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# Freezing and thawing resistance of air-entrained concrete incorporating recycled coarse aggregate: The role of air content in demolished concrete

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#### **Abstract**

This study aims to introduce new information on freezing and thawing resistance when air-entrained or non-air-entrained concrete is used as recycled coarse aggregate into air-entrained concrete. The laboratory produced air-entrained and non-air-entrained concretes with a water/cement (w/c) ratio of 0.45 were recycled at the crushing age of 1 year to obtain the coarse aggregates used in the investigations. The recycling process was performed in three stages to produce recycled coarse aggregates with different adhered mortar contents. The results showed that recycled coarse aggregate produced from non-air-entrained concrete caused poor freezing and thawing resistance in concrete even when the new system had a proper air entrainment. Microstructural studies indicated that non-air-entrained adhered mortar caused disintegration of the recycled coarse aggregate in itself and disrupted the surrounding new mortar after a limited number of freezing and thawing cycles. Minimizing non-air-entrained adhered mortar or enhancing the performance of new surrounding matrix could not give satisfactory results for a long freezing and thawing exposure.

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Keywords: Demolished concrete; Air content; Recycled coarse aggregate; Freezing and thawing; Microcracking

## 1. Introduction

Freezing and thawing resistance of recycled aggregate concrete is still questionable due to the great diversity in quality and composition of the demolished concretes. Dillmann [1] determined the effect of the strength level of the source concrete on the freezing and thawing resistance of concrete incorporating recycled coarse and fine aggregate in different replacement ratios. The authors reported that there was no noticeable difference in freezing and thawing durability of the recycled aggregate concretes with regard to strength level of the source concrete. Hosokawa et al. [2] investigated the influence of adhered mortar content of recycled coarse aggregate on the freezing and thawing performance of concrete. There was no clear effect of the process for reducing adhered mortar on freezing and thawing resistance. The results implied that if the quality of the

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source concrete is high enough, the adhered mortar may not be a factor influencing the freezing and thawing resistance negatively. Forster et al. [3] reported that freezing and thawing results differ depending on the original aggregate type. Recycled concrete containing freeze-thaw susceptible coarse aggregate performed better as aggregate in the new concrete than concrete containing that stone as coarse aggregate. Buck [4] found that freezing and thawing resistance of the concrete containing recycled concrete, where chert gravel was the original aggregate, increased in freezing and thawing tests. This is assumed to be the result of a reduction in frost susceptibility of the porous coarse aggregate particles. Kasai et al. [5] reported that freezing and thawing resistance became smaller as the replacement ratio of recycled coarse aggregate became larger. It is not recommended to use recycled coarse aggregate concrete where freezing and thawing action is severe. Mulheron and O'mahony [6] stated that the durability of lean concrete made using recycled aggregates, when subjected to freezing and thawing conditions, appears to be better than or similar to an equivalent control concrete made with natural gravel.

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Table 1 Properties of the original aggregates

Fraction	Origin	Code	Density (SSD) (g/cm <sup>3</sup> )	Water absorption (%)	Powder content (%)	Soundness loss (%)	Unit weight (kg/m³)	FM
Coarse	Sandstone	OCA	2.65	0.94	0.05	9.1	1700	6.48
Fine	River sand	NS	2.64	2.63	1.64	2.6	1570	2.87

Yamato et al. [7] investigated the freezing and thawing resistance of the concretes made with the recycled coarse aggregate produced in a recycling plant. The authors concluded that the resistance of recycled aggregate concrete was lower than that of the control concrete. For the replacement by the recycled aggregate of less than 30%, the reduction in the freezing and thawing resistance was small. Nishibayashi and Yamura [8] found that freezing and thawing durability of recycled aggregate concrete is very inferior and cannot be improved by air entrainment. Observations of Kawamura and Torii [9] on the disintegrated surface of the recycled aggregate concrete specimens subjected to freezing and thawing incorporating frost susceptible recycled coarse aggregate indicate that deterioration occurred along the interface between the mortar and original aggregate grains or within the adhered mortar. Their recommendation is reducing the adhered mortar content of the original aggregate grains as much as possible.

