

# Importance of Microscopy in Durability Studies of Solidified and Stabilized Contaminated Soils

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## ABSTRACT

Solidification/stabilization (S/S) is recognized by the U.S. EPA as a *best demonstrated available technology* for the containment of contaminated soils and other hazardous wastes that cannot be destroyed by chemical, thermal, or biological means. Despite the increased use of S/S technologies, little research has been conducted on the weathering and degradation of solidified and stabilized wastes once the treated materials have been buried. Published data to verify the performance and durability of landfilled treated wastes over time are rare. In this preliminary study, optical and electron microscopy (scanning electron microscopy [SEM], transmission electron microscopy [TEM] and electron probe microanalyses [EPMA]) were used to evaluate weathering features associated with metal-bearing contaminated soil that had been solidified and stabilized with Portland cement and subsequently buried on site, stored outdoors aboveground, or archived in a laboratory warehouse for up to 6 yr. Physical and chemical alteration processes identified include: freeze-thaw cracking, cracking caused by the formation of expansive minerals such as ettringite, carbonation, and the movement of metals from waste aggregates into the cement micromass. Although the extent of degradation after 6 yr is considered slight to moderate, results of this study show that the same environmental concerns that affect the durability of concrete must be considered when evaluating the durability and permanence of the solidification and stabilization of contaminated soils with cement. In addition, such evaluations cannot be based on leaching and chemical analyses alone. The use of all levels of microscopic analyses must be incorporated into studies of the long-term performance of S/S technologies.

SOILS, SLUDGES, AND INDUSTRIAL WASTES contaminated with heavy metals, such as As, Ba, Cd, Cr, Cu, Pb, and Zn, are commonly incorporated into cement matrices by means of S/S treatment processes (EPA, 1989; Gilliam and Wiles, 1996; Anderson, 1997). The treated wastes are considered nonhazardous if the solidified and stabilized material passes EPA's Toxicity Characteristic Leaching Procedure (TCLP) threshold values, and may then be buried on site or in a landfill (EPA, 1986, 1990).

Despite the increased use of S/S technologies on contaminated soils before they are disposed of in landfills, little published data are available to verify the durability and permanence of the solidified and stabilized wastes over time (Kirk, 1996; Glasser, 1997). Most knowledge about S/S technologies has been acquired through laboratory tests on small quantities of treated material, typically collected during treatability studies or technology

demonstrations (Klich, 1997). Past evaluations of durability commonly rely on standardized wet-dry or freeze-thaw cycling tests, as well as unconfined compressive strength tests (Horner and Slack, 1993; Kirk, 1996). However, the resistance of concrete to attack by chemical or physical agents is not solely related to its mechanical strength. A concrete material does not necessarily resist attack and disintegration in any environment in which it is placed simply because it has ample strength to fulfill the purpose for which it was designed (Bonen and Sarkar, 1994). Bulk chemistry and leaching tests also are frequently the only tests used to evaluate S/S treatment effectiveness (Badamchian et al., 1995). Such destructive analyses do not consider the mineralogical phases and microstructure of the materials being tested (Spence, 1993). Reports of failed solidified wastes suggest that unsuitable inorganic materials have been used widely without regard for long-term properties and field performance (Hills and Pollard, 1997). Still, there is a lack of fundamental field data related to long-term performance of solidified and stabilized products following their disposal (Stegemann et al., 1996).

The deterioration of solidified and stabilized wastes involves complex physical and chemical weathering processes. Chemical and physical stability of mineral phases present in the treated soil material can be determined best by in situ microscale analyses (Wakeley et al., 1992; Ivey et al., 1993). In this study, optical microscopy, scanning and transmission electron microscopy, and electron probe microanalyses were used to document and verify the effects of chemical and physical weathering on solidified and stabilized soil wastes and to evaluate the permanence of metals containment in several treated wastes that had been landfilled or stored aboveground for up to 6 yr.

## MATERIALS AND METHODS

Replicate samples from seven solidified and stabilized waste monoliths (1–10 m<sup>3</sup>) and smaller cast cylinders (15-cm diameter) that had been landfilled or stored aboveground for up to 6 yr were obtained from several EPA sources. Table 1 summarizes the source of soil contamination and environmental conditions of the treated waste storage locations. For the three treated wastes that included archived control samples, thin sections that were prepared at the time the treated wastes were cured (28 d following treatment) also were made available for this study. Previous studies documented the characteristics of the waste types under investigation, disposal environ-

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**Abbreviations:** BSE, backscattered electron; EDX, energy dispersive x-ray; SEM, scanning electron microscopy; EPMA, electron probe microanalyses; HDPE, high-density polyethylene; S/S, solidification/stabilization; TCLP, Toxicity Characteristic Leaching Procedure; TEM, transmission electron microscopy; UV, ultraviolet; WDX, wavelength dispersive x-ray.

**Table 1. Soil contamination and environmental conditions of treated waste storage.**

Treated waste	Source of soil contamination	Metal contaminants	Treated waste storage conditions	Critical soil characteristics of storage location
1	Metal plating; lagoon soils/sludge	Cd, Cr, Cu, Ni, Zn	Monoliths buried onsite in SC; fluctuating ground water table 0.5 to 1.5 m below surface.	Sandy; acidic to neutral pH; moderate to high risk of corrosion to concrete.
2	Metal plating; lagoon soils/sludge	Cd, Cr, Ni, Zn	Monoliths stored in aboveground landfill in NH; synthetic and soil liner/cover, freezing temperatures in winter.	Not available
3	Wood preserving; soil	As, Cr, Cu	Monoliths buried on site in SC; unknown water table level.	Not available
4	Metal salvaging; soil	Ba, Cd, Pb	Monoliths buried on site in FL; clay cap/cover; ground water table 9 m below surface.	Sandy; mildly to very acidic pH; moderate to high risk of corrosion to concrete.
5	Chemical processing; soil	As, Ba, Cd, Cr, Cu, Pb, Zn	Monoliths wrapped in HDPE and sorted above ground on pallets in NJ; freezing temperatures in winter; cast cylinders and control samples archived in TX warehouse.	Not applicable
6	Chemical processing; filtercake/sludge	As, Ba, Cd, Cr, Cu, Pb, Zn	Monoliths wrapped in HDPE and stored above ground on pallets in NJ; freezing temperatures in winter; cast cylinders and control samples archived in TX warehouse.	Not applicable
7	Transformer and metal recycling; soil	Cu, Pb, Zn	Cast cylinders and control samples archived in TX warehouse.	Not applicable

