彙　　報

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AN ELECTRICAL METHOD FOR MEASURING THE SETTING TIME OF PORTLAND CEMENT[1]

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Synopsis

The change in electrical resistance which occurs during the setting and hardening of Portland cement, has been measured for the purpose of determining the setting time. The effect of temperature on the rate of setting of Portland cement has also been studied; this method being more accurate than the mechanical and thermal methods commonly used, and more adapted for the purpose.

(1) Introduction

When the setting time of Portland cement is measured by a mechanical method, Vicat's needle is commonly used, but there are some disadvantages owing to the fact that setting and hardening of the cement are chemical processes. Gary, however, observed that at the moment when the final set occurs the temperature reaches a maximum[(2)], and defining this moment as the final set, he tried to measure the setting time thermally[(3)]. Professor S. Uchida also determined the moment corresponding to the maximum temperature with a thermo-couple, by the differential method, and measured the setting time[(4)]; but this thermal method is not regular, that is, the time of setting is greatly affected by the quantity of the material used for the specimen. In an experiment dealing with Portland cement, the writer hit upon the following method to determine the setting time by the change in electrical resistance.

It was observed by the writer that when the electrical conductivity was

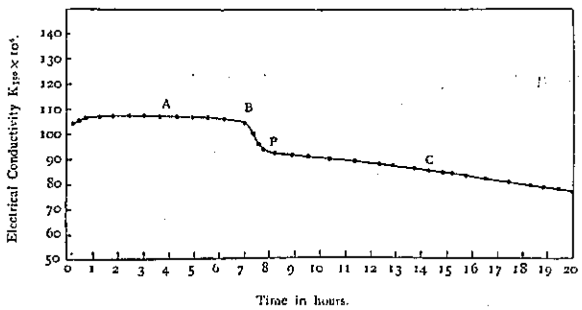
(1) The **180**th report from the Research Institute for Iron, Steel and Other Metals.

(2) W. and D. Asch consider that it is due to the separation and hydration of calcium oxide from the alumino-silicate molecule. W. Asch and D. Asch, The Silicates in Chemistry and Commerce, (1913), 189.

(3) Gary and Stradling, Conc. and Const. Eng., **1**, (1901-1907), 356, 432.

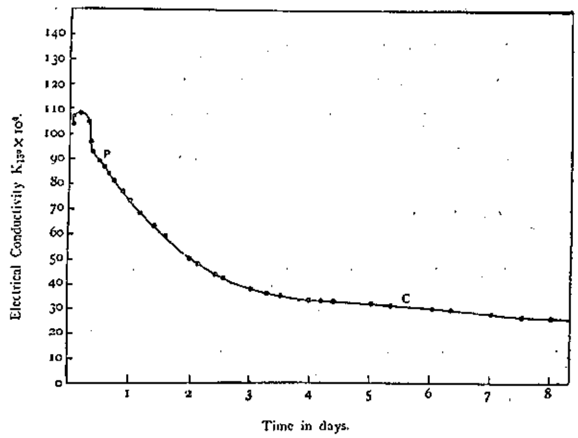
(4) S. Uchida, Sci. Rep. of the Sendai Higher Technical School, **1**, (1923), 17.

Fig. 1.



measured during the setting and hardening, the specimen being kept at a constant temperature, the electrical resistance suddenly changed at the moment corresponding to the final set, that is, to the maximum temperature described above (Fig. 1). In Fig. 1, the break point P in the curve $B\ P\ C$ is the point corresponding to the moment of the final set, i. e., to the maximum temperature, and the portion of curve $P\ C$ corresponds to the hardening, and, if the time in days is taken as the abscissa, the curve will become hyperbolic, as shown in Fig. 2. Therefore, the present writer defined the time required to reach P as the period of final set, and experiments were carried out with this method, to study the effect of temperature on the rate of setting, and also to test the accuracy of the method.

Fig. 2.



(2) Measuring Apparatus and Method of Measurement

A hardened Portland cement is electrically a semi-conductor. Owing to the phenomenon of polarisation, we cannot carry out measurements with a direct current and a galvanometer, and it is necessary to measure with an alternating current and a telephone-receiver or an electrodynamometer. Hence, for this purpose, Kohlrausch's alternating-current bridge[1] was used. Also, as the processes which occur in setting and hardening are accelerated and

(1) Kohlrausch, Lehrbuch der Praktischen Physik, 1914, 479.

electrical resistance is varied by temperature, it is absolutely necessary, for the determination of the setting time, to keep the specimen at a constant temperature, and a thermostat, therefore, was used. In Fig. 3, the experimental apparatus for this measurement is sketched. A glass tube *A* contains two platinum electrodes, and the details have been shown in Fig. 4. The dimensions of the tube are 2 cm. in diameter and 25 cm. in length, and two side tubes *U* and *T* are attached to it. After an experiment, hydrochloric acid was poured into the tube *T* to dissolve the specimen, and the dissolved specimen

Fig. 3.

