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Strength of C_3A paste containing gypsum and $CaCl_2$

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**STRENGTH OF C₃A PASTE CONTAINING
GYPSUM AND CaCl₂**

by

A. Traetteberg and P. J. Sereda

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STRENGTH OF C_3A PASTE CONTAINING GYPSUM AND $CaCl_2$

ANALYZED

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ABSTRACT

A study has been made of the strength of $3CaO.A\ell_2O_3$ and $3CaO.A\ell_2O_3$ + gypsum pastes as well as of the influence of the admixture $CaCl_2$ on these pastes. The methods employed were microhardness indentation and cone penetration. With 16 per cent $CaCl_2$ added to the $3CaO.A\ell_2O_3$ paste a significant gain in strength was obtained after 3 months, while an 8 per cent $CaCl_2$ addition to the $3CaO.A\ell_2O_3$ + gypsum system gave the highest strength at the same age. Morphological changes in the pastes were found to be related to changes in hardness. From a comparison of the two methods it was concluded that cone penetration measurements are useful up to a certain hardness level.

On a fait une étude de la résistance des pâtes de $3CaO.A\ell_2O_3$ et de $3CaO.A\ell_2O_3$ plus gypse ainsi que de l'effet de l'addition de $CaCl_2$ à ces mêmes pâtes. On a utilisé les méthodes de la microdureté par indentation et de la pénétration d'un cône. Lorsqu'on ajoute 16 pour cent de $CaCl_2$ à la pâte de $3CaO.A\ell_2O_3$, la résistance augmente considérablement en trois mois, tandis que l'addition de 8 pour cent de $CaCl_2$ au système $3CaO.A\ell_2O_3$ plus gypse produit la résistance la plus élevée pour cette même période. Les changements morphologiques de la pâte sont liés à des changements de dureté. Une comparaison des deux méthodes indique que les mesures de la pénétration d'un cône sont utiles jusqu'à un certain degré de dureté.

Presented at the VIth International Congress on the Chemistry of Cement, Moscow, September 1974.

Introduction

Information about strength developing during hydration of the pure cement components seems to be limited. An attempt to identify morphology and microstructure with strength was the object of this examination. Investigations carried out by Solov'eva and Segalova (1) and Segalova, Solov'eva and Rebinder (2) include examination of plastic strength developing in suspensions of C_3A^* and finely ground quartz sand ($C_3A/\text{quartz} = 5/95$) using a w/s ratio in the range 0.3 to 0.7. A conical plastometer was used. The plastic strength was found to decrease with an increase in temperature. This strength decrease was related to a decrease in the particle size of the hydroaluminates. The formation of the cubic C_3AH_6 caused a breakdown of the crystallization structure and thus a decrease in strength. The strength of the hydration products was found to decrease with increasing specific surface area of C_3A . A lowering of the w/s ratio caused more internal stresses in the hardened structure.

The structure formation of C_3A suspension and the effect of gypsum and $CaCl_2$ on the hydration structure of C_3A has been studied by Andreeva and Segalova (3). The addition of gypsum resulted in an increase in the strength of the structure formed, while the addition of $CaCl_2$ to the $C_3A + \text{gypsum}$ system caused expansion generating inner stresses and hence a reduced strength of the structure.

The conical plastometer that they used has been found convenient for strength measurements of plastic-viscous disperse systems. It has been evaluated theoretically in a paper by Gorazdovskii and Rebinder (4).

The technique of microhardness indentations has been found useful as a tool for strength measurements with the added advantage that, because the samples are not broken, a smaller piece is required and the same specimen can be used for several examinations. In earlier reports from the Division of Building Research (5,6,7), the results obtained by this method on gypsum and cement systems have been related to other physical properties such as modulus of elasticity, porosity, and fracture strength. The significance of microhardness of porous inorganic materials was discussed in a recent paper by Sereda (8) and it was concluded that a relation exists between microhardness and flexural strength for cement paste.

In an earlier report (9) the phase relations, microstructure and hydration mechanisms in the systems C_3A and $C_3A + \text{gypsum}$, with and without $CaCl_2$ added, were studied and discussed. The aim of the present work is to relate microstructure with mechanical strength as measured by the two methods, microhardness indentation and cone penetration, and to compare the two methods of measuring strength properties.

