Determination of Phosphorus in Low-Alloy Steels by Charged-Particle Activation Analysis

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Determination of small amounts of phosphorus in low alloy steels has always presented a rather difficult problem. Since this element in the low-alloy steels is usually determined by spectrophotometry, it is unavoidable that determination procedures applied in the analysis are relatively complicated. In analyzing small amounts of phosphorus in unknown samples, as a matter of course, it is best to use a nondestructive technique because of the difficulty of dissolving the material without losing the trace element or introducing the contaminant. It can be expected that charged-particle activation analysis has an interesting capability for nondestructive analysis.

In a previous paper[1)], we have studied fundamentally the charged-particle activation analysis of phosphorus in some biological materials using the $^{31}P(\alpha,n)$ ^{34m}Cl reaction. The knowledge of relevant thick-target yield curves obtained in there made it possible to choice the bombarding energy in order to produce the highest yield of the required activity and at the same time to suppress interfering activities as much as possible.

In the present work, nondestructive determination of phosphorus in some low-alloy steels were examined by charged-particle activation analysis using the same reaction as above.

Low-alloy steels were purchased from the National Bureau of Standard and the Iron and Steel Institute of Japan. All irradiation samples were rolled into metal plates having thick enough to stop the incident energy of alpha external beam. The low-alloy steel of NBS-461 was used as a most suitable calibration standard of phosphorus in a series of the present experiment. In order to reduce errors due to the nonhomogeneity and drift of beam, 12 samples were irradiated simultaneously by using a special designed rotating-target assembly. For measurements of the relative thick-target yields of interference elements in the low-alloy steel such as nickel, copper and iron, the target holder inserted 12 different aluminum absorbers was used to degrade the energy of incident alpha particles. The degraded energy of each absorber was calculated on the basis of the range-energy relationships given in literature. The rotating targets to measure the relative thick-target yields were bombarded with 1.5 μA beam of 22 MeV alphas for 32 min, whereas the sample to determine the phosphorus concentration were irradiated with 4 μA beam of 17 MeV for 32 min.

Radioactive nuclides produced were identified by gamma-ray spectrometry using a high-resolution Ge(Li) detector connected to a multichannel pulse height analyser. In the cases of relative thick-target yield measurements, the number of counts in the relevant area below photopeak were corrected by means of the counting efficiency of detector and the gamma-ray branching ratio.

A typical gamma-ray spectrum recorded from the NBS-461 low-alloy steel A is given in Fig. 1. As seen in Fig. 1, it is obvious that the Compton background of positron annihilation due to ^{61}Cu, ^{63}Zn, ^{66}Ga and ^{57}Ni, which are produced mainly through the ^{58}Ni(α,p)^{61}Cu plus (α,n)^{61}Zn $\rightarrow$ ^{61}Cu, ^{60}Ni(α,n)^{63}Zn, ^{63}Cu(α,n)^{66}Ga and ^{54}Fe(α,n)^{57}Ni reactions, in particular prevents the determination of phosphorus at low concentration levels. On the assumption that the low-alloy steel contains one percent of each element as a constituent, the relative thick-target yields of radioactive nuclides produced from nickel and copper were measured, and the results obtained as a function of alpha energy are shown in Fig. 2, together with those of the ^{31}P(α,n)^{34m}Cl reaction. In Fig. 2, those of ^{57}Ni produced from the sample in which no other elements contain are also given. From the analytical point of view, it is suggested that the determination of phosphorus seriously depends on the relative abundance of nickel in the sample. Since the presence of large amounts of nickel is undesirable as described above, it may be concluded that the present method is unsuitable for high-alloy steel samples.

On the other hand, the accuracy of the present method was examined by using a number of low-alloy steel samples with widely varing concentration of phosphorus. The results of analyses as given in Table 1 were excellent agreement with the certified values given by NBS and ISIJ. From these experimental data, it was confirmed that the phosphorus concentration in the low-alloy steel can be determined nondestructively and accurately by the present method. The detection limit for a 5 μA-one half-life irradiation was found to be 0.3 μg of phosphorus.

On the basis of the above results, it is concluded that the present method is applicable to a wide variety of low-alloy steels as a useful complement to other analytical methods.

Reference

1) Masumoto K. and Yagi M., J. Radioanal. Chem., (in press), CYRIC Ann. Rept. (1981) 106.

Table 1. Concentration of phosphorus in low-alloy steels.

