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# Exploratory Studies of Early Strength Development in Portland Cement Pastes and Mortars

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# Exploratory Studies of Early Strength Development In Portland Cement Pastes and Mortars

**R. L. Blaine and L. A. Tomes**

Building Research Division
Institute for Applied Technology
National Bureau of Standards
Washington, D. C. 20234

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# Exploratory Studies of Early Strength Development In Portland Cement Pastes and Mortars

R. L. Blaine* and L. A. Tomes*

A modified vane-shear apparatus was used to measure the shear resistance of neat cement pastes of normal consistency and 1:2.75 (cement to sand) mortars of standard consistency, and to measure the increase in shear resistance with time as the cements hardened. The hardening process appeared to occur in three stages. The rate of increase of shear resistance as well as the duration of the different phases differed with the different cements. The results were analyzed in terms of the various theories proposed to explain the hardening of cements.

Key words: Cement; cement mortar; cement paste; early strength; false set; hardening of cement; hydration; shear resistance; theory of cement hardening; time of set; vane-shear apparatus.

## 1. Introduction

There is extensive literature dealing with the early hydration reactions of portland cements and the development of structure which causes stiffening and hardening [1-6]. The plasticity as well as the rate of stiffening and hardening of cement pastes, mortars, and concretes are of great importance in the construction industry. The rheology of freshly mixed cement-water pastes, mortars, and concretes have been studied by various means, using viscometers [7], shear apparatus [8], penetrometers [9] as well as pull-out pins [10], soniscope [11], and other devices. A discussion of the various methods was presented by Kelly [12] and the reactions involved have been reviewed by Steinour [13] and more recently by Kondo and Ueda [6]. The Vicat and Gillmore needles are also used for determining time of set, that is, the time at which the neat cement pastes have attained strength adequate to prevent a penetration of the Gillmore needles or a limited penetration of the Vicat needle.

It has been stated [3] that "Published information on the viscosity or consistency of cement pastes during the early period of hydration is scanty, however, and somewhat lacking in agreement . . .". One of the problems has been the lack of apparatus sensitive enough to measure small differences in rheological properties but with a sufficient range to evaluate any strength from the time of mixing to the time of hardening. The present exploratory studies are reported at this time to indicate the application of a modified vane-shear apparatus in obtaining information on changes of rheological properties and apparent development of structure of cement pastes and mortars during the first two to three hours after mixing.

The vane-shear apparatus has been used in measuring the rheological properties of soils and many articles have been published with respect to its use [14-18]. These articles also give reference to many other publications in the field. One of the problems associated with its use has been the fact that the soil sample is disturbed when the vane is inserted. This would also happen if the vane were inserted in hardening cement paste. It was therefore necessary to modify the apparatus and techniques for use in studying the early hardening of cement pastes and mortars as indicated in the following sections and in the appendix A.

## 2. Apparatus

The commercially available vane-shear apparatus as modified is shown in figure 1. The vanes are placed in the freshly mixed mortar. The torque is applied by means of a helical spring through a couple of universal joints and a sheet of polytetrafluoroethylene is used as a base plate to minimize the friction be-

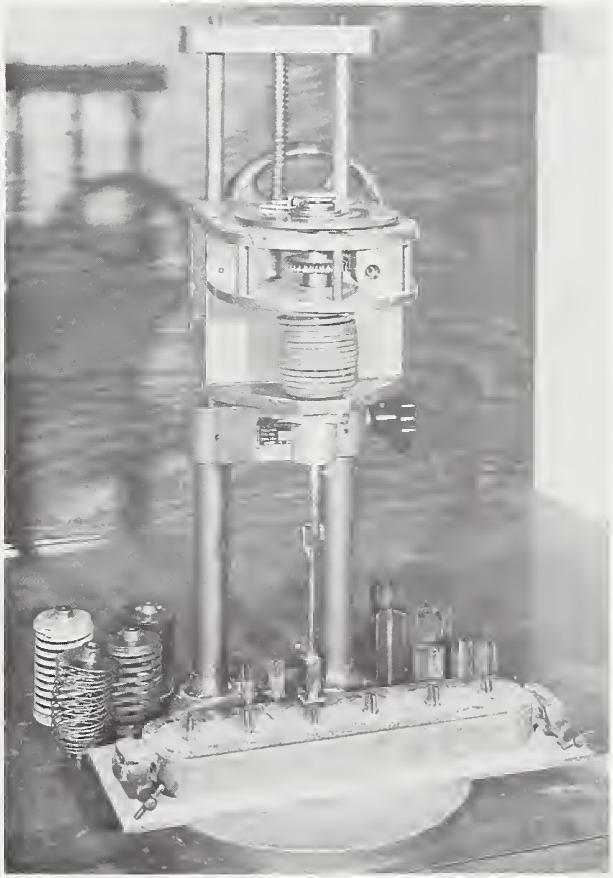

FIGURE 1. *Vane-shear apparatus and extra springs.*

[1] Figures in brackets refer to the literature references on page

*Deceased. Both formerly associated with the Building Research Division, Institute for Applied Technology, National Bureau of Standards, Washington, D.C. 20234.

tween the vane and base. It was also necessary to make a spring with about a tenth the strength of the weakest spring which came with the apparatus in order to make measurements on the plastic mortars.

Linkage between the shear apparatus and the vanes is indicated in figure 2 and described in appendix A.

