



Chemical Analysis of Welding Fume Particles

Airborne particle size is the most important factor in determining the accuracy of a method for chemical analysis

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ABSTRACT. Welding fume contains elements that, in their pure forms, can be hazardous to worker health if inhaled or ingested. Therefore, the chemical composition of welding fume must be examined when considering fume toxicity. Various chemical analysis techniques are presented and their applicability to airborne particles is described. Knowledge of particle size is important because a given characterization technique only provides accurate data for a specific size range. For the purpose of comparison and illustration, this paper uses several characterization techniques to analyze the chemistry of mild steel welding fume. X-ray diffraction (XRD) shows that mild steel gas metal arc welding (GMAW) fume is predominantly magnetite while the mild steel shielded metal arc welding (SMAW) fume contains both a mixed alkali fluoride phase and a Fe-Mn oxide spinel phase. Energy dispersive spectrometry (EDS) was found to be an effective technique for evaluating the elemental composition of welding fume, and the results of the elemental analyses of a few welding fumes are reported.

Introduction

Although it is almost impossible to compare enough iron oxide to cause a toxic effect (Ref. 1), steels contain alloying elements that, in their pure forms as found in other industries, could be hazardous to worker health if inhaled or ingested. All steels contain manganese while stainless steels also contain chromium and nickel. Although essential for health in small

doses, pure manganese is a neurotoxin that can cause manganese poisoning in large doses. Chromium and nickel can be carcinogenic. Welders are exposed to these elements if they inhale welding fume. Welding fume consists of metal oxide particles that form during welding. These particles are small enough to become and remain airborne and are easily inhaled.

Chromium, nickel, and manganese are not found as pure elements in welding fume. They are present as impure compounds, which do not present the same toxic risk as pure elements. The oxidation state of chromium and manganese also affects their toxicity. Trivalent chromium is inert, whereas hexavalent chromium can be carcinogenic. There is evidence that the oxidation state of welding fume manganese is less toxic than that used to set manganese exposure limits (Ref. 2). Therefore both the elemental and chemical composition of welding fume must be considered when assessing fume toxicity.

Information about the chemistry of mild steel welding fume can be used to compare the predicted toxicity of welding fume to that of elements found in other industries. It can also be used as an example of the methods used to study particle chemistry in general.

When analyzing airborne particles like

those in welding fume, the most important factor to consider is particle size. This is because a given characterization technique only provides accurate data for a specific size range. Many previous studies of particle chemistry do not take this fact into account when reporting chemical composition, and thus some of these studies present misleading conclusions. This has been fairly common in welding fume literature, particularly with regard to studies involving energy dispersive spectroscopy with scanning electron microscopes (SEM-EDS). For example, it is inaccurate to report compositional gradients measured with SEM-EDS across fume particles that are smaller than half a micrometer, because the SEM beam penetrates and samples the volume of such small particles rather than just their surface. Welding fume particles range in size from 0.005 to 20 μm , although less than 10–30% (depending on the welding process) of the fume mass is larger than 1 μm (Ref. 3). Therefore, it is important to carefully consider the techniques for analyzing welding fume.

Methods

Chemical Analysis of Particles

Table 1 lists the various analysis techniques that can be used to characterize welding fume (and other particulate matter). The information provided and the particle size ranges for which these techniques are applicable vary considerably. For further details, Refs. 2 and 4–6 contain reviews of the various techniques used to chemically characterize particles.

In practice, there are two types of elemental characterization techniques: those that measure proportional to the atomic number and those that measure proportional to the atomic mass. The former proportional

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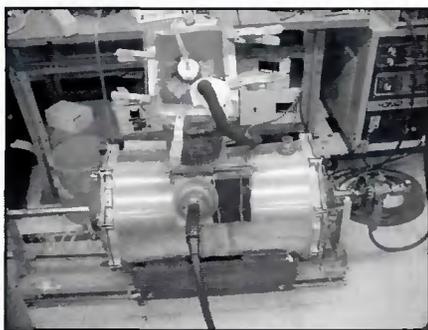


Fig. 1 — Welding fume collection chamber.

