

FREEZE-THAW DURABILITY OF CONCRETES WITH AND WITHOUT CLASS C FLY ASH

**Final Report
For
MLR-94-9 - Phase I**

January 1997

Project Development Division



**Iowa Department
of Transportation**

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WITH AND WITHOUT CLASS C FLY ASH

Final Report
for
MLR-94-9 - Phase 1

By
Chengsheng Ouyang
Transportation Engineer Associate
515-239-1088

and

John Lane
Testing Engineer
515-239-1237

FAX: 515-239-1092
Office of Materials
Project Development Division
Iowa Department of Transportation
Ames, Iowa 50010

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| Chengsheng Ouyang Transportation Engineer Assoc. John Lane Testing Engineer | Iowa Department of Transportation Materials Department 800 Lincoln Way Ames, Iowa 50010 |
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| <p>The freeze-thaw resistance of concretes was studied. Nine concrete mixes, made with five cements and cement-Class C fly ash combinations, were exposed to freeze-thaw cycling following 110 to 222 days of moist curing. Prior to the freeze-thaw cycling, the specimens were examined by a low-vacuum scanning electron microscope (SEM) for their microstructure. The influence of a wet/dry treatment was also studied.</p> <p>Infilling of ettringite in entrained air voids was observed in the concretes tested. The extent of the infilling depends on the period of moist curing as well as the wet/dry treatment. The concretes with 15% Class C fly ash replacement show more infilling in their air voids. It was found that the influence of the infilling on the freeze-thaw durability relates to the air spacing factor. The greater the spacing factor, the more expansion under the freeze-thaw cycling. The infilling seems to decrease effective air content and to increase effective spacing factor. The infilling also implies that the filled air voids are water-accessible. These might lead to concrete more vulnerable to the freeze-thaw attack.</p> <p>By combining the above results with field observations, one may conclude that the freeze-thaw damage is a factor related to premature deterioration of PCC pavements in Iowa.</p> | |
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DISCLAIMER

The contents of this report reflect the views of the author and do not necessarily reflect the official views of the Iowa Department of Transportation. This report does not constitute any standard, specification or regulation.

1. INTRODUCTION

Premature deterioration of some portland cement concrete (PCC) pavements in Iowa, other than D-cracking, has been reported for several years. Many efforts have been attempted to identify causes leading to the premature deterioration. These proposed causes include alkali-silica reaction^[1] and internal sulfate attack^[2].

Alkali-silica reaction (ASR) results from a chemical reaction between alkali contained in cement paste and certain reactive forms of silica within aggregates. This reaction causes expansion which may lead to cracking of concrete. It was reported that some Iowa sands contain up to 2.5% of shale particles which might have potential to cause ASR^[1]. However, these sands, with up to 2.5% shale particles, have good service records in many Iowa highways.

Calcium sulfoaluminate (or ettringite) is the reaction product of calcium, aluminum and sulfur components^[3]. The formation of ettringite is accompanied by increase in volume of materials. Since tricalcium aluminate (C_3A) is a very active major component in cement, it reacts with sulfate ions (SO_3 , also in cement) to form ettringite within hours after mixing with water. Because the concrete is plastic in this stage, the volume increase due to the ettringite formation might not result in cracking. However, the formation of excessive ettringite in hardened concrete may result in cracking. This is referred to as sulfate attack, and may happen when a mature concrete is exposed to a sulfate-rich environment, or when the formation of ettringite is excessive in concrete.

Several mechanisms have been proposed to explain freeze-thaw damage of concrete^[4]. It generally agrees that the freeze-thaw damage occurs when cement paste freezes and there is no free space within the concrete to relieve internal osmotic and hydraulic pressure. Entrained air voids are designed to relieve the pressure and to assure the freeze-thaw resistance of concrete.

Petrographic observations of cores from several concrete pavements in Iowa, using a low-vacuum scanning electron microscope, have found that many entrained air voids in matured and aged concrete are filled, to some extent, by sulfate-rich material which may be ettringite^{[2][5]} and will be referred to as ettringite later in this report. More infilling of ettringite was reported for the concrete mixes which have 15% the portland cement replaced by Class C fly ash. Currently, there are some arguments on whether or not the redeposit of ettringite in air voids of matured concrete results in sulfate attack^{[6][7]}. It is unclear if this infilling of ettringite reduces effective entrained air voids^[6]. The infilling of ettringite in entrained air voids might lead to the aged concrete becoming more vulnerable to the freeze-thaw attack. If this is true, the freeze-thaw resistance of concrete might decrease when concrete becomes more mature and when a certain amount of cement is replaced by Class C fly ash. This is because the infilling of entrained air voids is more significant for concrete with older age and with the Class C fly ash replacement. Certain portland cements alone may also have the potential to produce void infilling material.

Even though some contradictory results have been reported, most studies indicate that the use of suitable amount of Class C fly ash in concrete can improve its resistance to sulfate attack and alkali-silica reaction^[8]. Several investigators^{[9][10]} reported that concrete which used fly ash had satisfactory resistance to the freeze-thaw attack. However, in most of these laboratory studies, the concrete specimens were exposed to the freeze-thaw cycling at either 14 days or 28 days. Nasser and Lai^[11] tested the freeze-thaw resistance of concretes after 28 days and 80 days curing, respectively. A Class F fly ash with available alkalies (Na_2O equivalent) of 1.77% was used. They reported that concretes with 20%, 35% and 50% of fly ash had lower relative dynamic modulus and greater loss in mass compared to the control mix without fly ash. By considering the potential effect of the

infilling of air voids by ettringite previously discussed, more research is certainly needed to further assess the freeze-thaw resistance of concrete after a long period of moist curing.

This research is to study the freeze-thaw resistance of concretes with and without replacement of cement by Class C fly ash after up to 222 days of moist curing. Nine concrete mixes, made with five cements and cement-fly ash combinations, were exposed to the freeze-thaw cycling following 110 to 222 days of moist curing. The length change of the specimens was continuously monitored, from 24 hours after casting to the end of the freeze-thaw test. Prior to the freeze-thaw cycling, the specimens were examined by a low-vacuum scanning electron microscope (SEM) for their microstructure. The influence of a wet/dry treatment similar to that used for the Duggan Test^[12] was also studied.

Although many studies have been previously conducted to evaluate the freeze-thaw durability of concrete containing fly ash, the features of this study include: (i) testing the freeze-thaw resistance of concretes after relatively long periods of moist curing and wet/dry cycles. (ii) relating microstructure of concretes, which are based on their SEM photomicrographs taken immediately prior to the freeze-thaw cycling, to their freeze-thaw resistance.

2. MATERIALS, SPECIMENS AND TEST PROCEDURE

2.1 Materials and Specimens

The chemical properties of five cements that were used are listed in Table 1 were used. Chemical properties of the Class C fly ash used are given in Table 2. A total of nine mixes given in Table 3 were produced using these cements and the fly ash. The plastic entrained air contents

measured during casting are shown for each mix in Table 3. Iowa Department of Transportation Standard Mixes C-3 and C-3-C were used as indicated in Table 3.

2.2 Test Procedure

The goal of this research is to study the freeze-thaw resistance of concrete mixes after different periods of moist curing. The test procedure used is described in Fig. 1. Four 4" x 4" x 18" beams were cast for each mix, and stored in a standard 100% humidity and 73°F moist room. Sixteen 1 3/4" x 3 1/2" cores were drilled from two of these beams at the age of 59 days. The remaining two beams were stored in laboratory tap water and used later. At the age of 60 days, a core from each mix was examined by a SEM to observe microstructure of the concrete.

At the age of 102 days, the remaining fifteen cores were subjected to the wet/dry treatment, which consists of three consecutive wet (70°F in distilled water)/dry (180 F in oven) cycles, then cured in distilled water. This wet/dry treatment is similar to that used for the Duggan Test^[12]. Three of these cores were subjected to the freeze-thaw cycling at the age of 110 days. The remaining twelve cores were cured in distilled water.

At the age of 162 days, three cores from each mix were subjected to the freeze-thaw cycling, and one core was examined by SEM prior to the cycling. Among the remaining eight cores, four of them were continuously cured in distilled water, whereas the other four were switched to curing in 5% NaCl salt water. All these cores were cured until the age of 222 days. Then, for each curing condition, after one core was examined by SEM, the other three cores were subjected to

freeze-thaw cycling. Samples taken from mixes 3 and 6 at the age of 222 days, which have been subjected to the wet/dry treatment, were sent to the research laboratory of Lafarge Canada, Inc. for examining their microstructure.

Also at the age of 162 days, for each mix the two beams, which weren't subjected to the wet/dry treatment, were taken out of the laboratory tap water. After a dike was placed on the top surface of each beam, one beam was exposed to distilled water at the top surface, whereas the other was exposed to 5% salt water. At the age of 222 days, three cores were drilled from each of the beams. Since only the top surface of each beam were exposed to water during the period from 162 days and 222 days of age, these cores were expected to have lower degree of saturation compared to those subjected to the wet/dry treatment. The latter are under continuously moist curing after the treatment. While one core was examined by SEM, the other two cores were subjected to the freeze-thaw cycling. Samples was cut at the age of 222 days from each of these beams, which were not subjected to the wet/dry treatment. These samples were sent to Research Laboratories of Lafarge Canada, Inc. and Ash Grove Cement Company, respectively, for measuring entrained air parameters by linear traverse method (ASTM C457). The obtained results are given in Table 4. Since only empty and partially filled (by ettringite) air voids were counted in the measurement by the two laboratories, the results obtained in such a way are referred to as effective air parameters in this report, whose values may change in a hardened concrete as infilling progress. It is noted that these values of the effective air parameters listed in Table 4 are for the specimens which were exposed to extended moist curing but were not subjected to the wet/dry treatment. The samples from Mixes 4 and 6, which were subjected to both extended moist curing and the wet/dry treatment, were also sent to the Research Laboratory of Lafarge Canada, Inc. for testing. They reported that up to 80% of air

voids were filled by ettringite for these samples. The effect of ettringite infilling is obviously underestimated by the values of the air parameters given in Table 4 for the samples subjected to both extended moist curing and the wet/dry treatment. However, these values may provide a quantitative means to evaluate this effect.

The freeze-thaw cycling consists of two hours of freezing in air (0°F) and one and half hours of thawing in water (40°F). This is similar to ASTM C 666B. The cycling continues until the failure of the sample or after reaching 800 cycles.

3. EXPERIMENTAL OBSERVATIONS

3.1 History of Specimen Length Change

Length changes of all specimens (the beams and cores) were monitored during the entire test period. The length change of Mix 4, from 24 hours after casting to the end of the freeze-thaw cycling, is shown in Fig. 2. In this figure, the solid line represents the length change of the specimens without the wet/dry treatment, whereas the dashed line indicates the length change of the specimens with the treatment. Although the wet/dry treatment was performed at the age of 102 days, the length change was negligible until the freeze-thaw cycling started at the age of 222 days. The SEM photomicrographs for the specimens with the wet/dry treatment at ages of 60 days (point A), 162 days (point B) and 222 days (point C) are presented in Figs. 3, 4 and 5, respectively. As seen in Fig. 3a, at the age of 60 days (before the wet/dry treatment), the entrained air voids are quite clean. The elemental scan for chemical components of the same concrete sample is shown in Fig. 3b. The distribution of sulfate ions is basically uniform within the cementitious matrix. On the other hand, at the ages of 162 and 222 days, the SEM photomicrographs (Figs. 4a and 5a) indicate that the

entrained air voids are partially filled by some materials after the wet/dry treatment. The elemental scans shown in Figs. 4b and 5b indicate this infilling material is sulfate-rich, and accompanied by some alumina and calcium. This implies that the infilling material may be calcium sulfoaluminate compound. For easier discussion, this sulfate-rich infilling material will be regarded as ettringite hereafter. Although it is found that ettringite is more concentrated in the air voids, the expansion of the specimens with the wet/dry treatment is negligible until the beginning of the freeze-thaw cycling as shown in Fig. 2. Although some cracks are observed in Figs. 3a, 4a and 5a, most of these cracks pass through the interface between aggregates and the cementitious matrix. This suggests that they may primarily result from drying shrinkage and from the preparation of the sample, instead of expansion which usually generates three radiating cracks with approximately 120° apart^[13]. No evidence of sulfate attack was noticed up to the age of 222 days. Similar results were observed for other mixes.

3.2 Freeze-Thaw Response of Specimens With the Wet/Dry Treatment

The freeze-thaw response of specimens subjected to the wet/dry treatment is discussed in this section. The $1\frac{3}{4}'' \times 3\frac{1}{2}''$ cores were drilled from the $4'' \times 4'' \times 18''$ beams which were stored in 100% humidity moist room until an age of 59 days. The obtained cores were cured in laboratory tap water until the age of 102 days. All these cores were then subjected to the wet/dry treatment. After the treatment, these cores were stored in distilled water, and exposed to the freeze-thaw cycling at the age of 110, 162 and 222 days, respectively.

The freeze-thaw expansion of Mixes 1 and 4 is shown in Fig. 6. As indicated in Table 3, both mixes used Cement A. No fly ash was used in Mix 1, whereas 15% of the cement (by

weight) was replaced by the fly ash in Mix 4. Mix 1 has the air content of 5.2% and the spacing factor of 0.297 mm (see Table 4), whereas Mix 4 has the air content of 3.2% and the spacing factor of 0.318 mm. Unless specified, the air parameter measured by the Research Laboratory of Lafarge will be used hereafter in this report. As shown in Fig. 6, the specimens from the same mix subjected to the freeze-thaw cycling from the age of 222 days showed much greater expansion compared to those subjected to the cycling from the age of 110 days. On the other hand, at the same curing age Mix 4 has much greater freeze-thaw expansion than Mix 1. More discussion on the possible mechanisms will be included later.

Similar results were observed for Mixes 3 and 6, which are shown in Fig. I-1 (see Appendix I).

The freeze-thaw expansion of Mixes 2 and 5 is shown in Fig. 7. These two mixes used Cement B. For Mix 2 without the fly ash, the specimens subjected to different ages of moist curing prior to the freeze-thaw cycling have almost identical values of expansion. For Mix 5, the specimens exposed to 162-day moist curing have slightly lower expansion than those subjected to 110-day and 222-day moist curing, respectively. On the other hand, Mix 5 with the fly ash replacement generally shows slightly higher expansion compared to Mix 2 without the fly ash replacement. Similar observations were obtained for Mixes 8 and 9 as given in Fig. I-3 (see Appendix I).

The results presented in this section indicate that the freeze-thaw response of mixes using ordinary portland cements (Types I and II) may be divided into two groups. Group 1 includes Mixes 1, 3, 4 and 6. These mixes show greater expansion for longer period of the moist curing prior to the freeze-thaw cycling and when the replacement of 15% cement by the fly ash. Group 2 includes Mixes 2, 5, 8 and 9. When the period of moist curing was changed and when 15% cement was

replaced by the fly ash, no significant difference in the expansion was observed for this group. Potential mechanisms corresponding to these two groups will be discussed later.

The expansion of Mix 7, which used Cement D (Type IP), is presented in Fig. I-1 (see Appendix I). It is seen again that the longer period of moist curing prior to the start of the cycling, the greater the expansion. Although the spacing factor is 0.16 mm, 3.6% of the air content (measured by the Research Laboratory of Ash Grove Cement Company) may be somewhat responsible for relatively large expansion for this mix.

3.3 Freeze-Thaw Response of Specimens Without the Wet/Dry Treatment

The freeze-thaw response of specimens which were not subjected to the wet/dry treatment is discussed in this section. Two 4" x 4" x 18" beams for each mix were continuously stored in 100% humidity moist room. The beams were taken from the moist room at the age of 162 days, and a dike was placed on the top surface of each beam. Then, for each mix one beam was exposed to continuous distilled water on the top surface, whereas the other was exposed to a continuous 5% salt water. Cores were taken from these beams, and subjected to the freeze-thaw cycling at the age of 222 days. As afore-mentioned, these cores probably have lower degree of saturation than those subjected to the wet/dry treatment. This may partially explain why the former has lower freeze-thaw expansion compared to the latter.

The expansion of Mixes 1 and 4 without wet/dry treatment is presented in Fig. 8. Mix 4 with the fly ash replacement shows much greater expansion after approximately 500 cycles. The specimens exposed to the distilled water have slightly more expansion than those exposed to the 5% salt water. No significant difference was observed on the expansion values at 300 cycles for these specimens.

The results for Mixes 2 and 5 are shown in Fig. 9. These two mixes have much lower expansion than Mixes 1 and 4. The test results of the other mixes are presented in Appendix II. For these mixes, no greater difference in the expansion was observed, when 15% cement was replaced by the fly ash.

4. DISCUSSION

4.1 Effect of Ettringite Infilling

To further demonstrate the influence of Class C fly ash replacement and the wet/dry treatment on the freeze-thaw resistance of concrete, the results of Mixes 3 and 6 are presented in Fig. 10. The age of the specimens prior to the freeze-thaw cycling is 222 days. It is seen from Table 4, the two mixes have the same value of the spacing factor, where Mixes 3 and 6 have the air contents of 5.8% and 4.0%, respectively. The solid lines represent the results of Mix 3, whereas the dashed lines are for Mix 6 with 15% replacement of the fly ash. Note that Mix 6 shows greater expansion than Mix 3. This fact further confirms that the 15% replacement of cement by some Class C fly ash may reduce the freeze-thaw resistance of concrete after long period of moist curing. On the other hand, the specimens subjected to the wet/dry treatment show much greater freeze-thaw expansion than those without the treatment. This difference due to the wet/dry treatment is partially accounted for by the fact that the former has higher degree of saturation than the latter.

The SEM photomicrographs taken immediately prior to the start of the freeze-thaw may provide some hints to better understand the freeze-thaw response observed in Fig. 10. These photomicrographs are presented in Figs. 11, 12, 13, and 14 for Mix 3 without the wet/dry treatment, Mix 3 with the treatment, Mix 6 without the treatment and Mix 6 with the treatment, respectively.

Mix 3 without the treatment has the lowest expansion. Its entrained air system is quite clean (see Fig. 11a) and the distribution of sulfate is basically uniform (see Fig. 11b). Since sulfate is a major component of ettringite, the distribution of the sulfate component is thought to be related to the distribution of ettringite. Mix 3 with the treatment has the second lowest expansion. Minor infilling is found in its air voids (see Fig. 12a), and some concentration of sulfate component in the air voids is also observed (see Fig. 12b). Mix 6 without the treatment has the second highest expansion. The corresponding SEM photomicrograph (see Fig. 13a) shows that its air void system is partially filled. However, the elemental scan (see Fig. 13b) indicates that the infilling material is not sulfate-rich. Mix 6 with the treatment has the highest expansion. The SEM photomicrograph (Fig. 14a) indicates that most of its air voids have been filled by the sulfate-rich material (see Fig. 14b).