ments, and sampling procedures used to obtain samples for the microscopy analyses (Badamchian et al., 1995; Horner and Slack, 1993; Klich, 1997). Those studies also provided physical, chemical, and leaching data for the treated wastes; however, those test data and analyses do not address the possible mechanisms of concrete degradation in soil environments with time, nor do they adequately assess the durability of solidified and stabilized contaminated soils. Bulk x-ray diffraction analyses, sample preparation techniques, and quality assurance and quality control measures used in this study are described by Klich (1997). Microscopic analyses were conducted at laboratories of the U.S. Geological Survey in Reston, VA and at Texas A&M University.

### Petrographic Analyses

Small blocks of the solidified and stabilized wastes were impregnated with epoxy containing an ultraviolet (UV) fluorescent dye (Walker, 1992; Thaulow and Jakobsen, 1996). Thin sections were prepared by a professional petrographic laboratory using standard cement and concrete sample preparation techniques (St. John, 1990). Reflected light microscopy was used to evaluate opaque materials, such as the metallic waste contaminants. Plane and cross-polarized transmitted light was used to observe transparent and translucent concrete components of the treated waste and control samples, as well as the micromorphic relationship between the cement matrix and the constituents of the contaminated soils. In addition, UV-reflected light was used to determine porosity and evaluate the nature of voids within the samples of treated waste.

### Scanning Electron Microscopy

A JEOL (Model 840, JEOL, Ltd., Akishima, Japan) scanning electron microscope, equipped with a LaB<sub>6</sub> filament and an energy dispersive x-ray (EDX) spectrometer, was used to analyze epoxy-impregnated, highly polished thin sections of the treated waste samples. The advantage of using polished thin sections, rather than polished blocks, is that the same features can be observed and analyzed both under the scanning electron microscope and the petrographic microscope. Backscattered electron (BSE) images were used primarily to locate and differentiate metal contaminants having atomic numbers higher than that of the surrounding cement matrix

material. The EDX microanalyses provided nondestructive, qualitative chemical compositions of the cryptocrystalline and amorphous cement micromass, as well as the metal contaminants contained in the cement admixtures. Elemental distribution maps were used to provide detailed information on chemical zoning and textural relationships that were not always apparent from BSE images or optical photomicrographs.

### Transmission Electron Microscopy

A JEOL (Model JEM-2010, JEOL, Ltd.) transmission electron microscope, equipped with an EDX spectrometer was used to obtain electron diffraction patterns of selected metal contaminants and cement constituents. Chemical compositions obtained by EDX spectrometry aided in the identification of metal-bearing minerals through the use of selected area diffraction spot or ring patterns. Electron diffraction data obtained by the TEM analyses allowed for the nondestructive identification of the metal-bearing minerals. Such information cannot be obtained from SEM analyses alone.

### Electron Probe Microanalyses

A JEOL (Model 8900, JEOL, Ltd.) electron probe, equipped with a wavelength dispersive x-ray (WDX) spectrometer was used to chemically analyze samples containing metals with overlapping characteristic x-ray energy peak spectra, including As (K- $\alpha$ ) and Pb (L- $\alpha$ ); As (L- $\alpha$ ) and Mg (K- $\beta$ ); S (K- $\alpha$ ) and Pb (M- $\alpha$ ); Ba (L- $\alpha$ ) and Ti (K- $\alpha$ ); and P (K- $\alpha$ ) and Zr (L- $\alpha$ ). Those combinations of elements cannot be resolved by EDX spectrometry, as was the case with samples from this study. The spatial distribution of specific elements was recorded as two-dimensional maps, thus providing reliable representations of the distribution of the elements under investigation.

## RESULTS AND DISCUSSION

Evidence of chemical and physical weathering, as well as alteration of the treated waste samples, was observed in monoliths and cast cylinders of solidified and stabilized wastes that had been stored under diverse environmental conditions, such as direct exposure to freezing

and thawing, acidic soils prone to fluctuating groundwater tables, and archival storage under laboratory conditions. Examples of macro-, micro-, and submicroscopic features are presented below.

### Macrostructure

Large-scale features (up to 1 m) could be observed in the monoliths of treated wastes that were wrapped in high-density polyethylene (HDPE) and stored outdoors and aboveground on pallets (Treated Wastes 5 and 6). Field observations of those monoliths revealed abundant macroscale cracking after 6 yr. Macrovoids within those monoliths consisted predominantly of planar voids as large as 1 m long and 1 to 5 mm wide (Fig. 1a), as well as vesicles up to 6 mm in diameter. Planar voids commonly showed the characteristic "Isle-of-Man" or pattern cracking, also known as map cracking or crow's-foot cracking (Fig. 1b). It is recognized that such cracking patterns in concrete may result from changes in volume due to plastic shrinkage, as well as expansion due to chemical reactions within the concrete (Walker, 1992).

Macroscopic vertical cracks observed in the same monoliths of treated waste are characteristic of freeze-thaw action (Walker, 1992). In addition, macrovoids that consisted of irregular cavities as large as 3 cm long and 1 cm wide were commonly observed. Such irregular voids represent incomplete compaction of air space created during the pouring of semisolid cement admixtures into the frames that initially give form to the monoliths (Walker, 1992).

