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PREPRINT

**A DIFFERENT PERSPECTIVE
FOR INVESTIGATION OF
PCC PAVEMENT DETERIORATION**

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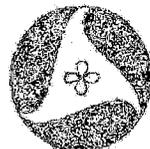
by
Vernon J. Marks
and
Wendell G. Dubberke

Interim Report
for
Iowa DOT
Research Project HR-2074

For Presentation at the
Transportation Research Board
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Project Development Division

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A Different Perspective
for Investigation of
PCC Pavement Deterioration

Interim Report
for
Iowa DOT
Research Project HR-2074

Vernon J. Marks
Research Engineer
515-239-1447
Office of Materials
Project Development Division
Iowa Department of Transportation
Ames, Iowa 50010

and

Wendell G. Dubberke
Geologist
515-239-1339
Office of Materials
Project Development Division
Iowa Department of Transportation
Ames, Iowa 50010

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DISCLAIMER

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

ABSTRACT

Many early Iowa Portland Cement Concrete (PCC) pavements provided good performance without deterioration for more than 50 years. In the late 1950's, Iowa was faced with severe PCC pavement deterioration called D cracking due to crushed limestone containing a bad pore system. Selective quarrying solved the problem.

In 1990, cracking deterioration was identified on a three year old US 20 pavement in central Iowa. The coarse aggregate was a crushed limestone with an excellent history of performance in PCC pavement. Examination of cores showed very few cracks through the coarse aggregate particles. The cracks were predominately confined to the matrix. A high resolution, low vacuum Hitachi Scanning Electron Microscope (SEM) with an energy dispersion detector was used to investigate the deterioration. Subsequent evaluation identified very little concentration of silica gel (silicon-Si), but did identify substantial amounts of sulfur-S and aluminum-Al (assumed to be ettringite) in the air voids. Some of these voids have cracks radiating from them leading us to conclude that the ettringite filled voids were a center of pressure causing the crack. The ettringite in the voids, after being subjected to sodium chloride (NaCl) brine, initially swells and then dissolves.

The research has led to the conclusion that the premature deterioration may be due to ettringite and may have been mistakenly identified as Alkali-Silica reactivity (ASR).

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INTRODUCTION

In the late 1950's, Iowa was faced with severe PCC pavement deterioration. Some pavements that were only 15 years old were exhibiting a substantial crack pattern. This deterioration was referred to as D cracking (Figure 1). After substantial research, the cause of this deterioration was related to a certain type of carbonate coarse aggregate. The initiating cause was identified as crushed limestone aggregate containing a predominance and substantial volume of pores in the 0.04 to 0.2 μm diameter pore size. Since the 1930's, Iowa has maintained an excellent record of the ledges and quarry locations of crushed stone used in Iowa PCC pavement and their performance by time to visible deterioration. The D cracking problem was solved by identifying the ledge or ledges in each quarry that would cause D cracking and prohibiting their use in Iowa Primary pavement. The Iowa limestone producers use selective quarrying and bench their operation to remove the undesirable ledges.

A TYPE OF PCC PAVEMENT DETERIORATION NEW TO IOWA

In 1990, the Iowa DOT encountered a very challenging problem. A ten mile section of new four-lane divided highway in central Iowa (US 20 from Fort Dodge to Webster City) was exhibiting distress cracking (Figure 2) at an age of only three years. In other

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premature PCC pavement failures, the primary factor relating to the deterioration was the coarse aggregate. In this case, the coarse aggregate was a crushed limestone (Gilmore City Ledge) from the Fort Dodge Mine that had an excellent performance record when used in PCC pavement. An examination of cores from the pavement and particularly one adjacent to the joint (Figure 3) revealed very few cracks passing through the coarse aggregate particles. The cracks were predominately confined to the matrix.

During construction of this pavement the contractor had made a number of changes in materials sources for the concrete mixture (Table 1 & Table 2). This provided six different PCC material combinations. These included two cement sources, three fly ash sources and two fine aggregate sources with the Fort Dodge coarse aggregate being used for the entire project. Two combinations of Lehigh cement with 15% Class C fly ashes exhibited severe distress.

There were a number of Iowa DOT personnel with substantial experience in evaluating PCC pavement performance and in identifying distress mechanisms that participated in the investigation (beginning in 1991) of this type of pavement deterioration which was new to Iowa. No consensus as to the

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cause of deterioration was reached. In 1992, a noted concrete consultant was retained to identify the cause of the US 20 deterioration. After an investigation, the consultant concluded in September 1992 that the cause of deterioration was ASR (1).

The Fort Dodge limestone is a very high quality limestone and does not contain siliceous material that would have potential to cause ASR. The consultant that identified the distress as being caused by ASR cited the shale particles in the fine aggregate as the reactive siliceous material. The two fine aggregates were natural sands with similar mineralogy. The Croft sand was an upland deposit with approximately two and one-half percent shale particles. The Yates sand is a river deposit with approximately one-quarter percent of the shale particles. The pavement with the Yates sand exhibited visible deterioration cracking in three years and severe map cracking in five years. We do not believe that one-quarter percent of the fine aggregate is sufficient to produce visible ASR cracking in three years nor severe deterioration in five years.

The Iowa DOT PCC pavement investigation team could not accept ASR as the initiating cause of the premature distress for a number of reasons. Even the noted concrete consultant did not identify any

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reactive silica in the crushed carbonate coarse aggregate. The fine aggregate, even though it has some chert and opaline shale particles, has provided very good performance history. The shale particles were identified as the major source of reactive silica that produced the expansive silica gel. Those shale particles will expand out of a cut surface very quickly after being wet diamond sawed. All central Iowa sands contain these shale particles and experience has shown that there is very little activity of these shale particles until they are cut and exposed on a surface. Shale particles tend to move up during PCC consolidation. There are a few, but for the most part, there are almost no shale popouts in the surface, even in the two severely distressed materials combinations of US 20. The Yates fine aggregate with approximately 0.25% shale particles exhibited distress cracking at three years of age (too fast for ASR) and essentially the same rate of deterioration as the Croft fine aggregate with approximately 2.5% shale particles. If the shale particles are the source of the expansive silica gel, the Croft sections should have a much more rapid rate of deterioration than the Yates sections.

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Thirty representative Iowa sands were subjected to the ASTM P214 ASR test. Based on the resulting expansions, only two were innocuous, 17 were inconclusive and 11 were potentially reactive. The current Portland Cement Association (PCA) guidelines for identifying potentially ASR aggregate is to first test them using ASTM C1260 (adopted from P214) and if they result in expansions greater than 0.10% subject them to the Canadian Prism Test. A very high concentration of sodium hydroxide is mixed into the plastic PCC mixture in the Canadian Prism Test. The two Iowa sands with the greatest P214 expansions were subjected to the Canadian Prism Test. The resulting expansions were well below the 0.04% that would identify them as reactive. The Croft and Yates sands have just recently been subjected to the Canadian Prism Test and meaningful expansion data is not yet available.

EVALUATION OF PCC PAVEMENT DETERIORATION USING NEW TECHNOLOGY

Wendell Dubberke of the Iowa DOT and Scott Schlorholtz of Iowa State University (ISU) conducted an evaluation of the US 20 concrete using an ISU high-vacuum scanning electron microscope (SEM). Unfortunately, the high-vacuum would cause cracks in the specimen and there was concern that vacuum induced cracks might distort the findings.

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On March 1, 1993 the Iowa DOT and ISU entered into a contract to conduct research project HR-358, "Evaluation of Microcracking and Chemical Deterioration in Concrete Pavement." The principal investigator of HR-358 is Scott Schlorholtz. Under HR-358 the Iowa DOT and ISU jointly purchased a low-vacuum, high-resolution Hitachi SEM with a germanium energy dispersion detector. It took substantial time for ordering and delivery of the SEM. Delivery was late and there were many issues requiring time to be resolved. The project finally began to progress about July 1994.

The current evaluation procedure is to obtain backscatter SEM, photomicrographs at 30X and 100X. Energy dispersion mapping plots of sodium, potassium, silicon, calcium, sulfur and aluminum are obtained on the same area at the same time of both the 30X and 100X micrographs. The germanium detector is top-of-the line and does an excellent job of identifying the elements present on the sawed and polished surfaces. The investigation has evaluated PCC concrete samples from more than 20 US 20 cores, 10 cores from other Iowa premature distress pavements and cores from six other states. Samples from the top, middle and bottom of the cores were evaluated.

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WHITISH MATERIAL IN THE PCC AIR VOIDS

Iowa US 20 Webster County Project

Examination of a sawed and polished face of PCC from the US 20 pavement (with a light microscope) identified a whitish material in many of the air voids. This whitish material may have been mistakenly identified as silica gel by the consultant, leading to the conclusion that the US 20 deterioration was ASR.