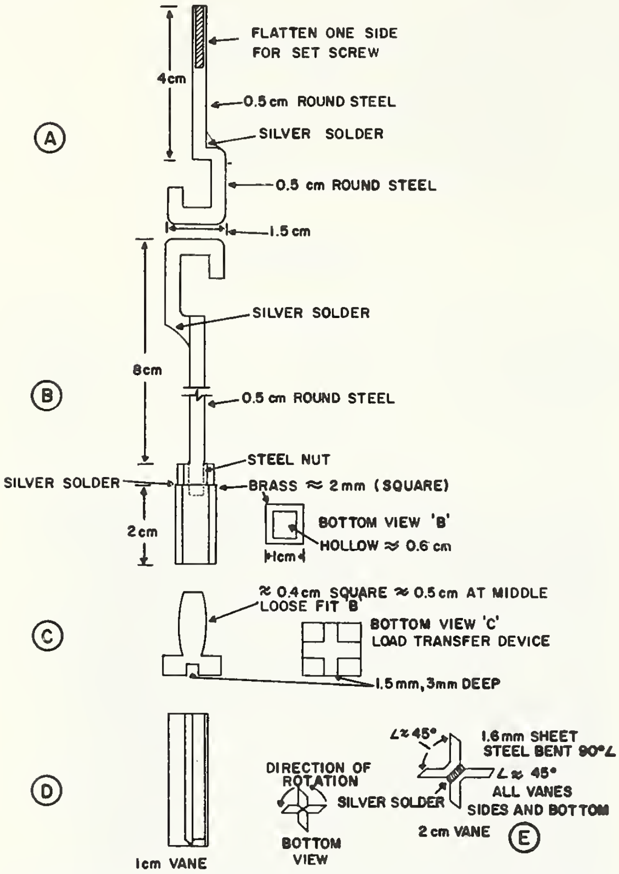


Figure 2a. *Vanes and linkage between shear apparatus and vanes.*

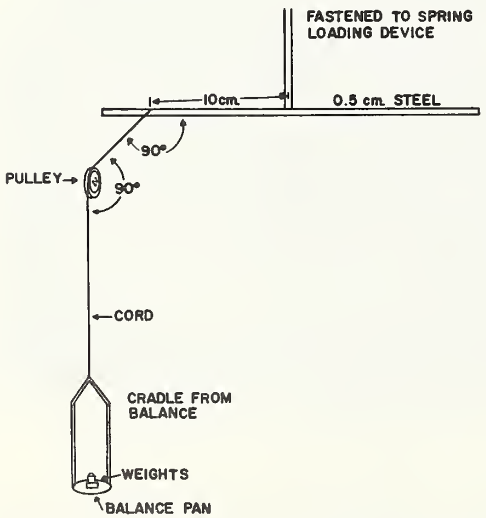


Figure 2b. *Calibration of springs in shear apparatus.*

The springs were calibrated using a cord attached to a horizontal rod 10 cm from the center of the spring. (see appendix B and Figure 2b) The cord passed over a pulley to a balance cradle and pan and a series of weights were placed on the pan noting the number of degrees of rotation required to bring the spring and weights to equilibrium. Plots of the rotation values versus the weights resulted in straight lines passing through the origin with each of the springs. The constants for the springs used in this study were calculated in terms of the grams per degree rotation at 1 cm radius. The weakest spring had a value of 1.91 g/deg rotation, the second spring 21.4 g/deg and the strongest spring 44.9 g/deg.

The vanes were made of 1.6 mm steel sheets bent at right angles and silver soldered together as indicated in figure 2. They were then machined to size ±0.05 mm and the side edges and bottom edges beveled (see figure 2a) to relieve any friction between the edges and mortar or paste when the vane was rotated. Vanes were made having a width or diameter of 1, and 2 cm. The 1 cm vanes were made for use in a 2.5 cm-deep mold* and the 2 cm vanes for use in a 4 cm-deep mold.** The lengths of the vanes were about 1 cm greater than the depth of the respective molds.

The equation used to compute the shear values was as follows:

$$S=\frac{\text{degrees rotation} \times \text{spring constant}}{\pi\ (D\ H\ r)}$$

where $S$ = shear resistance
$\pi$ = pi = 3.1416
$D$ = width of vane or diameter
$H$ = depth of vane in mortar or paste
$r$ = radius of vane.

This does not take into account any end effect. If effect of the bottom of the vane is included, the equation would be as follows:

$$S=\frac{\text{degrees rotation} \times \text{spring constant}}{\pi\ \left(D\ H\ r + \frac{D^2}{4} \cdot \frac{1}{3} D\right)}$$

If the depth in mortar and paste is constant and only one size vane used, the shear equals a constant times the degrees of rotation. The constants used with the different springs would be different depending on their calibration factor.

## 3. Cements

Tests were made on six portland cements which had previously been used in the Cement and Concrete Reference Laboratory (CCRL) interlaboratory reference sample program. These included two type I (CCRL Nos. 3 & 4), two type IA (CCRL Nos. 5 & 6), and two type III (CCRL Nos. 9 & 10) cements. The chemical composition and results of some of the

* ASTM Designation C190, Tensile Strength of Hydraulic Cement Mortars.
** ASTM Designation C348, Flexural Strength of Hydraulic Cement Mortars.

physical tests as determined in Cement and Concrete Reference Laboratory interlaboratory tests are presented in table 1. These values are the averages of values reported by all laboratories after outlying results from a few laboratories had been eliminated. There were 85 to 145 laboratories reporting chemical results and 104 to 162 laboratories reporting physical results. The estimated standard deviations of the time of set values determined by the different laboratories ranged from 14 to 28 min for Vicat time of set, from 22 to 36 min for Gillmore initial set and from 44 to 56 min for Gillmore final set.

## 4. Methods of Tests

A number of preliminary tests were made using the different sized vanes in different molds before adopting the techniques presented in this report. Further information about the preliminary tests will be presented in the discussion.

Tests were made on neat cement pastes mixed in a mechanical mixer in accordance with specification ASTM Designation C305-65 except that 1400 g of cement were used. The percentage water was that given in table 1. After mixing, a scoop of paste was placed in each of 3 molds of a 3-gang briquet mold and this was then vibrated sufficiently to cause the paste to flow to the edges,—about 5 to 10 s. The excess paste was cut off,—the mold trowelled once and then cut off with a sharp trowel using a sawing motion. One of the 1 cm vanes was placed in each half of the briquet at about the center of the broadest dimensions making six vanes for each mold. The mold with paste and vanes was then given 2 or 3