Experimental Work

The characteristics of the C_3A (supplied by Tetrattech International, San Diego) are the following: 61.6 per cent CaO , 37.8 per cent Al_2O_3 , 0.5 per cent MgO , and 0.1 per cent Na_2O . The C_3A had a surface area of $0.58 \text{ m}^2/\text{g}$. The gypsum and calcium chloride, $CaCl_2 \cdot 6H_2O$, were both Fisher certified reagents. The water used was decarbonated and de-ionized. Six different

* Standard cement nomenclature is used: $C = CaO$, $A = Al_2O_3$, $H = H_2O$ and w/s = water: solid ratio by weight. $G = \text{gypsum}$

C₃A PASTE, STRENGTH, GYPSUM, CaCl₂

pastes with a water/solid ratio of 1.0 were prepared: C₃A, C₃A + 8 per cent CaCl₂, C₃A + 16 per cent CaCl₂, C₃A + 25 per cent gypsum, C₃A + 25 per cent gypsum + 8 per cent CaCl₂, and C₃A + 25 per cent gypsum + 16 per cent CaCl₂. The w/s ratio of 1.0 was selected in an attempt to simulate the conditions for C₃A in the early hydration period of cement paste.

The paste hydrations were performed in specially designed plexiglass cylinders with a diameter of 2.5 cm and height of 1.3 cm. While curing under vapour-sealed conditions, the mixtures were rotated until the samples had completely set to prevent segregation (10). The same paste was tested repeatedly for microhardness at different intervals during the entire hydration time. The special shape was chosen to give a sufficiently large surface area to permit microhardness and cone penetration tests on the same sample. From a previous investigation (9) it was found that the shape of the sample can influence the thermal behaviour of the paste. To make use of data obtained previously it was ensured that the experimental conditions in the present case did not change the sequences and rates of formation of the different hydration products. This was verified by monitoring the temperature effect in the C₃A paste.

The strength of the materials was examined after hydrating for different periods up to 6 months.

Microhardness

A Leitz Miniload Hardness Tester was used. The test method employed the Vickers pyramid indenter. The hardness was found by the formula

$$HV(\text{kg/mm}^2) = \frac{1854.4 \times P}{d^2}$$

where

P = load in grams

d = mean value of the indentation diagonal.

Cone Penetration

In this method a cone is pressed into the paste with different pressures and the depth of penetration is measured. This method has been found particularly useful for strength investigations of cement paste in the early setting period. The significance of this method on different kinds of materials has been investigated by Sereda (11).

It is an open question whether the applied load per unit area should be calculated with an area corresponding to the base of the part of the cone that has penetrated into the material or the circumferential area of this part. The choice between the two areas might be considered to be dependent on the friction between the material and the cone during indentation. This leads to two possible formulae for calculating the hardness of the material:

$$H_B = \frac{\text{load (kg)}}{1.05 \cdot h^2} \quad \text{and} \quad H_C = \frac{\text{load (kg)}}{2.1 h^2} \quad \text{respectively}$$

for a 60° cone, where h = depth of penetration.

The load, which was produced by air pressure in a piston-cylinder arrangement, was supplied in steps of 3 lb and the penetration depth was measured at each level after reaching the equilibrium value. The plot of

load versus the depth of penetration squared generally gives two straight lines with the slope greater for the line at low penetration. The final slope of the plot representing the deeper penetrations includes the major number of measurements and is believed to represent the hardness of the three-dimensional structure.

Surface area was obtained by means of a Numinco-Orr surface area - pore volume analyzer with N_2 as the adsorbate.

Scanning Electron Microscopy

The morphology of pastes was studied by means of a Cambridge Stereoscan Mark 2A.

Results and Discussion

Microhardness

The indentation with the Vickers indenter was made on five spots on the surface of the paste sample and the ten diagonals measured were averaged to give a hardness value that was assumed to be representative for the sample involved. The variation in the measured values was sometimes large and sometimes insignificant, depending on the surface features of the particular sample. A typical set of measurements is given in Table I. No appreciable drying took place while measuring.

TABLE I

Measurements of Microhardness for Paste of $C_3A + G + 8$ per cent $CaCl_2$
after 3 Days of Hydration (load 500g)

Set of Diagonals	Microhardness (kg/mm^2)
162.3 - 169.8	2.10
160.6 - 163.0	2.21
153.9 - 150.8	2.50
159.2 - 151.0	2.28
157.0 - 178.0	2.06

Average diagonal: 160.5

Microhardness: 2.23 kg/mm^2

In connection with the formation of large amounts of C_3AH_6 accompanied by considerable shrinkage, the paste of C_3A became a thin slurry during the temperature effect after 5 to 6 minutes' hydration. This paste was not hard enough for any strength tests up to one week, and the surface of the specimen was too irregular for microhardness investigation.