Carbonate precipitation around pockets of the unmixed waste material and along the lower portions of some vertical cracks was observed at both the macro- and microscales. Such features typically are associated with carbonate alteration, or carbonation, as well as alkali-silica reactions in concretes where macroscale dissolution and reprecipitation of calcite ( $\text{CaCO}_3$ ) along vertical fractures are common (Thaulow and Jacobsen, 1996).

### Microstructure

Within all of the treated waste and control samples, microscopic and submicroscopic cracks were observed ubiquitously within the cement micromass itself, or in association with aggregates of cement and/or the waste (Fig. 1c–1f). Some cracks were unfilled with secondary weathering products, while others were filled or lined with silica gel, ettringite, or microcrystalline calcite. The percentage of filled cracks in deteriorating cement and concretes may increase with time, as well as with the extent and nature of chemical alteration (Walker, 1992). Cracking in concrete may be caused by a number of factors, including insufficient air-void content, drying and plastic shrinkage, chemical reactions that cause expansion, and freeze-thaw action (Walker, 1992). Cracking caused by chemical reactions is commonly the result of the formation of expansive minerals, such as ettringite [ $\text{Ca}_6\text{Al}_2\text{O}_3(\text{SO}_4)_3 \cdot 31\text{--}32\text{H}_2\text{O}$ ], monosulfate ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ ), thaumasite ( $\text{CaSiO}_3 \cdot$

$\text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$ ), gypsum, and alkali silica gel. Intergrowths of these minerals within the hardened cement micromass result in crystal expansion that exceeds the shear strength of the monolith and is therefore destructive to the concrete structure (Thaulow and Jacobsen, 1996). Amorphous to cryptocrystalline ettringite (Fig. 2a–2c) and thaumasite (Fig. 2d–2f) were identified within the cement micromass through the use of TEM, EDX spectroscopy, and selected area electron diffraction patterns. Alkali silica gel and microcrystalline gypsum also were observed frequently within the cement micromass. These mineralogical features are common indicators of physical and chemical alterations resulting from sulfate attack and alkali-aggregate reactions that can produce changes in volume, causing cracking of cement and concrete (Walker, 1992).

### Microchemistry

Chemical alteration of the cement micromass was apparent in all of the treated waste samples, as evidenced by the micromorphic features observed in thin sections. Microcrystalline precipitates of calcite within the cement matrix typically were observed in direct association with macro- and microfractures, as well as with packing voids of the control samples and treated waste samples. The presence of alkali silica gel-filled fractures suggests movement of fluids through the samples of treated wastes. Such features indicate that carbonate alteration, or carbonation processes have been active (Walker, 1992; Thaulow and Jackobsen, 1996). Carbonation is considered the most common form of environmental attack that can degrade the performance and structural integrity of cements and concrete with time (Bonen and Sakar, 1994).

Evidence for the movement of metals from waste aggregates into the cement micromass commonly was observed through the combined use of both optical and electron microscopy. For example, migration of Pb, Sn, and Ti from a dense, opaque waste aggregate into the surrounding cement matrix was not apparent under optical microscopy (Fig. 3a); however, BSE images and corresponding EDX analyses revealed the presence of a halo of Pb, Sn, and Ti in the cement matrix surrounding and closely associated with the waste aggregate (Fig. 3b). The TEM selected area diffraction patterns and associated EDX chemical analyses (Fig. 3c and 3d) verify the presence of microcrystalline  $\text{PbTi}_3\text{O}_7$  and  $\text{Pb}(\text{SnTi})\text{O}_3$ , as well as amorphous Pb oxide in the cement matrix surrounding the opaque waste fragment. The Ni peaks shown in each of the EDX spectra associated with TEM are the result of using Ni grids for mounting the TEM specimens. The BSE images together with electron microprobe data suggest that Pb (II) and Sn (II) cations in solution either diffused from the waste aggregate and substituted for Ca (II) in the cement hydrates, precipitated as complex Pb–Ti–Sn oxides within the micropores, or were adsorbed onto the amorphous cement micromass.

Alloys containing Zn along with varying amounts of Al, Cr, Cu, Fe, and Ni, also were identified (Plate 1a–1e).

