ENERGY DISPERSIVE X-RAY FLUORESCENCE (EDXRF) ANALYSIS OF STEELS

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***Abstract.** A powerful, simple and fast technique for non-destructive multi-element analysis of materials is energy dispersive X-ray fluorescence (EDXRF) technique. This method is based on radioisotope excited energy dispersive X-ray fluorescence (EDXRF) spectrometry and does not require any type-standards. For these reasons, the EDXRF method was chosen in this paper to analyse the different types of stainless steel (SS) samples. After non-destructive multi-element analysis of samples revealed that Si (0.1–0.52), S (0.01-0.12), K (0.25-0.86), Cu (0.19-0.24), Ca (0.01-0.03) etc are present as minor components while Cr (15.1-17.5), Mn (0.56-1.4), Fe (69.8-83.7), Ni (0.03-10.4) are present as major components.*

***Keywords:** EDXRF technique, non-destructive and standardless analysis of stainless steel samples.*

1. INTRODUCTION

X-ray fluorescence spectrometry (XRF) is a non-destructive analytical method; it is fast and can simultaneously measure many elements, both solid and liquid samples. There is a wide application area and XRF spectra cover most items on a field $100\% \div \mu g/g$ [1].

XRF is used in quality control of many products used daily, such as Fe determination in milk powder or determination of vitamins and essential minerals, as new examples of XRF applications in human health.

The application of energy-dispersive X-ray fluorescence (EDXRF) spectrometry in the quantitative analysis of samples is important, this method being able to evaluate the possibility of detecting different elements from the materials.

EDXRF is a relatively simple and inexpensive alternative compared to other techniques. The apparatus involves X-ray source represented by an X-ray tube (about 30-60 kV) and a liquid nitrogen cooled detector, Si (Li) or Si (PIN) or NAI detectors, CdTe or CdZnTe.

The fundamental parameter method (FP) using iterative method can work even with non-available type-standards. The analysis of multi-component alloys like stainless steel is somewhat complex and requires the matrix absorption-enhancement effects have to be corrected, but accurate peak intensities have to be determined from the complex spectra using specific procedures with helping the quantitative analysis program for energy dispersive X-ray fluorescence analysis [2]. In paper of Sawhney and G.S. Lodha [2], a FORTRAN-77 program, GEOXRF, is described for quantitative energy dispersive X-ray fluorescence analysis. The program employs the fundamental parameter method for quantitative analysis and corrects for absorption of X-rays, the result of inter-element effects, by iteration. To reduce computer storage requirements, parameterized equations for mass absorption

coefficients, emission-line energies and absorption-edge energies are used. The program is designed to be used in stand alone mode.

An X-ray fluorescence method without standards or reference materials for stainless and tool steel samples was used by Barrea and Mainardi [3]. They identified two fluorescence spectra for an unknown sample under two different sets of irradiation conditions providing a system of non-linear equations by means of the theoretical description of primary and secondary fluorescence intensities. The procedure has been tested with NIST standard reference samples, with excellent results.

In the papers of Tiwari and others [4] non-linear least square fitting (NL-LSF) procedures have been used to determine accurately the fluorescent peak intensities. The method has been tested by analyzing several standard reference samples and 304 and 316 steel samples assuming as unknown. The EDXRF results have also been compared with the results obtained same samples by vacuum emission spark spectrometry (VES). Obtained values for concentration in steel samples match quite well with their certified values.

R.M. Ion [5] and others have presented the results of a simple method of nondestructive and standard less analysis of stainless steel samples with EDXRF establishing the correlation between the calculated concentration and certified concentration values for various elements.

Seven stainless steel samples (70093, 86081, 87118, 104164-0.4; 104164-0.5, 104165 and X5CN18-10/1.1) have been analyzed in this paper by EDXRF. The samples have been cleaned and degreased. The composition ranges have been discussed in correlation with technological parameters.

2. EXPERIMENTAL PART

2.1. APPARATUS AND METHOD

For the experiments in this paper, was used X-ray fluorescence spectrometer PW 4025 - MiniPal - Panalytical. The MiniPal 2 energy dispersive XRF spectrometer performs non-destructive analysis of elements from sodium to uranium, in concentrations from 100% down to ppm levels. The sample preparation is simple, inexpensive and fast. Based on the latest Si-PIN technology, the MiniPal 2's detector combines high efficiency with a high resolution. The detector is thermoelectrically cooled, avoiding all the costs and inconvenience of liquid nitrogen cooling associated with other EDXRF systems.

The EDXRF measurement was performed with the following experimental parameters values: side window tube, high voltage tube: max. 30 kV; emission current: max. 1 mA; power: max. 9 W; air cooled [5].

