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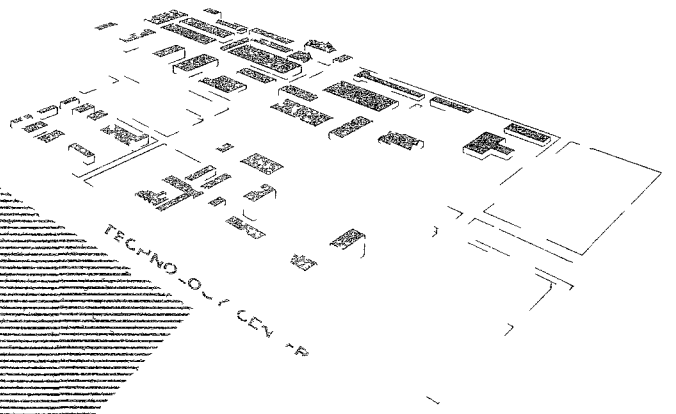

ARF-B6005-2
(Quarterly Report No. 1)

RESEARCH DESIGNED TO EVALUATE Zr-2.5Nb AND Zr-2.5Nb-0.5Cu ALLOYS FOR DELAYED FAILURE HYDRIDE SUSCEPTIBILITY

DISCLAIMER

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of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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RESEARCH DESIGNED TO EVALUATE Zr-2.5Nb AND Zr-2.5Nb-0.5Cu ALLOYS FOR DELAYED FAILURE HYDRIDE SUSCEPTIBILITY

October 15, 1962 - January 14, 1963

Contract No. AT(11-1)-578
Project Agreement No. 23

U.S. Atomic Energy Commission
Chicago Operations Office
9800 South Cass Avenue
Argonne, Illinois

January 21, 1963

RESEARCH DESIGNED TO EVALUATE Zr-2.5Nb AND Zr-2.5Nb-0.5Cu ALLOYS FOR DELAYED FAILURE HYDRIDE SUSCEPTIBILITY

ABSTRACT

The purpose of this program is to determine the extent to which Zr-2.5Nb and Zr-2.5Nb-0.5Cu exhibit delayed failure (static fatigue) under various conditions of hydrogen content, heat treatment, and ambient temperature. Specimens are hydrogenated to 200 ppm or 500 ppm by means of a modified Sieverts apparatus, heat treated or cold reduced, and then evaluated in dynamic tensile and delayed failure tests under a certain set of conditions. At the present time, data are insufficient to form any conclusions regarding static fatigue behavior of these alloys.

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I. INTRODUCTION

This is the first Quarterly Report, covering the period October 15, 1962 to January 14, 1963, on Contract No. AT(11-1)-578, Project Agreement No. 23. This investigation is being conducted under the auspices of the USAEC/AECL Collaborative Program.

Last year, under the program entitled "Delayed Failure Hydrogen Embrittlement of Zirconium," static fatigue evaluation of various zirconium alloys containing up to 500 ppm hydrogen indicated that one material was highly susceptible to this embrittlement phenomenon. These tests, which were performed under relatively fixed conditions, showed that Zr-1.25Al-1Sn-1Mo was highly sensitive to delayed failure whereas unalloyed zirconium and Zircaloy-2 were not susceptible. The alloy Zr-2.5Nb, while fairly notch-sensitive at ordinary rates of tensile testing, was only moderately susceptible to delayed failure when containing 500 ppm hydrogen. At 2000 ppm hydrogen, however, limited experimental data indicated that this alloy was severely sensitive to delayed failure. This material and the modification of this alloy (Zr-2.5Nb-0.5Cu) are presently intended for use as pressure tubes in the Canadian heavy-water moderated/cooled reactor. Thus, the present program is for evaluation of static fatigue susceptibility of these materials under various conditions of hydrogen content, heat treatment, and ambient temperature.

