Immersion Sampling: In Search of a Truer Metal Analysis

As foundries increase their dependence on spectrometer analysis, this steel-borrowed technique provides an even more consistent sample for element breakdown.

George P. Frigm
Heraeus Electro-Nite Co.
Philadelphia

The chemical analysis of metal samples in the steel industry has undergone a renaissance over the past decade with improvements providing more accurate analysis, faster turnaround time, lower costs and more reliable processing equipment. Optical emission spectrometers (OES) have become more cost effective, user friendly and accurate, as even the most basic of instruments analyze 15-20 elements, including carbon (C), nitrogen (N) and sulfur (S).

For foundries, which have begun to benefit from the advancements in OES technology and cost effective equipment, the tightening of customer specifications for chemical analysis coupled with the continued drive for ISO9000 and QS9000 certification accurate metal analysis has brought OES instruments in-house. The result has been an increase in business and overall casting quality with a reduction in scrap.

Table 1 compares the C content of samples analyzed by OES and combustion, and the difference in the results. As illustrated, with the increased quality of spectrometers available, their ability to analyze C is to the same level as combustion analysis.

But, the advancements in spectrometer technology are just one part of the chemical analysis of a foundry's metal. The ability of the technology to accurately provide analyses is still only as good as the molten metal sample taken from the furnace for breakdown. Sample contamination, sample matrix and surface quality all play significant roles in the accuracy of the chemical analysis. And these characteristics, which are affected by the procedures used to take the sample, can alter the resultant spectrometer analysis.

Spoon Sampling

Currently, foundry metal sampling, for the most part, is performed with a metal or fiber spoon that taps and pours the molten metal into a mold (generally made of steel, copper or graphite) to form a disc or coupon for OES analysis. While this method has been acceptable for many years, the inconsistency of its samples clash with the current tightening of customer specifications and the requirement for improved analysis.

These spoon-poured samples can have several deficiencies:

- impurities can be added as the sample molten metal is pulled back through the surface slag;
- varying chill rates of the sample (due to the differing sizes and materials of sample molds) can affect the matrix of the metal;
- irregular surface area can cause entrapment of impurities in the sample;
- extensive polishing and grinding of the sample, due to mold irregularity, must be performed to prepare the surface for the OES;
- transporting an open spoon of molten metal to an open mold can be dangerous.

These deficiencies result in the quality inconsistencies that plague the samples taken by the spoon method. These inconsistencies led to the development of technology for immersion sampling.

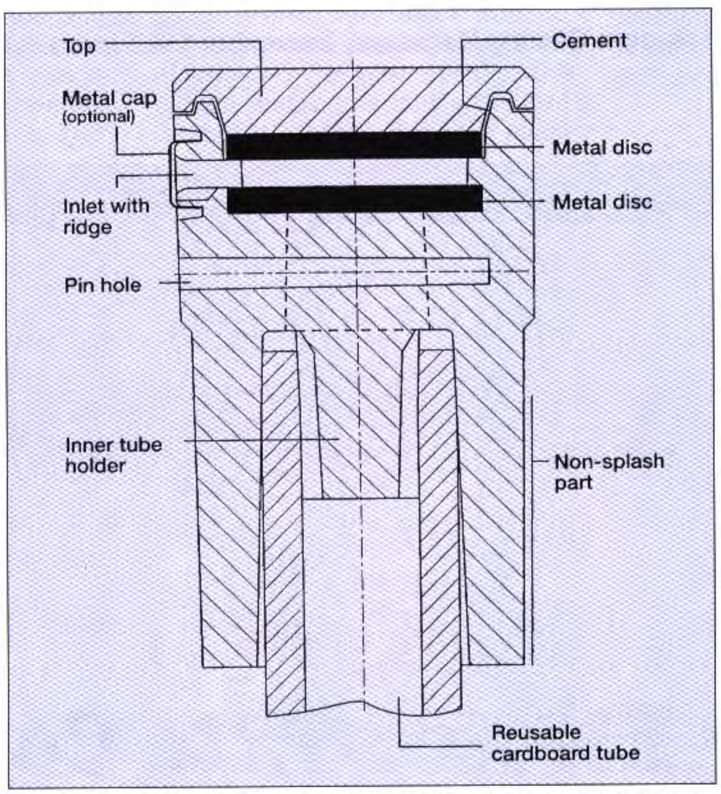


Fig. 1. The insert of the immersion sampler, as shown in the above cross section, is made of shell core sand. It has an inlet for the molten metal to flow and fill the cavity. Two steel plates surround the molten metal to provide consistent cooling for the sample disc. The insert also provides a pinhole for a second metal sample for combustion analysis. Just above the pinhole on the insert, the waste cardboard—thermocouple tube—is attached to allow the operator to submerge the immersion sampler into the molten metal.

