



Characterization study of heavy metal-bearing phases in MSW slag

Amirhomayoun Saffarzadeh^{a,*}, Takayuki Shimaoka^b, Yoshinobu Motomura^c, Koichiro Watanabe^a

^a Department of Earth Resources Engineering, Graduate School of Engineering, Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka 819-0395, Japan

^b Institute of Environmental Systems, Department of Urban and Environmental Engineering, Graduate School of Engineering, Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka 819-0395, Japan

^c Department of Earth and Planetary Sciences, Graduate School of Sciences, Kyushu University, 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581, Japan

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ABSTRACT

Slag products derived from the pyrolysis/melting and plasma/melting treatment of municipal solid waste (MSW) in Japan were examined for the characterization study of heavy metal-bearing phases using petrographic techniques. Detailed microscopic observations revealed that the shapes of heavy metal-rich inclusions are generally spherical to semi-spherical and their sizes range from submicron to scarcely large size spheres (over 100 μm). The experiments (both optical microscopy and electron probe microanalysis) indicated that Fe and Cu participate in mutual substitution and different proportions, and form mainly two-phase Fe–Cu alloys that bound in the silicate glass. This alloy characterizes the composition of more than 80% of the metal-rich inclusions. Other metals and non-metals (such as Pb, Ni, Sb, Sn, P, Si, Al and S) with variable amounts and uneven distributions are also incorporated in the Fe–Cu alloy. In average, the bulk concentration of heavy metals in samples from pyrolysis/melting type is almost six times greater than samples treated under plasma/arc processing. The observations also confirmed that slag from pyrolysis origin contains remarkably higher concentration of metallic inclusions than slag from plasma treatment. In the latter, the metallic compounds are separately tapped from molten slag during the melting treatment that might lead to the generation of safer slag product for end users from environmental viewpoint.

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1. Introduction

The incineration, melting and vitrification of municipal solid waste (MSW) has been seen as one of the most effective strategies in the MSW management in Japan. During the past three decades, several melting and vitrification technologies have come to operation and gained priorities over other treatment options nationwide. At present the total production capacity of melting plants in Japan amounts to 1500 tons/day of vitrified materials approximately.

It is believed that such treatment methods lead to a significant volume reduction and a destruction of >95% of dioxins. In addition, in some types of melting furnaces metals can be separated by evaporation or phase separation [1]. It is also expected that compared to nontreated residues, vitrified materials have much lower potential to mobilize metals [2]. However, the latter aspect has not been thoroughly investigated, particularly from the standpoint of characterization of hazardous metals.

Through literature survey, we understand that a great deal of attention has been given to the investigation on leach-

ing potential of toxic metals in ash and slag [3–8]. However, fewer studies have focused on the identification and characterization of metal-rich phases with respect to their spatial distribution, morphology and chemical compositions. Leaching studies are valuable analytical techniques for measuring the leaching behavior of the waste combustion residues, but they are largely limited to the evaluation of soluble compounds and provide little information about the insoluble compounds that may be present in the combustion products.

In this paper, it is intended to represent an ideal image of the characterization of heavy metal-bearing phases and their localization with respect to the glassy matrix of slag. During the observations, it was learnt that the slag residues are enriched in a number of heavy elements of environmental concern, including Zn, Cr, Cu, Pb and Ni as chief components [9].

Therefore, through the ongoing discussion, particular attention has been given to: (i) the preliminary identification of the heavy metal-bearing phases by using optical microscopy; (ii) the distribution pattern and morphology of those phases; and (iii) their chemical compositions. The latter two aspects were investigated using combined electron probe microanalysis (EPMA) and backscattered electron (BSE) imagery. To achieve the goals, slag

* Corresponding author. Tel.: +81 92 802 3311; fax: +81 92 802 3311.
E-mail address: a.saffarzadeh@yahoo.com (A. Saffarzadeh).

materials from: (1) pyrolysis gasification and melting, and (2) plasma torch-type incineration ash melting systems were selected for the characterization study of the heavy metallic compounds.

Such characterization understanding is required to evaluate the fate and transport of heavy metals from any sort of combustion products into a leaching medium, and to explore the possibility of controlling combustion process to ensure that the soluble forms of these metals in ash or slag products have minimized. A detailed knowledge of the phases can also lead to an improved understanding of the thermal processes.