The above results seem to indicate that the freeze-thaw response of concrete is somewhat related to the extent of infilling of entrained air voids. For the same period of the moist curing, the extent of infilling depends on whether or not some amount of cement is replaced by Class C fly ash, as well as on whether or not the wet/dry treatment is applied. The wet/dry treatment accelerates the infilling of ettringite in entrained air voids. That might result in greater freeze-thaw expansion for some concretes. Possible mechanisms to explain the above observed freeze-thaw response are discussed in the following.

4.2 Possible Mechanisms for Change of Freeze-Thaw Response

As afore-mentioned, the freeze-thaw response of mixes using ordinary portland cements (Types I and II) may be divided into Groups 1 and 2. Group 1 includes Mixes 1, 3, 4 and 6, which are underlined in Table 4. These mixes show greater expansion for longer period of the

moist curing prior to the start of the freeze-thaw cycling and when the replacement of 15% cement by the fly ash. Group 2 includes Mixes 2, 5, 8 and 9. For Group 2, no significant difference in the expansion was observed when the period of the moist curing was changed.

Two possible mechanisms are proposed here to explain the above observation. First, as indicated by Skalny^[6] and Mather^[14], ettringite normally re-deposits in entrained air voids through moisture migration. The infilling of ettringite in some air voids implies that these voids are somewhat water-accessible. Since only dry air voids can fully function to protect concrete from freeze-thaw attack, effectiveness of these water-accessible air voids might be already compromised before filled by ettringite.

Secondly, the infilling may increase the effective spacing factor. Since small air voids are usually first filled by ettringite, the infilling may have more significant effect on the spacing factor compared to the air content. When the spacing factor is smaller for a concrete, the increase may have negligible effect on its freeze-thaw durability. However, if a concrete has greater spacing factor, the increase may reduce its freeze-thaw resistance. That is evident by values of the spacing factor listed in Table 4. The mixes in Group 1 generally have greater values of the spacing factor than those mixes in Group 2 (except values of Mixes 3 and 6 measured by the Research Laboratory of Ash Grove Cement Company). The change of the freeze-thaw expansion at 300 cycles is plotted against the spacing factor in Fig. 15. The change of expansion is difference between the expansion of specimen with 222-day moist curing and that of specimen with 110-day moist curing. Since Mix 4 after 222-day moist curing failed far before 300 cycles (see Fig. 6), its expansion at 300 cycles is not available. This change increases with increasing of the spacing factor, which corresponds to more infilling. This fact indicates that a concrete with the smaller spacing factor will have less change in the freeze-thaw expansion, and may tolerate more infilling.

4.3 Effect of Cement Chemistry on Spacing Factor

The spacing factor is plotted against potassium alkali (K_2O) in cement in Fig. 16. When K_2O is up to approximately 0.55% in cement, the values of the spacing factor are almost constant. The spacing factors are much greater for Mixes 1 and 4 which used Cement A with $K_2O = 1.17\%$. Between $K_2O = 0.55\%$ and 1.17% , a linear relationship for the spacing factor and K_2O in cement is assumed for simplicity.

ACI Committee 201 proposed that the spacing factor should not exceed 0.2 mm for freeze-thaw durable concretes^[15]. This requirement is also plotted in Fig. 16. This requirement leads to that K_2O in cement should not exceed 0.66%. This indicates that the requirement of K_2O in cement $\leq 0.6\%$ for pavements by Iowa Department of Transportation is possibly suitable for the freeze-thaw durability.

The spacing factor is plotted against sulfur trioxide (SO_3) in cement in Fig. 17. A trend similar to the effect of K_2O is observed. It is seen from the figure that the ACI requirement results in that SO_3 in cement should not exceed 3.06%. This indicates that the requirement of SO_3 in cement $\leq 3.0\%$ for pavements by Iowa Department of Transportation is possibly also suitable for the freeze-thaw durability.

It is noted that the above discussion is relied on the experimental result used Cement A, which are high in both K_2O and SO_3 (see Table 1). Although influences of K_2O and SO_3 in cements on the spacing factors have been separately discussed above, the effects of two components are actually coupled in the experimental data. It is difficult to completely separate influences of K_2O and SO_3 in cements on the spacing factor based on the available results. From the practical purpose,

one may establish separate limits on these two components in cements. These have been recently properly proposed by Iowa Department of Transportation.

5. SUMMARY

The following conclusions can be reached based on this laboratory study.

(1) No significant expansion was recorded from the second day after casting until the start of the freeze-thaw cycling (up to 222-day of the moist curing) for all the concretes tested. The major cause of distress in these nine concretes is freeze-thaw damage after long period of the moist curing.

(2) Entrained air system is critical for the freeze-thaw durability of the concretes tested. Many air voids in concretes are filled by sulfate-rich component which may be ettringite. The infilling implies that these filled air voids are water-accessible. That may comprise effectiveness of these air voids to protect concrete from freeze-thaw attack. The infilling also increases the effective spacing factor in concrete. These may lead to concrete more vulnerable to freeze-thaw attack.

(3) Concretes with 15% Class C fly ash replacement usually show more infilling of ettringite in their entrained air voids, and have greater freeze-thaw expansion compared to similar mixes without the fly ash. The effect of fly ash is greater in some concretes than in others.

(4) More infilling ettringite is generally found in the air voids when the specimens were subjected to the wet/dry treatment and to the extended moist curing. A longer period of moist curing normally leads to lower freeze-thaw resistance for the concretes.

(5) The change of the concrete freeze-thaw response after different periods of the moist curing relates to the spacing factor in the concretes. The greater values of the spacing factor, the more change of the freeze-thaw response.

(6) The amount of ettringite in a concrete depends on amounts of sulfate ions and calcium aluminate which are available in mixture. These two components come from cement and fly ash used (other external sources are also possible). A further study to quantitatively determine the amount of the infilling ettringite in concrete is needed so that the extent of the infilling might be correlated to chemical compositions of the cement and fly ash used.

(7) By combining the above results with field observations discussed below, one can conclude that the freeze-thaw damage is a factor related to premature deterioration of PCC pavements in Iowa.

(8) Since the freeze-thaw damage is related to the infilling of ettringite in entrained air voids, the similar freeze-thaw test should be conducted to determine the extent of the infilling for different combinations of materials used in Iowa pavements. This will be done in Phase II study.

6. SOME FIELD EVIDENCE

Some field evidence from deteriorated pavements in Iowa is discussed in this section. This evidence may provide some correlation between the afore-discussed laboratory study and field performance of PCC pavements.

(1) By using a low-vacuum scanning electron microscope, Schlorholtz and Amenson^[5] recently examined cores taken from several PCC pavements in Iowa. Their results are briefly summarized in Table 4. Among six pavements with moderate or high distress, they reported that the freeze-thaw damage is the major failure mechanism for five of them (two pavements, US 169 and IA 25, are due to the use of freeze-thaw sensitive coarse aggregate). For Bettendorf Fast-Track using Type III cement, evidence associated with freeze-thaw, alkali-silica reaction and sulfate attack

were observed. They also observed the infilling of entrained air voids by sulfate-rich component for these deteriorated pavements.

(2) The result of a field survey for the evaluation of deterioration of US 20 in Webster County^[16] is presented in Table 5. The pavement sections using cements from Lehigh Cement Company (Mixes 1 and 3, these are different with Mixes 1 and 3 listed in Table 3) shows severe deterioration^[16]. As indicated in Table 4, the deterioration of this pavement section was identified as the freeze-thaw damage by Schlorholtz and Amenson^[5]. This field observation coincides with the results obtained this laboratory study which indicates that the concretes used the cement with high potassium alkali content have lower the spacing factor and is potential vulnerable to the freeze-thaw failure attack.

(3) Field observations indicate that deterioration is usually more severe in observable vibrator trails and along transverse joints for PCC pavements. This can be reasonably explained by the freeze-thaw mechanism. Concrete in the vibrator paths is sometimes over-vibrated. This results in low entrained air content, which has been reported in some Iowa pavements, and leads to the concrete being more vulnerable to the freeze-thaw attack. On the other hand, more moisture is usually present in the area next to a transverse joint. This results in the concrete more quickly reaching a higher degree of saturation which is critical for the freeze-thaw damage.

(4) The examination of field PCC pavement cores reveals both vertical and horizontal cracks^{[2][5]}. Past experience and a recent study^[7] have shown that horizontal cracks are usually associated with the freeze-thaw damage.

(5) Past field experience indicates that the use of deicing salt may accelerates the rate of pavement deterioration. This can also be accounted for by the freeze-thaw damage. The use of

salt can reduce the vapor pressure of the salt saturated pore liquid. This means that pores dry slowly or do not dry out at all^[6]. As a result, concrete may more easily reach a higher degree of saturation and lead to the freeze-thaw damage.

(6) Examinations of cores from Highways 14 and 34, which are durable for 45 and 67 years, respectively, indicate no concentration of ettringite in large voids of the concretes (no air entrainment was used in these two PCC pavements). On the other hand, although cores from some other pavements, such as I-80 Mitchellville rest area (with fly ash, placed in 1987), US 20 at Farley (with fly ash, placed in 1988), US 20 at Dyersville (no fly ash, placed in 1988), and the section of Mix 2 in Table 5 (US 20 in Westber County) show some infilling of ettringite in entrained air voids, no premature deterioration has been found in these pavements this date. This implies there may be a threshold value of the effective entrained air content. A concrete may be freeze-thaw durable if its effective air content is greater than this threshold value. More quantitative study will be conducted to verify this in the Phase 2 study.

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The authors appreciate the input of B. C. Brown, C. Narotam, J. Bergren, V. Marks, J. Grove, T. Hansen and W. Dubberke from Iowa DOT. The discussion from members of Iowa DOT Materials Quality Task Group are acknowledged. A special thank to Jerry Amenson of Iowa State University for taking SEM photography. Thanks are also due to Robert Suderman of LaFarge, Canada, Inc. and Greg Barger of Ash Grove Cement Company for their help on the linear traverse measurements of air parameters.

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TABLE TITLES

1. Chemical Properties of Cements Used
2. Chemical Properties of the Fly Ash Used
3. List of Mixes for Testing
4. Measured Entrained Air Parameters in Hardened Concretes (Linear Traverse method, ASTM C457)
5. Summary of Observation of Cores from PCC Pavements
6. Condition of Some US 20 Pavement Sections in Webster County (Based on 1996 Survey)

Table 1 Chemical Properties of Cements Used

| Cement sources | C ₃ A (%) | SO ₃ (%) | MgO (%) | Na ₂ O (%) | Al ₂ O ₃ (%) | Fe ₂ O ₃ (%) | SiO ₂ (%) | CaO (%) | K ₂ O (%) |
|---------------------|----------------------|---------------------|---------|-----------------------|------------------------------------|------------------------------------|----------------------|---------|----------------------|
| Dixon-Marquette | 7.55 | 3.67 | 3.85 | 0.08 | 4.71 | 3.34 | 19.54 | 61.73 | 1.17 |
| Lafarge | 8.55 | 2.93 | 2.97 | 0.17 | 4.60 | 2.66 | 21.05 | 63.67 | 0.55 |
| Ash Grove (Type II) | 6.73 | 2.46 | 3.50 | 0.15 | 4.28 | 3.31 | 21.64 | 62.90 | 0.54 |
| Ash Grove (Type IP) | - | 2.63 | 3.13 | 0.13 | 9.84 | 3.20 | 26.16 | 51.68 | 0.51 |
| Kaiser, California | 5.59 | 2.56 | 1.41 | 0.30 | 3.84 | 3.87 | 20.92 | 65.02 | 0.28 |

Table 2 Chemical Properties of Fly Ash (Ottumwa) Used

| SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₂ | SO ₃ | CaO | MgO | Available Equiv. Na ₂ O |
|------------------|--------------------------------|--------------------------------|-----------------|------|-----|---------------------------------------|
| 32.3 | 17.1 | 5.4 | 4.5 | 28.3 | 6.2 | 1.35 |

Table 3 List of Mixes for Testing

| Test No. | Cement Sources | Fly Ash (%) | Plastic Air Content (%) | Slump (in.) | Mix No. |
|----------|---------------------|-------------|-------------------------|-------------|---------|
| 1 | Dixon-Marquette | 0 | 6.0 | 2.5 | C-3 |
| 2 | Lafarge | 0 | 6.0 | 2.0 | C-3 |
| 3 | Ash Grove (Type II) | 0 | 6.8 | 2.5 | C-3 |
| 4 | Dixon-Marquette | 15 | 5.6 | 2.25 | C-3-C |
| 5 | Lafarge | 15 | 5.8 | 2.0 | C-3-C |
| 6 | Ash Grove (Type II) | 15 | 6.8 | 2.5 | C-3-C |
| 7 | Ash Grove (Type IP) | 0 | 5.0 | 1.75 | C-3 |
| 8 | Kaiser, California | 0 | 6.5 | 2.25 | C-3 |
| 9 | Kaiser, California | 15 | 6.2 | 2.0 | C-3-C |

Table 4 Measured Entrained Air Parameters in Hardened Concretes¹
(Linear Traverse Method, ASTM C457)

| Test No. | Cements | Larfage, Canada | | Ash Grove | |
|----------|----------------|-----------------|---------------------|-----------------|---------------------|
| | | Air content (%) | Spacing factor (mm) | Air content (%) | Spacing factor (mm) |
| 1 | A | 5.2 | <u>0.297</u> | 5.2 | <u>0.229</u> |
| 2 | B | 4.8 | 0.179 | 5.1 | 0.124 |
| 3 | C | 5.8 | <u>0.198</u> | 5.9 | <u>0.114</u> |
| 4 | A | 3.2 | <u>0.318</u> | 5.5 | <u>0.206</u> |
| 5 | B | 5.9 | 0.129 | 5.1 | 0.112 |
| 6 | C | 4.0 | <u>0.198</u> | 5.1 | <u>0.124</u> |
| 7 | D (Type IP) | 5.1 | 0.205 | 3.6 | 0.157 |
| 8 | E | 4.2 | 0.171 | 5.7 | 0.137 |
| 9 | E | 5.1 | 0.195 | - | - |

1. Samples of the age of 222 days, without the wet/dry treatment, were used for determining entrained air parameters in hardened concretes. Some of smaller voids were filled by ettringite in these samples. Only empty and partially filled (by ettringite) voids were counted in the measurement.

Table 5 Summary of Observation of Cores from PCC Pavements*
(Schlorholtz and Amenson^[5])

| Pavements | Year of Paving | Use of Fly Ash | Infilling of Ettringite | Observed Distress | Major Causes for Distress |
|--|----------------|----------------|---|-------------------|---|
| US 20, East Bound Webster County, F-520-3(12)--20-94 | 1987 | Yes | Many air voids, especially voids smaller than 100 μ m, filled with ettringite | High | Freeze-thaw. |
| I-35, North Bound, Story County, IR-35-5(40)121 | 1985 | Yes | Many air voids, especially voids smaller than 100 μ m, filled with ettringite | High | Freeze-thaw. |
| US 169, Madison County, FN-169-3(18)--21-6 | 1977 | No | Many small air voids filled with ettringite | High | Freeze-thaw of coarse aggregate. |
| IA 25, Ringgold County, F-451(9) | 1964 | No | Many small air voids filled with ettringite | High | Freeze-thaw of coarse aggregate. |
| I-80, East Bound, Dallas County, IR-80-3(57)106 | 1989 | Yes | Some small air voids filled with ettringite | Moderate | ASR, poor mixing and over-vibration. Difficult to identify the major cause. |
| Bettendorf Fast-Track, Scott County, (Used a Type III Cement) | 1987 | Yes | Ettringite fills all small voids and some gap between aggregate and cement paste | Moderate | Freeze-thaw, ASR and sulfate attack. Difficult to identify the major cause. |

* Schlorholtz and Amenson also examined cores taken from three other PCC pavements (IA 175, County Road B and Benton Fast-Track) with low distress. These results are not discussed here.

Table 6 Condition of Some US 20 Pavement Sections in Webster County
(Based on 1996 Survey)

| Mix Code | Mix Proportion | Material Sources | | | K ₂ O in cement (%) | SO ₃ in cement (%) | Distress |
|-------------|-------------------|------------------|---------------------------------|-------|--------------------------------------|-------------------------------------|------------------|
| | | Cement | Fly Ash | Sand | | | |
| 1 | C-3WR-C | Lehigh | Ottumwa | Croft | 1.06 | 3.38 | Severe |
| 2 | C-3WR | Lehigh | None | Croft | 0.65 | 3.50 | None |
| 3 | C-3-C | Lehigh | Port Neal #4 | Yates | 1.06 | 3.38 | Severe |
| 5 | C-3WR-C | Northwestern | Ottumwa & Port Neal #4 | Croft | 0.32 | 3.14 | Minor |
| 6a | C-3WR-C | Northwestern | Nebraska City | Croft | 0.32 | 3.14 | None |
| 6 | C-3WR-C | Northwestern | Nebraska City & Port Neal #4 | Croft | 0.32 | 3.14 | Minor to None |

FIGURE CAPTIONS

1. Flow Chart of Test Procedure
2. The History of Length Change for Mix 4
- 3a. SEM Photograph for Mix 4 at the Age of 60 Days Before the Wet/Dry Treatment
- 3b. Elemental Mapping of Figure 3a
- 4a. SEM Photograph for Mix 4 at the Age of 162 Days After the Wet/Dry Treatment
- 4b. Elemental Mapping of Figure 4a
- 5a. SEM Photograph for Mix 4 at the Age of 222 Days After the Wet/Dry Treatment
- 5b. Elemental Mapping of Figure 5a
6. Freeze-thaw Expansion of Mixes 1 and 4
7. Freeze-thaw Expansion of Mixes 2 and 5
8. Freeze-thaw Expansion of Mixes 1 and 4, no the Wet/Dry Treatment
9. Freeze-thaw Expansion of Mixes 2 and 5, no the Wet/Dry Treatment
10. Effect of Fly Ash Replacement and the Wet/Dry Treatment on Freeze-thaw Expansion
- 11a. SEM Photograph for Mix 3 Without the Wet/Dry Treatment
- 11b. Elemental Mapping of Figure 11a
- 12a. SEM Photograph for Mix 3 With the Wet/Dry Treatment
- 12b. Elemental Mapping of Figure 12a
- 13a. SEM Photograph for Mix 6 Without the Wet/Dry Treatment
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- 14a. SEM Photograph for Mix 6 With the Wet/Dry Treatment
- 14b. Elemental Mapping of Figure 14a
15. Relationship Between Change of Expansion and Spacing Factors
16. Relationship Between K_2O in Cement and Spacing Factors
17. Relationship Between SO_3 in Cement and Spacing Factors

