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The Relative Humidity Range for the Development of Irreversible Shrinkage in Hardened Cement Paste

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Abstract

For better understanding of irreversible shrinkage, nine hardened cement paste (hcp) samples with three different cement types and three different water to cement ratio were prepared. Four different relative humidity conditioning histories containing the first desorption, re-humidification and the second desorption are investigated for all the specimens to obtain the length change and water sorption isotherms. The irreversible shrinkage strain was developed when the specimen was dried up to less than 80% relative humidity (RH), while other previous experiments in literatures showed that the shrinkage strain between 40% RH and 11% RH is reversible. It is concluded that the irreversible shrinkage strain is developed between 80% RH and 40% RH, which is also supported by the change in water vapor BET surface area of hardened cement paste after long-term drying.

1. Introduction

The drying shrinkage of hardened cement paste has a large influence on the properties of concrete and concrete structures, in particular the stiffness of the structure (Maruyama 2016; Sasano et al. 2018; Satya et al. 2021) and the durability of the concrete, which may be affected by shrinkage-induced cracking (Wang et al. 1997; Aldea et al. 1999). Drying shrinkage of hardened cement pastes includes irreversible and reversible strain (Helmuth and Turk 1967). Reversible strain has been mainly investigated from a viewpoint of shrinkage mechanisms (Powers 1965; Feldman and Sereda 1968; Wittmann 1968, 1982, 2008; Ye and Radlińska 2016; Ye et al. 2017), while irreversible shrinkage accounts for approximately 50% of the total shrinkage strain of the hardened cement paste (hcp) (Helmuth and Turk 1967; Maruyama 2010) with a water-to-binder ratio of about 0.50 which is corresponding to the ordinary strength concrete level. Currently, a) the shrinkage strain of the specimen dried in the range between 11% and 40 to 50% relative humidity (RH) is reversible (Helmuth and Turk 1967; Maruyama 2010), b) the specimen strongly dried (10³ mm Hg approx.) shows hysteresis in the range between 1% and 40% RH (Feldman and Sereda 1964) and c) shrinkage strain dried at 11% RH and then re-humidified shows the deviation from the strain under the first desorption branch above 40 to 50% RH and not recovered at 95 to 100% RH (irreversible strain) (Helmuth and Turk 1967; Maruyama 2010).

In addition to these, recently, some shrinkage reducing agents (SRA) are known to play an important role in the first desorption process and reduce the irreversible shrinkage (Maruyama et al. 2016a, 2018a; Rahoui et al. 2021). The SRA is active in the range from 80 to 75% RH to 40% RH (Weiss et al. 2008; Maruyama et al. 2018a; Rahoui et al. 2021). It is suggested that the irreversible shrinkage starts to form around 80% RH, but there is no direct experimental evidence. Temperature also has a strong influence on the shrinkage strain in this region (Maruyama and Rymeš 2019).

The objective of this contribution is to clarify the RH range where the irreversible shrinkage occurs. For an improved understanding of irreversible shrinkage, nine hcp samples with three different cement types and three different water-to-cement ratios were prepared in this study, and the irreversibility of the length change was investigated.

2. Experiment

2.1 Materials and curing conditions of the specimens

Ordinary Portland cement (denoted as N), moderate-heat Portland cement (M) and low-heat Portland cement (L), which were provided by Taiheiyo Cement Corporation, were mixed as a paste with a water-to-cement ratio of 0.55, 0.40 and 0.30, respectively (denoted as 55, 40 and 30, respectively). The chemical compositions and properties of these cements are shown in Table 1. To minimize segregation, the paste was subjected to periodical mixing for six hours and was cast in the mold after a creamy consistency was achieved. Specimens were cured

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for one year in lime-saturated water at 20 ± 1°C. Therefore, sufficient hydration was expected. The chemically bound water and water adsorption of specimens are summarized in Table 2. Regarding the chemically bound water, blocks (approximate size 3 × 3 × 3 mm³) of the specimens were prepared and were submerged in isopropanol to extract the evaporable water, after which they were put under vacuumed condition by aspirator for 6 hours. They were then heated at 105°C for 2 days, followed by heating at 980 to 1000°C for 3 hours. The mass difference of the samples per ignited sample mass is defined as chemically bound water. A part of the specimens heated at 105°C was submerged into de-ionized water under the vacuumed condition for 2 to 4 hours. The mass of adsorbed water per the mass of the specimen heated at 105°C was defined as water adsorption.

