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DURABILITY OF CEMENT CONCRETE IN SULFATE ENVIRONMENT

CALIFORNIA UNIVERSITY

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DEPARTMENT OF CIVIL ENGINEERING

DURABILITY OF CEMENT CONCRETE IN SULFATE ENVIRONMENT

by

P. K. MEHTA

and

R.B. WILLIAMSON

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Abstract

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Introduction

Due to low cost, ready availability and wide applicability, portland cement concrete has become the work horse of construction industry today. Therefore, it is important that the factors capable of adversely affecting the service life of concrete are well understood by material scientists and engineers. In general, for the low concentration of sulfate ions usually found in many natural sulfate waters such as sea water, structures made with low permeability concrete should last indefinitely. However, sulfate water of concentrations higher than 0.2% SO_3 are capable of entering into destructive chemical reactions with the cementing constituents of portland cement concrete. In waters containing less than 0.2% SO_3, the possibility of sulfate attack is enhanced if the concrete is permeable through defective mix design, improper placement, or has subsequently cracked due to rusting of the reinforcement or other causes.

Destruction of concrete by sulfate waters can follow either or both of the two courses: (a) Cracking due to formation of a sulfoaluminate hydrate, (b) surface softening due to formation of gypsum. The first type of attack is usually prevalent in concretes made with portland cements containing significant proportion of reactive aluminate, i.e. greater than 3 percent $3\ CaO{\cdot}Al_2O_3$.

Several investigators including Atwood and Johnson (1), Stanton (2), and Miller and Manson (3) have clearly established that the susceptibility of portland cement concrete to attack by sulfate waters is increased with increasing permeability of concrete and with higher tricalcium aluminate content of the portland cement used. The reactive aluminate present in portland cement when attacked by sulfate ions can form the calcium sulfoaluminate hydrate, $3\ CaO \cdot Al_2O_3 \cdot 3\ CaSO_4 \cdot 32\ H_2O$, also called ettringite. A large quantity of $Ca(OH)_2$ is present in hydrated portland cements due to hydration of its major compounds, namely, $3\ CaO \cdot SiO_2$ and $2\ CaO \cdot SiO_2$ Recently, Mehta (4) has hypothesized that ettringite formed in the presence of $Ca(OH)_2$ is colloidal, and that the formation of colloidal ettringite in hardened concrete can lead to disruptive expansion.

The second type of sulfate attack, also called the acid type attack. is due to formation of gypsum. Generally, all sulfate solutions are somewhat acidic in nature. On prolonged storage to acidic conditions, the $Ca(OH)_2$ present in hydrated portland cement converts into gypsum.

Failures of concretes exposed to sulfate water have warranted numerous laboratory and field investigations, some of which are as follows. Feld (5) reported that in 1955 after 21 years of use, as a result of chemical action of sea water on concrete, about 70% of the 2500 concrete piles of the James river bridge at Newport News, Virginia, were found in need of repair. The repairs and replacement job cost $1.4 million. According to him, a similar deterioration was found in the precast concrete piles driven in 1932 near Ocean City, New Jersey, where by 1957 the 22-inch dimension had become reduced to 12-inch, and some 750 piles had to be repaired. Wakeman et al (6) reported that in 1925 after 12 years exposure to sea water, precast reinforced concrete piles in the Los Angeles Harbor showed extensive cracking and spalling.

One of the earliest recorded investigations in the United States on concrete test blocks exposed to sea water environment is due to Hughes as quoted by Wakeman et al (6). In 1905 Hughes supervised the making of eighteen 69" x 69" x 42" unreinforced concrete blocks with 6 different brands of portland cements (some containing 12-15% $3\ CaO \cdot Al_2O_3$) and three different types of concrete mixtures. These blocks were placed in a submerged position in the San Pedro breakwater of the Los Angeles Harbor. In 1932, after 27 years of seawater exposure, they were removed for inspection. Cores were taken for compressive strength testing and for chemical examination, and the blocks were returned to their original location. The test results showed that there was no evidence of disintegration and SO_3 take-up by concrete, however, some magnesia from the sea water had replaced a part of the lime present in the cements.

Present Investigation on the Concrete Test Blocks of the Los Angeles Harbor

In 1972, after 67 years of exposure to sea water, the writers arranged the inspection of these test blocks.* A 25-ton crane situated on a naval vessel hoisted the 7 ton blocks on board where 4-inch diameter cores were taken from the 6 blocks which were the only ones that could be retrieved from water depths varying between 10 to 30 feet at mean low tide. Five of the six blocks belonged to less permeable concrete Mix A (1:2:4), while only one belonged to more permeable concrete Mix B (1:3:6). A visual inspection of the blocks showed that Mix A concrete blocks were in excellent condition, with all edges and corners hard and sharp; whereas Mix B block, which was covered heavily with vegetable and animal marine growth, had dimensional

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* U.S. Naval Civil Engineering Laboratory, Port Hueneme, California collaborated with the University of California in some of the tests.

reduction from 1 to 3 inches, softer surface, and dull edges and corners. The possibility exists that some of the test blocks made with Mix B might have disintegrated during the period 1932-1972. Due to wide deviations in the compressive strength data from different cores from the same block, it was considered inconclusive.

