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Trace Element Detection at the Atomic Level by Atom Probe Microanalysis

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III. DETECTING AND LOCATING RESIDUAL ELEMENTS

Trace element detection at the atomic level by atom probe microanalysis*

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A computer-controlled atom probe time-of-flight mass spectrometer–field ion microscope system has been developed which permits chemical microanalysis of metals and alloys with approximately 1 nm spatial resolution, good quantitative accuracy and excellent sensitivity for the detection of trace elements. The mass resolution is sufficient to separate adjacent isotopes of the transition elements, and the detection efficiency for single atoms is approximately 90 %. Operation is carried out under ultra-high vacuum conditions to minimize background noise levels. Detection of local concentrations of trace elements and impurities of the order of 0.1 at. % is routinely obtained with this instrument.

An additional analytical facility, termed an imaging field desorption atom probe, (i.f.d.a.p.) has also recently been constructed. This produces a picture of the spatial distribution of a selected chemical element over the whole of the imaged region of the field-ion specimen; the information obtained is essentially complementary to that from the conventional time-of-flight atom probe.

Some examples of the applications of these instruments are as follows.

1. Steels. Pearlitic steels are being investigated to determine the distribution of alloying additions Mn, Cr and Si, and residuals Ni, Cu, etc., between the ferrite and cementite phases and the ferrite–cementite interface. Substantial partitioning of alloy elements is found to occur at transformation temperatures as low as 550 °C, particularly with Cr. Enrichment of Mn levels in the ferrite–cementite interface is also found, and there is some evidence of the enrichment of metallic residuals at the same interface (Miller & Smith 1977). The observed rates of partitioning of substitutional elements are much greater than expected on the basis of available bulk diffusion data, and imply the existence of an efficient short circuit diffusion path along the austenite–pearlite interface.

2. Nickel superalloys. A complex cast nickel-based superalloy, IN939 (Ni + 22.5 % Cr, 19 % Co, 2 % W, 1.4 % Ta, 1.0 % Nb, 3.7 % Ti, 1.9 % Al, 1.0 % Zr, 0.14 % C, 0.01 % B) is being studied to investigate the partitioning of alloy elements between the γ, γ' and carbide phases, and to identify the locations of the trace additions. Results for partitioning between γ and γ' phases have been published elsewhere (Beaven *et al.* 1977). The primary MC carbide phase has been shown to contain the metallic elements Ti, Nb, Ta, W, Zr and Cr; traces of N, B, and Si are also present in this phase, in addition to S. Enrichment of boron is found in the

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carbide–matrix interface, to at least 0.2 at. %. The spatial distribution of selected elements in this alloy, and in Nimonic 90A, is also being studied by using the i.f.d.a.p.

3. Tungsten. The segregation of impurities and dopant elements to grain boundaries and small precipitates in doped tungsten lamp wire is being investigated. Severe intergranular embrittlement often leads to specimen fracture in recrystallized material, but it has been possible to study the composition of small ($\leqslant$ 5 nm diam.) precipitates located both in the matrix and in grain boundaries. These precipitates are basically tungsten oxides, though containing traces of other elements, notably Al, Si, Na, K, and Fe. The solid particles may be of major importance in the retention of the more volatile dopant elements during the heat treatment of tungsten wire.

References (Beaven *et al.*)

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