This same sawed and polished piece of PCC was then observed using the Hitachi low vacuum SEM. A backscattered electron (BSE) photomicrograph of round air voids was obtained (Figure 4). The SEM identified smaller voids nearly filled and larger voids partially filled with the whitish material. Distress cracks passed through a series of these air voids.

The alkali in silica gel has been determined to be either sodium or potassium or a combination of sodium and potassium (2). This alkali combines with silicon and oxygen to produce silica gel. Elemental maps were obtained on the US 20 polished sample for sodium, potassium and silicon. In the elemental mapping a dark area would indicate very little of an element was present while a very white area would identify a concentration of the element. The particular interest in this case was "what elements are present in the whitish material filling the air voids?" An energy dispersion spectrum of the whitish material in the air

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voids was obtained (Figure 5). This spectrum showed major peaks for oxygen, aluminum, sulfur and calcium, supporting the conclusion that the whitish material in the voids is ettringite. There is a minor silicon peak, but no sodium or potassium peak; thereby, supporting the finding that the material in the void is not silica gel.

Samples were taken and evaluated from cores from all six US 20 materials combinations. All showed substantial ettringite in the air voids and silica gel only being formed in the shale particle locations.

The SEM also identified cracks radiating from some of these air voids (Figure 6). This would indicate that this air void was a center-of-pressure in the development of these cracks. There was another interesting feature of the ettringite in these voids with the radiating cracks. The ettringite was filling these voids, but the ettringite did not fill the crack inside or outside of the round voids. This led to the conclusion that the void was filled with ettringite before the cracks developed.

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The elemental maps (Figure 7) showed little or no silicon in the filled air voids. The round air voids could easily be identified because they were black on the silicon elemental map, showing that there was almost no silicon in the whitish material in the air voids and substantially more silicon in the PCC surrounding the air voids. This led to the conclusion that the whitish material in the air voids is not silica gel.

Elemental maps were obtained for 11 different elements (only a selected 5 are shown in Figure 7). The elemental map that outlined the round air voids the best was sulfur-S. In the map for sulfur, the air voids were very white showing a high concentration of sulfur in the whitish material in the air voids. The aluminum map also showed whiter than the surrounding PCC. The round air voids could also be identified in the calcium map. There was substantial calcium present in both the filled air voids and the surrounding matrix, but the whitish material in the filled air voids had less calcium than the surrounding matrix. These maps led to the conclusion that the whitish material in the filled air voids was very likely ettringite.

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Nebraska I-80 Aurora Project

Core samples were obtained from two Nebraska locations where premature deterioration similar to Iowa's had been identified. The deterioration at both of these locations (I-80 at Aurora (3) and the Omaha Airport) had also been identified by others as ASR. The aggregates at both of these locations contained sodium-potassium feldspars that others identified as slightly to moderately reactive in regard to ASR. Samples from both of these sites contained the whitish material in the filled air voids.

An investigation of the I-80 Aurora PCC (Figures 8 & 9) shows a high concentration of sulfur and very little silicon, again leading to the conclusion that the material is ettringite and not silica gel. An interesting note to make on the I-80 Aurora SEM data is that some of the air voids are in close proximity to these so called ASR reactive aggregates. If these reactive aggregate particles are producing silica gel, it would be a reasonable assumption that some of this expansive gel would be forced into the voids in close proximity. No concentration of silicon has been identified in these filled air voids.

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Nebraska Omaha Airport

The data from the Omaha Airport is very interesting for a couple of other reasons. First, for those who may question the capability of the energy dispersion detector to properly identify elements, a sodium-potassium feldspar aggregate will demonstrate the accuracy of the data. The photomicrograph using the backscattered detector (Figure 10) shows darker and lighter portions of the large aggregate in the left side of the picture. This is due to differences in atomic weight. In the elemental maps (Figure 11), the dark portions are shown to be sodium feldspar while the light portions are shown to be potassium feldspar.

There was also a rim deposit around an aggregate particle (Figure 10). Some petrographic researchers may identify this rim as silica gel. The elemental maps of Figure 11 show that this rim does not contain silicon, so it is not silica gel. The rim material contains substantial sulfur and is, therefore, probably ettringite.

The material in the Omaha Airport voids contains substantial sulfur, but almost no silicon, so it again is not silica gel, but is very likely ettringite.

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GROWTH OF ETTRINGITE IN THE LABORATORY

Additional research was conducted in the Iowa DOT Materials Laboratory to investigate the formation of ettringite in air voids. Concrete beams were made and left in the molds for approximately 24 hours. After removal from the molds, they were stored in a 100% humidity moist room at approximately 21°C (70°F). They are still being stored in the moist room. Slices are periodically sliced from the end of the beam. These slices were examined using the low vacuum SEM. At an age of two months, Mix #1 showed ettringite nodules either growing in the air voids or being extruded into the air voids from the capillary pore system. At an age of seven months, Mixes #2 & 3 exhibited ettringite nodules in the air voids. These ettringite nodules in the air voids developed in an environment where it would be almost impossible to have evaporation or to have an external source of sulfur.

INITIAL EXPANSION OF ETTRINGITE WHEN SUBJECTED TO NaCl BRINE

Canadian research at the University of Manitoba had shown that ettringite would dissolve in the presence of chloride ions (4). In an effort to substantiate this in regard to the ettringite in the voids, a polished PCC surface with large, tightly filled voids was selected. A photomicrograph and elemental maps were

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recorded prior to treatment with NaCl brine (Figure 12). The elemental maps (Figure 13) exhibited no silicon, but substantial sulfur and aluminum indicating that the material in the air voids was very likely ettringite. The NaCl treated, polished sample was put into the low vacuum SEM. After 45 minutes the ettringite in the large voids had either been dissolved or dislodged (Figure 14). Another very interesting finding was that the ettringite in the small voids had swelled and expanded. At 48 hours, the ettringite had dissolved from all of the air voids, but the expansion of the ettringite had been sufficient to lift the boundaries of the air voids (Figure 15). Another significant feature that became visible was the substantial cracking in the large air voids and the cracking through a series of air voids.

The SEM data shows that when PCC is soaked in a NaCl brine, ettringite will initially expand and then dissolve or be dislodged. Duggan cores (5) 25 mm (1 in.) in diameter and 51 mm (2 in.) long were drilled from the laboratory prepared PCC that had been stored in the moist room. This PCC had exhibited ettringite either growing in or being extruded into the air voids as described earlier in this paper. These four cores had exhibited a relatively constant rate of expansion through 50 days during a distilled water soak (Figure 16). At 50 days after

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pretreatment, the four cores were changed to a NaCl brine soak. From 50 to 60 days all four cores exhibited an increased rate of expansion. After 60 days there was very little expansion indicating that the expansive mechanism had been destroyed (or dissolved). There is speculation that the expansive pressure was due to ettringite through 50 days. When subjected to NaCl at 50 days, there was increased expansion of the ettringite. The ettringite then dissolved and there was very little additional expansion. Other researchers have identified ettringite filled or partially filled voids in deteriorated PCC pavement (6) and one of those has concluded it is the cause of concrete deterioration (7, 8, 9, 10, 11).

RELATED FINDINGS AND SPECULATION

Based primarily on the research contained in this report, there seems to be little data that would support ASR as the initiating cause of the deterioration. Substantial filling of air voids with ettringite from top to bottom of the PCC slab, even at midpanel, supports ettringite as the initiating cause. The most likely source of the sulfur and aluminum to produce the ettringite after initial set of the concrete is the cementitious materials. We speculate that the ettringite fills the capillary pore system and then extrudes into the air voids of the concrete.

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Other researchers contend that expansive ettringite is sufficient to induce deterioration cracking (11). They also contend that it can happen without a freeze/thaw environment. On US 20, we believe that freeze/thaw cycles accelerate the deterioration process.

Field reviews and laboratory investigations have identified excessive consolidation during PCC placement, the presence of deicing chemical and water freeze/thaw damage as contributing factors.

CONCLUSIONS

This low vacuum SEM research of PCC pavement deterioration supports the following conclusions:

1. There may be instances today where premature PCC pavement deterioration has been mistakenly identified as ASR.
2. The deterioration of some current premature PCC pavement distress locations may be caused by factors related to the formation of excessive ettringite.

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ACKNOWLEDGEMENTS

The authors wish to express their appreciation to a number of people who made substantial contribution to this paper. Much of the SEM investigation has been conducted by Scott Schlorholtz and Jerry Amenson of Iowa State University. In addition to the authors, the Iowa DOT PCC pavement investigation team included Bernard C. Brown, Jerry Bergren, John Lane, Jim Grove, Champ Narotam, Jim Myers and Chen Ouyang. Kathy Davis, Bob Steffes and Shane Tymkowicz made substantial contribution in the preparation of the paper. Gordon Smith of the Iowa Concrete Paving Association has been very supportive of this research.

