



CI-PREMIER PTE LTD

150 Orchard Road #07-14, Orchard Plaza, Singapore 238841.
Telephone: +65-6733 2922, Fax: +65-6235 3530
Company Reg. No. 4198/80

EFFECT OF TEMPERATURE TO MICRO-DISPLACEMENT OF INTERFACIAL TRANSITION ZONE IN CEMENTITIOUS MATERIALS BY ESPI MEASUREMENT

Huang-Hsing Pan*, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Jhan-Cheng Li, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Tsung-Chin Hou, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Kun-Sheng Liao, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan

35th Conference on **OUR WORLD IN CONCRETE & STRUCTURES**: 25 - 27 August 2010, Singapore

Article Online Id: 100035043

The online version of this article can be found at:

<http://cipremier.com/100035043>

This article is brought to you with the support of

Singapore Concrete Institute

www.scinst.org.sg

All Rights reserved for CI-Premier PTE LTD

You are not Allowed to re-distribute or re-sale the article in any format without written approval of
CI-Premier PTE LTD

Visit Our Website for more information

www.cipremier.com

EFFECT OF TEMPERATURE TO MICRO-DISPLACEMENT OF INTERFACIAL TRANSITION ZONE IN CEMENTITIOUS MATERIALS BY ESPI MEASUREMENT

Huang-Hsing Pan*, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Jhan-Cheng Li, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Tsung-Chin Hou, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan
Kun-Sheng Liao, Kaohsiung University of Applied Sciences, Kaohsiung, Taiwan

Abstract

We use electronic speckle pattern interferometer (ESPI) to measure the micro-displacement of interfacial transition zone (ITZ) in cementitious materials under various temperature conditions. ESPI is a non-contact measurement technique, and is capable of capturing the dynamic displacement of ITZ up to 10nm under temperature fluctuations. In this study, the ITZ micro-displacement is observed at top, bottom, left and right points of a single aggregate, respectively. Experimental results show that the maximum displacement of ITZ occurs at temperature 230°C to 330°C and is depending on the position around the aggregate. We observed that material properties (displacement) of ITZ experience an inverse change as the applied temperature goes beyond 230°C. The observed out-plane displacement of ITZ shows a more stable relationship with respect to the distance than in-plane displacement at all temperatures. A criterion to determine the ITZ is also presented based on ESPI measurements. We have concluded that the ITZ at bottom point of an aggregate is 80 μm for in-plane and 60 μm for out-plane evaluation. For all the other positions of an aggregate, the ITZ is 40 μm . The results also show that we can determine ITZ by out-plane evaluation at temperature above 50°C.

Keywords: ITZ, Cementitious materials, Displacement, ESPI

1. Introduction

In microscopic view, cementitious materials can be considered as a three-phase composite consisting of binder, aggregates and interfacial transition zone (ITZ). Among those, interfacial transition zone is considered as the weak point that dominates the strength of concrete [1]. Many microscopic measurement techniques have been used to investigate the compositions and microscopic structure of ITZ, such as scanning electron microscopy (SEM) [2-6], backscattered electron images (BSE) [7], environmental scanning electron microscopy (ESEM) and optical microscopy (OM) [8-9]. Other instruments such as X-ray diffraction test [4,10], mercury intrusion porosimetry (MIP) [9,11] and micro-indentation test [5,12] were also chosen to investigate the material properties of ITZ. Most of

them distinguish the realm of ITZ by exploiting image, the porous and loose characteristic in ITZ. Some researchers have also discussed the microstructural features, stress-strain relation and microcracks of ITZ at various temperatures [13-15].

In this study, we attempt to investigate the micro-displacement of ITZ in cementitious materials induced by the temperature from 30°C to 420°C. The composite specimens consist of cement mortar and one single sand stone (coarse aggregate). Interfacial transition zone lies in between the mortar and the coarse aggregate. The displacement in ITZ was measured at the material age of 28 days by ESPI (electronic speckle pattern interferometer), a non-contacted measurement different from SEM. The interfacial transition zone between the mortar matrix and coarse aggregate is also determined by ESPI approaches.