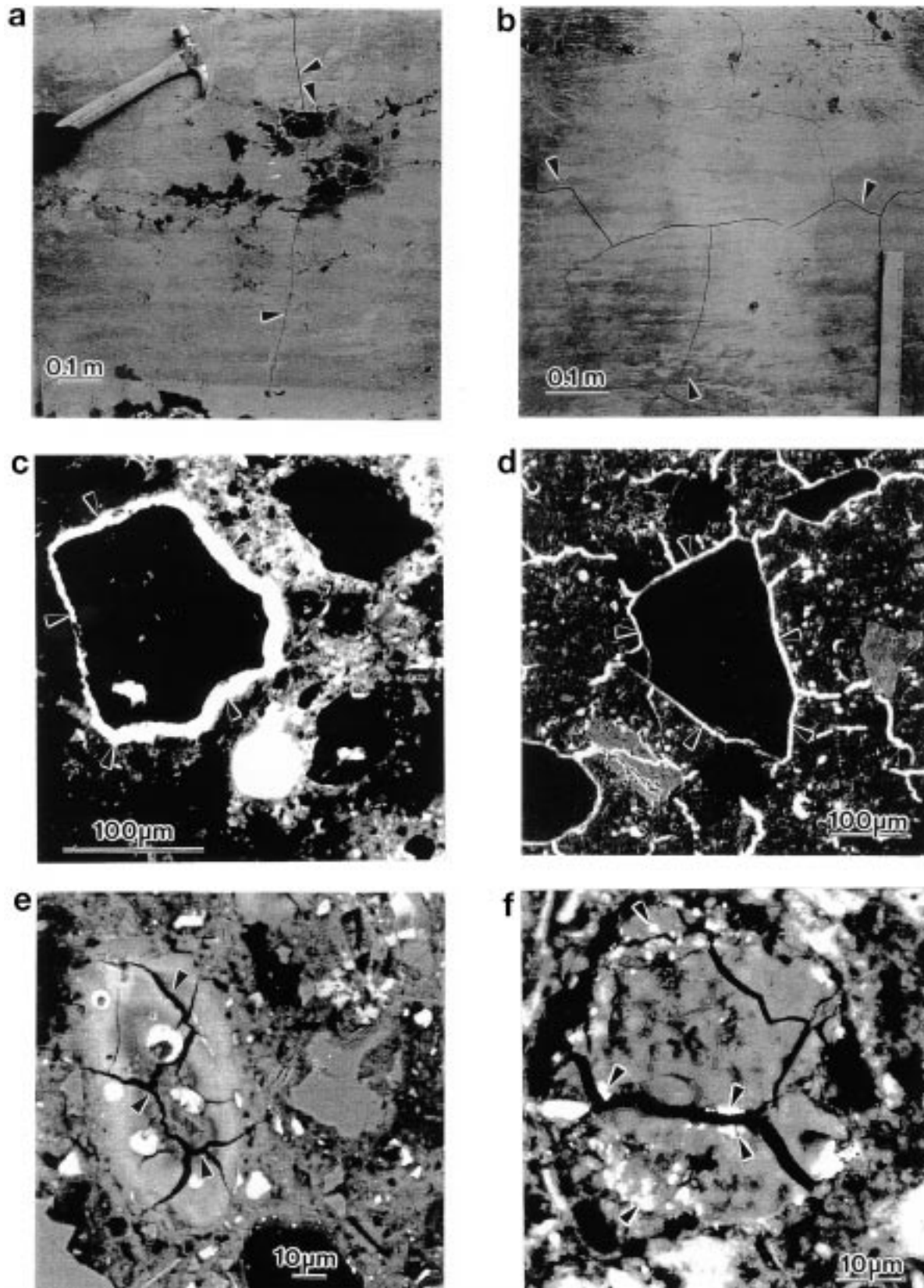


Fig. 1. Macrovoids (a–b) and microvoids (c–f) in solidified and stabilized contaminated soil. Hammer and rule length are  $\approx 30$  cm. Arrows show (a) vertical planar void from freeze–thaw cracking, Treated Waste 6; (b) characteristic “Isle-of-Man” pattern cracking, Treated Waste 5; (c) cement micromass expansion cracks under UV fluorescent light, Treated Waste 6, and (d) under SEM, Treated Waste 1; (e) plastic shrinkage cracks in cement micromass, Treated Waste 3; and (f) Pb and Ba contamination along plastic shrinkage cracks within clay waste aggregate, Treated Waste 4.

