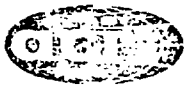


CEMENT INFORMATION

1. Menzel Symposium On High Pressure Steam Curing
2. Significance of Cement On Asbestos Cement Strength
3. A Correleation of Exaggerated Physical Effects of Additives In Mortar With The Chemistry Involved.

MENZEL SYMPOSIUM ON HIGH PRESSURE STEAM CURING

Compiled under the sponsorship of ACI Committee 516, High Pressure Steam Curing, and the Autoclave Division Committee, National Concrete Masonry Association, the 10 papers in this volume are the proceedings of a symposium held at the 65th ACI Annual Convention, Chicago, April 1969. Edited by W. H. Kuenning.



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PREFACE

Commercial high pressure steam curing (autoclaving) of concrete block in significant volume began about 1944. about 10 years after Carl Menzel developed the comprehensive background of technical information that gave this portion of the autoclaved products industry its first real impetus. Other portions of the industry have also drawn heavily on the results of Menzel's work.

Building products had been autoclaved as early as 1868 in Milwaukee by James Rowland. In Germany Michaelis initiated autoclaving as a method of testing of mortars in 1880. Interest in the autoclave process revived briefly in the United States in 1911 with the publication of a paper by Wig. In the 1920's and 1930's D. G. Miller published papers on high pressure steam curing, and Thorvaldson and various Canadian coworkers wrote of their investigations of several aspects of the subject during the same period. Woodworth reported on the properties of block made in an early autoclave plant in 1930, and Pearson and Brickett published the results of laboratory studies in 1932. These were the major autoclave forerunners of Carl Menzel.

High pressure steam curing is now applied to the manufacture of a number of products, each with their own special requirements. The most significant benefits that autoclaving contributes to the several major groups are:

Concrete block and brick - early strength, and reduction of drying shrinkage

Sand-lime brick – achievement of strength within a practical manufacturing period, and reduction of drying shrinkage

Asbestos-cement products – sulfate resistance, and early strength

Cellular concrete – high strength-to-weight ratio, and reduction of drying shrinkage

Hydrous calcium silicate heat insulation products – high strength-to-weight ratio.

Only the first, third, and fourth of these are specifically discussed in papers contributed to this volume.

We are fortunate to have three Russian papers included in this volume, on diverse subjects. In Russian practice high pressure steam curing has made it possible to utilize materials that become effective binders only at autoclave temperatures. The Russian literature therefore deals with a wider variety of raw materials than those used in the United States and Canada.

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PAPER SP 32-6

AUTOCLAVED ASBESTOS–CEMENT PRODUCTS

By JULIE C. YANG

Curing of asbestos-cement products by high-pressure autoclave, including saturated steam and superheated steam conditions is discussed and compared.

The binder produced under the various conditions were characterized by x-ray diffraction, DTA, microscopic point-count estimation of composition, pore spectra, uncombined $Ca(OH)_2$ analysis, density, and strength determinations. The desired high-strength binder was essentially tobermorite and some CSH_n gel. Al-substituted tobermorite and CSHII) may be present in small amounts, but C_2SH, Al_2O_3, Fe_2O_3, and SO_3-bearing hydrous phases were not detected.

Superheated steam conditions should be avoided at all time, and saturated steam autoclaving can be carried out usually at pressures around 100-110 psig (7.0 - 7.7 kgf/cm^2) for times of about 16 hr Curing in saturated steam, utilizing a water spray technique, is recommended because it permits the desired binders to be formed over a broad pressure range and also reduces significantly the autoclaving time.

An optimum autoclaving cycle is recommended that uses stepwise curing at two pressure levels in the presence of water spray: 5.5 hr at 115 psig (8.1 kgf/cm^2) followed by a 3.0 hr at about 130 psig (9.1 kgf/cm^2).

Keywords: *asbestos-cement products; autoclaving; binders (materials);* calcium silicate hydrates; chemical analysis; *compressive strength;* curing; differential thermal analysis; *flexural strength;* manufacturing; microscopy; pipes (tubes); porosity; presteaming period; research; superheated steam; supersaturation; tobermorite; xonotlite; x-ray diffraction.

Autoclaved asbestos-cement products were introduced commercially in 1935. The adoption of autoclaving was the culmination of a search for a curing method that would effect rapid strength development and provide a binder with good chemical stability and required physical properties.

As the demand for various autoclaved asbestos-cement products grows, capacity of the existing autoclaves, or the cost of installing new pressure vassels is usually the factor limiting increased rate of production. A significant improvement in production efficiency as well as reduction in cost will be achieved if the autoclaving cycle is shortened, while the quality of the product is maintained.

In this study, the effects on the binder properties and product strength of variations of autoclaving conditions to reduce the curing time are discussed.

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Dr. Yang is a member of the American Chemical Society, the American Ceramic Society, the American and British Mineralogical Societies, Sigma Xi (Science), Iota Sigma Pi (Chemistry), and Pi Mu Epsilon (Mathematics). As a member of the American Concrete Institute she currently serves on Committee 516, High Pressure Steam Curing.

MANUFACTURING PROCESS

Asbestos-cement products can be formed by several processes, the choice of which depends on the material being produced. Most large products such as pipe, corrugated, and flat sheets are made by a wet process that is similar to papermaking. A thin slurry containing about 10 percent thoroughly mixed solids and 90 percent water is spread into thin layers and filtered on a screen under vacuum, laminated, and compacted under pressure on a form until the desired thickness is built up. Flat sheets are stripped immediately after formation, cut, pressed, and stacked for a presteaming period,* whereas pipes are left on the mandrel for 30 to 45 min prior to stripping.

More recently, the extrusion technique has been adapted to produce desired shapes and intricate designs. In the extrusion technique a small amount of "hydromodifier" is used, a material that regulates the amount of water and the consistency of the asbestos-cement mixture without the dewatering operation that is necessary in the filtration process.

Materials

A typical formulation for autoclaved asbestos-cement products contains cement, asbestos fiber, and finely divided silica. For example, autoclaved asbestos-cement pipe contains about 20 percent chrysotile asbestos fiber, 30 to 35 percent of

* The term "presteaming period," defined in "Cement and Concrete Terminology, "ACI Publication SP-19, as" . . . the time between molding of a concrete product and start of the temperature rise period," is used here in place of the term "precure", used in the asbestos-cement industry.

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ground quartz flour with a Blaine surface area of about 2600 sq cm/g, and 45 to 50 percent of portland cement. The fine silica flour does not function as an aggregate, as in concrete, but as a chemically reactive silica that will react with the hydrolytic lime (formed during hydration of the cement) to provide additional binder material.

It is helpful to look upon the asbestos-cement system as a composite material consisting of a matrix of cement binder reinforced with asbestos fiber, in which the fiber component contributes to the high tensile, flexural, and impact strength of the product.

Sample Preparation

For this study, data were compiled by testing both 3x8x1/4-in. cake specimens and 6-in. diameter Class 150 (ASTM C 296) pressure pipe. The pipe was obtained from the production line immediately after stripping from the mandrel. A standard composition of 20 percent asbestos fiber blend, 30 percent New Jersey silex flour, and 50 percent Type I portland cement was employed. The composition of the portland cement is shown in Table 6-1.