2.2. MATERIALS

Seven stainless steel samples (70093, 86081, 87118, 104164-0.4; 104164-0.5, 104165 and X5CN18-10/1.1) have been analyzed in this paper by EDXRF. The samples have been cleaned and degreased.

## 3. RESULTS AND DISCUSSION

Fig. 1 shows the fluorescence spectrum of stainless steel samples (high-strength low-alloy steel) where elements like Fe, Cr, Ni, Mn, can be clearly identified; traces of Si, S, K, Cu in lower concentration can be also observed; the Fe peaks are due to the photons scattered from the tube target.

The composition ranges samples 70093, 86081, 87118, 104164-0.4; 104164-0.5, 104165 and X5CN18-10/1.1 are presented in Table 1 and EDXRF spectra of them are presented in Fig. 1.
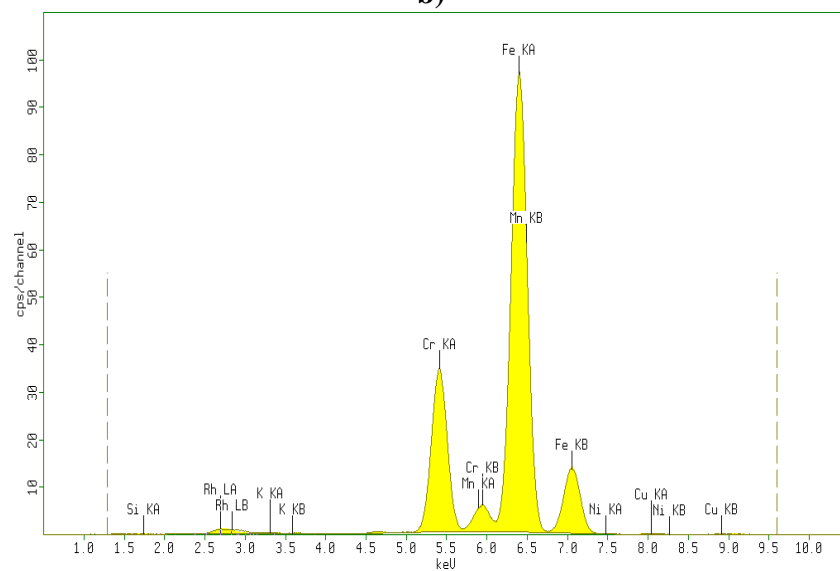
Table 1. Elemental composition of analyzed stainless steel

Sample / Element	70093	86081	87118	104164-0.4	104164-0.5	104165	X5CN18-10/1.1
Si [%]	0.2	0.2	0.1	0.52	0.4	0.5	0.2
S [%]	0.01	0.06	-	0.05	0.1	0.12	0.09
K [%]	0.31	0.86	0.39	0.51	0.25	0.35	0.6
Cr [%]	17.1	17.3	15.1	15.8	15.8	15.8	17.5
Mn [%]	1.0	1.1	0.56	1.1	1.3	1.2	1.4
Fe [%]	70.8	72.3	83.7	75.6	75.5	75.3	69.8
Ni [%]	10.4	8.28	0.03	6.39	6.44	6.47	7.77
Cu [%]	0.19	-	0.20	-	0.22	0.24	-
Ca [%]	-	-	-	-	0.01	-	0.03
Pd [%]	-	-	-	-	-	-	2.6