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TABLE TITLES

1. Mix Proportions for an Iowa DOT C3WRC PC Concrete
2. Material Combinations on US 20

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TABLE 1 Mix Proportions for an Iowa DOT C3WRC PC Concrete

Cement	233 kg	(513 lbs)
Fly Ash (Class C)	41 kg	(91 lbs)
Coarse Aggregate (Ft. Dodge)	755 kg	(1664 lbs)
Fine Aggregate	613 kg	(1352 lbs)
Water (approximate)	125 kg	(276 lbs)
Water Reducer (plastocrete 161)	510 g	(18 oz)

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TABLE 2 Material Combinations on US 20

<u>MIX</u>	<u>CEMENT</u>	<u>FLY ASH</u>	<u>FINE AGGREGATE</u>	<u>DISTRESS</u>
1	Lehigh	Ottumwa	Croft	Severe
2	Lehigh	None	Croft	None
3	Lehigh	Port Neal #4	Yates	Severe
4	Northwestern	Ottumwa & Port Neal #4	Croft	Minor
5	Northwestern	Nebraska City	Croft	None
6	Northwestern	Nebraska City & Port Neal #4	Croft	Minor

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FIGURE CAPTIONS

1. Typical D Cracking of PCC Pavement
2. The Cracking Pattern of US 20 in Central Iowa
3. A US 20 Core Showing Few Cracks in the Coarse Aggregate
4. A 200X BSE Photomicrograph of the PCC Air Voids on Iowa US 20
5. An Energy Dispersion Spectrum of the Whitish Material in the Air Voids
6. A 100X BSE Photomicrograph of Iowa US 20 Showing Cracks Radiating from the Ettringite Filled Air Voids
7. The Elemental Maps for the Figure 6 Photomicrograph
8. A 100X BSE Photomicrograph of a Nebraska I-80 Aurora PCC Sample
9. Elemental Maps for the Figure 8 Photomicrograph
10. A 100X BSE Photomicrograph of a Nebraska Omaha Airport PCC Sample
11. Elemental Maps for the Figure 10 Photomicrograph
12. A 100X BSE Photomicrograph of Air Voids Tightly Filled with Ettringite
13. Elemental Maps for the Figure 12 Photomicrograph
14. A 100X BSE Photomicrograph of the Figure 12 Air Voids 45 Minutes After Treatment with NaCl Brine.
15. A 100X BSE Photomicrograph of the Figure 12 Air Voids 48 Hours After Treatment with NaCl Brine.
16. Duggan Test Results with Sodium Chloride Brine Modification