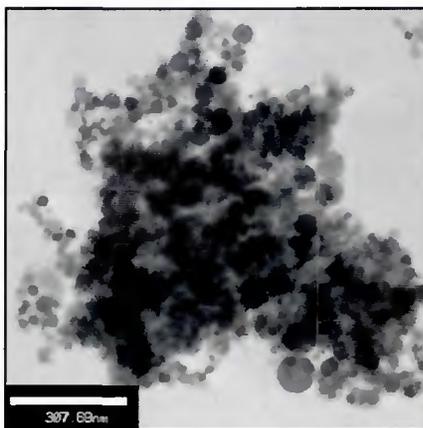


Fig. 2 — Transmission electron micrograph of fume created from 2%O₂-Ar-shielded gas metal arc welding with 0.045-in. mild steel ER70S-3 wire at 30 V and ~200 A.

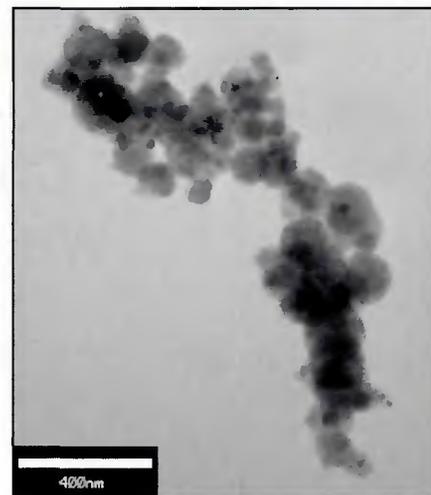


Fig. 3 — Transmission electron micrograph of fume created from shielded metal arc welding with 0.094-in. mild steel E7018-A electrode at 70 A.

duce data easily transformed into molar/atomic fractions, whereas the latter create data reported as weight percentages. If one has reliable values for each element present in the specimen being analyzed, then one can convert from atomic fraction to weight percent and back. However, no analysis technique is able to measure every element with the same accuracy, so inconsistencies exist when comparing the results of different tech-

niques. When this happens, conversion between weight and atomic percentages is only approximate, because the entire mass is not equally characterized. Therefore, the elemental data from multiple tech-

niques cannot be compared, except within the two major groupings presented herein.

The techniques that report mass in-

Table 1 — Chemical Composition and Size Characterization Techniques for Particles

Characterization Method	Size Range (µm)	Detection Limit (NA = not applicable)	Notes
<i>Particle Size Distribution</i>			
Impactors (various types)	0.1–20	NA	Size distribution by mass chemically analyze size groups
Electric aerosol analyzer (EAA) and differential mobility particle sizer	0.01–1	NA	Size distribution by number
Acrodynamic particle sizer	0.1–25	NA	Size distribution by number
Scanning electron microscope (SEM); high resolution (HRSEM)	0.5–50 0.002–1	NA NA	Particle sizes can be measured from micrographs
Electron probe microanalysis (EPMA)	0.5–50	NA	Particle sizes can be measured from micrographs
Transmission electron microscope (TEM)	0.001–1	NA	Particle sizes can be measured from micrographs
Light microscopy	1–400	NA	
<i>Elemental Composition</i>			
X-ray fluorescence spectrometry (XRF)	bulk	100 ppm	Atomic numbers > 10 very fast
Neutron activation analysis (NAA)	bulk	0.01%	Atomic numbers > 10, requires nuclear reactor
Optical emission spectrometry and mass spectrometry	bulk	1–10 ppm	Atomic numbers > 10
Atomic absorption spectrometry (AAS)	bulk	10 ppm	
Energy-dispersive spectrometry with SEM (SEM-EDS)	1–50	0.1%	Atomic numbers > 10
Wavelength-dispersive spectrometry with EPMA (EPMA-WDS)	1–50	0.1%	Atomic numbers > 4
Energy-dispersive spectrometry with TEM (TEM-EDS)	0.01–0.5	0.1%	Atomic numbers > 5 scanning TEM can map element distribution at nm resolution
Proton-induced X-ray emission spectrometry (PIXE)	>5	0.1%	Atomic numbers > 10
Laser microprobe mass spectrometry (LAMMS)	>1	10 ppm	All elements
Secondary ion mass spectrometry (SIMS)	>5	10 ppm	Light element capable
Auger electron spectrometry (AES)	>0.1	0.1%	Atomic numbers > 3 lower sample must be conductive
X-ray-induced photo-electron spectrometry (XPS or ESCA)	>5	0.1%	Surface composition (3–5 nm deep) contamination error common
<i>Chemical Speciation</i>			
X-ray diffraction (XRD)	bulk	NA	Only of crystalline material; particles must be >0.05 µm or they will seem amorphous
X-ray-induced photoelectron spectrometry (XPS or ESCA)	bulk	NA	Need appropriate standards Collect on noninteracting filter
Selected area electron diffraction with TEM (TEM-SAED)	~0.3	NA	Only of crystalline material