The morphology of the hydrogarnet phase appeared with an open

structure, Figure 1a, ostensibly with very weak bonding between the crystals. The shape of these crystals (rhombic dodekahedrons), however, is such that a quite dense packing is possible if the hydration takes place in compacts where the different particles are forced together by an outer pressure. Under such conditions the hydrogarnet phase yields stronger inter-particle bonding and, hence, a stronger material structure (12).

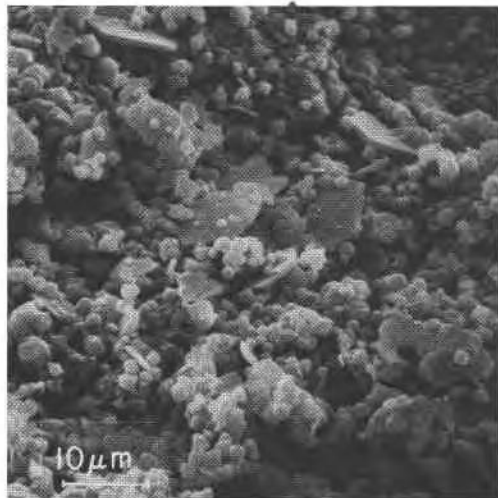


FIG. 1a
Paste of C₃A hydrated 28 days.

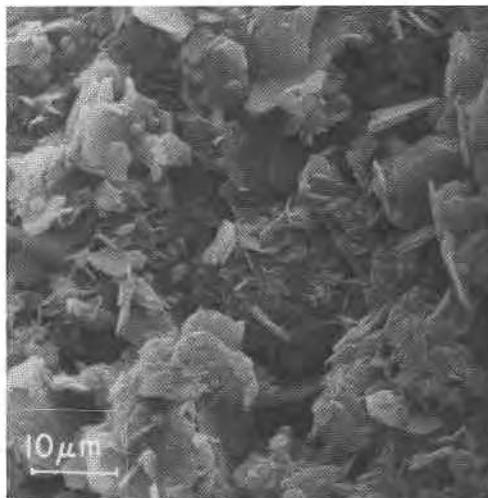


FIG. 1b
Paste of C₃A + 8% CaCl₂ hydrated
28 days.

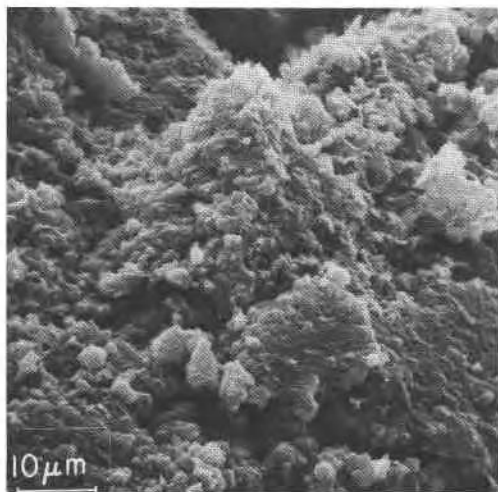


FIG. 1c
Paste of C₃A + 16% CaCl₂ hydrated
28 days.

The addition of 8 per cent CaCl₂ to the C₃A paste reduced the setting time so that after 2 hours the microhardness was measured at intervals up to 3 months. Table II gives the obtained values which show a slight increase by hydrating to 30 days and a great increase after 3 months. The minor reduction in strength observed in the period between 3 and 30 days might be due to a more active formation of the cubic C₃AH₆, an effect that has been confirmed in a previous work (9). The micrograph, Figure 1b, shows a dominant platy structure that is expected to result in improved inter-particle bonding compared with the cubic phase. An ageing effect might be the reason for the strength developed after 3 months. This is supported by the morphological change as demonstrated in Figure 2b and by the decreased surface area at this stage, Table III and Table IV.