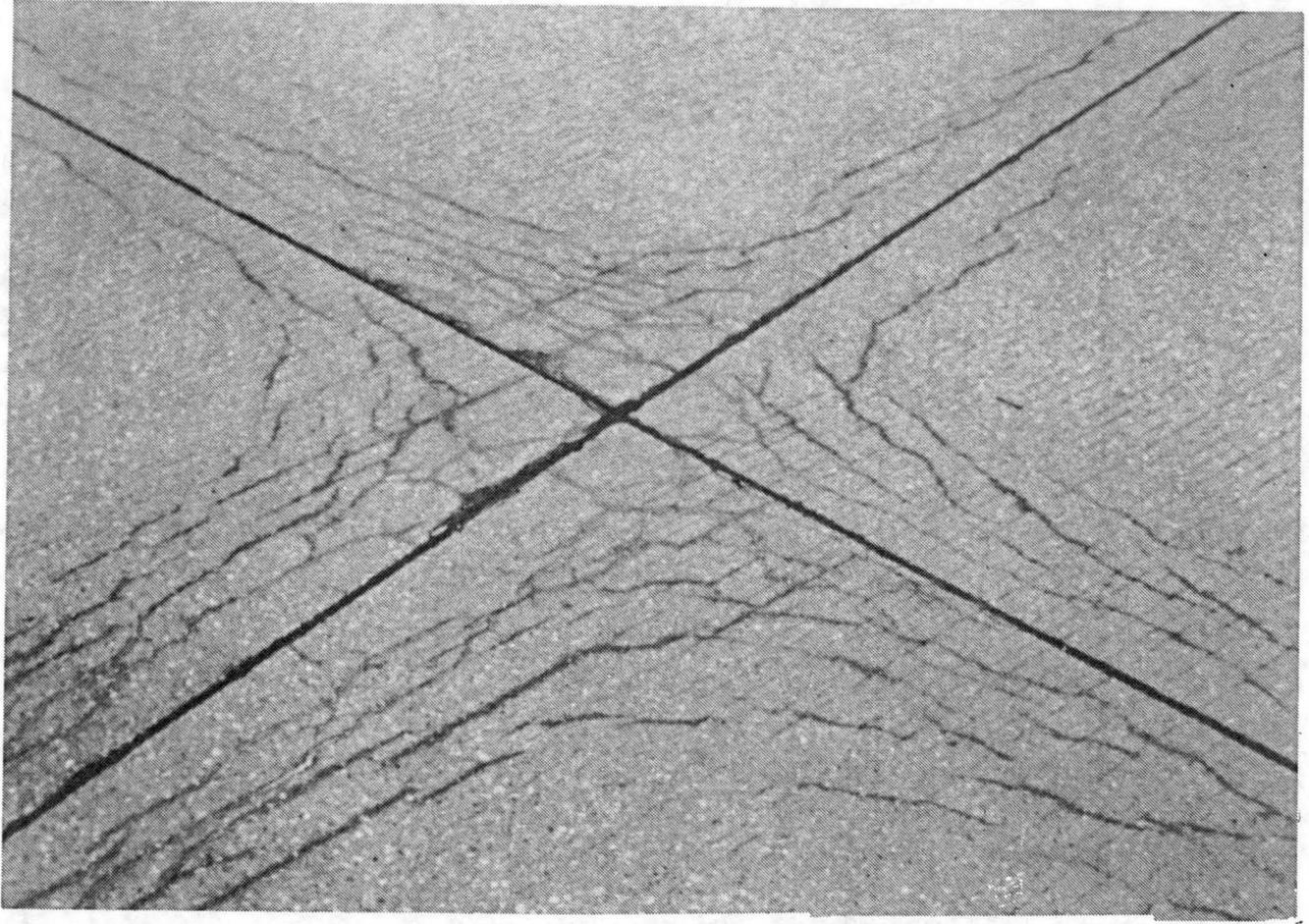


FIGURE 1 TYPICAL D CRACKING OF PCC PAVEMENT

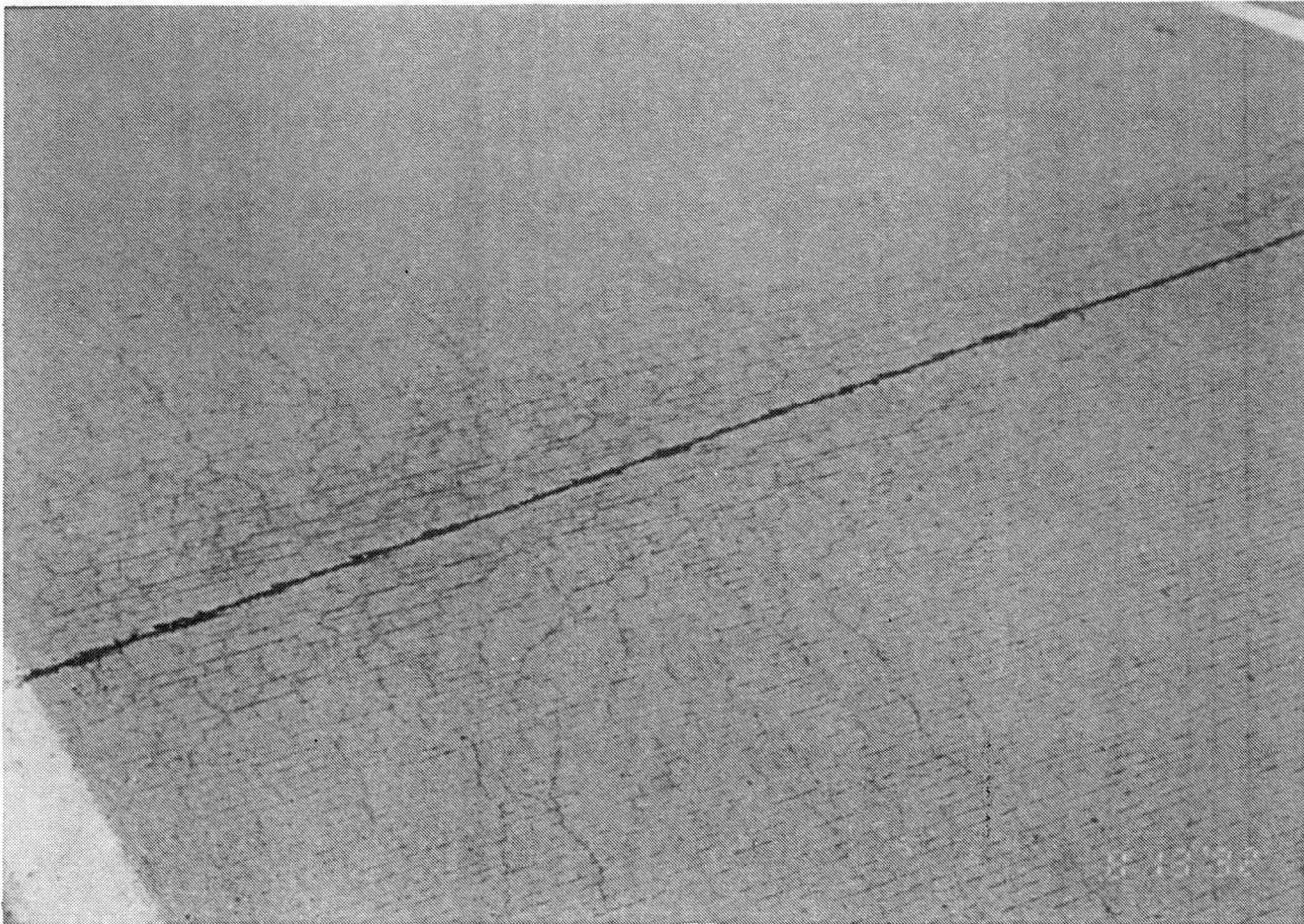
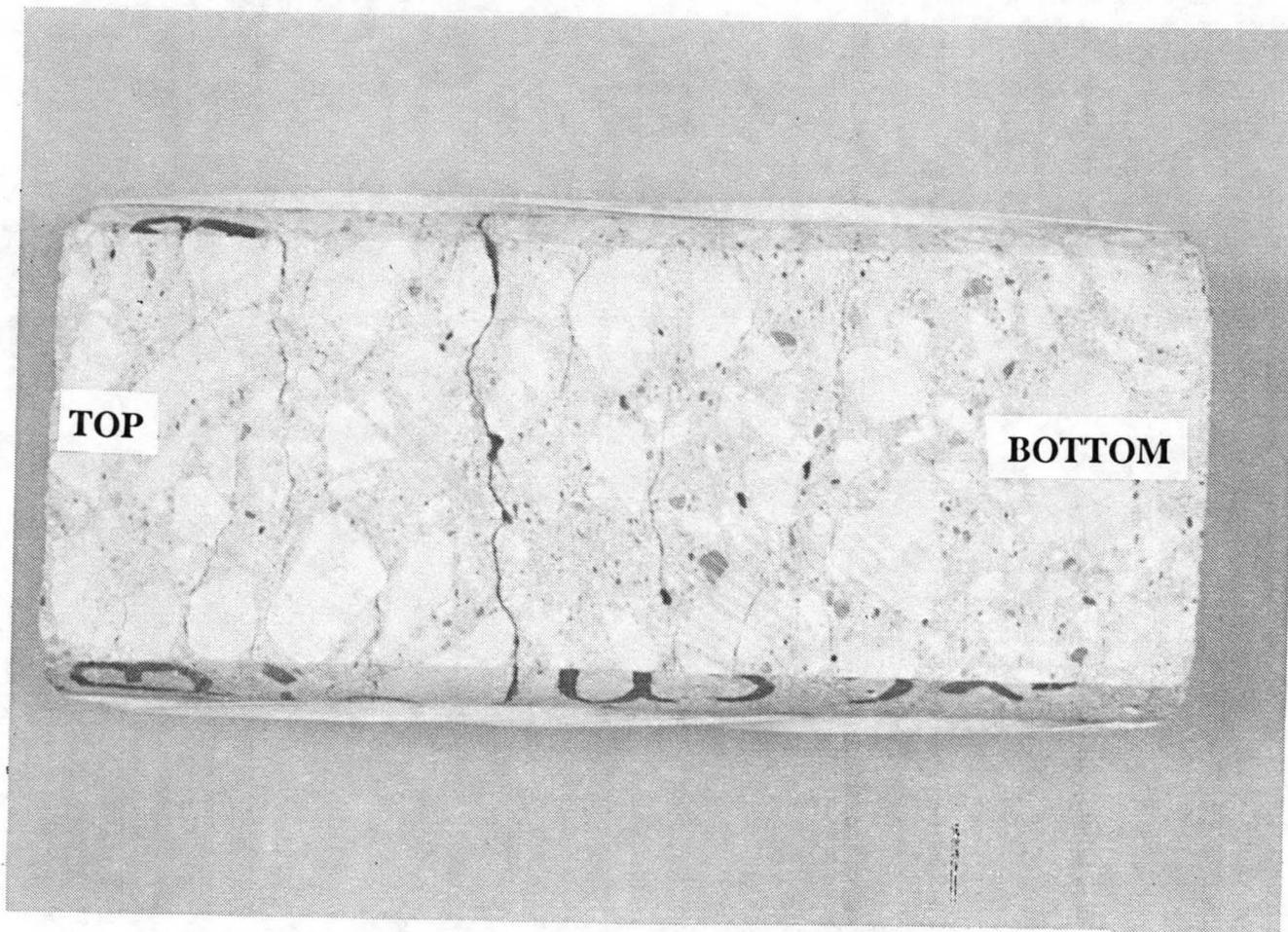


FIGURE 2 THE CRACKING PATTERN OF US 20 IN CENTRAL IOWA



**FIGURE 3 A US 20 CORE SHOWING FEW CRACKS
IN THE COARSE AGGREGATE**

Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Res: High
Label: 3T-2, US 520, EB lane (2 Nov 94 11:49:55)

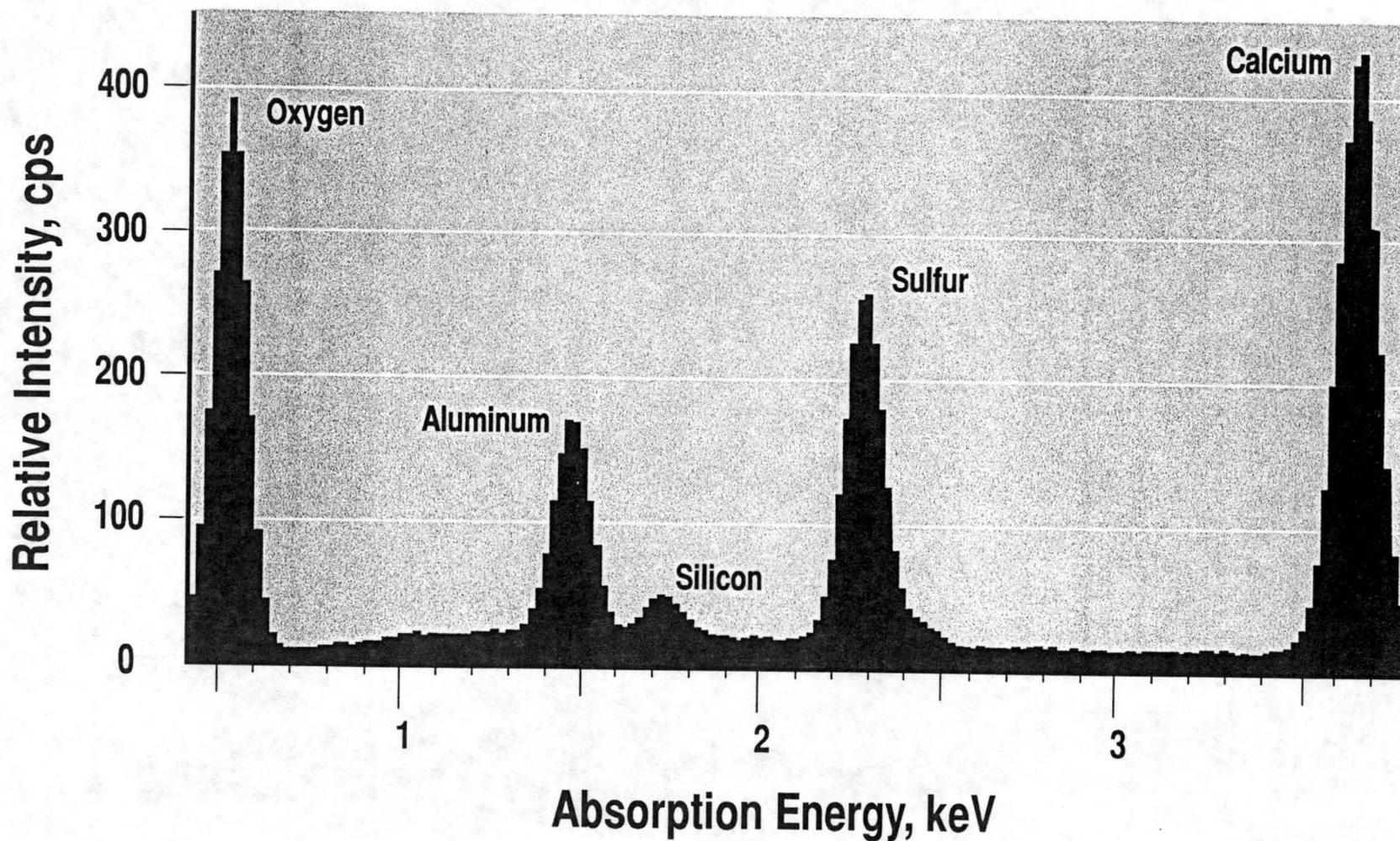
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**FIGURE 4 A 200X BSE PHOTOMICROGRAPH
OF THE PCC AIR VOIDS ON IOWA US 20**