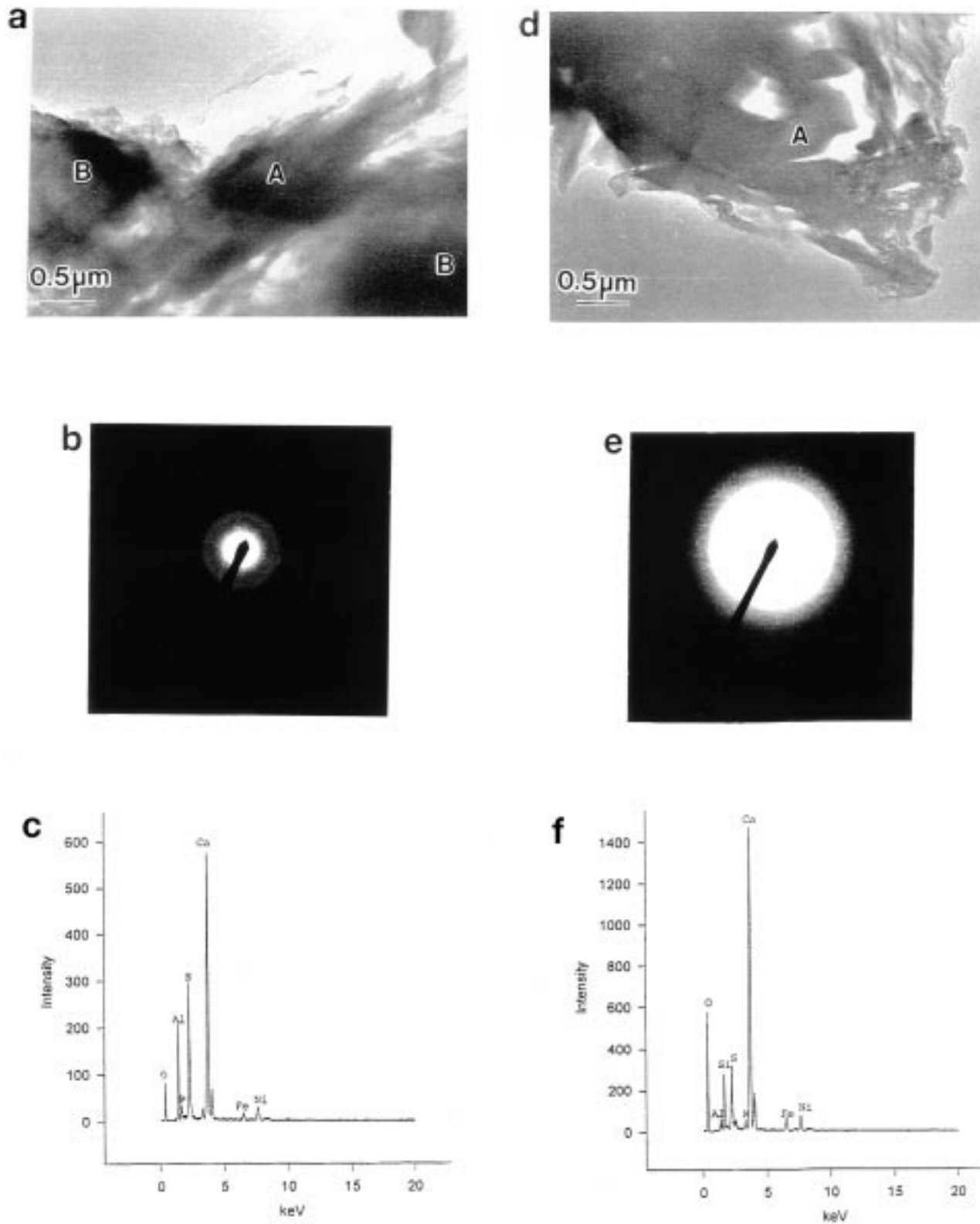


Fig. 2. Transmission electron microscope image, electron diffraction pattern, and energy dispersive x-ray spectra of (a-c) cryptocrystalline ettringite  $[\text{Ca}_6\text{Al}_2\text{O}_3(\text{SO}_4)_3 \cdot 31\text{-}32\text{H}_2\text{O}]$ , Treated Waste 1, and (d-f) thaumasite  $[\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}]$ , Treated Waste 4.