Laboratory specimens were prepared by mixing 150 g of premixed dry blend with 1000 ml of water for 1 min with a mechanical stirrer. The slurry was transferred to a 3x8-in. filter box and the water removed by suction. The filter cake was then placed on a press with a confined mold and pressed at 320 psi (22.5 kgf/cm^2) for 5 sec to remove excess water. The specimen was stored on a glass plate for the presteaming period. As based on previous testing results, the laboratory specimens prepared in this manner simulate the actual products formed by wet process in production.

Pipes obtained from production were selected randomly. A total of 12 sections of 10=ft (3.05 m) length was taken. Each pipe was cut into four 2-½ ft (0.76 m) lengths and the group was divided into four parts.

Curing

It is standard practice to cure asbestos-cement products in a moist atmosphere at ambient temperature before autoclaving. The actual length of the presteaming period varies from 12 hr to a week, depending upon the specific product. For example, autoclaved pipes and extruded products are given a presteaming period of about 16 to 24 hr, whereas the stacked flat sheets are given 48 to 72 hr.

For this investigation, all samples (both laboratory specimens and pipe) had a presteaming period of 16 hr at approximately 90 percent relative humidity and 20-25 C.

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The autoclaving process refers to the curing of articles for a predetermined time in a saturated steam atmosphere at a temperature above 100 C. The process is generally carried out in the temperature range of 160-190 C (320-374 F), with corresponding pressure of about 75 to 180 psig (5.3 to 12.7 kgf/cm^2) for 16 hr or more. Air was purged during heating according to normal practice, and heating was done at the rate of 80-100 psig (5.6 - 7.0 kgf/cm^2) per hr.

Laboratory samples were hydrothermally treated in a small 36 in. high autoclave with cam automatic pressure controller and recorder. In some cases, the samples were stored under water while being autoclaved.

Three quarters of the 2½-ft lengths of pipe were cured under various experimental autoclave conditions. The remaining specimens were used as controls and cured under conventionally accepted optimum conditions (16 hr at 100 psig in saturated steam).

One of the experimental modifications used for curing the pipe samples was the inclusion of a water spray in the autoclave (Fig. 6-1). Spray nozzles were positioned at the top of the autoclave, and were fed with condensate water from the autoclave or service water distributed by a pump at a temperature of about 170 C under pressure. The spray nozzles were calibrated to deliver approximately 1 gal of water per minute for each foot of autoclave body length.[2]

PROPERTY EVALUATIONS

In order to evaluate the effects of the various curing conditions, the samples were subjected to the following tests:

Phase Composition

Phase composition identifications were made on an x-ray diffractometer with filtered Cu radiation.

Differential Thermal Analysis (DTA)

DTA diagrams were obtained with a differential thermal analyzer using a 28-gage Pt-Pt, 13% relative humidity thermocouple and a corundum 1200 C high temperature cell. Sensitivity of this instrument is 0.025 or 10^{-3} cal.

Microsopic examination

Examinations were made on a polarizing microscope. Petrographic thin sections were prepared for evaluation of hydrated portland cement products.[3] Quantitive estimates of the amounts of gel and fiber, unhydrated cement, and silica present were made by a point-count method on polished sections. These

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...sections were impregnated with epoxy resin and etched with 1 percent ...ic acid in alchol,[3,4] Over 300 points at equal intervals were counted ...each section, using a point-counter attachment on the stage of the pet...graphic microscope, The ensity factors applied for weight precentage cal...ations were as follows: gel 2.2, quartz 2.65, unhydrated cement 3.15, ...d chrysotile asbestos 2.56.

...rosity

Porosity determinations were made on a 60,000 psi (4220 kgf/cm^2) mer...ury intrusion porosimeter covering the pore diameter range of 100 to 0.003 ...icrons.

Strength

Mechanical strength determinations on laboratory "sag cake" specimens were ...ade according to ASTM C 459 for flexural strength. Crushing strength of the ...ommercially produced 6-in. (15-cm) Class 150 pressure pipe was determined ...n accordance with ASTM C 500.

Uncombined $Ca(OH)_2$

Uncombined $Ca(OH)_2$ was determined by ASTM Method C 500.

RESULTS AND DISCUSSION

Effect of Water-Spray Curing

Physicochemical and mechanical properties of the hardened asbestos-cement products depend on the degree of hydration, density, phase composition, and microstructure of the binder material. In a well-autoclaved high-strength specimen, using proper proportions of raw materials, the principal binder is tobermorite ($5CaO\text{-}6SiO_2\text{-}5H_2O$). [4,5,6,7] Proper autoclaving requires that saturated steam conditions be maintained. If superheated steam is used, there is a severe strength reduction.

Friedlaender,[8] and Friedlaender and Camarda[9] related the strength reduction of the autoclaved asbestos-cement product under superheated conditions to the moisture loss in the specimen. The loss of the water that is required for proper hydration is directly proportional to the maximum superheat temperature.

Our findings (Table 6-2) showed that for a curing pressure of 100 psig and temperatures in the range 190-230 C (superheated steam conditions), marked decreases in strength and increases in porosity were obtained. X-ray diffraction identification of the binder phase showed the formation of xonotlite at the expense of tobermorite. In order to compensate for the rapid moisture depletion from the product while the hydration process proceeds, a technique

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utilizing a water spray, which simulates underwater curing, was developed to ensure a saturated steam environment[2] in large production autoclaves.

Strengths of specimens cured under various experimental conditions are given in Table 6-2. The determinations demonstrate that with the aid of underwater curing, autoclaving pressure can be raised from 100 psig (7.0 kgf/cm^2) to 135 psig (9.5 kgf/cm^2), yet the strength can be maintained even though the autoclaving duration is reduced from 16 hr to 9 hr.

Effect of High Pressure and Temperature Conditions in Underwater Curing

Experiments designed to study the optimum combination of presssure build-up, duration of the underwater curing period, and binder characteristics of experimentally cured specimens were carried out. Samples used for X-ray diffraction studies, DTA, and uncombined $Ca(OH)_2$ determinations were crushed immediately after autoclaving, ground, and stored in CO_2 free desiccators.

X-Ray Diffraction Data

Kalousek, et al,[7] described the x-ray identification of tobermorite in asbestos-cement products. Their method is based on the strong diffraction peaks at 11.3, 3.07, and 2.97 Å, and it introduces also an intensity ratio measurement of the w.97 and 3.07 Å peaks to differentiate between tobermorite and Taylor's CSH (1). Tobermorite has an intensity ratio of the 2.97 and 3.07 Å peaks in the range of 0.70 to 0.80. On the other hand, CSH (1), described by Taylor[8] can be identified by strong diffraction peaks at 12.5 and 3.07 Å but there is no peak at 2.97 Å so that the intensity ratio for CSH (1) should be 0.0. Diffraction peaks at 3.02 to 3.03 Å and at 14 Å suggested to Kalousek et al, the presence of CSH_n, a gel-like phase having the composition of (0.8 - 1.33) $CaO{\cdot}SiO_2{\cdot}nH_2O$.

