JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS Vol. 8, No. 1, February 2006, p. 222 - 224

Determination of carbon in steels using particle-induced gamma ray spectrometry

A. ENE*, I. V. POPESCU[a], T. BĂDICĂ[b]
Department of Physics, Faculty of Sciences, "Dunarea de Jos" University of Galaţi, Domneasca 111, 800201 Galaţi, Romania
[a]*Department of Physics, Faculty of Sciences, "Valahia" University of Târgovişte, Romania,*
[b]*"Horia Hulubei" National Institute of Physics and Nuclear Engineering, P.O. MG-6, Măgurele, Bucharest, Romania*

The analytical use of prompt gamma rays arising from the nuclear reactions ^{12}C (p, p'γ) ^{12}C, ^{12}C (p, γ) ^{13}N, ^{12}C (p, n) ^{12}N, ^{13}C (p, p'γ) ^{13}C, ^{12}C(d, p)^{13}C, ^{12}C(d, n)^{13}N induced by 5.5 MeV protons and 5 MeV deuterons during the bombardment of steel targets has been investigated. Being an on-line technique, the sensitivity of prompt γ-ray analysis using protons (p-PIGE) or deuterons (d-PIGE) critically depends on the level of background activities from the matrix nuclides under proton and deuteron irradiation. Carbon can be determined in steel from the very high yields of the 4439.1 keV (1,0) gamma-rays resulted respectively from the reaction ^{12}C (p, p'γ) ^{12}C, as well as the 3089.4 keV (1,0), 3684.5 keV (2,0) and 3853.8 keV (3,0) gamma-rays resulted from the reaction ^{12}C(d, p)^{13}C, the excellent peak to background ratio and the absence of other peaks in the 3-4 MeV energy range leading to a good sensitivity. From the PIGE spectra we have obtained a detection limit of carbon determination in a steel standard target (with a carbon concentration of 670 ppm) of 10 ppm in p-PIGE and 12 ppm in d-PIGE, which are much lesser than the reported values in the literature, of 800 ppm for pure carbon target in p-PIGE analysis, and 270 ppm in iron matrix in d-PIGE analysis.

(Received October 14, 2005; accepted January 26, 2006)

Keywords: Steels, Carbon determination, PIGE analysis, Nuclear reactions, Proton and deuteron irradiation

1. Introduction

The measurement of low concentrations of light elements is often useful in materials science, particularly in metallurgy. Metals, alloys and semiconductors have been actively investigated because of carbon's effect on both physical and electrical properties of its host matrix. Also, the properties of steels are critically dependent on their carbon content and its spatial distribution.

Some of the analytical procedures that have been employed in the quantitative determination of carbon include [1,2]: classical combustion techniques, Photon Activation Analysis (PAA) or Charged Particle Activation Analysis (CPAA) using ^{6}Li ions [1] and deuterons [2]. Some of the activation techniques, although sensitive, are problematic due to interferences from oxygen and nitrogen present in the sample. The combustion method can become unreliable at trace levels because of the limited sensitivity and blank-value problems.

Neutron Activation Analysis (NAA) is, in general, a very sensitive method but has limited use for light elements. Also, Particle-Induced X-ray Emission (PIXE) fails in the situation where the species of interest has a low atomic number because the low carbon K X-ray fluorescence yield is strongly attenuated by the absorption edge of higher atomic number elements present in the sample.

Ion beam Analysis (IBA) is often used and one of the first IBA technique on steel made use of the ^{12}C (p, γ) ^{13}N nuclear reaction [3]. The yield of this reaction is low and the sensitivity of the method is poor. The (d,n) activation reaction have very high sensitivities, but because the only γ-activities produced in these reactions, interfering activities (especially those from other light elements) might necessitate tedious time-decay analyses [4]. Prompt γ-ray analysis (PIGE – proton induced gamma emission) offers an alternative in measuring light elements and has the advantage that γ-rays from the different light elements can be easily distinguished by their energies [5, 6]. Nuclear Reaction Analysis (NRA) allows for the determination of light element concentrations in heavier element substrates, providing a depth distribution of the isotope of interest [7,8].

