RECEIVED BY DTIC AUG 19 1968

BNWL-SA-1996

CONF-680604--8

IRRADIATION-INDUCED EMBRITTLEMENT IN STAINLESS
STEEL AT ELEVATED TEMPERATURE

MASTER

by

M. Kangilaski, J. S. Perrin, and R. A. Wullaert

Discussion by H. R. Brager*
Battelle Memorial Institute
Pacific Northwest Laboratory
Richland, Washington

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

*This paper is based on work performed under United States Atomic Energy Commission Contract AT(45-1)-1830.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

fy

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

IRRADIATION-INDUCED EMBRITTLEMENT IN STAINLESS
STEEL AT ELEVATED TEMPERATURE

by

M. Kangilaski, J. S. Perrin, and R. A. Wullaert

Discussion by H. R. Brager
Battelle Memorial Institute
Pacific Northwest Laboratory
Richland, Washington

The authors are to be complimented on a most interesting and informative paper. The significant amount of effort put into interpreting the data is quite evident. We have some recent electron microscopy information¹ of type 304 stainless steel irradiated in the ETR G-7 water loop at 290°C to 6×10^{21} n/cm² ($E > 1$ MeV) which might be helpful in explaining some of the results described by the authors. The as-irradiated microstructure of the type 304 stainless steel we examined exhibited only normal displacement damage characteristic of low irradiation temperatures. A sample cut from a specimen tensile tested at 750°C did, however, contain about 2×10^{15} cavities/cc with an average diameter of 110 Å; these cavities were nearly uniformly dispersed throughout the matrix as well as on grain boundaries. Further investigations showed that the cavities behaved like gas-filled bubbles. It should be emphasized that the quantity of helium calculated to have been produced in our samples of type 304 stainless steel during irradiation was about 1/30 the amount of helium required to balance the surface tension of the observed bubbles. It was felt that hydrogen introduced in the steel by irradiating in the ETR cooling water, the same environment in which the authors' specimens were irradiated, was a possible explanation for the stability of the bubbles in the steel. We observed that

the bubbles were stable at the grain boundaries and there was no discernible change in the grain size to the highest temperature investigated (1090°C).

We propose that the authors' irradiated type 347 stainless steel contain similar gas bubbles. We found that annealing of the irradiated type 304 stainless steel at successively higher temperatures to 1090°C caused the bubble density to decrease, the average bubble size to increase, and the density of the sample to decrease, with no change in the macroscopic surface area of the bubbles. If indeed gas bubbles were also present in the authors' type 347 stainless steel, annealing to 1350°C should cause agglomeration and growth of the bubbles to a size sufficiently large to be observable by surface replica electron microscopy and be detectable by a density decrease of the bulk material.

Since a one hour postirradiation anneal of a sample of our 304 stainless steel to 1090°C did not cause a discernible change in the grain size, we suggest that the pinning of the grain boundaries observed by the authors in their type 347 stainless steel annealed to 1350°C might be due to gas bubbles analogous to the case we studied.

The authors stated that they did not expect voids to be in their stainless steel due to the low irradiation temperature and, therefore, discounted the elevated temperature embrittlement promoted by voids. We feel that due to the similar irradiation environment, similar material, and higher fluence, the authors' steel does contain bubbles which would be resolvable by transmission electron microscopy after a high temperature anneal. Then the bubbles could act as nucleation sites for crack formation and might explain the results reported by the authors, namely the low ductility, intergranular failure of the steel, particularly at high temperatures ($> 600^{\circ}\text{C}$).

The authors found it hard to explain why the extreme embrittlement observed in their specimens was not also observed in other investigations of stainless steel containing equivalent or higher amounts of helium. We feel that 1) the grain boundary pinning and 2) the intergranular failure mechanisms for the behavior of the authors' ETR irradiated type 347 stainless steel need not be related solely to the quantity of helium in the metal but should be related to the density and size of gas-filled bubbles in the metal which are partially filled with helium. Therefore, the authors' expectation that the degree of embrittlement should correlate with the amount of helium present in an irradiated steel (an inference contradicted by experimental observations) may be unwarranted.

The authors did not suggest a mechanism to explain the hardening of both the as-irradiated and the 980°C postirradiated annealed material over the 600°C to 750°C testing temperature range. A possible mechanism can be advanced to explain the high temperature hardening observed. Hardening in both conditions of the irradiated steel can be due to an Orowan interaction of dislocations with NbC particles that precipitate from the matrix at test temperatures above 600°C. During irradiation, the NbC particles are dissolved into the matrix as a result of displacement mechanisms as described for precipitates in Cu-2% Co by Piercy² and for fission gas bubbles in UO₂ by Whapham.³ We believe we have found further evidence for such a mechanism: we have found that commercial type 304L stainless steel irradiated at 400°C in the EBR-II to 5×10^{22} n/cm² (E > 0.1 MeV) contains small M₂₃C₆ precipitates (100-500 Å), whereas the same steel irradiated at 460°C in a lower flux region to 3×10^{20} n/cm² (E > 0.1 MeV) contains large (.2-.5 μ) M₂₃C₆ particles. It would appear that the large M₂₃C₆

carbides were dissolved during irradiation and at the high (400°C) irradiation temperature were continually re-precipitated. In the case of the authors' type 347 stainless steel irradiated at 50°C in the ETR, the NbC would be unable to precipitate during the low temperature irradiation. Since we have observed that a 1010°C anneal of unirradiated type 347 stainless steel will dissolve some NbC particles, it is proposed that during the 980°C vacuum anneal of the authors' ETR irradiated type 347 stainless steel, no significant precipitation of NbC particles occurs on rapid cooling. Therefore, both the as-irradiated and the 980°C postirradiation annealed material would contain a high concentration of dissolved NbC. During the higher temperature tensile tests ($600\text{--}750^{\circ}\text{C}$), the NbC would precipitate and harden the material.

A verification of this mechanism could be made by annealing the irradiated material one hour at 980°C and subsequently annealing one hour in the temperature range where the NbC would precipitate, for example at 750°C , and then testing at a temperature where the postirradiation hardening was not determined, for example at room temperature. This material should exhibit a higher yield strength than the control. Electron microscopy of the various states of the irradiated material should show whether this mechanism is applicable.

REFERENCES

1. H. R. Brager and R. E. Robbins, "Stabilized Cavities in Irradiated Austenitic Stainless Steel:", to be published in Trans. Aime.
2. G. R. Piercy, "The Detection of Displacement Spikes by Measuring the Effect of Particle Size on the Dissolution of Precipitate Particles by Fast-Neutron Irradiation", J. Phys. and Chem. of Solids, 23, 463 (1962).
3. A. D. Whapham, "Electron Microscope Observations of Fission Gas Bubble Distribution in UO_2 ", Nucl. Appl., 2, 123 (1966).