Table 6-3 shows the 2.97/3.07 peak intensity ratios for the experimentally cured asbestos-cement products in addition to the phase identification. A few x-ray diffraction patterns are shown in Fig. 6-2 and 6-3.

It has been found that the intensity ratios of the 2.97/3.07 lines for tobermorite can be applied only when no xonotlite or $Ca(OH)_2$ is present. When the product is cured at a pressure higher than 135 psig (9.5 kgf/cm^2), a partial transformation from tobermorite to xonotlite takes place. The strong peak at 3.07 Å for xonotlite causes a reduction of the value of 2.97/3.07 intensity ratio to around 0.5 or less depending upon the amount of xonotlite present. On the other hand, if the silica/cement ratio in the product is very low or the autoclaving condition is not adequate, an appreciable amount of $Ca(OH)_2$ is formed which in turn carbonates to form calcite. Calcite has a strong peak at 3.04 Å and also interferes with the 2.97/3.07 peak intensity computation. (This may be seen in the first two spectra of Fig. 6-2b which correspond to Samples 7 and 8 of Table 6-2.)

Taylor's CSH(1) may be present in very small amounts in these products. α-C_2SH, Al_2O_3, Fe_2O_3, and SO_3 bearing hydrous phases were not detected.

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It is difficult to determine whether the binder phase is partially Al or Fe-substituted, as described by Kalousek[11] and Diamond,[12] since the tobermorite formed under these conditions is not sufficiently well-crystallized to give sharp diffraction peaks in the 11.2 to 11.5Å region. The appearance of new peaks described by Diamond[12] at 2.67 to 2.79Å was found in most cases, even though 2.71 to 2.74Å peaks also represented the peaks of unhydrated portland cement. Other peaks that characterize the substitution at 4.20-4.25 and 3.60Å are interfered with by the quartz line (4.25Å) and the chrysotile peak (3.65Å), the essential ingredients of the autoclaved asbestos-cement product. It is, however, assumed that some substitutuion in the tobermorite lattice has taken place.

Differential Thermal Analysis and Uncombined Co(OH)

DTA tracings of all the laboratory autoclaved specimens in Fig. 6-4 show a small sharp endothermic deflection at 573 to 574C which characterizes the α–β quartz inversion of the unreacted silica flour present. The small endothermic peak at 823-825 C and the broad endotherm at around 650 C are due to the presence of chrysotile asbestos fibers. A small endotherm at 410-420 C is due to the presence of $Mg(OH)_2$, which occurs with asbestos fiber. Broad endotherms around 200-280 C, 650-700 C, and a sharp and high exothermic peak at 840-870 C indicate the presence of tobermorite and CSH_n binder compounds.[13] For the same curing duration a shift of the exotherm from 865 to 843 C was noted when the autoclaving pressure increased from 115 to 175 psig (8.0 to 12.3 kgf/cm^2). The only samples showing uncombined $Ca(OH)_2$ by DTA analysis were Samples 7 and 8 (pressure increased stepwise up to 150 psig (10.6 kgf/cm^2), in which a distinctive endothermic response at 510-515 C and a small one at 480 C were exhibited (last two tracings of Fig. 6-3).

The sensitivity of determining uncombined $Ca(OH)_2$ by DTA, using the equipment constants of the present analyzer, is not as high as by ASTM Method C 00 (Table 6-4). A calibration curve was employed for semiqualitative estimation of uncombined $Ca(OH)_2$ in the specimens. It was based on the area under the endotherm at 500-515 C versus percentage of $Ca(OH)_2$ added to a ground asbestos-cement sample with no detectable $Ca(OH)_2$.

Quantitative Estimation of the Components by Microscopic Point-Count Methdd

Point-count analysis was applied to a polished, etched asbestos-cement thin section to determine the quantity of components. Unhydrated cement and unreacted quartz grains are readily identifiable, whereas chrysotile asbestos may appear indistinguishable from the binder gel when viewed in polished sections. A photomicrograph of a polished section of a typical autoclaved asbestos-cement type is shown in Fig. 6-5. Therefore, gel and fiber were counted together in the specimen. For computations, the fiber component is assumed to be 18 percent of the total sample* since under those autoclaving conditions employed, no

* Fiber was 20 percent of the dry sample before hydration.

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visual interfacial reaction between cementitious material and chrysotile fiber takes place. (Interfacial reaction between saturated $Ca(OH)_2$ solution and asbestos was, however, detected by tracer technique in electron microscopic examinations of material autoclaved at 200 C for prolonged periods).

The results, listed in Table 6-5, show that the two-step autoclave curing with 5.5 hr at 115 psig (8.1 kgf/cm^2) and then 3 hr at 127 psig (8.9 kgf/cm^2) yielded the highest gel content and a low percentage of unhydrated cement. Other 8.5 hr underwater cured specimens showed a degree of hydration comparable with that of 100-psig (7.0 kgf/cm^2) autoclaved standard.

Porosity Measurement

The permaebility of wet-process asbestos-cement products is generally very low since they have been formed under vacuum pressure, or both. Porosity is one of the basic parameters that relates closely to mechanical and physical properties, so it gives an indication of the effectiveness of any experimental autoclave condition.

Pore spectra and the relative percentage distribution of several experimentally cured specimens are given in Fig. 6-6.

When the autoclaving pressure exceeds 135 psig (9.5 kgf/cm^2) as for the top sample in Fig. 6-6 porosity increases despite the use of underwater curing. Underwater curing in the range 127 to 135 psig (8.9 to 9.5 kgf/cm^2) spectra 3 and 4 from top) yielded impermeable products compared with the control. The fifth spectrum from the top, which underwent 8.5 hr underwater two-step curing, showed the least poroxity and highest percentage of fine pores of all the specimens tested.

From the evaluation of binder material and strength of the underwater cured specimens, the optimum condition is 8.5 hr at a pressure of 100 to 130 psig. (7.0 to 9.1 kgf/cm^2). A slow, stepwise pressure buildup using two steps, or pressure levels, (Sample 5 in Table 6-3) is preferred, i.e., a 5.5 hr first step at 115 psig (8.1 kgf/cm^2) followed by a 3 hr second step at 127 psig. (8.9 kgf/cm^2). Properties of the binders are very similar and often superior to those of the control cured 16 hr at 100 psig (7.0 kgf/cm^2).

APPLICATION TO COMMERCIAL PRODUCT

Water-spray autoclaving of 6-in (15-cm) Class 150 pipe with single step or two-step curing was tried under actual production conditions. The crushing strength of the pipe is plotted against single-step autoclaving time, as shown in Fig. 6-7. A strength increase of about 7 percent was achieved when single-step water-spray curing was carried out 9 hr at 115 psig (8.1 kgf/cm^2) which demonstrates the feasibility of employing water spray. A comparison of the

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strengths obtained in single-step and two-step curing with and without water spray is shown in Table 6-6.