TABLE II

Microhardness (kg/mm^2) for Pastes of C_3A
 With Addition of 25 Per Cent Gypsum and CaCl_2
 Hydrated for Different Periods

Hydration Time	C_3A	$\text{C}_3\text{A} + 8\%$ CaCl_2	$\text{C}_3\text{A} + 16\%$ CaCl_2	$\text{C}_3\text{A} +$ gypsum	$\text{C}_3\text{A} + \text{G} +$ 8% CaCl_2	$\text{C}_3\text{A} + \text{G} +$ 16% CaCl_2
2 hours	-	0.46	-	-	0.98	-
4 hours	-	0.60	0.75	-	1.54	-
8 hours	-	0.98	1.28	-	2.42	-
1 day	-	1.00	1.77	0.58	2.60	2.84
2 days	-	1.08	2.26	0.94	3.14	3.16
3 days	-	1.77	2.39	1.11	2.26	3.05
7 days	-	1.38	3.52	1.32	2.63	2.91
10 days	-	1.37	3.32	1.45	2.67	3.10
14 days (15)	-	1.30	4.29	1.73	2.90	3.54
30 days	-	1.85	10.20	1.65	3.41	3.65
3 months	-	8.74	14.07	6.24	12.64	9.52

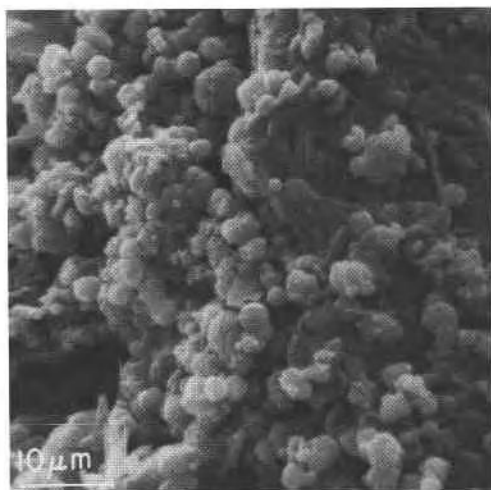


FIG. 2a
 Paste of C_3A hydrated 3 months.

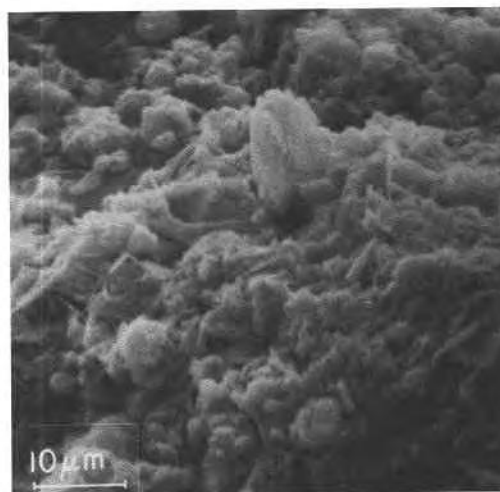


FIG. 2b
 Paste of $\text{C}_3\text{A} + 8\% \text{CaCl}_2$ hydrated
 3 months.

C₃A PASTE, STRENGTH, GYPSUM, CaCl₂

The paste of C₃A + 16 per cent CaCl₂ showed a similar trend in strength development as the previous mixture. While higher values in microhardness were obtained in this series, the formation of C₃AH₆ was further retarded by this addition. This corresponds to the delay in the small decrease in strength that, in this case, occurred after about 10 days. The ageing effect seemed to take place at an earlier stage for this mixture. This might be suggested from the micrographs, Figures 1b and c, that demonstrate a denser packing and a smaller crystal size of the hydroaluminates in the C₃A + 16 per cent CaCl₂ paste. The increased addition of CaCl₂ to paste of C₃A improves the strength of the structure of the hydration products up to 1 month, an effect which might be related to morphological features (Figure 2c).

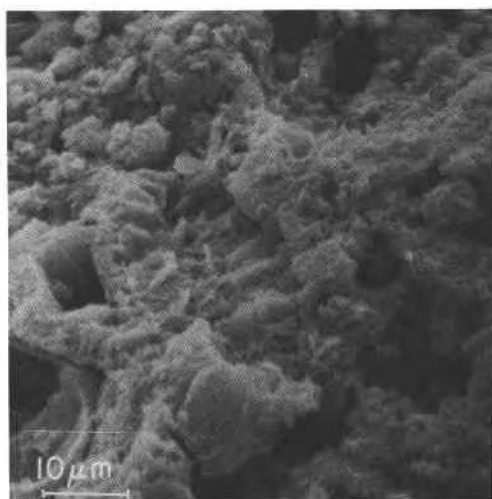


FIG. 2c
Paste of C₃A + 16% CaCl₂ hydrated
3 months.