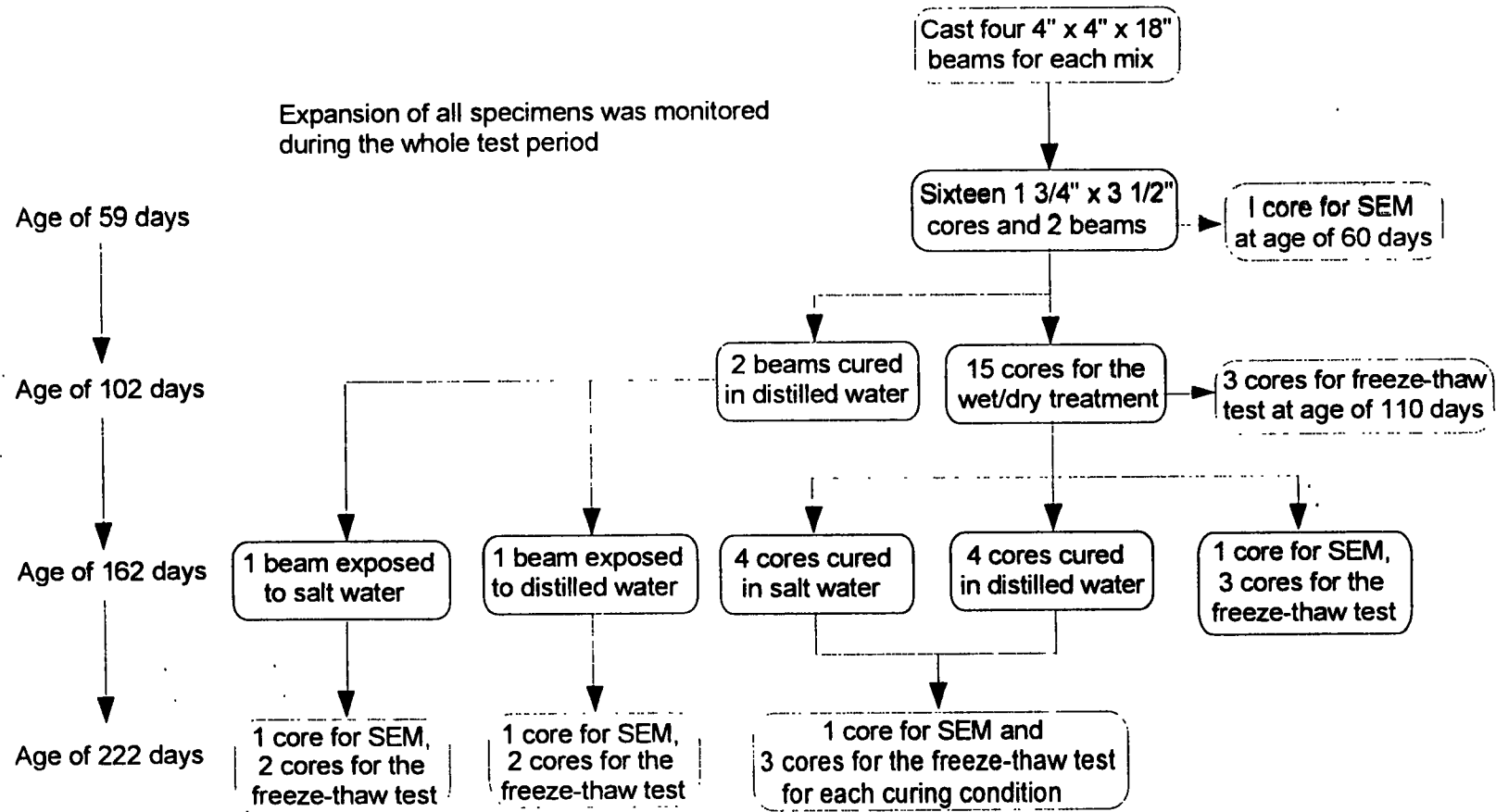


Fig. 1 Flow chart of test procedure

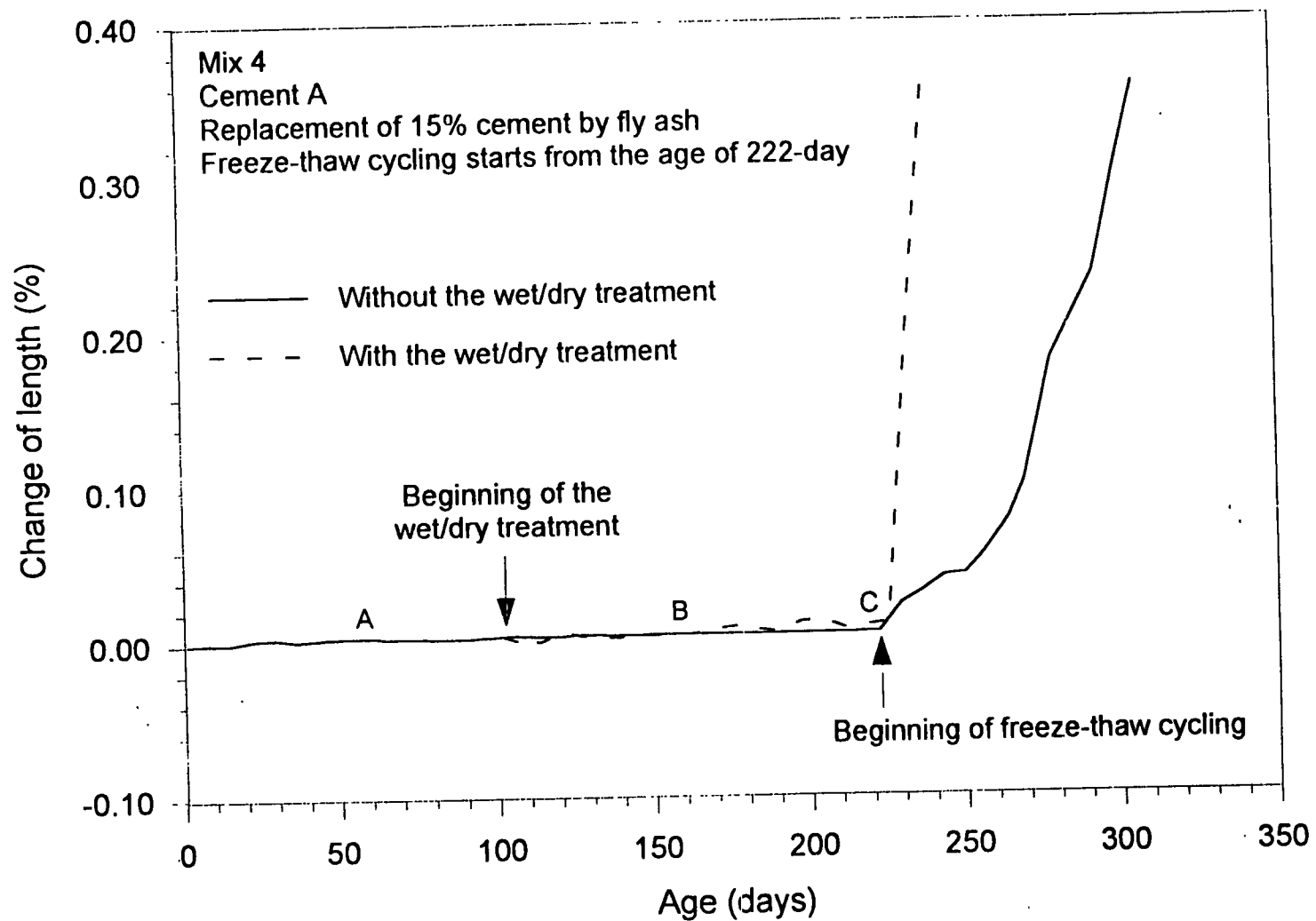


Fig. 2 The History of Length Change for Mix 4

Operator: Jerry Amenson

Client: Cheng

Job: IOWA DOT Cheng

Res: High

Label: Mix 4, 60 Day, 100x

(1 May 95 12:56:56)

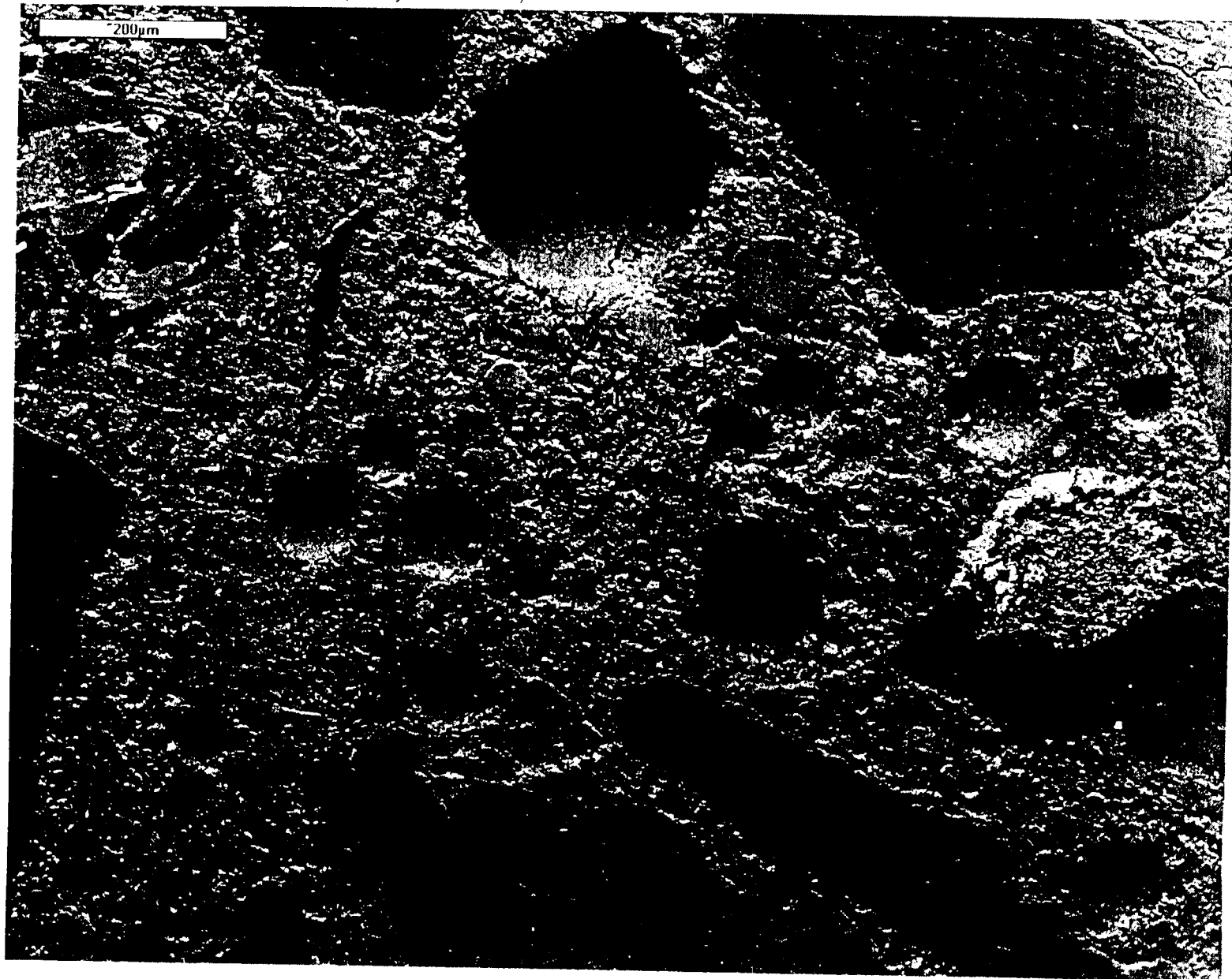


Fig. 3a SEM Photograph for Mix 4 at the age of 60 days before the wet/dry treatment

Operator: Jerry Amenson
Client: Cheng
Job: IOWA DOT Cheng
Label: Mix 4, 60 Day, 100x (1 May 95 14:47:38)

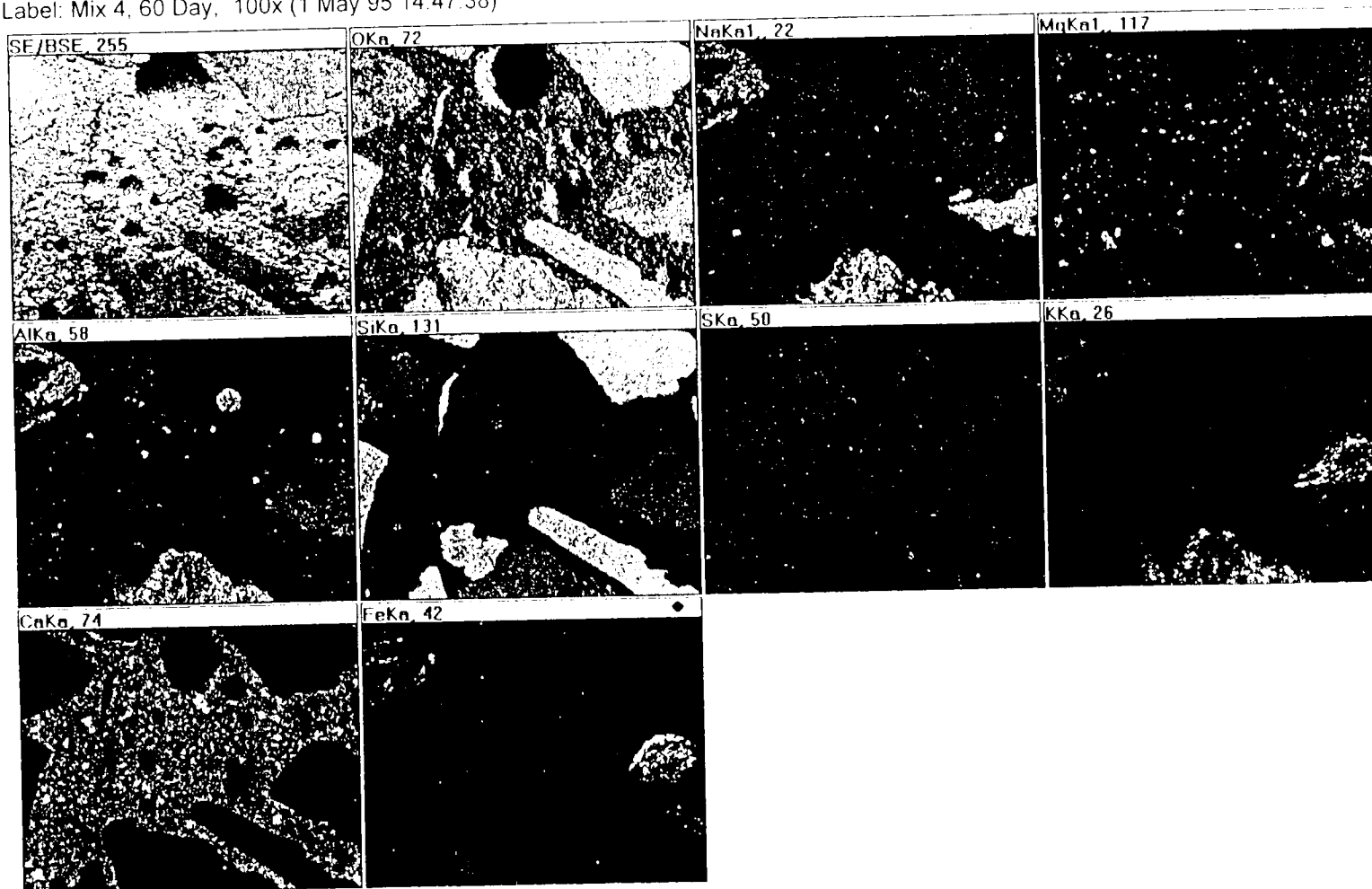


Fig. 3b Elemental Mapping of Fig. 3a

Operator: Jerry Amenson

Client: Cheng

Job: IOWA DOT Cheng

Res: High

Label: Mix 4, 162 Day @ 100x

(21 Aug 95 10:17:07)

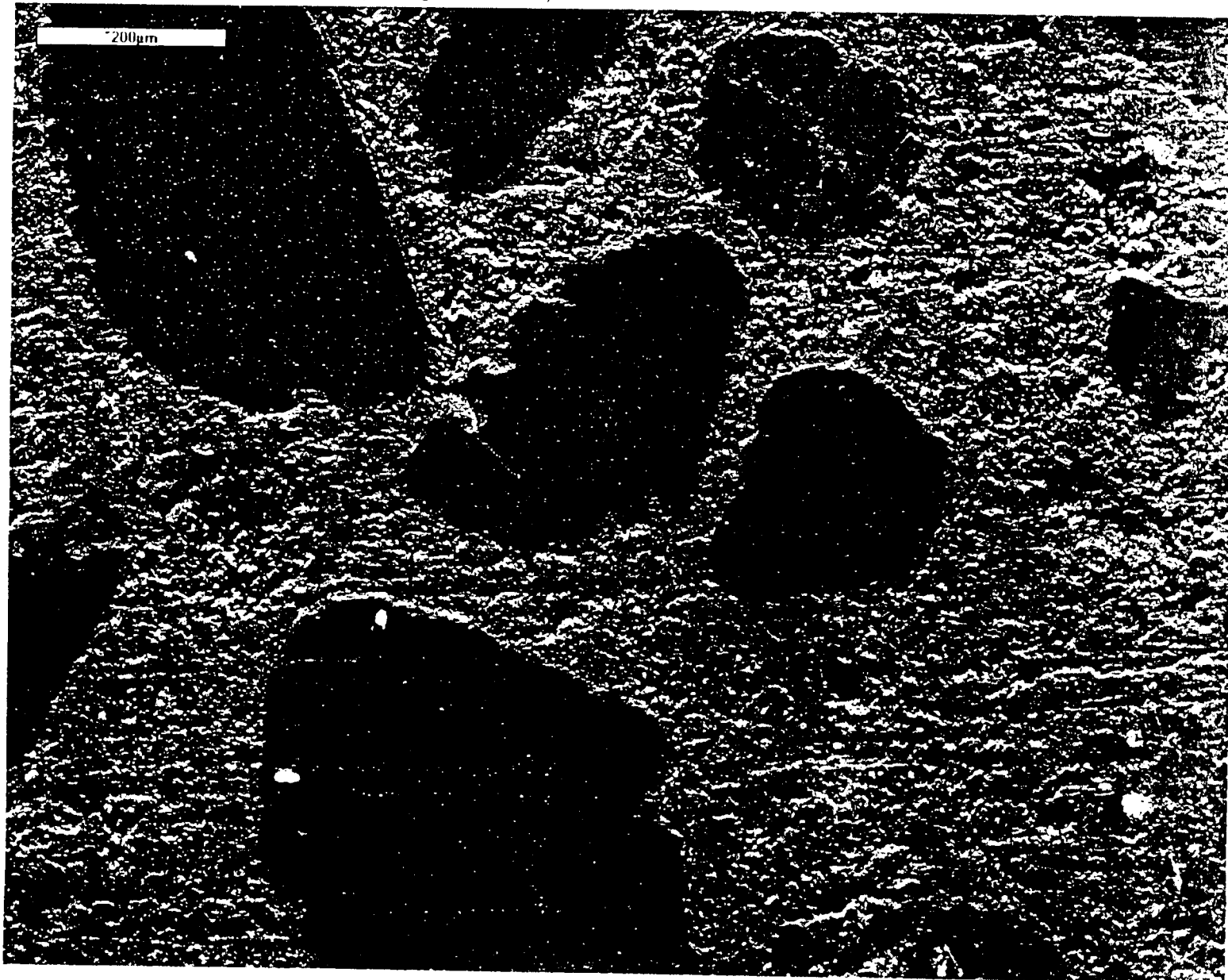


Fig. 4a SEM Photograph for Mix 4 at the age of 162 days after the wet/dry Treatment

