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DIFFUSION AND PRECIPITATION OF INERT GASES IN METALS

to

United States Atomic Energy Commission
Washington, D.C. 20545

by

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Significant progress has been made in all areas of work on this project. The results will be presented in two categories, the work on aluminum-helium done by the research associate plus the principal investigator, and the work on niobium-helium done by the graduate student. During this term the principal investigator has devoted roughly one-third or more of his time to this project since the beginning of the term and expects to continue at this same level during the remainder of the current term.

The following communications relating to this work have been prepared:


C00-1799-5, "Inert Gases in Metals," J. R. Cost, (Research project review article for Materials Sciences Newsletter, Purdue University).
Progress has been made in determination of precipitation kinetics in this system using three different experimental techniques: residual electrical resistance, electron microscopy, and low temperature heat capacity. These combined techniques have shown that the precipitation to form bubbles is more complicated than was originally envisioned. The formation of a pre-precipitated structure at temperatures roughly from 350°C to 550°C is indicated. This pre-precipitate does not appear to form into bubbles until annealing temperatures are within 50°C of the melting point. This unexpected result was obtained from heat capacity measurements in which a specimen isochronally annealed from room temperature to 625°C did not show the lambda peak at 2.15K which must be present when helium exists as a separate phase. It had been originally thought that the marked changes in the lattice parameter and electrical resistance near 400°C were due to formation of bubbles which were too small to be seen by electron microscopy. These changes must now be interpreted as being due to formation of a pre-precipitate, which upon formation, decreases the lattice parameter and the extra electrical resistance introduced when helium atoms were injected into the lattice.

To characterize the kinetics of bubble formation in light of these new findings, electrical resistance measurements and electron microscopic examinations are being made upon specimens annealed at much higher temperatures than were formerly considered important. Electrical resistance results for a specimen isothermally annealed at 550°C show a classical form for a precipitation system. There is marked increase to a maximum resistance which occurs after 48 hours of annealing and then a subsequent decrease with further annealing. This behavior can be interpreted as being due to scattering by precipitates with a size near the wavelength of conduction electrons. Further examination
and correlation with electron microscopy is in progress.

By electron microscopy, bubbles have been resolved which are less than 50Å in diameter. They have been shown to have a size range from 50Å to 250Å which roughly, fits a normal distribution centered near 150Å diameter. By examining specimens irradiated to produce varying helium concentrations it has been shown that median bubble size increases with helium concentration. Bubbles have been observed which appear to be faceted, but the condition for this has not yet been determined. It is planned, with the aid of a heating stage, to examine mobility of faceted and unfaceted bubbles and to observe changes in the bubble size distribution with annealing.

The planned low temperature heat capacity measurements of precipitated helium have been successfully made. This effect was found to be as large as predicted. The technique will allow detection of amounts of helium gas as little as one-third of a microgram depending upon the host material. The annealing study using this technique has been written up in preliminary form and will be presented and published as a part of the International Conference on Radiation-Induced Voids in Metals.

In brief, the following results were obtained. (See C00-1799-4 for a more complete description.) First, a one to one correspondence in the presence of helium bubbles was found between the heat capacity method and electron microscopy; i.e., whatever results one method showed, the other method was in agreement. This gives us confidence in the heat capacity method. Second, bubble formation does not occur (for a one hour anneal) at annealing temperatures below 600°C. Third, once nucleation of helium bubbles occurs, precipitation takes place very rapidly. This is probably because helium diffusion is very fast at the high temperatures required to produce bubbles. Evidence for this
is that at 625°C there was no evidence for the precipitation reaction after a one hour anneal, but the reaction was complete after ten hours.

Niobium-Helium System

An inverted torsional pendulum had to be built before the internal friction measurements planned for this system could be made. This is because the weight of the inertia arm of the normal pendulum puts too much tensile stress on the 10 mil diameter niobium wires. Such a pendulum and an associated vacuum system has been constructed. Results obtained for the internal friction at roughly 1 Hz for alpha-irradiated niobium are shown in Figure 1. Some temperature hysteresis due to variations between specimen and furnace temperature may be observed in these results. Also, the vacuum system is not yet tight as is indicated by the increase during the cooling run of the height of a damping peak near 165°C which is due to dissolved oxygen.

The result of interest is a large peak at roughly 445°C. This peak did not exist for the single non-irradiated specimen examined. Several possible explanations for the peak are possible. Some of these involve radiation-produced point defects. Another involves the possible stress-induced motion of interstitial helium atoms. If this mechanism is involved, then it will be possible for the first time to obtain a reliable measurement of the diffusivity of helium in a metal. Furthermore, we do not expect it to be difficult to establish whether or not the peak is due to helium since its presence can be correlated with the kinetics of bubble precipitation as determined by the other techniques we have made use of. This work is being conducted by Mr. Dave Johnson as a part of his Ph.D. thesis. It is expected to be completed roughly one year from now.
Figure 1. Internal friction of niobium irradiated with alpha particles.