches long, were impregnated in conjunction with each pipe section. After polymerization, the samples were examined for unpolymerized monomer and to determine the depth of penetration, and all seemed to be fully impregnated. Compressive strength measurements were also made. These data are summarized in Table 42. Prior to testing, the ends of the cores were made parallel by capping with a high-strength, fast-setting, sulfur-based compound. For the impregnated cores, an average compressive strength of 17,370 psi with a coefficient of variation of 14.9 percent was calculated. Compared with an average control strength of 6,625 psi (Table 43), this corresponds to an increase in strength of 162 percent. The test results are in good agreement with the value of 18,000 psi previously obtained for CP-type concrete impregnated with t-butyl styrene.

As indicated by the coefficient of variation, the data exhibit considerable scatter, probably because the diameter of the core specimen was small compared with the size of the aggregate. To obtain a sample with a length over diameter of about 2, the core diameter was limited to 1-5/16 inches. Examination of many of the cores that produced low strengths showed pieces of aggregate $> 3/4$ inch.

Therefore, the ratio of specimen diameter to aggregate size is < 2 , considerably lower than the ratio of 3 specified by ASTM Designation: C 42-68. The results indicated, however, that strengths comparable to those obtained in laboratory tests can be expected.

Three-edge Bearing Tests.—The three-edge bearing tests were conducted according to ASTM Designation: C 497. The ultimate load values for all specimens and the load values when a 0.01-inch crack was obtained (cracking load) for the reinforced specimens are given in Table 44. Figure 46 shows a typical test and Figure 47 shows failure of a specimen.

All the pipe specimens were oriented in the test so the point A, refer to Figure 39, was the top of the pipe.

Evaluation of Three-edge Bearing Test.—The largest increase between concrete and PIC pipe in three-edge

Figure 46. Three-edge bearing test of ACPA pipe.

Figure 47. Failure of ACPA pipe in three-edge bearing test.

bearing was in the 24-inch inside diameter thin wall specimens. Refer to Table 46 for the comparison between the control and PIC pipe in each group in three-edge bearing tests.

In comparing pipe groups A-24 and R-24, which have the same dimensions, group R-24 reinforced PIC pipe did not yield a significant increase over the A-24 PIC pipe. Hence, in 24-inch PIC pipe with a wall thickness of 2 inches, reinforcement is not very effective. The reinforcement is effective in concrete pipe as evidenced

Table 42

SUMMARY OF RESULTS FOR IMPREGNATED CORE SAMPLES

ACPA Pipe

Pipe ¹	Identification Core	Polymer loading, percent of dried unimpregnated weight	Compressive strength, psi
	Controls	0	² 6,625
A-12-3	T-5	4.1	12,560
	T-6	4.0	13,710
A-12-5	T-7	4.5	18,460
	T-8	4.5	17,220
A-12-2	T-9	4.4	19,670
	T-10	4.4	19,680
B-12-3	T-11	4.5	
	T-14	4.9	
A-12-4	T-12	4.9	20,600
	T-13	5.0	17,200
B-12-4	T-15	4.8	
	T-16	4.5	
B-12-2	T-17	4.0	18,890
	T-18	4.9	16,000
B-24-4	T-19	4.5	19,600
	T-20	4.5	16,125
B-24-3	T-21	4.5	19,850
	T-22	6.1	15,500
B-24-2	T-23	4.9	17,550
	T-24	4.1	18,450
R-24-2	T-25	2.0	15,500
	T-26	2.0	16,900
A-24-1	T-27	4.6	18,000
	T-28	3.8	18,950
R-24-4	T-29	4.6	20,500
	T-32	3.9	20,200
R-24-1	T-33	4.7	16,550
	T-34	4.3	19,750
A-24-3	T-35	4.8	19,825
	T-36	4.5	17,950
R-24-3	T-37	4.5	15,950
	T-38	4.0	15,340
A-24-4	T-39	4.4	15,725
	T-40	4.0	14,045
A-24-2	T-42	4.9	12,210
Mean		4.4	17,370
Deviation		±0.7	±2,590
Coefficient of variation		16.0	14.9

