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# Electro-mechanical impedance based monitoring for the setting of cement paste using piezoelectricity sensor

Jun Cheol Lee[1a], Sung Woo Shin[2b], Wha Jung Kim[1c] and Chang Joon Lee[*3]

*[1]School of Architecture and Civil Engineering, Kyungpook National University, 80 Daehak-ro, Buk-gu, Daegu 702-701, Korea*
*[2]Department of Safety and Engineering, Pukyong National University, 45Yongso-ro, Nam-gu, Pusan608-737, Korea*
*[3]Department of Architectural Engineering, Chungbuk National University, 52 Naesudong-ro, Seowon-gu, Cheongju 362-763, Korea*

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**Abstract.** The evolution of the electro-mechanical impedance (EMI) of a piezoelectricity (PZT) sensor was investigated to determine the setting times of cement paste in this study. The PZT sensor coated with non-conductive acrylic resin was embedded in fresh cement paste and the EMI signatures were continuously monitored. Vicat needle test and semi-adiabatic calorimetry test were also conducted to validate the EMI sensing technique. Significant changes in the EMI resonance peak magnitude and frequency during the setting period were observed and the setting times determined by EMI sensing technique were relevant to those measured by Vicat needle test and semi-adiabatic calorimetry test.

**Keywords:** electro-mechanical impedance; piezoelectricity sensor; setting; cement paste

## 1. Introduction

The chemical reactions between the cementitious materials and water lead to the result that they are subsequently transformed into rigid structures. Setting is the transitional period that the cementitious materials change from fluid to solid. As soon as the cement and water are combined, the cement dissolves quickly, releasing calcium and hydroxide ions into the water. When the calcium and hydroxide ion concentrations reach a critical value, the hydration product, calcium hydroxide and calcium silicate hydrates, start to crystallize from solution and tricalcium silicate ($C_3S$) has begun to react with the renewed vigor. At this time, the cement paste starts to solidify hence the rate of stiffness gain becomes larger compared to that in prior period. The beginning of solidification is called to the initial set. $C_3S$ continues to hydrate rapidly for a few hours, and the hydration of $C_3S$ becomes diffusion controlled. By this time, the final set has passed. At the final set, the cement paste has completely solidified and starts to gain strength (Neville 1996).

∗Corresponding author, Assistant Professor, E-mail: cjlee@cbnu.ac.kr
[a] Postdoc Research Associate, E-mail: uggenius@hanmail.net
[b] Associate Professor, E-mail: shinsw@pknu.ac.kr
[c] Professor, E-mail: kimwj@knu.ac.kr


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The setting time is an important characteristic for the cementitious materials because it affects the time at which finishing and curing operations can be initiated, steam curing can be applied, cold joints can be avoided, and load can be applied to the structures (Christensen 2006).

The most widely used methods for determining the setting time of cemenetitious materials are Vicat needle test and penetration resistance test. These test methods have the advantages that the test equipments are compact, easy to operate and available at cost effective prices. However, applying the test methods to the cast-in-place cementitious materials has some limitations. One of the limitations of the test methods is that they cannot be applied to cast-in-place materials, i.e. the tests should be conducted in laboratory by sampling the cast-in-place materials but the laboratory test cannot consider the cast-in-place environmental condition such as temperature and humidity variations. Due to this, many new test methods applicable to cast-in-place conditions have been developed; modified finisher's foot test, thermal method, electrical method, and rheological method (Abel and Hover 2000, Carino 1995, Pinto and Hover 1999 and Struble and Lei 1995). The ultrasonic method based in wave propagation have been studied to evaluate the setting time of cast-in-place cementitious materials, particularly over the recent year (Chung *et al.* 2012, Trtnik *et al*. 2013 and Yim *et al.* 2014).

Very recently, the EMI sensing technique using the PZT sensor has been investigated to monitor the mechanical properties of cementitious materials like the strength gain and the damage evolution (Shin *et al*. 2008, Shin and Oh 2009, Tawie and Lee 2010 and Wang and Zhu 2011). In most EMI sensing technique, the PZT senor is adhesively bonded on the surface of solidified cementitious materials to couple the sensor and the cementitious materials. The change of mechanical properties affects the coupled system and could be monitored by the EMI signal change of the PZT (Crawley and Edward 1987 and Liang *et al.* 1994).

