C112-0510

Analysis of Cast Iron Using Shimadzu OES-5500

The analysis of low and high alloy cast iron by optical emission spectrometry is presented. Cast iron alloys are classified by their mechanical properties. The characteristics of cast iron, such as good ductility, hardness, wear resisting property, etc., are the reasons for the broad field of applications. In many cases specific mechanical properties cannot be reached without perfect control of the chemical composition of the cast iron. The addition of one or more alloying elements modifies and optimizes the properties of cast iron to a particular application.

Cast iron alloys contain more than 2% carbon and are named depending upon their metallurgical structure as follows:

- **White cast iron** for the Cast iron with cementite structure.
- **Gray cast iron** for the Cast iron with lammelar structure.
- **Nodular cast iron** for the Cast iron with nodular structure.
- **High alloy cast iron** for the Cast iron with austenitic structure.

Two main factors have a direct influence on the metallurgical structure:

1. The cooling speed of the melted metal A fast cooling speed contributes to a White structure, in contrary a slow cooling speed contributes to the precipitation of the graphite.
2. The chemical composition For example carbon, silicon, manganese, phosphorus and sulfur are the most important alloying elements.
   - Silicon promotes the precipitation of graphite
   - Manganese impedes the precipitation of graphite
   - Sulfur decreases the mechanical properties and increases the tendency of the formation of tension cracks
   - Phosphorus increases the fluidity of the melt. Phosphorus is an undesirable element for the production of cast iron with nodular graphite. The content of phosphorus higher than 0.15% reduces the tensile strength and creep characteristics, therefore the concentration of phosphorus is kept below 0.08% and respectively for extra impact strength material below 0.05%.

Cast irons are classified as unalloyed, when elements such as Ni, Mo, Si, Cr, Mn and V do not exceed the concentration indicated in the following table.

Nickel 0.80%	Molybdenum 0.12%	Silicon 2.0%
Chromium 0.60%	Manganese 1.50%	Vanadium 0.2%

Other alloying elements as magnesium, copper, titanium, aluminum, tin, niobium, boron, etc. in the addition of accompanying elements, enhances the corrosion resistance or the mechanical properties.

Nodular Cast Iron

The addition of magnesium at 0.1 % to 0.15 % contributes to the production of Cast iron with nodular structure. About 50% of this amount burn out during the inoculation process. Meanwhile, during casting, the magnesium concentration in the melt continues to decrease. It is highly recommended anytime in the process, the critical concentration of 0.03 % of magnesium should be avoided. The content of 0.03% or less of Magnesium in the liquid metal prohibits the formation of graphite with the nodular structure. Therefore, to produce nodular cast iron, the continuous control of the magnesium concentration is necessary.

High Alloyed Cast Iron

Massive additions of chromium or nickel provides a stable austenitic structure. The structure of the carbon graphite in high alloyed cast iron can be lammelar or nodular, depending on the chemical composition. One typical application of chromium alloyed white cast iron is the production of balls used in mining for

crushing ores. "Nickel-resistant alloys" can contain up to 36% nickel. This type of cast iron is temperature and corrosion resistant, and used for example as a non magnetic material in shipbuilding.
Even if low and high alloyed cast irons are designated by their physical properties, it is also necessary to evaluate their chemical composition. In addition to the testing methods such as tensile strength and hardness, the chemical composition is often an important factor to determine the suitability of the material for a particular application. Spark emission spectrometry is the most popular method to determine chemical composition of a metal, a technique that is a very fast while providing a very reliable analysis.

Instrumentation

The metal analyzer, OES-5500 is a high performance optical emission spectrometer with the distinct advantage of simultaneous determination of many elements. The OES-5500 with incorporated argon flushed spark stand and high performance spark source, has been designed for accurate and quantitative analysis of low and high alloyed cast iron. The simultaneous analysis of up to 32 elements including trace and gaseous elements is performed in less then one minute. Equipped with a 600 mm spectrometer, the OES-5500 analyzes up to 64 elements. Pulse height distribution analysis method (PDA) and the time resolved integration method (TRS) are sequenced in the measuring cycle. The combination of these two methods improves the analytical precision of trace elements, particularly for those elements that tend to form precipitations in cast iron (for example: Al, S, Pb, B, Ca.) The improvement of accuracy, detectability, reproducibility and higher sample throughput results in higher productivity.

Samples And Sample Preparation

Ladle analysis requires that a small amount of the melt is taken out of the molten metal bath using a test spoon and poured into a copper mold. The copper mold insures high cooling speed necessary for the production of homogeneous samples. Homogenous samples are basic requirements to avoid lack of precision and accuracy during the analysis by optical emission instruments.. A rapid cooling in a special mold prevents the formation of larger precipitations and phase separations. A sample with white solidification microstructure and good homogeneity (fine-grained and homogeneous crystalline structure) provides good reproducibility and accuracy of the analyses. To carry-out an analysis by spark emission spectrometry the surface should be prepared accordingly. The unnecessary parts of a casted sample are cut using a grinder and/or cutter. The sample is ground with a pendulum grinding machine, abrasive belt or disk grinder (with grid is #40 or #60)

Argon

Argon purity: 99.999% (Highly recommended)
Oxygen: < 2 ppm
H_2O: < 3 ppm (< -70°C dew point)
Nitrogen: < 10 ppm
For nitrogen analysis, argon should contain less than 3 ppm of Nitrogen.
Upon request, Shimadzu can provide argon purifiers. A pressure of four bars at the entrance is necessary. During pre-spark and analysis time, the argon flow rate is approximately 7 l/min..