In the literature, a study reporting whether the demolished concrete is air-entrained or not can be rarely found. The important goal of this investigation is to compensate for this insufficient knowledge and provide a better understanding of resistance to freezing and thawing when air-entrained or non-air-entrained concrete is used as recycled coarse aggregate in air-entrained concrete. Furthermore, dealing with the characterization of the freezing- and thawinginduced damage patterns for a concrete incorporating recycled coarse aggregate is more difficult and needs special care, a versatile technique and experience due to the complex structure of the system. Compared with an ordinary aggregate, the recycled coarse aggregate introduces to the new concrete three different types of interfacial transition zones and doubles the matrix system [10]. There is almost no microstructural level investigation explaining the question of "which constituent(s) of the system leads freezing and thawing deterioration." The importance of the different processes involved in the crack formation in concrete subjected to freezing and thawing environment has not yet

been fully evaluated [11]. This study deals with the evaluation of the freezing-and thawing-induced internal damage mechanism by focusing on the microstructure of each material phase constituting the recycled coarse aggregate concrete. The classification of the crack formations in recycled coarse aggregate concrete makes it easy to diagnose the constituent(s) of the system responsible for freezing and thawing damage.

# 2. Experimental

#### 2.1. Source and properties of the recycled coarse aggregates

Laboratory-produced air-entrained and non-air-entrained concrete blocks were fabricated with a water/cement (w/c) ratio of 0.45 as the source of the recycled coarse aggregates. The virgin aggregates used in the concretes were sandstone coarse aggregate and natural sand from Sagami River. The properties of the original aggregates and mixture proportions of the source concretes are shown in Tables 1 and 2, respectively. Slump, total air content, and compressive strength of the concretes are presented in Table 3. The concrete blocks were demolded at the age of 28 days and stored at outdoor conditions until the crushing age of 1 year.

The recycled coarse aggregates were produced by crushing of the concrete blocks with a combination of jaw crusher and impact crusher. Also, higher quality recycled coarse aggregates having low adhered mortar content were produced by further processing of the crushed material twice with a mechanical grinding equipment. As a result, recycled coarse aggregates belonging to each source concrete were classified according to adhered mortar content. The detailed information on the recycling technology used for this investigation was presented in another report [12]. The properties of the recycled coarse aggregates are shown in Table 4. The adhered mortar content of the recycled coarse aggregate was determined by hydrochloric acid dissolution

Table 2 Mixture proportions of the source concretes

Туре	w/c (%)	s/a (%)	Mixture composition (kg/m³)						
			Water	OPC	Coarse	Sand	Admixture		(mm)
					aggregate		AEHRWR <sup>a</sup>	$AD^{b}$	
Air entrained	45	43	170	378	1004	749	3.40	_	20
Non-air-entrained	45	43	175	388	1031	769	3.10	1.94	20

<sup>&</sup>lt;sup>a</sup> Air-entraining high-range water reducer.

<sup>&</sup>lt;sup>b</sup> Air-detraining agent.

Table 3
Properties of the source concretes

Туре	Slump (mm)	Air (%)	Compressive strength at 28 days (N/mm <sup>2</sup> )
Air entrained	100	4.0	49.0
Non-air-entrained	20	1.2	55.8

method and corrected taking into account the dissolved original coarse aggregate [13].

#### 2.2. Fabrication of concrete specimens

#### 2.2.1. Materials

The natural aggregates of the present study were the same crushed sandstone coarse aggregate and river sand used in the production of the source concretes. An ordinary Portland cement was supplied for this investigation. The silica fume contains more than 90%  ${\rm SiO_2}$ . Metakaolin was a commercial product with an average particle size of 1  $\mu$ m and specific gravity of 2.5. An air-entraining admixture and a standard type air-entraining water reducer were included in the mixtures.