X-ray diffraction analyses of the mortars obtained from crushed concrete cores showed that the cementing constituents remained essentially unaltered in Mix A concretes, but brucite, gypsum, ettringite, aragonite, and hydrocalumite, $C_{16}Al_{18}(OH)_{54}CO_3 \cdot 21\ H_2O$, were present in Mix B concrete. The presence of aragonite and hydrocalumite showed that in addition to the acidic attack by sulfate (i.e. conversion of lime into gypsum by $MgSO_4$ of sea water), there was additional acidic attack by carbonation. An upper core from concrete of Mix B showed that virtually no $Ca(OH)_2$ and tobermorite (the calcium silicate hydrate which is the binding constituent in hydrated portland cements) were present. This is consistent with the following chemical reactions proposed by Lea (7) regarding acid-type of sulfate attack:

$$Ca(OH)_2 + MgSO_4 \cdot 7\ H_2O = Mg(OH)_2 + CaSO_4 \cdot 2\ H_2O$$

$$3\ CaO \cdot 2\ SiO_2 \cdot 3\ H_2O + 3\ MgSO_4 \cdot 7\ H_2O = 3\ Mg(OH)_2 + CaSO_4 \cdot 2\ H_2O + 2\ SiO_2 \cdot H_2O.$$

Idorn (8) reported similar surface softening of Oddesun Bridge piers concrete exposed to sea water The chemical alternation of the cement paste was associated with decomposition of $Ca(OH)_2$ and tobermorite, and was accompanied by the formation of gypsum, brucite, ettringite, and aragonite. This is also illustrated in Fig. 1 which is based on an unpublished study by Mehta on exposure of 3-inch diameter concrete cylinders made with $3\ CaO \cdot SiO_2$ for 6 years, to a 10% $MgSO_4 + Na_2SO_4$ solution. The exposure caused no expansion, but significant disintegration and softening of the surfaces.

Recent Reports on Sulfate Attack

Gjorv (9) reported a 30-year study on 2500 concrete test blocks stored in sea water near Trondheim Harbor, Norway. In this study, 18 different kinds of cements, and water-cement ratios from 0.55 to 0.65 were employed to make (1 : 3.08 : 3.29) concrete mixtures. Concrete made with high alumina cement, super-sulfated cement, and blast furnace slag portland cement were found to be unaffected. Concretes made with portland cements were affected; those made with ordinary portland cements (7-11 percent 3 $CaO \cdot Al_2O_3$) were severely affected, but the ones made with sulfate-resisting portland cements (0-3 percent 3 $CaO \cdot Al_2O_3$) were also significantly affected. During the immersion period 1-30 years, the flexural strengths of concretes made with sulfate-resisting portland cements dropped from about 1350 psi to 900 psi. Addition of trass or pozzolan generally improved the durability of the portland cement concretes. Also, Kalousek et. al (10) predict a life expectancy of less than 50 years for majority of concretes made with sulfate-resisting portland cements which contain no pozzolanic additions. Apparently, the presence of a low 3 $CaO \cdot Al_2O_3$ content by using a sulfate-resisting portland cement, does not offer adequate protection against long term sulfate attack of the acid-type. Thus, reduction of available $Ca(OH)_2$ in hydrated cement by adding pozzolan to concrete mixture was found helpful by both Gjorv (9), and Kalousek et al (10).

Swenson (11) reported premature deterioration of high quality concrete cribbings in the shaft of a potassium sulfate mine in Saskatchewan, Canada. The concrete was made with a sulfate-resisting portland cement of relatively high 3 $CaO \cdot SiO_2$ content. The failure through surface softening was apparently due to the acid-type of sulfate attack.

On the basis of above observations with regard to long-time durability of concrete exposed to sulfate water, it appears that in addition to low permeability of concrete, and low 3 $CaO \cdot Al_2O_3$ content of the portland cement, it is useful to reduce the available $Ca(OH)_2$ content in the hydrated cement. Consequently, along with low 3 $CaO \cdot Al_2O_3$ portland cements, blast furnace slag cements and pozzolan cements (including trass cement) are being marketed for sulfate-resistant applications.

Laboratory Tests for Sulfate Resistance

In spite of a large number of methods which are available for testing sulfate resistance of cements, there is general agreement that none of the present methods is entirely satisfactory.

Many test methods are based on expansion measurements of cement mortar or cement concrete prisms immersed in sodium or magnesium sulfate solutions. It is obvious that the expansion measurements ignore the surface deterioration type of sulfate attack. Secondly, due to the slow rate of diffusion of sulfate ions into the specimens stored in stagnant solutions, it takes several months, and often several years before significant expansions are discernible to indicate the vulnerability of a particular cement type to sulfate attack. Some acceleration in sulfate attack is realized by using concentrated sulfate solutions, but from practical standpoint, these methods are still considered slow.

The ASTM method (12) involves determination of expansion in water of 1 x 1 x 10 in. mortar prisms prepared with cement mixed with additional gypsum so that the SO_3 content of cement is 7 percent. Although it is quicker than the sulfate solution immersion methods, obviously, again the method is aimed at separating the high 3 $CaO \cdot Al_2O_3$ portland cements from the low 3 $CaO \cdot Al_2O_3$ portland cements by virtue of the magnitude of expansion caused by ettringite formation. Thus, the method ignores the long-time acid type of attack by sulfate waters on the available $Ca(OH)_2$ in a hydrated cement. It is also obvious that the method is not applicable to slag cements, pozzolan cements, supersulfated cements, and high alumina cements, which also find application in sulfate resistant concretes, and which are not usually susceptible to sulfate expansion phenomenon.

A New Test Method For Sulfate Resistance

Ideally, a test method for evaluating sulfate resistance should meet with the following requirements:

1. It should be simple and short. To be of practical value to the construction industry the testing should be complete within a few weeks.
2. It should be applicable to all types of sulfate resistant cements, i.e. portland cements, slag cements, possolan cements, high alumina cement, supersulfated cements, etc.
3. It should determine the susceptibility of a cement to both types of sulfate attack, i.e. ettringite-formation-and-expansion-type attack as well as gypsum-formation-and-surface-deterioration-type attack. The second type of attack can be ascertained by strength measurements

In collaboration with Gjorv, one of the writers (Mehta) has developed a new method for testing sulfate resistance. The method is applicable to all types of cements, and is based on the following concepts:

(a) Acceleration of sulfate attack can be realized if the tests are planned to take expansion measurements at age 7, 14, and 28 days, and to conduct strength measurements at age 14 and 28 days.

Results and Discussion

Figures 2 and 3 show photographs of cement paste cubes exposed to sulfate solutions for 28 days. Fig. 4 shows photographs of ordinary portland cement paste prisms at age 7, 15 and 28 days. Figs. 5 and 6 show changes in compressive strength of cubes with age of immersion in sulfate solutions for gypsum, and for sodium sulfate solutions respectively. Fig. 7 shows the expansion data for all the 5 cements for both types of sulfate exposure.