TABLE 1. *Average values for chemical analyses and physical tests reported for Cement and Concrete Reference Laboratory interlaboratory tests on six cements*

| Cement No. | 3 | 4 | 5 | 6 | 9 | 10 |
|---|---|---|---|---|---|---|
| $SiO_2$ % | 20.52 | 21.31 | 20.21 | 21.68 | 20.14 | 20.09 |
| $Al_2O_3$ % | 5.05 | 4.49 | 6.12 | 5.51 | 4.84 | 5.42 |
| $Fe_2O_3$ % | 2.96 | 2.89 | 2.17 | 2.34 | 2.26 | 2.43 |
| CaO % | 62.79 | 63.11 | 63.61 | 63.42 | 62.89 | 65.46 |
| MgO % | 4.23 | 4.33 | 2.77 | 2.35 | 3.90 | 1.66 |
| $SO_3$ % | 2.42 | 2.29 | 2.65 | 2.81 | 3.52 | 3.12 |
| Loss % | 1.14 | 0.83 | 1.15 | 1.00 | 1.76 | 1.06 |
| Insol % | 0.19 | 0.15 | 0.15 | 0.22 | 0.33 | 0.13 |
| $Na_2O$ % | .13 | .19 | .35 | .31 | .19 | .15 |
| $K_2O$ % | .82 | .64 | .96 | .79 | .57 | .63 |
| Normal consistency, $H_2O$ % | 24.0 | 25.62 | 25.49 | 25.57 | 28.53 | 26.87 |
| Vicat time of set, min | 147 | 207 | 123 | 103 | 155 | 77 |
| Gillmore initial set, min | 177 | 223 | 158 | 145 | 196 | 120 |
| Gillmore final set, min | 300 | 335 | 283 | 270 | 316 | 221 |
| Air content, 1:4 mortar, % | 9.6 | 8.8 | 18.6 | 18.3 | 10.0 | 10.8 |
| $H_2O$, 1:2.75 mortar, % | 46.6 | 47.0 | 45.2 | 45.5 | 50.0 | 47.8 |
| Comp str, ld psi | — | — | — | — | 3318 | 3590 |
| Comp str, 3d psi | 3060 | 3310 | 2913 | 2665 | 5041 | 5058 |
| Comp str, 7d psi | 3990 | 4320 | 3615 | 3504 | — | — |
| Fineness (air perm), $cm^2/g$ | 3379 | 3537 | 3358 | 3782 | 5784 | 4857 |
| $C_3A$ ($3CaO \cdot Al_2O_3$), %* | 8 | 7 | 13 | 11 | 9 | 10 |
| $C_3S$ ($3CaO \cdot SiO_2$), %* | 55 | 54 | 55 | 45 | 57 | 65 |
| $C_3S$ ($2CaO \cdot SiO_2$), %* | 18 | 20 | 17 | 28 | 15 | 9 |
| $C_4AF$ ($4CaO \cdot AL_2O_3 \cdot Fe_2O_3$), %* | 9 | 9 | 7 | 7 | 7 | 7 |

* Potential compound composition calculated from average values reported.

short "bursts" of vibration (1 to 2 s) to settle the paste back around the vanes. The molds and vanes were then covered with a thin transparent plastic sheet. Four 3-gang molds were filled as rapidly as possible. This usually required about 6 to 7 min.

In vibrating the molds, a vibrator table with frequency of 60 Hz was used with an amplitude of 0.4 mm. The molds were held on the vibrating table by hand—i.e. no clamps were used, hence the amplitude of vibration of the mold was less than that noted on the dial indicator. A minimum of vibration was used in order to avoid as much as possible the bleeding and segregation that may occur with excessive vibration.

The shear tests were normally started at 10 min after making the first specimen. In making the shear test, the load-transfer device was placed on top of a vane and universal joints and spring section lowered onto the load-transfer device. Torque was then applied to the spring by means of a worm gear and the number of degrees of rotation required to cause a shear failure (that is the maximum torque developed) was observed and noted. Measurements were made on the other vanes at various intervals as noted in the figures until the capacity of the apparatus was approached or no more vanes remained to be tested.

Tests were also made of 2 cements using 3 percentage points more water than that required for normal consistency.

A series of tests was also made on 1:2.75 (cement to graded Ottawa sand) mortars using the standard mixing procedure and the amount of water noted in table 1. The mortar was vibrated only a few seconds, cut off, trowelled once, sawed off, and the 1 cm vanes inserted and a "burst" or two of vibration was used to consolidate the mortar around the vanes. One test was made with one of the cements which had a moderate amount of premature stiffening. In this case the sand and cement were mixed dry, the water added in 5 s and the mortar mixed for only 30 s at the medium speed.

A third series of tests on these cements was made using the 2 cm vanes in 4 × 4 × 16-cm molds, placing 3 vanes in each mold.

## 5. Results of Tests

The results of tests on the neat cement pastes are presented in figures 3, 4 and 5, plotting individual shear-strength values in g/cm² versus time in minutes on a log-log scale. A series of three straight lines giving the best visual fit were drawn through the values obtained with each of the cements; the values appeared reasonably close to the lines drawn. There were, as may be noted, some values especially in the 10 to 50 min measurements which were erratic.

The slopes of the lines for the different phases of the hardening process were different with the different cements and were of different duration. Increasing the water content by 3 percentage points delayed by 10 to 20 min the start of the second phase and about 20 min the start of the third phase with cements 5 and 9.

Indicated on these graphs are the values for Vicat time of set and the times of Gillmore initial and final set values as obtained in table 1. At the time of the Vicat initial set, the shear resistance ranged from 400 to 1200 g/cm² with four of the cements (Nos 3, 6, 9, and 10); this was shortly after the start of the third phase. With two of the cements, (Nos. 4 and 5) the Vicat time of set came somewhat later. At the time of initial set as determined by means of the Gillmore needle, the six cements had a shear resistance ranging from 1300 to 3200 g/cm² with an average of about 2100 g/cm². With the apparatus used together with the size of the vanes and depth of the paste, it was not possible to obtain the shear resistance at the time of the Gillmore final set. Extrapolating the third phase lines to the time of final set would indicate a shear resistance of 10,000 to 20,000 g/cm².