### 2.2.2. Design and production of the concrete mixtures

Including reference concrete made with natural coarse and fine aggregates, a total of 8 mixtures with a w/c ratio of 0.55 was produced. The first series of the recycled aggregate concretes incorporated the recycled coarse aggregates with and without air entrainment in different adhered mortar contents. The second series of the mixtures were produced with the recycled coarse aggregates in the blended form of air-entrained and non-air-entrained ones in different blending ratios [percentages of non-air-entrained recycled coarse aggregate (B' 1) in the total coarse aggregate population: 12.5, 25, and 50]. In addition to these concretes, a mixture incorporating non-air-entrained recycled coarse aggregate (B' 1) was designed with a water/binder (w/b) ratio of 0.30. This mixture was also modified by including silica fume or metakaolin with a replacement ratio of 10%.

The slump was kept at  $80\pm20$  mm and air content was adjusted to  $4.5\pm1\%$ . The specimens to be used in microstructural investigations were colored with a red pigment to be able to distinguish the new mortar from the adhered mortar. The mixture proportions and measured fresh properties are given in Table 5. The concrete specimens were cured in accordance with the standard conditions until the age of 28 days.

#### 2.3. Test program and procedure

# 2.3.1. Freezing and thawing tests

Testing of the freezing and thawing resistance was started at the age of 28 days and carried out in accordance with ASTM C 666 procedure-A on two  $100\times100\times400\text{-mm}$  prismatic specimens. The changes in weight and dynamic

modulus of elasticity of each specimen were measured for 500 cycles or when the relative dynamic modulus of elasticity had decreased below 60%.

# 2.3.2. Microscopic examination of freezing and thawing damage

In order to determine the role of the air void content of the source concrete on freezing and thawing resistance of air-entrained recycled aggregate concrete, a microscopic investigation was carried out on three concrete mixtures. Those were control concrete made with natural aggregates (OCA-55) and the ones incorporating air-entrained and nonair-entrained recycled coarse aggregates (B1-55 and B' 1-55) with a w/c ratio of 0.55. As soon as the freezing and thawing exposure was terminated, sampling was done by cutting slices from the middle portion of  $100 \times 100 \times 400$ mm concrete prisms. The observation area was arranged to be parallel to the side surface (perpendicular to the casting surface) with a depth of 5 mm. Two plane sections with the dimensions of  $20 \times 40 \times 15$  mm were prepared for each type of concrete in accordance with the procedure developed by Gran [14]. The concrete sections were examined using an optical microscope equipped with a fluorescent light source and an image analyzer. A Japanese image processing software application (Himawari) was used to collect the crack images. Before the analysis, a transparent grid was taped on each sample dividing the surface into 40 observation fields. The area of one observation field was 2.85 mm<sup>2</sup>. The images of the fields obtained by fluorescent microscopy were transmitted to an image processor to analyze the net area occupied by microcracks. The detailed information on fluorescent microscopy and procedure of image analysis had been given in the earlier reports [15-19].

In the microstructural level evaluation of freezing and thawing damage, the crack formations were classified under six constitutional zones. The constituents of a concrete incorporating recycled coarse aggregate are illustrated in Fig. 1. Two microstructural damage parameters were defined in the quantitative assessment of the cracking patterns: crack density ( $\Delta_{cr}$ ) and interface cracking ratio (ITZ<sub>cr</sub>). Crack density was taken into account in assessment of the damage within the constituents of new mortar (NM), ad-

Table 4
Properties of the recycled coarse aggregates

Code	Air	Adhered	$D^a$	$W^b$	$S^c$	$C^{d}$	FM
	entrainment	mortar	$(g/cm^3)$	(%)	(%)	(%)	
	in source	content					
	concrete	(%)					
B1	yes	55.0	2.41	5.58	48.3	5.19	6.57
B3		32.4	2.50	3.19	18.4	1.73	6.39
B' 1	no	55.7	2.44	5.57	47.0	3.65	6.67
B' 3		45.5	2.47	5.07	36.8	2.06	6.60

<sup>&</sup>lt;sup>a</sup> D: Density (ssd).