Immersion Sampling

Within the steel industry, an almost universal switch has been made from spoon sampling to immersion sampling technology. Now, based on the success in the steel industry, immersion sampling is slowly gaining the attention of ferrous and nonferrous metalcasters. The technology can help to provide the consistency in

Table 1. Analysis of Carbon: Spectrometer vs. Combustion

Sample #	OES % C	Combustion % C	Difference
1	4.61	4.68	-0.07
2	4.44	4.63	-0.19
3	4.80	4.79	0.01
4	4.55	4.69	-0.14
5	4.61	4.59	0.02
6	4.63	4.50	0.13
7	4.63	4.58	0.15
8	4.59	4.76	-0.17
9	4.75	4.69	0.06
10	4.66	4.68	-0.02
11	4.60	4.61	-0.01
12	4.71	4.77	-0.06
13	4.66	4.63	0.03
14	4.49	4.60	-0.11
15	4.74	4.70	0.04

analysis that customers demand. The homogeneity, improved surface quality and dimensional stability of the immersion sample make it a fit for OES analysis and automated sample preparation.

The process of immersion sampling begins with the sampler insert—a specially designed sand core (Fig. 1) molded with chill plates. The insert is attached to a cardboard tube (often a used thermocouple tube) and submerged into a molten metal bath, where an inlet hole on the sand core takes in the molten metal to form a sample. After solidification (usually 4-5 sec), the sand core is struck against a hard surface to remove the disc. The disc is then prepared for OES analysis by polishing it on a belt or rotary sander (see Using an Immersion Sampler sidebar for further description of process).

The benefits of immersion sampling are:

- consistent sample surface, matrix and dimensional stability;
- the sample mold is contained within the immersion sampler, eliminating the dangers of spoon sampling.
- preparation time for analysis is reduced as the immersion sampler metal inlet discs provide a cleaner surface finish, without the excess grinding and polishing.
- the sampler only taps the molten metal for the sample—no waste.

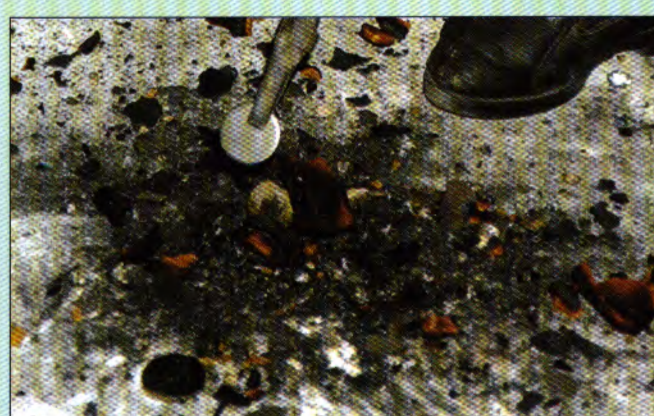
Sample preparation plays an important role in the quality of analysis, and a sample with a clean, pit-free surface will prepare quicker and more efficiently than a sample that has surface irregularities. This easier preparation saves time, and wear and tear on the polishing equipment,

Proper Use of an Immersion Sampler

Step 1—The cardboard thermocouple tube is firmly inserted into the sand insert. The tube is then given a quarter twist to lock in place.

Step 2—The sand insert is immersed into the molten metal. The immersion angle shouldn't be any more than 20° off of vertical to form an accurate sample.

Step 3—The immersion sampler is held in the metal for typically 5 sec. (Times vary according to the thickness of the sample required).



Step 4—The immersion sampler is withdrawn from the molten metal, and struck against a hard surface to retrieve the spectrometer sample disc. The sample is then prepared for OES analysis. ▼

Table 2. Silicon Analysis Comparison: Immersion Sample vs. Commercially Available Sample