¹ Pipe section that accompanied core samples throughout impregnation process.² Average of 12 control core specimens; data points given on Table 43

Monomer, tertiary-butyl styrene

Radiation-induced polymerization

Specimens dried at 302° F (150° C) to constant weight

Specimen size, 1-5/16-inch diameter by 2-1/2 inches long

Table 43

SUMMARY OF RESULTS FOR UNIMPREGNATED
CORE SAMPLES

ACPA Pipe	
Sample	Compressive strength, psi
T-1	9,700
T-2	9,100
T-43	6,900
T-44	7,800
T-45	5,520
T-46	7,640
T-47	5,640
T-48	5,090
T-49	6,500
T-50	6,450
T-51	4,550
T-52	4,940
Mean	6,625
Deviation	±1,576
Coefficient of variation	23.7 percent

Specimen size, 1-5/16-inch diameter by 2-1/2 inches long.

Table 45

EVALUATION OF THREE-EDGE
BEARING TESTS
ACPA PIPE

Specimen group	Average polymer loading (weight percent)	Average ultimate load (pounds/foot)	Percent increase over control
A-12	0.0	1,780	—
A-12	6.5	3,760	111
B-12	0.0	5,020	—
B-12	4.7	12,480	149
A-24	0.0	1,730	—
A-24	4.8	4,400	154
B-24	0.0	3,410	—
B-24	4.9	7,200	111
R-24	0.0	3,660	—
R-24	5.0	4,990	36

Table 44

RESULTS OF THREE-EDGE
BEARING TESTS
ACPA PIPE

Specimen	Polymer loading (weight percent)	Cracking ¹ load (pounds/foot)	Ultimate load (pounds/foot)
A-12-1	0.0	—	1,550
A-12-3	6.5	—	3,760
A-12-6	0.0	—	2,000
B-12-2	4.7	—	12,480
B-12-5	0.0	—	4,910
B-12-6	0.0	—	5,120
A-24-1	4.7	—	4,320
A-24-2	5.0	—	4,480
A-24-5	0.0	—	1,720
A-24-6	0.0	—	1,720
A-24-7	0.0	—	1,630
A-24-8	0.0	—	1,850
B-24-2	5.4	—	5,920
B-24-3	4.6	—	7,660
B-24-4	4.8	—	8,030
B-24-5	0.0	—	3,180
B-24-6	0.0	—	3,320
B-24-7	0.0	—	3,620
B-24-8	0.0	—	3,500
R-24-1	4.4	2,560	5,380
R-24-2	4.5	2,180	4,440
R-24-3	5.5	3,320	5,040
R-24-4	5.6	2,640	5,120
R-24-5	0.0	1,620	4,320
R-24-6	0.0	1,910	3,370
R-24-7	0.0	1,870	3,770
R-24-8	0.0	1,850	3,160

¹Cracking load is the load when a 0.01-inch crack formed.

Table 46

**RESULTS OF HYDROSTATIC TESTS
ACPA PIPE**

Specimen	Polymer loading (percent)	Ultimate pressure (psi)	Average ultimate pressure (psi)	Percent increase over control
A-12-4	7.2	*90	170	102
A-12-5	6.4	170		
A-12-7	0.0	84		
A-12-8	0.0	84	84	—
B-12-3	4.7	230	252	133
B-12-4	4.7	275		
B-12-7	0.0	92		
B-12-8	0.0	125	109	—
A-24-3	4.7	117	110	—
A-24-4	5.2	104		

* Defective specimen, not included in averages.

by R-24 concrete pipe having a strength of 3,660 pounds/foot and A-24 concrete pipe having a strength of 1,730 pounds/foot (a strength increase of 112 percent). These limited tests indicate that polymer impregnation of unreinforced pipe is as effective in increasing three-edge bearing strength as is the use of reinforcement.

Hydrostatic Tests.—The hydrostatic tests were conducted according to ASTM Designation: C 97. The ultimate pressure values are given in Table 46. Figure 48 shows the equipment used in the test of a pipe specimen. Figure 49 shows failure of a specimen.

Figure 48. Hydrostatic test of ACPA pipe.

Figure 49. Failure of ACPA pipe in hydrostatic test.

