Steel analysis

ARL X900 XRF Spectrometer with universal goniometer

Keywords

ARL X900, X-ray fluorescence, XRF, WDXRF, steel, ferrous base, goniometer

Goal

Describe the analytical performance of Thermo Scientific™ ARL™ X900 Series for steel analysis using the Moiré fringe goniometer.

Introduction

Ferrous base materials are very important products in this world because they are the foundation in many applications such as building, automotive and many manufacturing processes. It is important to accurately analyze these materials to confirm compliance with their chemical specifications and allow for high quality and efficient production.

Irons

There are several kinds of irons which are distinguished by their composition and use. They belong to two main categories:

- Pig irons, also called hot metal, form the basic production for the manufacture of steel
- Cast irons, are used to produce semi-manufactured products

From a metallographic point of view, a distinction can be made between white cast iron with a cementite structure and grey cast iron which contains free graphite either in the form of laminae or nodules. These make grey cast iron inhomogeneous and therefore difficult to analyze. Alloy cast irons also exist where alloying elements such as nickel, chromium, manganese, copper, etc. are added to improve hardness, corrosion resistance, or engineering properties.

ARL X900 Simultaneous/Sequential X-Ray Fluorescence Spectrometer.

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Low alloy steels

This category covers steels which are destined for a wide variety of uses, such as the production of:

- Steel castings, rails, axels, boiler and ship plates, automobile bodies
- Girders, all kinds of bridge and structural sections
- Wires, nuts, bolts, and forgings of any description
- Springs, cutting steels

From a compositional point of view, these steels can be distinguished by the fact that the alloying elements generally total less than 5 to 7 %. Typically, the main alloying elements are present at less than the following concentrations:

Mn 2 % ; Cr 3 % ; Ni 5 % ; Cu 1.5 % ; Mo 1.5 % ; V 1 %.

High alloy steels

High alloy steels contain, in addition to iron and carbon, notable quantities of one or more of the following elements: nickel, chromium, manganese, silicon, cobalt, tungsten, molybdenum and vanadium. Included under this heading are: Stainless steel types such as 18/8, austenitic, maraging, martensitic and all types of special stainless steels, tool steels, high speed steels and high manganese steels.

Instrument parameters and conditions

The ARL X900 XRF Spectrometer is equipped with the unique proprietary **Moiré fringe goniometer**. Speed, flexibility, and reliability of analysis are guaranteed thanks to the ingenious friction-free positioning system. Up to nine crystals and four collimators can be fitted. With the two detectors (flow proportional and scintillation counters) precise elemental analysis from boron to californium is possible. Additionally, the spectrometer can accommodate up to 24 fixed monochromator channels alongside the goniometer or up to 32 fixed monochromator channels when no goniometer is fitted.

The ARL X900 XRF Spectrometer can be calibrated using commercially available certified reference material (CRM) standards or well analyzed samples from the user.

It should be stressed that an XRF spectrometer is a very accurate comparator, but the accuracy of the final analysis is entirely dependent on the quality of the standards used for calibration and on the care and reproducibility of sample preparation which must be identical for CRMs and for routine samples as well.

Typical performance in low alloy steel samples using the goniometer

Table 1 is a summary of typical limits of detection. A set of international steel standards were used for calibration with recommended goniometer parameters regarding crystal, detector, collimator, and the highest power of 4200W. Phosphorus was measured with two different crystals to compare the performance.

All elements from B to Cf can be analyzed if needed, but Table 1 includes only a small selection of usual elements measured in steels. Limits of detection (LoD) are calculated from the calibration curve for 10 seconds and 100 seconds counting time per element for comparison. As the goniometer measures sequentially one element after the other, it is often interesting to use shorter counting times in order to produce a final result within a few minutes.