Operator: Jerry Amenson
Client: Cheng
Job: IOWA DOT Cheng
Label: Mix 4, 162 Day Map @ 100x (21 Aug 95 10:49:36)

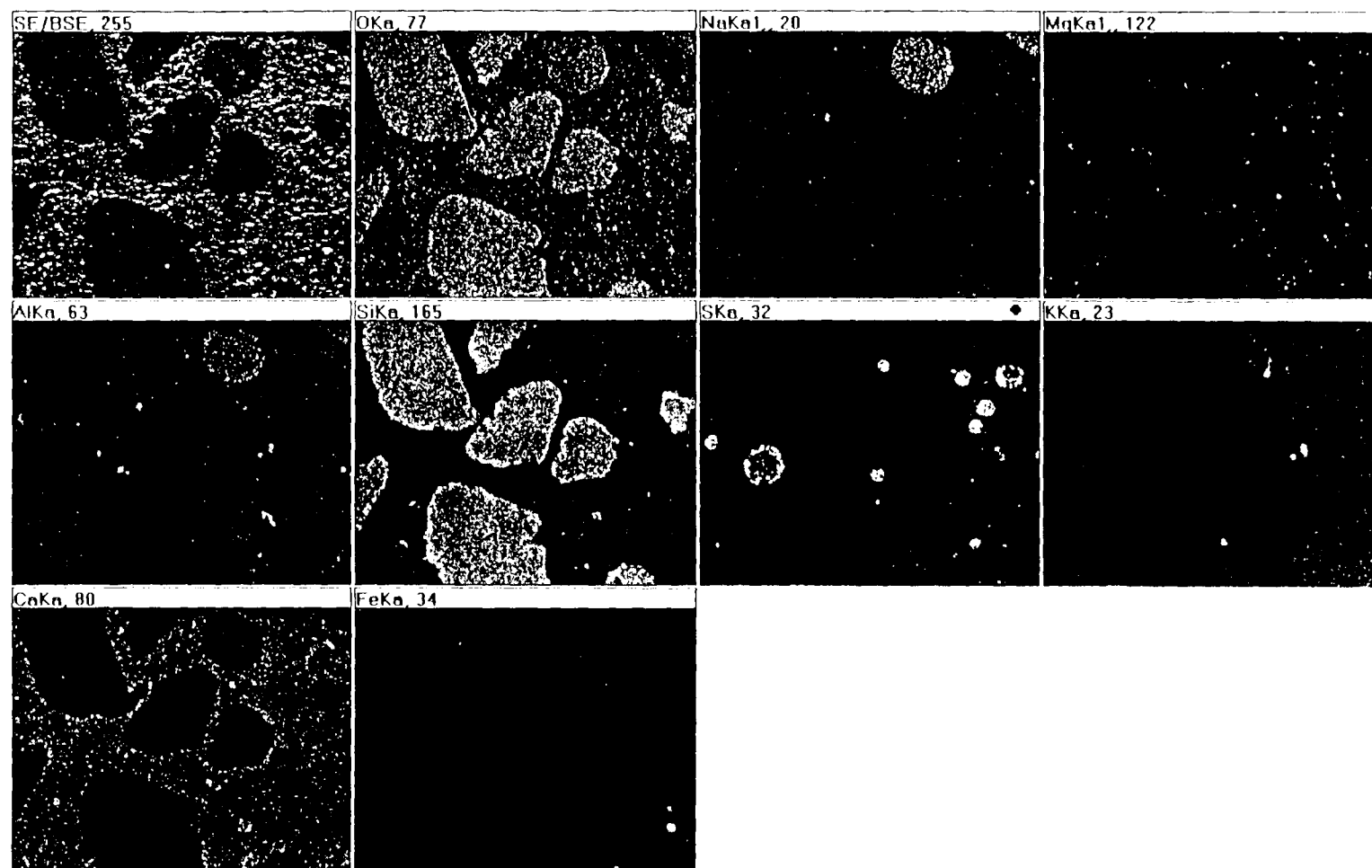


Fig. 4b Elemental mapping of Fig. 4a

Operator: Jerry Amenson
Client: Cheng
Job: Iowa DOT
Res: High
Label: M4 , 222d, dist water, @ 100x (12 Oct 95 08:27:32)

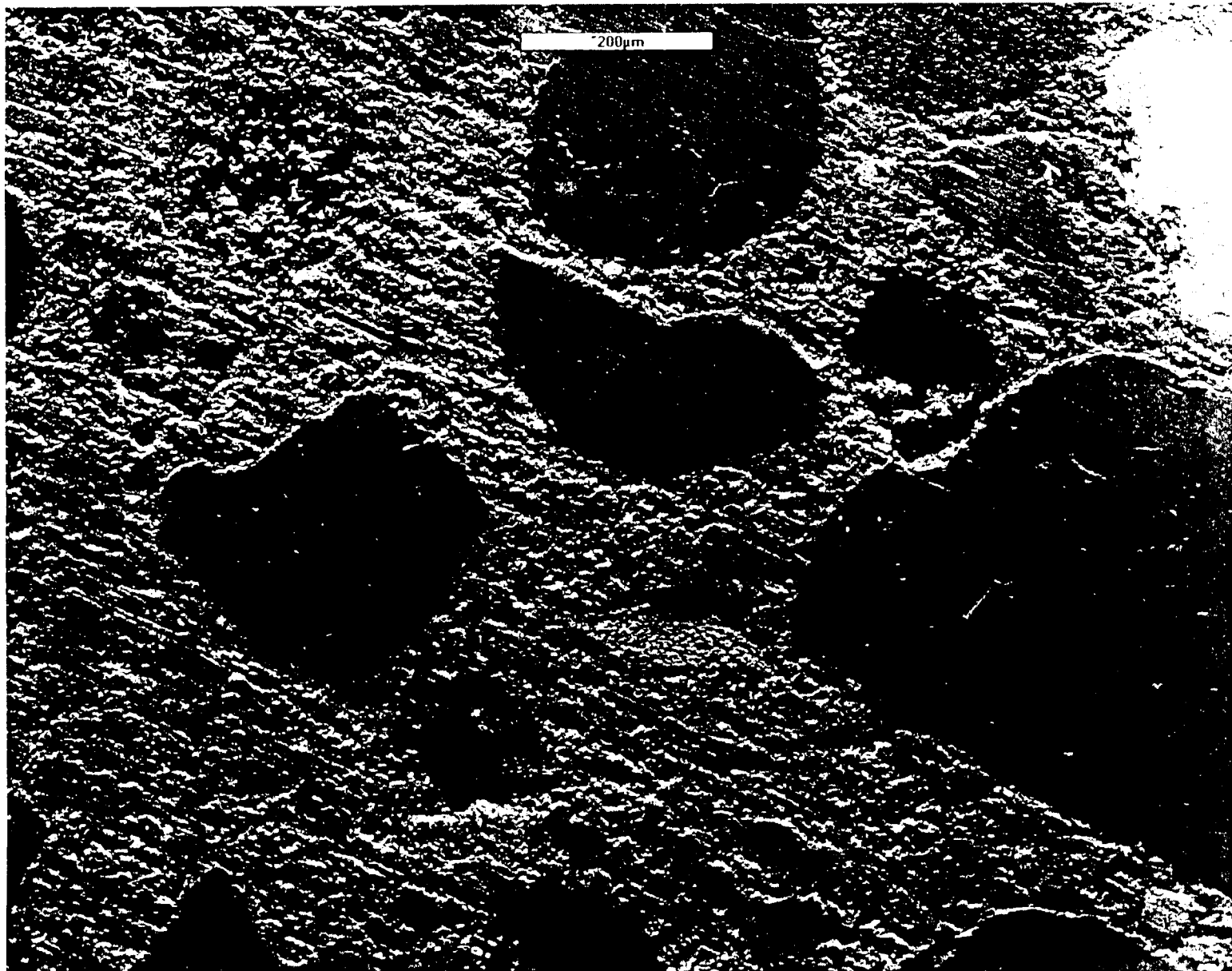


Fig. 5a SEM Photograph for Mix 4 at the age of 222 days after the wet/dry Treatment

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Label: M4 , 222d, dist water, @ 100x m (12 Oct 95 08:51:26)

