5. DISTRIBUTION STUDIES OF CARBON AND OXYGEN IN METAL SURFACES BY MEANS OF PROTON AND DEUTERON INDUCED REACTIONS

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5.1 Carbon analysis

Depth distribution studies of carbon in steel were carried out in the concentration range 0.003 - 1 %, using proton and deuteron activation analysis and utilizing the reactions:

$$^{12}C(p,\gamma)^{13}N \text{ and } ^{12}C(d,n)^{13}N$$

Evaluations of depth distribution were mainly based on resonances in the excitation function of the reaction $^{12}C(p,\gamma)^{13}N$, which occur at the proton energies of 0.46 and 1.70 MeV, respectively [1]. The carbon determinations were made with the aid of the positron emitter ^{13}N, which has a half-life of 10 min, using either single-peak or coincidence measurements.

The (p,γ) reaction exhibits small cross-section values for the two resonances utilized (127 and 35 μb), and its usefulness is therefore restricted to carbon depth distribution measurements of industrial steel products with fairly high carbon contents. The sensitivity of the method is estimated to be about 0.01 %. The determination of carbon concentration profiles in the concentration range 0.03 - 0.08 % is of importance, for example in connection with carburization processes applied to stainless steels.

Various concentration profiles for carbon in steel, obtained using the (p,γ)-reaction, are given in Fig. 5.1. The resolution of the layers investigated amounted to 0.3 - 2 μm, depending on the depth from the surface.

During proton irradiation the positron emitter ^{15}O with a half-life of 2.05 min is produced from nitrogen through the reaction:

$$^{14}N(p,\gamma)^{15}O$$

This reaction [2] also exhibits resonances suitable for depth distribution evaluations. Experiments utilizing these resonances are in progress.

The reaction $^{12}C(d,n)^{13}N$ was also utilized for the analysis of carbon in steel surfaces. The nuclide ^{13}N formed was used for the identification of carbon. This reaction does not exhibit resonances in the excitation function, which complicates the evaluation of depth distribution. In this case, therefore, an attempt was made to determine the concentration profiles using the principle of convolution, but the method did not yield reproducible results. However, preliminary studies suggest that the carbon concentration profiles may be determined using the (d,n) reaction in combination with neutron time-of--flight measurements [3].

A device for automatic sample exchange which is remotely controlled has been developed. With this arrangement prompt gamma-rays as well as gamma-rays from induced radionuclides can be measured. Charged particle spectroscopy can also be carried out using this device. The arrangement is suitable for routine analysis of carbon or oxygen in metals.

During the irradiation procedure considerable amounts of energy are dissipated into rather small volumes due to the slowing-down of protons or deuterons. Experiments have revealed that when thin iron foils are irradiated, they often melt in the zone exposed to the particles. The heat evolved might give rise to carbon diffusion, resulting in carbon losses from the volume analysed. In steel a transport effect is already noticeable at a few hundred degrees C. At 500°C, carbon may move a distance of 5 μm in a few minutes [4]. In depth distribution analysis layers are studied, which have a resolution of the order of magnitude of 1 μm. Consequently diffusion effects have to be considered, and for this purpose the temperature rise in the irradiated volumes has been both calculated and measured. The temperature distribution around the irradiated zone was measured in detail by means of thermocouples.

5.2 Oxygen analysis

The reactions $^{16}O(d,n)^{17}F$ and $^{16}O(d,p)^{17}O$ were utilized for the surface analysis of oxygen in zircaloy samples. The carbon content was determined with the aid of either the positron emitter ^{17}F, which has a half-life of 66 sec, or the prompt gamma-rays of 0.87 MeV arising from the (d,p)-reactions. An attempt was made to carry out

depth distribution analysis using the principle of convolution, since there are no resonances in the excitation functions of these reactions [5]. However, the technique of deuteron irradiation combined with neutron time-of-flight measurements seems to be more suitable for these studies [3].

For samples with higher oxygen contents, the reaction $^{18}O(p,\gamma)$ ^{19}O was utilized. This reaction features resonances in the excitation function [6], and could thus be used as an internal standard for the determination of oxygen concentration profiles.