The values shown are considered to give a reliable comparison between curing combinations because comparative pieces were taken from the same length of pipe. Experience has shown individual pipes to be uniform form one end to the other. Although a larger variation than might be expected can be seen between standard-cured pipes No. 2 and 3, for example, it must be remembered that the individual pipes were selected at random from commercial production, where the between-pipe variation is much larger than the within-pipe variation.

Products employing water-spray curing have consistently higher strength than those cured in the absence of water spray. The density change of asbestos-cement pipe is negligible.

SUMMARY

Mechanical, physical, and chemical properties of asbestos-cement products (both laboratory-prepared specimens and commercial asbestos-cement pipe) cured under various autoclaving conditions were evaluated.

The desired binder was essentially tobermorite and some CSH_n gel. Very small amounts of uncombined $Ca(OH)_2$ (usually less than 0.2 percent) were found in satisfactory autoclaved products. α-C_2SH, dicalcium-silicate alpha hydrate, and hydrated phases containing Al_2O_3, Fe_2O_3, and SO_3 were not detected.

Saturated steam-curing conditions were compared with superheated steam treatments. In regular saturated steam curing, long durations of about 16 hr at 100 to 110 psig (7.0 to 7.7 kgf/cm^2) are preferred. With increasing curing temperature and pressure, products formed under high saturated steam pressure ar are found to be more porous lower in strength, and to have an increasing amount of xonotlite.

Superheated steam environments should be avoided. Curing under such conditions produces binder that contains xonotlite, and has low strength and high porosity.

Autoclaving procedures that employ water spray to ensure saturated steam yield superior products in both strength development and reduced permeability. Meanwhile, the total autoclaving time can be reduced significantly from 16 hr to about 9 hr.

Underwater curing was used in the laboratory to achieve such conditions; but for commercial operation, a water-spray technique was developed to assure saturation.

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In underwater curing, or in the presence of water spray, the workable pressure range seems to be broader. Products can be cured at elevated pressures up to 135 psig (9.5 kgf/cm^2) with a slow rate of pressure buildup. The optimum conditions comprises two-step pressure curing with water spray: 5.5 hr at 115 psig (8.1 kgf/cm^2) followed by 3.0 hr at about 130 psig (9.1 kgf/cm^2). Binder characterization indicates the abundance of tobermorite and CSH_n gel, which is quite dense and impermeable, with 80 percent or more of the pore size diameters less than 0.04 microns.

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The author wishes to thank Mr. R. Zukoski for his assistance in making many of the measurements reported here, Mr. D. Bailey for the point-count determinations and microscopic examinations, Mr. T. Sopoci for the uncombined $Ca(OH)_2$ analysis by ASTM C 500, and Mr. R. H. Adams for the pilot plant evaluation of pipe.

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TABLE 6-1–CHEMICAL ANALYSIS OF PORTLAND CEMENT NATIONAL PIONEER, TYPE I CEMENT*

Components	Amount, %
Loss on ignition (1800 F)	0.4
SiO_2	21.1
Fe_2O_3	2.8
Al_2O_3	5.0
TiO_2	0.19
Mn_2O_3	0.005
CaO	64.7
MgO	3.2
Na_2O	0.18
K_2O	0.67
SO_3	1.92
	100.22

Probable composition	Amount, %
C_3S	60.0
C_2S	15.0
C_3A	8.5
C_4AF	9.0

* In addition to the ASTM C 150 requirements, the cement meets the following Johns-Manville requirements:

Fineness, specific surface (Blaine), cm^2/g 3600
Sulfur trioxide (SO_3), max., percent 2.0

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ND CEMENT
T*

Amount, %
0.4
21.1
2.8
5.0
0.19
0.005
64.7
3.2
0.18
0.67
1.92
100.22

Amount, %
60.0
15.0
8.5
9.0

e cement

3600
2.0

TABLE 6-2–EFFECT OF HIGH PRESSURE AUTOCLAVING, SUPERHEATED STEAM AND UNDERWATER CURING ON FLEXURAL STRENGTH OF ASBESTOS–CEMENT 3 x 8 x 1/8-IN. "SAG CAKE" SPECIMENS

Experimental Conditions						
Description	Temp., C	Saturated steam pressure, psig	Time hr	Product identified besides asbestos*	Flexural strength, psi	Change from control strength,%
	160	75	16.0	(T) + Q	3320	- 32.7
	170	100	16.0	T + (Q)	4940	(Control)
Saturated	181	135	8.5	T + (X) + (Q)	4620	- 6.5
	190	175	5.0	(T) + X + (Q)	2970	- 40.0
steam	200	210	16.0	(T) + X + (Q)	2930	- 40.7
	230	385	7.0	T† + X + Q	3050	- 38.2
cure						
Superheated	190	100	16.0	(T) + (Q) + (X)	2500	- 49.4
steam	200	100	7.0	T + (Q) + X	3860	- 21.8
cure	230	100	7.0	T + Q + (X)	3360	- 30.9
Saturated	170	100	16.0	T + (Q)	5080	+ 2.8
steam	175	115	5.5	T + Q	4600	- 6.8
cure	175	115	8.0	T + (Q)	4860	- 1.6
under water	179	127	8.5	T + (Q)	4910	- 0.6
	181	135	8.5	T + (Q)	5020	+ 1.6
	186	150	5.5	(T) + (X) + (Q)	4750	- 3.8
	186	150	8.5	T + (X) + (Q)	4620	- 6.4
	192	175	5.5	(T) + (X) + Q	4590	- 7.1

* symbols:

T = tobermorite gel binder
Q = quartz
X = xonotlite
() denotes minor quantity
† denotes trace quantity

(Strength determinations made on 4 to 6 samples of each.)

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TABLE 6-3–BINDER PHASE IDENTIFICATION OF ASBESTOS–CEMENT PRODUCTS BY X-RAY DIFFRACTION METHOD[a] AND STRENGTH PROPERTIES

Sample No.	Autoclaving conditions, psig-hr	Under water curing	Flexural strength, psi	Change from control strength %	Hydration product identified[a]	Peak intensity ratio 2.97/3.07 Å
1	100-16 (control	No	4940	----	T + G	0.77
9	100-16	Yes	5080	+ 2.8	T + G	0.81
2	115-5.5	Yes	4600	- 6.9	(T) + G	0.93
10	115-8.0	Yes	4860	- 1.6	T + (G)	0.77
11	127-8.5	Yes	4910	+ 0.6	T + G	0.88
12	135-8.5	Yes	5020	+ 1.6	T + G + (X)	0.68
3	150-5.5	Yes	4750	- 3.8	(T) + G + X	0.58
13	150-8.5	Yes	4620	- 6.5	(T) + (G) + X	0.50
4	175-5.5	Yes	4590	- 7.1	X + (T) + G*	0.47
5	115-5.5, 127-3[b]	Yes	5200	+ 5.2	T + G	0.76
6	115-5.5, 150-3[b]	Yes	5090	+ 3.0	T + G	0.77
14	100→ 127-8.5 (100-2, 110-2, 120-2, 127-2.5)[c]	Yes	5090	+ 3.0	T + G	1.09
15	100→ 135-8.5 (100-2, 110-2, 120-1.5, 130-1.5, 13501.5)[c]	Yes	4750	- 3.8	(T) + G	0.92
7	100→ 150-5.5 (100-1, 110-1, 120-1, 130-1, 140-1, 150-0.5)[c]	Yes	3870	- 21.6	T* + (G) + C	0.93
8	100→ 150-8.5 (100-1, 110-1, 120-1, 130-1, 140-1, 150-3.5)[c]	Yes	4550	- 7.9	T* + G + (C)	0.83

[a] Symbols:

T = tobermorite
X = xonotlite
G = CSH_n gel
C = $CaCO_3$ (Calcite)
() () = denotes minor quantity
* denotes trace quantity

[b] Stepwise curing, 5.5 hr at 115 psig, followed by 3 hr at the higher pressure indicated.