<sup>&</sup>lt;sup>b</sup> W: Water absorption.

<sup>&</sup>lt;sup>c</sup> S: Soundness loss.

<sup>&</sup>lt;sup>d</sup> C: 100 kN crushing value.

Table 5 Mixture proportions and fresh concrete properties

Mixture	w/b	Materials (kg/m <sup>3</sup> )									Air	Slump
	(%)	Water	Binder		Aggregate		Admixture			(%)	(mm)	
			OPC	SF	MK	Fine	Coarse	WRAE	AE	SP		
OCA-55	55	165	300	_	_	813	1039	0.94	0.7	_	4.3	90
B1-55	55	160	291	_	_	819	955	0.91	0.5	_	4.1	80
B3-55	55	157	285	_	-	825	998	0.89	0.5	_	3.8	65
B' 1-55	55	160	291	_	_	822	970	0.91	0.5	_	4.7	85
B' 3-55	55	158	284	_	_	817	996	0.89	0.6	_	4.1	80
B' 1 (% 12.5)-55	55	160	291			856	115	0.91	0.5	_	3.8	80
							816					
B' 1 (% 25)-55	55	160	291			856	230	0.91	0.5	_	4.0	90
, , ,							699					
B' 1 (% 50)-55	55	158	286			833	485	0.89	0.5	_	4.0	60
, , ,							484					
B' 1-30	30	156	520	_	_	658	955	_	1.0	3.1	4.0	80
B' 1 (SF)-30	30	156	468	52	_	653	948	_	0.9	3.7	3.9	100
B' 1 (MK)-30	30	156	468	_	52	652	946	_	0.9	4.2	4.0	80

hered mortar (AM), or original coarse aggregate (OCA), which can be expressed as

$$\Delta_{cr} = \frac{A_{\text{cr,const}}}{A_{\text{tot,const}}} \times 100 \quad (\%) \tag{1}$$

where  $A_{cr,const}$  is the total crack area within the constituent, and  $A_{\text{tot,const}}$  is the total area of the constituent within 80  $(2 \times 40)$  observation fields.

Eq. (1) cannot be applied to the identification of the cracks developed in the three different interfacial transition zones (NM-AM, OCA-AM, or NM-OCA) due to the uncertainty of the specific area occupied by the interface. Therefore, ITZ<sub>cr</sub> was employed as the second damage evaluation criteria. In equation form this is

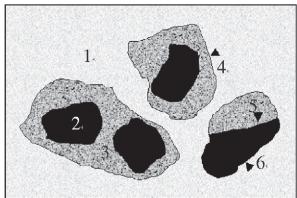
$$ITZ_{cr} = \frac{L_{cr,ITZ}}{L_{tot,ITZ}} \times 100 \quad (\%)$$
 (2)

where  $L_{cr,ITZ}$  is the total crack length through the interfacial transition zone, and  $L_{\text{tot,ITZ}}$  is the total length of ITZ within 80 observation fields.

#### 3. Results and discussion

# 3.1. Freezing and thawing resistance

Freezing and thawing durability of the concretes made with the recycled coarse aggregates produced from non-airentrained concrete was quite poor, although the required air entrainment was accommodated in the matrix of the new concrete. The relative modulus of elasticity decreased below 60% at 90 cycles. It seems that recycled coarse aggregate particles including adhered mortar with insufficient air void content convert the total pore system of the concrete to a partial non-air-entrained void system causing serious durability loss under frost attack. Further processing of the frost susceptible recycled coarse aggregate (B' 1) to reduce its adhered mortar content slightly improved the durability, but this limited contribution was not enough to attain the desired freezing and thawing resistance. On the other hand, the concretes made with the recycled coarse aggregates originated from air-entrained concretes were highly frost resis-