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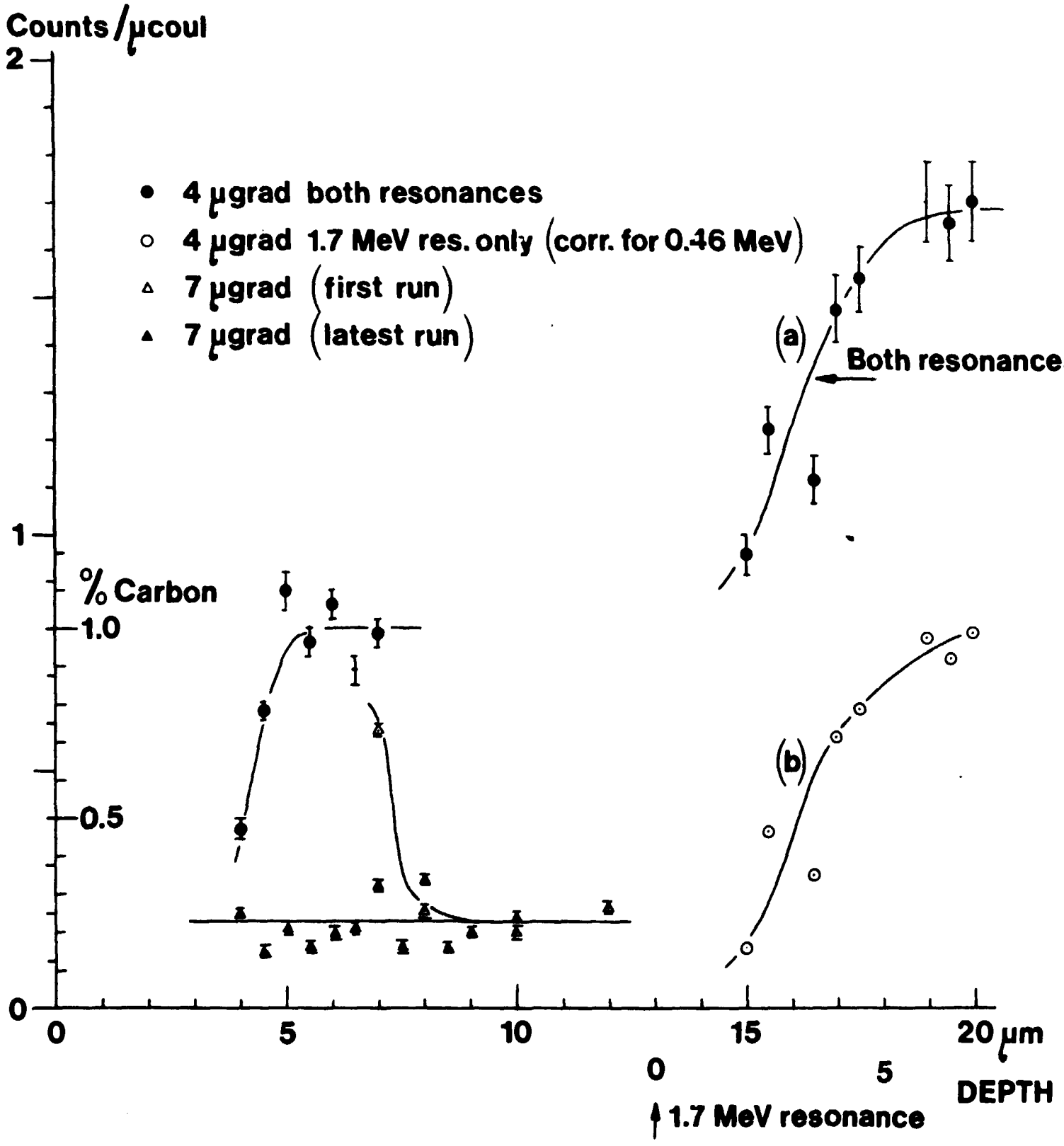

Fig. 5.1 Profiles of carbon gradients at 4 μm resp. 7 μm depth. Integral yield from 0.46 MeV resonance (left half) and 1.70 MeV resonance (right half) for $^{12}C(p,\gamma)\ ^{13}N$ reaction.