This study is intended to monitor the setting behavior of cementituous material by using the EMI sensing technique which could be applied to cast-in-place environmental condition. The only deficiency of the current EMI sensing technique for setting monitoring of cementitious material is the lack of solid surface of cement paste since the material status in setting period is transformed from fluid to solid. Therefore, in this study, the PZT sensor was embedded in hydrating cement paste, and the EMI signature change was monitored during the setting period of cementitious material.

## 2. Experimental program

In this study, the evolution of the EMI of PZT sensor embedded in cement paste samples was investigated to evaluate the setting time of cementitious materials using the EMI sensing technique.

A buzzer type PZT sensor (Daeyoung electric co., Ltd, CBCG2035BAL-2) was used to monitor the EMI response. When the PZT sensor embeds in cement paste, it has short-circuited because of ions dissolved in cement paste. Therefore, the insulating coating on the surface of the PZT sensor is material to prevent a short-circuit of the embedded PZT sensor. Acrylic resin was thinly coated on the surface of the PZT sensor in this study. Fig. 1 shows the PZT sensor used in this study and Fig. 2 shows the EMI signal of PZT sensor after coating with the acrylic resin under various conditions.

As seen in Fig. 2, the EMI signatures of non-coated PZT sensor were extremely different. Whereas in case of the EMI signatures of coated PZT sensor, EMI peak magnitudes were slightly different, but clear peaks were shown. The difference of EMI peak magnitudes is mainly caused by the difference of the surrounding material of the PZT senor, i.e., the air, distilled water, cement paste and saturated calcium chloride solution. This means that a short circuit has been successfully removed, and EMI sensing can be possible even if the PZT sensor is embedded in cement paste having dissolved ions.

For preparing cement paste sample, ordinary portland cement equivalent ASTM C150 type portland cement was used (ASTM C150/C150M-12, 2012). Table 1 shows the chemical composition of the cement provided by manufacturer. Distilled water was chosen as mixing water for the cement paste sample to reduce other effects on the cement hydration. Cement pastes of various water to cement ratio (0.3, 0.4 and 0.5) were prepared in accordance with ASTM C305 (ASTM C305-14, 2014).

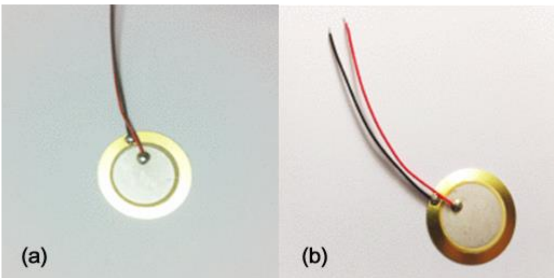
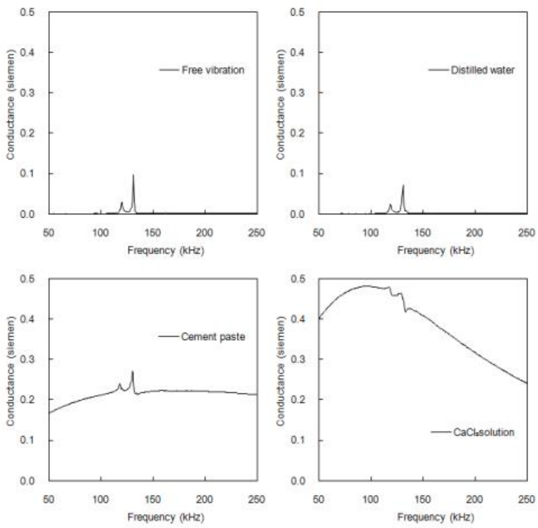