At 14th day of immersion, some surface deterioration and cracking was noticed in the ordinary portland cement cubes in both the sulfate solutions. The cubes exposed to gypsum solution lost about 40% of their original strength, whereas the cubes exposed to Na_2SO_4 solution lost about 60% of their original strength. In both cases the specimens were badly cracked and showed a great loss in compressive strength at 28th day of immersion. Similarly, the bending and cracking of prisms (Fig. 4) was associated with over 0.3% expansion in gypsum solution and over 0.5% expansion in sodium sulfate solution at 14th day of immersion. Thereafter, the expansion measurements were discontinued due to cracking of the prisms during the immersion period 14-28 days. X-ray diffraction analyses of the samples from the deteriorated surfaces showed presence of significant amounts of ettringite, and reduced $Ca(OH)_2$ content.

Regarding the sulfate resisting portland cement (zero-3 $CaO{\cdot}Al_2O_3$), the specimens did not exhibit significant strength loss and expansions during the testing period in either of the two sulfate solutions. Consider-

After normal curing in the molds for 4 hours, the specimens were moist cured at 50°C for additional 20 hours before demolding. Subsequently, the moist curing at 50°C was continued until age 7 days. This accelerated curing procedure ensured that the compressive strength of specimens from all types of cements were similar before immersion in sulfate solution at age 7 days, and at the same time without changing the nature of hydration products which would have formed by normal curing. Thus, the average compressive strengths before sulfate immersion ranged from 450 to 550 K_{gf}/cm^2

In this preliminary study two types of sulfate solutions were investigated, namely, a saturated gypsum solution (0.12% SO_3), and an approximately 4.0% Na_2SO_4 solution (2.1% SO_3). The measured pH value of both the solutions was about 6. To ascertain a reasonably acidic sulfate environment during the test, Bromothymol Blue indicator was added to the solutions. In the presence of this indicator, a solution with 6.0 or less acidity should remain yellow, whereas it turns blue during the pH range 6.0-7.6. The bulk solutions containing the indicator were acidified with a few drops of sulfuric acid to give a starting pH of slightly under 6.0.

After the 7-day curing period, initial length measurements were made and a set of six prisms were stored in each of the two solutions. In the same container, a set of cement paste cubes (10 for each test) meant for strength measurements were stored. Separate plastic containers were used for each of the 5 cements. In order to simulate flowing water conditions, the containers were stored on a vibrating table, and were continuously subjected to light vibration. At 24 hour intervals, a 1 N solution of sulfuric acid was added slowly to the sulfate solutions until the blue coloration changed back to yellow. This procedure ensured that the specimens were always exposed to acidic sulfate solutions of pH about 5.5 to 7.5. It was planned to take expansion measurements at age 7, 14, and 28 days, and to conduct strength measurements at age 14 and 28 days.

Results and Discussion

Figures 2 and 3 show photographs of cement paste cubes exposed to sulfate solutions for 28 days. Fig. 4 shows photographs of ordinary portland cement paste prisms at age 7, 15 and 28 days. Figs. 5 and 6 show changes in compressive strength of cubes with age of immersion in sulfate solutions for gypsum, and for sodium sulfate solutions respectively. Fig. 7 shows the expansion data for all the 5 cements for both types of sulfate exposure.

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Regarding the sulfate resisting portland cement (zero-3 $CaO \cdot Al_2O_3$), the specimens did not exhibit significant strength loss and expansions during the testing period in either of the two sulfate solutions. Considerable volume of sulfuric acid was, however, needed to neutralize the excessive lime which was being continuously leached out of the specimens made with this cement. Therefore, under more severe acidic conditions such as under the combined effect of sulfate and carbonate attack, this cement may be vulnerable.

The test data regarding response to sulfate attack of portland slag cements confirms other field and laboratory studies in that the susceptibility to sulfate attack varied with the slag content of the cement. Locher(14) reported that irrespective of the 3 $CaO \cdot Al_2O_3$ content of the portland clinker, portland slag cements are stable to sulfate attack if they contain at least 65% granulated blast furnace slag. In this investigation, the cement containing 30% slag (German, EPZ) showed significant strength loss and expansion during 28 days of immersion in both the sulfate solutions, although the deterioration was somewhat at a slower rate in the gypsum solution. On the contrary, both the strength and the expansion data show that the cement containing over 70% slag exhibited excellent resistant to sulfate attack. Furthermore, due to no free $Ca(OH)_2$ present in it (against about 13-15% free lime present in the 7-day cured pastes of both the ordinary portland cement and the sulfate resisting portland cement), hardly any sulfuric acid was needed to maintain the acidic pH of the immersion solutions used for the 70% slag cement investigation. Although both the zero-3 $CaO \cdot Al_2O_3$ sulfate resisting portland cement, and the 70% slag portland cement were found equally resistant to sulfate attack in this investigation, it appears desirable from the standpoint of long-time durability to use the latter for applications where acidic conditions may be present simultaneously.

The 20% trass cement (representing a portland pozzolan cement) did not exhibit adequate sulfate resistance in this test. The attack was at a slower rate in the gypsum solution, since at 28th day of sulfate exposure there was about 40% strength loss in the gypsum solution as compared to about 70% strength loss in the Na_2SO_4 solution. Thus the test confirmed the theoretical and field data that the type of pozzolan used and its proportion in this cement were not adequate for providing protection against sulfate attack.

In conclusion, it appears that the above described experimental procedure is capable of evaluating the chemical resistance of all types of cementitious materials to attack by sulfate solutions usually occurring in nature. The technique is quick and simple. It may need further modifications before finding final acceptance by the construction industry, but at least certain new ideas are advanced for solving the problems which have so far frustrated the development of a generally acceptable, quick, and reliable physical test of sulfate resistance of cements.