In this paper the analytical use of prompt gamma rays arising from the nuclear reactions ^{12}C (p, p'γ) ^{12}C, ^{12}C (p, γ) ^{13}N, ^{12}C (p, n) ^{12}N, ^{13}C (p, p'γ) ^{13}C, ^{12}C(d, p)^{13}C, ^{12}C(d, n)^{13}N induced by 5.5 MeV protons and 5 MeV deuterons during the bombardment of steel targets has been investigated. Being an on-line technique, the sensitivity of PIGE critically depends on the level of background activities from the matrix nuclides under proton or deuteron irradiation.

2. Experimental

The 5.5 MeV proton and 5 MeV deuteron beams have been generated with the help of the 7 MV FN tandem accelerator of the "Horia Hulubei" National Institute of Physics and Nuclear Engineering (NIPNE) Bucharest. The beam current was below 10 nA. Prompt γ-rays produced in the steel plate samples and standards provided by specialists from ISPAT-SIDEX Iron and Steel Works of Galați (Romania) and prepared as thick targets, were measured with a GeHP detector having an active volume of about 100 cm^3 and an energy resolution of 2 keV at 1.33 MeV, placed at 90° with respect to the beam.

Attempting to enhance some weak reaction channels by eliminating other channels or decreasing the spectral background, coincident gamma spectra were measured simultaneously with the conventional ones, by means of a multiparameter analyzer system [9-11] and processed off-line.

3. Results and discussion

Details of the prompt γ-rays spectra obtained during the bombardment of a steel standard having a carbon content of 670 ppm, with 5.5 MeV protons and 5 MeV deuterons are presented in Figs. 1 and 2, respectively, and the γ-ray lines in the spectra used to identify carbon in steel are labelled in accordance with the convention ^{A}X (p,b) (t, s) where ^{A}X is the target nuclide; b – the emitted nuclear product particle in the nuclear reaction induced by incident particle p (protons or deuterons); t and s – the level numbers in the heavy product nucleus between which the γ transition occurs.

Gamma energies corresponding to the transitions in the nuclei of interest were extracted from the nuclear level schemes [12] and from the yields of prompt gamma-rays resulted from the irradiation of pure elemental targets with protons [13] and deuterons [14].

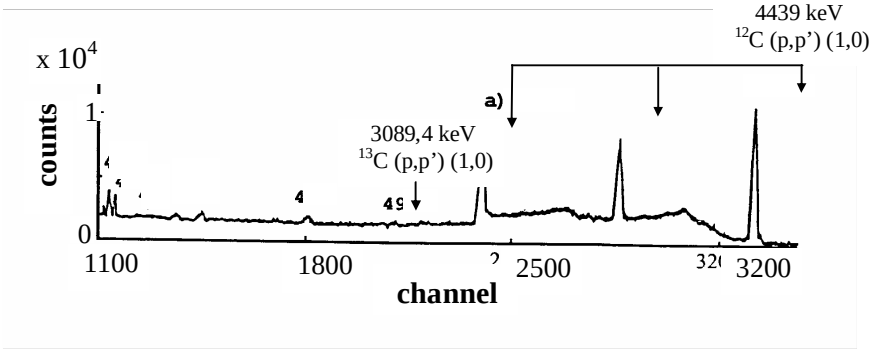

Fig. 1. Detail of the prompt γ spectrum obtained during the proton bombardment of a standard steel sample.

Carbon can be determined in steel from the very high yields of the 4439 keV (1,0) gamma-rays resulted respectively from the reaction ^{12}C (p, p'γ) ^{12}C (Fig. 1), as well as the 3089.4 keV (1,0), 3684.5 keV (2,0) and 3853.8 keV (3,0) gamma-rays resulted from the reaction ^{12}C(d, p)^{13}C (Fig. 2), the excellent peak to background ratio and the small number of peaks in the 3-4 MeV energy range leading to a good sensitivity. In Fig. 1 the peak of 4439 keV, showing Doppler broadening, is easily recognizable due to the appearance of the escape peaks of 3417 keV and 3928 keV, marked in the figure by a link to the full energy peak. The peak of 3089.4 keV (1,0) resulting from the ^{13}C (p, p'γ) ^{13}C nuclear reaction is very weak, as can be seen from the same figure. The peaks of 2365 keV(1,0) and 2313 keV(1,0) gamma-ray from the reactions ^{12}C (p, γ) ^{13}N and ^{13}C (p, γ) ^{14}N, respectively, do not appear in p-PIGE spectra of the steel samples, as in the case of γ spectra of pure carbon targets irradiated with protons with energy 1-2.4 MeV, presented in [13].