The results of tests of the 1:2.75 mortars are presented in figures 6, 7, and 8. The results obtained for cements 4 and 5 were somewhat erratic,—especially in the first and second phases of the hardening process. The time of inflection in the curves obtained with the mortars are close to those obtained in the curves for the neat cement. If the lines drawn through the values obtained on the 1:2.75 mortar are superimposed on the lines drawn through the values obtained with

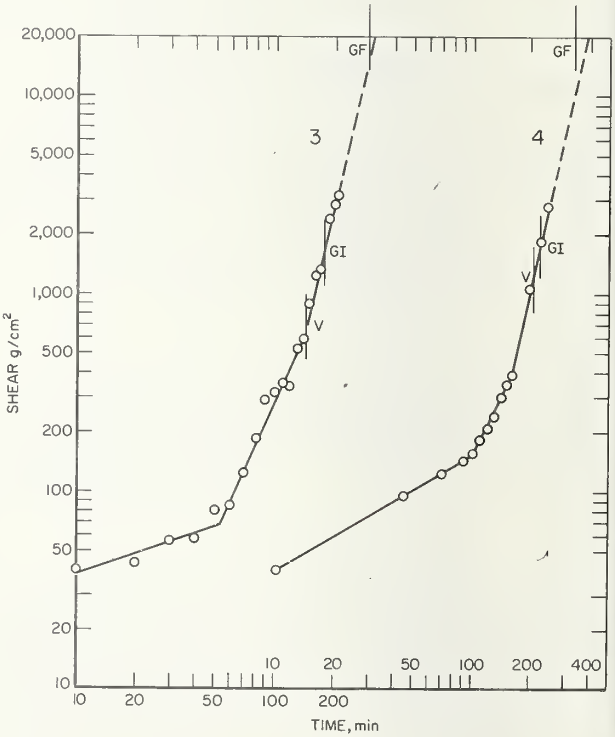


FIGURE 3. *Graph indicating shear strength in g/cm² versus time in minutes for neat pastes of normal consistencies of cements 3 and 4.*
*V*=Time of Vicat time of set, *GI*=Gillmore initial set, *GF*=Gillmore final set.

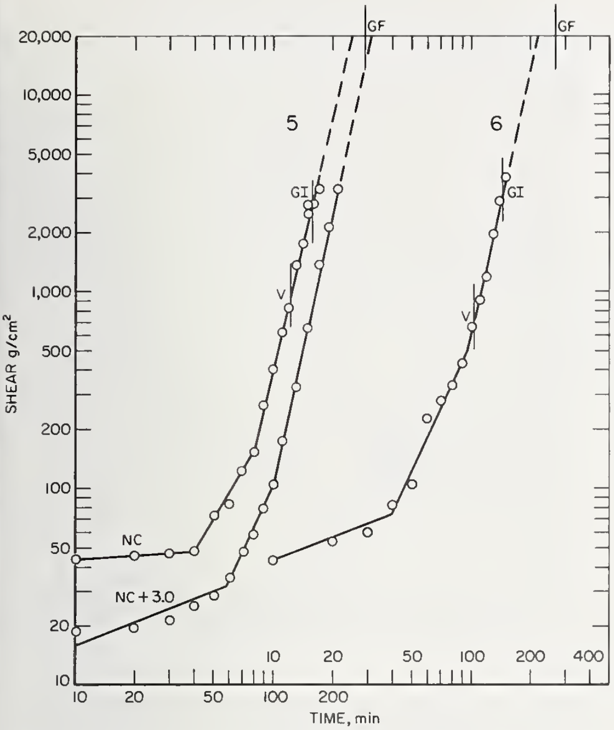


FIGURE 4. *Graph indicating shear strength in g/cm² versus time in minutes for neat pastes of normal consistency of cements 5 and 6.*
*V*=Time of Vicat time of set, *GI*=Gillmore initial set, *GF*=Gillmore final set, *N.C.*=Normal consistency,N.C. +3=Normal consistency plus 3.0 percentage points extra water.

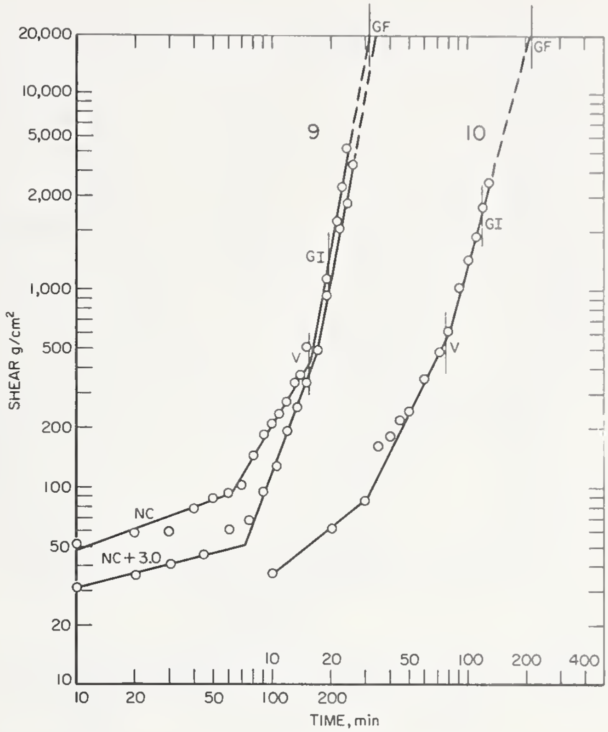


FIGURE 5. *Graph indicating shear strength in g/cm² versus time in minutes for neat pastes of normal consistency of cements 9 and 10.*
*V*=Time of Vicat time of set, *GI*=Gillmore initial set, *GF*=Gillmore final set, *N.C.*=Normal consistency,N.C. +3=Normal consistency plus 3.0 percentage points extra water.

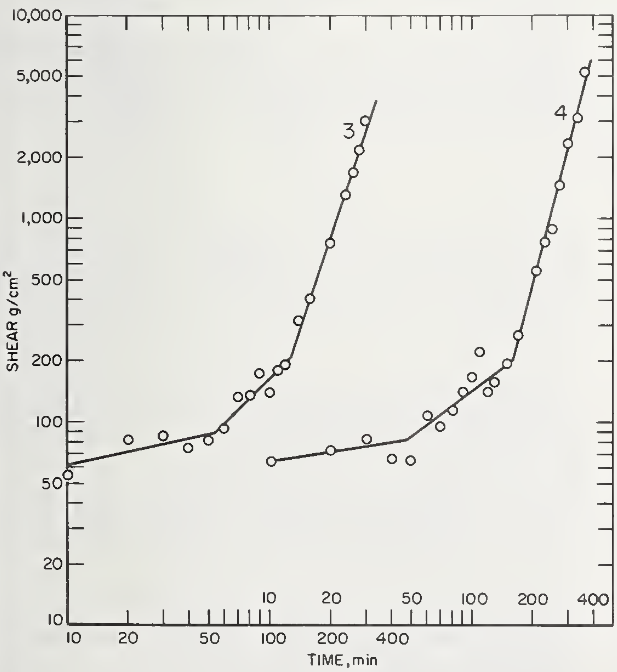


FIGURE 6. *Graph indicating shear strength in g/cm² versus time in minutes of 1:2.75 mortars of standard consistency, cements 3 and 4.*