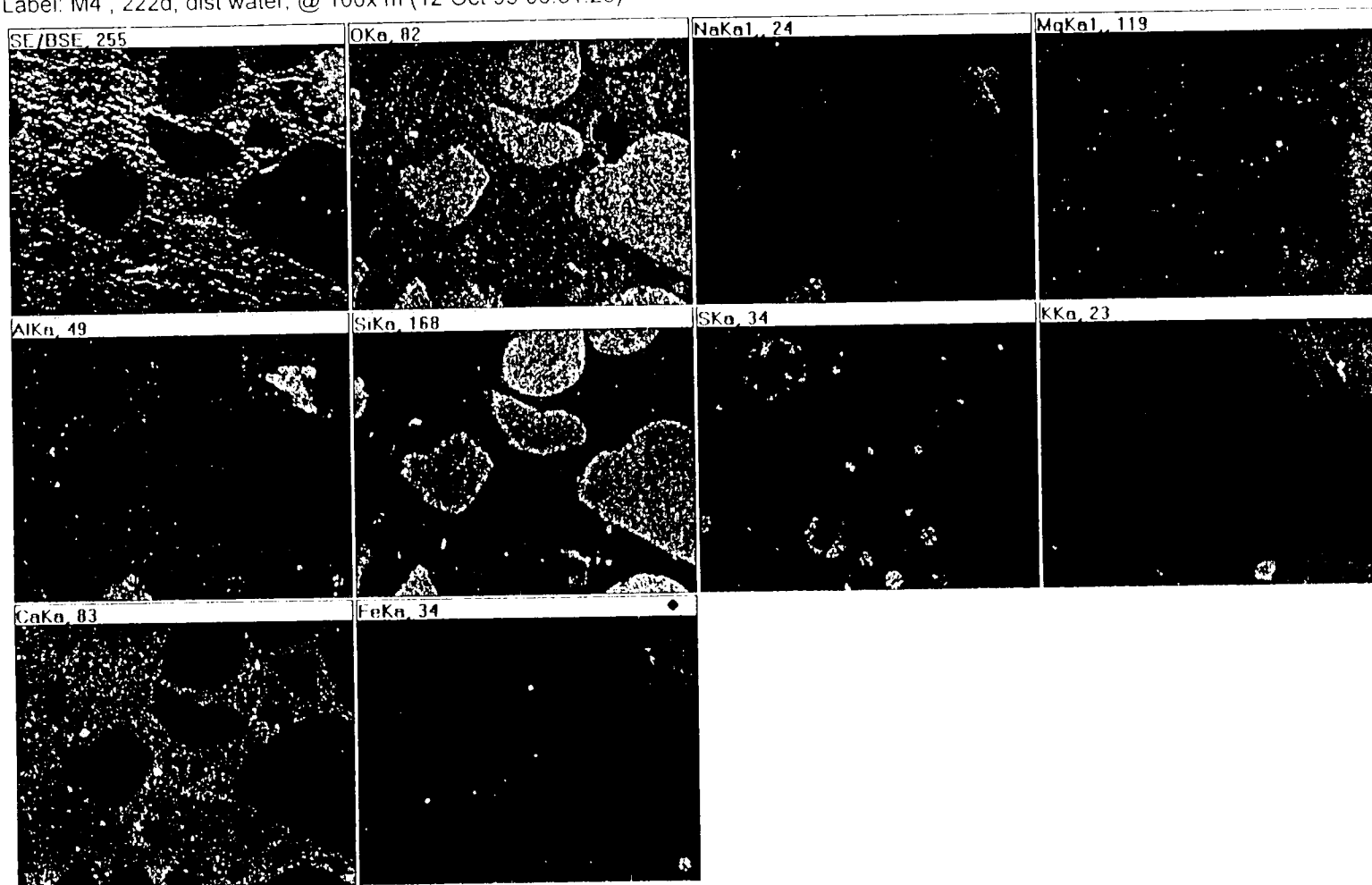


Fig. 5b Elemental Mapping of Fig. 5a

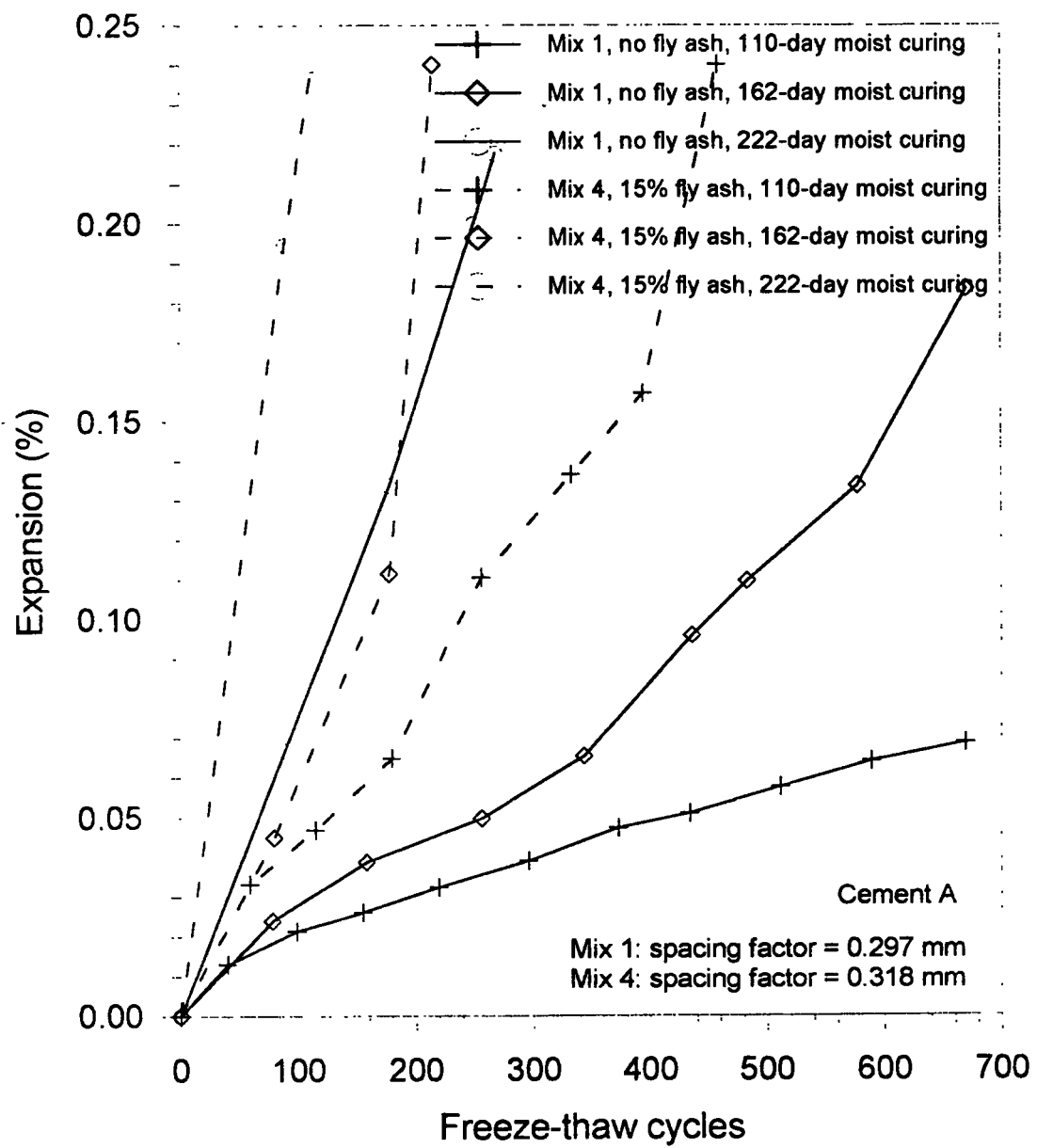


Fig. 6 Freeze-thaw expansion of mixes 1 and 4

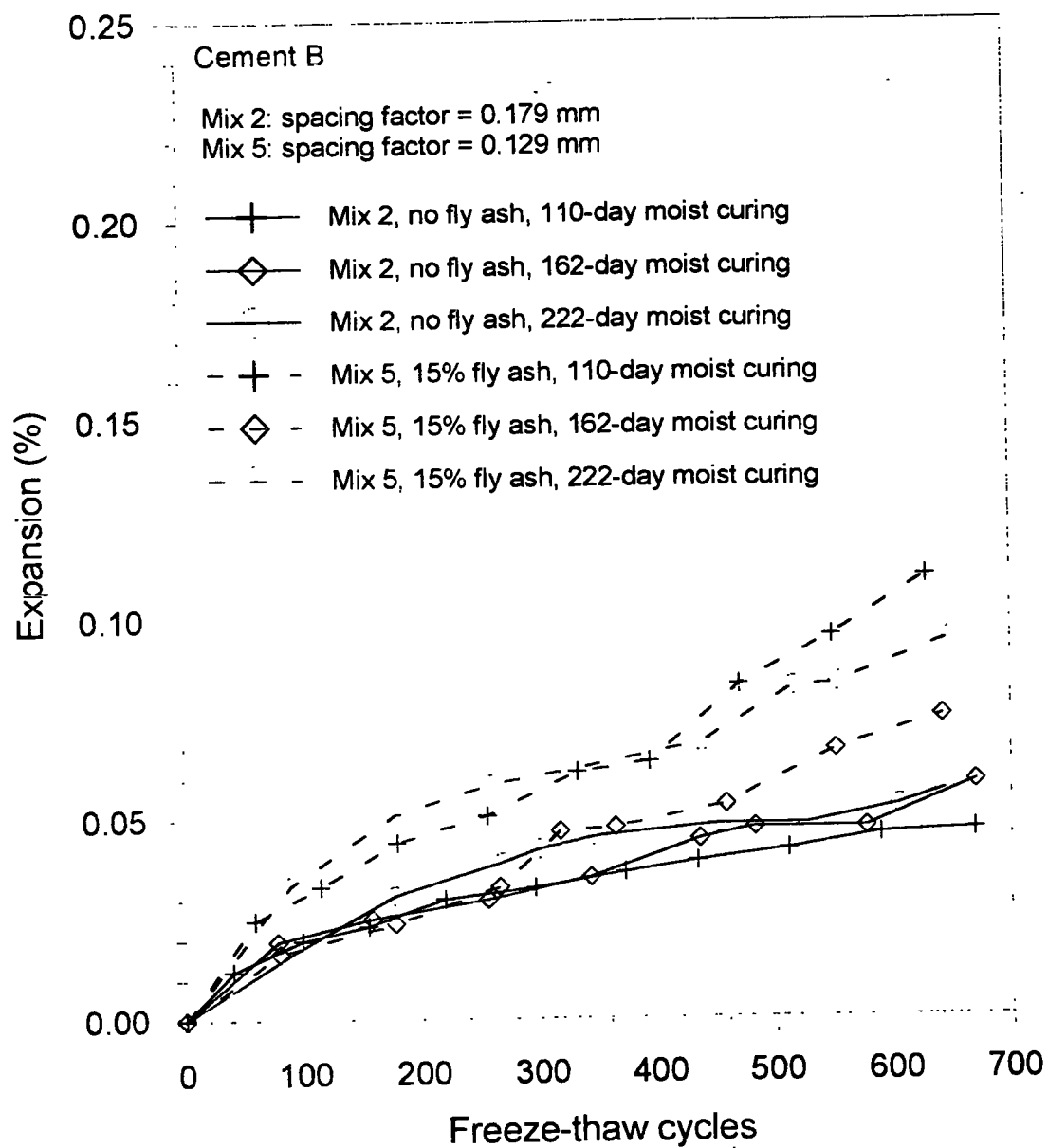


Fig. 7 Freeze-thaw expansion of mixes 2 and 5

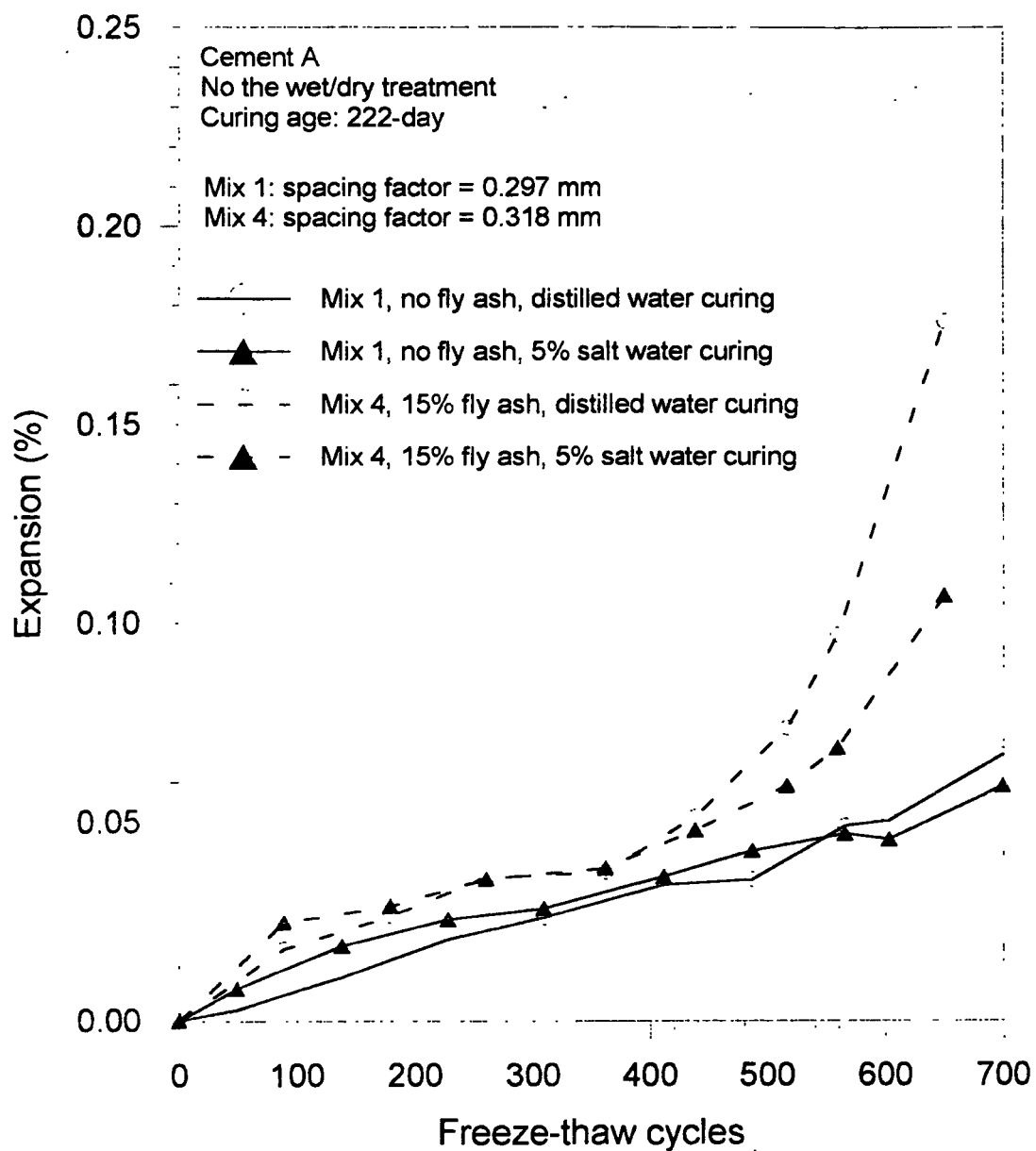


Fig. 8 Freeze-thaw expansion of mixes 1 and 4, no the wet/dry treatment

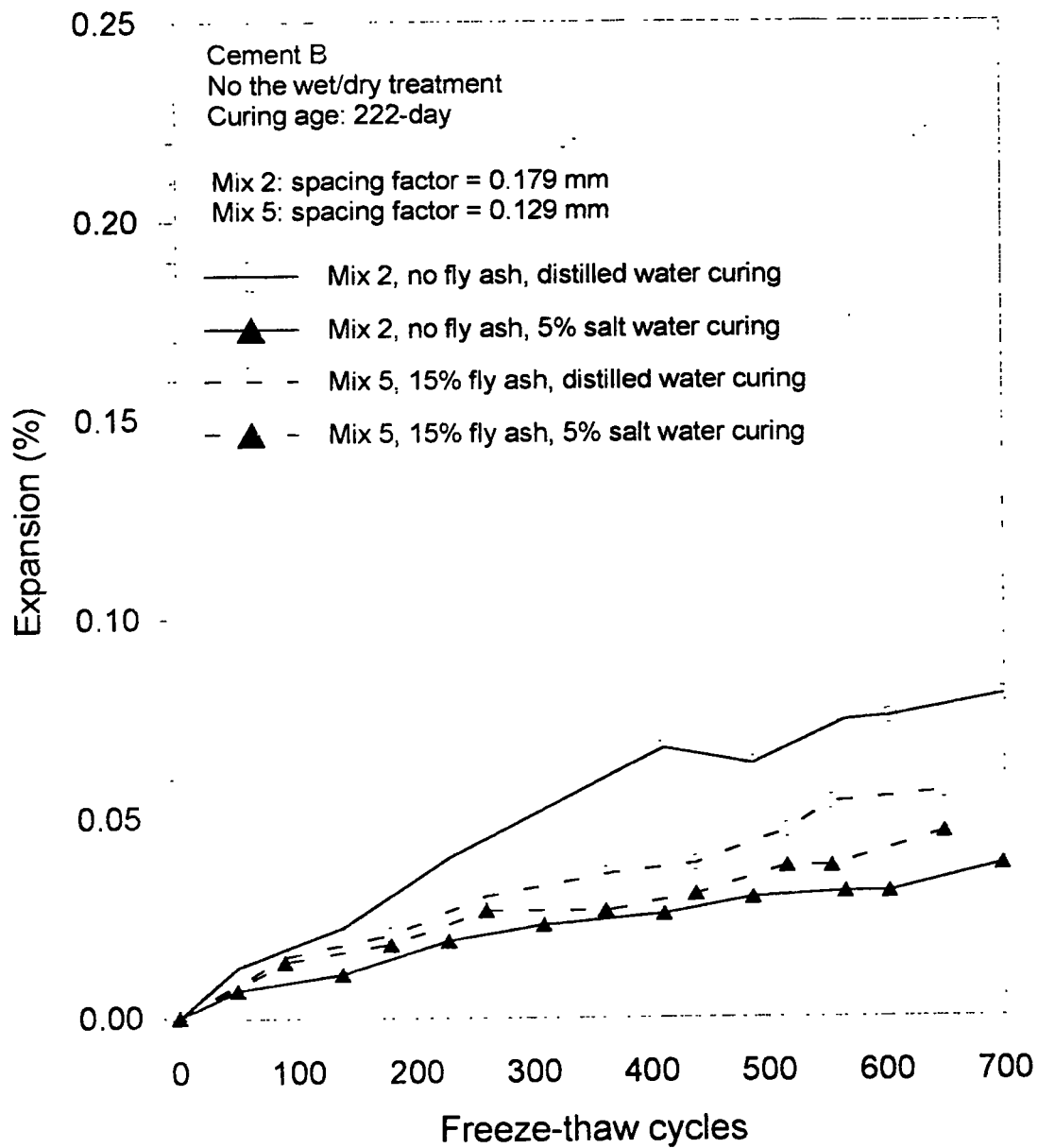


Fig. 9 Freeze-thaw expansion of mixes 2 and 5, no the wet/dry treatment

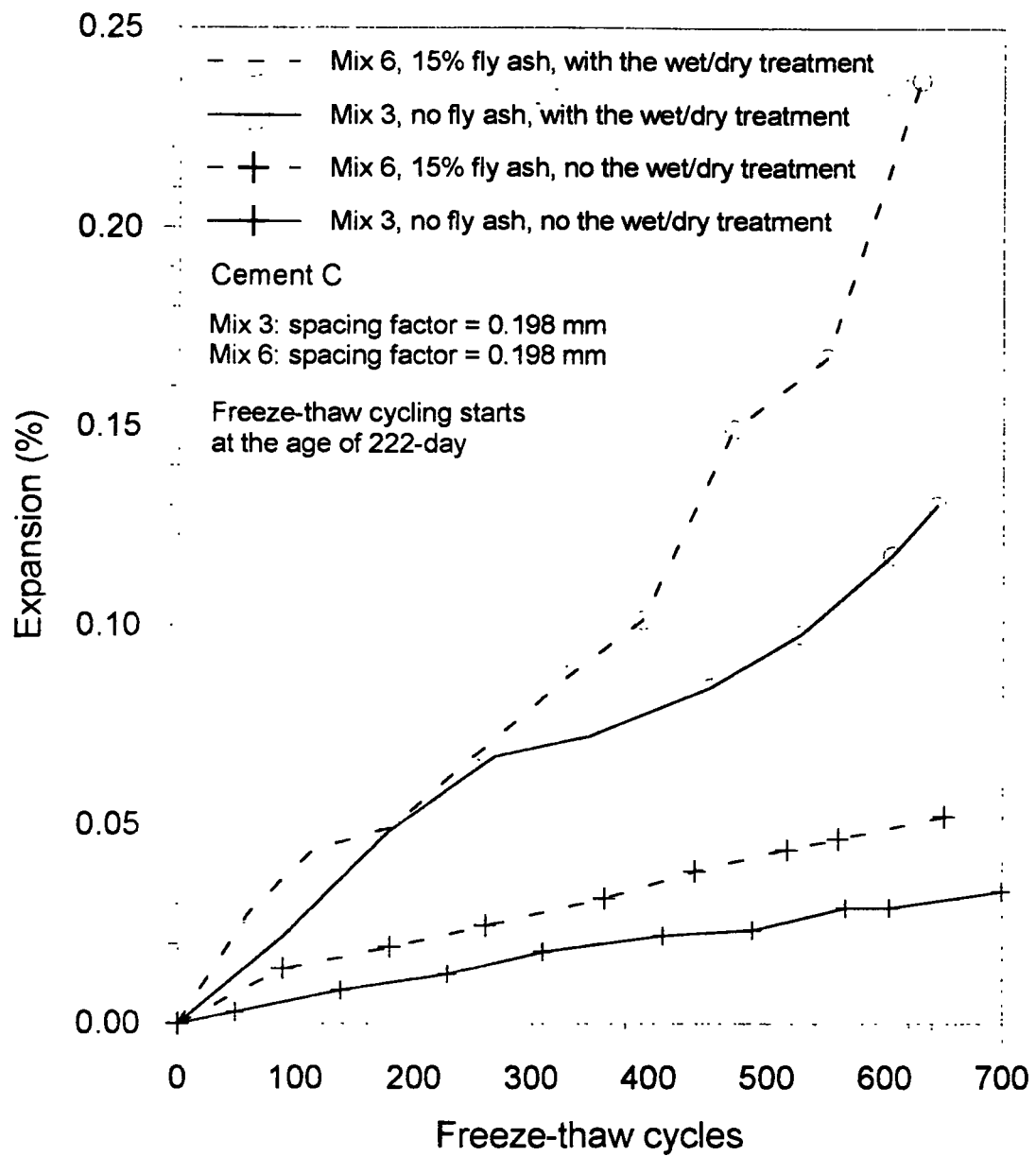


Fig. 10 Effect of fly ash replacement and the wet/dry treatment on freeze-thaw expansion

Operator: Jerry Amenson
Client: Cheng
Job: Iowa DOT
Res: High
Label: M3, 222d, dist, no w/d trt @ 100x (6 Oct 95 18:06:26)

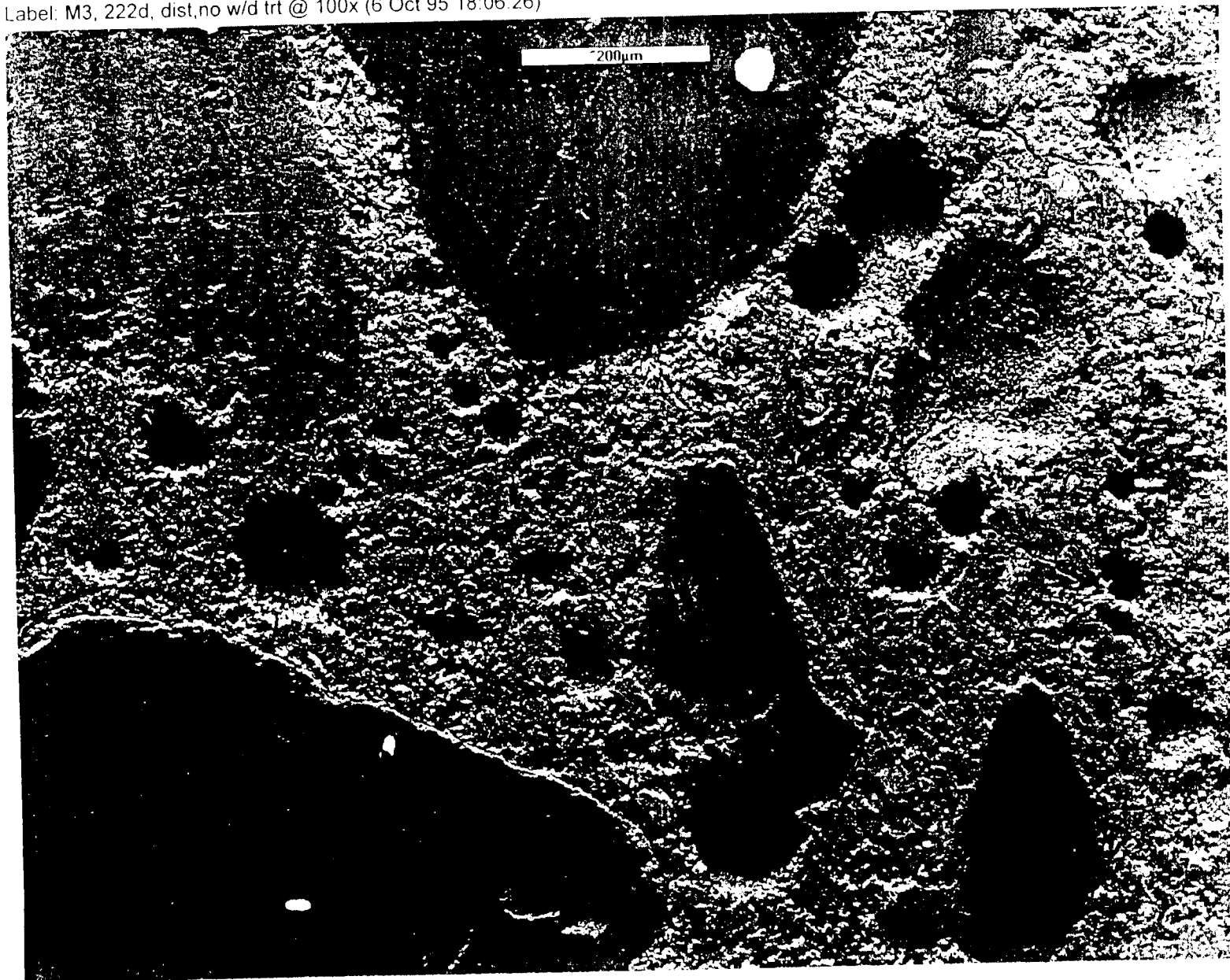


Fig. 11a SEM Photograph for Mix 3 without the wet/dry treatment

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Label: M3, 222d, dist, no w/d trt 100x m (6 Oct 95 18:26:52)

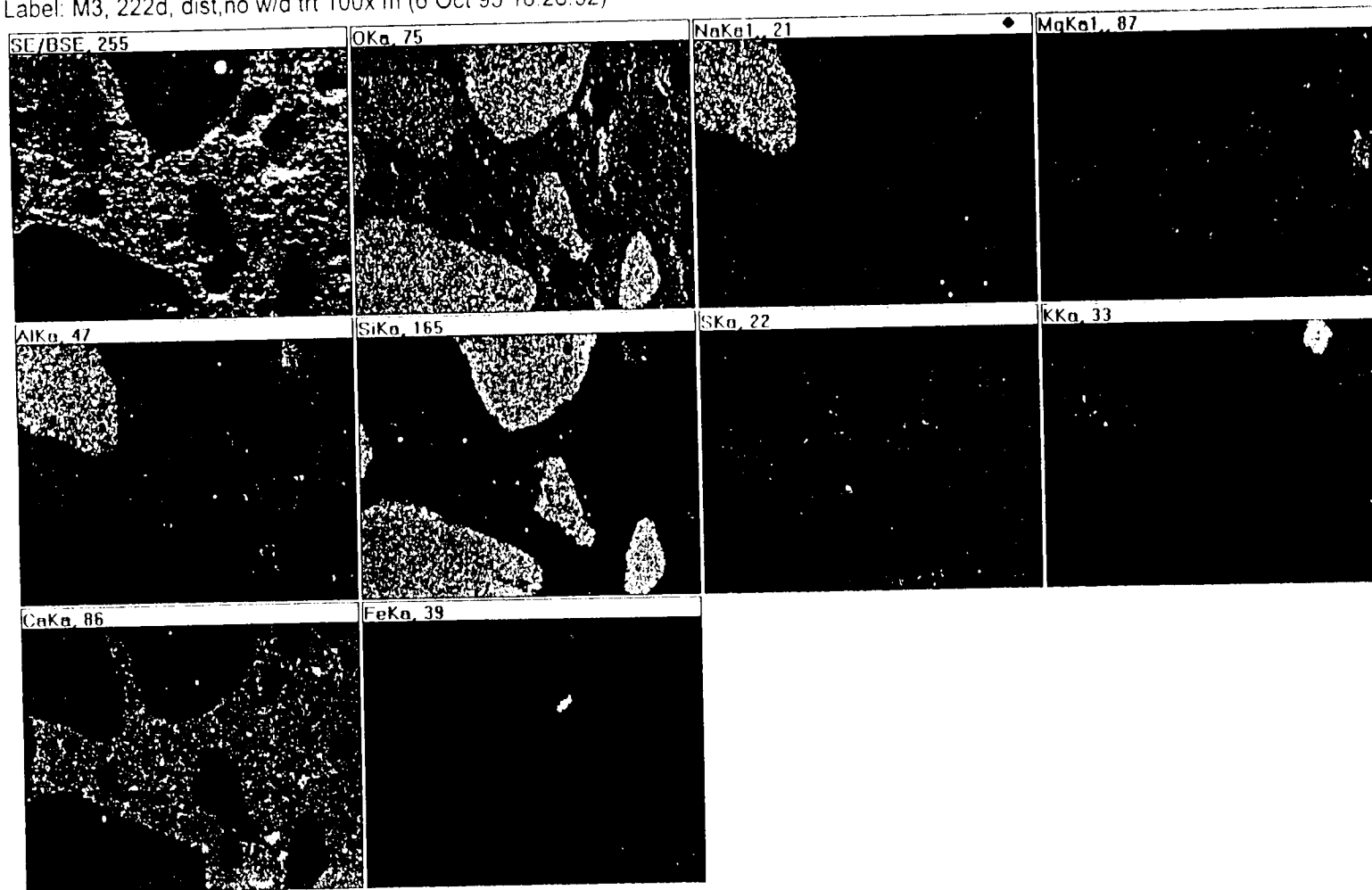


Fig. 11b Elemental mapping of Fig. 11a

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Res: High

Label: M3, 222d, distilled water, 100x (6 Oct 95 18:53:57)