2. Experimental program

2.1 Materials

The mortar used in this study composed of Type I Portland cement with $w/c=0.35$ and 20% quartz sand with the particle size about 0.7-1.0mm and the specific gravity of 2.65. A 50×50×50mm specimen cube was made with mortar matrix and a cylindrical sand stone (aggregate) with ϕ 10×15mm located at the center of the specimen, as shown in Fig.1. The specific gravity of the sand stone aggregate is 2.74 with absorption of 1.24%.

The cast specimens were then cured at room temperature (25°C) for 28 days before temperatures applied onto them. In this study, we investigate the micro-displacement of the cement specimens at six kinds of temperature degrees- 30°C, 50°C, 130°C, 230°C, 330°C and 420°C, respectively. Temperatures were applied by surrounding the specimens into a chamber of heater with heating rate of 2°C/min starting at room temperature. The investigated area of displacement is depicted in Fig.2.

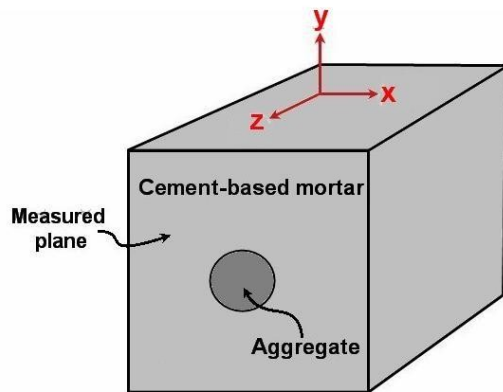


Fig.1 Cubic specimen with the size of 50×50×50mm

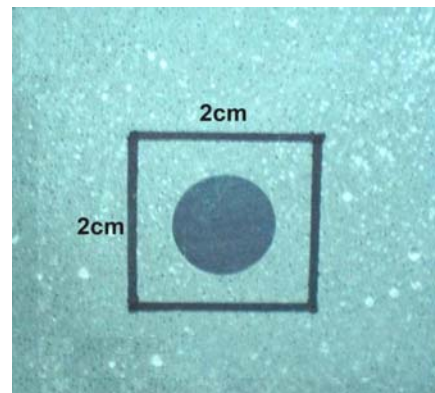


Fig.2 Investigated area (20×20mm)

2.2 ESPI measurements

The micro-displacement of the specimen was measured by ESPI with a wavelength of 532μm and precision of 25nm at various temperature degrees mentioned previously. The in-plane and out-plane displacement within the measured plane were both observed, where the displacement in x-y plane

represents in-plane displacement, and in the z-direction is out-plane, as shown in Fig.1. The displacement of the specimen can be found by calculating speckle pattern observed by ESPI shown in Figs.3-4. The image converted from ESPI can display the displacement with 10nm per contour line. It should be mentioned that in Fig.3 and Fig.4, U-field interfere pattern was used to measure the in-plane displacement and W-field measures the out-plane displacement of the specimens.

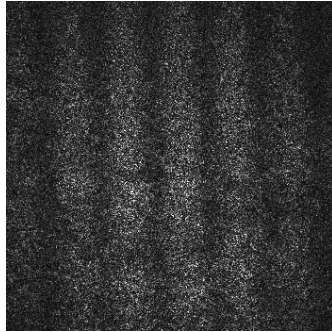


Fig.3 Interferogram in U-field

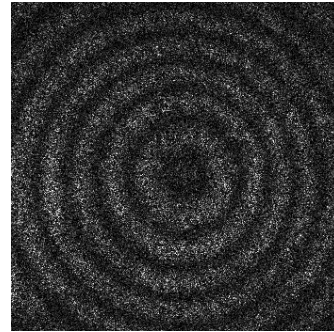


Fig.4 Interferogram in W-field

Our experiments focus on observing the micro-displacement in a 20x20mm investigated area, particularly in the range of ITZ in between mortar matrix and single sand stone aggregate, as marked by T (top), B (bottom), L (left) and R (right) in Fig.5. The specific investigating positions, for example, at R point are d_0 (right on the aggregate edge), d_1 (10 μm away from d_0), and sequentially up to d_{12} (120 μm away from d_0), as shown in Fig.6. The corresponding observed micro-displacements with respect to each observing position are denoted as $\delta_0, \delta_1, \delta_2, \dots$ etc.