- 1- New mortar (NM)
- 2- Original coarse aggregate (OCA) 3- Adhered mortar (AM)
- 4- Interface between new mortar and adhered mortar (NM-AM)
- 5- Interface between original coarse aggregate and adhered mortar (OCA-AM)
- 6- Interface between new mortar and original coarse aggregate (NM-OCA)

Fig. 1. Structural constituents of a concrete incorporating recycled coarse aggregate.

tant regardless of the adhered mortar content. Their overall performance at the end of 500 freezing and thawing cycles was even superior to that of the control mixture incorporating sandstone coarse aggregate, although the recycled aggregate concretes had noticeably higher values for volume of permeable pores and water absorption [10]. The explanation of this paradoxical result is given in the next section focusing on the microstructural aspect of the phenomena. Fig. 2 illustrates the variation of the relative dynamic modulus of elasticity of the control and the concretes incorporating comparable quality non-air-entrained and air-entrained recycled coarse aggregates during freezing and thawing exposure.

In real cases, it is often difficult to say that the origin of the recycled aggregate is purely air-entrained or non-airentrained due to the storage of the demolished concretes in the mixed form in the recycling site regardless of their mixture characteristics. This situation normally creates a product that is a blend of air-entrained and non-airentrained type of recycled aggregates. Regarding this point, some special mixtures were designed by blending the recycled coarse aggregates originated from the source concretes with and without air entrainment. The blending percentages (B' 1+B1) were decided as 50+50, 25+75, and 12.5 + 87.5 by weight. Fig. 3 compares the variation in relative dynamic modulus of elasticity of the concretes incorporating pure type or blended recycled coarse aggregates during freezing and thawing exposure. It is obvious that very limited amount of non-air-entrained type recycled coarse aggregate in the total aggregate population (12.5%) was enough to cause a sharp decrease in freezing and thawing resistance of the concrete. It can be concluded that non-air-entrained type recycled coarse aggregate should be completely excluded from the mixture if the concrete will be exposed to freezing and thawing environment.

As another important result, long-term freezing and thawing resistance could not be achieved by enhancing the matrix quality of new air-entrained concrete (w/

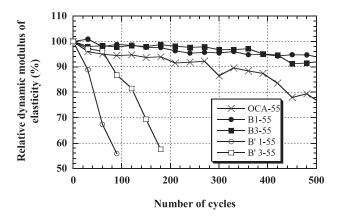


Fig. 2. Comparison of the variation in relative dynamic modulus of elasticity for the mixtures made with virgin, air-entrained, and non-air-entrained recycled coarse aggregates.

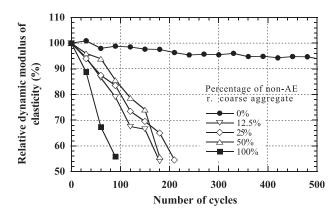


Fig. 3. Relative dynamic modulus of elasticity versus number of frost cycles for the concretes incorporating pure type or blended recycled coarse aggregates.

b = 0.30) when it was made with recycled coarse aggregate produced from non-air-entrained concrete (B' 1). As Fig. 4 shows, only the mixture containing metakaolin performed relatively well and satisfied the durability limits resisting over 300 cycles. Inclusion of silica fume in concrete decreased freezing and thawing resistance compared with the performance of the concrete containing only cement as binder. There was no noticeable scaling for the concretes with a w/b of 0.30. On the contrary, a weight increase was observed during the measurements showing the typical signs of the internal microcracking. As soon as microcracking takes place, the deteriorated zones fill with the surrounding water causing an increase in the weight of the specimen. Fig. 5 illustrates the change in weight of the concrete specimens having enhanced matrix properties during freezing and thawing exposure.