TABLE III

Characteristics of Pastes of C₃A and C₃A + Gypsum With and
Without CaCl₂ Hydrated for 30 Days

Paste	Phases Present	Surface Area (m ² /g)	Micro-hardness (kg/mm ²)	Cone Penetration (kg/mm ²)
C ₃ A	C ₃ AH ₆ , minor amounts of C ₂ AH ₈ and C ₄ AH ₁₃	4.2	-	0.28
C ₃ A + 8% CaCl ₂	C ₃ AH ₆ , C ₂ AH ₈ and (ss) C ₄ AH ₁₃ - C ₃ A.CaCl ₂ .H ₁₀	8.2	1.85	0.96
C ₃ A + 16% CaCl ₂	C ₃ AH ₆ , C ₂ AH ₈ and (ss) C ₄ AH ₁₃ - C ₃ A.CaCl ₂ .H ₁₀	9.9	10.20	7.53
C ₃ A + gypsum	(ss) C ₃ A.CaSO ₄ .H ₁₃ - C ₄ AH ₁₃ , C ₂ AH ₈ and traces of C ₃ AH ₆	11.0	1.65	1.00
C ₃ A + G + 8% CaCl ₂	(ss) C ₃ A.CaSO ₄ .H ₁₃ - C ₃ A.CaCl ₂ .H ₁₀ - C ₄ AH ₁₃ and C ₂ AH ₈	13.3	3.41	3.60
C ₃ A + G + 16% CaCl ₂	(ss) C ₃ A.CaSO ₄ .H ₁₃ - C ₃ A.CaCl ₂ .H ₁₀ - C ₄ AH ₁₃ and C ₂ AH ₈	12.6	3.65	5.31
(ss) = solid solution				

TABLE IV

Characteristics of Pastes of C_3A and C_3A + Gypsum
With and Without $CaCl_2$ Hydrated for 3 Months

Paste	Surface Area (m^2/g)	Micro-hardness (kg/mm^2)	Cone Penetration (kg/mm^2)
C_3A	1.0	-	23.00
$C_3A + 8\% CaCl_2$	2.4	8.74	6.85
$C_3A + 16\% CaCl_2$	4.2	14.07	25.66
$C_3A + 25\% \text{ gypsum}$	3.7	6.24	4.94
$C_3A + G + 8\% CaCl_2$	3.9	12.64	25.50
$C_3A + G + 16\% CaCl_2$	4.2	9.52	14.20

The microhardness of the $C_3A + 25$ per cent gypsum paste appeared to be close to that for the $C_3A + 8$ per cent $CaCl_2$ system. Traces of C_3AH_6 have been detected at late stages in the hydration of the $C_3A +$ gypsum paste (9), and this effect might also be the reason for the slight decrease in microhardness around 30 days. The microstructure of these two mixtures is also quite similar, both at 30 days (compare Figures 1b and 3a) and at 3 months (Figures 2b and 4a). The increase in strength with the addition of gypsum to C_3A is in agreement with the result of Andreeva and Segalova (3).

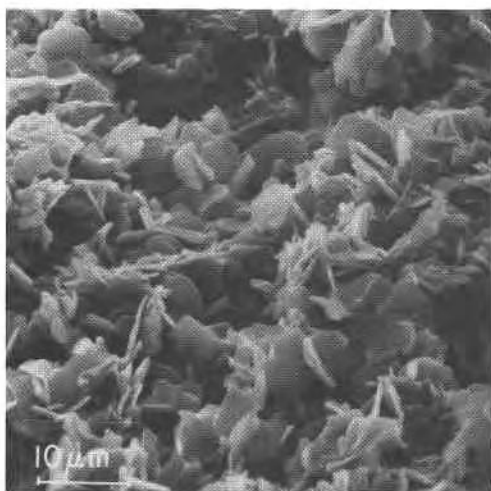


FIG. 3a
Paste of $C_3A + 25\% \text{ gypsum}$
hydrated 28 days.

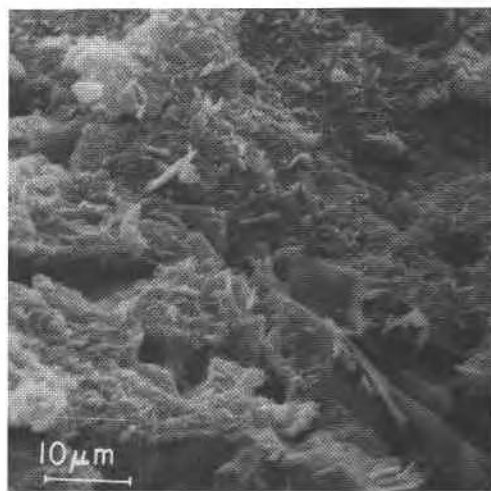


FIG. 3b
Paste of $C_3A + g + 8\% CaCl_2$
hydrated 28 days.