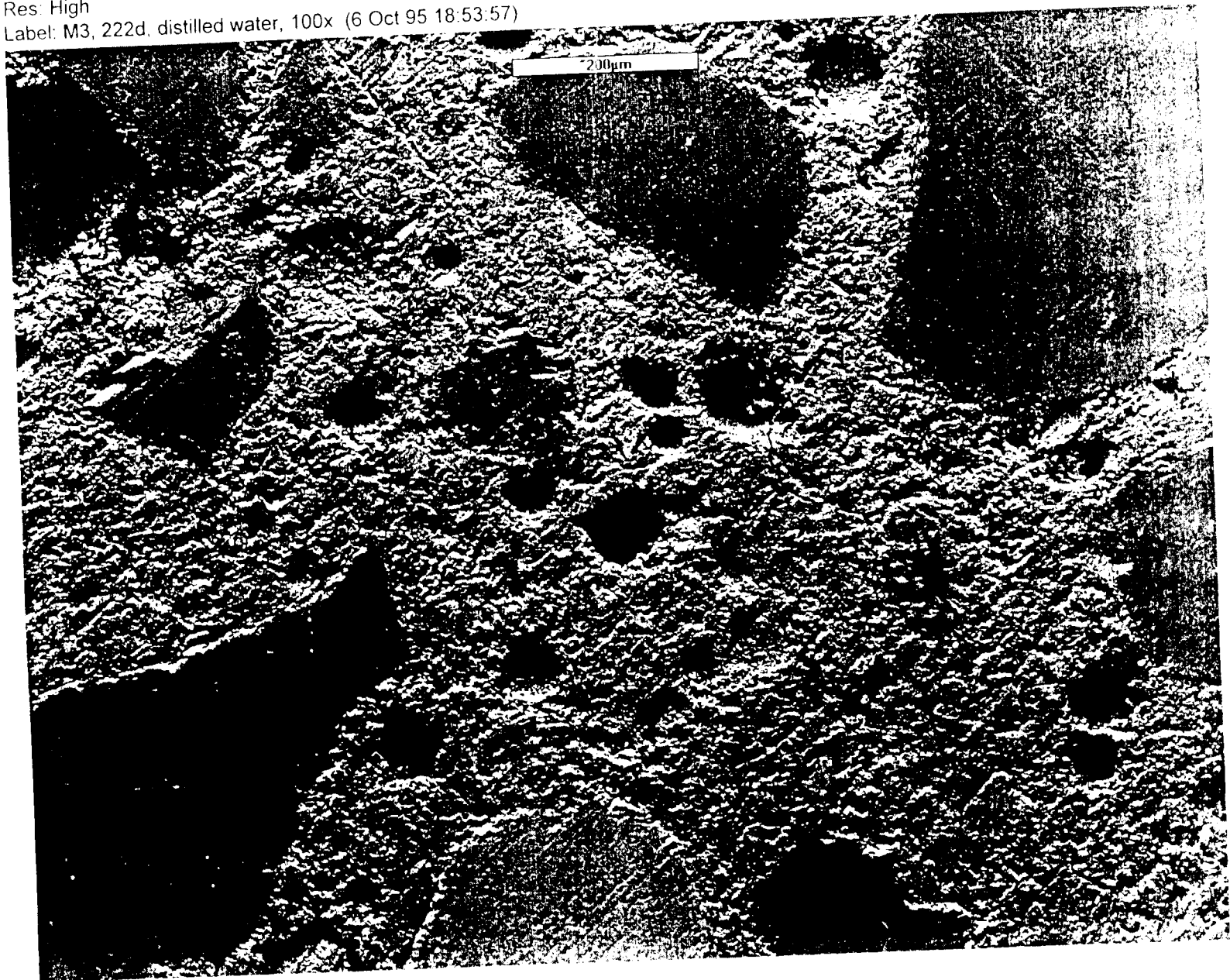


Fig. 12a SEM Photograph for Mix 3 with the wet/dry Treatment

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Label: M3, 222d, distilled water, 100x m (6 Oct 95 19:16:28)

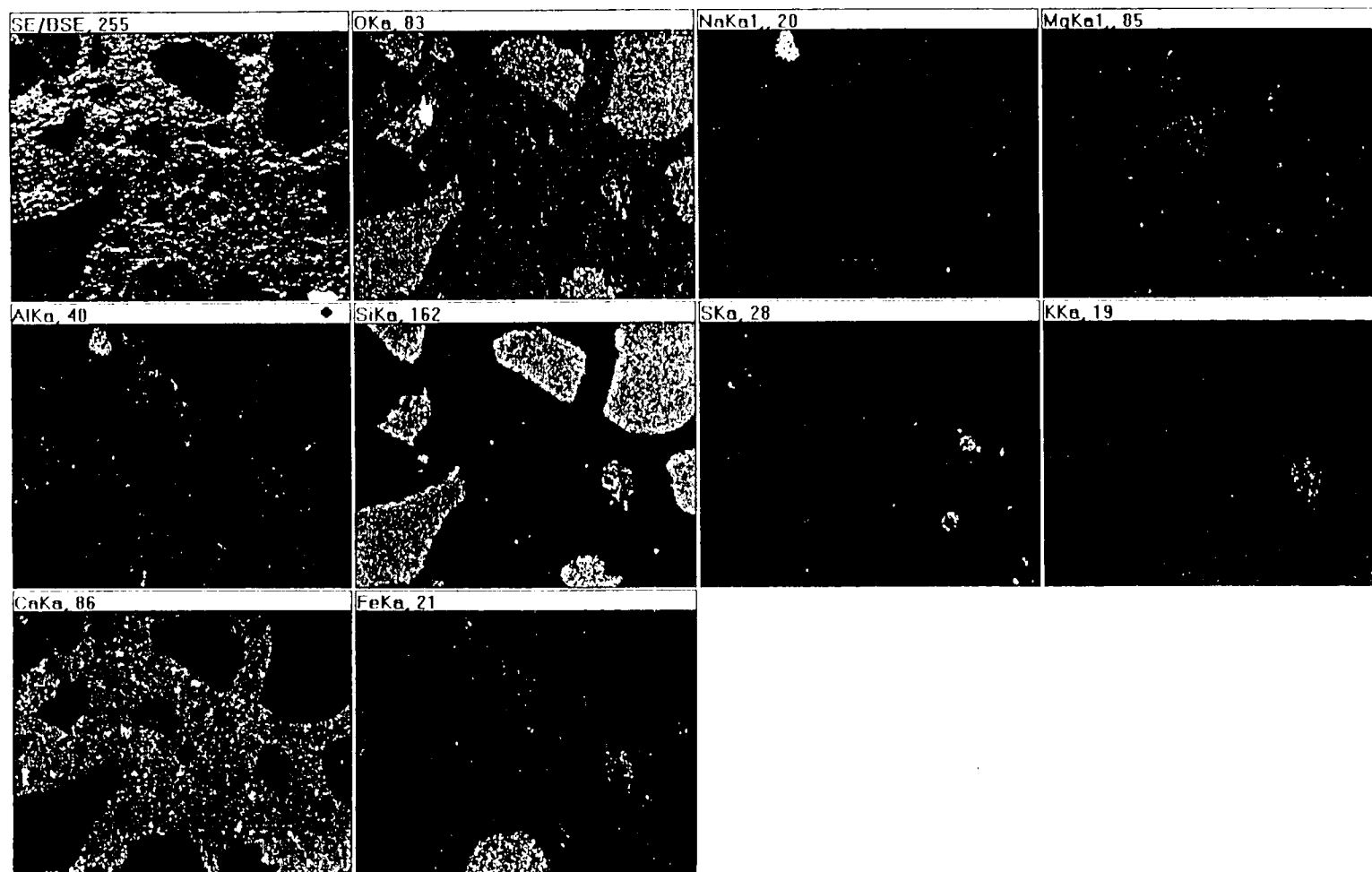


Fig. 12b Elemental Mapping of Fig. 12a

Operator: Jerry Amenson
Client: Cheng
Job: Iowa DOT
Res: High
Label: M6,222d.dist.no w/d trt @ 100x (12 Oct 95 12:04:23)

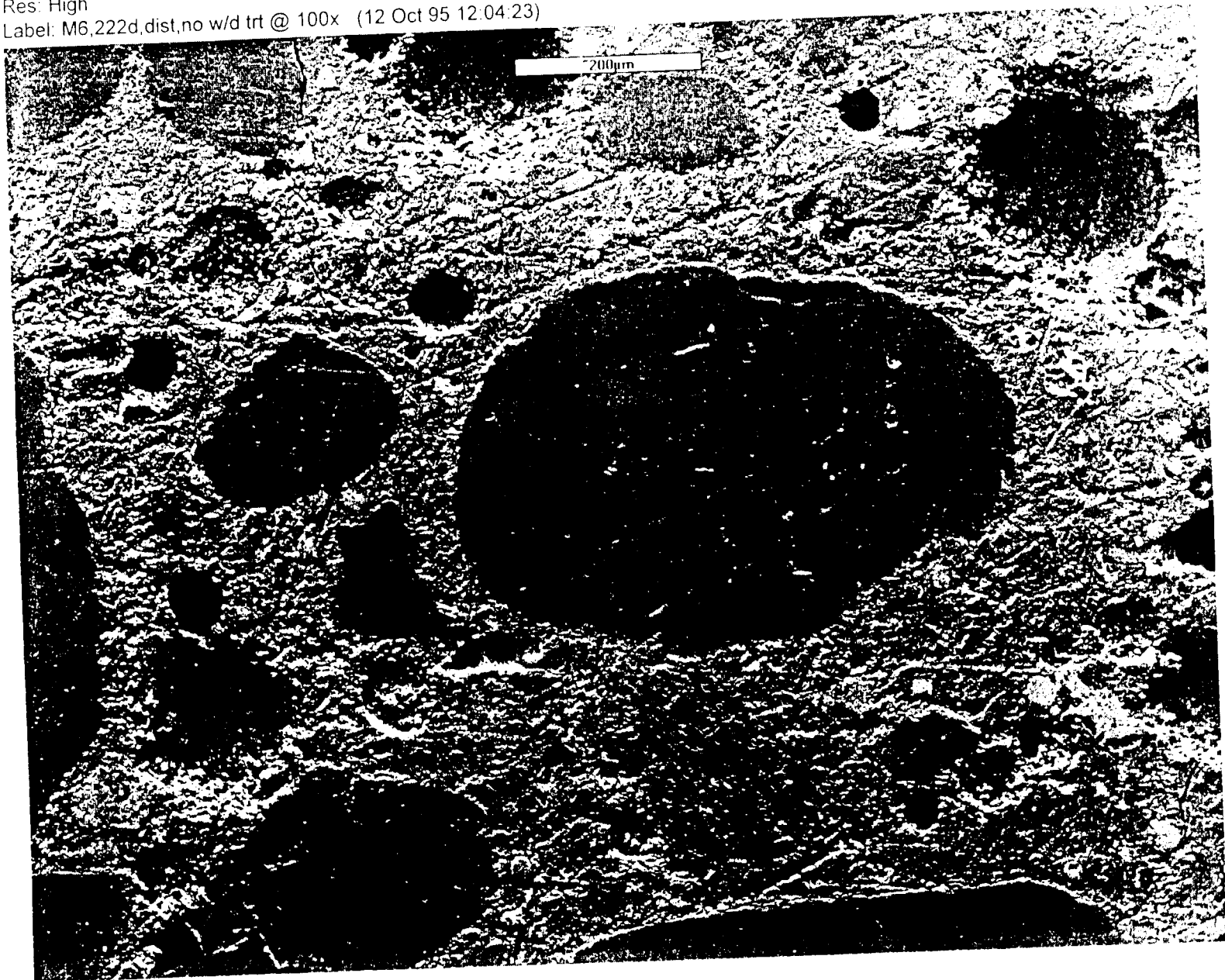


Fig. 13a SEM Photograph for Mix 6 without the wet/dry Treatment

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Label: M6,222d,dist,no w/d trt @ 100x m (12 Oct 95 12:25:09)

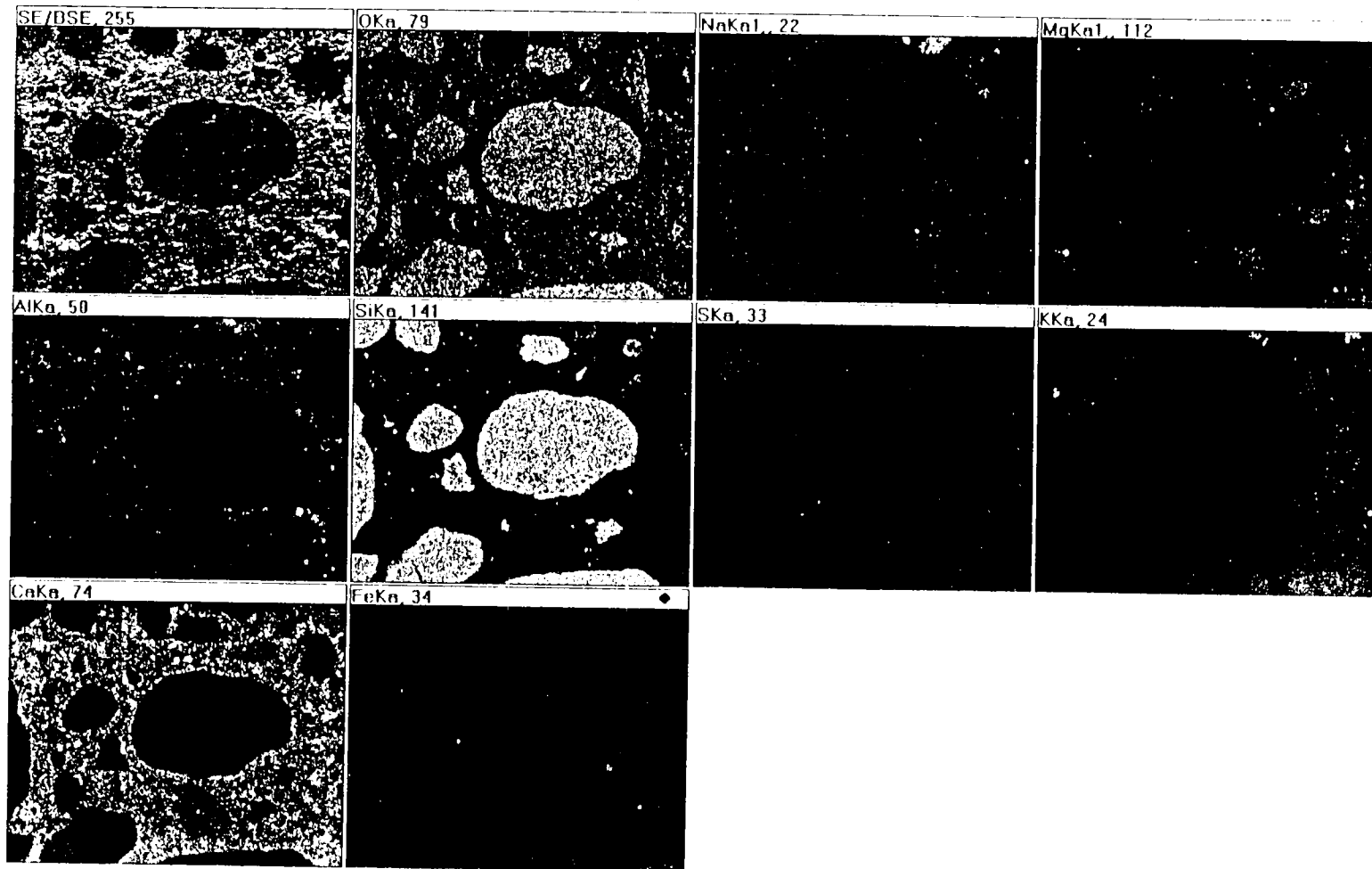


Fig. 13b Elemental Mapping of Fig. 13a

Operator: Jerry Amenson
Client: Cheng
Job: Iowa DOT
Res: High
Label: M6,222d,distilled water @ 100x (12 Oct 95 13:02:12)

