



A Spectrometer Discussion

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2017 SFSA T&O

Overview

A spectrometer is a chemical analytical tool that is used by most foundries, regardless of alloys poured, and those who do not have one typically still send out a final analysis to verify the chemistry of the metal that they are pouring. The purpose of this paper is to review the results of a survey that SFSA conducted and discuss various aspects of spectrometers and the analysis they provide.

Types of Spectrometers

From the survey there were three types of spectrometers mentioned: OES, XRF, Glow Discharge. These three will be the focus of this discussion.

For all of these spectrometers there are three things that are needed: a source to excite the atoms; a detection method; a software package to interpret what is being detected.

For the OES spectrometer, there is a high voltage source in the spectrometer that generates an arc between an electrode and the sample. A small part of the sample is heated to thousands of degrees Celsius which causes the surface to vaporize and excite the outer shells of the various atoms.

This excitation emits light which travels down a fiber optic path into the spectrometer. The light then passes through a diffraction grating where the element-specific wavelengths of light are separated. The light intensity of the element-specific wavelengths are then measured with a detector specific to that element. The detectors can be photomultiplier tubes or charge coupled devices (CCD's).

A computer software package then converts the measured intensities into weight percentages of the various elements.

A glow discharge spectrometer (GDS) is also an Optical Emission Spectrometer, that operates in a very similar manner as described above; however the source is different. Without delving too deeply, with glow discharge, an argon plasma sputters (or removes) material uniformly for the sample surface. Some of the sputtered material diffuses into the plasma where it becomes excited and emits light. Similar to above the

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light intensity is measured and then converted into weight percentage via a software package.

X-ray Fluorescence (XRF) uses an x-ray tube as the x-ray source that emits an x-ray beam into the sample. Different from above where the outer shell electrons are excited, with XRF the inner shell electrons are excited to an outer shell or removed completely. The outer shell electrons “fill” the inner shell. The excess energy is emitted as a fluorescent (or secondary) x-ray. For a given element the energy difference is specific to that element and always the same. XRF commonly uses a Silicon Drift Detector (SDD) to measure the energy and then a software package converts the values into weight percentages. Many of the handheld spectrometers utilize XRF methodology to provide an analysis.

From the survey and input from various suppliers, listed below are advantages and disadvantages. Some of these are not absolutes and are based on people’s perceptions and experiences.

Method	Advantages	Disadvantages
OES	Lower cost Speed Wider range of elements Dependability	Not as accurate as other methods
GDS	Accuracy of analysis Wide range of elements	Complicated instrument with a number of moving parts to maintain Must maintain a vacuum chamber Sample prep
XRF	Accuracy of analysis Speed of the analysis- fastest method	Higher cost Fewer elements

Survey Results

A survey was sent to SFSA member foundries. Listed below are the questions and a brief summary of the survey sent out by SFSA.

1. What type of spectrometer(s) do you use (OES, XRF, Atomic Absorption, etc.), why did you choose this type, and for what purposes do you use it for?
2. What is the make(s) and model(s)?
3. Are you happy with the performance of your instrument? If not, what are your issues and are you considering replacing it? If you bought a spectrometer in the last 7 years, please comment on why?
4. Do you use type standards to make adjustments for various alloys? If not, what do you do?



5. Are you able to find proper standards for the alloys you pour? If not, what is your standard practice?
6. How often do you standardize (or drift) the base element line?
7. How do you validate your spectrometer results are correct over the range of alloys you produce (separate from drift or control standards, do you perform Gage R&R, blind test samples, etc.)?

The most common type of spectrometer used is OES with most choosing it for cost, reliability, and range of analyzable elements. A number of foundries using XRF spectrometers are using hand held models to check incoming materials, conduct PMI, sort mixed metal.

Thermo Scientific is the most common manufacturer with Spectro being a close second. Overall the foundries in the survey are happy with their OES and XRF spectrometers and list the reason to replace was due to age of the instrument or difficulty in finding replacement parts.

In general most of the foundries use some method of type standardization or checking against known certified reference materials or standards. Many foundries check these standards daily and make small adjustments to the analysis; however a number also check at a longer interval and only make adjustments as needed. Most foundries did not have trouble finding certified reference materials. When standards did not exist most foundries would create their own and send to an outside lab. The level of outside analysis varied from sending them out to multiple labs or just one.

There was a lot of variation in the frequency of when the spectrometer is standardized or drifted for the base element line. The answers ranged from when needed based on a result to daily.

As far as validating our spectrometers, there are a few who participate in round robin analysis or complete a statistical analysis; however, most rely on PM's and checking against reference standards.

Conclusion

The 3 types of spectrometers focused on have 3 general things needed for the analysis with some similarity, yet some significant differences. The pros and cons and not absolute in all cases and are based on the experience, opinions and needs of the people using them. There is a fair amount of variation in how we standardize (or drift) our spectrometers and validation of results. These topics would be good topics for more in depth discussions at a later date.