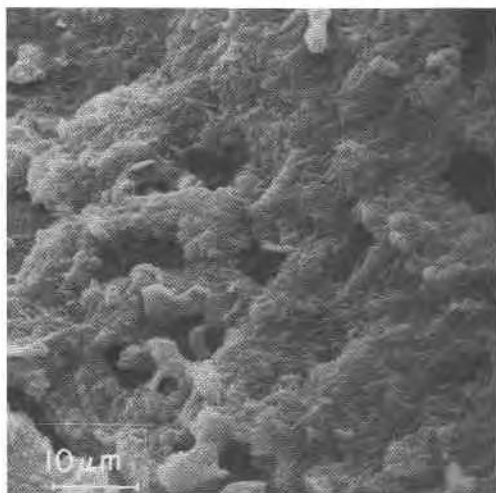


FIG. 3c
Paste of C₃A + G + 16% CaCl₂
hydrated 28 days.

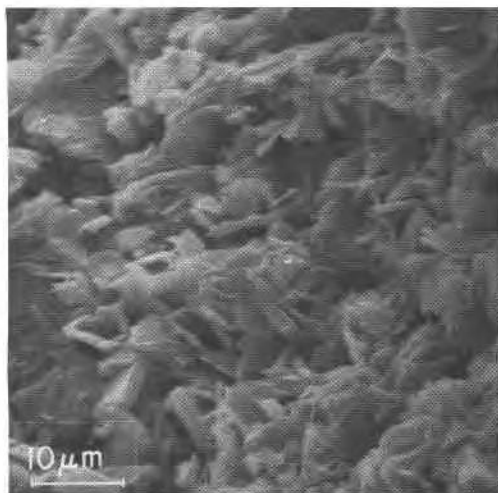


FIG. 4a
Paste of C₃A + 25% gypsum
hydrated 3 months.

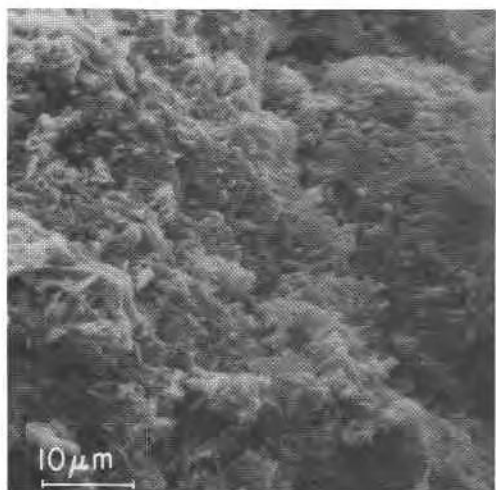


FIG. 4b
Paste of C₃A + G + 8% CaCl₂
hydrated 3 months.

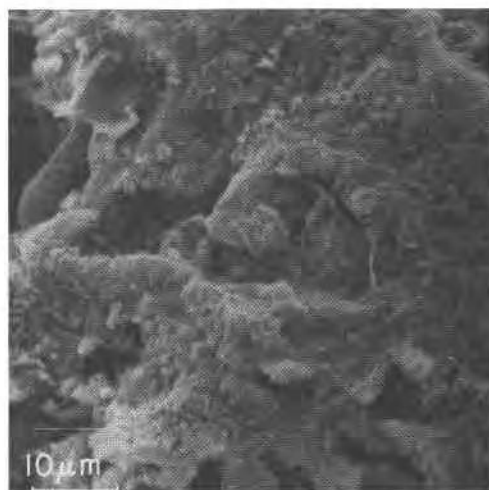


FIG. 4c
Paste of C₃A + G + 16% CaCl₂
hydrated 3 months.

The C₃A + gypsum + 8 per cent CaCl₂ mixture set within the first 10 minutes of hydration. The hardness increased steadily up to 2 days, when a small decrease took place. The effect in this case cannot be explained by C₃AH₆ formation which is prevented with the addition of CaCl₂ to the C₃A + gypsum paste (9). The reason for the small reduction in strength is not known, but it might be due to some reorganizing of the bonding between the crystallites. The ageing effect after 3 months is especially evident in this case. The increased strength in this paste might arise from an improved

bonding or better packing of the smaller crystals, as is illustrated in the micrographs, Figures 3b and 4b and possibly due to some carbonation. This improved strength with the addition of CaCl_2 is in contradiction with the result of Andreeva and Segalova (3). The reason for the different result might arise from their use of suspensions of C_3A and quartz compared with a pure paste hydration of C_3A in our work.