The specimens had a size of 3 × 13 × 300 mm³, as in a previous study (Maruyama 2010), and were conditioned in relative humidity (RH) chambers, each with a target RH of 90%, 80%, 70%, 60% and 50% at 20 ± 1°C. The RH conditions were created using sodium hydrate solutions (Stokes and Robinson 1949). This RH controlling method was selected to avoid carbonation. In this experiment, the environment temperature was 20 ± 1°C, which is different from the 25°C reported by Stokes and Robinson (1949), but this difference was not significant in this measurement, the same concentration for 25°C was adopted to this experiment. As this technique is different from the case using saturated salt solution, solution was changed after one month of RH conditioning. In the previous experiment (Maruyama 2010), significant carbonation was not confirmed in similar experiments conducted recently by the authors using saturated salt solution (Rahoui 2018; Rahoui et al. 2021), carbonation was not confirmed either. Therefore, it was not expected that carbonation reaction occurred in the present study. The RH in the chamber at the time of the mass and length change measurements was within 2% of the target RH.

2.2 Length change and water sorption isotherms
The length change was measured with a contact displacement meter with an accuracy of 0.001 ± 1 mm. For the measurement, small contact force (about 1 N) and round contact tip were applied to measure the same contact position of the specimen. The elongation was determined by the difference using an Invar reference with a length of 300 mm. The deformation strain was given as,

\[ \epsilon = \frac{L_{\text{dry}} - L_{\text{ini}}}{L_{\text{ini}}} \]  

where \( L_{\text{ini}} \) is the length of the specimen at the beginning of the experiment, \( L_{\text{dry}} \) is the length of the specimen after conditioning and \( \epsilon \) is the strain of the specimen.

The evaporable water content at the beginning of the experiment was determined by calculating the difference in mass between the wet condition and the dried condition at 105°C for 24 hours according to,

\[ w_{\text{ini}} = \frac{M_{\text{ini}}}{M_{\text{dry,105}}} - 1 \]  

where \( w_{\text{ini}} \) is the evaporable water content at the beginning of the experiment, \( M_{\text{ini}} \) is the mass of the specimen at the beginning of the experiment and \( M_{\text{dry,105}} \) is the mass of the specimen after drying at 105°C for 24 hours. After the RH-conditioning, the mass of the specimen was recorded. Based on this value, the evaporable water after the RH-conditioning was calculated according to,

\[ w_{\text{RH}} = \left( w_{\text{ini}} + \frac{M_{\text{dry,105}} - M_{\text{ini}}}{M_{\text{ini}}} \right) - 1 \]  

where \( M_{\text{RH}} \) is the mass of the specimen after the RH-conditioning.

There are four different RH-conditioning histories for each mixture, as summarized in Table 3. The specimens were stored at 80%, 70%, 60% and 50% RH for two months, and the changes in length and mass were recorded. This first drying step, which is followed by the re-humidification steps, is referred to as the “turnback”
The specimens were then moved to the chambers with higher RH conditions and stored for one month, after which changes in length and mass were recorded. The process was repeated up to the conditioning at 95% RH. Finally, after conditioning at 95% RH, the specimens were again stored in the original RH chamber for one month, and the changes in length and mass were recorded. Based on our preliminary experiments, even after the 1 year, it is difficult to obtain the complete equilibrium state on mass and length change. Therefore, 2 months of drying, which covers more than 80% of mass and length change after drying, is considered sufficient to observe the irreversible shrinkage of C-S-H.