stead of molar quantity are also those with the more frequent inaccuracies in reporting data. For example, iron and aluminum cannot be determined with great accuracy with inductively coupled plasma mass spectrometry (ICPMS) when in high concentrations (greater than 5 wt-%). The bags used to contain the sample during neutron-activated analysis (NAA) often contain aluminum, which distorts the analysis of that element. The acids used to dissolve material for emission spectrometry/spectroscopy often cannot dissolve everything, so such analyses are often incomplete. The accuracy of X-ray fluorescence spectrometry (XRF) and neutron activation analysis (NAA) is dependent on the standards used to calibrate the measurements. Therefore each of these methods has limitations.

When energy-dispersive spectrometry (EDS) is used with transmission electron microscopy (TEM), the resolution of the microanalysis should be considered. Because of electron interactions with the material (i.e., beam broadening), the spatial resolution decreases with increasing atomic number. Thus, it may not be possible to measure the change in composition in a very small area. However, the thinner the sample analyzed, the less beam broadening will occur. For a review of the limitations involved in electron microscopy, see Ref. 7.

These weaknesses are in addition to the inability of nearly all techniques to accurately analyze any elements lighter than sodium. Even when a method can analyze elements as light as carbon, problems like water condensation on the filament (as in transmission electron microscopy) or surface contamination with carbon dioxide

(as with X-ray-induced photoelectron spectrometry) can obscure the presence of elements such as oxygen or fluorine. High-powered electron beams can also break down organic contaminants, causing a film of carbon to deposit that obscures the sample. Because welding fume always contains oxygen, lack of information about oxygen concentration makes it impossible to determine the entire molar/mass quantity of the fume.

Therefore the most convenient way to analyze welding fume is by the molar fraction of the metal cations. Since energy dispersive spectrometry (EDS) and X-ray-induced photoelectron spectrometry (XPS) report data in this form, these techniques are well suited for welding fume analysis, especially when one considers the respective size limitations with respect to ultrafine particles.

Since welding fume can be collected as a powder, it is easily prepared as a bulk material, e.g., for X-ray diffraction (XRD) analysis; it only becomes difficult to prepare if one wishes to analyze individual discrete particles, although for transmission electron microscopy, the small sizes aid sample preparation enormously in comparison with samples that must be thinned by ion sputtering and similar processes.

Therefore, there are many studies using X-ray diffraction and electron microscopy to analyze welding fume particles (Refs. 8-17). There are also several that use surface characterization techniques, mostly XPS (Refs. 18-24). There are a few that use multiple techniques (Refs. 2, 25-30) for a complete welding fume analysis.

For the purpose of comparison and il-

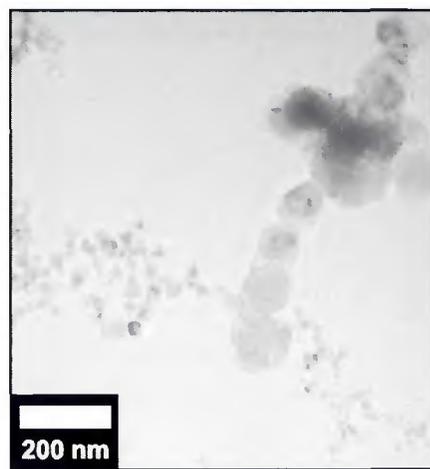


Fig. 4 — Transmission electron micrograph of fume created from self-shielded flux cored arc welding with 0.045-in. mild steel E71T-GS wire at 30 V and ~170 A.

lustration, this paper uses several characterization techniques to analyze the chemistry of mild steel welding fume. These include inductively coupled mass spectrometry (ICPMS), X-ray fluorescence spectrometry (XRF), neutron activation analysis (NAA), X-ray-induced photo-electron spectrometry (XPS), X-ray diffraction (XRD), and energy dispersive spectrometry with scanning and transmission electron microscopy (SEM-EDS, TEM-EDS).