An increased addition of CaCl_2 does not seem to give any significant improvement to the hardness of the paste. The slight decrease in strength after 2 days is also detected here. Figures 3b and 3c also demonstrate a similar morphology. The microhardness after 3 months is lower than with the 8 per cent addition and the micrograph, Figure 4c, indicates a denser packing of the structure but more cracks and pores than in the previous case. The lower strength might be due to some stress developed with the additional amount of CaCl_2 .

It is known that carbon dioxide may cause surface hardening of cement. A doubt therefore arose that the increase in strength at later hydration periods may not only be due to an ageing effect but also partly to carbonation of the surface. A 2-mm-thick piece from the surface of the samples was cut off after 6 months' hydration and analyzed for CO_2 content. Microhardness was measured on the old and new surfaces of the samples and the measurements are recorded in Table V. The results reveal that some carbonation had taken place. The effect does not seem to have had a significant effect on the microhardness, however, except in the cases of samples with 8 per cent CaCl_2 added to the C_3A and C_3A + gypsum pastes where a lower microhardness was obtained on the inner surface. Experience has shown that the effect of carbonation is first observed on the surface and it is reasonable to consider the value of microhardness for the new (cut) surface as representing the bulk property.

TABLE V

Carbon Dioxide Content and Microhardness for Pastes
of C_3A With and Without Admixtures, Hydrated for 6 Months

Paste	CO_2 content (%)	Microhardness (kg/mm^2)	
		original surface	cut surface*
C_3A	0.95		17.6
$\text{C}_3\text{A} + 8\% \text{CaCl}_2$	2.24	8.89	4.36
$\text{C}_3\text{A} + 16\% \text{CaCl}_2$	4.24	15.77	13.30
$\text{C}_3\text{A} + 25\% \text{gypsum}$	3.42	6.03	5.74
$\text{C}_3\text{A} + \text{G} + 8\% \text{CaCl}_2$	3.33	12.22	8.62
$\text{C}_3\text{A} + \text{G} + 16\% \text{CaCl}_2$	2.21	9.71	10.27

* Original surface was removed by cutting a section 2 mm thick

C_3A PASTE, STRENGTH, GYPSUM, $CaCl_2$ Cone Penetration

Figure 5 shows a typical result of the cone penetration measurement that includes a number of load versus depth determinations. High humidity was maintained during measurement to prevent drying. In a few cases there were three determinations carried out to establish the reliability of the measurements. It was found that in this range of hardness only a slight scattering in the values took place and one determination was, therefore, considered sufficient to give a measure of the strength of the material. The hardness, calculated per unit circumferential area of the cone, is found to be the most suitable for these samples.

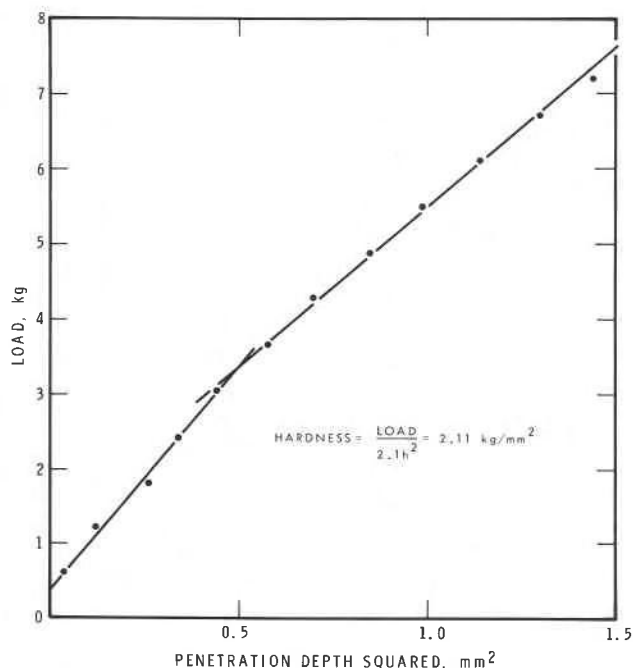


FIG. 5
Cone penetration measurements for
paste of C_3A + gypsum + 8% $CaCl_2$
hydrated for 3 days.