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CHEMICAL ANALYSES OF CEMENTS, PERCENT

	No. 1 NORWEGIAN ORDINARY PORTLAND	No. 2 GERMAN SULFATE RESISTANT	No. 3 GERMAN EPZ (30% SLAG)	No. 4 GERMAN HOZ(>70% SLAG)	No. 5 GERMAN TRASS (>20% TRASS)
SiO_2	20.60	20.50	23.50	28.88	21.89
Fe_2O_3	2.68	5.83	1.94	0.61	3.22
Al_2O_3	4.97	3.53	7.63	12.00	6.39
CaO	65.12	65.31	58.23	47.68	51.46
MgO	1.50	0.80	2.74	4.24	1.42
SO_3	2.62	2.91	2.68	2.61	3.04
INSOLUBLE RESIDUE	0.20	0.10	0.66	0.75	10.40
LOSS ON IGNITION	1.30	1.34	2.21	1.54	4.03
C_3A	8.6	0	-	-	-

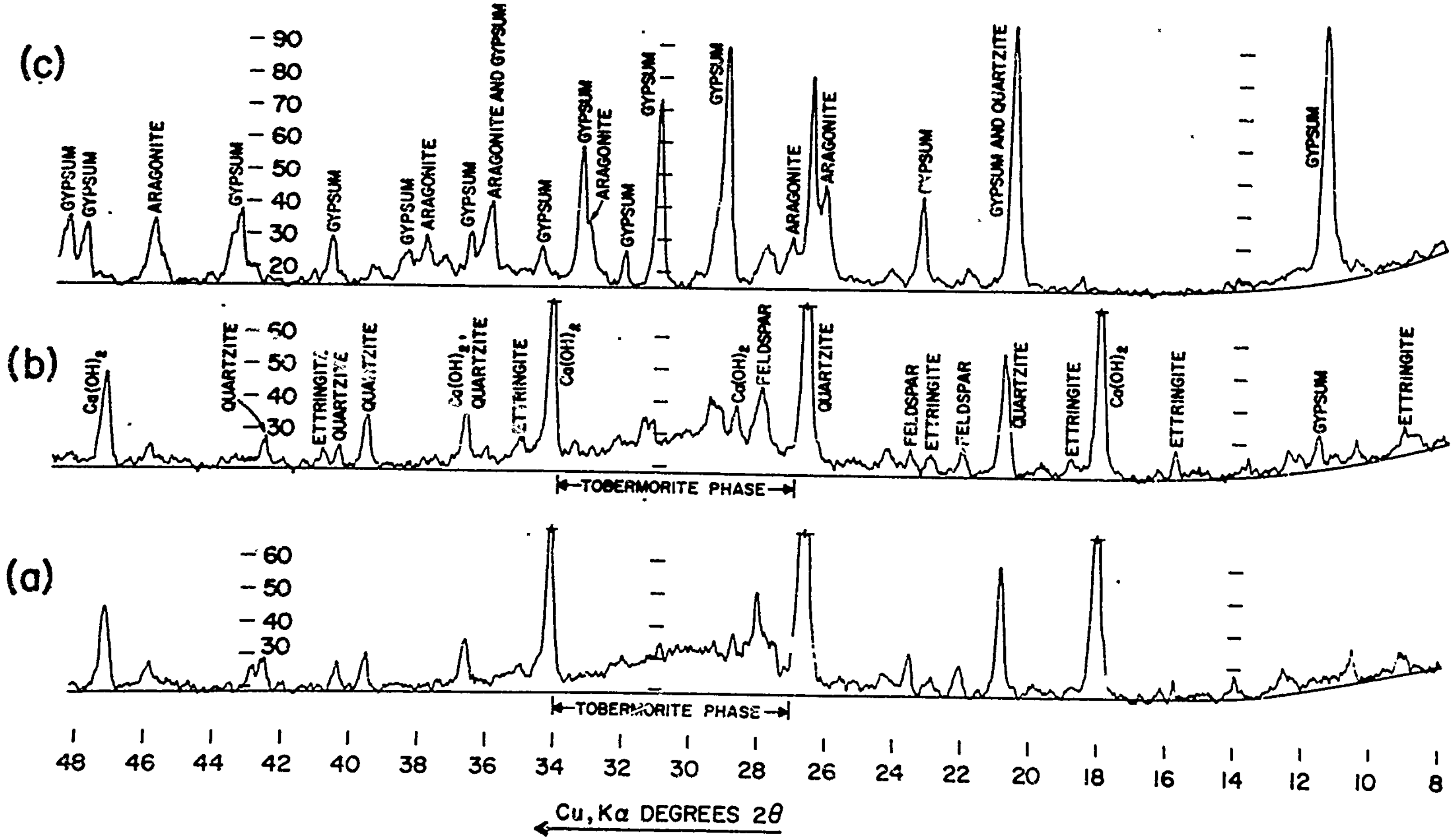

FIG. 1. X-RAY DIFFRACTION ANALYSES OF 3 $CaO \cdot SiO_2$ CONCRETE SAMPLES EXPOSED FOR 6 YEARS TO A 10% SOLUTION OF $MgSO_4 + Na_2SO_4$:

(A) INTERIOR SAMPLE, (B) ONE-INCH FROM THE SURFACE, (C) EXTERIOR SAMPLE.

