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Energy Dispersive X-Ray Fluorescence Spectroscopy and Applications in Material Science

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Abstract

Energy dispersive X-ray fluorescence spectroscopy (EDXRF) is a powerful non-destructive technique used to determine the elemental composition of materials. The current work focuses on the installation and optimization of the operation of the EDXRF system developed by Amptek Inc. Furthermore, XRF elemental analysis results of alloys candidate for fusion reactor applications are presented

Keywords: X-ray fluorescence, fundamental parameters method

1. Introduction

Energy dispersive X-ray fluorescence spectroscopy is a powerful, non-destructive technique used for qualitative and quantitative elemental analysis of materials. Typically, EDXRF covers all the elements of the periodic table, from $_{11}\text{Na}$ to $_{92}\text{U}$. As for the concentrations, the detection range varies from 100% down to ppm and even sub-ppm levels under certain conditions.

In this paper, the installation and optimization of operation of EDXRF system developed by Amptek Inc. is presented. The effect of the different parameters, that refer to signal and spectrum analysis, on the accuracy of the quantification of the results is discussed. The quantitative analysis of the spectra is based on fundamental parameters (FP) method. Standards were used for the calibration of the parameters used in the FP analysis method. Moreover, experimental results of samples candidate for fusion reactor applications are presented showing the applicability of the XRF technique.



Figure 1: EDXRF system located at NCSR “Demokritos”

X-ray tube
Voltage/Current: 10-50 kV / 5-200 A
Collimators diameter: 1 and 2 mm
Various filters: Available
Detector
Type: Silicon drift detector
Resolution: 125-140 eV FWHM at 5.9 keV
Maximum count rate $> 5.6 \cdot 10^5$ cps

Table 1: Characteristics of the EDXRF system at NCSR “Demokritos”

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2. Energy dispersive X-ray fluorescence technique

The EDXRF system (Figure 1) consists of the X-ray tube with $_{47}\text{Ag}$ anode and the detection system which includes a silicon drift detector, a digital pulse processor and a multi-channel analyser. The sample mount was designed to provide vertical motion so as samples of different sizes to be positioned with high accuracy. The system was placed inside a metal shield and a safety system of two micro switches and a light indicator was installed in order to prevent accidental exposure to radiation during systems operation.

Irradiating a sample with high energy photons, such as X-rays, results in the emission of characteristic fluorescent X-rays from the elements present in the sample. The EDXRF spectrometer directly measures these energies. Each event that occurs is sorted into the corresponding energy channel of the multi-channel analyser and the result is an XRF energy spectrum. The peak positions of the spectrum define which elements are present (qualitative analysis) while the peak intensities designate their concentrations (quantitative analysis).

Various parameters that refer to signal and spectrum processing need to be assigned to obtain the best performance of the system for a specific application. The most important parameters are the gain that gives the channel number of the multi-channel analyser in which a specific energy that occurs will be sorted and thus it determines the energy range of the spectrum and the peaking time which is defined as the time required for the pulse to go from the baseline to the peak. Artefacts present in the XRF spectrum due to X-ray scattering and detector effects are removed at the processing stage. The processed spectrum consists only of the characteristic peaks of the elements present in the sample. Fundamental Parameters (FP) method is used for quantitative analysis, where the concentration calculations are based on the Sherman equation [1]:

$$C_i = R_i \left(\frac{1 + \sum_j a_{ij} C_j}{1 + \sum_j \epsilon_{ij} C_j} \right)$$

where C_i is the concentration of the analyte i , R_i is the relative peak intensity of the analyte i , C_j is the concentration of the matrix element j and a_{ij} , ϵ_{ij} are the influence coefficients correcting the secondary absorption effect and the enhancement effect of the matrix element j on the analyte i , respectively.

3. Results

To determine the performance of the EDXRF system, the energy resolution and throughput were measured for a variety of counting rates and peaking times. By increasing peaking time the electronic noise decreases and the resolution increases. In addition, for each peaking time the resolution is constant within error bars (Figure 2). Throughput is the ratio of the valid events to the total events recorded. Increasing peaking time leads to the reduction of throughput (Figure 3).