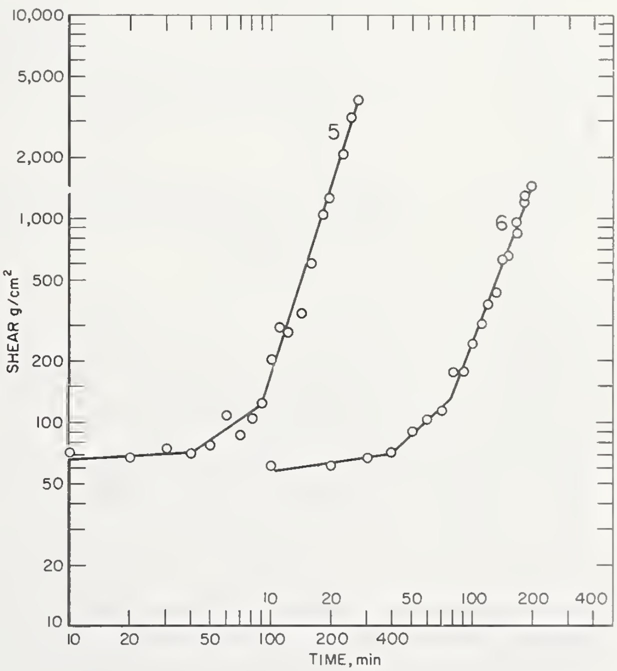


FIGURE 7. *Graph indicating shear strength in g/cm² versus time in minutes of 1:2.75 mortars of standard consistency, cements 5 and 6.*

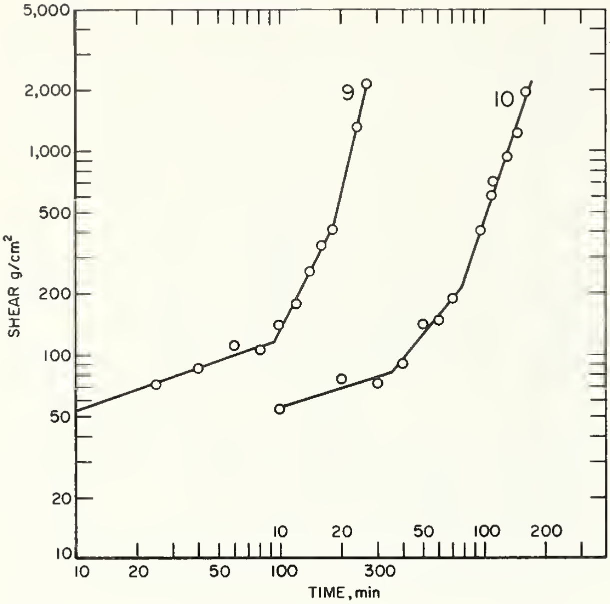


Figure 8. *Graph indicating shear strength in g/cm² versus time in minutes of 1:2.75 mortars of standard consistency, cements 9 and 10.*

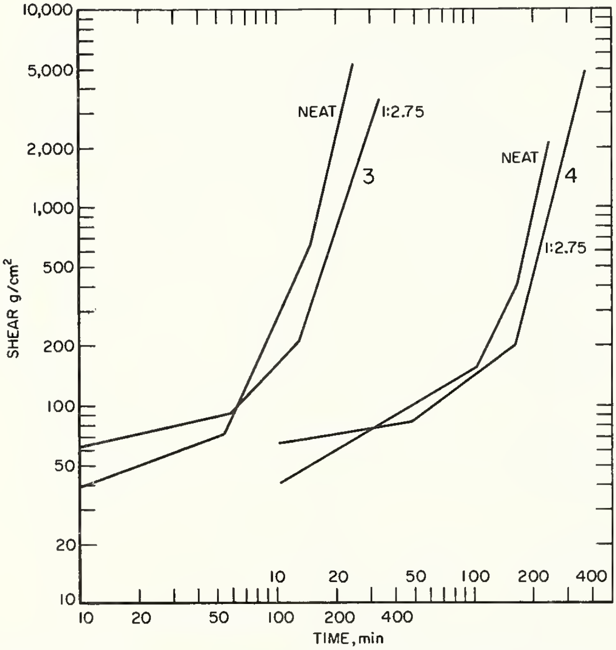


Figure 9. *Graph superimposing the shear strengths of neat cements and mortars of cements 3 and 4.*

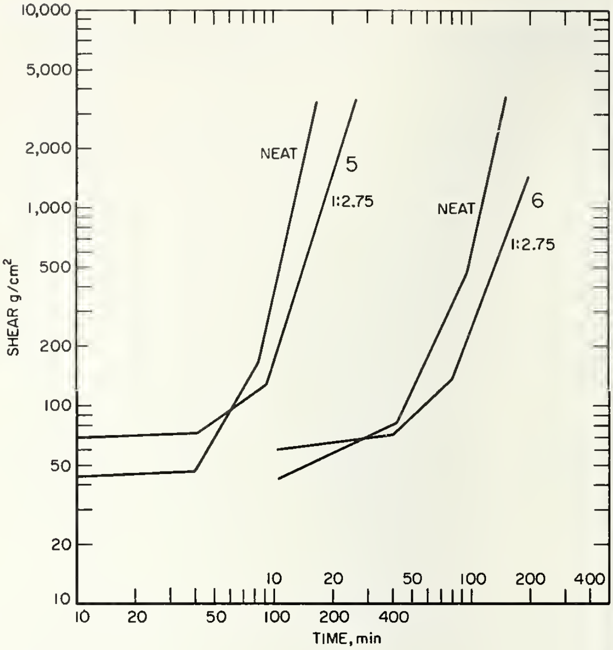


Figure 10. *Graph superimposing the shear strengths of neat cements and mortars of cements 5 and 6.*