Fig. 1 PZT senor (a) before coating and (b) after coating with acrylic resin




(a) before coating

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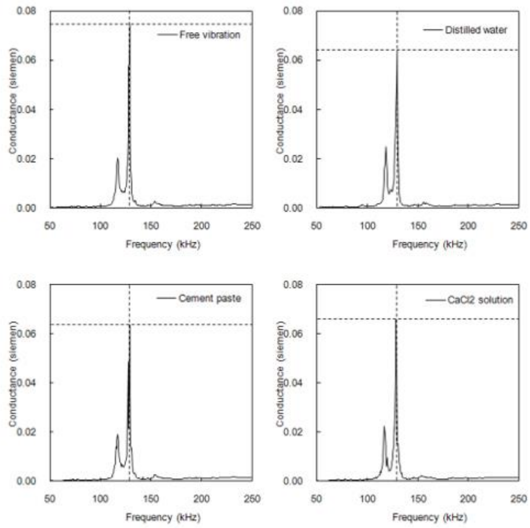



(b) after coating

Fig. 2 EMI signatures of PZT sensor under various conditions

Table 1 Chemical composition of the cement (% by weight)

| Chemicals | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $K_2O$ | Ig.Loss |
|---|---|---|---|---|---|---|---|---|
| Contents | 21.45 | 5.15 | 3.85 | 61.63 | 3.08 | 2.10 | 1.32 | 1.40 |

As soon as cement paste sample was prepared, it was cast into a cylindrical container (having a diameter of 60 mm and a height of 72 mm) for the measurement of EMI. The PZT sensor thinly coated by acrylic resin was placed at the centre of the container. After placing the PZT sensor at the container, initial measurement of EMI of PZT sensor was conducted immediately and subsequent measurement was conducted every 10 minutes for 12 hours. The EMI of PZT sensor was measured using a commercial LCR meter (HIOKI, 3235-50 LCR HiTESTER), and all measured data was recorded using a personal computer though a GP-IB interface connected to the LCR meter. The frequency range from 20 kHz to 250 kHz (500 Hz intervals) was swept for measuring EMI. Fig. 3 shows the testing setup for the measurement of EMI of PZT sensor.

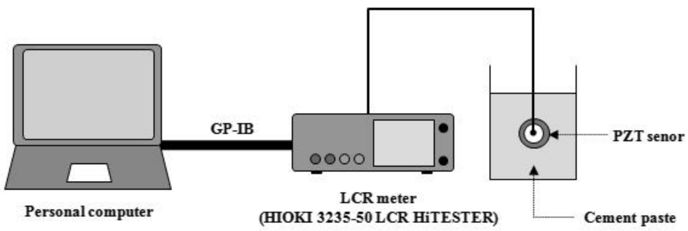



Fig. 3 Test setup for measuring EMI of PZT sensor

Vicat needle test and semi-adiabatic calorimetry test were also conducted in this experimental program in order to justify the validity of EMI sensing technique in setting monitoring of cementitious materials. Vicat needle test was conducted in accordance with ASTM C191 (ASTM C191-13, 2013). A cement paste sample was prepared and placed in a conical ring (having a height of 40 ± 1 mm, an inside diameter at the bottom of 70 ± 3 mm, and an inside diameter at the top of 60 ± 3 mm ). The steel needle with a diameter of 1.00±0.05 mm and length of 50 mm was fixed on a moveable rod of mass 300±0.5 g, and it placed on the top of cement paste sample. After the needle fell down under gravity, the penetration depth of needle was measured. The initial setting time is considered as the time when the needle penetration is 25±0.5 mm. The final setting time corresponds to less than ±0.5mm of penetration.

To measure the hydration heat of cement paste using semi-adiabatic calorimetry test, cement paste samples were poured into a container (having a diameter of 60 mm and a height of 72 mm) that was encapsulated within the polystyrene foam (having a thickness of 100 mm). A K-type thermocouple was immersed inside the cement paste samples to measure the temperature rise of the cement paste during early hydration period.