2. Description of the melting processes

As mentioned earlier, slag materials from different technologies (pyrolysis melting and plasma melting systems) were selected for the characterization study of the heavy metallic compounds. The pyrolysis melting technology is an innovative pyrolysis gasification and melting process for the proper treatment of municipal waste. In this type of MSW melting treatment that operate under *semi-oxidizing* to *oxidizing* atmosphere, MSW-derived substances (pyrolysis char and gas) produced at a pyrolysis drum (reactor) is introduced into a high temperature combustion chamber (HTCC) and co-fired at a controlled temperature rate ($\sim 1300^\circ\text{C}$) to generate molten slag material (see [9–11] for further technical details). Slag and remaining metallic compounds are simultaneously tapped from the tapping hole of HTCC. It is therefore presumed that an appreciable amount of metallic compounds are preserved in the silicate matrix of slag.

The plasma melting technology is another treatment method that has been developed for detoxifying and vitrification of incineration ash produced by municipal waste incinerators. In this technology, high temperature condition ($>1400^\circ\text{C}$) provided by the system's plasma arc (nitrogen type), significantly scale down the ash volume, and treats hazardous constituents in the ash. Ashes of different types (bottom and fly ashes) are produced in an attached incineration facility prior to vitrification process. The prevailing condition during the treatment of ash is *reducing*. The reducing atmosphere in the plasma furnace causes the metals to be reduced to the elements, and sink down to form a layer of metallic alloy, or to be evaporated through the off-gas system (see [10,11] for further details).

Detailed knowledge about the melting processes applied for MSW in Japan has been published elsewhere [1,12–14].

3. Materials and methods

MSW slag products were obtained from four undisclosed pyrolysis/melting facilities (K, N, T and Y) from different geographic locations in Japan with the total capacity of 260, 210, 200 and 240 tons/day of fresh waste receiving from the surrounding municipal areas, respectively. Three suites of water-quenched (WQ) slag and two suites of air-cooled (AC) slag samples were collected from facility K at different periods. A pair of WQ and AC samples was obtained from facility N, while only WQ slag was received from facilities T and Y. Slag of plasma type origin (WQ) was sampled from facility S (undisclosed) with the total treatment capacity of 140 tons/day of incineration ash. The original feed material delivered to the entire facilities is the non-combustible fraction of MSW whose composition may somewhat vary on a daily or seasonal basis.

The standard petrographic and polished thin sections of a variety of collected samples were prepared for the preliminary identification of metal-bearing phases and their elemental associations. This was completed by a petrographic microscope (Nikon ECLIPSE E600WPOL) at different magnifications.

Backscattered electron and characteristic X-ray images were obtained by using a JXA-733 electron probe microanalyzer (EPMA-WDS), and the qualitative and quantitative spot analyses of the samples were conducted under accelerating voltages of 20 and 15 kV, respectively, with 0.1 μA electron probe current. Under such conditions, the minimum detection limit of EPMA technique is about 100 ppm for the existing heavy elements (e.g., Cu, Pb, Ni and most transition metals). Thus the quantitative results are obtainable in the order of several hundreds of ppm. BSE images were used to visually detect the dimension, distribution pattern and spatial positioning of metal-bearing phases within the glassy background of slag.

Selected samples were pulverized by an automated vibrating milling machine model CMT (TI-100) for bulk analyses. Semi-quantitative bulk chemical analyses of the powdered samples were completed by using a Rigaku RIX3100 X-ray fluorescence spectrometer (XRF).

Optical microscopy associated with BSE imaging and characteristic X-ray mapping provided a practical tool to determine the morphology, distribution pattern and chemical composition of the heavy metal-bearing phases and their relation with the surrounding materials. The detailed description of both heavy elements of environmental significance and the accompanying metals and non-metals has been provided with relevant to their positioning in the described phases.

4. Results and discussion

The bulk analyses of the slag products (pyrolysis and plasma types) revealed that they are silicate-based materials with variable amounts of other major, minor and trace elements. As shown in Table 1, SiO_2 , CaO and Al_2O_3 are the main constituents and can be considered as the basis of the silicate melt. Such products also contain appreciable amounts of heavy metals of environmental concern such as Zn, Cu, Cr and Pb (Table 1). The results obviously distinguish the differences between the individual and total contents of trace elements in MSW slag products (pyrolysis vs. plasma type). It is understood that the total amount of trace elements in the pyrolysis slag is significantly higher than the other.