Fume Collection

The fume for this study was collected in a chamber (Fig. 1) designed by Prof. Gael Ulrich and coworkers at the University of

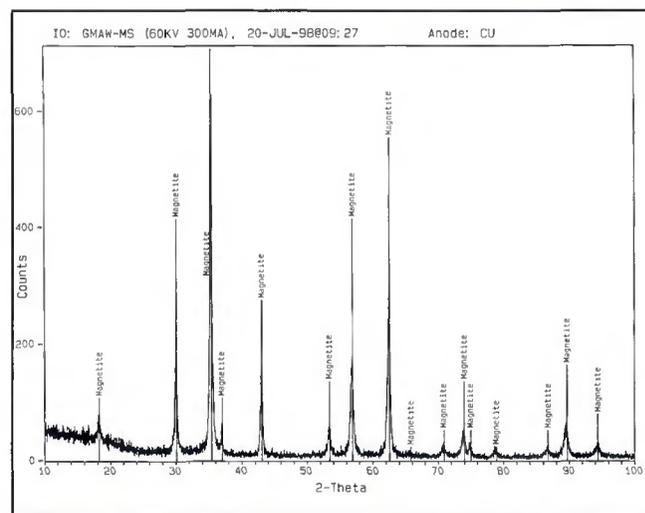


Fig. 5 — X-ray diffraction spectrum of fume created from 2%O₂-Ar shielded gas metal arc welding with 0.045-in. mild steel ER70S-3 wire at 30 V and ~200 A.

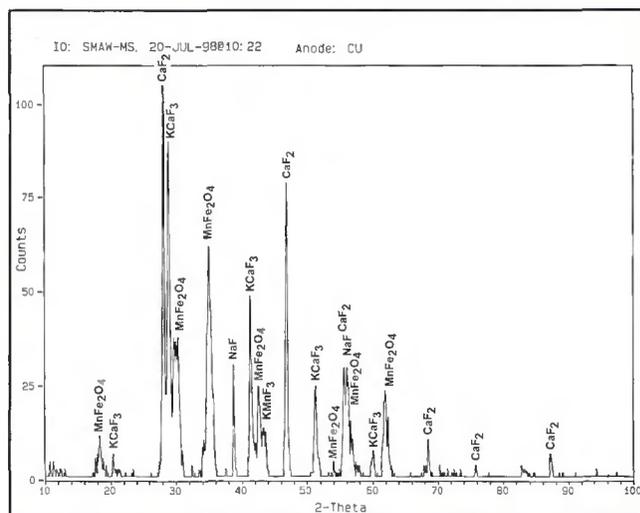


Fig. 6 — X-ray diffraction spectrum of fume created from shielded metal arc welding with 0.094-in. mild steel E7018-A electrode at 70 A.

Table 2 — Parameters for Welding Fume Generation

Welding Parameter	Condition
Welding Travel Speed	~16 mm/s (14 in./min)
Contact Tip to Work Distance	~19 mm (0.75 in.)
Electrode Angle	~10 degree drag angle
Wire Diameter	0.094 in. (2.4 mm) (SMAW) or 0.045 in. (1.2 mm) (GMAW, FCAW)
Shielding Gas Flow Rate	unshielded (SMAW, FCAW) or 16.5 L/min (35 CFH) (GMAW)
Shielding Gas Composition	unshielded (SMAW, FCAW) or Ar-2% O ₂ (GMAW)
Work Piece Composition	A500 carbon steel
Electrode Designations	E7018-A (SMAW) ER70S-3 (GMAW) E71T-GS (FCAW)
Wire Feed Speed (wfs)	noted
Voltage	positive electrode; magnitude noted
Current	steady; magnitude noted

Table 4 — Composition of the Metal Cations in Bulk Welding Fume with Techniques Based on Atomic Numbers

Mole Fraction of Cations Only	SEM-EDS	TEM-EDS ^(a)	XPS
2% O ₂ , Ar-shielded GMAW with 0.045-in. mild steel ER70S-3 wire at 30 V, ~200 A			
Fe	0.877	0.887	0.767
Mn	0.098	0.104	0.233
Si	0.025	0.008	<0.01
SMAW with 0.094-in. mild steel E7018-A electrode at 70A			
Fe	0.270	0.280	<0.01
Mn	0.099	0.103	0.052
Si	0.095	0.057	0.131
K	0.276	0.295	0.575
Ca	0.251	0.252	0.241
Na	0.011	0.013	<0.01

(a) Compositional average of 3 fume agglomerates
Although not listed, oxygen is present in all fume, and fluorine is present in SMAW fume.