## 3. Test results

### *3.1 Setting time of cement paste by Vicat needle test*

Fig. 4 shows the penetration depth of needle for cement pastes of water to cement ratio of 0.3, 0.4 and 0.5 measured by Vicat needle test. According to ASTM C191, the initial set occurs when the penetration depth of needle reaches to 25 mm and the final set occurs when the needle dose not penetrate the cement paste anymore. The times at which the penetration depth decreases below 40mm were 180 minutes, 270 minutes, 300 minutes for the cement paste of water to cement ratio of 0.3, 0.4 and 0.5 respectively. Table 2 shows the setting time of cement pastes evaluated by ASTM C191. The results show that the initial and final setting times delayed and the time lag from the initial set to the final set increased as water to cement ratio increased.

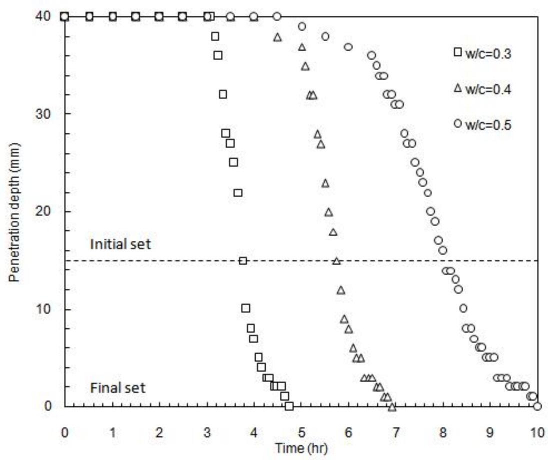



Fig. 4 Penetration depth of needle as a function of material age

Table 2 Setting times of cement pastes determined by ASTM C191 (minutes)

| Water to cement ratio | 0.3 | 0.4 | 0.5 |
|---|---|---|---|
| Initial setting time | 225 | 345 | 485 |
| Final setting time | 285 | 415 | 600 |

### 3.2 Temperature rise of cement pastes

The temperature rise of cement pastes of water to cement ratio of 0.3, 0.4 and 0.5 during early hydration period is presented in Fig. 5.

The temperature of cement pastes did not show a significant change in the early stage of hydration, and then rapidly increased at a certain time and finally reached to maximum temperature. The time at which the temperature started to increase delayed as water to cement ratio increased, and the maximum temperature of cement pastes decreased when water to cement ratio was increased. The time of the maximum temperature also delayed when water to cement ratio was increased. The times at which the temperature started to increase were 178 minutes, 198 minutes and 229 minutes for the cement pastes of water to ratio of 0.3, 0.4 and 0.5 respectively. The maximum temperatures of cement pastes were 75.2℃ at 527 minutes, 66.5℃ at 603 minutes and 56.8℃ at 705 minutes for the cement paste of water to ratio of 0.3, 0.4 and 0.5 respectively.

### 3.3 Evolution of EMI of PZT sensor

Fig. 6 shows the EMI signatures of PZT sensor embedded in cement pastes of water to cement ratio of 0.3, 0.4 and 0.5. The EMI signatures were different with the age of cement paste. Significant changes in the EMI resonance peak were observed. The magnitude of EMI resonance peak continuously decreased and the EMI resonance peak frequency continuously shifted toward higher frequency region with age, and then the EMI resonance peak disappeared after a certain time.