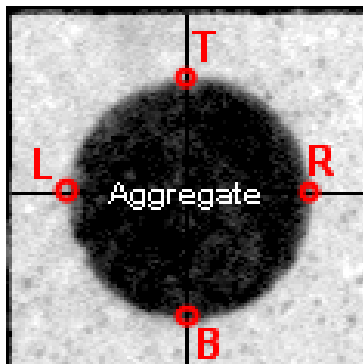


Fig.5 Measuring points near ITZ

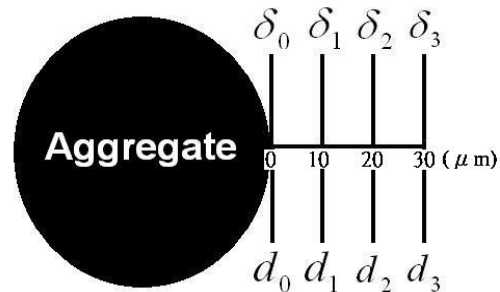


Fig.6 Schematic diagram of R observing position

3. Results and discussion

The displacement within ITZ caused by external temperature effects will change with respect to six designated temperatures, i.e. at 30°C, 50°C, 130°C, 230°C, 330°C and 420°C respectively. The displacement at each observing position around ITZ is measured by ESPI when the configured temperatures are reached. This kind of measurement is categorized as non-contact technique and can obtain the dynamic displacement of ITZ at various temperatures.

3.1 Displacement of ITZ

The displacements δ were measured at several discrete points from the edge of aggregate (d_0) to the anticipated edge of ITZ (d_{12}). The in-plane displacements (U-field) caused by temperature at T, B, L and R points (Fig.5), are summarized in Figs.7-10, respectively. As shown in Fig.7, the in-plane displacements on top of the aggregate (T point) decrease at 130°C and 330°C as the distance from the interface increases. In Fig.8, the behaviors of displacement over 230°C show no rational patterns due to dissipation of the bonding water within cement gel. The ITZ displacements at 30°C and 50°C increase with the temperature, and also slightly with the distance from the interface at top, left and right points of the aggregate, whereas decrease with the distance at bottom point. The caused ITZ displacements at L and R points (Figs.9-10) are very similar, especially at 130°C and 230°C the displacements reduce with the distance from the interface.