Table 1
Representative bulk chemistry of three WQ slag samples from pyrolysis/melting facilities (K and Y) and one from a plasma-arc type (S)

Facilities	K ₁	K ₂	Y	S
Major elements (wt%)				
SiO ₂	43.06	38.60	39.58	42.33
TiO ₂	1.56	1.76	2.09	1.18
Al ₂ O ₃	13.67	12.51	13.54	19.54
FeO	4.31	6.12	4.50	0.51
MnO	0.16	0.18	0.20	0.09
MgO	3.44	3.64	4.02	4.37
CaO	22.53	23.72	26.04	23.36
Na ₂ O	5.38	6.01	4.46	5.47
K ₂ O	2.58	2.61	1.31	1.57
P ₂ O ₅	2.06	2.82	2.21	0.84
LOI	0.45	0.54	0.41	0.00
S	0.07	0.09	0.04	0.38
Cl	0.03	0.01	0.02	0.15
Total	99.29	98.60	98.42	99.81
Dominant trace elements (ppm)				
Zn	4582	6254	7465	367
Cu	1919	2411	2002	257
Cr	1088	1429	2055	184
Pb	1226	972	637	12
Ni	112	126	152	1

K₁ and K₂ present repeated sampling from facility K.

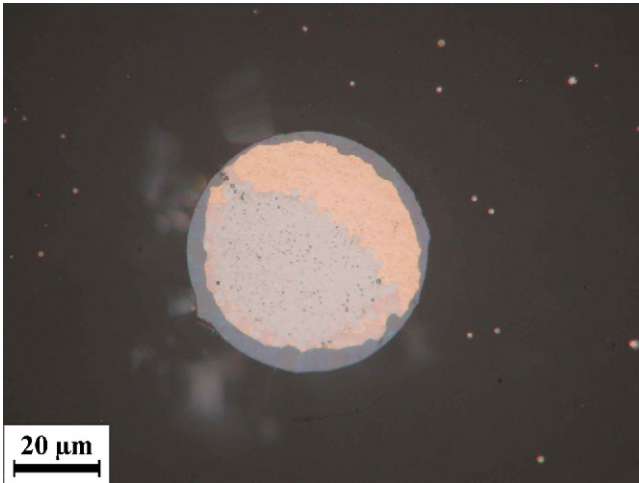


Fig. 1. Photomicrograph of a typical heavy metal inclusion in WQ slag (from facility T). Bright spots are minute inclusions and dark area is glass-reflected light.

4.1. Pyrolysis-type slag

Petrographic thin sections prepared from slag samples of pyrolysis origin indicated a vesicular glassy texture with visible thin-layered flow pattern. These observations in association with EPMA investigation showed that the glassy matrix of this type of slag (both WQ and AC) was essentially composed of low and high-silica glasses (typically 30% and 50% of SiO_2 , respectively) that are juxtaposed along sharp boundaries. Several mineral phases were also identified by petrographic studies. The major mineral phases are spinel (Zn- and Cr-rich), melilite, pseudowollastonite, tiny quench crystallites and heavy metal-rich inclusions that may reach up to 3% in total (see [9] for detailed petrography).

Comprehensive microscopic observations revealed that the shapes of heavy metal-rich inclusions are generally spherical (Fig. 1) to semi-spherical and their sizes vary chiefly from submicron to barely large size spheres (up to $100\ \mu\text{m}$ in diameter typically in AC samples). Larger inclusions are preferably located very close to or right on the margins of the vesicles (Fig. 2) due to the surface tension effect of the molten liquid. The BSE imaging (Fig. 3) indicates that the metallic inclusions are preferentially bonded in the high-silica glasses (bright spots in the dark bands); so that