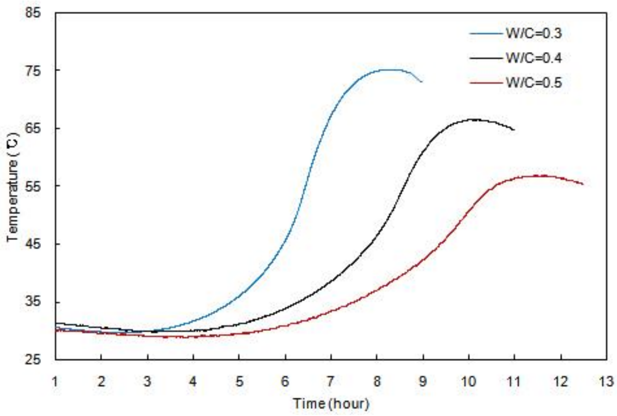
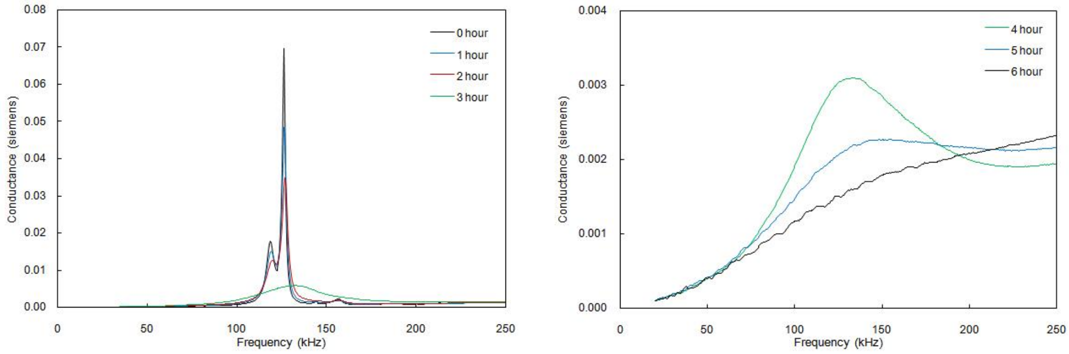
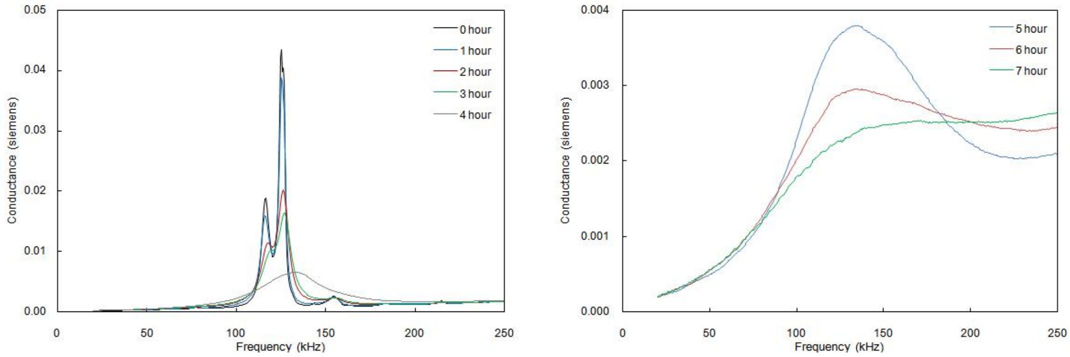


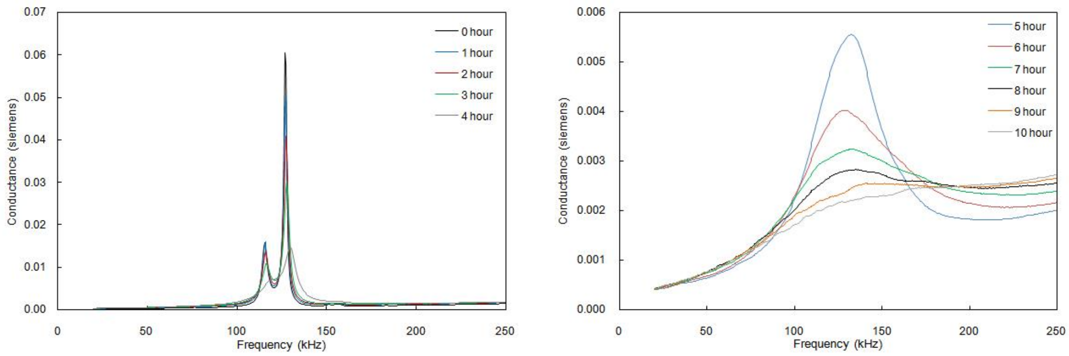

Fig. 5 Temperature of cement paste as function of material age




(a) water to cement ratio of 0.3




(b) water to cement ratio of 0.4




(c) water to cement ratio of 0.5

Fig. 6 EMI signatures of PZT sensor embedded in cement pastes

Fig. 7 shows the magnitude of EMI resonance peak with the age of cement paste. The magnitude of EMI resonance peak rapidly decreased in the early stage of hydration, and then started to decrease gradually at a certain time until the peak disappeared. In other words, there are turning points in the magnitude change curves and the curves are clearly bi-linear. Two linear regression lines were overlapped in the figure to present the abrupt slope change of the curves. The intersections of these two lines were presumed as the initial setting time of cement paste specimens in this work.

