Chemical Analysis at Materion's Spectrometry Laboratories

Materials for high reliability components require precise control of their chemical constituency for successful manufacture and service. The metals Materion Performance Alloys Group produces to meet these needs are typically copper based, and contain alloying ingredients such as beryllium, nickel, cobalt, tin, and lead in various combinations to impart the desired mechanical and physical benefits. In addition to these alloying ingredients, the content of a variety of impurities must be monitored and controlled at low limits. Nickel and aluminum alloys are also produced; alloying ingredients and impurities similarly must be monitored.

This paper describes the methods Materion uses in production of our high reliability alloys.

INTRODUCTION

Materion's copper beryllium alloys are melted and direct chill cast in high capacity induction furnaces. Pure copper, beryllium-containing master alloy, alloying ingredients, and recycled scrap are melted together to provide cast billets for further processing into strip or bulk products. Each "heat" must be carefully controlled to permit a certified composition.
Production to rigorous quality standards such as ASTM E255 requires chemical analysis at several stages. A "pot check" assures that the melt meets applicable specifications and allows billet casting to proceed. Each pot check sample requires a precise analysis within 15 minutes or less. A sample for certification is taken at the start of the pour, and a confirmation sample is taken at the end.
In addition to billets for strip and bulk products, Materion produces master alloys and casting ingot for worldwide markets, and purchases copper and alloying ingredients (typically nickel and cobalt) and scrap for recycling. These require chemical analysis, and although the need for rapid response is not as great, high precision is essential.

OPTICAL EMISSION SPECTROSCOPY

Most chemical analyses performed by Materion are done by Optical Emission Spectrometry (OES), also referred to as atomic emission spectroscopy. These analyses are primarily done by **Spark Spectrometry**, but **Direct Current Plasma** (DCP) and **Inductive Coupled Plasma** (ICP) are also used. The scientific principles of these are similar but sample preparation differs.
For both methods the metal is converted to individual atoms in the spark or plasma. On exposure to a temperature between 6,000 and 12,000 degrees Kelvin, electrons in the outer atomic orbits (optical electrons) transition to higher energy states as they absorb thermal energy (excitation). During random quantum fluctuations, these electrons fall to back to their ground state or other lower energy orbitals. In falling to a lower state the electrons emit photons of specific wavelengths characteristic of the energy differential and therefore of the specific element. The wavelength intensity compared with a standard of known concentration accurately determines the element's concentration.

For **spark spectrometry**, a representative sample is obtained by casting a small disc and machining one face. The disc is inserted into a chamber where the machined face is exposed to a carbon arc discharge.

For **plasma spectrometry** chips are produced by turning a rod or milling a plate. These chips are dissolved in acid for injection into the high temperature argon plasma of the ICP or DCP unit.

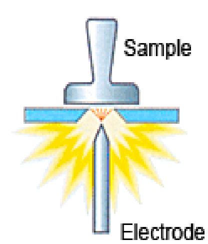

Figure 1 *Spark Excitation.*

SPARK OPTICAL EMISSION SPECTROMETRY

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This technique involves applying electrical energy in the form of a DC spark generated between a carbon electrode and a metal sample as shown in Figure 1.

The energy of the spark causes atoms to emit light at distinctive wavelengths. By measuring the intensity of the peaks in this spectrum, a direct reading analyzer quantifies the weight percentages of alloying ingredients and impurities with accuracy to parts per billion.

For illustration, Figure 2 shows sparked samples of Materion's copper beryllium alloys.