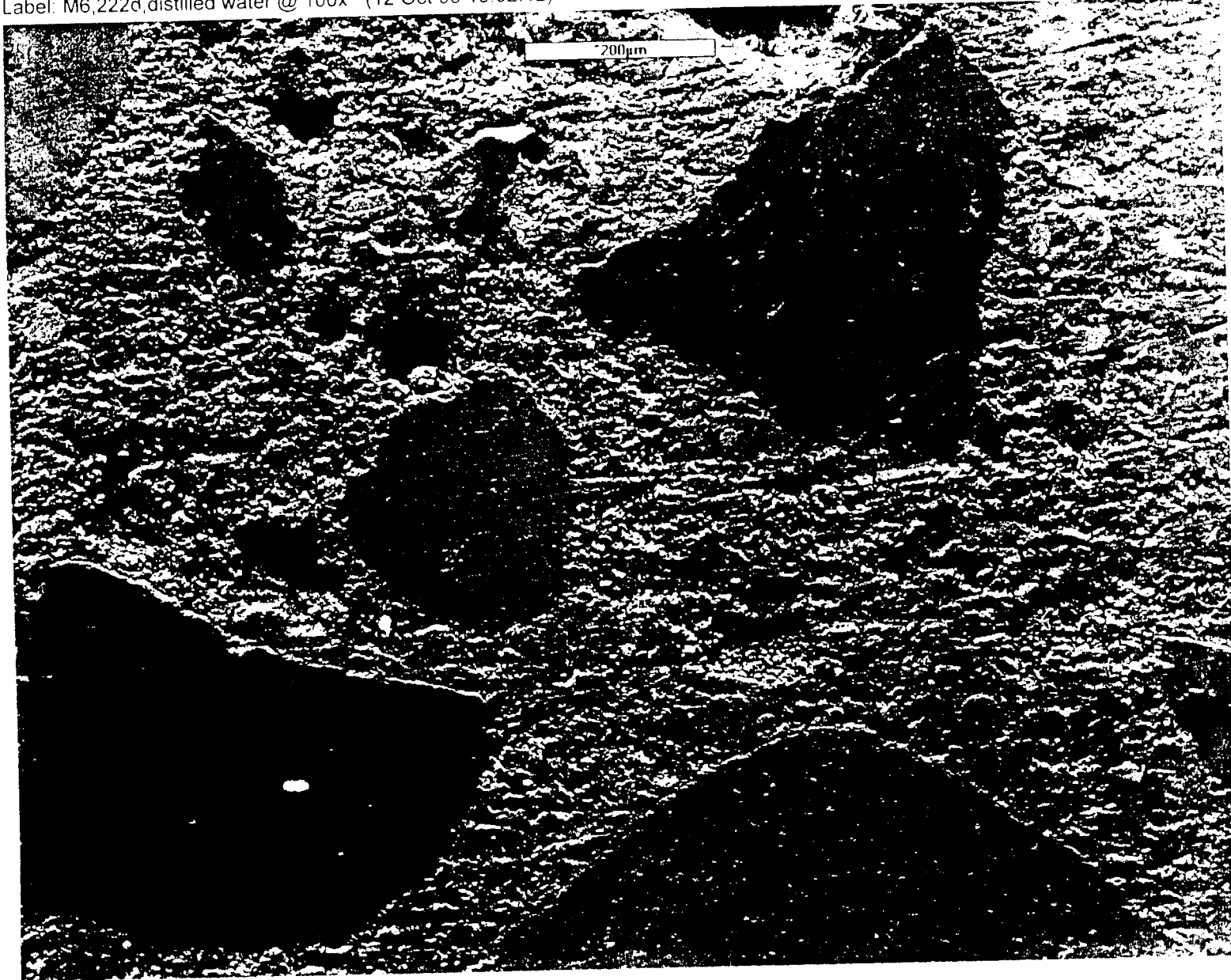


Fig. 14a SEM Photograph for Mix 6 with the wet/dry Treatment

Operator: Jerry Amenson

Client: Cheng

Job: Iowa DOT

Label: M6,222d,dist water @ 100x m (12 Oct 95 13:25:51)

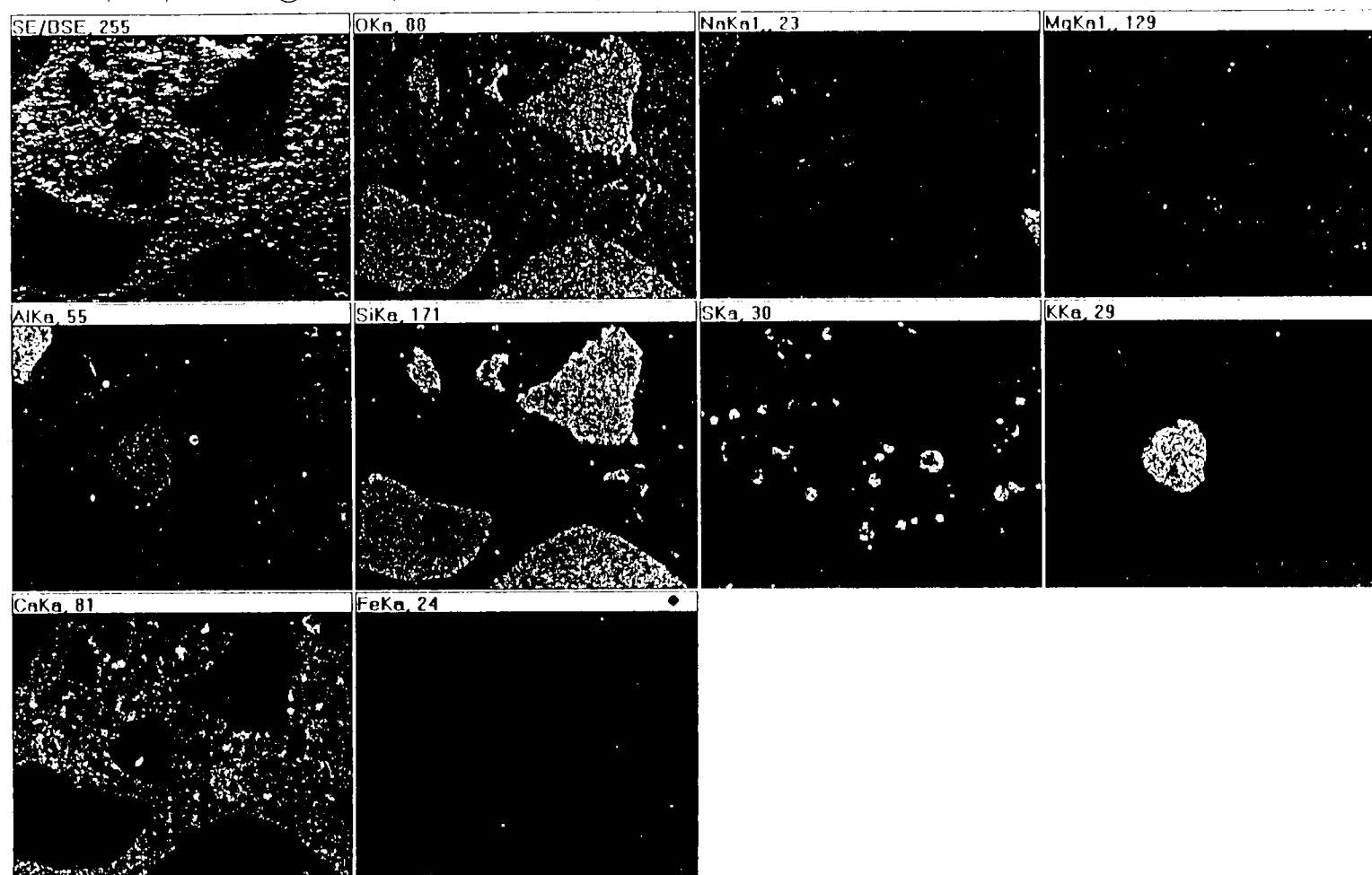


Fig. 14b Elemental Mapping of Fig. 14a

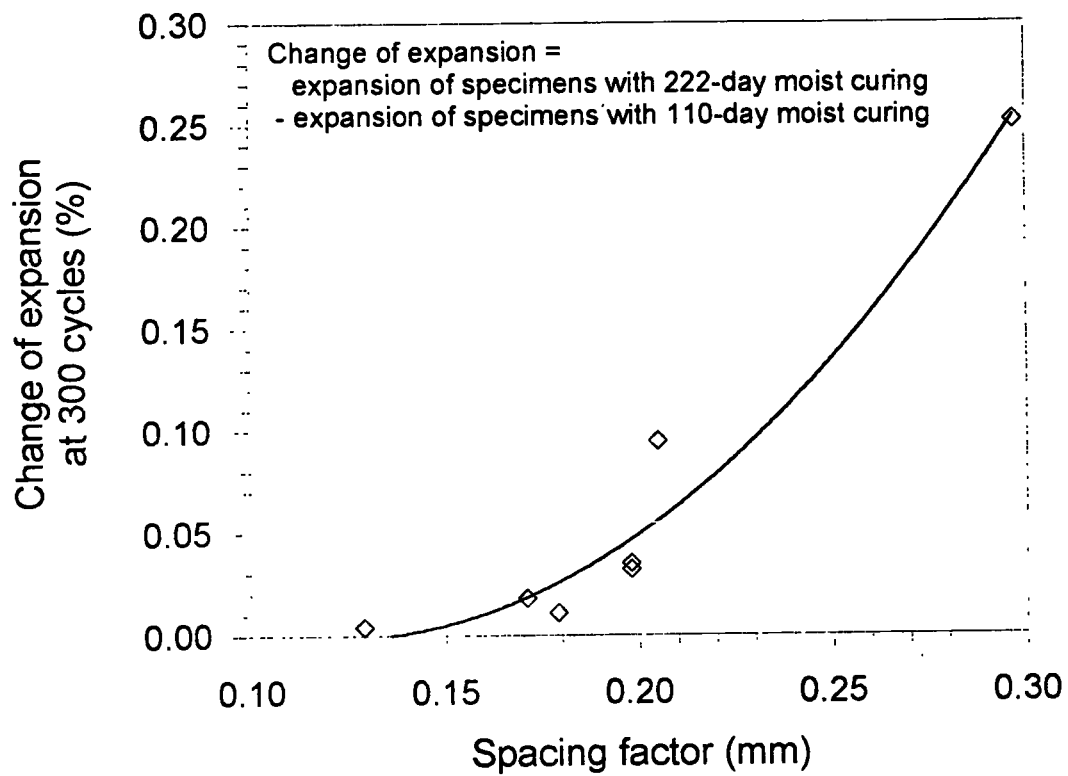


Fig. 15 Relationship between change of expansion and spacing factors
(the spacing factor measured by Lafarge)

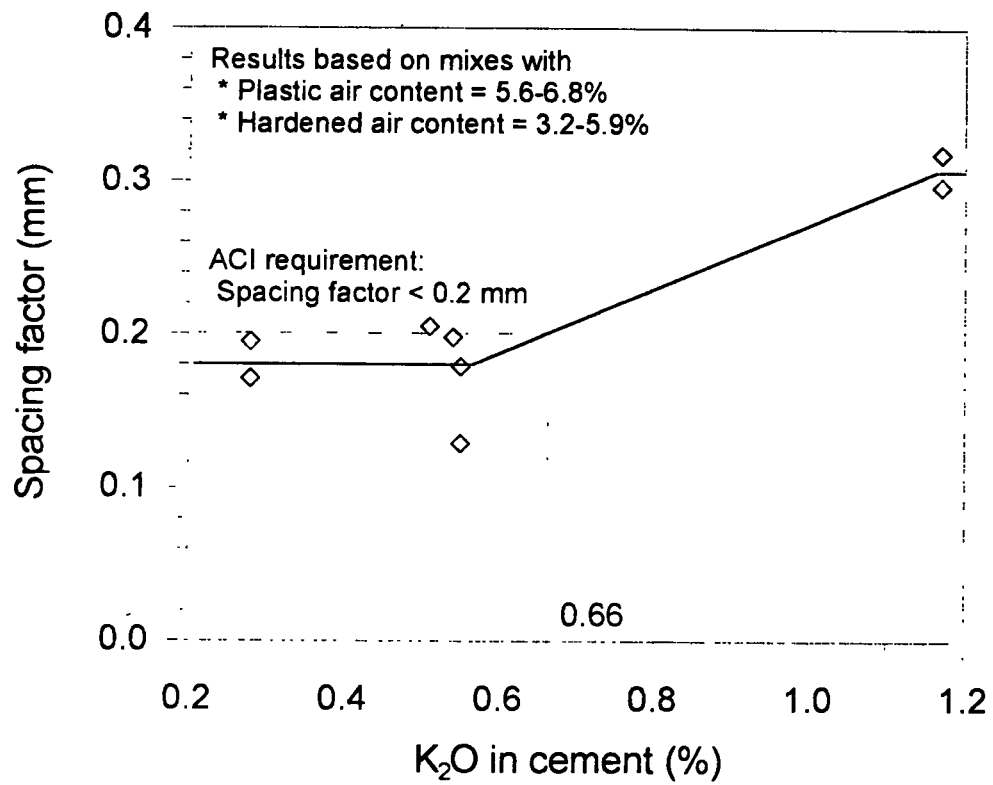


Fig. 16 Relationship between K₂O in cement and spacing factors
(the spacing factor measured by Lafarge)

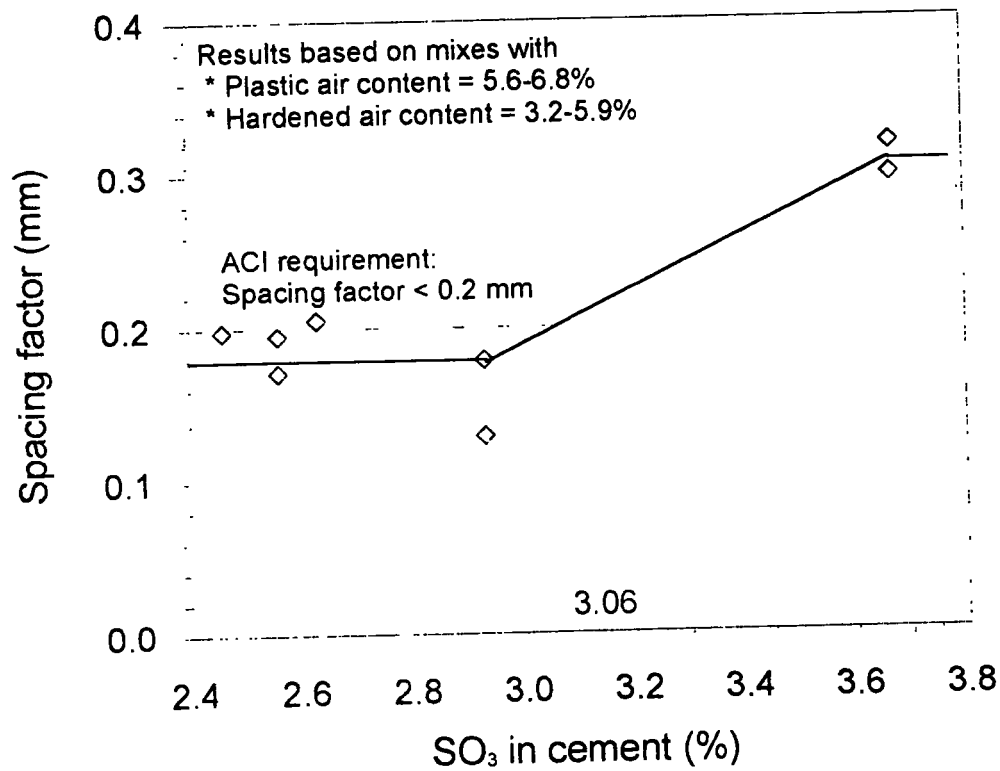


Fig. 17 Relationship between SO₃ in cement and spacing factors
(the spacing factor measured by Lafarge)

APPENDIX I

Supplemental Results on Freeze-Thaw Durability of Concrete Mixes with the Wet/Dry Treatment