After 7 days the initially weak structure of the C_3A paste reached a hardness sufficiently large to permit cone penetration measurements to be done, Table VI. The micrograph of the 30 days' sample, Figure 1a, shows a loose morphology mainly of the cubic phase C_3AH_6 . The ageing effect was very pronounced in this paste as the hardness increased to 23 kg/mm² after 3 months' hydration. The micrograph, Figure 2a demonstrates a denser structure of the crystals and the decreased surface area of this sample (Table IV) evidently corresponds to a coalescence of the individual particles.

The hardness values obtained by cone penetration in the C_3A + 8 per cent $CaCl_2$ paste as a function of hydration time show a similar pattern as those obtained by microhardness measurements.

The observed decrease in strength, considered to be due to more active formation of C_3AH_6 , is also detected in the paste of C_3A + 16 per cent $CaCl_2$, but was slightly delayed in this case. Both methods show a marked strength increase in the 3 month samples, but the absolute values differ. It is believed that after a certain hardness level the cone penetration method gives values that are too high.

For the C_3A + gypsum system, cone penetration gave hardness values close to those from the microhardness tests. This was also the case for the samples with the addition of 8 per cent $CaCl_2$ to the system. However, the cone penetration method resulted in higher hardness values with the 16 per cent addition of $CaCl_2$.

TABLE VI

Hardness Values (kg/mm^2) Obtained by Cone
Penetration Measurements for Pastes of C_3A With
Addition of 25 Per Cent Gypsum and CaCl_2 Hydrated
for Different Periods

Hydration Time	C_3A	$\text{C}_3\text{A} + 8\% \text{CaCl}_2$	$\text{C}_3\text{A} + 16\% \text{CaCl}_2$	$\text{C}_3\text{A} + \text{gypsum}$	$\text{C}_3\text{A} + \text{G} + 8\% \text{CaCl}_2$	$\text{C}_3\text{A} + \text{G} + 16\% \text{CaCl}_2$
$\frac{1}{2}$ hour	-	-	-	-	1.02	-
1 hour	-	-	-	-	1.44	-
2 hours	-	0.48	-	-	1.19	0.065
4 hours	-	0.86	1.46 (6h)	-	1.52	-
8 hours	-	0.90	1.30	-	2.07	0.112
24 hours	-	0.85	3.08	0.75	1.22	4.88
2 days	-	0.89	2.40	0.98	2.30	3.18
3 days	-	1.20	1.68	0.69	2.11	3.82
7 days	0.24	0.84	3.08	0.58	2.47	5.15
10 days	0.16	0.63	4.00	0.59	2.94	5.50
14 days	0.13	0.60	3.87	1.44	2.84	5.58
30 days	0.28	0.96	7.53	1.00	3.60	5.31
3 months	23.00	6.85	25.60	4.94	25.50	14.20

Table III demonstrates the composition of the pastes at 30 days' hydration as obtained in another report (9), together with corresponding data of surface area and hardness.

A comparison between the data at 30 days and 3 months (Tables III and IV) clearly demonstrates the ageing effect for all samples, i.e., an increase in strength accompanied by a decrease in surface area. The ageing might be due partly to the high humidity conditions of storage.

Conclusions

The investigation of strength in different C_3A systems by means of microhardness and cone penetration has shown that changes in these properties can be identified with morphological changes. The denser morphology for all six pastes after 3 months' hydration is believed to be the result of an ageing effect involving coalescence of the individual crystals by increasing the area of contact due to dissolution and recrystallization. The reduced surface area obtained for all the pastes at this stage and evidence from micrographs support this assumption.

The influence of the admixture CaCl_2 on the strength of C_3A and $\text{C}_3\text{A} + \text{gypsum}$ pastes is significant up to 1 month hydration where an increase of CaCl_2 means an increase in strength. However, its influence in connection with the ageing effect is less clear. The 16 per cent addition to the C_3A

C_3A PASTE, STRENGTH, GYPSUM, $CaCl_2$

paste gave the highest hardness value but it is of the same magnitude as the one for the C_3A paste alone. For the C_3A + gypsum system the 8 per cent $CaCl_2$ addition gave the hardest structure.

A comparison between the two testing methods, microhardness indentation and cone penetration, shows good agreement in some mixtures and only fair agreement in others. It might be concluded that cone penetration is a convenient method for softer materials, but has its limitation on materials with hardness above a certain level.

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