### Table 3: History of the RH-conditioning for each mixture.

| Notation           | Initial condition | Step 1 (2 months) | Step 2 (1 month) | Step 3 (1 month) | Step 4 (1 month) | Step 5 (1 month) | Step 6 (1 month) | Step 7 (1 month) |
|--------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| 80% RH-turnback    | Saturated condition | 80%               | 90%               | 95%               | 80%               | -                 | -                 | -                 |
| 70% RH-turnback    | Saturated condition | 70%               | 80%               | 90%               | 95%               | 70%               | -                 | -                 |
| 60% RH-turnback    | Saturated condition | 60%               | 70%               | 80%               | 90%               | 95%               | 60%               | -                 |
| 50% RH-turnback    | Saturated condition | 50%               | 60%               | 70%               | 80%               | 90%               | 95%               | 50%               |

### 3. Experimental results and discussion

#### 3.1 Length change isotherms

The length-change isotherms are summarized in Fig. 1. As it is mentioned in Section 2.2, complete equilibrium is not obtained in the first desorption process. Based on a preliminary experiment (Rahoui 2018), re-humidification for 2 weeks is enough to obtain sufficient strain recovery at equilibrium. As the strain recovery is one-month step, sufficient recovery strain must be obtained. The following characteristics were found from Fig. 1(a): 1) the 80% RH-turnback shows a linear relationship between the RH and shrinkage strain, suggesting a lack of hysteresis; 2)
the second desorption for the 80% RH-turnback showed a smaller shrinkage than the first desorption shrinkage at 80% RH (the second desorption plot is above the first desorption plot at 80% RH); 3) at the re-humidified 80% RH condition, the 70% RH-turnback showed nearly equal shrinkage to that of the first drying shrinkage at 80% RH (at 80% RH, the shrinkage strain of 70% RH-turnback is at or above the shrinkage strain of 80% RH-turnback), while for the higher RH conditions, the shrinkage strain of 70% RH-turnback was larger than that of the 80% RH-turnback at 90% and 95% RH; 4) the second desorption plot of the 70% RH-turnback was nearly equal to that of the first desorption plot; 5) the 60% and 50% RH-turnbacks showed a greater shrinkage than for the 80% and 70% RH-turnbacks.

These trends are almost consistent with those observed for N40, N30, M55, M40, M30, L40 and L30. Based on these trends, it was concluded that the length-change irreversibility was developed at a relative humidity of less than 80% RH. In general, qualitative trends of the impacts of water to cement ratio on drying shrinkage and irreversible shrinkage is consistent to the previous studies (Helmuth and Turk 1967; Bentur et al. 1979), while a new finding is that the shrinkage at 80% RH can be recovered. It should be noted that the almost linear relationship between RH and drying shrinkage strain at the target RH from the saturated condition in the range above 80% RH at 20 ± 1°C was confirmed. In case of low heat cement, sudden drop of drying shrinkage strain from the saturated condition to 95% RH (Maruyama 2010).

Comparing the results of N55, N40 and N30, there is a clear trend showing that shrinkage is reduced when the water-to-cement ratio is reduced. For a turnback RH, the irreversibility can be defined as the difference between the shrinkage strain of the 80% RH-turnback at 95% RH [denoted as \( \varepsilon_{irv}(95\%) \)] and the shrinkage strain of the corresponding sample at 95% RH [denoted as \( \varepsilon_{irv}(95\%_{s}) \)], given as,

\[
\varepsilon_{irv}(RH_{turn}) = \varepsilon_{irv}(95\%) - \varepsilon_{irv}(95\%_{s})
\] (4)

It can be seen from Fig. 2 that the irreversible shrinkage is reduced as the water-to-cement ratio is reduced. For water-to-cement ratios of 0.55 and 0.4, with a decrease in the turnback RH corresponded to a faster increase in the irreversible shrinkage for L compared with N and M. However, for samples with water-to-cement ratios of 0.30, the highest increase in irreversible shrinkage was observed in M. Interestingly, the ratio of irreversible shrinkage of the sample turnback at RH = 50% to the shrinkage at 50% RH was almost 50% for N and M. The RH range in which irreversible shrinkage developed seems to have shifted to lower values, since the shrinkage at the 80% and 70% RH-turnback were similar for N40, M40, M30, L40 and L30.