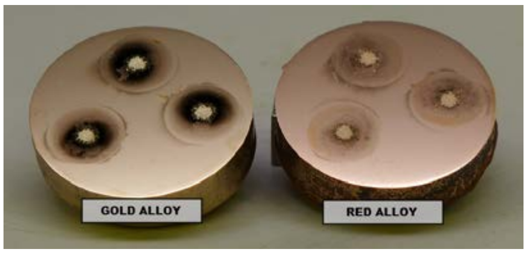

Figure 2 *Sparked samples. A high strength composition, Alloy 25, is on the left. A high conductivity version, Alloy 3, is on the right.*

DCP and ICP Optical Emission Spectrometry

Direct Current Plasma (DCP) and Inductively Coupled Plasma (ICP) rely on an argon gas plasma to stimulate emission of photons from excited atoms. DCP employs a direct current discharge among three electrodes. ICP uses a radio frequency (RF) discharge. Figures 3 and 4 illustrate these principles.

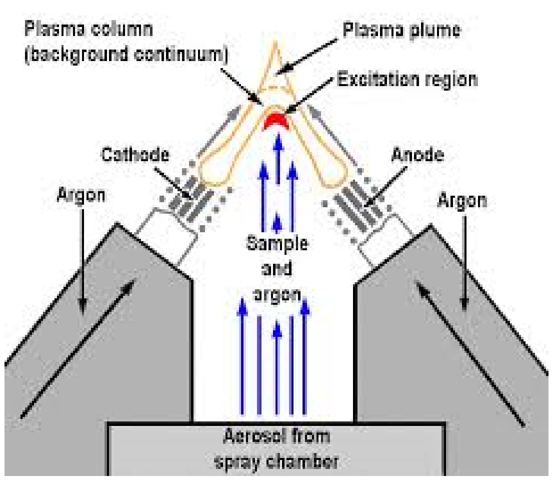

Figure 3 *Direct Current Plasma Excitation and Sample Injection*

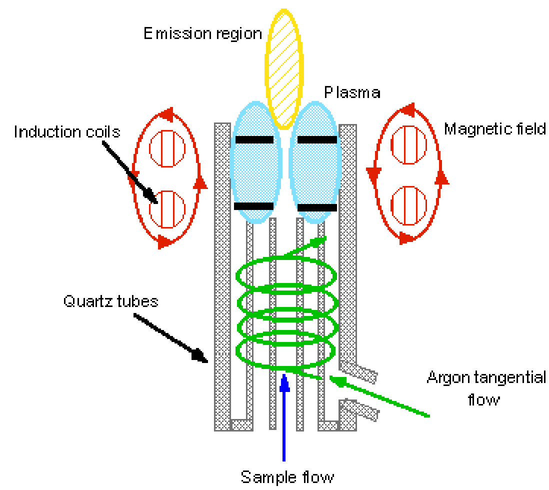

Figure 4 *Inductive Coupled Plasma Excitation and Sample Injection.*

Spectral Analysis

In each of these methods a monochromator separates the specific wavelengths and a sensitive detector measures the intensity of each. This intensity compared to a standard is used to calculate the concentration of each element. An example spectrum is shown in Figure 5.

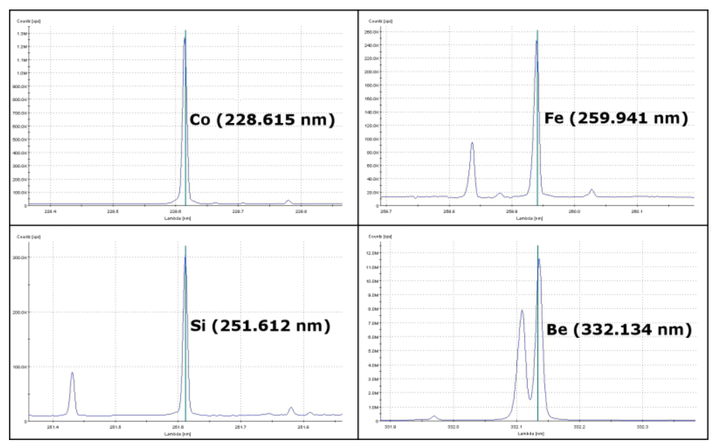

Figure 5 *Spectra from Copper Beryllium sample IARM 71B showing wavelengths of the primary ICP emission peaks. The secondary peaks in each graph are from the same element.*

For DCP and ICP OES, the constituents of the acid solvent, such as oxygen, nitrogen, chlorine and fluorine, will be present in spectrum and prevent analysis of those elements. The analyst must also be aware of interference effects from adjacent peaks that can bias results if not properly treated.