Evaluation of Hydrostatic Test.—Hydrostatic testing of the pipe specimens was limited to a few specimens because the pipe used in this program is not considered

pressure pipe. The result of the hydrostatic test on Specimen A-12-4 showed a very small increase; this specimen failed at the points where the Schmidt hammer impact test was made. It is believed this originally was a plane of weakness and not caused by the Schmidt hammer; this specimen was not included in the comparison of test results. The ultimate pressure sustained by the test specimens shows an increase for PIC A-12 pipe of 102 percent, and an increase for PIC B-12 pipe of 133 percent over control pipe.

9.1.3. Sewer Pipe, Joint Program with city of San Diego

Based on the improvements in structural and durability properties as measured in laboratory tests, a field test of PIC pipe in a sewage line was undertaken. In this test, the effect of hydrogen sulfide produced by bacterial action will be determined.

Six lengths of 8-inch-diameter by 24-inch-long pipe were impregnated with t-butyl styrene and radiation polymerized. (Reasons for selecting of t-butyl styrene were discussed in Section 9.1.2.) The pipe contained Type II cement and nonreactive aggregate, and was fabricated according to ASTM Designation: C 14 specifications. Experimental data for the series are given in Table 47. In cooperation with the city of San Diego, four sections of PIC pipe were installed in their sewage system during July 1970. The pipe specimens will be examined periodically for evaluation of resistance to chemical attack.

Table 47

POLYMER IMPREGNATED SEWER PIPE

City of San Diego

Specimen	Polymer loading, percent of unimpregnated weight
H-1	4.3
H-2	4.2
H-3	4.3
H-4	4.4
H-5	4.4
H-6	4.3

Monomer, tertiary butyl styrene
Radiation-induced polymerization
Average radiation intensity, 8.5×10^5 rads/hr
Average radiation dose, 2×10^7 rads
Specimen size, 8-inch diameter by 24 inches long
Specimens oven-dried at 302° F (150° C) prior to
impregnation

9.2. HOUSING

The use of concrete-polymer materials for a number of housing applications is being investigated. Potential applications include beams, wall and floor panels, and building block.

In cooperation with the AEC and BNL, the Agricultural Research Service of the USDA, Athens, Georgia, is studying the feasibility of using concrete-polymer materials for precast panels in low-cost rural and urban housing. Steel-fiber reinforced and nonreinforced concrete specimens were tested. In general, an increase in flexural strength by a factor of about 5 was obtained with fiber reinforcing and polymer impregnation.² Similar increases in compressive strength were obtained. No additional tests were performed during the past year.

Lightweight concrete would be more widely used for housing applications if higher strengths could be obtained. The results from preliminary experiments indicate that large improvements in compressive, tensile splitting, and flexural strengths can be obtained by impregnation with MMA and radiation polymerization.

Specimens containing perlite, vermiculite, and foamed-glass aggregates are being tested. Preliminary results from these tests have been published.³ The large increases in strength combined with the expected thermal insulation and acoustical properties and the maintenance-free surface, would appear to make these composites a potential material for many building applications.

The Construction Engineering Research Laboratory (CERL), U.S. Army Corps of Engineers, Champagne, Illinois, has started a program for developing lightweight concrete-polymer materials for housing panels. Insulating lightweight concretes have been partially impregnated with polyester-styrene and compressive strengths of 2,300 psi at 40 pound/foot³ density have been obtained. For panel construction only the outer layers, where maximum stress occurs, need be impregnated; the center can remain unimpregnated for purposes of thermal and acoustic insulation.

Tests conducted at Lehigh University on Inset building block (Concrete Products Magazine, page 52, June 1970), indicated a 250-percent increase in compressive strength and improved durability properties in block impregnated with MMA to 14 weight percent loading. The Universal Building Products Corporation of Washington, D.C., is

recommending this block for below water bridge piers and retaining walls as well as for housing applications.

9.3. MARINE APPLICATIONS

Concrete is being considered as a material of construction for underwater habitats because of its low cost, high strength, and excellent resistance to chemical attack. The greatly improved structural properties, and negligible water absorption and permeability would appear to make PIC excellent materials for underwater applications.