The results for L55 differed significantly from those of N55 and M55. The shrinkage for L55 at 80% RH was more than 3000 \( \mu \)m/m, while for N55 and M55, it was approximately 2000 \( \mu \)m/m. The results suggest that the irreversibility of L55 was developed even at 80% RH. A strong inflection point was apparent for L55 at 80% RH, which can be observed to an extent in the other cases.

### 3.2 Water sorption isotherms

The water sorption isotherms are shown in Fig. 3. For N55, the following characteristics can be observed: 1) a slight hysteresis occurred in the 80% RH-turnback at 95% RH; 2) the first re-humidifying process (Step 2 in Table 3) of the 70%, 60% and 50% RH-turnbacks demonstrate smaller water sorption compared with the water sorption at the first desorption (Step 1 in Table 3), 3) an irreversible water content at 95% RH, which is defined by the water content of the 80% RH-turnback, and the water content of the other specimens increased as the RH of the first desorption decreased (Step 1 in Table 3), and 4) the water sorption of the second desorption (the rightmost step in Table 3) was always smaller that of the first desorption (Step 2 in Table 3).

These tendencies were confirmed by the other cases. Only for the 70% RH-turnback for N50, M30 and L30 was the water sorption of the second desorption slightly higher than for the first desorption. While in general, the trends are consistent with those observed in previous studies (Feldman 1968; Baroghel-Bouny 2007; Ranaivomanana et al. 2011; Jennings et al. 2015; Pinson et al. 2015; Saeidpour and Wadso 2015), it is likely that the measurements from this study were not at a complete equilibrium state, especially in the desorption branch; In our previous research on white Portland cement with
water-to-cement ratio of 0.55, it was confirmed that a further slight change in the water vapor Brunauer-Emmett-Teller (BET) surface area ($S_{BET}$) developed from three months to twelve months of drying (Maruyama et al. 2014a).

The mass reduction in the re-humidifying process can be explained by the hypothesized inequivalent condition associated with the dynamic microstructural change. In previous experiments using $^1$H NMR relaxometry, it was confirmed that layered C-S-H agglomeration structures contain gel pore spaces and interlayer spaces (McDonald et al. 2007), and that the slit size in the pore structure dynamically changes according to the drying state (Maruyama et al. 2019, 2020). Due to the narrowing of gel pores in the higher relative humidity range above 80% RH followed by the narrowing of the interlayer spaces below 80% RH, the moisture transport changes dramatically, as confirmed by previous studies [refer to Figs. 4 and 5 of Poyet (2009), Fig. 18 of Maruyama et al. (2014b) and Fig. 11 of de Burgh and Foster (2017)].

Regarding the C-S-H sheets’ local structure, it is possible that the end members of the C-S-H slits, which are outlets facing the large pores located between C-S-H agglomerations, have narrowed spaces and the water inside the C-S-H sheets structure is trapped like in an ink bottle. It should be noted that all the present experimental results are in transient conditions and that the water is moving toward the equilibrium state. Due to the narrowing of the outlet slit width and the high pore solution viscosity in narrowed C-S-H slits, the rate of drying of the entrapped water inside the C-S-H agglomeration is very small. This explains why the water transport in the mid-RH range (40 to 70% RH) was slower than in other RH ranges, presenting a challenge for reaching equilibrium experimentally. Consequently, in the re-humidifying process, the outlet slit spaces increased during humidification, and a fraction of the entrapped meta-stable water (which could be in equilibrium at more than 80% RH when considering the gel pore size) was exuded through the widened outlet slit. The instantane-

![Fig. 3 Water sorption isotherms.](image-url)
ous transition when the saturated specimen was placed in the mid-RH range may be the reason for the increase in the meta-stable water content in the specimen. This meta-stable water causes cavitation when the specimen reaches 35% RH at room temperature (Maruyama et al. 2018b).