	Crystal	Detector	Collimator	4200W LoDs 10s	4200W LoDs 100s
Si	PET	FPC	0.6°	17.2	5.9
P	PET	FPC	0.6°	10.1	3.5
P	Ge111	FPC	0.6°	5.6	1.9
V	LiF200	FPC	0.15°	4.8	1.7
Cr	LiF200	FPC	0.15°	8.1	2.8
Mn	LiF200	FPC	0.15°	10.9	3.8
Cu	LiF200	Scint	0.15°	9.7	3.4
Ni	LiF200	Scint	0.15°	9.2	3.2
Mo	LiF200	Scint	0.15°	4.7	1.6

Table 1: Typical limits of detection in ferrous matrix for the goniometer at 10s and 100s counting time.

Typical precision tests

The stability of an instrument reflects the precision that can be obtained. A short-term, repeatability test consisting of 11 runs was performed on 2 low alloy steel samples over 1 hour (table 2a and 2b). Counting time of 10 seconds was used for all elements at each run. A power level of 2500W was used for this test.

A long-term, repeatability test consisting of 18 measurements of a carbon steel sample over 60 hours was performed (table 3).

Counting time of 10 seconds was used for all elements. The power level was set at 2500W for this test.

We can see that the standard deviation over 60 hours is less than twice the standard deviation obtained over one hour. This is due to the excellent stability of the ARL X900 WDXRF Spectrometer.

Counting time	10s	10s	10s	10s	10s	10s	10s	10s
Run #	Cr Kα	Cu Kα	Mn Kα	Mo Kα	Ni Kα	P Kα	Si Kα	V Kα
Crystal	LiF200	LiF200	LiF200	LiF200	LiF200	Ge111	PET	LiF200
Detector	FPC	Scint	FPC	Scint	Scint	FPC	FPC	FPC
1	1.902	0.259	0.577	0.871	0.907	1.402	0.0181	0.0209
2	1.902	0.256	0.573	0.874	0.902	1.401	0.0183	0.0209
3	1.907	0.258	0.574	0.873	0.903	1.413	0.0186	0.0222
4	1.909	0.258	0.575	0.873	0.904	1.412	0.0185	0.0232
5	1.905	0.258	0.575	0.874	0.902	1.406	0.0189	0.0222
6	1.901	0.256	0.576	0.873	0.904	1.398	0.0180	0.0226
7	1.903	0.256	0.576	0.877	0.900	1.406	0.0184	0.0228
8	1.909	0.257	0.580	0.870	0.904	1.407	0.0192	0.0224
9	1.909	0.259	0.580	0.873	0.903	1.406	0.0180	0.0223
10	1.907	0.256	0.572	0.875	0.902	1.408	0.0190	0.0233
11	1.901	0.256	0.575	0.870	0.901	1.400	0.0186	0.0222
Average %	1.905	0.257	0.576	0.873	0.903	1.405	0.0185	0.0223
% std deviation	0.0034	0.0014	0.0024	0.0021	0.0018	0.0048	0.0004	0.0008

Table 2a: Sample 1—short term precision test over one hour using the goniometer at 2500W—low alloy steel.

Counting time	10s	10s	10s	10s	10s	10s	10s	10s
Run #	Cr Kα	Cu Kα	Mn Kα	Mo Kα	Ni Kα	P Kα	Si Kα	V Kα
Crystal	LiF200	LiF200	LiF200	LiF200	LiF200	Ge111	PET	LiF200
Detector	FPC	Scint	FPC	Scint	Scint	FPC	FPC	FPC
1	2.695	0.365	0.1885	0.754	0.496	0.0413	0.667	0.1562
2	2.696	0.365	0.1875	0.753	0.498	0.0408	0.668	0.1564
3	2.699	0.361	0.1854	0.752	0.496	0.0401	0.666	0.1562
4	2.698	0.371	0.1874	0.752	0.500	0.0406	0.666	0.1552
5	2.697	0.368	0.1867	0.753	0.500	0.0412	0.669	0.1568
6	2.694	0.369	0.1873	0.750	0.493	0.0407	0.671	0.1576
7	2.700	0.371	0.1863	0.752	0.497	0.0406	0.670	0.1572
8	2.708	0.364	0.1865	0.752	0.493	0.0407	0.660	0.1566
9	2.699	0.370	0.1848	0.753	0.497	0.0409	0.665	0.1570
10	2.699	0.365	0.1861	0.750	0.497	0.0416	0.667	0.1564
11	2.706	0.363	0.1877	0.754	0.499	0.0414	0.671	0.1556
Average %	2.699	0.366	0.187	0.752	0.497	0.041	0.667	0.156
% std deviation	0.0044	0.0033	0.0011	0.0014	0.0024	0.0004	0.0030	0.0007

Table 2b: Sample 2—short term precision test over one hour using the goniometer at 2500W—low alloy steel.