Operating Parameter

Counter electrode: Tungsten
Argon flush time: 3 seconds
Pre spark time: 15 seconds (Sulfur < 0.2%)
Integration time: 4 seconds

Analytical Data / Precision

The following table reports typical precision data for low and high alloyed cast iron. The precision data were evaluated using suitable test samples. These samples covered a large variety of grades of the different cast iron alloy types.

Element	Detection Limit ppm 3 sigma	Quantitative Working Range %	Content %	Precision ±% 1 sigma
Al	3	0.001 -- 0.5	0.005	0.0005
			0.01	0.001
			0.1	0.002
As	3	0.0015 -- 0.1	0.01	0.0007
			0.05	0.0015
B	<1	0.0002 -- 0.1	0.001	0.00005
			0.01	0.001
			0.05	0.0015
C		1.7 -- 4.5	2.0	0.015
			3.5	0.03
			4.0	0.04
Ca	<1	0.0003 -- 0.01	0.001	0.0001
			0.01	0.0005
Ce	10	0.003 -- 0.1	0.01	0.0015
			0.05	0.005
			0.1	0.008
Co	2	0.001 -- 0.3	0.01	0.001
			0.05	0.005
			0.1	0.002
Cr	5	0.0015 -- 36.0	0.01	0.0004
			0.05	0.0007
			0.5	0.005
			1.0	0.006
			5.0	0.03
			10.0	0.03
			20.0	0.06
			30.0	0.09
Cu	1	0.0005 -- 10.0	0.01	0.0003
			0.05	0.0005
			0.5	0.004
			1.0	0.008
			3.0	0.03
			5.0	0.05
Mg	3	0.001 -- 0.3	0.005	0.0004
			0.01	0.0006
			0.1	0.004
Mn	4	0.001 -- 6.0	0.01	0.0004
			0.05	0.0006
			0.5	0.005
			1.0	0.01
			2.0	0.015
			5.0	

Element	Detection Limit ppm 3 sigma	Quantitative Working Range %	Content %	Precision ±% 1 sigma
Mo	4	0.001 -- 5.0	0.01	0.0003
			0.05	0.005
			0.5	0.004
			1.0	0.009
			3.0	0.03
N	15	0.003 -- 0.01	0.003	0.0003
			0.005	0.0004
			0.01	0.0005
Nb	5	0.0015 -- 0.3	0.01	0.001
			0.05	0.003
			0.1	0.005
Ni	3	0.001 -- 40.0	0.01	0.0004
			0.05	0.0007
			0.5	0.005
			1.0	0.006
			5.0	0.02
			10.0	0.04
			20.0	0.08
			30.0	0.12
P	3	0.001 -- 1.5	0.005	0.0002
			0.01	0.001
			0.05	0.002
			0.1	0.005
Pb	5	0.002 -- 0.1	0.01	0.0006
			0.05	0.001
			0.1	0.002
S	2	0.0006 -- 0.2	0.005	0.0005
			0.01	0.001
			0.05	0002
			0.15	0.02
Sb	8	0.0025 -- 0.2	0.01	0.0015
			0.05	0.004
			0.1	0.008
Si		0.1 -- 5.0	0.01	0.0003
			0.05	0.001
			0.5	0.007
			1.0	0.01
			2.0	0.015
			3.0	0.02
Sn	5	0.001 -- 0.5	0.01	0.0003
			0.05	0.0009
			0.1	0.002
Te	10	0.003 -- 0.1	0.005	
			0.01	
Ti	1	0.0002 -- 0.35	0.01	0.0003
			0.05	0.001
			0.1	0.002

Element	Detection Limit ppm 3 sigma	Quantitative Working Range %	Content %	Precision ±% 1 sigma
V	3	0.001 -- 0.5	0.01 0.05 0.1 0.5	0.0002 0.0007 0.0015 0.005
W	15	0.003 -- 0.2	0.01 0.05 0.1	0.001 0.002 0.003
Zn	5	0.0015 -- 0.1	0.005 0.01	0.0005 0.0007
Zr	4	0.001 -- 0.1	0.01 0.05 0.15	0.001 0.004 0.006

Detection Limit

The detection limit is defined as three times the standard deviation (σ) calculated on the background. The standard unit is in ppm (parts per million). These values are valid only for low and medium alloyed cast iron. Values at the detection limit level can only be considered semi quantitative. (Relative standard deviation 33%.)

Precision

Precision is defined as the Standard deviation on 10 successive measurements. The precision is related to the distribution of the elements in the samples.

Calibration

Factory calibration for low and high alloyed cast iron is available on request. For the specific alloy types, please see the factory calibration summaries. The factory calibration is based on certified reference material (ISO 9000), and provides high accuracy. The factory calibration set up, provides for a rapid installation time with the spectrometer immediately available for production control. For more detailed information please contact your local Shimadzu office or dealer.