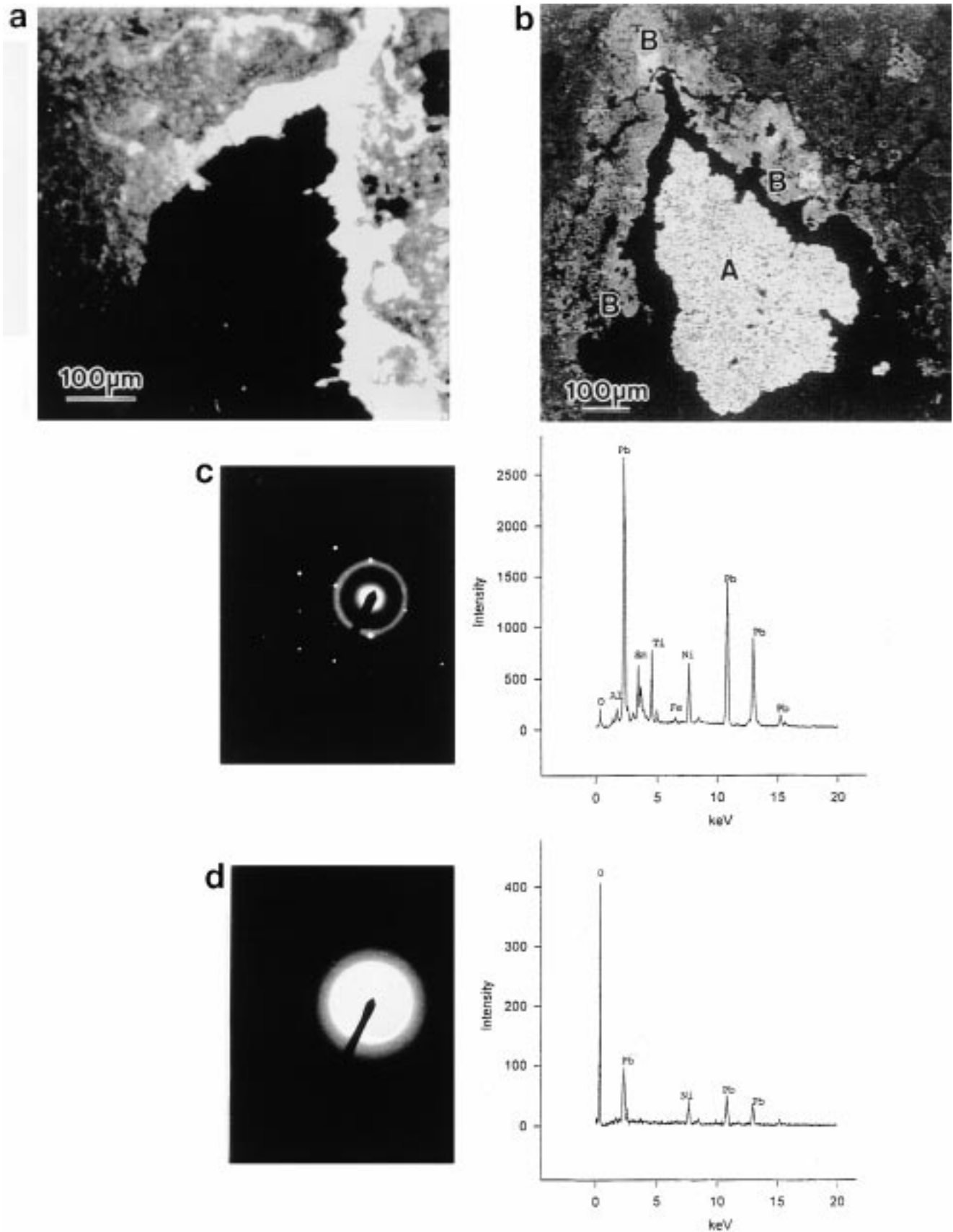
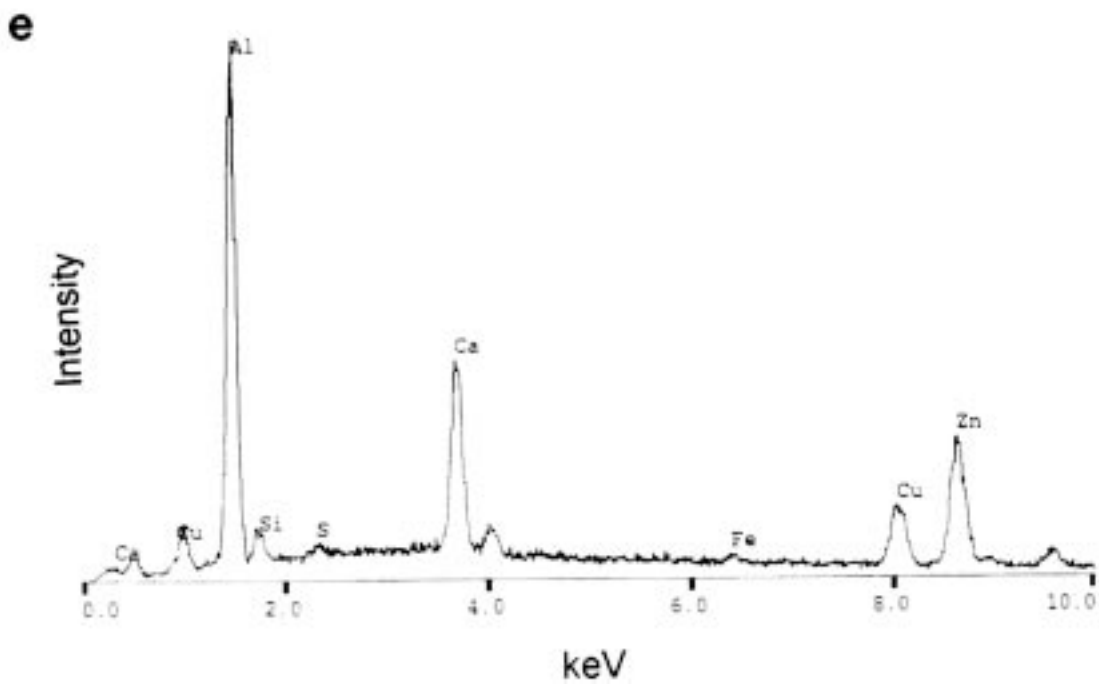
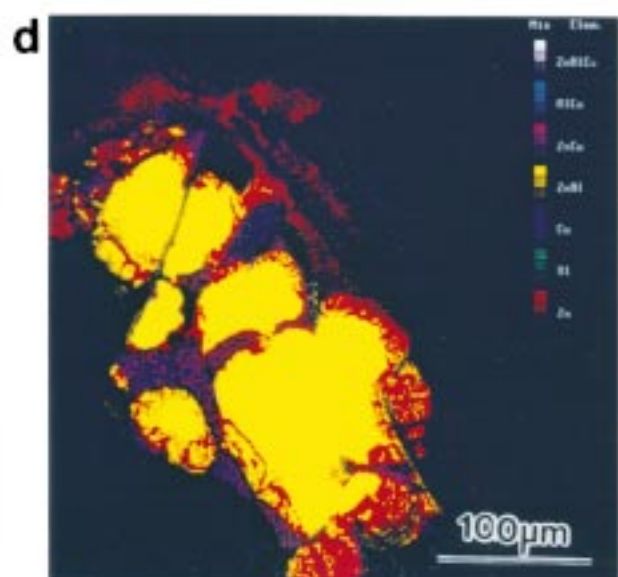
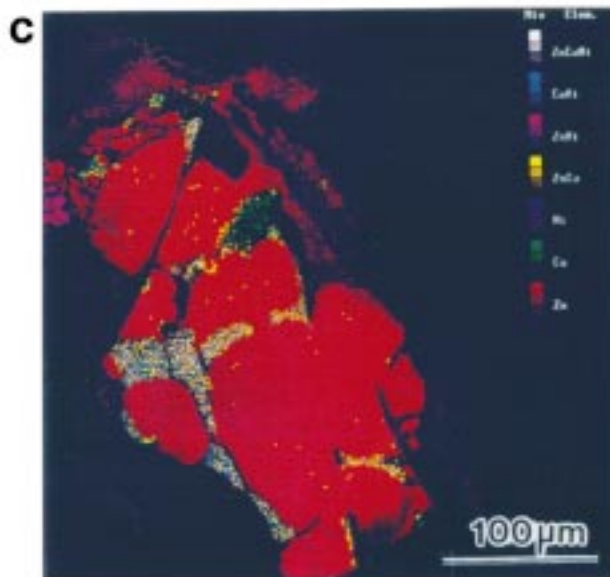
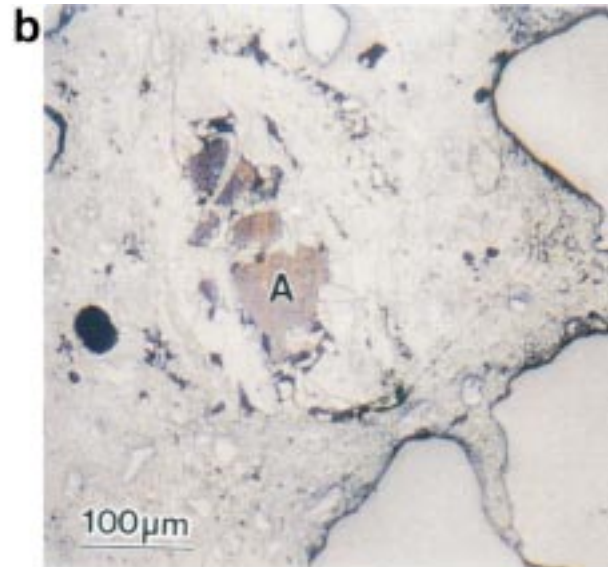
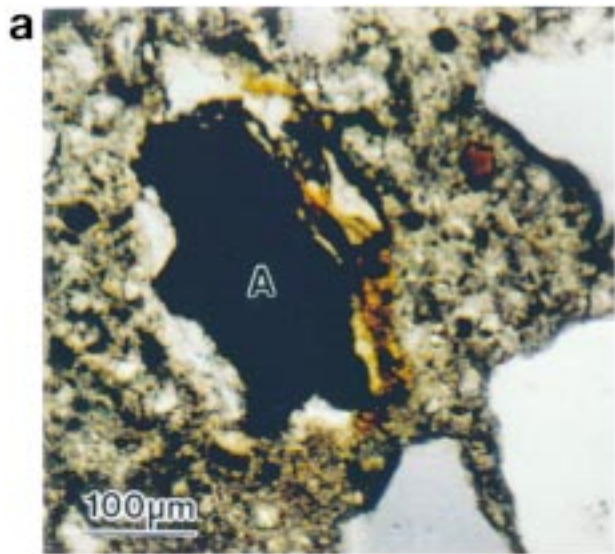