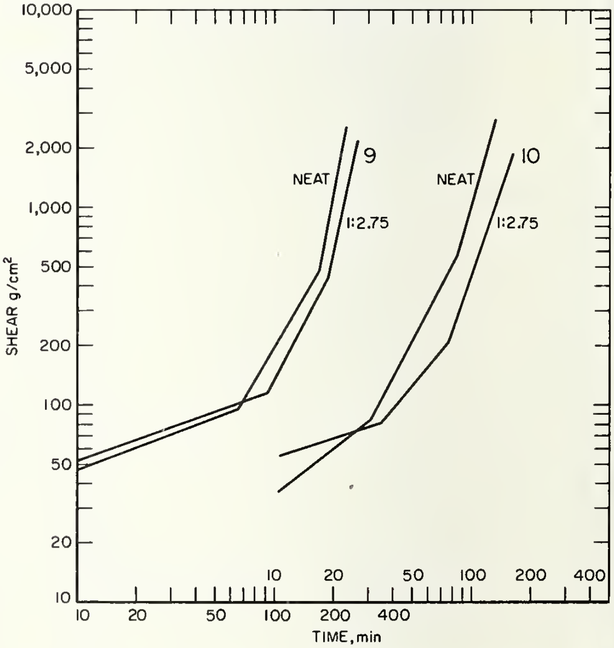


Figure 11. *Graph superimposing the shear strength of neat cements and mortars of cements 9 and 10.*

the respective neat cement pastes as indicated in figures 9, 10, and 11, it may be noted that the curves cross. Whereas, the mortars had greater shear resistance at the start of the test, the shear resistance developed more slowly for the first two phases of the hardening process and with some of the cements the lines representing the third phase appeared less steep.

A single test was made of the cement having moderate false set. The shear resistance of the mortar mixed for 30 s was higher than that occurring when the normal mixing procedure was used as indicated in figure 12. There was no increase in shear resistance for 50 to 60 min. The slope of the second phase was

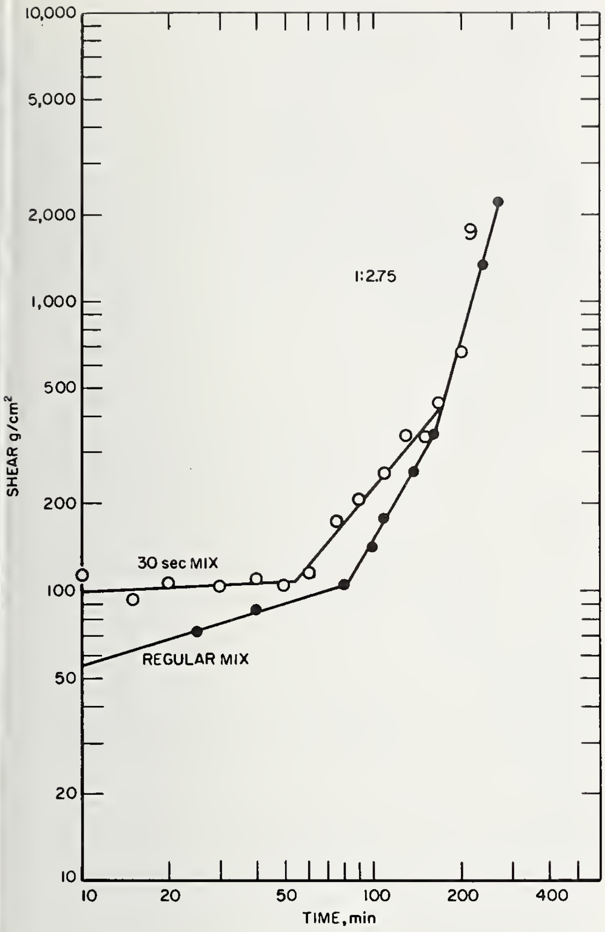


**Figure 12.** *Graph indicating the effect of time of mixing a 1:275 mortar (cement to graded Ottawa sand) on the shear strength versus time using a cement having a moderate amount of false set.*

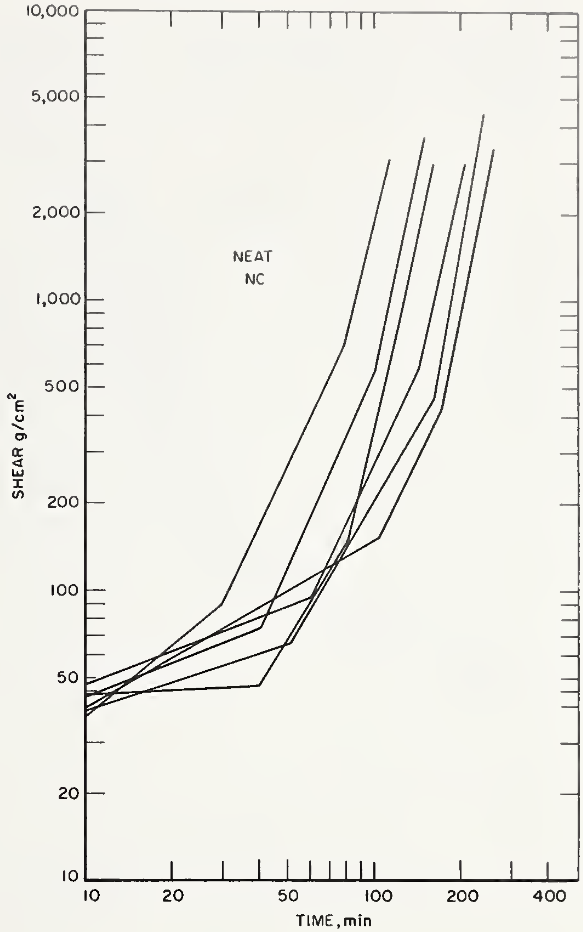


**Figure 13.** *Graph indicating the shear strength in g/cm² versus time in minutes of the six neat cements of normal consistency.*

less than that of the mortar mixed for the time required in specifications. Only one value was obtained in the third phase and this was close to the line obtained with the mortar mixed according to specification requirements.

In figures 13 and 14 the results obtained with the six cements have been traced on the same graphs. The lines of the first two phases appear to cross to a greater extent than was the case in the third phase. Most of the lines in the third phase were fairly parallel on the log-log plot but were displaced with respect to the time the phase started.

Although a log-log plot was used to show the overall picture of the hardening process, the first phase may well be presented on a linear plot graph as in figure 15. The early stiffening as measured by means of the vane shear apparatus resulted in straight lines. The slopes of the lines obtained with the different cements differ to some extent and if a shear test is used to measure the plasticity of neat cement paste or mortar, attention must be given to the time after mixing, or placing, that the test is made.

