A Neutron Small-Angle Scattering Investigation of Voids in Neutron-Irradiated Aluminum Single Crystals*

By R. W. Hendricks,† J. Schelten and W. Schmatz

Institut für Festkörperforschung der Kernforschungsanlage Jülich, Germany (BRD)

Large single crystals of high-purity aluminum (99·9999 + %), in the form of cylinders 25 mm dia × 40 mm long, were irradiated in the removable beryllium shield of the Oak Ridge High-Flux Isotope Reactor to fluences between 5×10^{20} and $1·5 \times 10^{21}$ n/cm² ($E > 0·1$ Mev).‡ Six of these crystals have been examined at the neutron small-angle scattering facility at the FRJ-2 reactor in Jülich. Scattering curves have been measured in essentially a point geometry at specimen-to-detector distances of 12, 5, 2, and 1 m, which cover a K range of 2×10^{-3} to 2×10^{-1} Å in overlapping steps. The scattering from the irradiated specimens is well above the scattering from the unirradiated single crystals except at the largest K vectors, where the intensity is principally the weak incoherent scattering from Al. Non-symmetric scattering effects have been observed in the high-angle tails of the curves from the unirradiated specimens. Throughout, especial attention has been paid to the scattering curves in the regions of relatively high K where a previously small-angle X-ray investigation has suggested that there may be some effects from voids or other scattering centers which are below the limit of resolution of the electron microscope. These data are compared with electron microscopy, small-angle X-ray scattering, and immersion density measurements. The void-size distribution function and its first three moments are computed.

Application of SAXS to the Study of Microporosity of Graphitic Materials for High-Temperature Nuclear Reactors

By P. Krautwasser, K. Koizlik and H. Nickel

Kernforschungsanlage Jülich GmbH, D-517 Jülich, Germany (BRD),

P. Koss

Reaktorzentrum Seibersdorf, A-1082 Wien VIII, Austria

and W. Ruland

Universität Marburg, 355 Marburg, Germany (BRD)

Measurements of microporosity changes in graphitic materials for high-temperature reactors – pyrocarbon, graphite and matrix materials – during heat treatment and irradiation were expected to be of interest because of the following reasons: (*a*) porosity and pore-size distribution seem to be primarily responsible for fission-product transport, (*b*) irradiation experiments show a strong influence of density, *i.e.* of porosity, on the irradiation behaviour. First investigations showed the presence of a pore-size distribution in the range of about 5 Å (vacancies) up to more than 5000 Å diameter in all these graphitic materials. It can be assumed that there exists a large variety of pore shapes, from spherical to wedge-shaped pores. Study on pyrocarbon: The mechanical behaviour of pyrocarbon coating layers during heat treatment and irradiation is partly governed by creep processes. Creep processes are generally explained by stress-directed motion of vacancies or small clusters of vacancies which cause an increase in anisotropy. This should result in an overall change in the pore-size distribution. Therefore the first

part of the paper compares changes in texture and pore size distributions. The results are (a) during heat treatment a significant growth of the energetically preferred bigger pores, consuming the small ones, can be noted, (b) the changes in texture increase with increasing concentration in vacancies or small vacancy clusters and internal or external stresses, (c) creep in pyrocarbon leads to a strong decrease in pore concentration of 10 Å pores which in turn should lead to a decrease in creep processes. Study on matrix material and graphite: In contrast to graphite, matrix material additionally contains disordered, non-graphitized binder regions. In these regions an irradiation-induced graphitization takes place at irradiation temperatures far below the usual graphitization temperature. This graphitization leads to a higher order in the binder which should be perceptible by a decrease in vacancy-cluster concentration. The change of microporosity in matrix material and graphite was measured as a function of the dose of fast neutrons. The results are: The concentration of small pores is reduced in the binder by irradiation-induced graphitization. The final value is caused by a balance of two contrarily acting processes. The production of new defects during irradiation leads to a lower state of order in the material. On the other hand, the degree of order increases by the use of irradiation-produced Frenkel defects for atomistic transport processes. Therefore in the low-ordered binder an irradiation-induced graphitization is predominant at low doses. With increasing dose and order an increasing disordering process is superimposed until an equilibrium is reached. The concentration of vacancy clusters therefore decreases to a limiting value. Unlike in matrix material, the order in graphite is reduced; the concentration of vacancy clusters increases from the beginning of the irradiation.

Cluster Arrangement of Point Defects in γ-Irradiated LiF

BY W. WAGNER, W. WAIDELICH AND O. WOHOFSKY

Physikalisches Institut der Technischen Hochschule Darmstadt and Physik-Department der Technischen Universität München, Germany (BRD)

The clustering of point defects in γ-irradiated LiF single crystals was investigated by measuring the small-angle X-ray scattering after various γ doses. The curves were obtained by means of a Kratky camera on a 30 kW X-ray generator with rotating anode. With increasing irradiation time the scattered intensity shows a maximum at 10^{-2} Å^{-1}. It is suggested that this maximum is due to a regular arrangement of the defect clusters. The mean distance is 500 Å.

Inhomogeneities in Molten Alloys

BY S. STEEB

MPI-Sondermetalle, 7000 Stuttgart-1, Seestrasse 92, Germany (BRD)

Molten alloys consisting of two components under thermodynamic-equilibrium conditions can be classified into one of three groups: compound, segregation and solubility groups. This classification can best be made by using diffraction methods. In compound-forming alloys often agglomerates are detected, *i.e.* regions with a structure similar to that of the intermetallic compound at the corresponding concentration. Agglomerates only show small differences in electron density or density of scattering length *versus* the matrix and therefore neither from small-angle X-ray scattering nor from small-angle neutron scattering can the shape and magnitude of the agglomerates be determined. Within segregation melts, on the other hand, regions can exist which contain atoms of one kind only, and which are called concentration fluctuations or clusters. In the author's laboratory to date melts from the following systems have been investigated by SAXS and SANS: In–Zn, Bi–Cu, Ag–Pb, Cd–Ga, Bi–Ga, Al–Sn, Al–In, Al–Zn, Mg–In, Al–Ga, Ag–Al and Cu–Sb. A SAS intensity which surmounts the contribution of density fluctuations and therefore can be ascribed to concentration fluctuations was found with melts from the systems Al–Sn, Al–In and Cu–Bi. From Guinier plots particle diameters were obtained of about 12 Å, *i.e.* the inhomogeneities or clusters contain one coordination sphere only. Concerning the structural aspects of these melts, the observed inhomogeneities are identical with the embryos or nuclei necessary for the understanding of solidification. Of course, the run of intensity can also be interpreted with the aid of an Ornstein–Zernike plot which finally yields a correlation length.