II. EXPERIMENTAL

The scope of work to be performed during the current year includes study of Zr-2.5Nb and Zr-2.5Nb-0.5Cu with three different metallurgical histories--fully annealed, annealed and cold worked, and beta-quenched and tempered. For these conditions, delayed failure evaluation

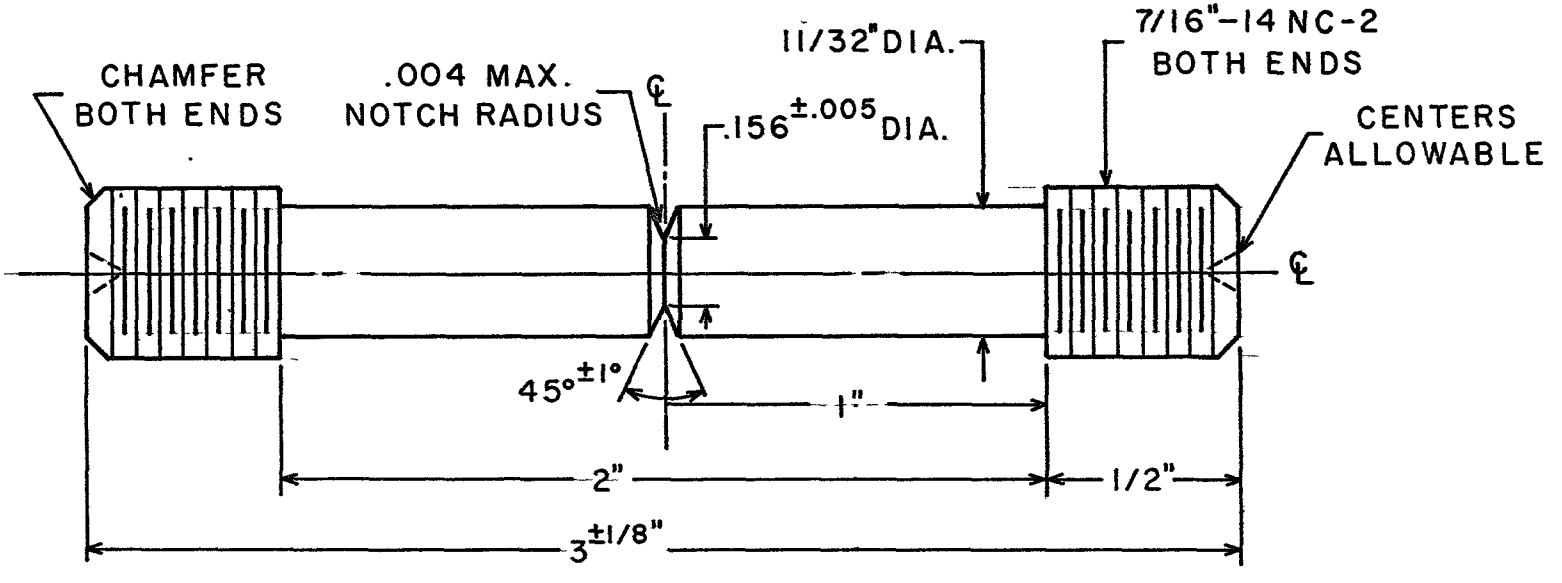
will be performed at room temperature, 250° C, and 300° C, and the specimens will be vacuum annealed or contain 200 ppm and 500 ppm hydrogen. The applied stress will be 95 per cent of the yield strength (at temperature) as determined by dynamic tensile tests. Thus, approximately 250 specimens are to be evaluated during the current year.

It is obvious that a certain combination of variables represent more favorable conditions for delayed failure than others. The program will be initiated by employing the most severe conditions; if static fatigue failure is not observed, then future work can be evaluated in the light of these results. Hopefully, the number of specimens for testing might be diminished without affecting evaluation of delayed failure susceptibility.

The experimental program also includes a cursory study of static fatigue susceptibility of Zircaloy-2 containing hydrides oriented perpendicular to the stress axis. This investigation results from the very interesting studies at Savannah River Laboratory which showed that Zircaloy-2 with oriented hydrides is extremely brittle in tensile tests carried out at ordinary strain rates.