New Hampshire. The design is published in a thesis and a paper (Refs. 31, 32) in which an excellent critique of fume collection methods can also be found.

A weld was laid on a cylindrical pipe about 25 cm (10 in.) in diameter and 30 cm (12 in.) long. The pipe rotated inside a chamber that moved horizontally around the pipe. An arc welding gun was inserted into the chamber through a hole so that a weld bead could be laid on the pipe forming a spiral as the pipe rotated and as the chamber moved transversely. The power source was a primary inverter machine current-rated at 500 A/40 V at 100% duty cycle.

Negative pressure was applied to the chamber so that fume could be collected while the pipe was welded, and shortly thereafter. This pulled the fume out of the chamber at a rate of ~2 L/min (4 ft³/h) through filters (0.2 μm Corning [Nucleopore] PC filter, 37 mm diameter) stationed approximately 30 cm (12 in.) above the arc. The fume was then removed from the filters for analysis. Table 2 lists the welding parameters used.

Results and Discussion

Figures 2–4 show TEM images of welding fume agglomerates. Tables 3–6 and Figs. 5 and 6 contain chemical data from welding fumes collected from mild steel electrodes using shielded metal arc welding (SMAW), gas metal arc welding (GMAW), and flux cored arc welding (FCAW).

The results from various chemical characterization techniques can be compared. Despite the extremely different resolution of the two types of electron microscopy as shown in Table 4, and despite the much larger sampling of particles in SEM-EDS, the compositions reported by TEM-EDS and SEM-EDS are very similar for both GMAW and SMAW fumes.

X-ray-induced photoelectron spectrometry (XPS) reports quite a different composition. There are two possible reasons. XPS probes only the top few nanometers of the surface of the analyzed material. So, as previous research on welding fume with XPS has reported, the surface of the individual fume particles may

Table 3 — Composition of GMAW and FCAW Wire Used To Create Welding Fume

wt-%	ER70S-3 GMAW mild steel	E71T-GS FCAW mild steel
Fe	98.04	91.69
Cr	0.046	0.190
Ni	0.038	0.028
Cu	0.160	0.002
Co	0.000	0.001
Mn	1.162	0.731
Si	0.551	0.270
Al	0.000	3.143
Ti	0.000	0.001
Zr	0.000	0.000
Mo	0.005	0.002
K	0.000	0.025
Na	0.000	0.007
Ca	0.000	0.340
Mg	0.000	0.941
Ba	0.000	2.463
O	0.000	0.160
F	0.000	<0.001
CL	0.000	0.001

Measured with emission spectrometry and inert gas fusion.

be enriched in the more volatile elements, like manganese or the alkali metals, forming core-shell particle morphologies (Refs. 18–24). This would be supported by the presence of multiple phases as reported by the XRD. However, although it is possible that the surface of welding fume particles is enriched in certain elements, it is doubtful that XPS can detect this phenomenon in nanoparticles. As noted in Table 1, XPS is only effective analyzing the surfaces of individual particles larger than 5 μm. When it is used to analyze the bulk chemistry of particles that can be as small as 5 nm, it is not clear what surface XPS is analyzing, because the XPS beam could be interacting with multiple particles. Therefore XPS is not a reliable analysis technique for the bulk or surface chemistry of ultrafine particles. It does, however, provide data on chemical bonds that can be used to determine the valence states of the elements it detects.

Another possibility for the difference in composition is that XPS analysis may preferentially report the composition of large particles, those near 5 μm in diameter. It is possible that liquid weld slag forms micrometer-sized spatter droplets, or “microspatter,” more readily than liquid metal does (Ref. 3). Slag microspatter contains more manganese and silicon than microspatter formed from liquid metal. The larger particles in welding fume would therefore have a composition more like that reported by XPS than by EDS. This theory could be tested by separating fume in size groups and analyzing separately with XPS or other analytical techniques.