Counting time	10s	10s	10s	10s	10s	10s	10s	10s
Element—line	Cr Kα	Cu Kα	Mn Kα	Mo Kα	Ni Kα	P Kα	Si Kα	V Kα
Crystal	LiF200	LiF200	LiF200	LiF200	LiF200	Ge111	PET	LiF200
Detector	FPC	Scint	FPC	Scint	Scint	FPC	FPC	FPC
Run #1—July 19	2.706	0.363	0.1877	0.754	0.499	0.0414	0.671	0.1556
2—July 19	2.695	0.365	0.1885	0.754	0.496	0.0413	0.667	0.1562
3—July 20	2.705	0.364	0.1878	0.752	0.503	0.041	0.667	0.1582
4—July 20	2.695	0.368	0.1871	0.751	0.498	0.0407	0.667	0.1566
5—July 20	2.704	0.368	0.1879	0.752	0.499	0.0405	0.669	0.1555
6—July 20	2.701	0.365	0.1888	0.754	0.496	0.0407	0.669	0.1582
7—July 20	2.700	0.364	0.1871	0.749	0.498	0.0417	0.672	0.1571
8—July 20	2.695	0.369	0.1872	0.750	0.496	0.0412	0.677	0.1576
9—July 21	2.703	0.368	0.1882	0.751	0.499	0.0416	0.674	0.1575
10—July 21	2.701	0.366	0.1889	0.749	0.499	0.0411	0.673	0.155
11—July 21	2.695	0.364	0.1877	0.747	0.499	0.0412	0.680	0.1566
12—July 21	2.695	0.364	0.1884	0.749	0.502	0.0407	0.682	0.1556
13—July 21	2.694	0.364	0.1878	0.752	0.496	0.0415	0.673	0.1561
14—July 21	2.697	0.367	0.1864	0.747	0.497	0.0414	0.678	0.1569
15—July 21	2.702	0.368	0.1878	0.748	0.498	0.0413	0.681	0.1551
16—July 22	2.700	0.367	0.1864	0.752	0.500	0.0415	0.675	0.1564
17—July 22	2.689	0.370	0.1871	0.752	0.495	0.0416	0.680	0.1561
18—July 22	2.693	0.367	0.1872	0.753	0.499	0.0410	0.677	0.1549
Average %	2.698	0.366	0.18767	0.751	0.498	0.0412	0.674	0.1564
% std deviation	0.0047	0.0021	0.0007	0.0022	0.0021	0.0004	0.0050	0.0010

Table 3: Long term repeatability over 60 hours using the goniometer at 2500W—carbon steel.

Conclusion

Analysis of steels can be performed with ease using the ARL X900 Simultaneous-Sequential XRF Spectrometer. The performance of the Moiré fringe goniometer is such that it can be used for analysis of any elements that are not fitted as fixed channels. The analysis on the goniometer is done while the fixed channels are also measuring. In addition, it can be used as a backup in case of failure of any of the fixed channels.

Appropriate calibrations for steel alloys can be delivered turnkey from Thermo Fisher Scientific. In this case, the commissioning time of the spectrometer is reduced to a minimum.

The precision is excellent in these matrix types for routine or R&D analysis, especially when an innovative, high counting fixed channel monochromator is used for elements like Ni, Co or Mo.

Furthermore, operation is made easy through the advanced Thermo Scientific™ OXSAS™ Software that operates with the latest Microsoft Windows® package.