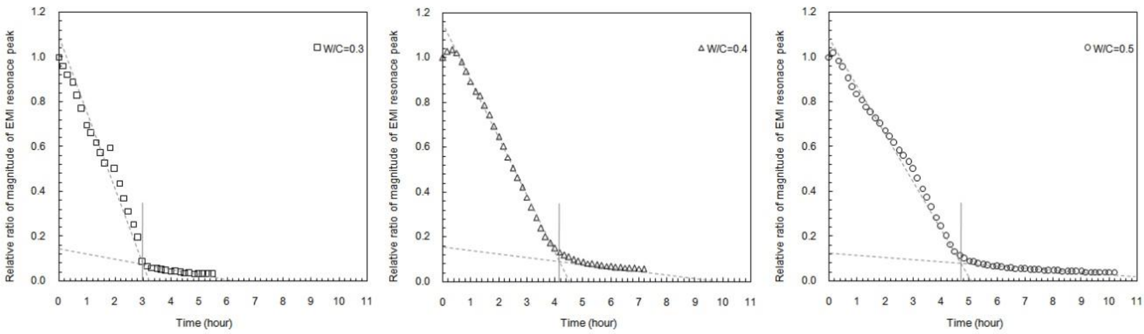



Fig. 7 Magnitude of EMI resonance peak as function of material age

The time of the turning point delayed as water to cement ratio increased. The times of the turning point were 180 minutes, 250 minutes and 280 minutes for the cement pastes of water to cement ratio of 0.3, 0.4 and 0.5 respectively. The time of disappearance of EMI resonance peak also delayed as water to cement ratio increased. The times of disappearance were 330 minutes, 430 minutes and 610 minutes for the cement paste of water to cement ratio of 0.3, 0.4 and 0.5 respectively.

Fig. 8 shows the curve of the EMI resonance peak frequency with the age of cement paste. The EMI resonance peak frequency gradually increased in the early stage of hydration until a certain time, and then started to increase rapidly until the peak disappeared. There were turning points in the peak frequency change curves. The time of turning point delayed as water to cement ratio increased. The times of the turning point were 180 minutes, 250 minutes and 280 minutes for the cement paste of water to cement ratio of 0.3, 0.4 and 0.5 respectively.