Table 5 — Composition by Weight of Bulk Welding Fume with Techniques Based on Atomic Masses

wt-%	ICPMS ^(a)	XRF	NAA
2% O ₂ Ar-shielded GMAW with 0.045-in. mild steel ER70S-3 wire at 30 V, ~200A			
Fe	37.8	55.0	NA
Mn	8.8	12.8	NA
Si	NA	NA	NA
Cu	0.9	1.1	NA
Self-shielded FCAW with 0.045-in. mild steel E71T-GS wire at 30 V, ~170A			
Fe	18.1	23.7	32.4
Mn	0.5	2.7	1.6
Ba	NA	NA	9.8
Mg	NA	NA	8.8
Al	56.3	NA	(b)

(a) % SMAW fume undissolved in acid: 25
 (b) aluminum present strongly but obscured by aluminum in sample bag.
 NA = not analyzed because of experimental difficulties.
 Oxygen and fluorine were also not analyzed and are not listed here because they are anions.

Thus, of the three analysis techniques reported in Table 4, it is probable that SEM-EDS and TEM-EDS provide the most accurate measure of the mole fraction of the elements in the fume.

The atomic-mass-based analysis techniques listed in Table 5 yielded a fairly broad set of data. It should again be emphasized that these data cannot be compared with the previously discussed data from atomic-number-based analysis techniques. As noted, without data for each element present in the fume (and, as noted, the data for oxygen and other anions are usually lacking) one cannot convert between molar fraction and weight percent. Secondly, the weight percent reported here represents the total fume, not just the metal cations, as in Table 4. Also, these methods can be imprecise because of calibration sensitivities. Nevertheless, the values for manganese from reports by other researchers (Refs. 33–35) vary between 1 and 15 wt-% of the total welding fume, hence, the data presented herein are believed to be typical.

From the XRD spectra, it is clear that the GMAW fume is predominately magnetite (an oxide spinel phase dominated by iron, but which can contain manganese impurities), while the SMAW fume contains both a complex alkali-alkali earth fluoride phase and an oxide spinel phase containing a mixture of manganese and iron. This agrees with previous XRD studies of welding fume (Refs. 8–17). Voitkevich (Ref. 23) used X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD), while Minni et al. (Ref. 24) used electron spectroscopy to study welding fume. They concluded that Mn²⁺, or Mn³⁺, as opposed to Mn⁴⁺, are the most probable oxidation states of manganese in

Table 6 — X-ray Diffraction of Mild Steel GMAW and SMAW Fume

	Chemical Phases Detected	Approximate Crystalline Amount of Sample (%)	Approximate Amorphous Amount of Sample (%)
GMAW (ER70S-3, 30 V 300 in./min)	Fe ₃ O ₄ (spinel)	64	36
SMAW (E7018-A 3/32 in., 70A)	(K, Na, Ca, Mn) F ₂ (fluoride) MnFe ₂ O ₄ (spinel)	71	29

See Figs. 5 and 6 for X-ray diffraction spectra

welding fume. This also agrees with the XRD data presented herein. Manganese in welding fume is present as part of the spinel phase. Such phases contain metal ions (e.g., predominately iron mixed with the manganese) that are +2 or +3.

The amorphous fraction reported by X-ray diffraction may be generated by small nanoparticles in the welding fume because all the atoms in particles smaller than 50 nm are essentially on the surface, and therefore cannot form standard crystal lattices. Such particles are essentially amorphous.

Previous X-ray diffraction studies have reported many phases in welding fume and have suggested that each single fume particle contained three or more phases. This is obviously not the case for ultrafine particles; a particle less than a micrometer in diameter cannot contain many phases because its small volume will homogenize very rapidly. In addition, because it is difficult for XRD to detect crystals that are smaller than 50 nm, a particle would have to be at least half a micrometer to contain multiple detectable phases. Most welding fume particles are smaller than that. Transmission electron microscopy shows that fume can be heterogeneous with many phases contained individually in separate particles (Ref. 3). Therefore, if XRD reports that there are five phases, this means that there are at least five different types of particles, not that there is one particle that has each of the five phases contained in an extremely small volume. It is possible that large particles (greater than 100 nm) that have coalesced or sintered from smaller particles may contain multiple phases (Ref. 36). It is also possible that some large particles have a core-shell morphology with a metal oxide core and a slag exterior; this may be the case with the SMAW fume (Ref. 3). However, it is doubtful there are particles with more than two phases present.