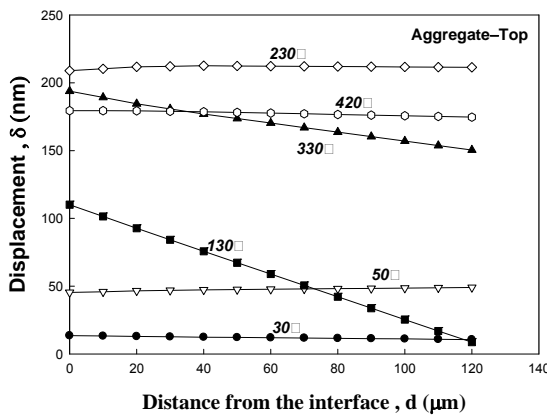


Fig.7 U-field displacement at T point

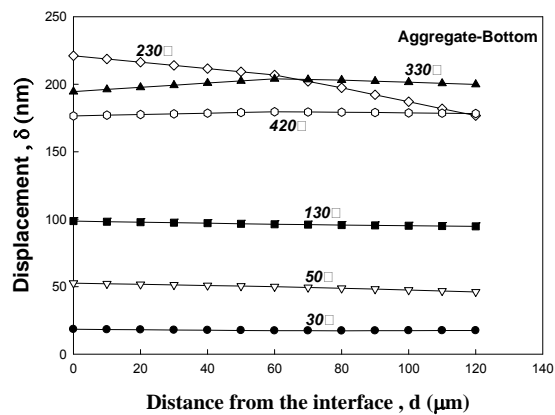


Fig.8 U-field displacement at B point

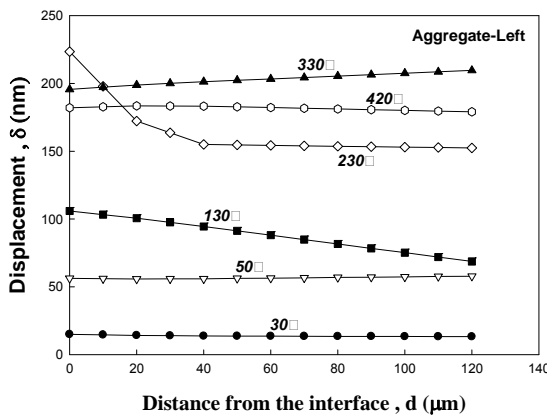


Fig.9 U-field displacement at L point

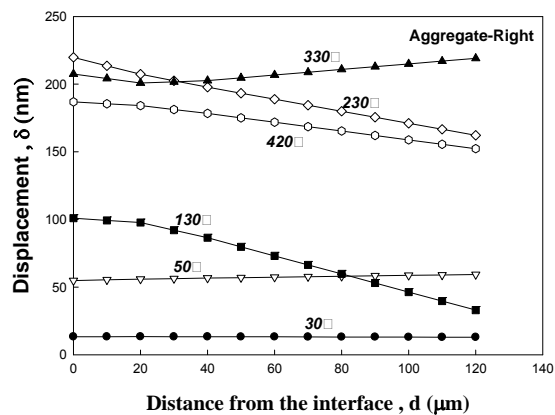


Fig.10 U-field displacement at R point

The W-field (out-plane) displacement are shown in Figs.11-14. It can be observed that the temperature effects on ITZ micro-displacement are less prominent when the temperature goes beyond 130°C. Generally, the W-field displacement increases with temperature till 330°C at T point (Fig.11) and 230°C at the others (Figs.12-14). These trends are then reversed as the temperatures continue to increase. It appears that the changes of W-field displacement are more stable than of U-field with

increasing temperatures. This may explain why the mortar (or binder) and the aggregate are easy to split each other since the thermal stress (the difference of the thermal strain) within ITZ in the radial direction is greater than that in the longitudinal direction. The results also suggest that the ITZ experiences certain inherent changes of material properties when temperature goes beyond 230°C.

