Contract AT(11-1)-2206

VOID NUCLEATION AND GROWTH IN HEAVY
ION AND ELECTRON BOMBARDED PURE METALS

Progress Report

For Period: 1 March to 1 November 1972

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Summary

High purity aluminum has been irradiated in a 1 MeV transmission microscope at 100, 150, 175, and 200°C from 0-8.1 dpa. The damage consisted of a large number of unfaulted, faulted and doubly faulted loops after the 100 and 150°C irradiations. Very little damage was observed during the 175°C study and none during the 200°C test. No voids were observed at any of the above conditions.

A 2 MV electrostatic generator has been modified to accelerate heavy ions. A Physicon heavy ion source has been purchased, modified, bench tested, and installed in the accelerator. Thus far a beam of 20 microamps, principally nitrogen-14, has been extracted from the source. On this basis it is reasonable to expect 2-3 microamps per cm^2 on the target which would correspond to displacement rates of $3-5 \times 10^{-3} \text{ sec}^{-1}$. Three sample chambers have been built and operated at temperatures of 20-200, 20-700, 20-1100°C. A total of 49 samples have been doped with 0.1, 1 and 10 appm He.

A 2 MeV electron accelerator has been modified to deliver a high current of finely focussed electrons to samples on a specially designed sample chamber. Beam currents up to 500 microamps per cm^2 have been observed at 2 MeV. Such an electron flux produces a calculated damage rate of 7×10^{-7} dpa per second.

Objective

The objective of this study is to clarify the important parameters for nucleation and growth of voids in pure aluminum and vanadium. The factors governing the production of voids during neutron irradiation of these metals are difficult to separate because both transmutation reactions and displacement spike cascades are superimposed on the pure displacement damage during irradiation. This study will use a high energy beam of Al^+ or V^+ ions to produce the necessary damage while eliminating the transmutation effects. A high energy beam of electrons will be used to eliminate both the transmutations and displacement spike effects. Selected doping of the samples with helium, hydrogen, Si, Cu, Zn will help to understand the role of these impurities in nucleating voids. This study will concentrate on high purity V and samples of 99.999% Al already extensively analyzed by ORNL scientists after neutron irradiation.

Introduction

This progress report covers approximately the first eight months of the irradiation study of void nucleation and growth in Al. The reasons for this study are given in detail in Reference 1 and will not be discussed here.

The majority of the initial work on this program has been directed toward modifying existing accelerators at the University of Wisconsin. The details of these modifications are given elsewhere.^{1,2} Because the time period between the last progress report² (15 September 1972) and this one is so short, extensive referencing of previous reports will be made here to avoid unnecessary duplication, and in the interest of brevity. The information in this report will try to cover where we have been and where we are going during the remainder of this contract term. Future plans beyond March 1973 will be contained in other proposals.

Progress in Experimental Programs

1) High Voltage Electron Microscopy (HVEM)

The first samples of high purity aluminum obtained from ORNL² have been irradiated by 1 MeV electrons at nominal temperatures of 100, 150, 175, and 200°C. Photomicrographs were taken of the irradiated area before the irradiation and at calculated dpa values of² 0.25, 0.54, 0.84, 1.6, 3.0, 4.1,

5.7, and 8.1 dpa. The first irradiations were conducted at WADCO Corporation in collaboration with Dr. J. J. Laidler because the University of Wisconsin 1 MeV EM-7 microscope has run into unexpected acceptance problems. The studies at WADCO were funded by the University of Wisconsin Graduate School and no contract funds were expended on the actual irradiations. The program will shift to the UW scope once the operation difficulties have been solved.

The analysis of the Al samples is still in progress and only a few new facts have emerged in the month since the past progress report.²

Preliminary analysis reveals that the number density of loops varies from $1.4 \times 10^{14} \text{ cm}^{-3}$ at 0.14 dpa to $5.5 \times 10^{13} \text{ cm}^{-3}$ at 5.7 dpa. The reason for the density decrease is the coalescence and growth of the loops.

Secondly, it is quite apparent that our decision to use the high purity Al in exactly the same fabrication conditions as used by the ORNL workers for neutron irradiation studies bears some further investigation. The dislocation network introduced by pre-irradiation cold rolling does not anneal out in 4-5 hours at 200°C. This network obviously is affecting the nature of the defect structure at the cell boundaries which complicates interpretation of the results. We may have to pre-anneal the samples to provide a relatively dislocation-free sample and sacrifice some of our direct correlation with the ORNL neutron irradiated samples..