# 3.2. Microstructural evaluation of freezing and thawing damage

It should be pointed out that the possibility of preexposure cracks in the recycled coarse aggregate makes it

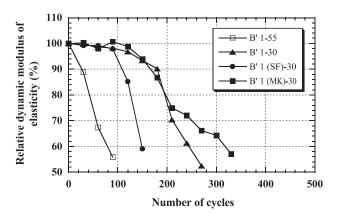


Fig. 4. Relative dynamic modulus of elasticity versus number of frost cycles for the concretes incorporating non-air-entrained recycled coarse aggregate (B' 1).

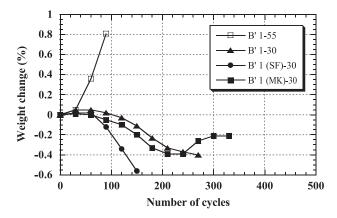


Fig. 5. Weight change of the concrete specimens incorporating non-air-entrained recycled coarse aggregate (B' 1) during frost exposure.

necessary to start the microstructural investigations prior to freezing and thawing action. Otherwise, it would be impossible to distinguish the unbound aggregate cracks from the others propagated by the frost attack. The first step of any microstructural study on recycled aggregate concrete subjected to freezing and thawing has to examine the integrity of the unbound aggregate. The detailed discussion of the recycling process induced damage sensitivity of the recycled aggregates used in this investigation was done in an earlier study [10]. It was demonstrated that double-crushing noticeably reduced the original coarse aggregate defects detected before recycling. This result implied that during crushing of the source concretes, fracture paths followed the weakest zones such as cracked or porous sandstone coarse aggregate particles. On the other hand, recycling of the source concretes did not introduce detectable adhered mortar or bond crack in recycled coarse aggregate. In conclusion, all the recycled coarse aggregates had no integrity loss before use in concrete to be subjected to freezing and thawing action.

Microscopic examination of the concrete made with non-air-entrained recycled coarse aggregate (B' 1-55) clarified that the main constituent in the system propagating microcrack development was the adhered mortar, which has no

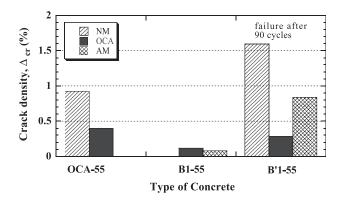


Fig. 6. Crack density of new mortar, original coarse aggregate, and adhered mortar in the control and recycled coarse aggregate concretes.

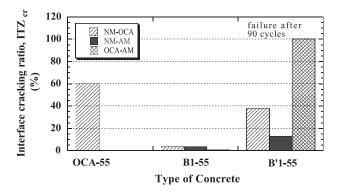


Fig. 7. Cracking ratio for interfacial transition zones in the control and recycled coarse aggregate concretes.

appropriate air void system to resist freezing and thawing exposure. The relatively higher crack density in this type of adhered mortar than in that of the comparable air-entrained one shows the severity of the damage in itself (Fig. 6). The results also indicate that following the deterioration of the adhered mortar, a full disintegration occurs between the original coarse aggregate particle and adhered mortar. This disintegration results in 100% cracking through the interface (Fig. 7). The photomicrograph in Fig. 8 demonstrates the severity of the microstructural damage within non-airentrained recycled coarse aggregate after only 90 freezing and thawing cycles. The bond between adhered mortar and original coarse aggregate is absolutely lost. Once the recycled coarse aggregate particle loses its integrity, it negatively affects the surrounding new mortar. The deteriorated particles constituting the local defects joined extending cracks with one other throughout the new mortar. As Fig. 6 shows, an extensive crack network developed in the new mortar phase of the concrete incorporating non-airentrained recycled coarse aggregate. After this final stage of the deterioration mechanism, an absolute failure occurred in the system.