Figure 5
An Energy Dispersion Spectrum
of the Whitish Material in the Air Voids

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Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Res: High
Label: 3T-2, US 520, EB lane (2 Nov 94 12:17:29)

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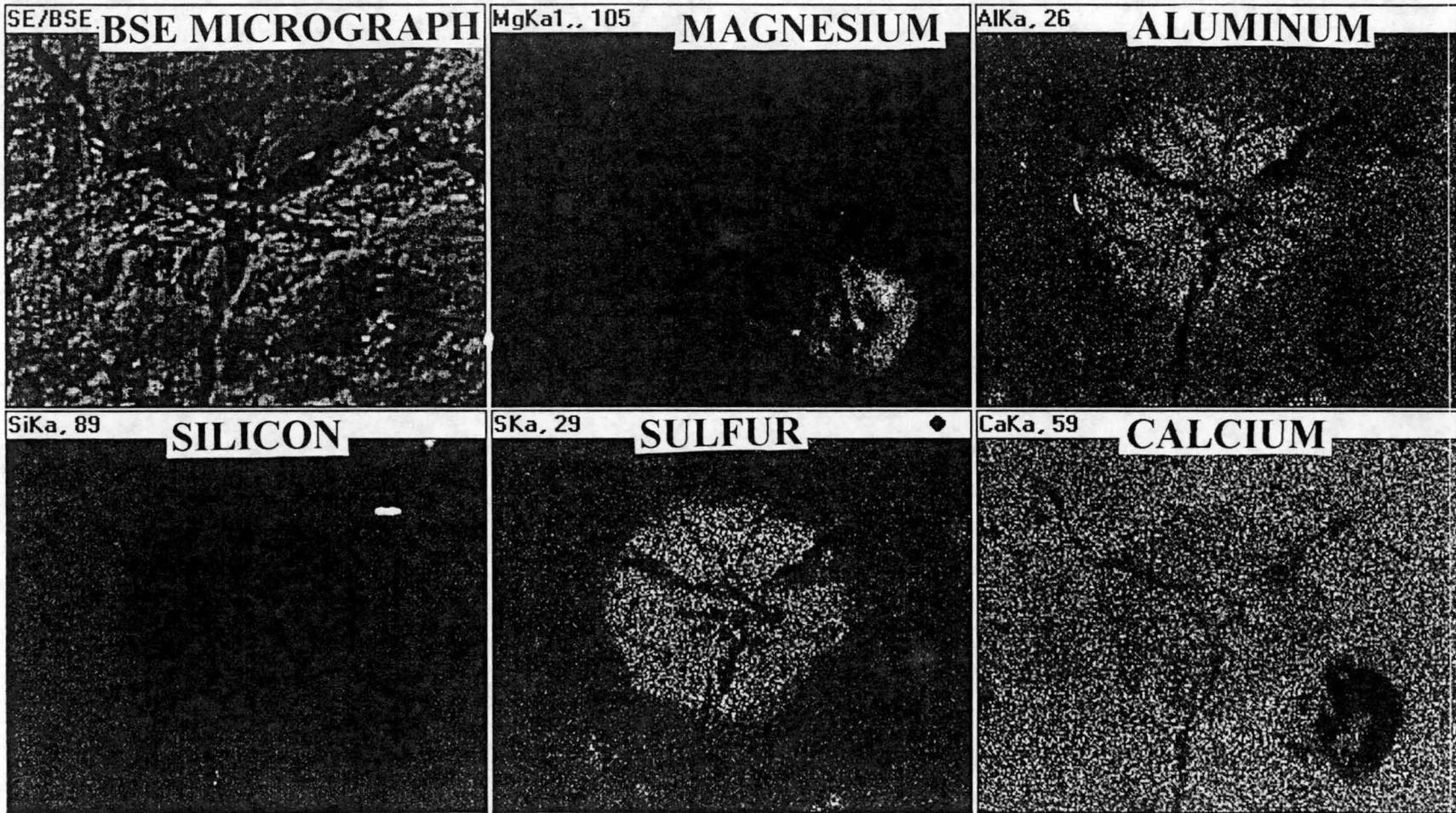


FIGURE 6 A 100X BSE PHOTOMICROGRAPH OF IOWA 20 SHOWING CRACKS RADIATING FROM THE ETTRINGITE FILLED AIR VOIDS

Operator: Scott S
Client: Iowa Dept. of Trans.
Job: HR-358 Task 1
Label: 3T-2, 1500X, US 520, EB LANE (2 Nov 94 12:39:57)

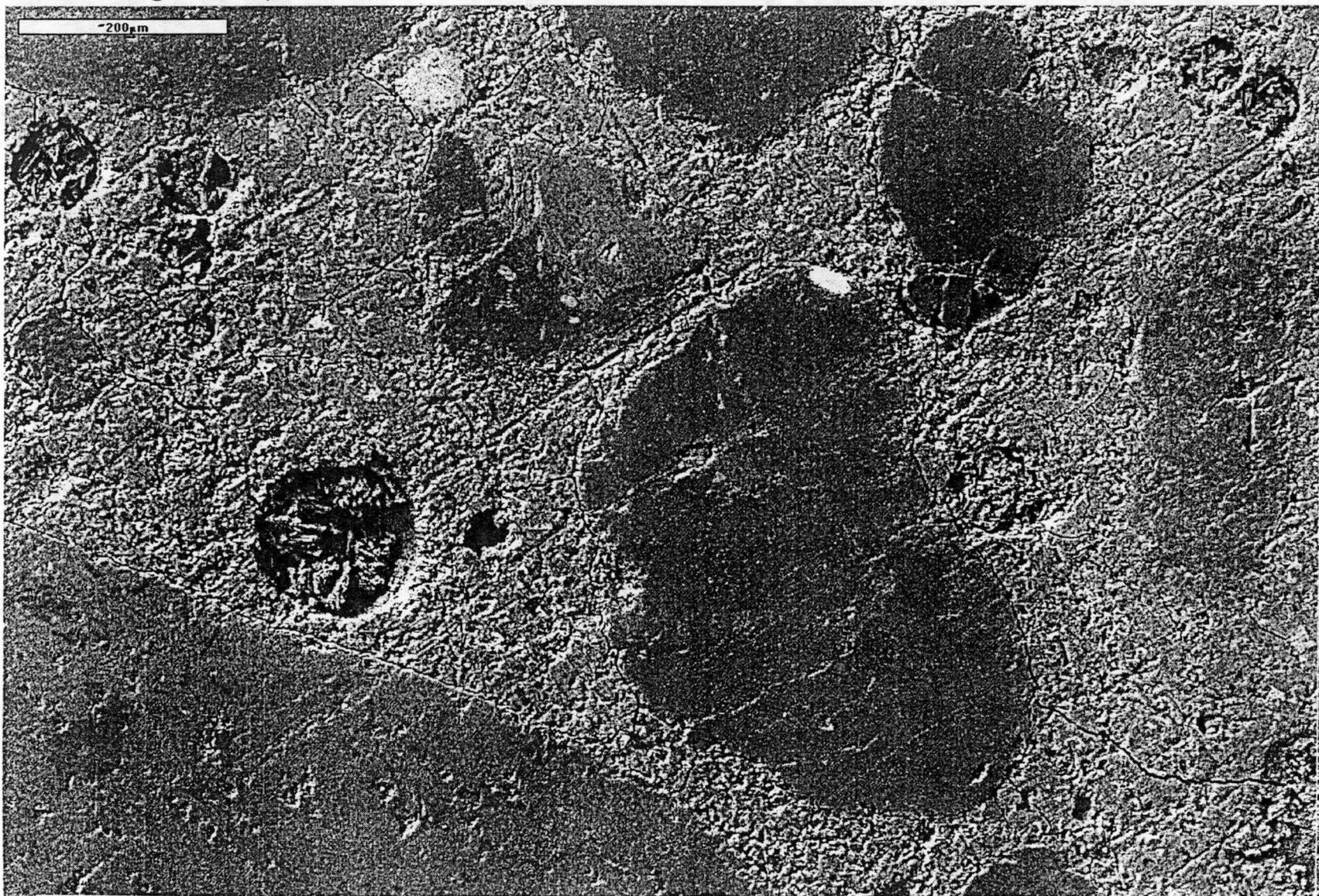
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**FIGURE 7 THE ELEMENTAL MAP FOR THE
FIGURE 6 PHOTOMICROGRAPH**