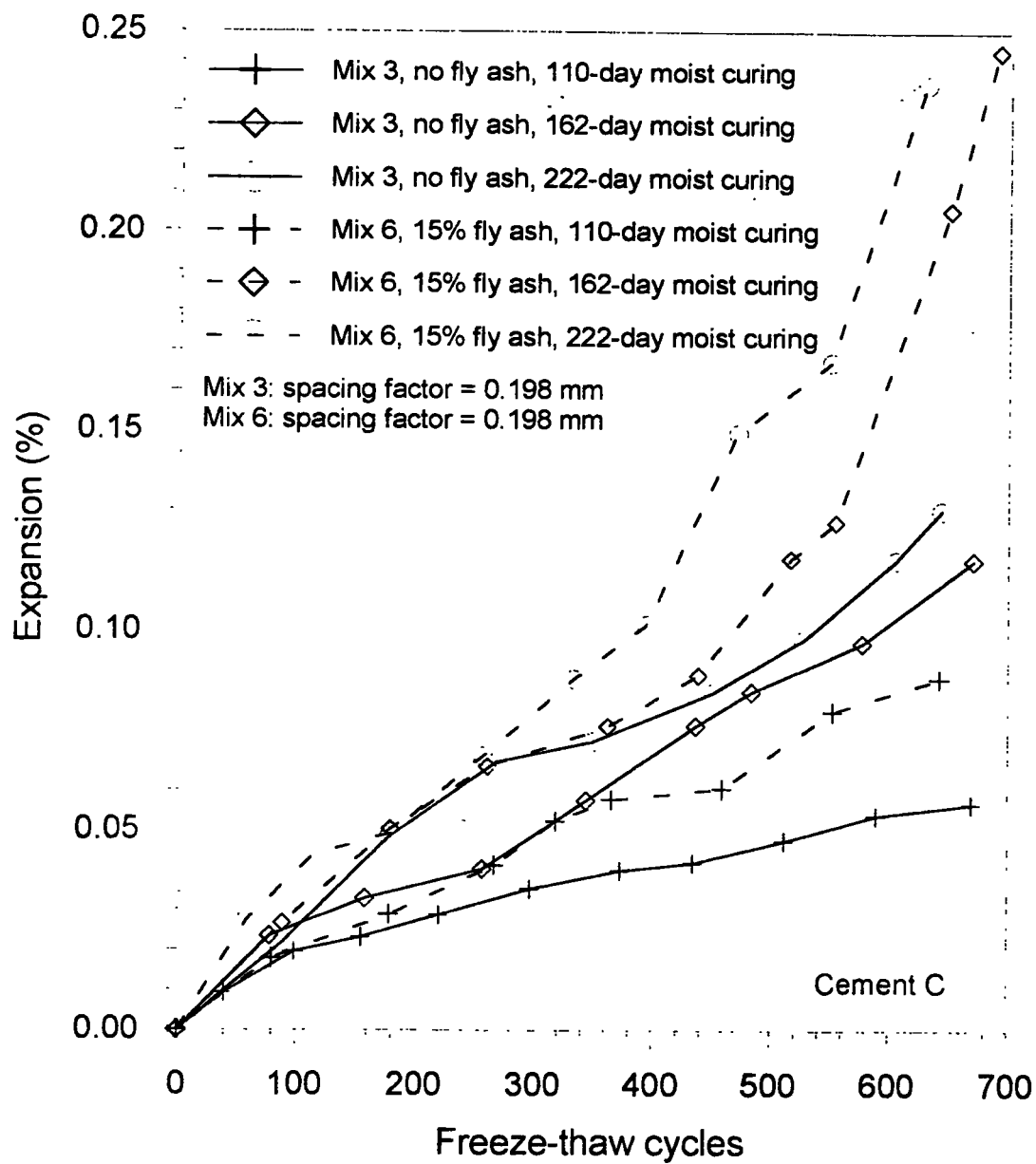


Fig. I-1 Freeze-thaw expansion of mixes 3 and 6

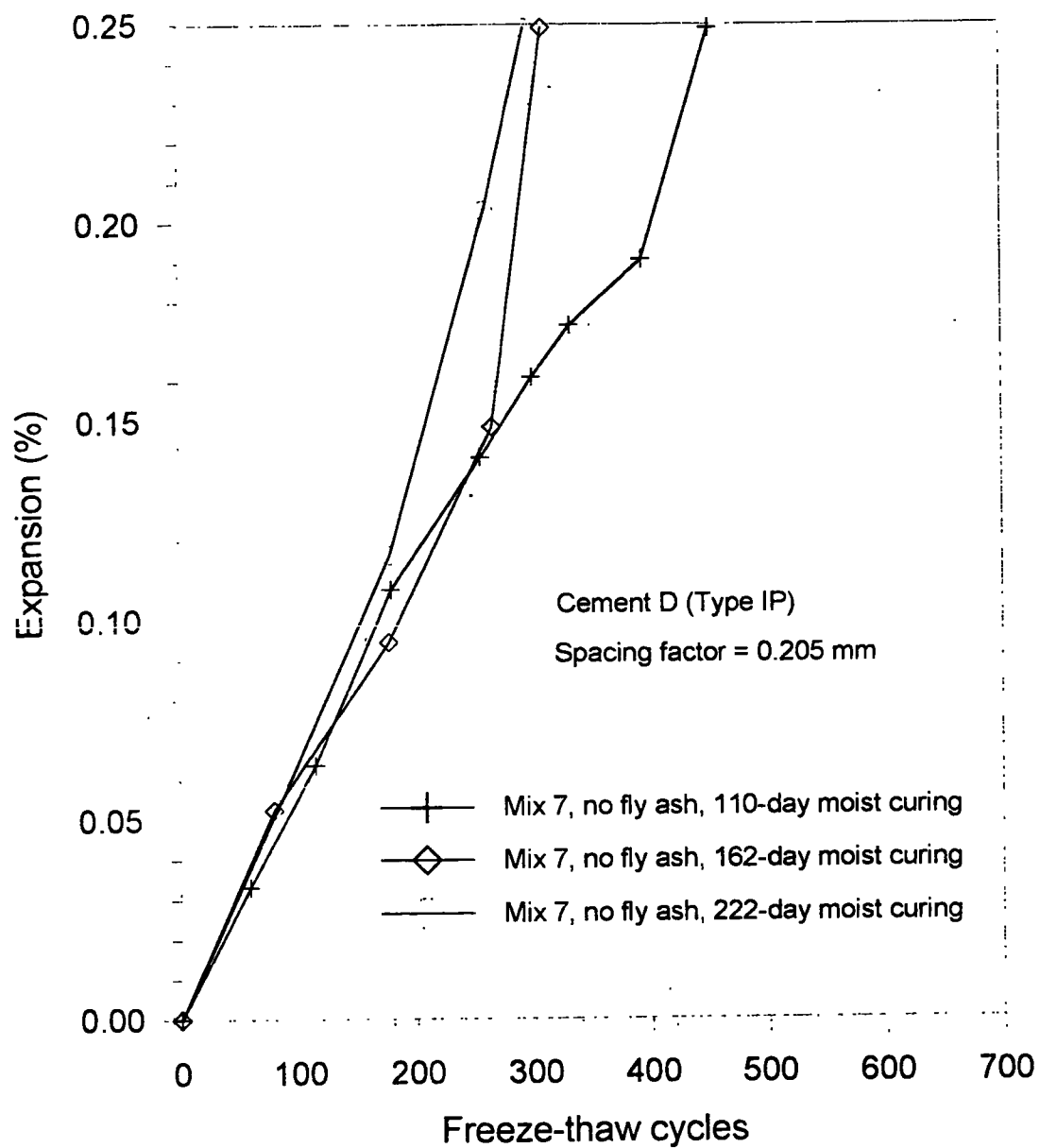


Fig. I-2 Freeze-thaw expansion of mix 7

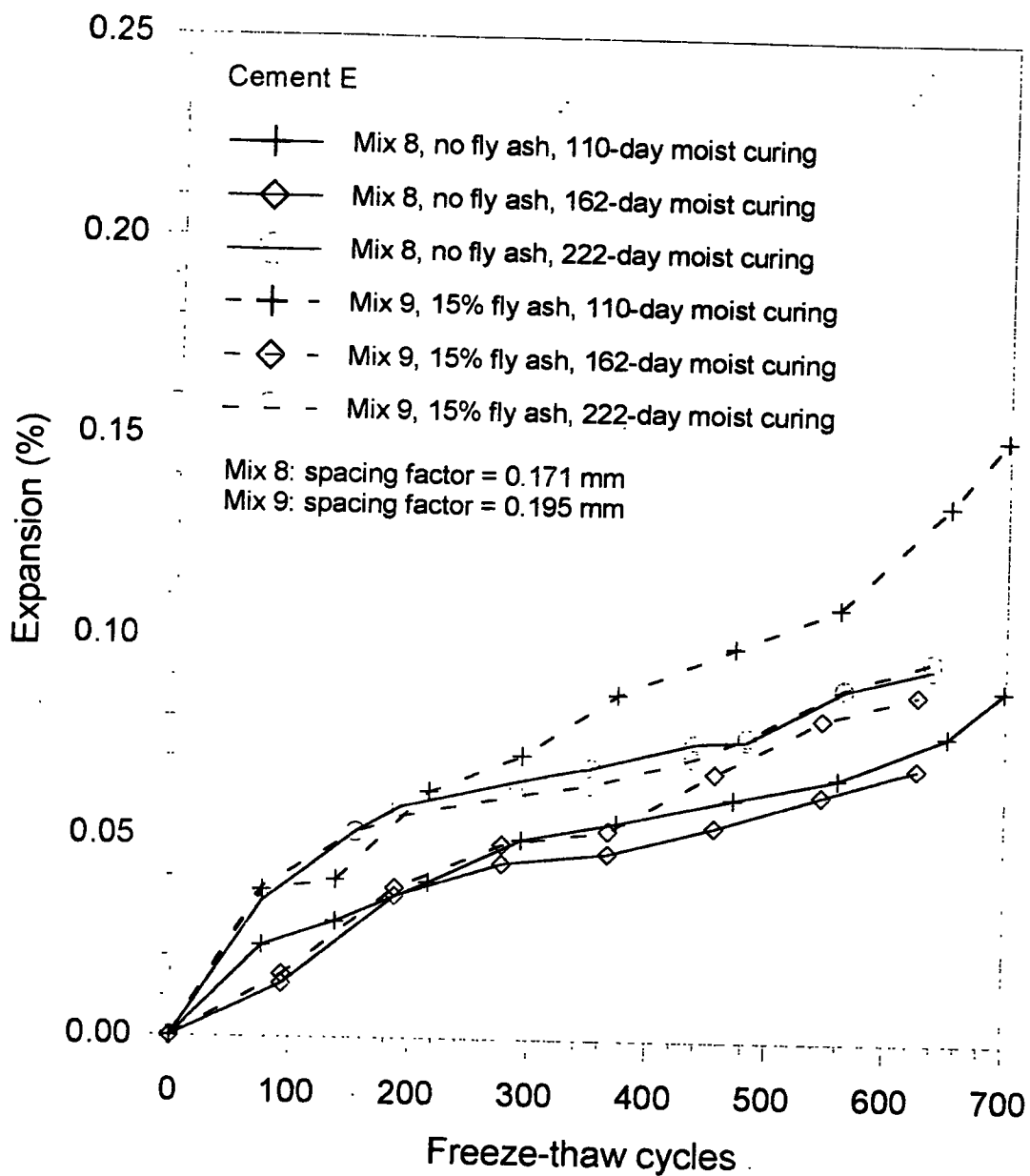


Fig. I-3 Freeze-thaw expansion of mixes 8 and 9

APPENDIX II

Supplemental Results on Freeze-Thaw Durability of Concrete Mixes without the Wet/Dry Treatment

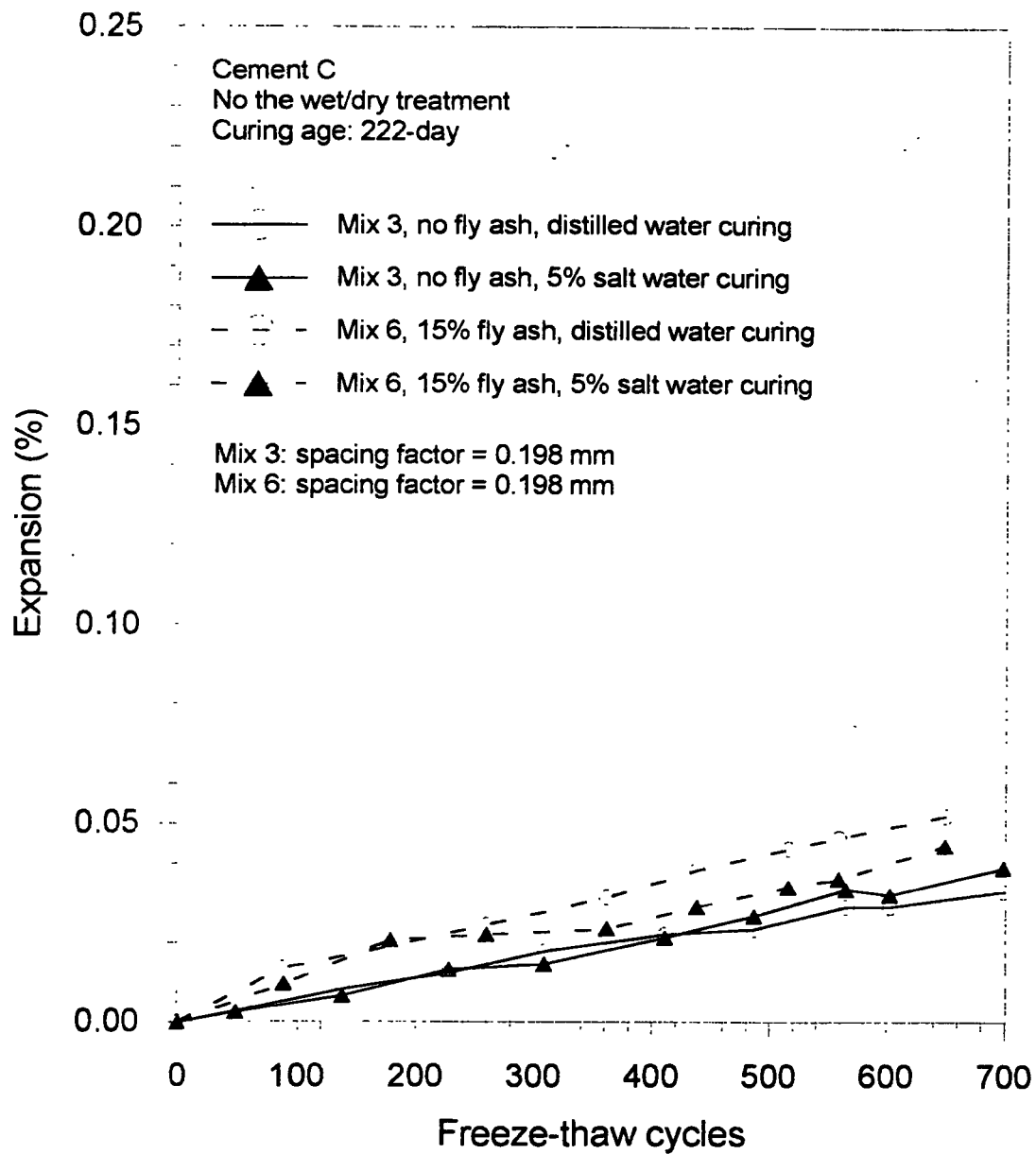


Fig. II-1 Freeze-thaw expansion of mixes 3 and 6, no the wet/dry treatment

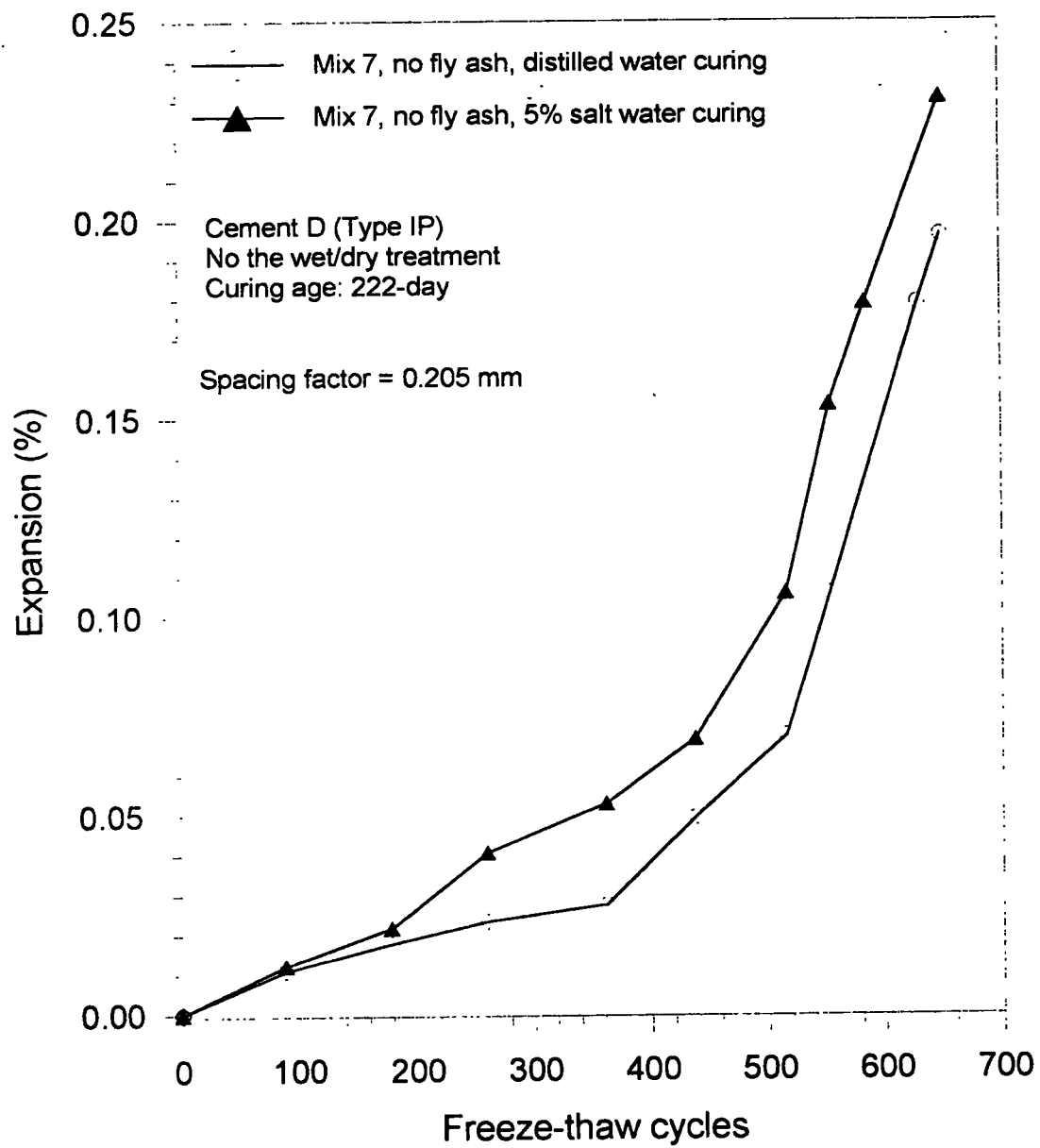


Fig. II-2 Freeze-thaw expansion of mix 7, no the wet/dry treatment

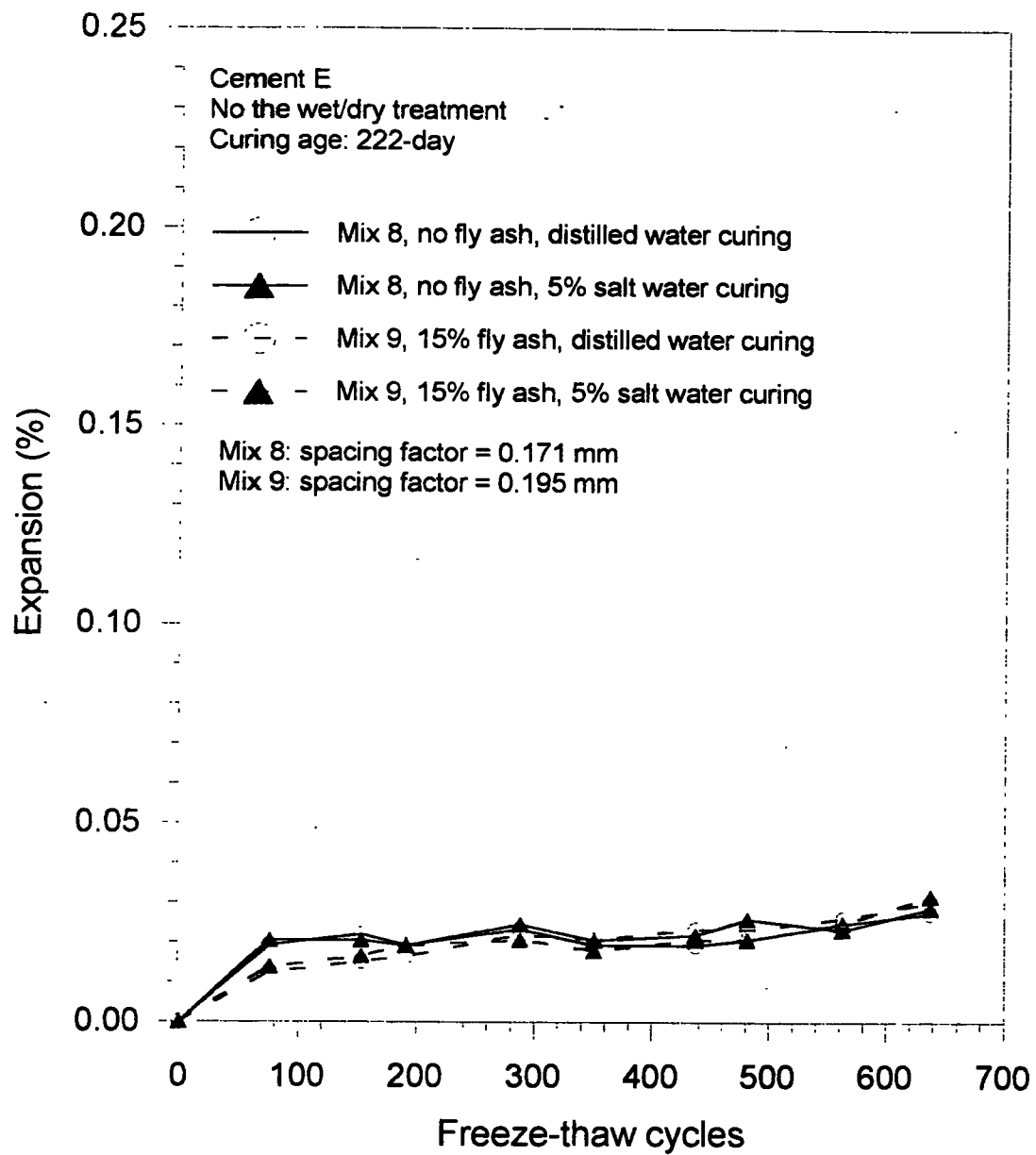


Fig. II-3 Freeze-thaw expansion of mixes 8 and 9; no the wet/dry treatment