3.3 Microstructural changes
Irreversible shrinkage has been associated with the microstructural change caused by the rearrangement of the C-S-H agglomeration structure (Helmuth and Turk 1967; Bentur et al. 1979; Maruyama et al. 2014a, 2015, 2016b; Bogner et al. 2020; Soja et al. 2020). It has been proposed that the water vapor sorption isotherms obtained by the volumetric (or manometric) method using rapid pre-treatment can capture the re-arranged C-S-H microstructure after long-term drying. Even though a complete understanding of pores that are accessible to the water molecules is still lacking [discussions can be found in Feldman and Sereda (1968) and Brunauer et al. (1970)], a difference in the sorption isotherms after different drying conditions can be obtained. An example is shown in Fig. 4. In this figure, the $S_{\text{H}_2\text{O}}$ of the samples after long-term drying (approximately three years) is normalized to that of the sample under the saturated conditions. From this figure, three trends are confirmed. First, from the saturated state to 80% RH, the $S_{\text{H}_2\text{O}}$ did not change; however, from 80% RH to 30% RH, the $S_{\text{H}_2\text{O}}$ decreased. Below 30% RH, there was no change in $S_{\text{H}_2\text{O}}$. When the strain data, BET surface area change after long-term drying, and recent data of $^1$H NMR relaxometry results (Maruyama et al. 2019, 2020) are compared, a new view of C-S-H alteration is obtained. Based on the $^1$H NMR relaxometry, above and equal to 80% RH, $T_2$ (a decay constant for transverse components of magnetization decay process) of interlayer space in C-S-H will not be changed during the drying. This means that the distance of C-S-H sheet is not changed at relative humidities of 80% and above, which indicates that there is no possibility of chemical reaction between C-S-H sheets, such as forming Si-O-Ca-O-Ca-O-Si due to drying, and that thermodynamic equilibrium under the drying condition above and equal to 80% RH will not produce the condition that the water in C-S-H sheets is removed. Naturally, due to the additional hydration and resultant change in pore solution chemistry, additional aging / progress of reaction can happen to C-S-H, but it is not due to the drying. This hypothesis is evidenced by that the shrinkage strain at 80% RH was reversible and the BET surface area of hcp conditioned above and equal to 80% RH is not changed.

It should be noted that this measurement is a relatively quick measurement, while long-term shrinkage is associated with microstructural rearrangement. Helmuth and Turk (1967) showed that the length change between 47% RH and 11% RH is reversible, and previous measurements have suggested that the reversibility occurs between 40% RH and 11% RH (Maruyama 2016). Based on these considerations, it was concluded that the irreversibility of long-term drying shrinkage is developed between 80% RH and 40% RH.

4. Conclusions
To improve the understanding of the irreversible shrinkage in hardened cement paste, measurement of shrinkage and mass change of hcp specimens, water vapor BET surface area of hcp conditioned in the different RH are shown. The results are discussed with the recent $^1$H NMR relaxometry data in which the interlayer distance of C-S-H sheets will not change under the drying above and equal to 80% RH, and it is concluded that the starting of creation of irreversible shrinkage or irreversible C-S-H alteration is in the range less than 80% RH. These results are well consistent with the RH range where the shrinkage reducing agent played an important role to reduce the drying shrinkage. With the previous research results, it is concluded that the irreversibility for long-term drying shrinkage develops between 80% RH and 40% RH.

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