The experimental procedures have not significantly changed from last year. A modified Sieverts apparatus is still employed for hydriding to the desired amounts; however, the temperature used is now 500° C rather than 800° C. This change was necessitated by the required heat treatments; at 500° C, approximately 24 hours is necessary for hydrogen to be absorbed and homogeneously distributed in the specimen.

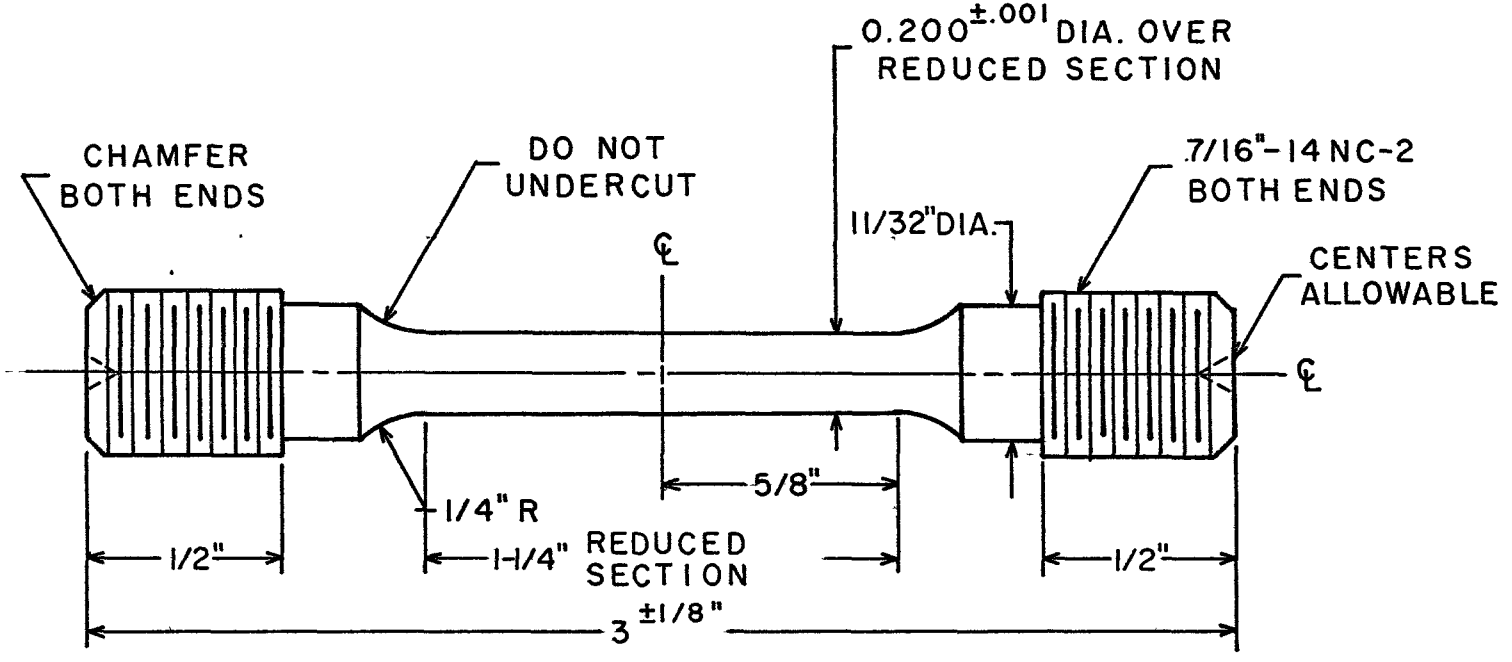
The materials are supplied by Atomic Energy of Canada Limited (Chalk River Laboratory) in the form of 1/2 inch diameter rod in the as-fabricated condition. Three-inch sections are cut from the rods, pickled in a $HF-HNO_3$ solution, and then hydrogenated; heat treatment is then performed in sealed, helium-filled capsules. Tensile and delayed-failure specimens, both notched and unnotched, are machined from these rods to the dimensions shown in Figures 1 and 2. For material which is cold-worked, smaller diameter specimens--but with the same general design--are prepared. The notch radius is further reduced by cutting a 0.001 inch slot at the base; this