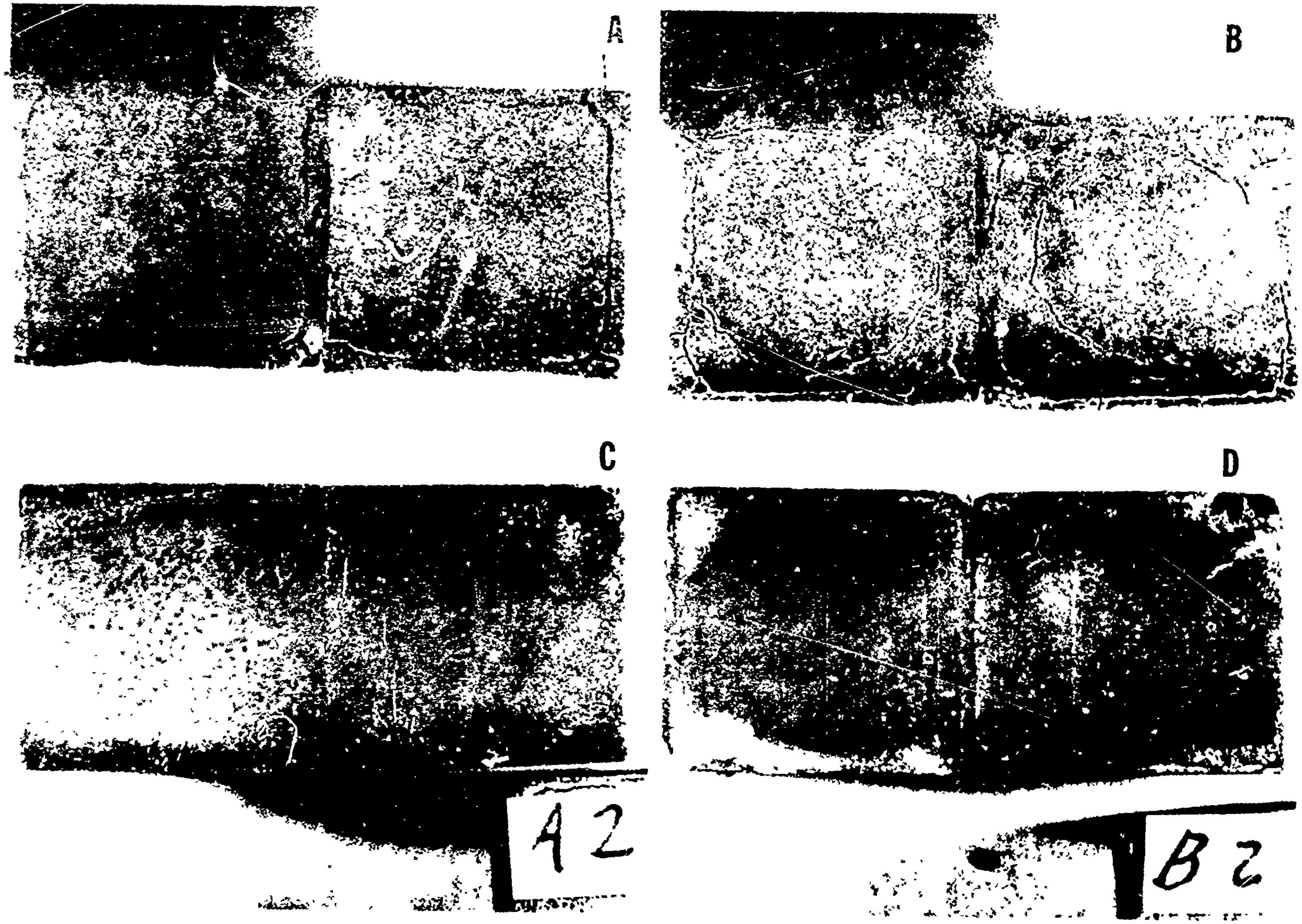

Fig. 2. Photographs of Cement Paste Cubes Immersed in Sulfate Solutions for 28 Days.
A. Ordinary Portland Cement in Gypsum Solution (top, left).
B. Ordinary Portland Cement in Sodium Sulfate Solution (top, right).
C. Sulfate-Resisting Portland Cement in Gypsum Solution (bottom, left).
D. Sulfate-Resisting Portland Cement in Sodium Sulfate Solution (bottom, right).