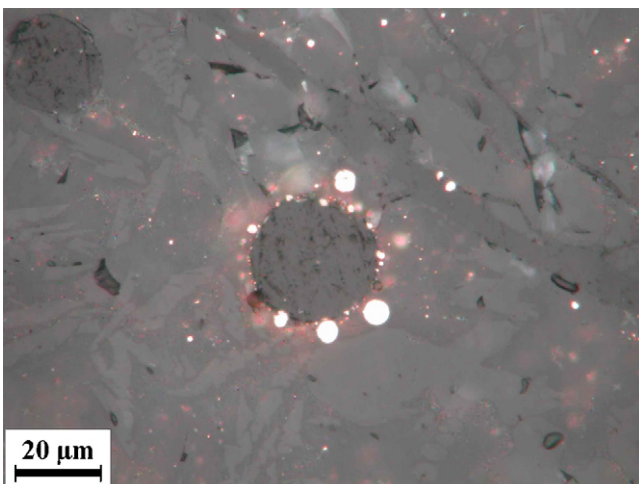


Fig. 2. Aggregates of metallic inclusions around a vesicle (center) in an AC slag sample (from facility N)-reflected light.

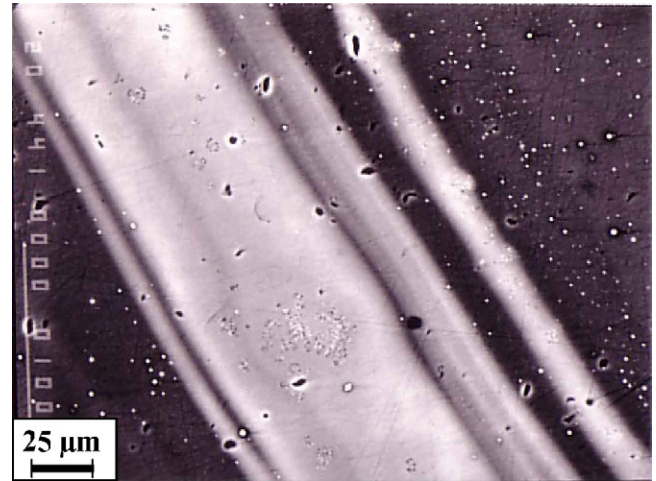


Fig. 3. BSE image of low (bright bands) and high (dark bands) silica glasses. Bright spots in the dark bands are metallic inclusions (WQ slag from facility K).

the low-silica glass (light bands) is almost depleted from such inclusions.

The experiments (both optical microscopy and electron probe microanalysis) indicated that Fe and Cu participate in mutual substitution and different proportions, and form mainly two-phase Fe–Cu alloys ubiquitously. This alloy characterizes the composition of more than 80% of the existing metal-rich inclusions. Additional metals and non-metals (such as Pb, Ni, Sb, Sn, P, Si, Al and S) with variable amounts and uneven distributions also incorporated in the Fe–Cu alloy.

The AC samples provided appropriate targets for qualitative and quantitative analyses. Fig. 4 represents a compositional BSE image from a typical Fe–Cu complex in an AC slag from the pyrolysis/melting facility (Y). The bright area (point 1) is chiefly composed of metallic Cu ($\sim 97\%$) with insignificant amount of Fe (Fig. 5), whereas the darkest zone (point 2) is almost entirely enriched in Fe. The intermediate zone (point 3) is Fe–Cu–Ni alloy (Fig. 5) for which the chemical formula of about $\text{Fe}_{0.85}\text{Cu}_{0.09}\text{Ni}_{0.06}$ may be proposed based on microprobe data.

A blow-up image from Fig. 4 (represented in Fig. 6) shows the scattered combinations of Pb–Cu–Sb in the Cu-rich zone. Pb has not been identified as individual metallic inclusion and is essentially