## 6. Discussion

Many theories have been proposed relating to the mechanism of the early development of structure in hardening cement pastes. These have been reviewed by Green [3] and more recently by Kondo and Ueda [6]. Among these theories were those of Segalova [19] and Rehbinder [20] who proposed, on the basis of work by a previous Russian research worker, A. A. Baikov, that there were three phases in the hardening process which may overlap in time. The first phase was a colloidization of the cement particles, especially the $C_3A$ (tricalcium aluminate) after which there is a coagulation of cement particles and reaction products and finally the development of new hydrated compounds through crystallization which brings about the strengthening.

Sivertsev [21] considered that the initial phase of the setting process was the absorption of water by the

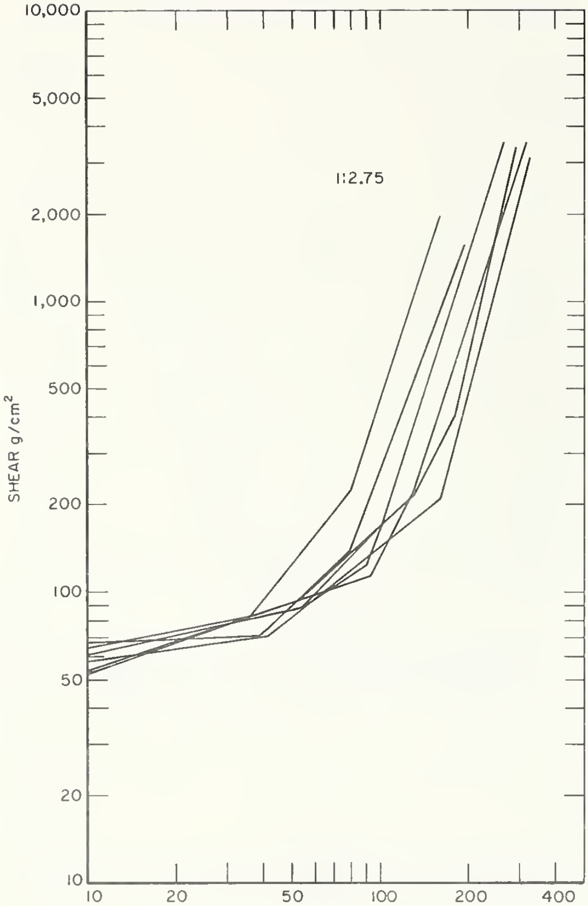


Figure 14. *Graph indicating the shear strength in g/cm² versus time in minutes of the six 1:2.75 (cement to graded Ottawa sand) mortars of standard consistency.*

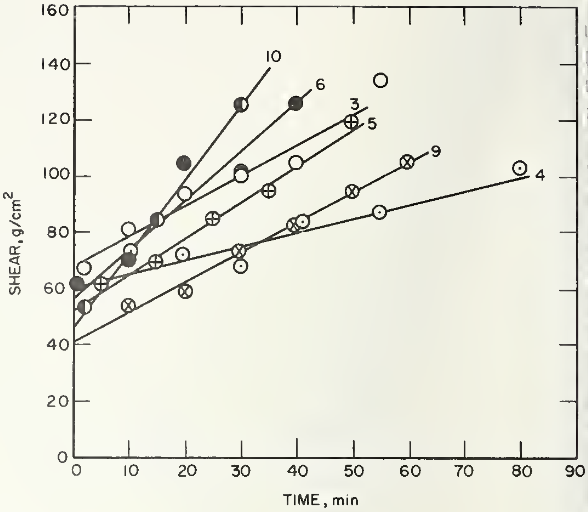


Figure 15. *Graph indicating the shear strengths in g/cm² versus time in minutes of neat cement pastes of normal consistency.*

cement particles, which was followed by formation of micelles which later coagulate into structures. Budnikov and Strelkov [22] have shown that a fibrous material is formed within the first few seconds. These fibrous materials were noted in extracts from cement-water mixtures. The extracts were dried and observed using a microscope.

Schwietes, Ludwig and Jager [23] have presented electron micrographs indicating that ettringite is formed on the $C_3A$ in the form of needles within a few minutes and lance-like foils of $Ca(OH)_2$ had also formed on the CaO.

Even after a few minutes contact with water there is (1) an apparent roughening of the cement particles (especially the $C_3A$) [23] making it more difficult for the particles to move past each other in the shear test, (2) a reduction in the amount of free water because of the approximately 31 molecules of water taken up per molecule of ettringite, lowering the water/cement ratio of the paste, and (3) a solution of some of the compounds in cement which may be absorbed on the grains of cement [22]. All or combinations of these factors may be responsible for the slight increase in shear resistance in the first phase of the hardening process.

The second phase which coincides approximately with the time of the start of the second increase in heat generated by cements may be, as suggested by Sivertsev [21], the formation of micelles which then filled the space occupied by the free water. Increasing the water/cement ratio with cements 5 and 9 increased the time at which the second phase started by 10 to 20 min. The 1:2.75 mortars with a higher water/cement ratio had generally a slightly longer first period than the neat cements with the lower W/C. In some instances, however, the times of the first inflection of the curves were very close. Early investigators in this field have indicated that the solutions are supersaturated which may account for the formation of the micelles.

The third phase, starting at 80 to 160 min with different cements after mixing with water, may indicate the start of chemical bonding of the hydration products. Cement pats at this stage will normally have lost their sheen, indicating the end of the bleeding period and the continuing uptake of water by the hydration process. It was of interest to note in comparing the curves of the 1:2.75 mortars and those of the neat cements, that the time of the start of this phase was, with each of the cements, within 10 to 20 min, irrespective of the differences in water/cement ratios and the presence of sand grains in the mortars.

It was observed in conducting the shear test that the method of failure was not always the same. In the first phase the paste or mortar built up in front of the vane and a space was noted in back of each vane. There were no visible cracks extending into the rest of the paste from the edge of the vane. As the paste became stiffer, as in phase two, cracks were noted from the edges of the vanes in the direction the blades were turning. The V-shaped cracks extended half a centimeter in some instances and the paste or mortar at time of failure was not attached to the back of the vane. This effect in soil tests has previously been noted by Wilson [16d]. No cracks were noted at the higher shear strengths and the pastes and mortars appeared attached to both sides of each vane.