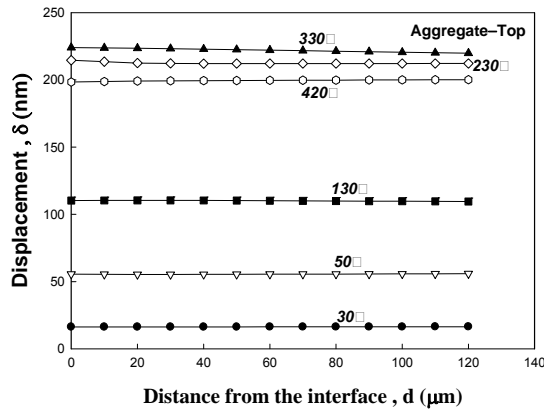


Fig. 11 W-field displacement at T point

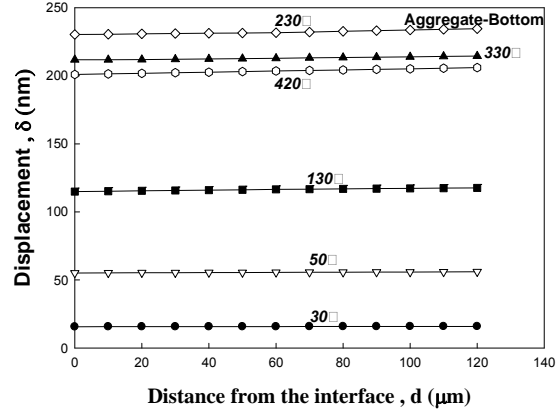


Fig. 12 W-field displacement at B point

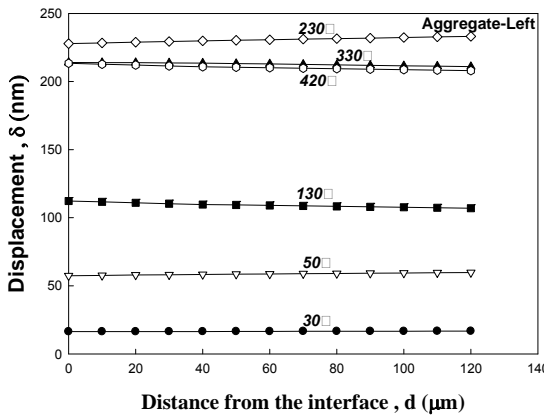


Fig. 13 W-field displacement at L point

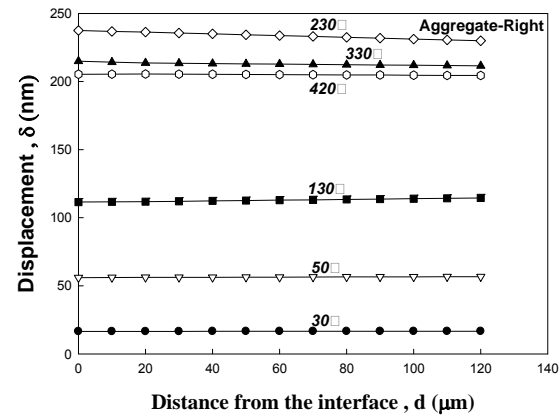


Fig. 14 W-field displacement at R point

3.2 Determination of ITZ

There are many measurement techniques such as SEM, OM, X-ray diffraction, micro-indentation and EDX to determine the range of ITZ. However, these methods can only observe an object with sizes that require sophisticated manufacturing processes. In addition, it is also challenging to maintain the object's original state of the interface when using these measurement techniques. As an alternative, we use ESPI technique here to determine the range of ITZ.

It is well recognized that the mechanical properties of ITZ are weaker than those of mortar (binder) and aggregates. Thereby, the displacement of ITZ is supposed to be sensitive to the applied temperatures that would alternate material's mechanical properties. From the results in Figs.7-14, we define a non-unit formula to calculate the difference of the displacement per unit distance:

$$\frac{\Delta\delta}{\Delta d} = \frac{\delta_{i+1} - \delta_i}{d_{i+1} - d_i} \quad (1)$$

The formula would give a constant value beyond ITZ and thus can be used as a criterion to determine the range of ITZ.

The ITZ determination results are depicted in Figs.15-18 (in-plane, U-field) and Figs.19-22 (out-plane, W-field), respectively. The range of ITZ is shown to be 40 μm for the top, left and right points of the aggregate (Fig.15, 17-18), and 80 μm for the bottom point (Fig.16). The calculation result is also supported by previous observations showing that the microstructure of ITZ at the bottom of aggregate is more porous comparing to other locations of the aggregate. We can also observe that the ITZ range of B point can be determined at room temperature. However, to more properly determine the ITZ of T, L and R points, the temperature should be increased to 130 $^{\circ}\text{C}$.