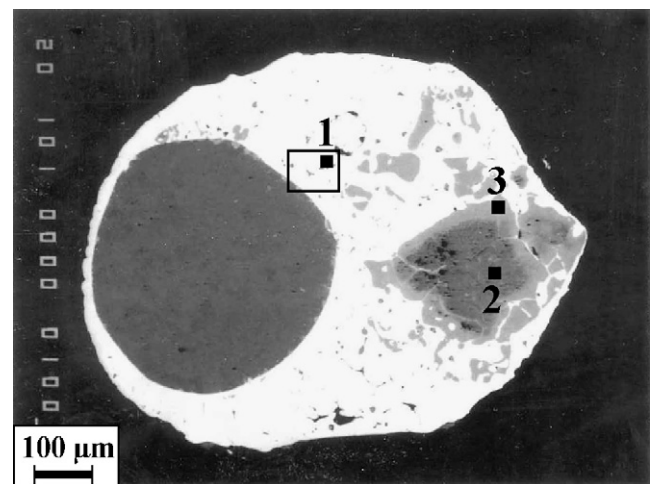


Fig. 4. BSE image of a complex metallic inclusion surrounded by interstitial glass (AC slag from facility Y). See text and Fig. 5 for detailed chemistry of points 1–3. The selected field is magnified in Fig. 6.

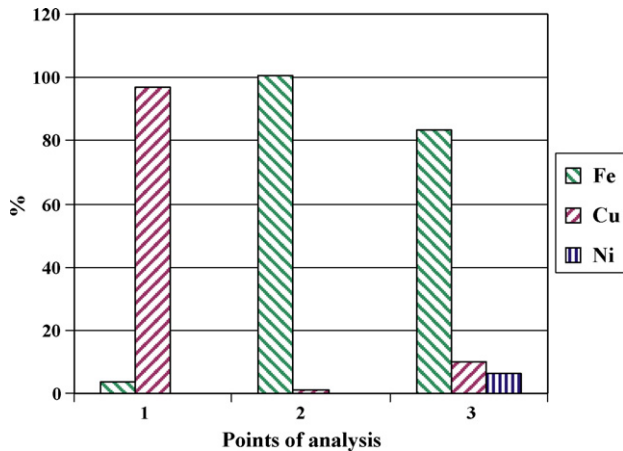


Fig. 5. EPMA results of points 1–3 as marked on Fig. 4.

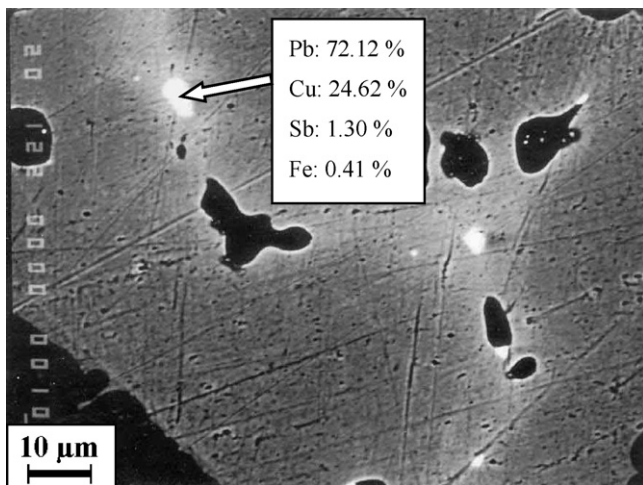


Fig. 6. BSE image of the selected field from Fig. 4 that represents the Pb-rich phase (bright areas) bound in the Cu-rich zone. Data on the image show the composition of the Pb-rich phase.

locked up as sporadic phases in the Cu-rich zone, occasionally associated with Fe and Sb (e.g., Fig. 6). Although Pb is expected to be volatile in air as chloride species, it partly exists as PbO having relatively high boiling point (1470 °C), and it partially vaporizes depending on its vapor pressure at all temperatures [15]. Hence, the remaining lead that is not vaporized is co-precipitated with other heavy metals to form complex metallic compositions in the metal inclusions. The electron probe microanalysis of the Pb–Cu-rich alloy in Fig. 6 (results shown on the image) yields the stoichiometric formula of approximately $Pb_{0.47}Cu_{0.51}(Fe,Sb)_{0.02}$ for this phase.

Cadmium (Cd), as an environmentally hazardous element was not identified by bulk and EPMA analyses and it is almost impossible to make a statement about the speciation and distribution of this element. This can be attributed to the strong volatility effect of Cd [16,17] due to low boiling point (~765 °C) and partly due to the detection limits of the employed techniques.