[c] Stepwise curing, Data may be read as follows, e.g., for Sample 14: Pressure steps from 100 to 127 psig for a total of 8.5 hr, made up of individual steps of 100 psig for 2 hr, and finally 127 psig for 2.5 hr.

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TABLE 6-4–ANALYSIS OF UNCOMBINED $Ca(OH)_2$

Sample No.	Autoclaving conditions		Uncombined $Ca(OH)_2$	
	Psig- hr	Under water	DTA method	ASTM method C 500
1	100-16	No	0	0.2
9	100-16	Yes	0	0.2
2	115-5.5	Yes	0	0.2
10	115-8.0	Yes	0	0.2
11	127-8.5	Yes	0	0.2
12	135-8.5	Yes	0	0.2
3	150-5.5	Yes	0	0.2
13	150-8.5	Yes	0	0.1
4	175-5.5	Yes	0	0.1
5	115-5.5, 127-3[a]	Yes	0	0.2
6	115-5.5, 150-3[a]	Yes	0	0.2
14	100→127–8.5[b]	Yes	0	0.2
15	100→135–8.5[b]	Yes	0	0.1
7	100→150–5.5[b]	Yes	1.5	1.7
8	100→150–8.5[b]	Yes	0.5	0.8

[a] Stepwise curing, 5.5 hr at 115 psig, followed by 3 hr at the higher pressure indicated.

[b] Stepwise curing at various pressures from 100 psig to pressure at right of arrow for period of time indicated by number after dash. (For further details, see Table 6-3.)

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TABLE 6-5–MICROSCOPIC POINT–COUNT DETERMINATION OF THE COMPOSITION OF ASBESTOS–CEMENT PRODUCTS

Autoclaving conditions (psig - hr)	Underwater curing	Composition		
		Unhydrated cement, %	silica, %	Gel and fiber, %
100-16	No1	2.8	7.4	89.8
100-16	Yes	0.9	8.1	91.0
115-5.5	Yes	3.0	7.8	89.2
127-8.5	Yes	2.6	8.3	89.0
135-8.5	Yes	3.2	8.8	88.0
150-8.5	Yes	2.6	6.8	90.6
115-5.5, 127-3	Yes	1.7	5.1	93.2
100–>150-8.5 hr Stepwise*	Yes	3.2	6.0	90.6

* For details see Table 6-3, Sample 8.

TABLE 6-6–EFFECT OF WATER SPRAY AND AUTOCLAVE CONDITIONS ON CRUSHING STRENGTH OF ASBESTOS–CEMENT PIPE (6-IN., CLASS 150)

No.	Total time	Water spray	Experimental autoclaving conditions		Density, pcf	Crushing strength		Change in strength of exp vs. std, %
			First step, psig-hr	Second step, psig-hr		Experimental curing, psi a	Standard curing, psi a	
1	8.5	Yes	115-6.5	127-2	110.7	8660	8520	+ 2
2	8.5	Yes	115-5.5	137-3	109.1	8970	8770	+ 2
3	8.5	Yes	115-5.5	132-3	111.2	8010	7360	+ 9
								+ 5

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DETERMINATION OF
EMENT PRODUCTS

Composition		
ated %	silica, %	Gel and fiber, %
	7.4	89.8
	8.1	91.0
	7.8	89.2
	8.3	89.0
	8.8	88.0
	6.8	90.6
	5.1	93.2
	6.0	90.6

TABLE 6-6 EFFECT OF WATER SPRAY AND AUTOCLAVE CONDITIONS ON STRENGTH OF ASBESTOS–CEMENT PIPE (6-IN., CLASS 150)

No.	Total time	Water spray	Experimental autoclaving conditions		Density, pcf	Crushing strength		Change in strength of exp vs. std, %
			First step, psig-hr	Second step, psig-hr		Experimental curing, psi [a]	Standard curing, psi [a]	
1	8.5	Yes	115-6.5	127-2	110.7	8660	8520	+ 2
2	8.5	Yes	115-5.5	137-3	109.1	8970	8770	+ 2
3	8.5	Yes	115-5.5	132-3	111.2	8010	7360	+ 9
4	8.5	Yes	115-5.5	127-3	110.3	8030	7640	+ 5
5	8.5	Yes	115-5.5	122-3	110.9	8720	8660	+ 1
6	7.5	Yes	115-5.0	132-2.5	110.1	6730	6210	+ 8
7	8.5	Yes	115-4.5	127-4	108.2	7410	7100	+ 4
8	7.5	Yes	115-4.5	132-3	112.0	9380	9000	+ 4
9	7.5	Yes	115-4.0	132-3.5	110.3	8850	8710	+ 2
10	9.5	Yes	132-9.5	----	113.7	10220	9770	+ 5
12	16.0	No	100-16	Control	110.8	----	8170 [c]	Standard
13	8.5	No	115-6.5	127-2	111.2	7770	7860	- 1
14	8.5	No	115-5.5	137-3	110.9	7320	7360	- 0.5
15	8.5	No	115-5.5	127-3	110.3	7420	7640	- 3
16	8.5	No	115-5.5	122-3	110.9	8190	8660	- 7
17	7.5	No	115-5	132-2.5	109.9	6080	6210	- 2
18	7.5	No	115-4.5	132-3	110.6	8830	9000	- 2
19	7.5	No	115-4	132-3.5	----	8180	8710	- 6
20	9.5	No	165-9.5	----	----	8010	8520	- 6
21	9.5	No	155-9.5	----	----	7940	8660	- 8
22	9.5	No	135-9.5	----	----	7500	7640	- 2
23	9.5	No	125-9.5	----	----	6870	7100	- 3
24	9.5	No	115-9.5	----	----	7450	7360	+ 1

[a] Cured as in columns 4 and 5
[b] Cured 16 hr at 100 psig
[c] Average value of all control runs.