Regardless of the interpretation and/or significance of the three phases of hardening of neat cement pastes and mortars observed in these exploratory tests, it appears that the vane shear apparatus offers an excellent means for studying this phenomenon. It also appears that the apparatus would offer a means for studying the effects of different admixtures on cements. The apparatus may also be used to determine the consistency of cement pastes and mortars, obtaining the values in terms of $g/cm^2$ instead of percent flow on a flow table or penetration of a cylinder or needle of specified weight and diameter.

As indicated earlier, many papers [14-18] have been published on the use of the vane-shear apparatus for measuring the shear strength of soils. Among the variables that have been studied and reviewed by Brand [18] are the vane geometry (height to diameter ratio) and the number of blades, the effect of the rate of strain, the mode of failure, the effect of inhomogeneity, anisotropy, and effect of soil disturbance. The last three items would possibly not affect the tests on neat pastes or mortars as conducted in the present studies.

More work is necessary and certain refinements in both apparatus and techniques appear desirable. In correlating these values for rate of hardening with existing time of set tests it would be desirable to have an apparatus rugged enough to measure a shear strength of 20,000 to 30,000 $g/cm^2$ (about that corresponding to final set as determined by the Gillmore apparatus) after which compressive strength measurements could be made to follow further strength development. It would have been possible to increase the range of the instrument used by using smaller vanes and a shallower depth of paste or mortar. It appeared desirable to use only the one size vane (1 cm diameter) in each series of tests, changing the springs as necessary to make the measurements of the more mature pastes. With the neat cement pastes, there was the problem of relatively large occluded air bubbles making some of the measurements of questionable value when the air bubbles were in the shear plane. The use of larger vanes would give a better ratio of diameter of vane to diameter of bubbles of occluded air, but more air bubbles would be present in the shear plane and it would not be possible with the apparatus used to follow the strength development past the time of initial set. Another problem with the small vanes was their tendency to tilt as the mortar or paste was vibrated into place. This could possibly be remedied by using some type of jig for holding them in place until after the mortar or paste had been vibrated in place around the vanes. For measurements of consistency of freshly mixed pastes or mortars, the larger vanes (2 or 3 cm) appear desirable because they do not tilt as readily as the 1 cm vanes.

In preliminary tests using a $2.5 \times 2.5 \times 28$ cm mold, the specimen cracked lengthwise instead of shearing out a cylinder. This occurred only at the higher shearing values but prompted the use of briquette molds with more rigid sides in later tests. This also helped to isolate the vanes to a greater extent.

In calculations of shear resistance the effect at the end of the vane was ignored. This may or may not have had an effect. Brand [18] indicated that with a vane of height twice the diameter the end effect would account for only 3 to 4 percent of the shear value. With a $2\frac{1}{2}$ to 1 ratio for height to diameter, the end effect should be less. Although polytetrafluoroethylene was used as a base, there was some bond between the cement paste and the plastic at the later ages.

The effect of method of placement and amount of vibration used in forming the paste and mortar specimens and setting of the vanes requires further study. Some preliminary tests with lighter gage brass vanes indicated that they were deformed at high shear values.

During the past 20 years, much research has been conducted relating the shear of soils as measured by the vane-shear apparatus and shear tests conducted by other means. There have also been published discussions of the nature of the shear, the stresses involved, the effect of pore-water pressure or dilatency at the edge of the vane, and how the linear stresses are translated to a circular stress to shear out a cylinder. Very little appears to be known with respect to the distribution of the stresses on the boundary between soil and the testing device. The authors are not aware of any photoelastic studies of the stresses generated by a turning vane of the type used. Although the apparatus appears very simple, there are, as indicated by a review of many articles on soil mechanics, still many unknown factors which need further study and clarification.

Although a four-bladed vane was used in these tests on pastes and mortars, a shear-vane having the configuration of a spline shaft may possibly be better for use in concrete in order to avoid having large aggregates in the shear plane. The edges away from the lead edges of the splines would have to be cut back or beveled to avoid friction with the mortar.

## 7. Summary and Conclusions

The results of exploratory studies indicated that a modified vane shear apparatus offers a means for determining the early strength development in portland cement pastes and mortars. The shear tests indicated what appear to be three distinct phases in the setting and early hardening process as postulated earlier by various authors as cited. Three phases were evident in each of the six cements tested and in both neat cement pastes and in cement-sand mortars. The rate of increase of shear resistance with time as well as the duration of the different phases differed with the different cements.

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# Appendix A

Modifications of the shear apparatus linkage are indicated in figure 2a.

Part "A" of figure 2a is attached to the spring portion of the apparatus by means of a set screw. Part "B" is hooked over part "A" and acts as a universal joint when torque is applied. Actually the contact is on the sides of the hook when the spring portion and part "A" are lowered onto the load transfer device "C". The upper section of part "C" is square in cross section and somewhat larger in the midsection than at the top or bottom and fits rather loosely in order to act as a universal joint with the bottom hollow portion of part "B". The bottom of part "C" has notches at right angles to fit over and engage the vanes, part "D", and transfers the torque to the vanes. Some details of the 2 cm vane are indicated in part "E".

The worm-gear for loading the spring in the apparatus used had a 30 to 1 ratio. The gear was turned at the rate of 1 revolution per second. With different strength springs, the rate of loading would then be different and no account was taken of this in computations of shear resistance.

In making a shear test, the load transfer device "C" was placed on the vane "D" which was previously imbedded in cement paste or mortar. The mold with mortar and vane were aligned under part "B" and the spring (with screw loading device and indicator) together with parts "A" and "B" were lowered onto the parts "C" and "D" until part "B" lost contact with part "A" on the horizontal part of the hook. Torque was then applied, noting the maximum number of degrees that the spring was rotated before shear failure occurred.

## Appendix B, Calibration

An inverted Tee with arms each approximately 12 cm long as indicated in figure 2b was attached to the shear apparatus in place of the vane. The cord was attached to the inverted Tee at 10 cm from the center. The cord was horizontal from the Tee to the pulley and at right angles to the Tee. The balance cradle and vertical portion of the cord from the pulley to the cradle were weighed and additional weights added, noting the combined weights and the number of degrees of spring rotation necessary to raise the balance pan off its support. The results of spring rotation with the different weights were plotted for each of the springs and the values for grams force at 1 cm radius per degree rotation calculated for use in expressing experimental shear values.



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