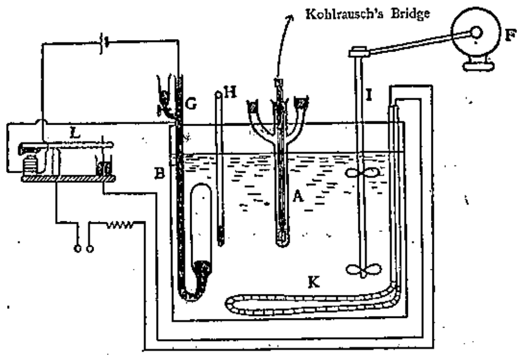
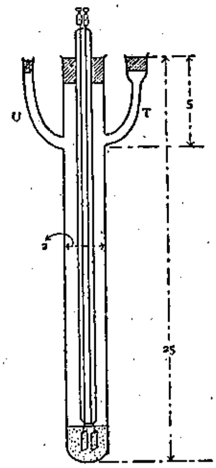


Fig. 4.



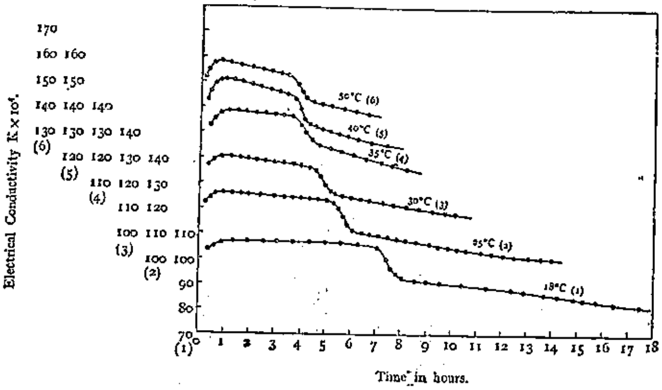
was then allowed to flow out through the other tube *U*. Also in order to avoid the loss of the gauging water, the upper ends of the glass tube were each closed with a cork. In Fig. 3, *B* is a thermostat and *F* a small motor for a stirrer *I*. *G* is a temperature regulator, *H* a thermometer, *K* a heater made of nichrome wire, and *L* a current relay, which in co-operation with *G* makes or cuts the current passing through *K* automatically, whenever the temperature is too high or too low to keep the specimen at a constant temperature.

The Portland cement used in this experiment was manufactured by the Asano Cement Co.

Before the experiment the cement water and all the other tools used for gauging were warmed for about 24 hours, in an airthermostat at a constant temperature. The quantity of the cement used in each experiment was 12 gr. and this was gauged for about 3 minutes with a quantity of water 27 % in weight. The specimen gauged with water was put into the bottom of the glass tube A, and two platinum electrodes were inserted vertically into it, thus gauged. These were set in the thermostat B, and the platinum electrodes were connected with a Kohlrausch bridge; the measurement was then begun and the readings were recorded every 5 or 10 minutes.

(3) Results of the Experiments

Fig. 5.



The results of the experiments are shown in Fig. 5 and given in table 1—6, the arm ratios in the second columns being proportional to the electrical resistance. In Fig. 5, specific electrical conductivity $K \times 10^4$ is taken as the ordinate and the time from the beginning of the gauging as the abscissa. As seen from the figure, the rate of the setting and hardening is greatly influenced by the temperature, and hence the time of the final set is remarkably shortened with a rise of temperature. At various temperatures, the changes in electrical resistance at its setting and hardening are all similar.

Table 1; 18° C.

Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	12.5	265	12.3	565	14.4	885	15.2
15	12.5	295	12.3	595	14.5	925	15.4
20	12.4	335	12.3	625	14.5	945	15.5
35	12.2	375	12.3	655	14.6	975	15.8
55	12.2	405	12.4	685	14.6	1015	16.0
75	12.2	435	13.5	715	14.7	1045	16.0
95	12.2	455	13.7	745	14.7	1075	16.1
135	12.2	475	14.0	775	14.8	1125	16.4
175	12.2	495	14.1	805	14.9	1155	16.5
205	12.3	515	14.2	825	15.0	1205	16.5
235	12.3	545	14.4	855	15.1	1235	16.8

Table 2; 25° C.

Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	11.0	250	10.5	480	12.0	850	13.0
30	10.3	270	10.5	540	12.3	880	13.0
50	10.3	290	10.6	560	12.4	910	13.1
70	10.3	310	10.6	580	12.5	940	13.2
90	10.3	330	11.2	600	12.5	970	13.3
110	10.4	340	11.5	640	12.6	1000	13.4
130	10.4	360	11.5	680	12.7	1030	13.5
150	10.4	380	11.7	700	12.7	1060	13.6
170	10.4	400	11.8	730	12.7	1090	13.8
190	10.4	420	11.8	760	12.8	1120	13.8
210	10.5	440	11.9	790	12.8	1150	13.9
230	10.5	460	12.0	820	12.9	1180	14.0

Table 3; 30° C.

Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	10.8	185	10.7	345	11.9
25	10.6	205	10.7	365	12.0
45	10.5	225	10.8	385	12.1
65	10.5	245	10.8	445	12.3
85	10.5	265	11.2	475	12.4
105	10.5	285	11.5	505	12.5
125	10.6	295	11.6	535	12.6
145	10.6	305	11.8	595	12.8
165	10.6	325	11.8	655	13.0

Table 4; 35° C.

Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	10.2	160	9.8	270	10.8
20	10.2	180	9.8	290	10.9
30	9.8	200	9.9	350	11.2
60	9.7	220	10.2	380	11.3
80	9.7	230	10.5	410	11.4
100	9.7	240	10.6	440	11.5
120	9.8	250	10.7		
140	9.8	260	10.8		

Table 5; 40° C.

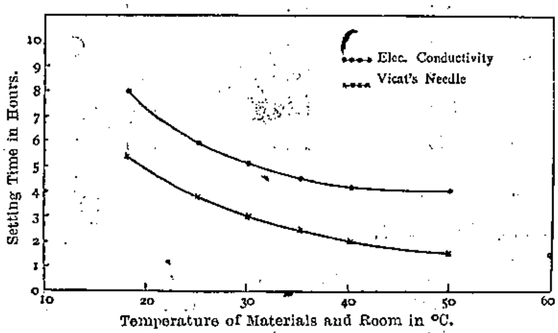
Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	9.4	165	8.9	295	10.1
20	9.0	185	8.9	315	10.2
30	8.9	205	9.0	335	10.3
45	8.7	215	9.2	355	10.3
65	8.7	225	9.4	385	10.4
85	8.7	235	9.7	465	10.7
105	8.8	245	9.9	495	10.8
125	8.8	255	9.9		
145	8.8	275	10.0		

Table 6; 50° C.

Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)	Time (Minutes)	Reading (Arm Ratio)
10	8.6	100	8.3	220	8.6	260	9.1
15	8.4	120	8.3	225	8.7	280	9.2
20	8.3	140	8.4	230	8.8	300	9.2
30	8.2	160	8.4	235	8.9	330	9.3
40	8.2	180	8.5	240	9.0	360	9.4
60	8.2	200	8.5	245	9.1	390	9.5
80	8.3	210	8.5	250	9.1	420	9.6

The relation between the setting time determined by the electrical resistance and temperature is graphically shown in Fig. 6. This curve is nearly parallel to that obtained by Professor S. Uchida with Vicat's needle(1).

Fig. 6.



This method for determining the setting time by electrical resistance is better adapted and more accurate than the mechanical and thermal methods commonly used. This method is also applicable not only to neat cement, but also to mortar and concrete.

The writer is now studying the setting time of blast-furnace cement, alumina cement, mortar, and concrete. In the case of blast-furnace cement,

(1) S. Uchida, Sci. Rep., of the Sendai Higher Technical School, **1**, (1923) 41.

two breaks of Conductivity-time curve, which were undectable by the mechanical or thermal methods, have first been observed by this method, the one corresponding to the setting time of Portland cement and the other to that of the blast-furnace slag which together constitute blast-furnace cement. It has also been found that these two breaks combine to give only one, when the quantity of Portland cement clinker becomes greater than that of blast-furnace slag and at the same time the blast-furnace cement greatly increases in compressive strength. The details regarding this point will be described in the next paper.

In conclusion the writer wishes to express his cordial thanks to Professor K. Honda and also Assistant Professor S. Uchida, under whose kind guidance the present experiment has been carried out.

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