a)
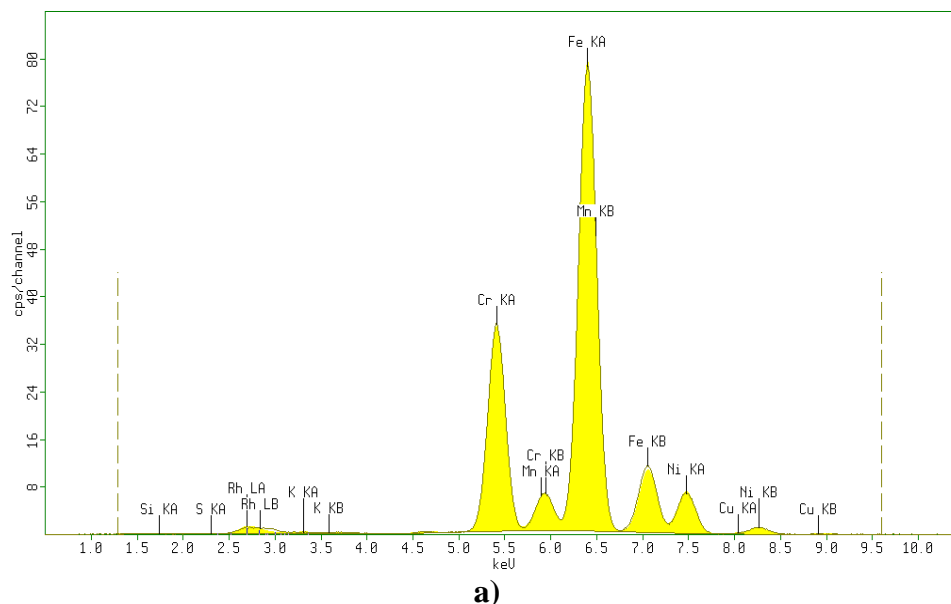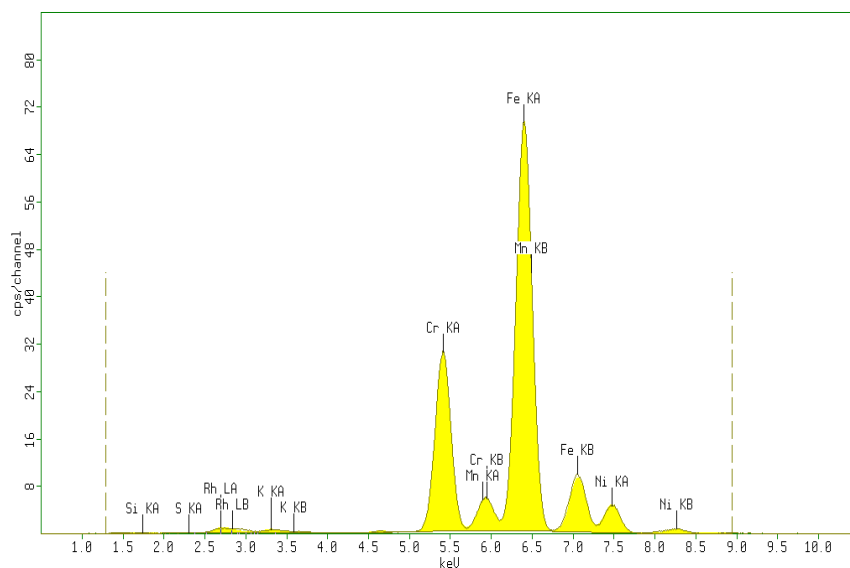
b)

c)

d)