Operator: Wendell D
Client: Iowa DOT
Job: Iowa DOT
Res: High
Label: Neb I-80 @ Aurora 10 yrs 100x (22 Nov 94 10:05:39)



**FIGURE 8 A 100X BSE PHOTOMICROGRAPH OF
A NEBRASKA I-80 AURORA PCC SAMPLE**

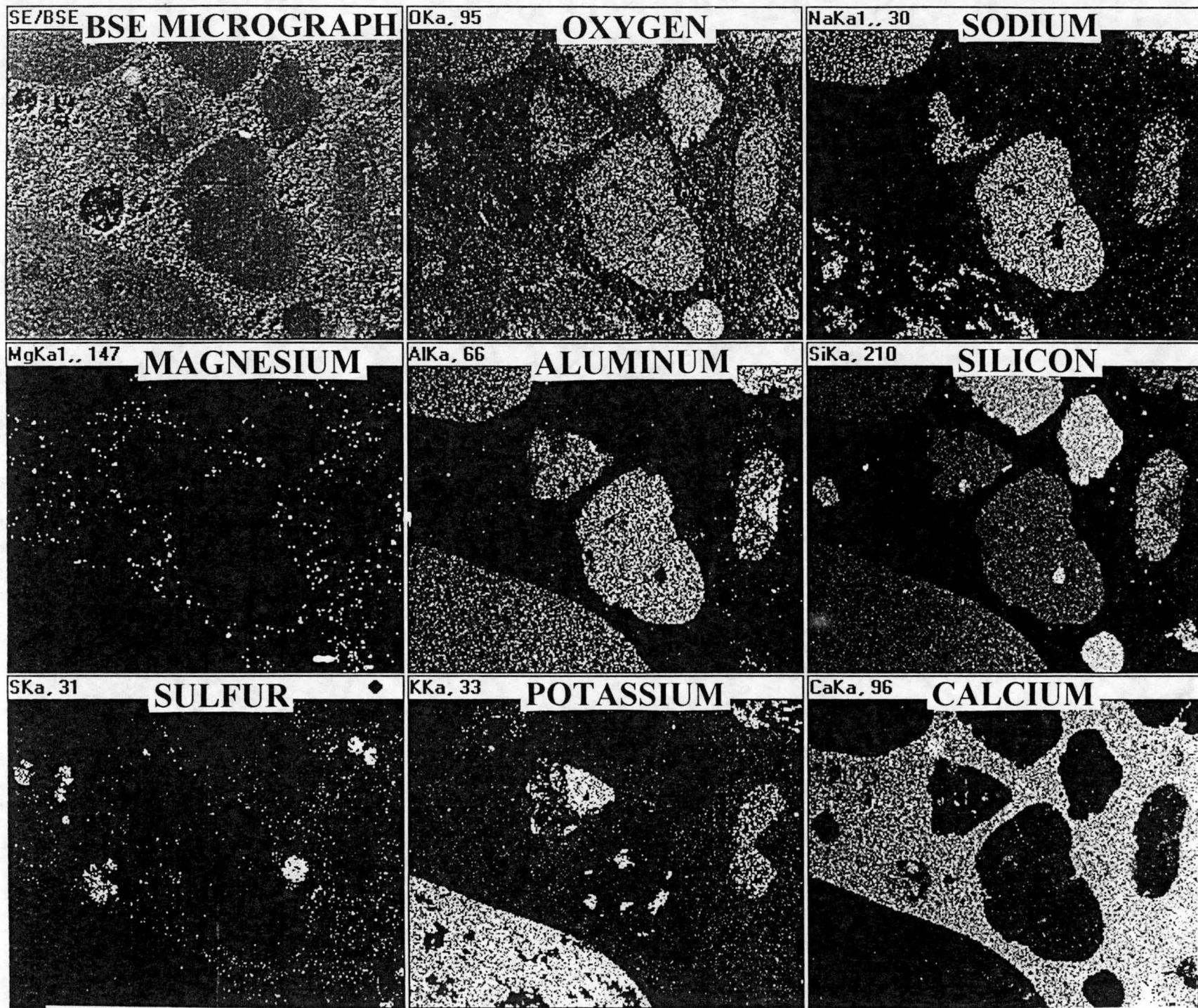
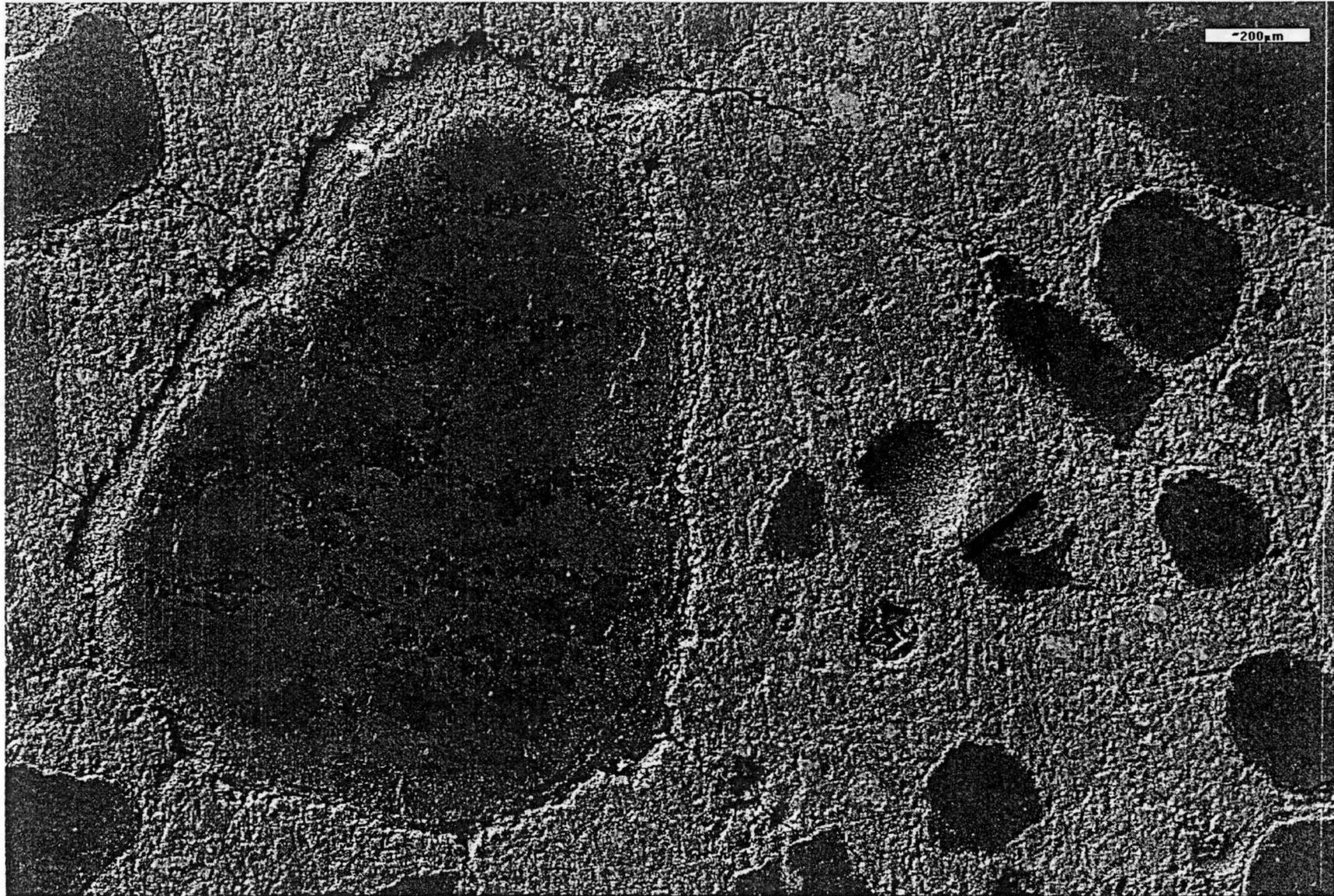


FIGURE 9 THE ELEMENTAL MAPS FOR THE FIGURE 8 PHOTOMICROGRAPH

Operator: Wendell D
Client: Iowa DOT
Job: Iowa DOT
Res: High
Label: Omaha Airport 50x

(22 Nov 94 15:10:12)



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**FIGURE 10 A 100X BSE PHOTOMICROGRAPH OF A
NEBRASKA OMAHA AIRPORT PCC SAMPLE**