The analyses presented herein did not take different particle sizes into account; therefore, the results represent the bulk only (with the exception of TEM-EDS, where the reported composition repre-

sents a very small sample of individual fume particles). There may be a change in chemistry with particle size that is not detected by an analysis that averages over many particles simultaneously. This is important for inhalation toxicology, because particles of different sizes are inhaled differently (Ref. 37). If the chemistry varies with particle size, the reported bulk chemistry may not be an accurate average of all particles, because some analytical techniques may sample data more strongly from certain particle size groups than others.

Because of the size-related complications of chemically analyzing welding fume particles that range from a few nanometers to a few micrometers (Ref. 38), a better procedure would be to separate welding fume into size groups and to analyze the size groups separately. This can be done with a cascade impactor (Ref. 39), with stages separating fume into groups of particles. A suggested grouping would separate particles smaller than 0.1 μm from those 0.1 to 0.5 μm, those 0.5 to 1 μm, and those larger than 1 μm.

There are five reasons for this suggested grouping. First, most impactors cannot distinguish between particles smaller than 0.1 μm (Table 1). Second, the particles that deposit in the alveolar passages of the lung when inhaled are predominantly smaller than 0.1 μm, while the inhaled particles that deposit in the nose and throat are generally larger than 1 micrometer (Ref. 40). Separating these size groups aids inhalation toxicology analysis. Third, analysis techniques tend to have an upper or lower boundary of 0.5 μm (Table 1). Fourth, separating it into four groups instead of two helps prevent overloading on the impactor stages. Fifth, airborne particles smaller than 0.1 μm are formed through vapor condensation, while airborne particles larger than 1 micrometer are created through liquid droplet ejection. Particles from 0.5 to 1 μm are a mixture of the two types of particles (Ref. 3). These formation paths strongly influence the particle chemistry, so it is important to separate them and to analyze both types of

particles with multiple techniques.

The smallest two groups can be analyzed with transmission electron microscopy, energy dispersive spectrometry, and electron diffraction. The largest two can be analyzed with scanning electron microscopy, energy dispersive spectrometry, and X-ray diffraction. Each size group should also be digested in acid and analyzed with emission spectroscopy. Care must be taken to dissolve all of the fume in the acid; otherwise, the results will not be valid.

Conclusions

Various chemical analysis techniques are presented in this paper and their applicability to welding fume particles is discussed. The range in their size complicates the analysis of airborne particles like those in welding fume. Particle size is the greatest factor when determining the utility of an analytical method. If this is not taken into account, erroneous conclusions can be easily made.

There are the two groups of elemental characterization techniques: those that report molar quantities and those that report atomic mass. The elemental data from various analytical techniques cannot be easily compared, except within these two groups. There seem to be fewer complications with the molar analytical techniques. Because of this and because oxygen analysis of small particles is difficult, the most convenient way to analyze the elemental composition of welding fume (which is comprised of oxides and halides) is by the molar fraction of the metal cations. This is most easily done with SEM-EDS and TEM-EDS.

Other techniques used on welding fume by previous researchers such as XPS, AES, XRF, and PIXE, yield data that are problematic to analyze. However, XRD is well suited for determining the phase composition of welding fume.

It was shown that mild steel GMAW fume is predominately magnetite in which the cations are approximately 10% Mn. Mild steel SMAW fume contains both a complex alkali-alkali earth fluoride phase and an oxide spinel phase. The metal cation fraction of that fume was approximately 27% Fe, 10% Mn, 10% S, 28% K, 25% Ca.

The analyses presented herein did not take different particle sizes into account; therefore, these results represent the bulk only. However, it has been shown that fume can be heterogeneous with many phases contained individually in separate particles. It would prove useful to separate welding fume into size groups and to analyze the size groups separately. This can be done with a cascade impactor, with stages

separating fume into groups of particles. The smallest groups can be analyzed with transmission electron microscopy, energy dispersive spectrometry, and electron diffraction. The largest groups can be analyzed with scanning electron microscopy, energy dispersive spectrometry, and X-ray diffraction. All groups can be analyzed by spectrometry of acid-digested fume. In this way, welding fume chemistry would best be characterized by size and by bulk (which would be a simple mass-weighted average of the chemistry of the size groups). This would aid in determining the toxicity of welding fume inhalation and shed light into how welding fume forms.

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