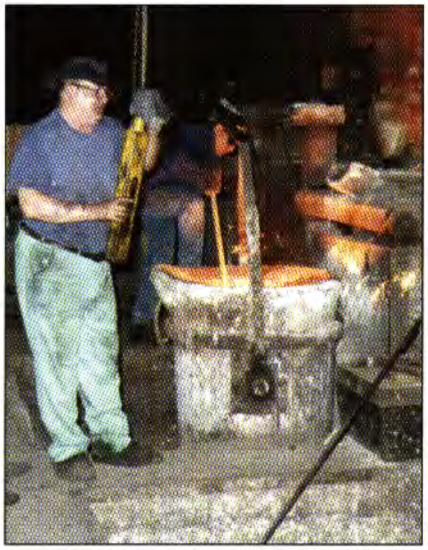
Immersion Sample			Commercially Available Standard		
1st Burn % Si	2nd Burn % Si	Difference	1st Burn % Si	2nd Burn % Si	Difference
0.333	0.334	-0.001	0.442	0.387	0.025
0.803	0.798	0.005	0.424	0.462	-0.038
0.823	0.810	0.013	0.399	0.397	0.002
0.497	0.510	-0.013	0.377	0.377	0.000
0.585	0.595	-0.010	0.356	0.336	0.020
0.728	0.718	0.010	0.981	0.986	0.000
0.435	0.429	0.006	0.740	0.730	0.010
0.573	0.583	-0.010	0.272	0.262	0.010
0.541	0.544	-0.003	0.477	0.493	-0.015
0.356	0.377	0.021	0.476	0.481	-0.005
0.599	0.601	-0.002	0.382	0.420	-0.038
0.571	0.552	0.019	0.460	0.467	-0.007
0.557	0.551	0.006	0.451	0.467	-0.016
0.718	0.703	0.015	0.415	0.335	0.080
0.451	0.458	-0.007	0.451	0.386	0.065

resulting in increased popularity for immersion sampling.

The importance of the cleanliness of the sample is best reflected in its element analysis. Table 2 shows data from burns done on an immersion sample of iron and a commercially available standard. The statistical difference and consistency between the first and second burn is comparable between the immersion sample and the commercially available standard.

With iron, the style of an immersion sampler also yields a sample with 100% iron carbide matrix. This matrix allows for complete element analysis including C, Si and S. In addition, immersion samplers for iron give a consistently white iron structure. This allows the complete analysis of the sample, including percent C. If the sample is homogenous, and has a completely white structure, then the OES instrument can measure percent C very accurately, even in very high C iron.

Following the lead of the steel industry over the past decade, the foundry industry is moving toward the better analysis of molten metal samples. The combination of the advanced and cost-effective OES technology and the introduction of immersion samplers has allowed overall improvement in the accuracy and speed of chemical analysis. ▼



Penn-Mar Castings, Inc., a gray iron foundry in Hanover, Pennsylvania, has been using an immersion sampler both in the furnace for base iron chemistry control and in the ladle to verify the final iron. "Using the immersion sampler, we are able to measure percent C consistently to +/-0.04, which is an accuracy we never received with the old spoon and mold method," said OES operator Craig Rang. ▼

Wagner Casting's Experience with Immersion Sampling

Wagner Casting Co., a large ductile iron casting producer based in Decatur, Illinois, has traditionally poured its chemistry lug samples from a hand held spoon into a water or air cooled copper (Cu) chill book mold—which were costly to buy ($700) and rebuild ($300). Deterioration of this mold over time caused poor surface quality and cooling problems with the sample, and left open the possibility for explosion if the lug pot was water-cooled.

In the past few years, Wagner has moved toward thinner lug samples for OES analysis. But this change caused problems with gating the sample for a proper fill, and lugs were more prone to stress cracking. These cracks made the analyses inconsistent or impossible. In addition, a hammer and a chisel were occasionally required to remove sticking samples. The result was a dented soft cooper mold, which gripped poured lugs even tighter, eventually leading to early retirement for a costly item.

At the present time, there is a movement toward immersion sampling. Currently, they are used with the Presspour furnaces at the Decatur and Havana plants, as well as the melt deck at Havana. The result since switching from book molds to the immersion samplers is a more consistent lug, both in quality and dimensions. The samples are less prone to stress cracks from cooling and water quenching, and the consistency and repeatability between shots on the spectrometer is much better with all elements. The technicians at both plants feel that the more consistent and accurate C analysis (as a result of good chill throughout the sample) is the most noticeable difference after switching from book molds. In addition, there are fewer spectrometer burns with the immersion samples that are obviously way out of the actual range. Cracking on the surface of the book mold samples sometimes would give grossly high final Magnesium (Mg), which isn't seen with immersion samples.

This is a cross-section of four fractured iron samples. At far left, the immersion sample shows a white iron matrix with a graphite free matrix. The other samples (from l to r) show varying degrees of white iron. This inconsistency greatly affects accuracy and precision of C analysis.

The elements of most concern to Wagner in final chemistry are Cu, Si and Mg. Since we began using the immersion sampling, the element analyses from the spectrometer are at the same percent the calculator says they should be. This saves the shop floor foundryman from making unnecessary changes to the chemistry that could be making molten iron worse, instead of better.

—Scott Davis, Wagner Casting Co.