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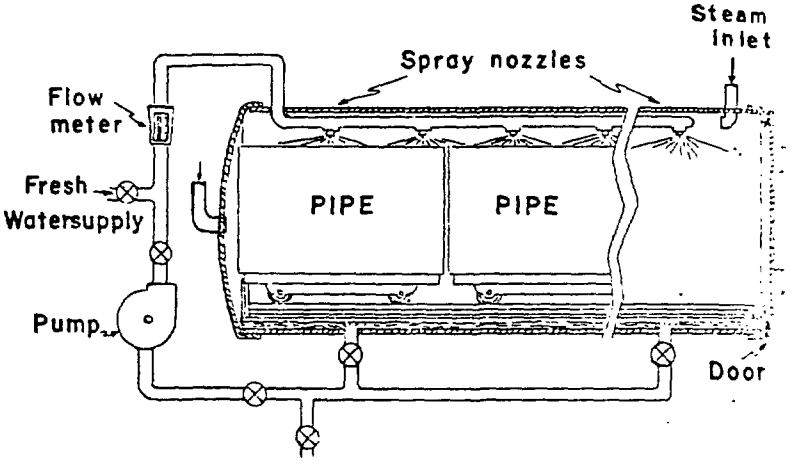
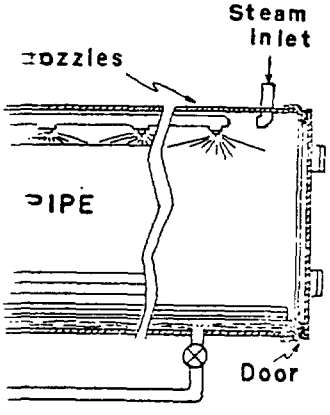


Fig. 6-1—Diagram of autoclave equipped with water spray

CTD000988



h water spray

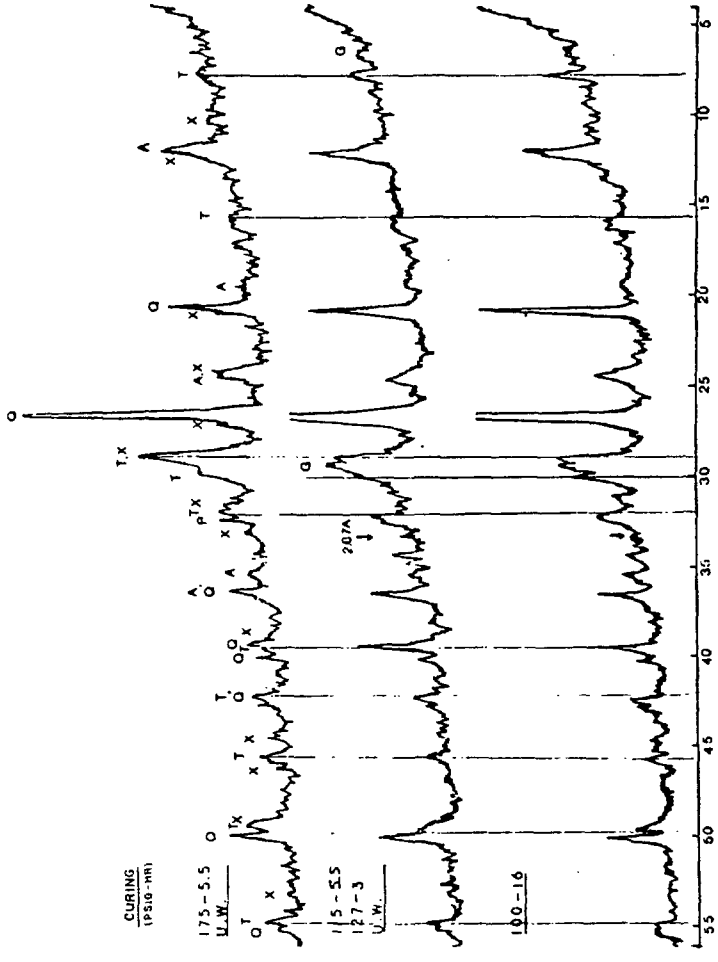


Fig. 6-2–X-ray diffraction patterns of some autoclaved asbestos-cement products: in 2θ = 4 to 56 deg range; Q-quartz, A-chrysotile asbestos, G-CSH_n, T-tobermorite, X-xonolite, P-unhydrated portland cement, U.W.-underwater curing

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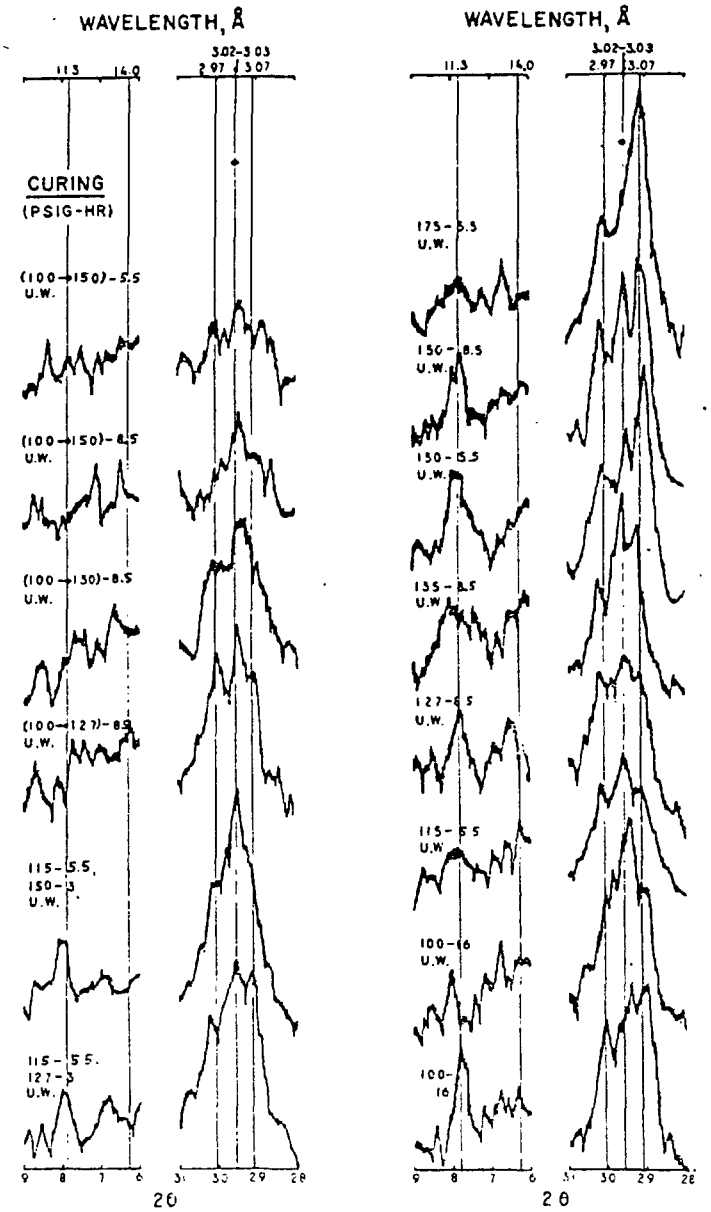
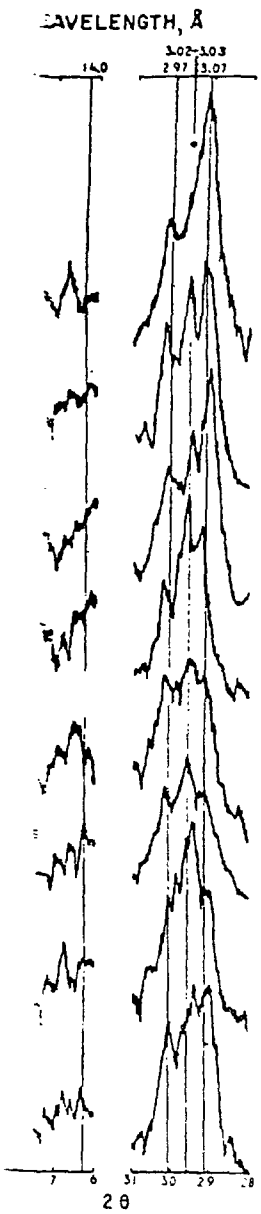