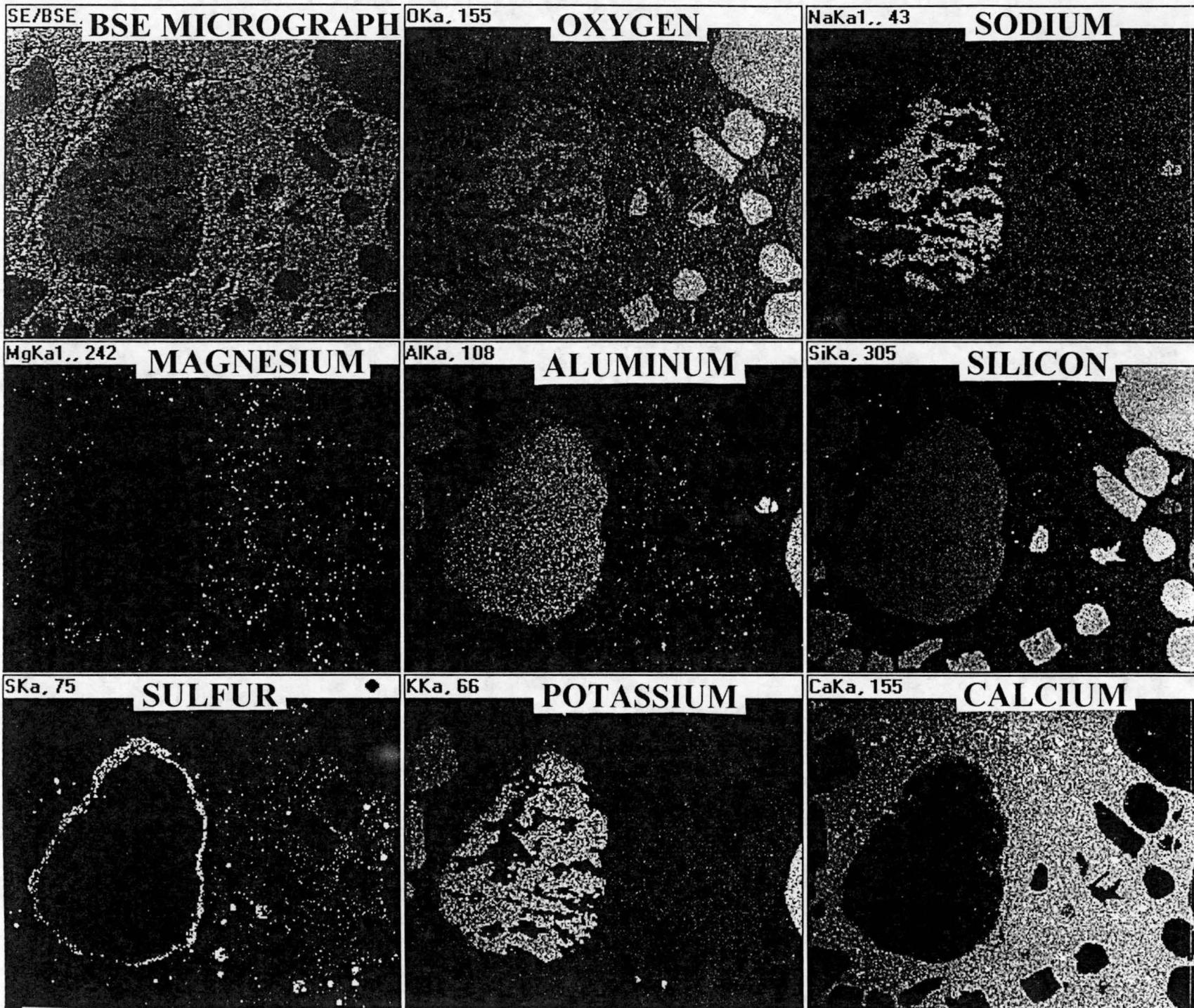


FIGURE 11 THE ELEMENTAL MAPS FOR THE FIGURE 10 PHOTOMICROGRAPHS

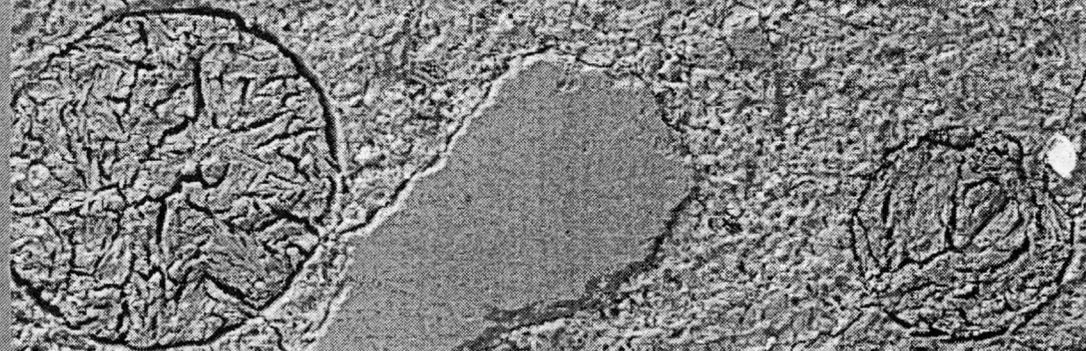
FIGURE 12 A 100X BSE PHOTOMICROGRAPH OF AIR VOIDS TIGHTLY FILLED WITH ETTRINGITE