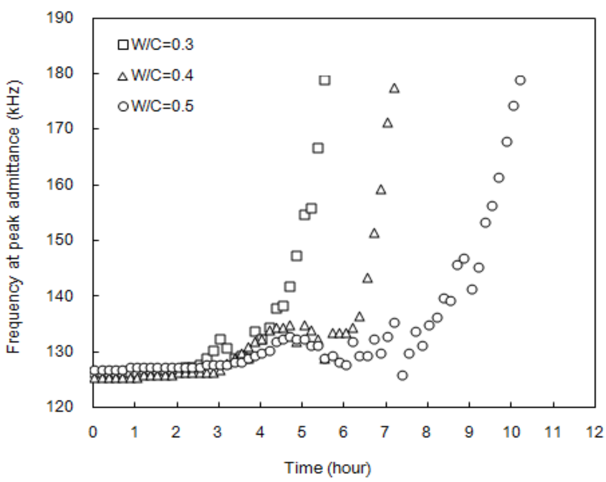



Fig. 8 EMI resonance peak frequency as function of material age

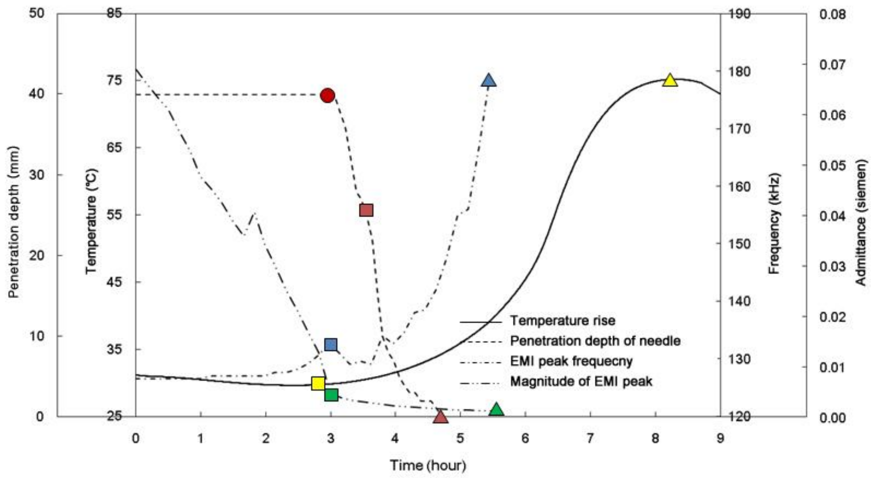
## 4. Discussion

The times of turning point in EMI peak magnitude curves and EMI peak frequency curves seem to be related with the initial setting time by Vicat needle test. The times of disappearance of EMI peak seem to be related with the final setting time by Vicat needle test. For discussion purpose, we defined the time of turning point as the initial setting time by EMI sensing technique, and also the time of disappearance of EMI peak as the final setting time by EMI sensing technique.

Fig. 9 shows the initial and final setting times by the EMI sensing technique, Vicat needle test and the temperature rise of cement paste. As shown in the figure, the final setting times by the EMI sensing technique and Vicat needle test were well matched, whereas the initial setting times by the two testing methods were not. The initial setting time by the EMI sensing technique was earlier than that by Vicat needle test.

ASTM C191 defines that the initial setting time is when the penetration of the needle is measured to be 25 mm. However, the penetration resistance of cement paste for the needle occurs earlier than the initial setting defined by ASTM C191. As seen in Fig. 4, the time at which the penetration depth decreased below 40mm, i.e., the time at which the cement pastes initially had the penetration resistance for the needle, was earlier than initial setting time defined by ASTM C191. The time at which the penetration depth started to decrease below 40mm is well matched with the initial setting time by the EMI sensing technique.

It is the most difficult problem to investigate when the setting of cement paste clearly occurs due to complex chemical reactions during the hydration process. The setting time defined by ASTM C191 dose not clearly represent the real setting of cement paste because the criteria for determining the setting of cement paste are chosen arbitrary by long experiences (Mindess *et al*. 2003).




(a) water to cement ratio of 0.3

Initial setting time by Vicat needle test
Starting time for penetration resistance for the needle
Final setting time by Vicat needle test
Initial setting time by semi-adiabatic calorimetry test
Final setting time by semi-adiabatic calorimetry test
Initial setting time by EMI resonance peak frequency
Final setting time by EMI resonance peak frequency
Initial setting time by magnitude of EMI peak
Final setting time by magnitude of EMI peak