In a cooperative program with the Naval Civil Engineering Laboratory (NCEL), Port Hueneme, California, a series of 16-inch outside diameter by 1-inch wall hemispheres is being impregnated and evaluated.

Previous work² with MMA-impregnated specimens indicated compressive strengths of about 20,400 psi, compared with about 10,000 psi for the controls.² One impregnated sphere imploded at a pressure of about 3,020 psi, compared with a burst pressure of about 2,600 psi for the control sphere. Visual observation of the fractured wall indicated that both the inner and outer surfaces were void of polymer to a depth of about one-eighth inch. Voids extending to greater depths were also apparent. The probable cause of the voids was evaporation of monomer prior to

polymerization. Reimpregnation of a second sphere resulted in an increase in burst pressure to 4,700 psi, but loss by evaporation from the surface was still apparent.

Based upon these observations, it was decided that in future work low vapor pressure monomers would be used.

Survey-type experiments were conducted with NCEL-type concrete impregnated with DAP as chlorostyrene. Compressive strengths of 20,000 and 17,300 psi, respectively, were measured. Void-free surfaces were also obtained with the DAP-impregnated specimens.

Twelve 16-inch outside diameter by 1-inch wall hemispheres and thirty-six 3-inch-diameter by 6-inch-long cylinders were impregnated with DAP and are being tested. The results to date are given in Tables 48 and 49. The ratio of the implosion pressure to compressive strength (P_{im}/f'_c) for the PIC spheres was approximately 25 percent lower than that for the control spheres (Table 49). This difference in behavior was unexpected. Strain data indicate that the spheres may have undergone a buckling mode of failure instead of the normal material failure experienced by plain concrete spheres. Additional tests will be performed on spheres with 2-inch-thick walls. These data should provide additional information on the structural behavior of PIC spheres.

Table 48

MECHANICAL PROPERTIES OF POLYMER-IMPREGNATED CONCRETE

NCEL Concrete

Cylinder designation	Polymer loading ¹ percent by weight	Compressive strength, ² f'_c , psi	Modulus of elasticity 10^6 psi
730C	8.3	20,960	³ 4.76
730C	0	9,940	⁴ 3.78
724C	8.5	21,550	5.16
724C	0	9,750	3.78
731C	7.9	23,070	6.70
731C	0	11,220	3.78
725C	8.1	22,350	5.10
725C	0	10,000	3.78

¹ Average of three specimens.

² Average of three specimens.

³ Value from one specimen.

⁴ Average elastic modulus for control cylinders obtained from previous work.

Monomer diallyl phthalate.

Table 49

HYDROSTATIC LOADING TESTS ON POLYMER-IMPREGNATED CONCRETE SPHERES

NCEL Concrete

Sphere number	Wall thickness, inch	Implosion pressure P_{im} (psi)	Control cylinder compressive strength f'_c psi	Ratio P_{im}/f'_c
A	1.0	4,670	20,960	0.223
B	1.0	5,065	22,350	0.227
Control ¹	1.0	3,000	10,000	0.300

¹ Result from previous work on plain concrete spheres.

Test results on 16-inch outside diameter spheres.

Average diallyl phthalate polymer loading of 8.2 percent by weight.

10. CONCLUSIONS AND RECOMMENDATIONS

The work to date has indicated that remarkable improvements in the structural and durability properties of concrete can be obtained by monomer impregnation and in situ polymerization by either radiation or thermal-catalytic means. Five monomer systems in concrete are presently under test for normal temperature applications. Two systems are being studied for high-temperature applications.

The monomers tested for normal temperature applications are MMA, styrene, acrylonitrile, MMA plus 10 weight percent TMPTMA, and chlorostyrene.

All of the monomer systems under study have shown improvement in structural properties and durability as compared with unimpregnated concrete. The degree of improvement is a function of polymer loading. Concrete impregnated with MMA and MMA plus 10 percent TMPTMA have given the greatest improvements, resulting in compressive strengths as high as 22,500 psi and tensile strengths of 1,700 psi (increases of over 300 percent). Test cylinders have given no failures after more than 2 years' exposure to sulfate attack and 15 percent HCl. One specimen has exhibited essentially no deterioration after 7,220 cycles of freezing and thawing.