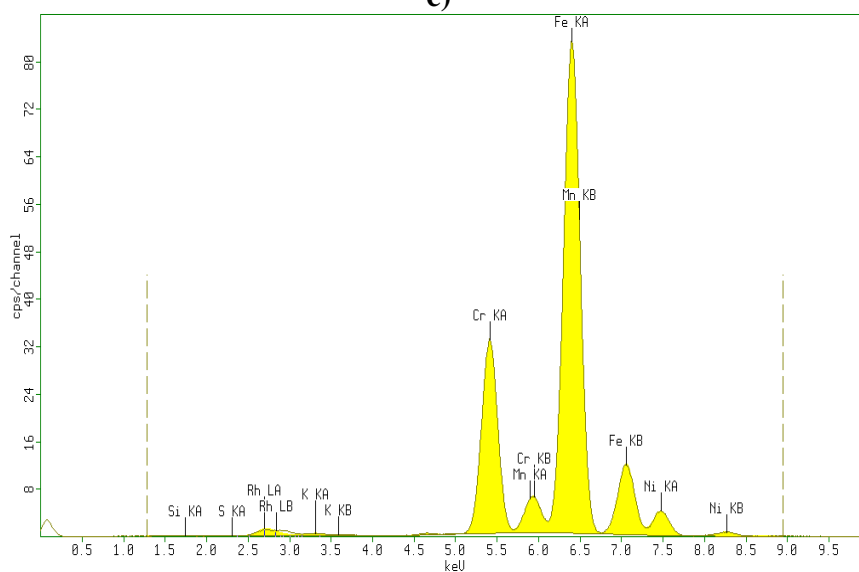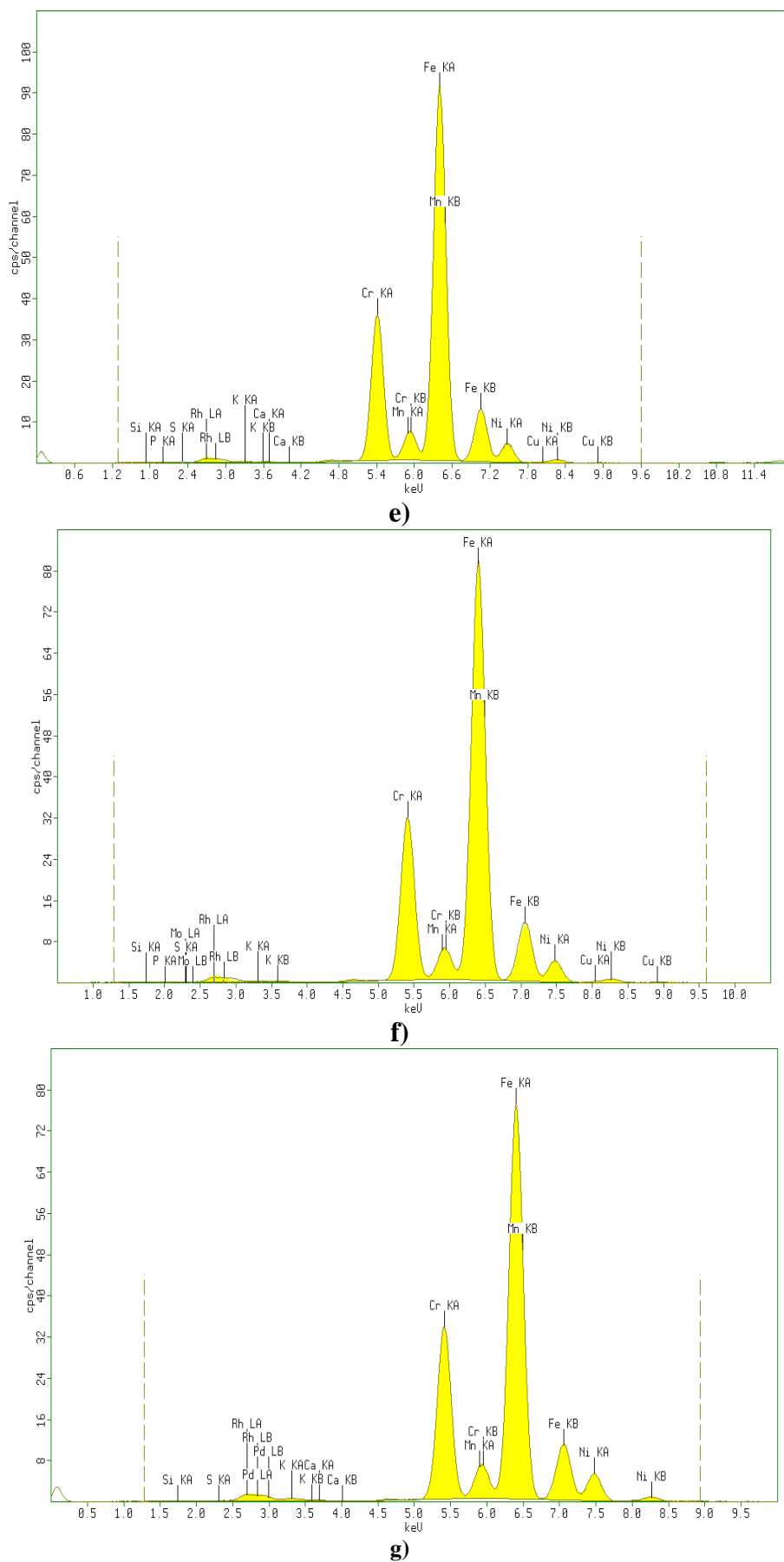**Fig. 1. EDXRF specter of unknown elemental composition of stainless steel: a) 70093; b) 86081; c) 87118; d) 104164-0.4; e) 104164-0.5; f) 104165 and g) X5CN18-10/1.1.**

The results of measuring of fluorescence spectra (quantitative analysis) for seven types of unknown elemental composition [%] high-strength low-alloy stainless steel analysis give us certain results for analysis of trace elements like Si(0.1–0.52), S (0.01-0.12), K (0.25-0.86), Cu (0.19-0.24), Ca (0.01-0.03) and Cr (15.1-17.5), Mn (0.56-1.4), Fe (69.8-83.7), Ni (0.03-10.4) as major elements in stainless steel.

4. CONCLUSIONS

Energy dispersive X-ray fluorescence (EDXRF) technique has become a powerful technique for non-destructive multi-element analysis of materials. A simple method for the analysis of different stainless steel samples is presented in this paper, which is based on radioisotope excited energy dispersive X-ray fluorescence (EDXRF) spectrometry and does not require any type-standards. In these stainless steel (SS) samples have been detected the following element concentrations [%] Si(0.1–0.52), S (0.01-0.12), K (0.25-0.86), Cu (0.19-0.24), Ca (0.01-0.03) as minor components, while Cr (15.1-17.5), Mn (0.56-1.4), Fe (69.8-83.7), Ni (0.03-10.4) are present as major components. EDXRF is a relatively simple and inexpensive alternative compared to other techniques.

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