Fig. 6-3—X-ray diffraction patterns of some autoclaved asbestos-cement products: in 2θ = 6 to 9 deg and 28 to 31 deg range; U.W. - underwater curing

CTD000990



autoclaved asbestos-
deg and 28 to 31 deg range;

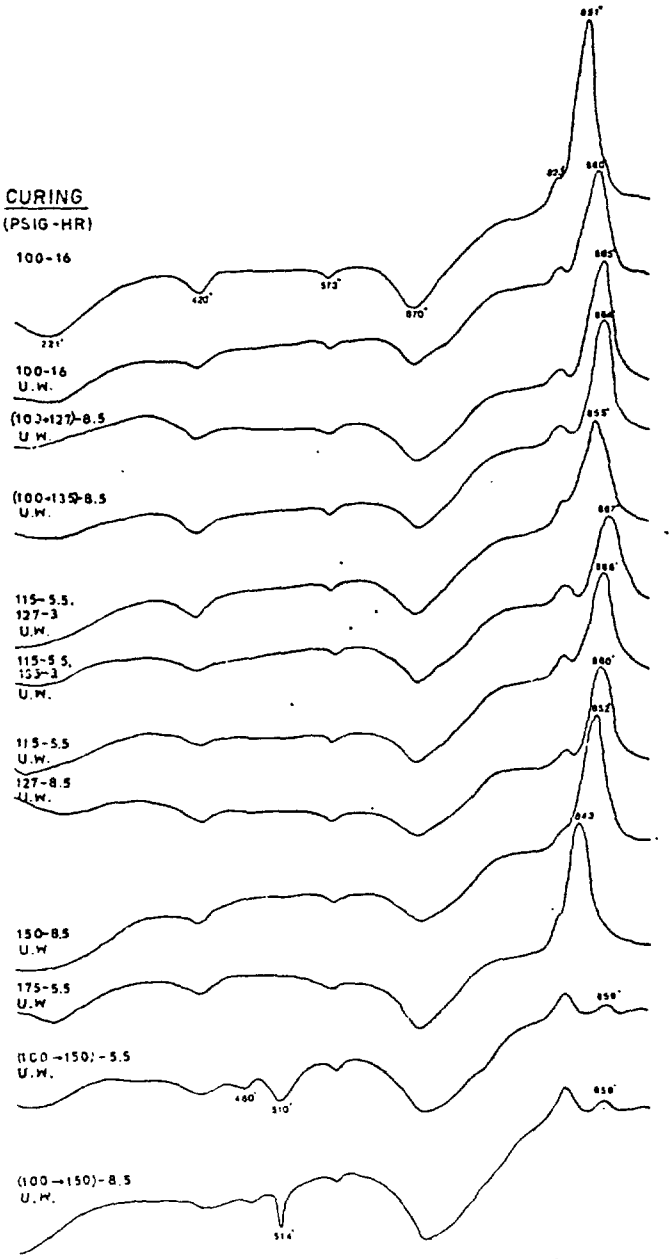


Fig. 6-4—DTA tracings of asbestos-cement compositions: U.W. underwater curing

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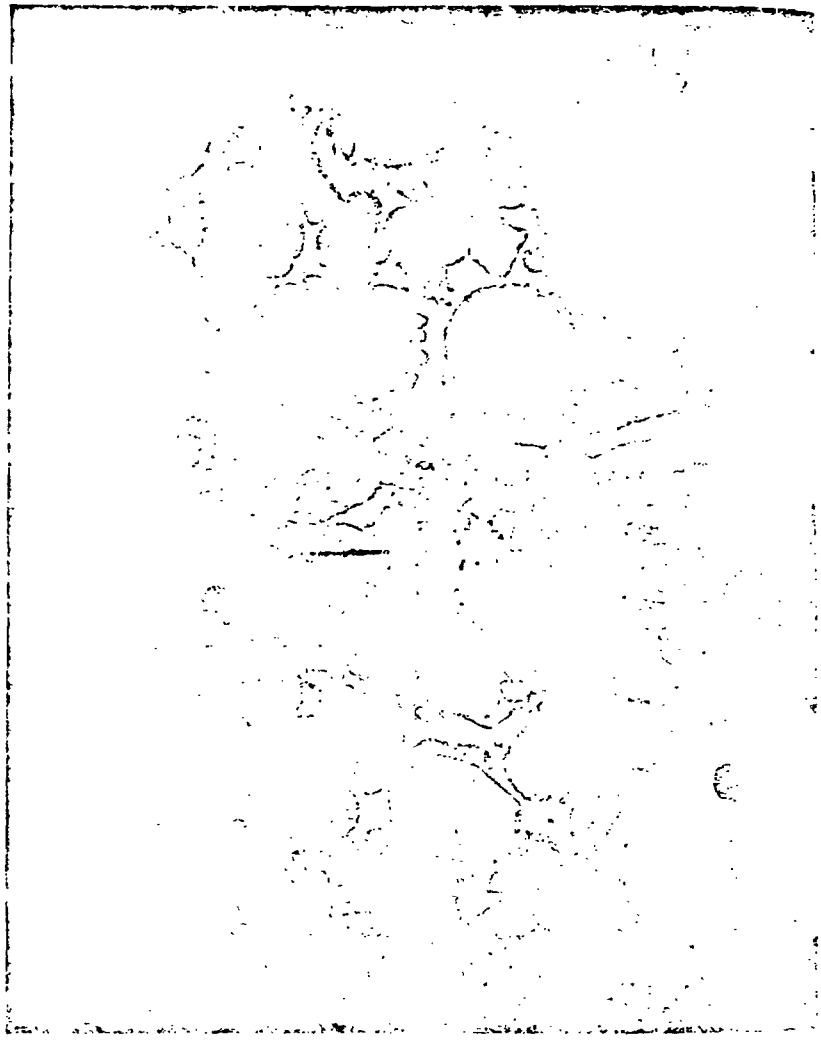

Fig. 6-5—Polished section of autoclaved asbestos-cement pipe as viewed by reflected light; crystals which show cross hatching are C_2S; smooth gray crystals are C_3S; "Q" deontes quartz grains; "C", chrysotile; magnification, 800 X