Tests are continuing to determine the high-temperature structural properties and durability for applications as would be encountered in desalting plants.

Two OSW monomer systems, 60 percent styrene plus 40 percent TMPTMA and 90 weight percent DAP plus 10 percent MMA, are being evaluated. Tests with CP-type concrete, impregnated with these monomers, show essentially the same high-temperature structural properties as those obtained with the more expensive OSW-type concrete. Creep tests at 290° F (143° C) at sustained compressive loads of 7,000 psi show a reduction in creep of about 90 percent as compared with unimpregnated control concrete.

A 28-inch-diameter by 12-inch-thick specimen was fully impregnated with 60 percent styrene plus 40 percent TMPTMA. This is the largest specimen that has been impregnated to date. Results indicate that walls of the thickness required for large desalting vessels can be successfully impregnated.

Resistance to chemical attack by hot brine and distilled water at temperatures up to 290° F is being measured. Data are not yet available for reporting.

Development of PC and PCC is continuing. To date, greater success has been obtained with PC. PC made with an aggregate consisting of portland cement and sand, and containing a 10 percent by weight of a 10 percent polyester plus 90 percent styrene mixture gave a compressive strength of 23,500 psi. Additional work appears warranted for PC and PCC because of a large number of potential applications for premixed-type concrete-polymer materials.

A number of potential applications for PIC are being evaluated in cooperative programs with several governmental agencies. The applications include the use of PIC for desalting plants, beams and wall panels in housing, underwater structures, bridge decks, roads, and luminaries.

Field tests of PIC drain tile have been in progress for 1 year. Sections are scheduled for examination in July 1971. PIC sewer pipe has been installed in the city of San Diego sewer system. The pipe will be examined periodically.

A joint program with the ACPA for polymer-impregnated sewer and culvert pipe has been completed. Three-edge bearing test results show an average improvement of 131 percent for unreinforced pipe and an improvement of 36 percent for reinforced pipe. Hydrostatic pressure tests show an average improvement of 94 percent for unreinforced pipe. These results are highly significant and warrant an expanded program to obtain the data required for full utilization of these improved properties in the design of polymer-impregnated pipe.

The following recommendations are made for the future program:

- (1) Applications should be identified and evaluated in both laboratory and field investigations. Such applications would include precast concrete pipe, precast tunnel supports and lining, and onsite impregnation of concrete for protection or repair.
- (2) Process technology investigations should be directed toward development of production-line methods.
- (3) Preliminary process and product designs for specific applications should be made with the available data on concrete-polymer materials.
- (4) Further work should be done on evaluating PC and PCC and the use of both radiation and thermal-catalytic techniques of polymerization.

(5) Concretes of varying composition should be used to study the effect of porosity and aggregate density on the ultimate strength of the concrete-polymer material. Various curing methods should also be investigated.

(6) Durability tests started on the normal temperature PIC should be completed. Studies of additional durability properties such as fire resistance, weatherability, resistance to bases, and soil micro-organisms should be initiated.

(7) Long-term durability tests on the high-temperature concrete-polymer materials should be initiated, and high-temperature strength measurements should be continued.

(8) Comprehensive strength data should be obtained on concrete-polymer systems to yield data on maximum and minimum strength curves for a given monomer.

(9) Structural data should be obtained on unreinforced and steel-reinforced PIC to permit development of design criteria.

(10) Investigations should be made to develop reliable methods for the quality control of concrete-polymer products.

(11) Fundamental studies on the basic nature of concrete-polymer materials should be continued to determine areas for further improvements.

(12) Construction methods for the utilization of concrete-polymer materials should be developed and tested.

(13) Methods for joining structural members should be developed and tested.

11. REFERENCES

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CONVERSION FACTORS--BRITISH TO METRIC UNITS OF MEASUREMENT

The following conversion factors adopted by the Bureau of Reclamation are those published by the American Society for Testing and Materials (ASTM Metric Practice Guide, F 383-59) except that additional factors (*) commonly used in the Bureau have been added. Further discussion of definitions of quantities and units is given in the ASTM Metric Practice Guide.