Even in the case that (d,n) reaction channel would be selected using neutron-gamma ray coincidences measurement, the gamma-rays of 2365 keV(1,0) and 1146 keV(2,1), resulted from the ^{12}C(d, n)^{13}N reaction, were too weak to be used for carbon analysis [9] and traditional PIGE measurements provided better determination of this element. The (p,n) reaction on the most abundant isotope of carbon, ^{12}C, gives no gamma lines in the coincident spectra because it has a reaction energy Q = - 18.1 MeV.

An advantage of this non-destructive method is that carbon can be determined without any background or interference due to matrix activity.

From the PIGE spectra we have obtained a detection limit of carbon determination in a steel standard target (with a carbon concentration of 670 ppm) of 10 ppm in p-PIGE and 12 ppm in d-PIGE, which are much lesser than the reported values in the literature, of 800 ppm for pure carbon target in p-PIGE analysis [5], and 270 ppm in iron matrix in d-PIGE analysis [4].

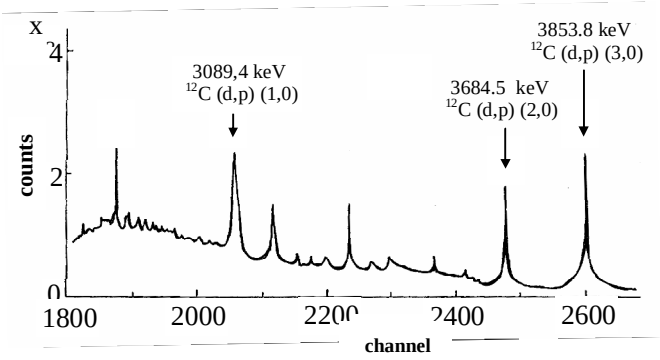

Fig. 2. Detail of the prompt γ spectrum obtained during the deuteron bombardment of a standard steel sample.

4. Conclusions

This work shows the applicability of Particle-Induced Gamma-ray Emission (PIGE) analysis method using beams of 5.5 MeV protons or 5 MeV deuterons for the sensitive determination of carbon in steels relying on the spectrometry of prompt γ-rays from the ^{12}C (p, p'γ) ^{12}C and ^{12}C(d, p)^{13}C nuclear reactions.

References

[1] J. F. Ojo, B. D. Lass, E. A. Schweikert, Determination of carbon in steel by ^{6}Li activation, J. Radioanal. Chem. **60**(1), 261 (1980).

[2] C. S. Sastri, G. Blondiaux, H. Petri, Nucl. Instr. and Meth. B **124**, 558 (1997).

[3] F. Papillon, P. Walter, Nucl. Instr. and Meth. B **132**, 468 (1997).

[4] N. S. Chen, J. H. Fremlin, Radiochem. Radioanal. Letters **4**(6), 365 (1970).

[5] D. Gihwala, M. Peisach, J. Radioanal. Chem. **70**, 287 (1982).

[6] A. Ene, Ph. D Thesis, University of Bucharest, Romania, 1997.

[7] P. N. Johnston, ANALNRA-charged particle nuclear reaction analysis software for the IBM PC, Nucl. Instr. and Meth. B **79**, 506 (1993).

[8] W. Jiang, V. Shutthanandan, S. Thevuthasan, D. E. McCready, W. J. Weber, Carbon analysis using energetic ion beams, Nucl. Instr. and Meth. B **222**, 538 (2004).

[9] A. Ene, T. Badica, A. Olariu, I. V. Popescu, C. Besliu, Nucl. Instr. and Meth. **B179**, 126 (2001).

[10] A. Ene, I. V. Popescu, T. Badica, A. Olariu, Rom. Journ. Phys. **48**(1-4), 445 (2003).

[11] I. V. Popescu, T. Badica, A. Ene, A. Olariu, C. Besliu, Rom. Journ. Phys. **49**(3-4), 385 (2004).

[12] R. B. Firestone, Table of Isotopes, 8th ed., Wiley, New York, 1996.

[13] A. Antilla, R. Hänninen, J. Räisänen, J. Radioanal. Chem. **62**(1-2), 293 (1981).

[14] Z. Elekes, A. Z. Kiss, I. Biron, T. Calligaro, J. Salomon, Nucl. Instr. Methods **B168**, 305 (2000).

*Corresponding author: aene@ugal.ro