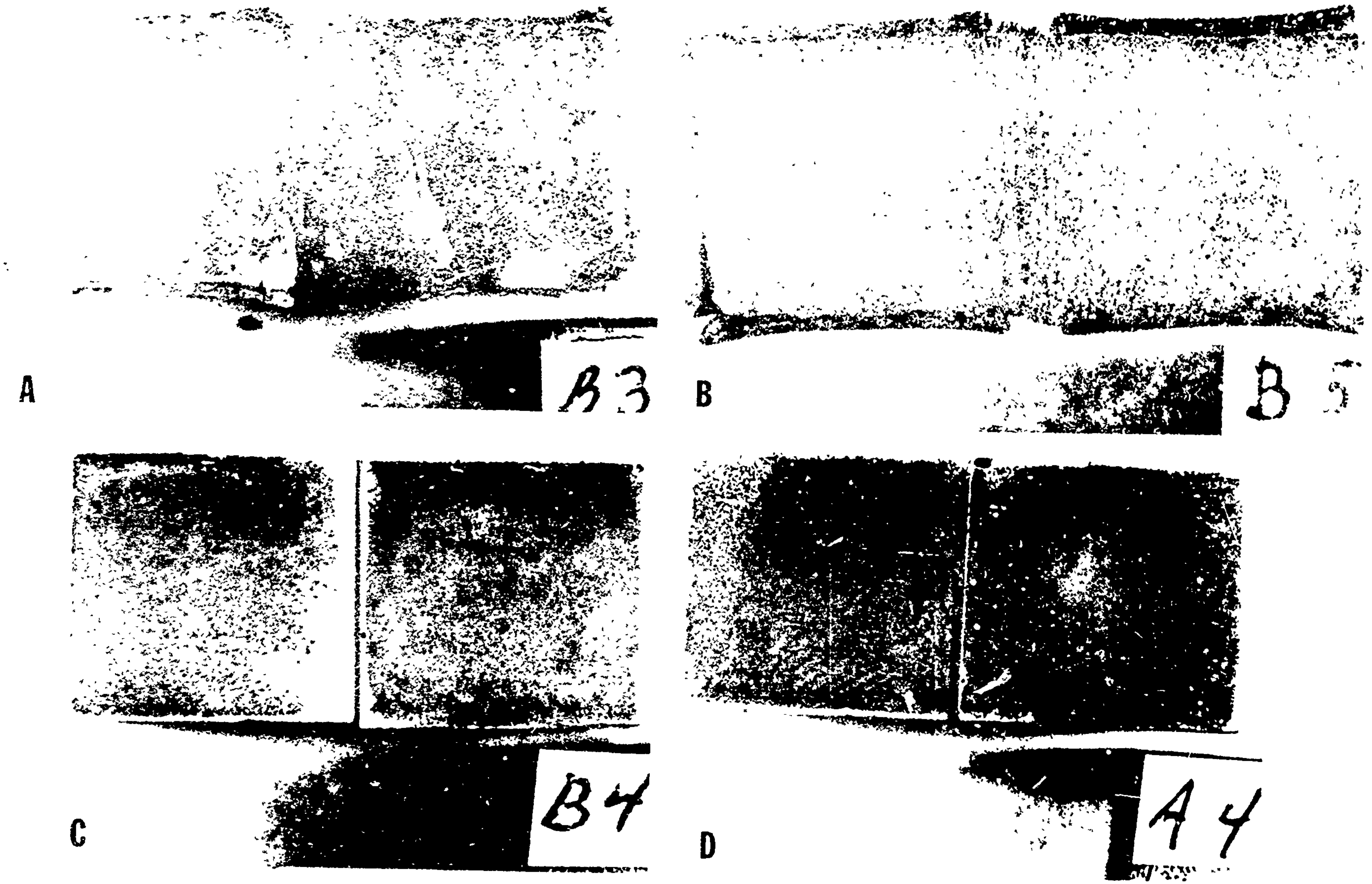

Fig. 3. Photographs of Cement Paste Cubes Immersed in Sulfate Solutions for 28 Days.
A. 30% Slag Portland Cement in Sodium Sulfate Solution (top, left).
B. 20% Trass Portland Cement in Sodium Sulfate Solution (top, right).
C. 70% Slag Cement in Sodium Sulfate Solution (bottom, left).
D. 70% Slag Cement in Gypsum Solution (bottom, right).

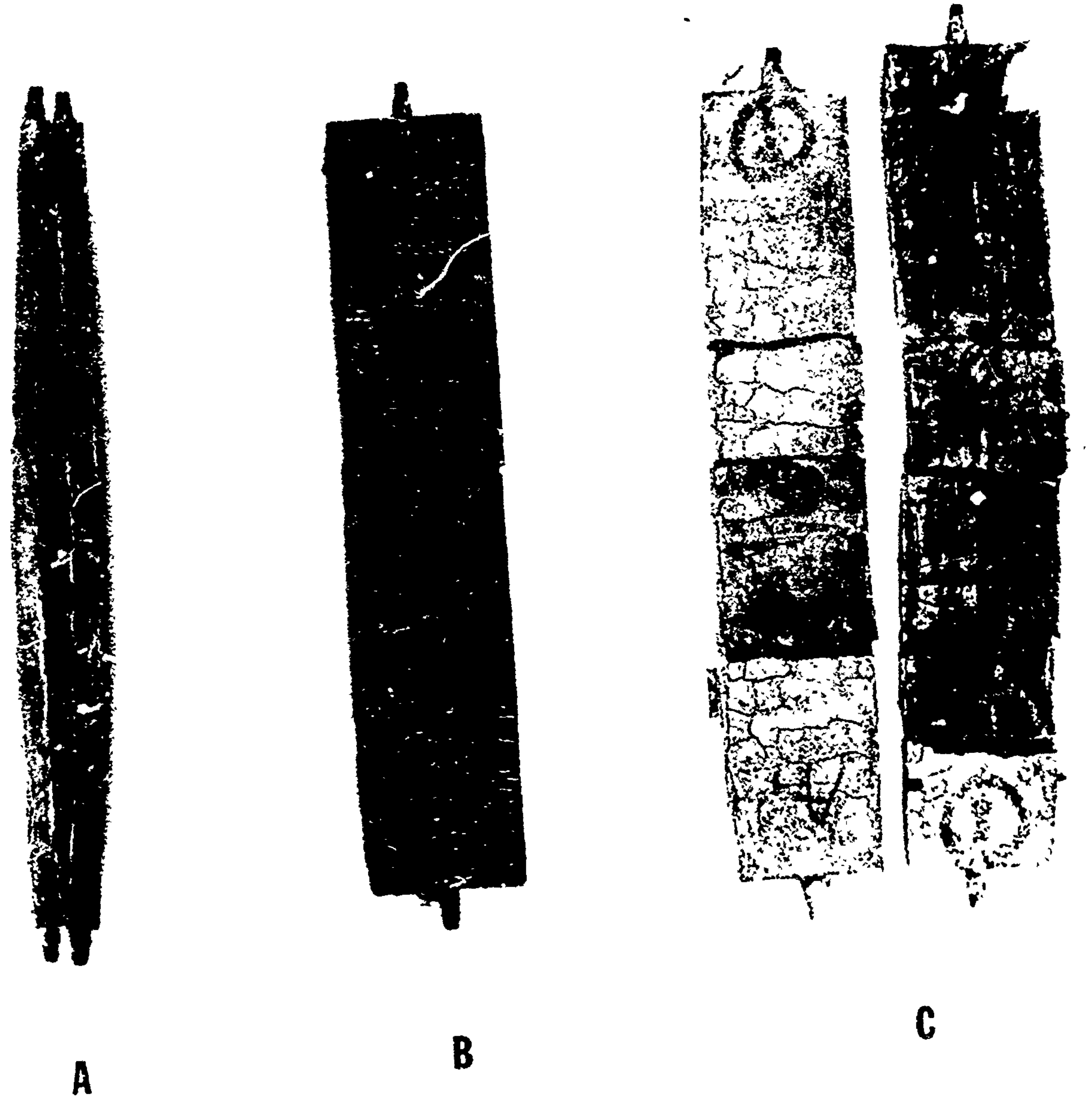

Fig. 4. Photographs of Ordinary Portland Cement Paste Prisms in Gypsum Solution.
A. Age 7 Days. B. Age 15 Days. C. Age 28 Days.