Fig. 3. (a) Photomicrograph and (b) backscattered electron image of opaque waste aggregate “A” encapsulated in cement, Treated Waste 7; transmission electron microscope selected area diffraction pattern and energy dispersive x-ray analyses verify presence of (c) microcrystalline  $PbTiO_3$  and  $Pb(SnTi)O_3$ , and (d) amorphous Pb oxide within the cement micromass “B”.



The Zn commonly was observed to be disseminated throughout the cement micromass as halos around metallic waste fragments that contained Zn and other metals such as Al, Cu, and Ni. As in the case of Pb contaminants, such Zn-rich halos, observed on electron microprobe WDX elemental maps, were not as easily recognizable when the same opaque fragments were viewed under visible or reflected light. The lack of apparent alteration of the cement micromass, as seen in thin sections under visible light, suggests that Zn cations also may have diffused from the original metal contaminant and adsorbed to the surrounding cement micromass.

Migration of Cu also was observed in samples of solidified and stabilized waste that contained Cu-rich waste aggregates encapsulated in cement. Various stages of dendritic overgrowths were observed during a 2-yr period (Fig. 4a and 4b). As a result of exposure to fluctuating humidity conditions in the laboratory (as high as 90%), hydration of the Cu-rich waste aggregate formed the hydrated Cu-sulfate mineral chalcantite. Scanning electron microscopy BSE imaging (Fig. 4c) and EDX elemental mapping (Fig. 4d) show movement of Cu without the associated S into a crack within the Ca- and Cu-rich waste aggregate.

### Implications for Durability of Landfilled Solidified and Stabilized Wastes

The same environmental issues that affect the durability of concrete must be considered when evaluating the durability and permanence of solidified and stabilized wastes that later are buried, either on site or off site in hazardous waste landfills. Such evaluations can only be made if one considers the micromorphology of the treated wastes and the soil conditions into which the treated wastes are to be disposed. Currently, EPA's regulatory criterion for toxicity characteristic requires the use of the TCLP to determine whether a waste is hazardous (EPA, 1990). In addition, EPA also requires the use of the paint filter liquids test (PFLT) to determine whether free liquids are present in a waste before it is placed in a hazardous waste landfill. Neither criterion is adequate for determining the durability or permanence of the containment of cement-stabilized and solidified wastes once the treated wastes have been buried on site under diverse environmental conditions. However, such tests continue to be inappropriately used to evaluate the long-term effectiveness of S/S technologies.

Viewed mineralogically, solidified and stabilized wastes, like hardened concrete, are metastable, both physically and chemically, under ambient conditions of service. Durability of concrete is governed by the material's quality, as defined by its resistance to chemical and physical degradation, and the harshness of the environmental conditions to which it is exposed during its

service life. All through its service life, hardened cement and concrete attempts to reach equilibrium with the surrounding exposure conditions, which, even under the harshest environments, would not approach its original curing conditions consisting of a relative humidity of 100% and a pH of 13. In addition, even the best-quality concrete cannot be expected to last for centuries under extremely harsh conditions, such as in acidic or saline environments (Harrison, 1987; Bonen and Sarkar, 1994). Therefore, when considering the durability and permanence of cement-based solidified and stabilized wastes, the expected service life of such treated wastes must be defined. Furthermore, the service life will naturally depend on the site-specific environmental conditions in which the treated wastes are placed. Environmental conditions that would be likely to enhance the disintegration of concrete include acid or saline soils, as well as areas with shallow, fluctuating groundwater tables (National Cooperative Soil Survey, 1989; American Concrete Institute, 1990). Current regulations governing the burial of solidified and stabilized wastes that are not classified as hazardous because the treated wastes do not exhibit the toxicity characteristic do not restrict their on-site placement in areas in which shallow fluctuating groundwater, or saline or acid soils occur. Therefore, additional microscopy evaluations of actual field samples of treated wastes from such diverse settings are urgently needed to accurately assess and verify the long-term stability and durability of buried solidified and stabilized wastes with time.

### CONCLUSIONS

The primary advantage of micromorphic and mineralogical analyses in evaluating the effects of weathering on solidified and stabilized wastes is the ability to observe the exact area of chemical analysis without destroying the character of the sample being analyzed, as is the case in bulk chemistry and leaching analyses. In addition, microscopic features and mineral relationships can be viewed and analyzed directly at several scales of resolution. These microscopic and mineralogical analyses also show specifically what is being altered, the mode of alteration, alteration pathways, and secondary products of alteration. Such findings are important in evaluating the permanence and leaching potential under given environmental conditions. Evaluations of cement and concrete quality and its durability over time, therefore, can be made based on observed mineralogical data.

Results of this microscopy study show that micromorphic features found in aged, cement-based, solidified and stabilized soils are similar to those documented for deteriorating concretes. Cement-based solidified and stabilized wastes will naturally be vulnerable to the same physical and chemical degradation and weathering processes as soil, concrete, and other cement-based materi-

**Plate 1.** Photomicrograph of opaque waste aggregate "A" under (a) plane polarized light and (b) reflected light, Treated Waste 2; (c-d) wavelength dispersive x-ray elemental maps show (c) Zn in red, with combinations of Zn, Cu, and Ni in white; Cu and Ni in blue; and Zn and Cu in yellow; and (d) combinations of Zn and Al are shown in yellow. (e) Energy dispersive x-ray spectra verify presence of Zn, Cu, Ni, and Al in the waste aggregate.