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Application

Spark spectrometry is used for nearly all analyses of conventional copper beryllium alloys, including wrought and cast compositions of both the high strength and high conductivity versions. These analyses are for pot checks to approve the composition of a heat prior to pouring, a certification sample at the start of the pour and a validation sample at the end.

DCP is used in Materion's lab for analysis of aluminum and nickel alloys, master alloys remelted scrap heats, and raw materials. It also serves to cross check the Spark Spectrograph, for analysis of non-standard alloys, and for R&D and product development work.

ICP is used for detailed analysis of impurities in all metals including high purity beryllium and copper as well as alloying constituents such as cobalt and nickel.

Alternate Techniques and Different Uses

Materion copper alloys produced in Elmore, Ohio, are classified by the Copper Development Association as High Copper Alloys. In other words, they are dilute in the sense that alloying additions typically totaling less than 2.5% are sufficient to impart the very high strength needed for their demanding applications. Other Materion alloys, notably our ToughMet, achieve comparable properties but have higher alloy content, often approaching 20% or more. These are best analyzed with techniques that include X-Ray Fluorescence for cost effective production analysis.

Portable spectrographs are available commercially for sorting alloys. These are highly useful for that intended purpose. They can accurately separate Materion's high strength alloys from the high conductivity versions and they can reliably sort various bronzes that are not visually distinguishable. But they do not have, nor are they intended to have, accuracy suitable for material certification.

Note that emission spectrometry is not the favored method for certain elements, especially nonmetallic and gases. For example, carbon, sulfur, oxygen, and others are best analyzed by combustion or other techniques. Materion performs these analyses on a sampling basis of production heats or as requested for specific applications.

Standards and Protocols

Certified reference materials traceable to the National Institute of Standards and Technology are readily available for a number of representative Materion alloys. Commercial sources for these reference materials include:

- Analytical Reference Materials International (ARMI), Manchester NH, USA
- Bureau of Analysed Samples Ltd, Middlesbrough, UK
- Brammar Standard Company, Houston TX, USA
- National Institute of Standards, Gaithersburg, MD, USA

Materion's labs participate in proficiency testing and interlaboratory measurement campaigns to be sure that the precision and bias of our techniques are in line with the most rigorous industry standards.

Materion's laboratory is an ISO/IEC A2LA 17025 accredited facility. It functions within a manufacturing environment under an AS9100 Quality Management System with an ISO14001-2004 certification.

Independent Laboratory Analysis

Commercial laboratories with no ties to Materion, in the United States and abroad, analyze copper beryllium alloys for producers and users worldwide.

Occasionally an independent laboratory performs an analysis that yields results that differ from values reported by Materion. In more than 50 years of experience, we know of no instance where this issue has not been resolved by using a proper reference standard obtained from one of the sources named above. Selection of appropriate wavelengths to avoid interferences, selection of appropriate reference standards, and matrix matching criteria are among the topics that should be examined in advance to avoid potential confusion.

Materion is pleased to work with independent laboratories to ensure the highest precision and lowest bias for alloy composition determination. We encourage our customers and independent laboratories to coordinate analytical procedures with us at any time.

Safe Handling of Copper Beryllium

Handling copper beryllium in solid form poses no special health risk. Like many industrial materials, beryllium-containing materials may pose a health risk if recommended safe handling practices are not followed. Inhalation of airborne beryllium may cause a serious lung disorder in susceptible individuals. The Occupational Safety and Health Administration (OSHA) has set mandatory limits on occupational respiratory exposures. Read and follow the guidance in the Safety Data Sheet (SDS) before working with this material. For additional information on safe handling practice, contact Materion Performance Alloy's Product Stewardship Group at 1-800-862-4118.

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