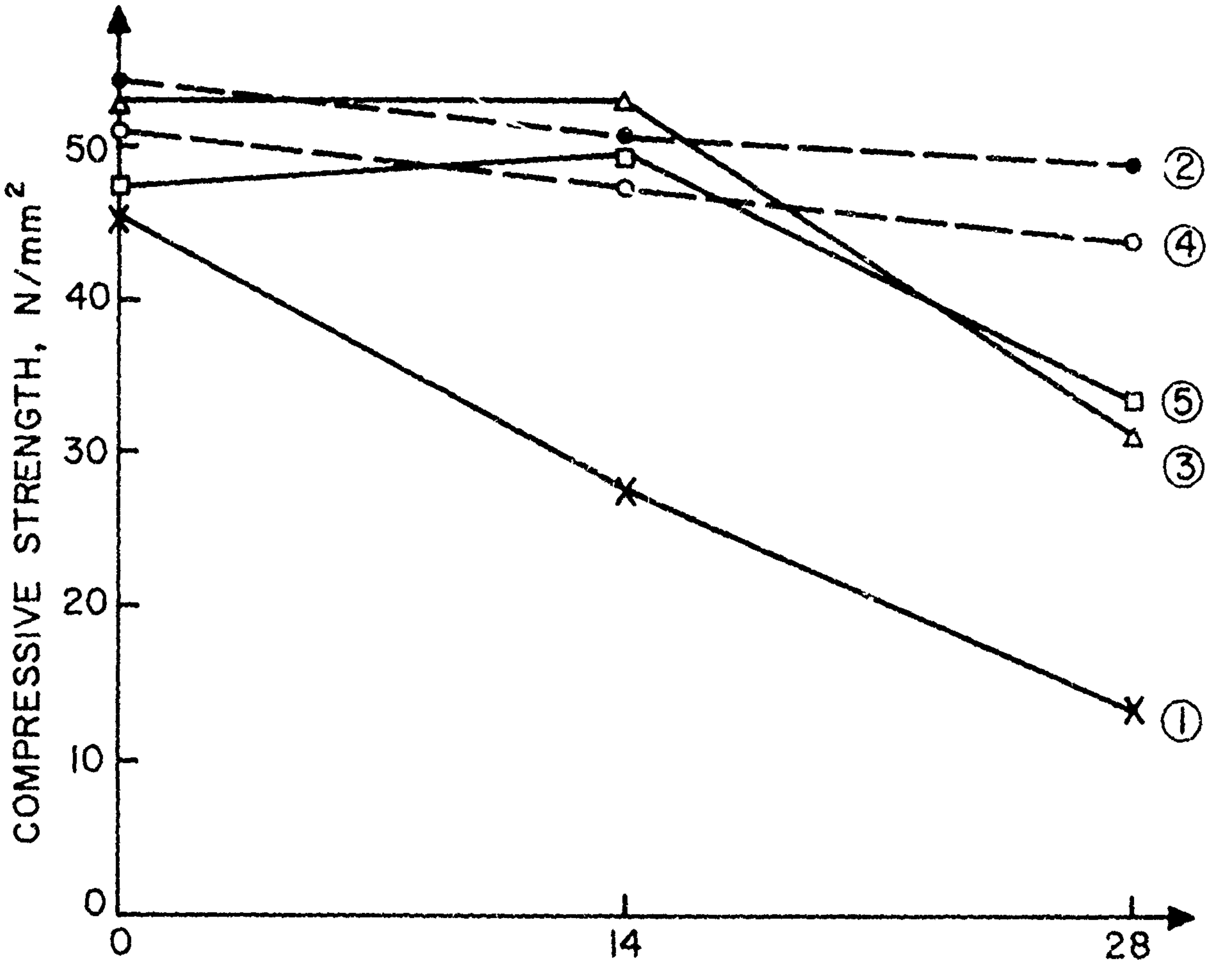

FIG.5 COMPRESSIVE STRENGTH OF CEMENT PASTE CUBES IN GYPSUM SOLUTION

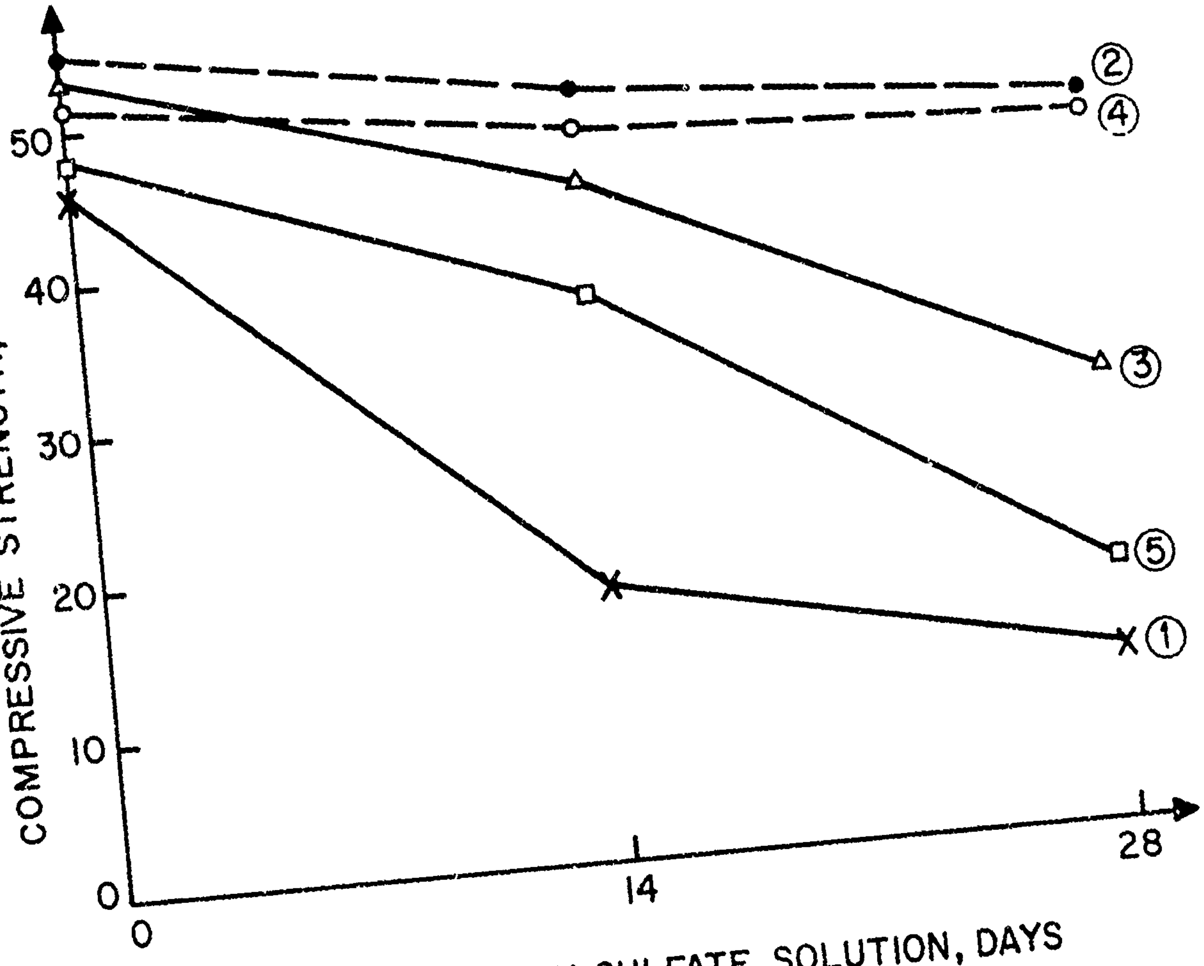