In this melting system (more oxidizing), sulfur is not commonly identified in the inclusions, as most of it is expected to volatilize and release from the HTCC as gaseous species through the off-gas system. However, because of complex melting condition, trace amount of sulfur might survive and reside in the melt. In such a case, it was realized that it is strongly in bond with Cu and partly with Fe to form sulfide species in the main part or preferably in the rim of the inclusion. Fig. 7a exhibits the BSE image of a Cu–S rich inclusion. Fig. 7b (characteristic X-ray image) shows the homogenous distribution pattern of S in the inclusion, which is consistent with that of Cu (Fig. 7c). The brighter spots (Fig. 7a) display the Ni–Sb–P-rich blebs. The microprobe analysis yields the chemical formula of Cu_2S for the Cu–S phase. Regarding Cu, in general the majority of this element has been identified as pure Cu (or alloyed with other metals), and at most 5% might exist as sulfide inclusions whose characterization may be due to a moderately local reducing condition in the HTCC.

Zn and Cr show particular behaviors. Despite elevated concentration of Zn in the bulk composition of pyrolysis-type slag (K and Y, Table 1), the microanalytical studies demonstrated that a significant amount of Zn has been markedly incorporated into the existing spinel crystals and in lesser amount into the glassy matrix itself. The EPMA data also indicate the incorporation of small amounts of Zn into the existing melilite. In spite of relatively high concentration of Cr in the bulk composition of pyrolysis slag (Table 1), the

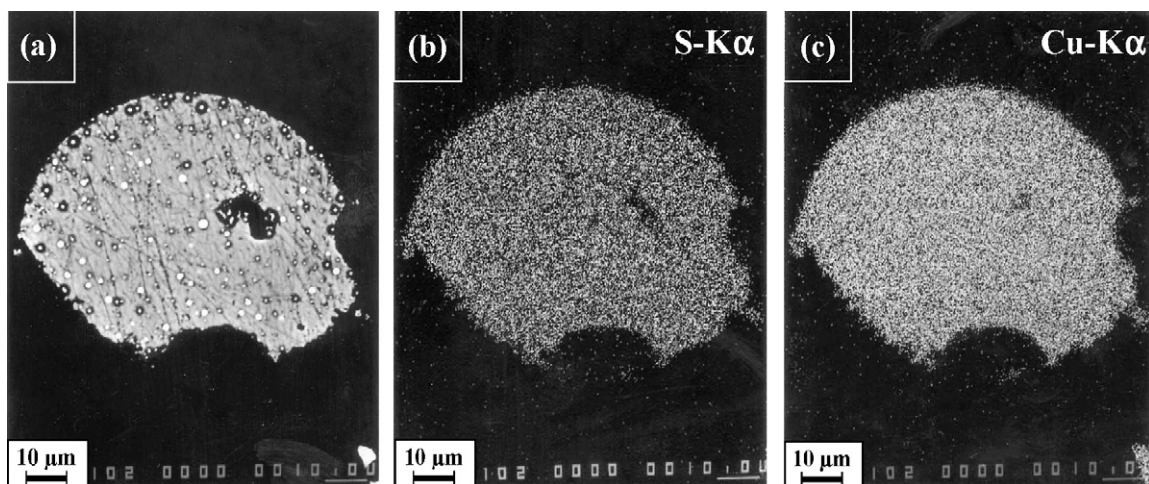


Fig. 7. Backscattered electron image (a) and characteristic X-ray images (b and c) of a metal-rich inclusion within the background glass of a WQ slag (from facility K). (b and c) Represent the distribution pattern of sulfur and copper, respectively.

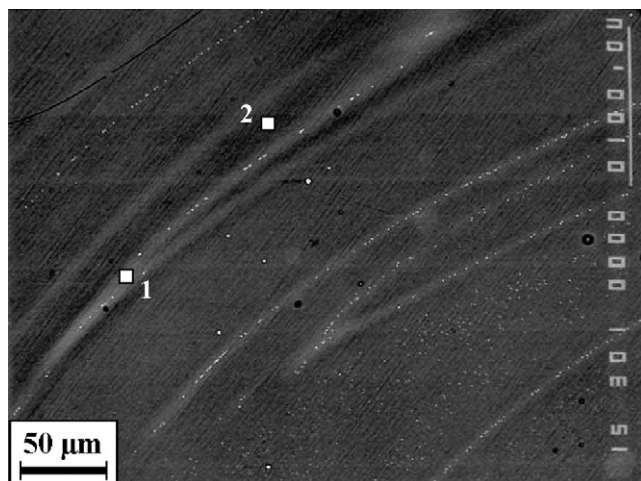


Fig. 8. BSE image from the glassy matrix of slag from plasma-arc system. Analytical results of points 1 and 2 are listed in Table 2.