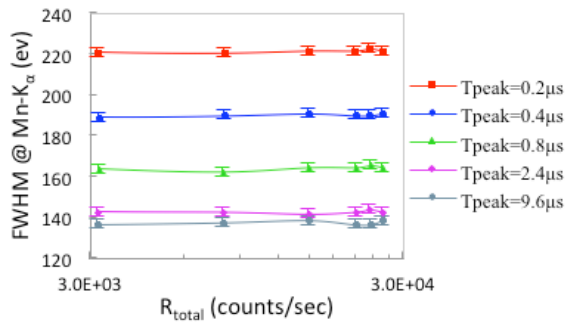


Figure 2: Energy resolution of the EDXRF system

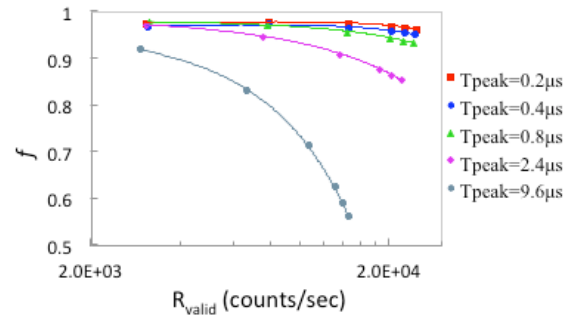


Figure 3: Throughput of the EDXRF system

For the calibration of the instrument, a stainless steel 316 (NIST) sample was measured. To calibrate the parameters used by FP analysis, standard samples of pure iron, molybdenum and nickel were used while standardless approach was applied to the rest elements present in the sample. The results are in good agreement with NIST values (Figure 4). Only in the case of Cu there is a discrepancy. The overestimation in the Cu concentration is attributed to additional signal coming from the brass collimator of the X-ray tube.

Furthermore, elemental compositions of alloys candidate for fusion reactor applications were measured with XRF technique. In the Tables 2 and 2 the results are presented.

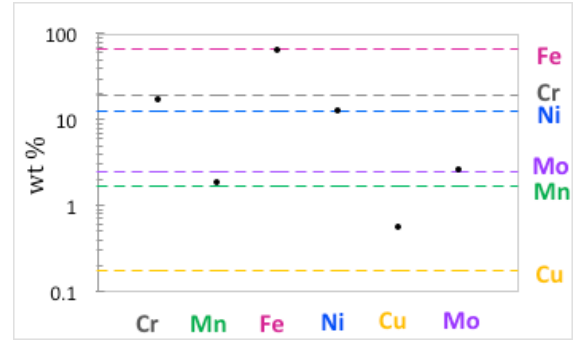


Figure 4: Elemental composition of SS316 standard sample measured with XRF technique. The dashed lines represent the elements nominal compositions given by NIST

Code	Element	Concentration (wt%)	Nominal Concentration (wt%)
Fe- 14.25%Cr	Cr	14.29±0.13	14.25
	Fe	85.71±0.26	85.75
Fe- 10.1%Cr	Cr	10.25±0.12	10.1
	Fe	89.75±0.26	89.9
Fe- 5.4%Cr	Cr	5.32±0.05	5.4
	Fe	94.68±0.25	94.6

Table 2: FeCr alloys elemental composition as determined by XRF

Element	Concentration (wt%)	Nominal Concentration (wt%)
²⁴ Cr	13.08±0.10	13.5
²⁶ Fe	85.11±0.28	84.5
⁷⁴ W	1.81±0.06	2

Table 3: FeCrW alloy elemental composition as determined by XRF

4. Conclusions

The installation and optimization of operation of EDXRF system was accomplished successfully. Measurements of stainless steel 316 (NIST) were in good agreement with NIST values. A number of alloys candidate for fusion reactor application were measured with EDXRF technique.

References

- [1] The Quest for a Fundamental Algorithm in X-Ray Fluorescence Analysis and Calibration. **Rousseau R.M.** s.l. : The Open Spectroscopy Journal, 2009, volume