FIG.6 COMPRESSIVE STRENGTH OF CEMENT PASTE CUBES IN SODIUM SULFATE SOLUTION

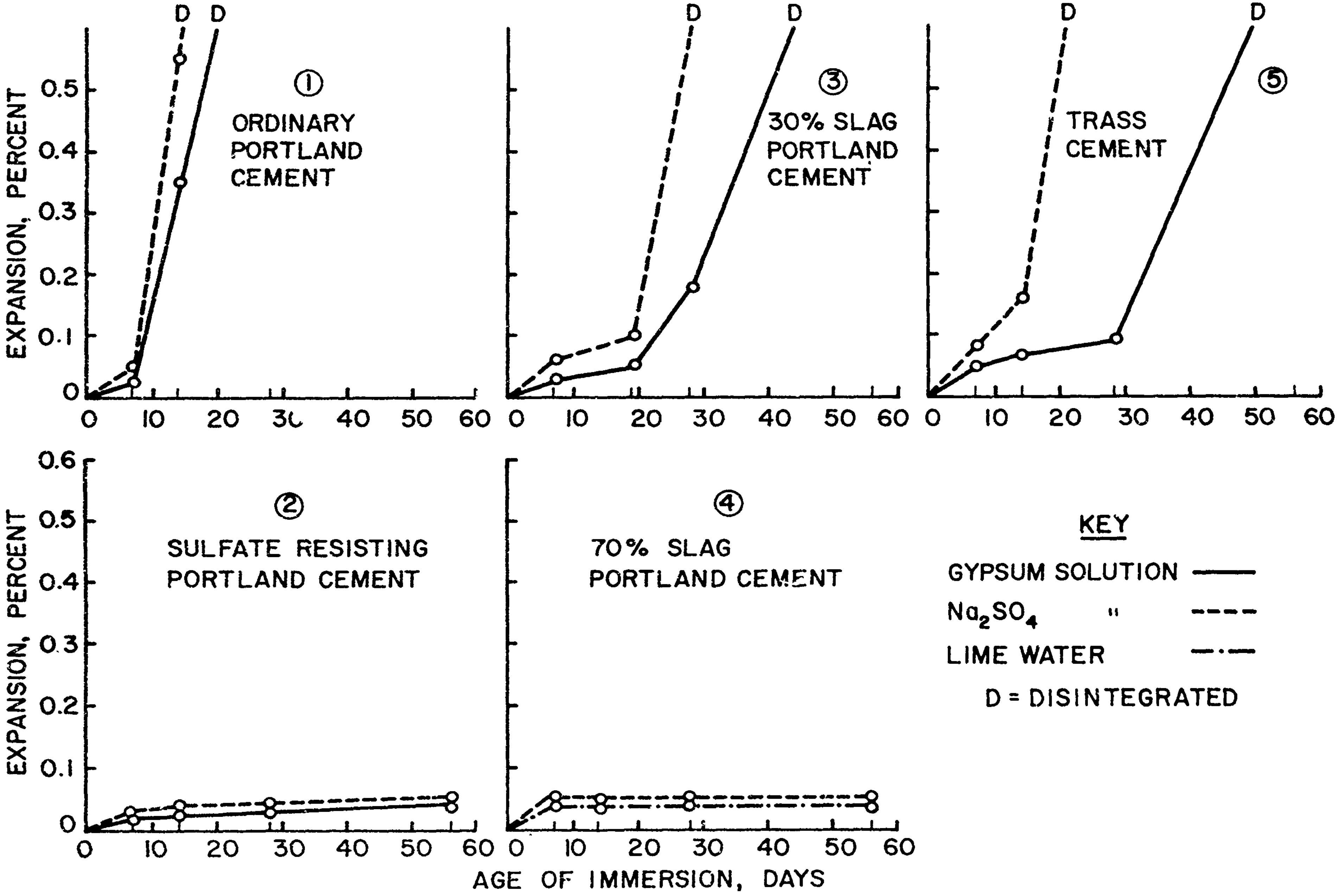

FIG.7 EXPANSION OF CEMENT PASTE PRISMS IN SULFATE SOLUTIONS