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MEASUREMENT OF THE MICROSCOPIC DISTRIBUTION OF OXYGEN AND CARBON IN METALS BY He^3 ACTIVATION*

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Standard methods allow the determination of oxygen and carbon in macroscopic metal samples. However, it often is not possible to obtain detailed information on the distribution within a small sample containing these elements in low concentration. This paper describes how the microscopic distribution of oxygen and carbon can be qualitatively determined in some metals by He^3 activation⁽¹⁾ and subsequent autoradiography.

The polished metallographic sample was covered with a thin (0.00002") nickel foil to minimize carbon deposition during irradiation from diffusion pump oil. The sample was placed inside the vacuum system of the accelerator and irradiated with a defocused beam for about 20 minutes with 0.5 μA of 6.5 Mev He^3 . Immediately after irradiation, the foil was removed and the sample placed on a microscope slide with a 10 micron photographic emulsion. A series of exposures was made during a time interval of about five hours. The composite decay curve was measured during the emulsion exposures by placing the sample and the microscope photographic plate between two NaI crystals in coincidence to detect annihilation radiation. Figure 1 shows a typical decay curve in which the sample was irradiated for 2.5 hours in order to enhance the F^{18} activity. An auxiliary integrating scaler, operated from the same signal, was used as an exposure meter, and exposure corrections

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were made, if necessary, after viewing the developed slides. The composite decay curve was analyzed on a numerical computer to determine the relative contributions of the individual components. This information was utilized to deduce the principal isotope which caused any particular exposure. By calibrating with known samples, it was possible to determine the amount of carbon, nitrogen, and oxygen, as well as the microscopic distributions of these elements.

Pertinent information on the reactions employed are:

<u>Reaction</u>	<u>Half-Life</u>	<u>Positron Energy</u>
$O^{16} (He^3, He^4) O^{15}$	125. min	1.7 Mev
$O^{16} (He^3, p) F^{18}$	110. min	0.65 Mev
$N^{14} (He^3, He^4) N^{13}$	10.1 min	1.2 Mev
$C^{12} (He^3, He^4) C^{11}$	20.4 min	0.97 Mev

The important advantages of He^3 activation are the high sensitivity for low "Z" elements in high "Z" matrices and the short range of penetration (about 0.001"). This makes it possible to obtain adequate autoradiographic exposures for impurity levels in the order of 100 ppm. Concentrations at grain boundaries are visible and bulk diffusion can be studied. Several parameters can be varied for optimizing sensitivity for detection of a particular element, and the distributions of other elements can be determined sequentially.

Some of the difficulties with the method are: surface contamination, interferences between different isotopes, and uneven beam exposure. The surface contamination can be reduced by controlled etching. Interferences can be minimized by selection of the proper irradiation and film exposure parameters, and the beam inhomogeneity can be ameliorated by defocusing and sweeping the beam in two dimensions.

REFERENCE:

1. Samuel S. Markowitz and John D. Mahony, "Activation Analysis for Oxygen and Other Elements by Helium-3-Induced Nuclear Reactions", Analytical Chemistry, Vol. 34, March 1962, pp. 329-35