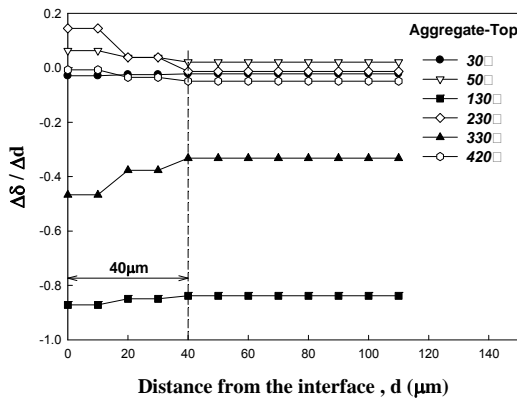


Fig.15 Range of ITZ from U-field at T point

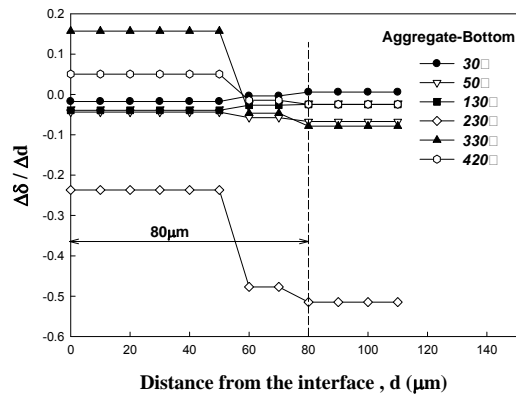


Fig.16 Range of ITZ from U-field at B point

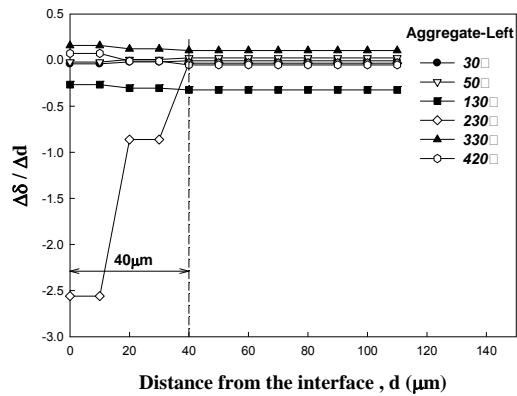


Fig.17 Range of ITZ from U-field at L point

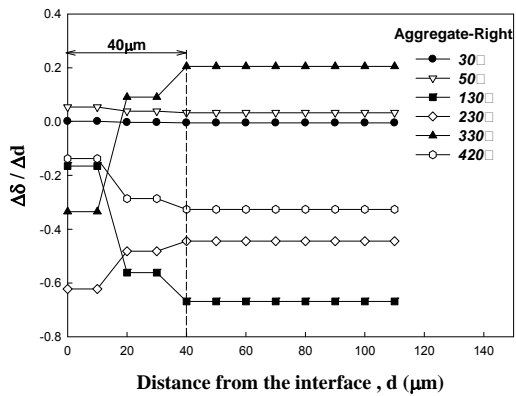


Fig.18 Range of ITZ from U-field at R point

Figs.19-22 show the results of out-plane ITZ determination using W-field displacement. Similar to U-field, the ITZ range obtained from W-field is also 40 μm at T, L and R points (Fig.19, 21-22). However, W-field ITZ at B point (Fig.20) appears to be 60 μm , which is less than U-field ITZ (80 μm). It can also be

observed that at 50°C, the W-field ITZ determination for all the four points is sufficiently sensitive.

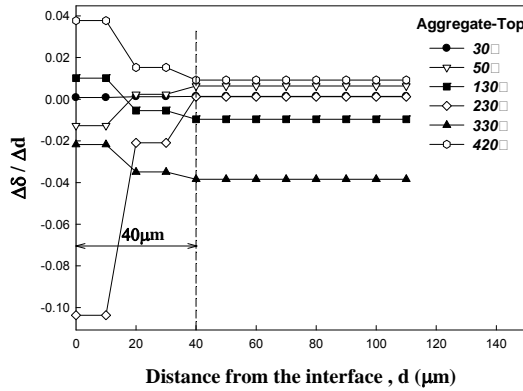


Fig.19 Range of ITZ from W-field at T point

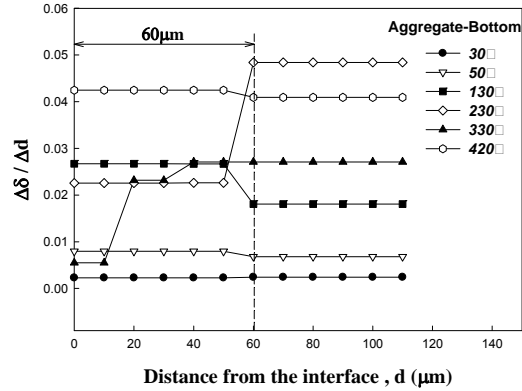


Fig.20 Range of ITZ from W-field at B point

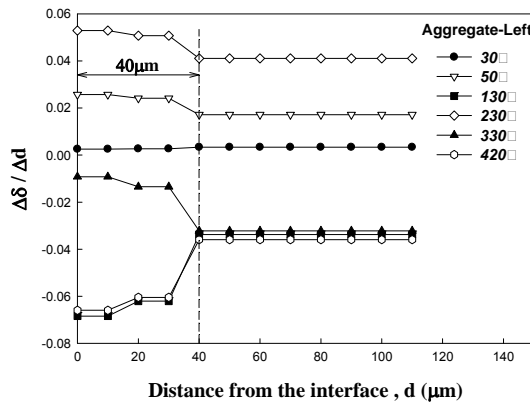


Fig.21 Range of ITZ from W-field at L point

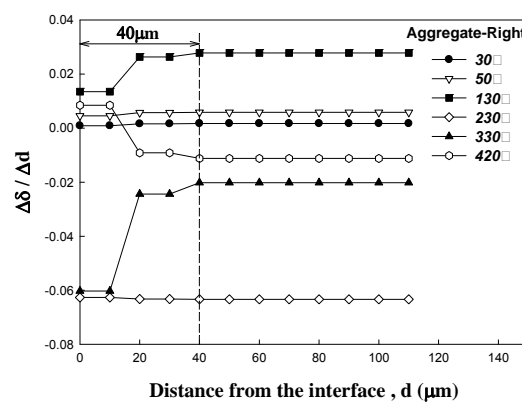


Fig.22 Range of ITZ from W-field at R point

4. Conclusions

The micro-displacements of ITZ are investigated under six degrees of temperature (from room temperature to 420°C) using ESPI measurement technique. We conclude this study as follows.