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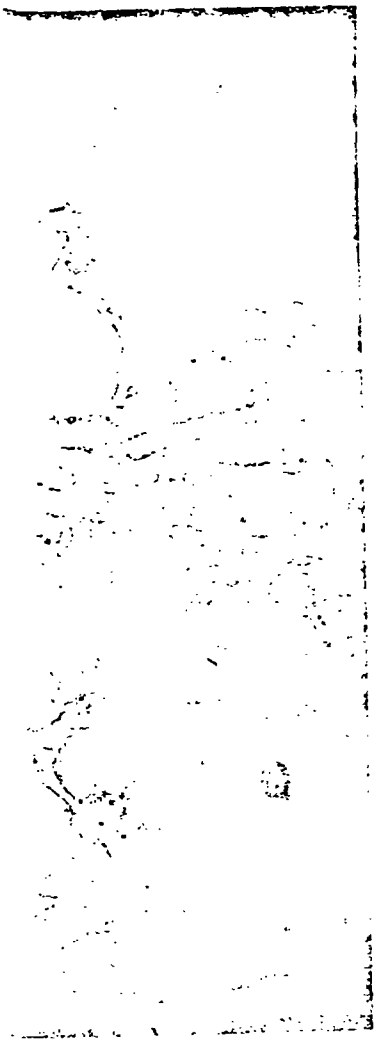

s-cement pipe as viewed by re-
s hatching are C_2S; smooth gray
grains, "C", chrysotile; magnifica-

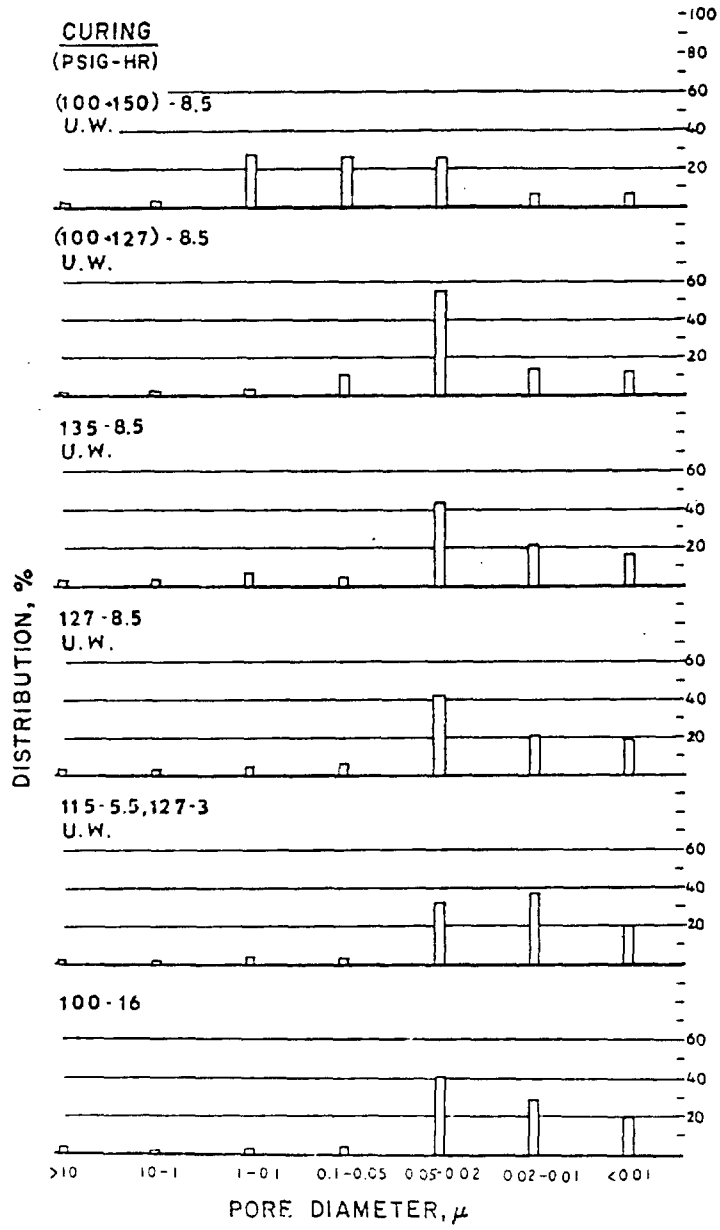

Fig. 6-6--Pore spectra of autoclaved asbestos-cement products; U.W. -underwater curing.

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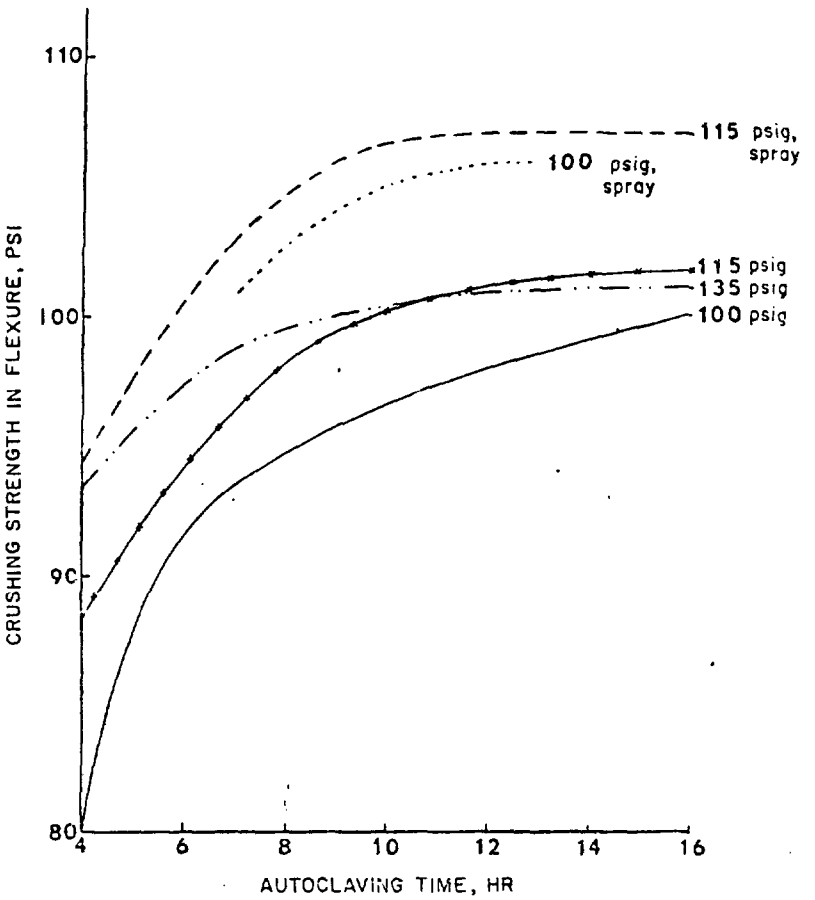

Fig. 6-7—Effect of curing conditions on crushing strength of asbestos-cement pipe (6-in. Class 150)

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