EPMA data acquired from several targets (both glass and minerals) demonstrated that Cr has the least compatibility with silicate glass and metallic inclusions; majority of which has been incorporated into the Cr-rich spinels. As a consequence, there remains very insignificant amount of Zn and Cr in metallic state to contribute to the characterization of metal-rich inclusions [9].

4.2. Plasma-type slag

The bulk chemistry of a characteristic sample (WQ) from facility S was measured by XRF identical to the pyrolysis samples (K and Y). Table 1 displays the differences of the major, minor and trace elements between the two suites of samples (pyrolysis vs. plasma). In facility S, slag and metal alloys are tapped separately during the melting treatment, and as a consequence the concentration of heavy metals has significantly diminished in slag product. In average, the concentration of heavy metals in K and Y samples is almost six times greater than sample S (1.34 vs. 0.23 wt%).

Minute metal inclusions were examined in several petrographic thin sections. Most of them are too small (normally at the sub-micron size) to be analyzed quantitatively by EPMA without interaction effect from the surrounding glass (Fig. 8). The larger inclusions however were analyzed qualitatively. Results demonstrated that they essentially consist of Fe–Cu phase (identical to pyrolysis-type slag) generally bound with sulfur to form metal-sulfide inclusions.

A reducing environment in the plasma furnace can be assumed due to the presence of additional sulfur comparing with K and Y samples (Table 1). However, sulfur is not thermodynamically stable in the slag of any commercial combustion or gasification process; consequently, it will completely volatilize and pass out of the system, except for the small fraction that reacts to form stable metal sulfates or sulfides [18].

Chlorine also shows slightly elevated concentrations (Table 1) comparing with pyrolysis-type slag. Very likely during plasma treatment as a result of prevailing reducing condition, the majority of heavy metals with relatively low boiling point (such as Cd, Zn, Hg and Pb) does not form chloride compounds and mostly vaporize as metallic state. As a result, slightly additional chlorine may still be preserved in the molten material. However significant amount of chlorine content is expected to discharge as gaseous phase from most MSW high temperature treatment systems.

Table 2

Representative electron probe microanalyses of matrix glass as marked on Fig. 8

	Point 1	Point 2
SiO ₂	41.43	45.06
TiO ₂	1.06	0.97
Al ₂ O ₃	19.94	20.67
FeO	0.35	0.28
MnO	0.00	0.00
MgO	3.11	2.99
CaO	21.67	20.08
Na ₂ O	5.74	5.56
K ₂ O	1.57	1.67
Total ^a	94.87	97.28

^a Low total value for each analysis point is due to the non-measurement of minor constituents.

Likewise, large part of present Fe very likely exists as divalent iron due to the reducing atmosphere. However, comparing the amount of Fe-rich inclusions and low concentration of bulk Fe in sample S (Table 1), the majority of Fe must have been reduced to form the present metal-rich inclusions instead of being incorporated into the glassy matrix. Representative microprobe analysis of selected glasses (from Fig. 8) is indicative of further chemical homogeneity in terms of major constituents (Table 2).

The samples from this treatment technique appear to be more homogeneous with considerably lesser amount of toxic elements. The inclusions made up less than 0.5 vol.% of the slag material. No other crystalline phase such as spinel was found in the studied samples. It is therefore hypothesized that the present heavy metals must have been incorporated into the glassy matrix (e.g., Zn) or coexisted with the metal-rich inclusions (Fe, Cu, Pb).

4.3. Discussion

High-temperature treatment of MSW concentrates the original, nonvolatile inorganic elements into a solid slag with a composition that is variably dominated by Si, Ca, and Al. This typically leads to the development of a slag phase consisting of Si-rich glass matrix with immiscible droplets of metallic iron (rarely sulfide) and/or euhedral crystals of spinel and/or silicates. MSW-based slags show irregularity with regard to some elements (major and minor) and exhibit a much more complex array of phases.