(1) The maximum displacement of ITZ occurs at temperature 230°C to 330°C and is depending on the position around the aggregate. The temperature effects on both U-field and W-field ITZ displacement are similar at bottom, left and right points, but slightly different for top point.

(2) From the displacement observed by ESPI, material properties (displacement) of ITZ experience an inverse change at 230°C.

(3) The W-field ITZ displacements at all observing points are more stable to temperature changes than U-field ITZ displacements.

(4) The determined ITZ range at bottom point of the aggregate is 80μm for U-field observation, and 60μm for W-field observation, respectively. The ITZ ranges at top, left and right points of the aggregate are determined as 40μm in this study.

Acknowledgements

This research was partially supported by the Taiwan National Science Council under Grant NSC 98-2221-E-151-054.

References:

- [1] Mindess, S., Tests to determine the mechanical properties of the interfacial zone, *Interfacial Transition Zone in Concrete*, Edited by J. C. Maso (1996) 47-48.
- [2] Diamond, S., Considerations in image analysis as applied to investigations of the ITZ in concrete, *Cement Concrete Composites* 23 (2001) 171-178.
- [3] Liaoa, K. Y., Chang, P. K., Peng, Y. N., Yang, C. C., A study on characteristics of interfacial transition zone in concrete, *Cement Concrete Research* 34 (2004) 977-989.
- [4] Zhang, J., Sun, H., Wan, J., Yi, Z., Study on microstructure and mechanical property of interfacial transition zone between limestone aggregate and Sialite paste, *Construction Building Materials* 23 (2009) 3393-3397.
- [5] Diamond, S., Huang, J., The ITZ in concrete—a different view based on image analysis and SEM observations, *Cement Concrete Composites* 23 (2001) 179-188.
- [6] Leemann, A., Loser, R., Münch, B., Influence of cement type on ITZ porosity and chloride resistance of self-compacting concrete, *Cement Concrete Composites* 32 (2010) 116-120.
- [7] Elsharief, A., Cohen, M. D., Olek, J., Influence of aggregate size, water cement ratio and age on the microstructure of the interfacial transition zone, *Cement Concrete Research* 33 (2003) 1837–1849.
- [8] Leemann, A., Münch, B., Gasser, P., Holzer, L., Influence of compaction on the interfacial transition zone and the permeability of concrete, *Cement Concrete Research* 36 (2006) 1425–1433.
- [9] Cwirzen, A., Penttala, V., Aggregate–cement paste transition zone properties affecting the salt–frost damage of high-performance concretes, *Cement Concrete Research* 35 (2005) 671–679.
- [10] Gao, J. M., Qian, C. X., Liu, H. F., Wang, B., Li, L., ITZ microstructure of concrete containing GGBS, *Cement Concrete Research* 35 (2005) 1299-1304.
- [11] Ollivier, J. P., Maso, J. C., Bourdette, B., Interfacial transition zone in concrete, *Advanced Cement Based Materials* 2 (1995) 30-38
- [12] Zhu, W., Bartos, P. J. M., Application of depth-sensing microindentation testing to study of interfacial transition zone in reinforced concrete, *Cement Concrete Research* 30 (2000) 1299-1304.
- [13] Mouret, M., Bascoul, A., Escadeillas, G., Microstructural features of concrete in relation to initial temperature—SEM and ESEM characterization, *Cement Concrete Research* 29 (1999) 369-375.
- [14] Fu, Y. F., Wong, Y. L., Poon, C. S., Chun-An Tang, C. A., Lin, P., Experimental study of micro/macro crack development and stress–strain relations of cement-based composite materials at elevated temperatures, *Cement Concrete Research* 34 (2004) 789-797.
- [15] Wang, X. S., Wu, B. S., Wang, Q. Y., Online SEM investigation of microcrack characteristics of concretes at various temperatures, *Cement Concrete Research* 35 (2005) 1385-1390.