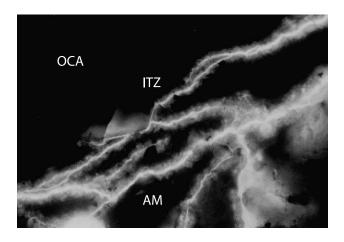


Fig. 8. View of disintegrated non-air-entrained recycled coarse aggregate through the bond (OCA-AM) due to the severe microcracking in adhered mortar. Height of the field is 3.24 mm.

On the other hand, the concrete made with recycled coarse aggregate originated from air-entrained concrete (B1-55) showed no signs of frost damage in microcracking analysis. The extent of cracking in the control concrete incorporating natural aggregates was more severe (Fig. 6). Also, serious interface cracking was another typical damage pattern in the natural coarse aggregate concrete as shown in Fig. 7. Sixty percent of the interface path was totally cracked. The lower cracking resistance of control concrete compared with that of the recycled aggregate concrete made with air-entrained recycled coarse aggregate can be explained with a couple of reasons. Although both recycled aggregate concretes and the reference concrete contained the sandstone aggregate from the same source, the recycled aggregate concretes showed important differences in connection with the modification of the original coarse aggregate characteristics in the recycling process: The most important difference is the absence of weak and porous sandstone particles in recycled aggregate concretes due to the elimination of these defects in a great extent during the recycling process. The second difference is the considerably lower concentration of the original coarse aggregate in the system of the recycled aggregate concrete because of the presence of adhered mortar. Finally, crushing of the source concretes led an important reduction in the size of the original coarse aggregate. Almost all the sandstone particles in recycled aggregate concretes were under the critical size at which they are not capable of provoking freezing- and thawing-induced deterioration.

### 4. Conclusions

- The investigations demonstrated that non-air-entrained concrete is a serious handicap to achieve a good freezing and thawing resistance when it is used as recycled coarse aggregate in air-entrained concrete. Improper air void system of each independent aggregate particle converts the total void system to a partial non-air-entrained system causing a poor freezing and thawing resistance. However, if an air-entrained concrete incorporates also air-entrained recycled coarse aggregate, this concrete has an entirely air-entrained void system showing an excellent performance under freezing and thawing exposure.
- 2. Although the beneficial effect of the further processing to reduce adhered mortar content was observed in the concrete made with non-air-entrained recycled coarse aggregate, this limited contribution was not enough to attain desired freezing and thawing resistance. On the other hand, reducing the adhered mortar content of a sound recycled concrete aggregate did not create a clear contribution to freezing and thawing resistance of the concrete.

- 3. The presence of a small amount of non-air-entrained recycled coarse aggregate in the aggregate population would be enough to drastically reduce freezing and thawing resistance of the concrete. To ensure high freezing and thawing durability, only pure air-entrained recycled coarse aggregate with enough quality should be used in the mixture.
- 4. Even though the matrix performance of the concrete incorporating non-air-entrained recycled coarse aggregate was considerably improved with a low w/b ratio of 0.30, the freezing and thawing resistance for a long-term exposure could not be achieved. Only the concrete containing metakaolin performed relatively well and satisfied the durability limits resisting over 300 cycles.
- 5. Microscopic level investigation of the damage mechanism for the concretes incorporating non-air-entrained recycled coarse aggregate showed that deteriorated adhered mortar with heavy cracks first caused disintegration of the recycled coarse aggregate itself, then the damaged particles behaved as local defects to distress the new mortar. After development of the crack network, the concrete failed with heavy damage.
- 6. If adhered mortar was frost resistant (air-entrained), recycled aggregate concrete did not show a serious sign of cracking. The severity of cracking was even lower than that of natural aggregate concrete. This was due to the preexposure defect potential of the sandstone coarse aggregate. The recycling process reduced the size of the original coarse aggregate particles under a critical value. The porous particles were mostly eliminated during crushing as well. Thus, sandstone coarse aggregate particles reduced in size and amount could not propagate microcracking within sound recycled coarse aggregate and into the other constituents of the recycled aggregate concrete subjected to freezing and thawing action.

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