The metric units and conversion factors adopted by the ASTM are based on the "International System of Units" (designated SI for Systeme International d'Unites), fixed by the International Committee for Weights and Measures; this system is also known as the Giorgi or MKSA (meter-kilogram (mass)-second-ampere) system. This system has been adopted by the International Organization for Standardization in ISO Recommendation R-31.

The metric technical unit of force is the kilogram-force; this is the force which, when applied to a body having a mass of 1 kg, gives it an acceleration of 9.80665 m/sec/sec, the standard acceleration of free fall toward the earth's center for sea level at 45 deg latitude. The metric unit of force in SI units is the newton (N), which is defined as that force which, when applied to a body having a mass of 1 kg, gives it an acceleration of 1 m/sec/sec. These units must be distinguished from the (inconstant) local weight of a body having a mass of 1 kg; that is, the weight of a body is that force with which a body is attracted to the earth and is equal to the mass of a body multiplied by the acceleration due to gravity. However, because it is general practice to use "pound" rather than the technically correct term "pound-force," the term "kilogram" (or derived mass unit) has been used in this guide instead of "kilogram-force" in expressing the conversion factors for forces. The newton unit of force will find increasing use, and is essential in SI units.

Where approximate or nominal English units are used to express a value or range of values, the converted metric units in parentheses are also approximate or nominal. Where precise English units are used, the converted metric units are expressed as equally significant values.

Table 1

QUANTITIES AND UNITS OF SPACE

Multiply	By	To obtain
LENGTH		
Mil.	25.4 (exactly).	Micron
Inches	25.4 (exactly).	Millimeters
	2.54 (exactly)*.	Centimeters
Feet	30.48 (exactly).	Centimeters
	0.3048 (exactly)*.	Meters
	0.003048 (exactly)*.	Kilometers
Yards	0.9144 (exactly).	Meters
Miles (statute).	1,609.344 (exactly)*.	Meters
	1.609344 (exactly).	Kilometers
AREA		
Square inches	6.4516 (exactly).	Square centimeters
Square feet	929.03*.	Square centimeters
	0.092903.	Square meters
Square yards	0.836127.	Square meters
Acres	0.404689*.	Hectares
	4,046.89*.	Square meters
	0.00404689*.	Square kilometers
Square miles	2.58999.	Square kilometers
VOLUME		
Cubic inches	16.3871.	Cubic centimeters
Cubic feet	0.0283168.	Cubic meters
Cubic yards	0.764555.	Cubic meters
CAPACITY		
Fluid ounces (U.S.)	29.5737.	Cubic centimeters
	29.5729.	Milliliters
Liquid pints (U.S.)	0.473179.	Cubic decimeters
	0.473166.	Liters
Quarts (U.S.)	946.358*.	Cubic centimeters
	0.946351*.	Liters
Gallons (U.S.)	3,785.43*.	Cubic centimeters
	3,78543.	Cubic decimeters
	3,78533.	Liters
	0.00378543*.	Cubic meters
Gallons (U.K.)	4.54609.	Cubic decimeters
	4,54609.	Liters
Cubic feet	28.3160.	Liters
Cubic yards	764.55*.	Liters
Acre-feet	1,233.5*.	Cubic meters
	1,233,500*.	Liters