Iowa DOT

No NaCl Brine

100x

100 um



35

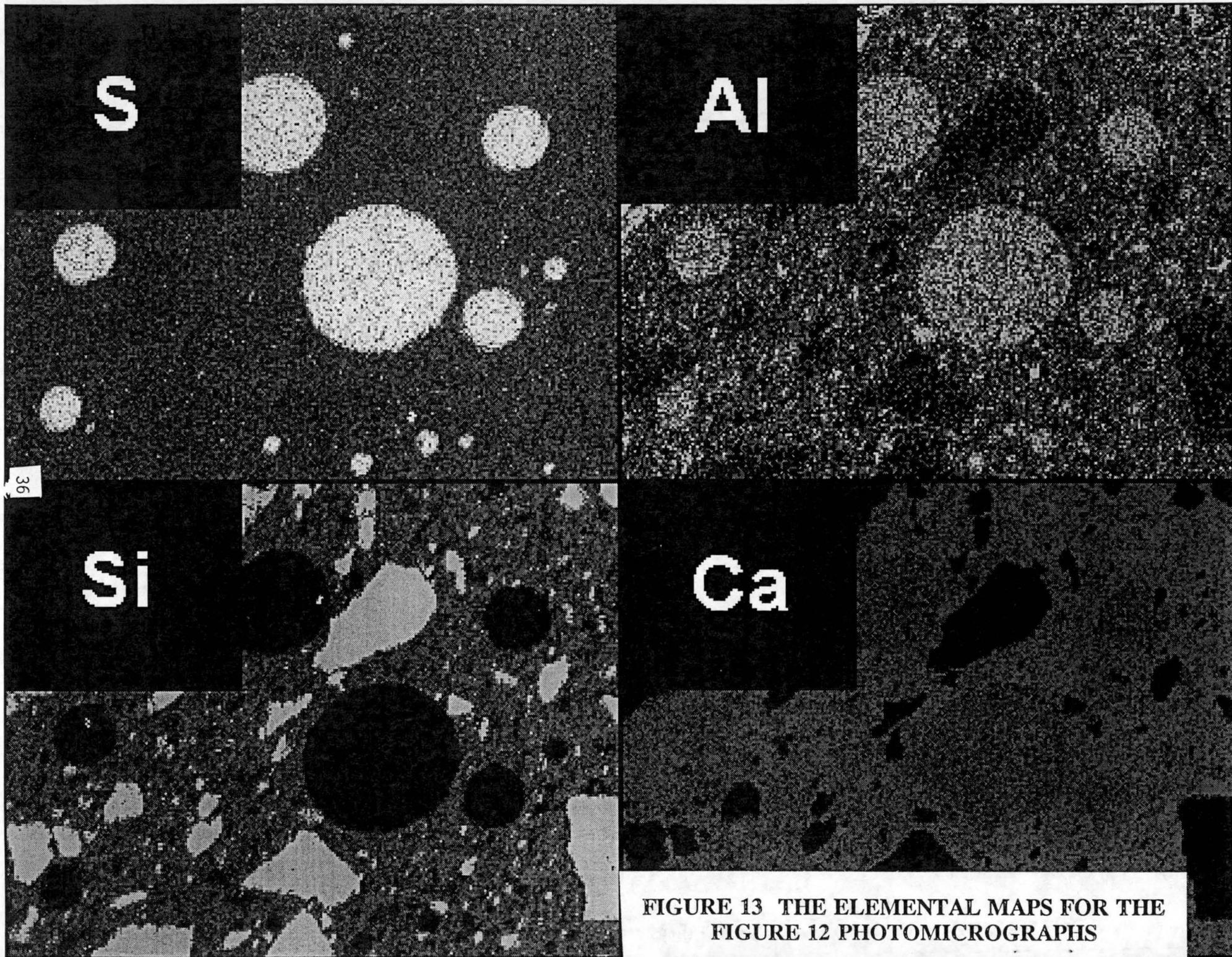


FIGURE 13 THE ELEMENTAL MAPS FOR THE FIGURE 12 PHOTOMICROGRAPHS

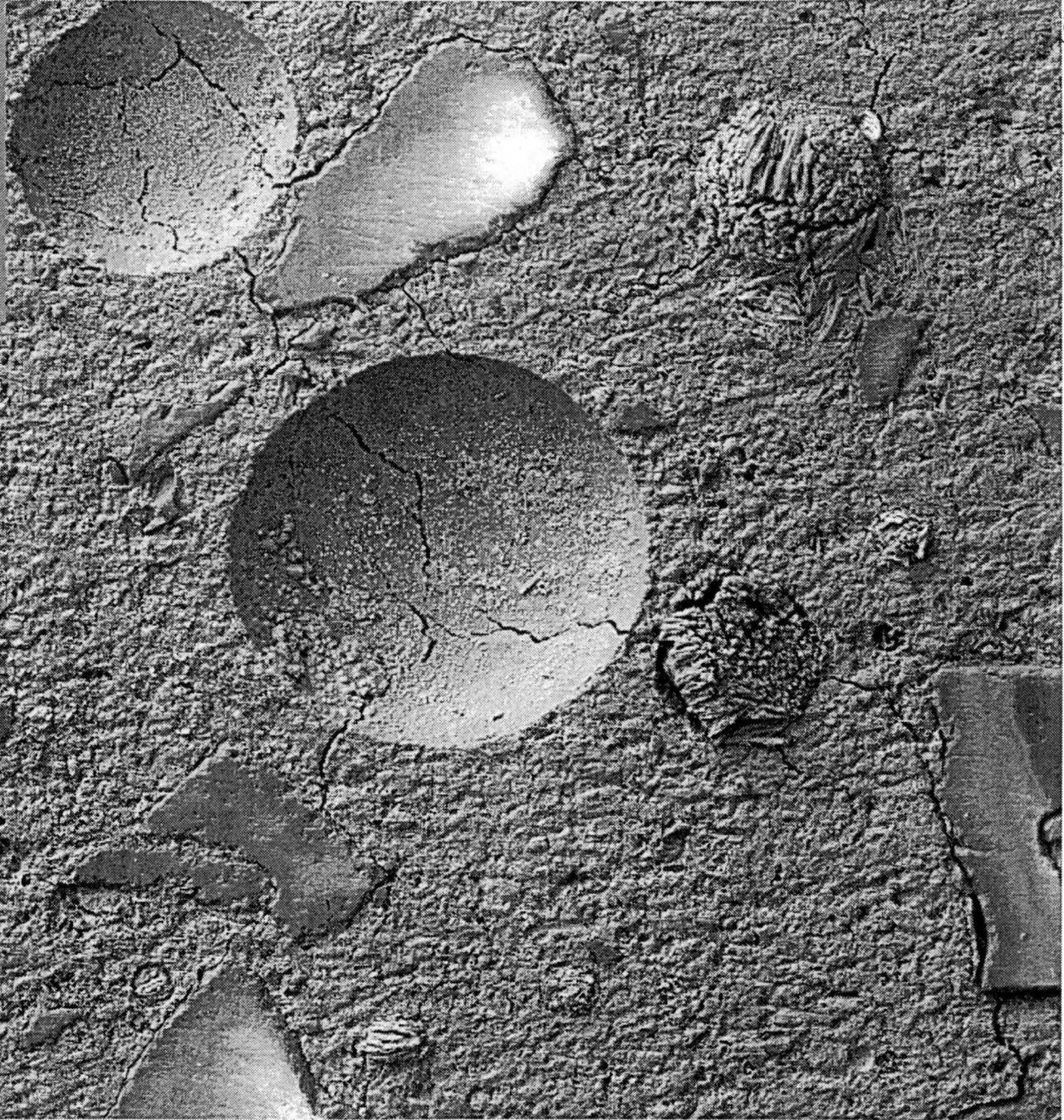
Iowa DOT

FIGURE 14 A 100X BSE PHOTOMICROGRAPH OF THE FIGURE 12 AIR VOIDS
45 MINUTES AFTER TREATMENT WITH NaCl BRINE

In NaCl Brine
45 min

100x

100 um



37

FIGURE 15 A 100X BSE PHOTOMICROGRAPH OF THE FIGURE 12 AIR VOIDS 48 HOURS AFTER TREATMENT WITH NaCl BRINE

Iowa DOT

**In NaCl Brine
48 hours**

100x

100 um

38

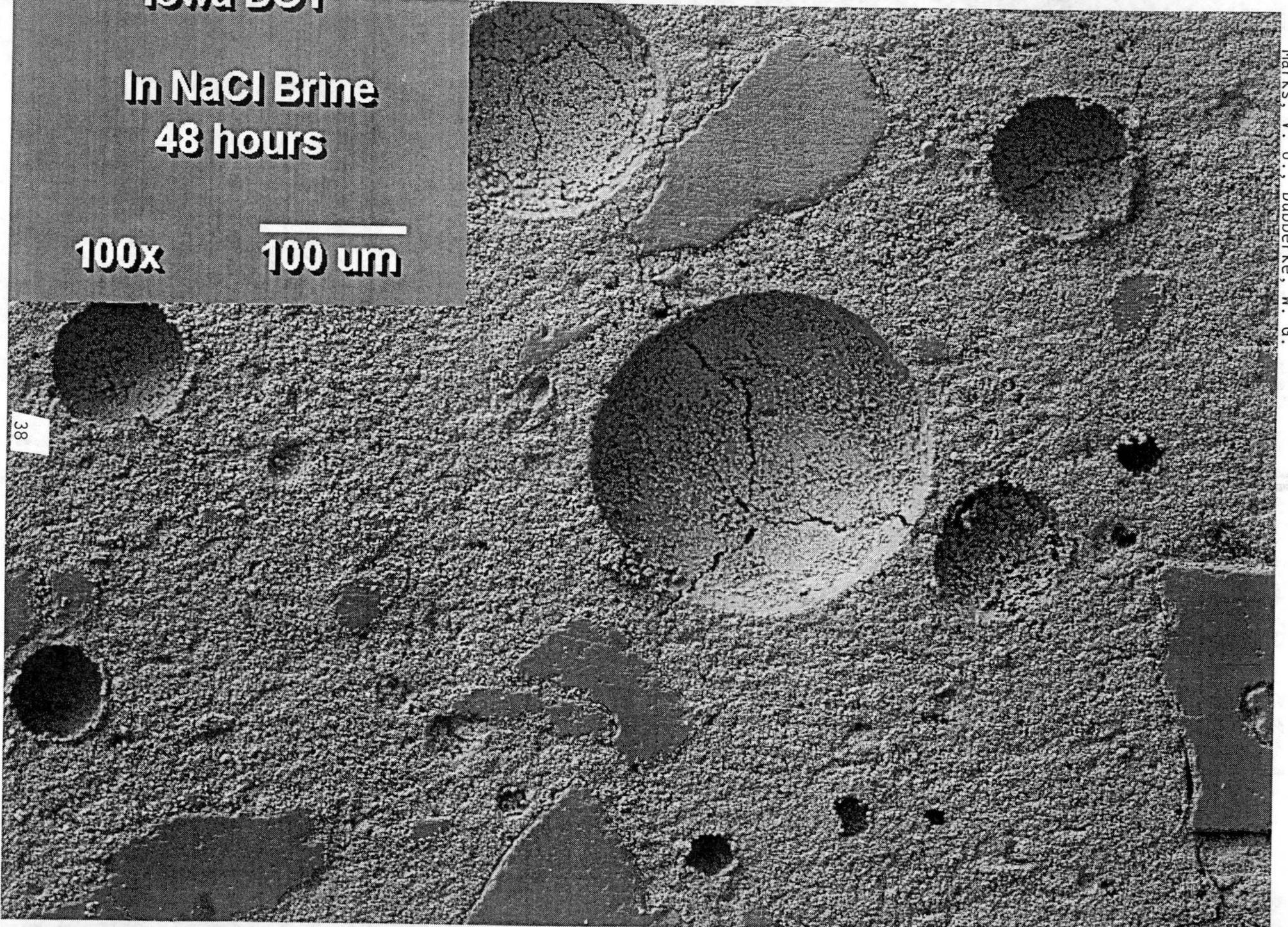


Figure 16 DUGGAN TEST RESULTS
WITH SODIUM CHLORIDE BRINE MODIFICATION

