II. 2 Determination of Phosphorus in Low-alloy Steels by Charged-particle Activation Analysis

Masumoto K. and Yagi M.
Laboratory of Nuclear Science, Tohoku University

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 \(^{31}\text{P}(α,ν)\) \(^{34}\text{Cl}\) reaction. The knowledge of relevant thick-target yield curves obtained in there made it possible to choose 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($\alpha$,p)$^{61}$Cu plus ($\alpha$,n)$^{61}$Zn $\rightarrow ^{61}$Cu, $^{60}$Ni($\alpha$,n)$^{63}$Zn, $^{63}$Cu($\alpha$,n)
$^{66}$Ga and $^{54}$Fe($\alpha$,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 mea-
sured, and the results obtained as a function of alpha energy are shown in Fig.
2, together with those of the $^{31}$P($\alpha$,n)$^{34}$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 varying concentration of phospho-
rus. 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 deter-
mined non-destructively 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
Table 1. Concentration of phosphorus in low-alloy steels.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Found</th>
<th>Average</th>
<th>Certified value</th>
</tr>
</thead>
<tbody>
<tr>
<td>NBS-464</td>
<td>0.0166, 0.0160</td>
<td>0.1016±0.0004</td>
<td>0.017</td>
</tr>
<tr>
<td>NBS-466</td>
<td>0.0156, 0.0166</td>
<td>0.0116±0.0007</td>
<td>0.012</td>
</tr>
<tr>
<td>JSS-150-6</td>
<td>0.0109, 0.0117</td>
<td>0.0116±0.0009</td>
<td>0.044</td>
</tr>
<tr>
<td>JSS-151-6</td>
<td>0.0282, 0.0286</td>
<td>0.0284±0.0002</td>
<td>0.028</td>
</tr>
<tr>
<td>JSS-152-6</td>
<td>0.0286, 0.0242</td>
<td>0.0264±0.0022</td>
<td>0.026</td>
</tr>
<tr>
<td>JSS-153-6</td>
<td>0.0072, 0.0100</td>
<td>0.0086±0.0014</td>
<td>0.012</td>
</tr>
<tr>
<td>JSS-154-6</td>
<td>0.0062, 0.0064</td>
<td>0.0063±0.0001</td>
<td>0.007</td>
</tr>
<tr>
<td>JSS-155-6</td>
<td>0.0073, 0.0057</td>
<td>0.0065±0.0008</td>
<td>0.006</td>
</tr>
<tr>
<td>JSS-163-3</td>
<td>0.0228, 0.0218</td>
<td>0.0223±0.0005</td>
<td>0.020</td>
</tr>
<tr>
<td>JSS-164-3</td>
<td>0.0581, 0.0646</td>
<td>0.0614±0.0035</td>
<td>0.058</td>
</tr>
</tbody>
</table>

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)