Table II
QUANTITIES AND UNITS OF MECHANICS

Multiply	By	To obtain
MASS		
Ounces (avoirdupois)	61.7469 (exact)	Milligrams
Troy ounces (troy)	31.1035	Grams
Ounces (avoirdupois)	28.3495	Grams
Pounds (avoirdupois)	453.59237 (exact)	Kilograms
Pounds (avoirdupois)	453.592	Kilograms
Short tons (avoirdupois)	907.185	Metric tons
Long tons (avoirdupois)	1,016.05	Kilograms
FORCE/AREA		
Pounds per square inch	0.00000706	Kilograms per square centimeter
Pounds per square inch	0.00000706	Newton per square centimeter
Pounds per square foot	4.88243	Kilograms per square meter
Pounds per square foot	4.88243	Newton per square meter
MASS/VOLUME DENSITY		
Ounces per cubic inch	1.72900	Grams per cubic centimeter
Pounds per cubic foot	16.0185	Kilograms per cubic meter
Pounds per cubic foot	0.0160185	Grams per cubic centimeter
Pounds per cubic foot	1.60185	Grams per cubic centimeter
MASS/CAPACITY		
Ounces per gallon (U.S.)	7.4893	Grams per liter
Ounces per gallon (U.S.)	7.4893	Grams per liter
Pounds per gallon (U.S.)	119.826	Grams per liter
Pounds per gallon (U.S.)	83.454	Grams per liter
SPRING MOMENT OR TORQUE		
Foot-pounds	0.0135582	Meter-kilograms
Foot-pounds	1.35582 x 10 ³	Centimeter-dynes
Foot-pounds	0.135582	Meter-kilograms
Foot-pounds	1.35582 x 10 ³	Centimeter-dynes
Foot-pounds per inch	5.4432	Centimeter-kilograms per centimeter
Foot-pounds per inch	5.4432	Gram-centimeters
VELOCITY		
Foot per second	30.48 (exact)	Centimeters per second
Foot per second	0.3048 (exact)	Meters per second
Foot per second	0.3048 x 10 ³	Centimeters per second
Miles per hour	1.60934 (exact)	Meters per second
Miles per hour	0.44704 (exact)	Meters per second
ACCELERATION*		
Foot per second ²	0.3048	Meters per second ²
FLOW		
Cubic feet per second (second-foot)	0.0283168	Cubic meters per second
Cubic feet per second (second-foot)	0.0283168	Liters per second
Gallons (U.S.) per minute	0.06309	Liters per second
FORCE*		
Pounds	4.44822	Kilograms
Pounds	4.44822	Newton
Pounds	4.44822 x 10 ⁻⁵	Dynes

Multiply	By	To obtain
WORK AND ENERGY*		
British thermal unit (Btu)	0.252	Kilogram-calories
Btu per pound	1,055.06	Calories
Foot-pounds	1.35582	Calories per gram
Foot-pounds	1.35582	Calories
POWER		
Horsepower	746.700	Watts
Btu per hour	0.293071	Watts
Foot-pounds per second	0.7457	Watts
HEAT TRANSFER		
Btu in./hr ft ² deg F (thermal conductivity)	1.488	Milliwatts/cm deg C
Btu in./hr ft ² deg F (thermal conductivity)	0.1761	Kilocalories/m deg C
Btu/hr ft ² deg F (thermal conductance)	1.488	Kilocalories/m deg C
Btu/hr ft ² deg F (thermal conductance)	0.1761	Milliwatts/cm deg C
Deg F hr ft ² /Btu (thermal resistance)	1.761	Deg C cm ² /milliwatt
Btu/hr deg F (thermal capacity)	4.1868	Joules deg C
Btu/hr deg F (thermal capacity)	1.000	Cal/gram deg C
ft ² /hr (thermal diffusivity)	0.258	cm ² /hr
ft ² /hr (thermal diffusivity)	0.0258	m ² /hr
WATER VAPOR TRANSMISSION		
Grains/hr ft ² water vapor (transmission)	16.7	Grams/hr m ²
Pervs (permittance)	0.056	Metric perms
Pervs (permittance)	0.056	Metric perms

Table III

Multiply	By	To obtain
OTHER QUANTITIES AND UNITS		
Cubic feet per square foot per day (seepage)	304.8	Liters per square meter per day
Pound-seconds per square foot (viscosity)	1.48816	Kilogram seconds per square meter
Square feet per second (viscosity)	0.02203	Square meters per second
Fahrenheit degrees (change)	5/9 exactly	Celsius or Kelvin degrees (change)
Volts per mill	0.03047	Volts per millimeter
Lumens per square foot (foot-candle)	10.764	Lumens per square meter
Ohm-circular mils per foot	0.001682	Ohm-square millimeters per meter
Milliamps per cubic foot	35.3147	Milliamps per cubic meter
Millamps per square foot	10.7639	Millamps per square meter
Calories per square yard	4.52719	Liters per square meter
Pounds per inch	0.17858	Kilograms per centimeter