The significance of our first test is that voids do not form in heavily damaged electron-irradiated Al. This is supported by recent Japanese work,³ but is in direct conflict with neutron irradiations at the same temperature but a much lower damage level.⁴

There are two immediate possibilities for this behavior: the lack of neutronicallly produced helium for void stabilization, or the lack of sufficiently large displacement spikes which can produce depleted zones for void nucleation. One other possibility exists and that is the effect of surface denuding. Our samples were only ~2 microns thick in the irradiated zone and the higher temperature of the disappearance of loop damage in our samples (175°) versus that observed in thinner samples³ (70°) may support this.

We intend to test the helium nucleation theory by irradiating the same Al at the same temperatures, with the same damage rate and to the same damage levels with one difference, the new samples will contain 10 appm helium. The injection techniques have been described elsewhere.²

The spike theory will be tested by irradiating the Al, with and without He, at the same temperature with the same displacement rates and to the same damage levels with 2 MeV Al⁺ ions instead of 1 MeV electrons. If voids can be produced in high purity Al with 2 MeV Al⁺ ions we might gain some insight into the true role of the displacement spike for nucleating voids.

The effect of sample thickness will be tested, at low dpa values, by bombarding 75 micron thick samples with 1 MeV electrons at $\sim 7 \times 10^{-7}$ dpa per second. It is hoped that the combination of all of the above studies will clearly illuminate the path to take in future experiments.

2) Heavy Ion Bombardment

The major emphasis in this part of the program has been to perfect the experimental facilities. Modifications to the accelerator, too numerous to mention here, consumed several months of time. The purchase, modification and bench testing of a Physicon heavy ion source^{1,2} took longer than was expected. The source, its power supplies, the telemetry system and cooling system are now operative. The einzel lens, which was designed to provide optimum beam optics over the voltage range 500 kV to 2MV, is working properly.

Since the last progress report² an ion beam of 20 micro-amperes, principally nitrogen-14, has been collected at the entrance to the electrostatic analyzer,¹ while the accelerator was operating at 750 kV. Work is presently underway to align the beam handling system (magnetic quadrupole lenses, electrostatic analyzer, magnetic momentum selector and the target chamber) for selection of the aluminum beam. Based on the beam characteristics of the ion source when it was studied on the optical bench, it is reasonable to predict a beam of Al^+ ions of 2-3 μA on the target. This intensity will allow for

defocussing of the beam to minimize localized hot spots while still achieving a final current density of $2-3 \mu\text{A}/\text{cm}^2$. Such a beam corresponds to a displacement rate of $3-5 \times 10^{-3} \text{ sec}^{-1}$.

After a short optimization study, it is expected that the first samples can be irradiated under closely controlled conditions. Sample chambers to control the temperature from 20-200, 20-700, 20-1100°C have already been built.¹

A considerable amount of time has been devoted to injecting 49 samples with helium at concentrations of 0.1 to 10 appm. These samples will be used in HVEM, heavy ion, and electron irradiated Al studies.

Our immediate plans are to duplicate the HVEM studies on pure aluminum to pin down the effect of displacement spikes. The microscopy techniques for analyzing these samples with the 2 microns damage zone have already been developed.¹

3) 2 MeV Electron Bombardment Studies

Like the heavy ion part of this study, most of the early months were spent on accelerator modifications and sample chamber construction.^{1,2} The major problem was to maximize the displacement rate by careful focussing of a 100 microampere, 2 MeV electron beam onto a 5 mm diameter sample. Such a high energy input ($> 1 \text{ kw}/\text{cm}^2$) presented severe temperature control problems which are still being solved.²

It is interesting to note that it is possible to get

dpa rates with the UW accelerator that approach those in the core of the EBR-II reactor ($\sim 10^{-6}$ dpa/sec). Future plans are to begin irradiation of the high purity Al under identical conditions incurred during the HVEM studies. The effect of surface denuding at high temperature will be explored.

Other Work

We intend to use the internal progress reports to document our experimental data collection and calculational procedures. The reader is referred to the first two in this series for some of the work already reported in this area.^{1,2}

Level of Effort in this Program

For the past 8 months and for the remaining 4 months of this contract, the level of effort for the investigators in this program are as follows:

G. L. Kulcinski 50%

J. M. Donhowe 50%

We also have 5 graduate students participating at various levels on this program. Their combined effort will amount to ~ 2.5 man years for the full years.

References

1. COO-2206-1 Progress Report, 1 March to 1 June 1972.
2. COO-2206-2 Progress Report, 1 June to 15 September 1972.
3. K. Shiraishi, A. Hishimumu, Y. Katano, and T. T. Taoka, J. Phys. Soc. Japan, 32, 964, 1972.
4. N. H. Packen, J. Nucl. Mat., 40, 1, 1971.