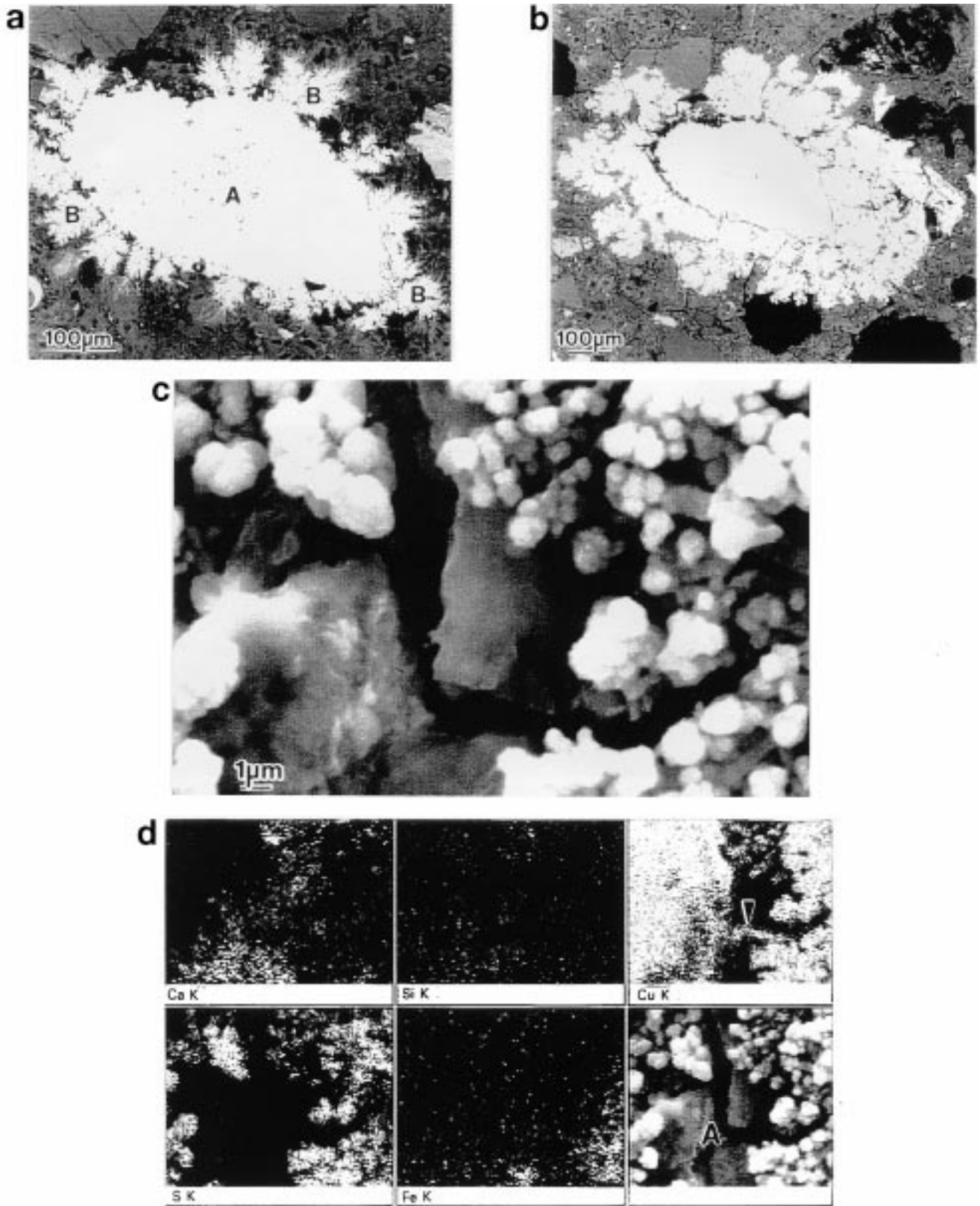


Fig. 4. Backscattered electron image of dendritic overgrowth of microcrystalline chalcantite "B" on waste aggregate "A", containing Cu after (a) 6 mo and (b) 2 yr, Treated Waste I; (c) scanning electron microscope image and (d) corresponding energy dispersive x-ray elemental map show movement of Cu within crack at location "A" on waste aggregate shown above.

als. The vulnerability of the treated wastes to chemical and physical attack depends to a large extent on factors such as permeability, chemical and mineralogical composition, and microstructure of the cement micromass, as well as the cement type and incorporated waste aggregates. After the treated wastes had undergone 6 yr of exposure to varying environmental conditions, including (i) on-site burial at four different locations, (ii) above-ground storage simulating landfill conditions, and (iii) archival storage under laboratory conditions, the nature and extent of physical and chemical weathering and degradation of the treated wastes was evaluated. Physical and chemical alteration processes identified included: freeze-thaw, carbonation, acid attack, alkali-aggregate reaction, and  $\text{SO}_4$  attack on the solidified and stabilized metal-bearing wastes. Aggressive solution attack on cement-based materials results in secondary mineralization that ultimately may decompose a concrete or treated waste structure, releasing toxic compounds. Although the extent of degradation of the treated wastes after 6 yr, as indicated by the micromorphic features observed in thin section, is considered only slight to moderate, the consequences are significant if one considers that concrete can disintegrate completely in 50 to 100 yr or less.

Future studies should incorporate the results of these findings into models that evaluate the durability and permanence of solidified and stabilized wastes on the basis of actual physical and chemical weathering and alteration changes that occur in the environmental setting in which the treated wastes are to be placed. Such models must be site-specific because environmental conditions of burial vary. However, it must be emphasized that such studies cannot be based on leaching and chemical analyses alone; the use of all levels of microscopic analyses must be included in such studies.

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