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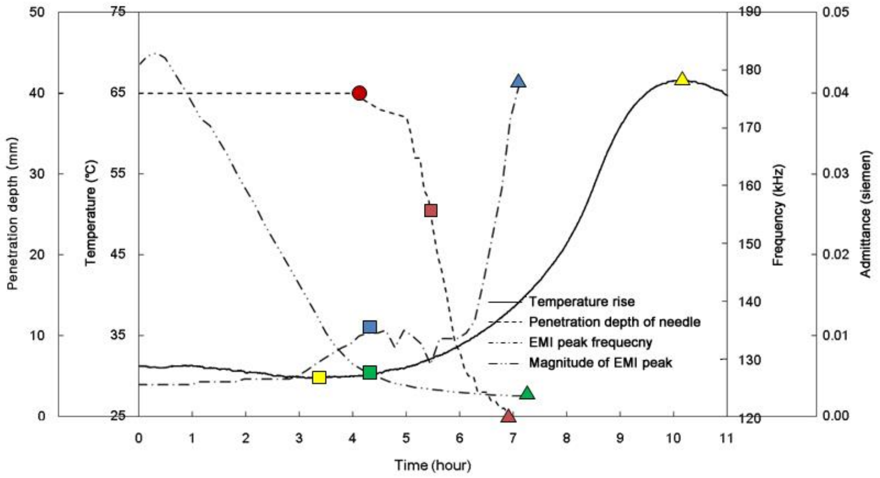
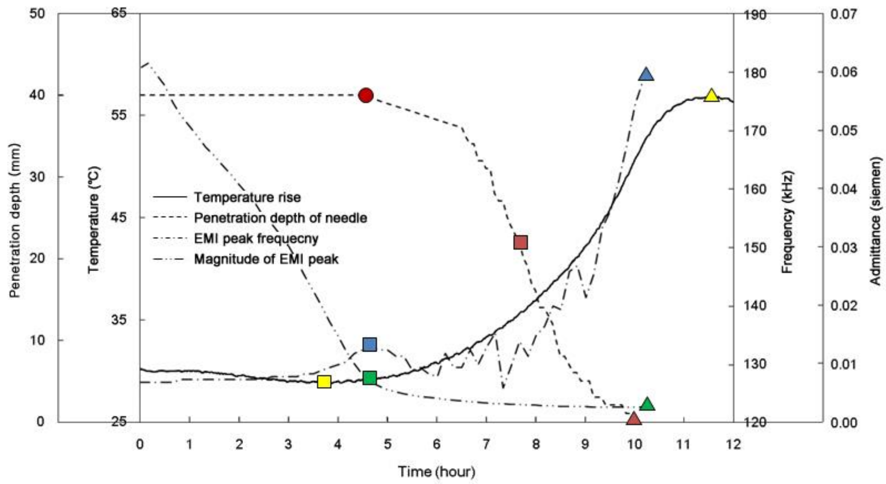



(b) water to cement ratio of 0.4




(c) water to cement ratio of 0.5

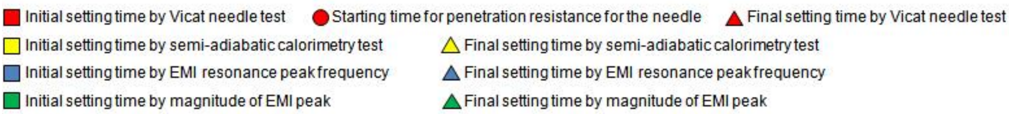



Fig. 9 The setting times by various testing methods

When the testing results of EMI sensing technique are compared with those of the semi-adiabatic calorimetry test, the setting times by the EMI sensing technique are well posed within the setting period estimated by the semi-adiabatic calorimetry test (see Fig. 9). In the cement hydration process, the temperature rise begins at the time tricalcium silicate ($C_3S$) has begun to react again with renewed vigor at the end of the induction period. At this time, initial set occurs. The hydration temperature reaches a maximum temperature at the end of the acceleration period because the silicate hydrates rapidly. By this time, final set has been passed and early hardening has begun (Neville 1996 and Mindess *et al.* 2003). The period between the beginning of temperature rise and the reaching of maximum temperature can be considered as the setting period of cement paste. Therefore, the initial and final setting times by EMI sensing technique could be one of the candidates for estimating the real setting times of cement paste.

## 5. Conclusions

This study investigated the evolution of EMI of the PZT sensor embedded in hydrating cement paste with the age. The PZT sensor coated with the acrylic resin was embedded in fresh cement paste at the time of casting and the EMI signatures were continuously monitored. During the hydration period, the magnitude of the EMI resonance peak decreased, and then the peak disappeared at a certain time. The peak frequency shifted toward higher frequency region with age. The initial setting time related with the time at which the EMI resonance peak frequency starts to increase rapidly and the final setting time related with the time of disappearance of the EMI resonance peak. The final setting times by the EMI sensing technique and Vicat needle test were well matched, but the initial setting times by the two testing methods were not. Whereas, the setting times by the EMI sensing technique are well posed within the setting period estimated by the semi-adiabatic calorimetry test. Based on the results and discussion, it can be concluded that the setting time of cement paste can be effectively monitored through the EMI sensing technique using an embedded PZT senor. This method could be utilized to determine setting times of cast-in-place cementitious materials.

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