Petrographic thin section study combined with EPMA provided valuable approaches in order to locate, identify, and determine the chemical composition of heavy metal-rich phases in slag products. In order to evaluate such characteristics at different treatment atmospheres, molten slag materials from pyrolysis/melting (moderately oxidizing) and plasma arc/melting (reducing) processes were studied in detail.

The investigations show that the plasma melting process generates a more homogeneous product with noticeably lower heavy metal concentration as a result of reducing atmosphere in the high temperature furnace and gravity settlement of the heavy metal fraction and its separate tapping from slag compared to pyrolysis/melting process. In average, the bulk concentration of heavy metals in samples from pyrolysis/melting type is almost six times greater than samples treated under plasma/arc processing. The BSE images and X-ray maps ascertained that some heavy metals such as Cu (regularly bonded with Fe) and Pb significantly partition into metallic alloys or metal-sulfide inclusions with variable concentrations of other metals and non-metals such as Sb, Ni, Si and P, whereas elements such as Zn and Cr have been extensively incorporated into the melt glasses or minerals.

Such metallic complexes were the most important part of the conducted research, especially with regard to their long-

term leaching potential and their environmental sustainability. For instance, in the case of existing Pb, care should be taken about its speciation. Because the solubility of Pb-oxide is very high (around 40 mg/l), even a small amount of oxidized lead, might contribute to the amount of lead released [5]. In addition, the existence of contiguous pores (vesicles) may allow a leaching medium to contact heavy element-containing phases distributed within the matrix.

It appeared that under oxidizing atmosphere, the majority of heavy metals have been fixed into the silicate matrix by different reactions as a result of the prevailing complex conditions. In contrast, under reducing atmosphere, volatile metals have been significantly extracted as gaseous species (e.g., Cd, Pb, Zn, Hg and their salts) or separated and tapped from the melt according to their specific gravity (e.g., Fe, Cu, Ni).

Although the redox condition is considered as the crucial factor in controlling the behavior of heavy metals in various melting systems, attention should be paid to the effective role of additional factors. The interaction of other major and minor constituents, treatment temperature, melt flow speed, density and viscosity of the molten material, the presence of scavengers such as halogens, melt residence time in the furnace and the overall structure of each plant certainly affect the fate and transport of the heavy metals. The influences of the entire factors need to be investigated in further details for generating more homogenous and inherently safer slag products.

5. Conclusions

The current research provided advantageous knowledge from the characteristics (morphology, distribution and composition) of heavy metal-bearing phases in MSW slag products using optical microscopy and microanalytical technique. In order to evaluate such characteristics, molten slag from pyrolysis/melting and plasma arc/melting processes were studied in detail.

In the pyrolysis treatment, considerable amount of heavy metallic compounds are retained in the glassy matrix, whereas during plasma melting treatment most of heavy metals are reduced into metallic state. This favors easier evaporation and/or precipitation of significant amount of metal species from the system through either off-gas (e.g., Pb, Zn, Cd) or separate tapping from the melt (e.g., Fe, Cu, Ni) into smaller fraction of heavy metal condensates.

Knowledge from the current study verified that a large quantity of Zn and Cr partition into spinel and to a minor extent into melilite and glass phases of slag products processed by pyrolysis/melting technique (under oxidizing condition). Under such condition, a notable amount of other heavy metals (Cu, Fe, Pb, Sb, Ni) are significantly enriched in metal-rich inclusions and preserved in the silicate glass matrix. In contrast, slag from a system operating under reducing atmosphere (plasma), exhibited more homogenous matrix and lower bulk concentration of heavy metallic compounds. In average, the concentration of heavy metals in slag from pyrolysis/melting system was almost six times greater than slag treated by plasma with separate slag/metal tapping.

Experimental evidences from this research combined with results from other investigations may allow the design of a thermal

process that can effectively eliminate hazardous contents from a toxic residue (e.g., fly ash, char) by alternative reducing/oxidizing atmospheres (even without vitrification). Such processes can produce safer glassy residues and a small heavy metal condensate (metallic alloy) that should be suitable for metal recycling, if economically and technically feasible. Therefore, care should be taken about the selection of the most reasonable techniques (oxidizing vs. reducing) for the thermal treatment of MSW with regard to any environmental and economic prospects.

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