Sample	Concentration of P (%)		
	Found	Average	Certified value
NBS-464	0.0166, 0.0160 0.0156, 0.0166	0.0161±0.0004	0.017
NBS-466	0.0109, 0.0117 0.0125, 0.0111	0.0116±0.0007	0.012
JSS-150-6	0.0448, 0.0430	0.0439±0.0009	0.044
JSS-151-6	0.0282, 0.0286	0.0284±0.0002	0.028
JSS-152-6	0.0286, 0.0242	0.0264±0.0022	0.026
JSS-153-6	0.0072, 0.0100	0.0086±0.0014	0.012
JSS-154-6	0.0062, 0.0064	0.0063±0.0001	0.007
JSS-155-6	0.0073, 0.0057	0.0065±0.0008	0.006
JSS-163-3	0.0228, 0.0218	0.0223±0.0005	0.020
JSS-164-3	0.0581, 0.0646	0.0614±0.0035	0.058

JSS: Japanese standard of iron and steel

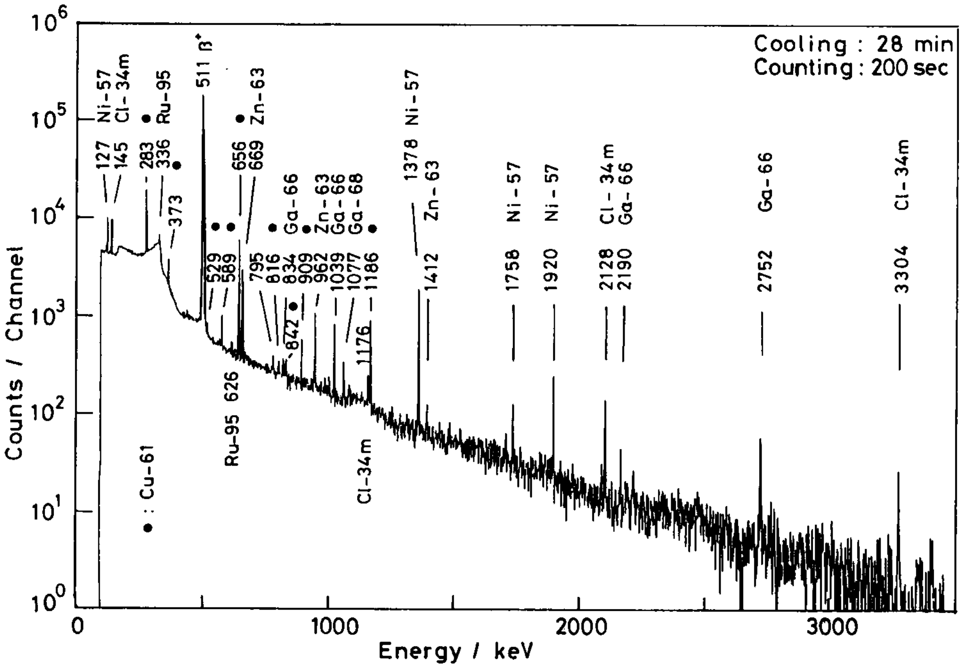

Fig. 1. Gamma-ray spectrum of irradiated NBS-461 low-alloy steel.

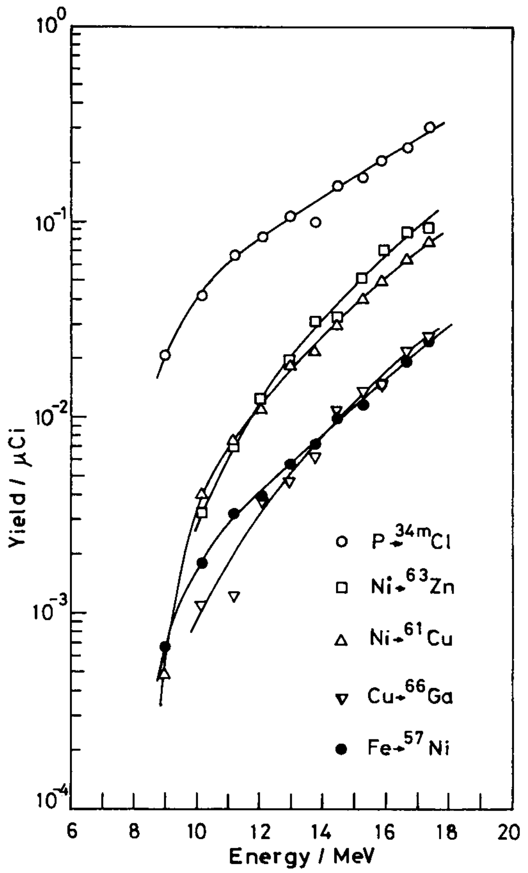

Fig. 2. Relative thick-target yield curves of radioactive nuclides produced from the constituent elements in the low-alloy steel. (Content of element: 1 % = P, Ni, Cu, 100 % = Fe)