MAKE SYMMETRICAL ABOUT ℄
ALL DIMENSIONS IN INCHES

SCALE: DOUBLE SIZE
TOLERANCES: ±1/64" EXCEPT AS NOTED
FINISH: 16 MICRO-INCHES (NOTCH ONLY)

FIG. 1 - NOTCHED TENSILE AND DELAYED FAILURE SPECIMEN



MAKE SYMMETRICAL ABOUT ℄
ALL DIMENSIONS IN INCHES

SCALE: DOUBLE SIZE
TOLERANCES: ±1/64" EXCEPT AS NOTED
FINISH: 63 MICRO-INCHES EXCEPT AS NOTED

FIG. 2 - UNNOTCHED TENSILE AND DELAYED FAILURE SPECIMEN

is accomplished with a 0.001 inch diamter, abrasive-coated tungsten wire moving within the notch while the specimen rotates.

III. RESULTS AND DISCUSSION

Since the materials are to be studied in the heat-treated or cold-worked condition, hydrogenation in the Sieverts apparatus must take place prior to these operations; if not, then the heat treatment effect would be lost or recrystallization of cold reduced specimens would occur. A number of specimens--both binary and ternary materials--have been prepared, and tensile tests have been carried out. However, the results thus far are too cursory to report. A ten-foot rod of the ternary alloy has been received, and tensile and delayed failure specimens are being prepared. The initial set of conditions to be investigated are annealed and beta quenched and tempered at 535° C and 400° C. Evaluation of material will take place at room temperature, and the specimens will be notched and contain 500 ppm hydrogen. Such a set of parameters represents conditions most favorable for delayed failure--with perhaps the exception of cold-worked material.

Studies were performed on the cold swaging (to a 30 per cent reduction in area) of binary and ternary alloys in the annealed condition with 500 ppm hydrogen content. Thus far, fine surface cracks have resulted on every rod, and in some cases an internal center crack along the specimen axis was observed. However, by preparation of a smooth surface finish prior to swaging, beveling of edges, and smaller reductions in area per pass it is felt that satisfactory cold-worked material can be obtained by swaging. If not, the possibility of tensile straining is being considered.

Four specimens of Zircaloy-2 were received from Savannah River Laboratory; two of these had randomly distributed hydrides (about 150 ppm) and the others had hydrides oriented essentially perpendicular to the tensile axis. The accompanying data indicated that the fracture stress of the former group of specimens was 80,000 psi whereas the latter group was 50,000 psi. These specimens were placed in static fatigue at room temperature, and in all cases fracture occurred at the grips which had been welded

to the Zircaloy sheet. From the remaining material, small size sheet specimens were prepared, and loading for delayed failure study was as follows:

random hydride distribution:	70, 000 psi 60, 000 psi
oriented hydride distribution:	40, 000 psi 30, 000 psi

For the above specimens, the one loaded at 70, 000 psi fractured immediately and the one at 60, 000 psi fractured in approximately 150 hours. From our experience on Zircaloy-2 containing 200 ppm hydrogen, the reported fracture stress of 80, 000 psi is perhaps lower than actual. One must also suspect that the reported value of 50, 000 psi for hydride-oriented specimens is not as accurate as desired, for at these applied stresses no fractures have been observed up to 300 hours. Thus, a dynamic tensile test on a rather small specimen of material with oriented hydrides will be conducted which should be very valuable in judging whether or not oriented hydrides lead to increased susceptibility of Zircaloy-2 to delayed failure.

IV. FUTURE WORK

This program is one of a rather straightforward evaluation of Zr-2.5Nb and Zr 2.5Nb-0.5Cu for sensitivity to delayed failure. The scope of work has been outlined, and the work for the next quarter will involve accumulation of tensile and static fatigue data on available material. Additional study of oriented hydrides in Zircaloy-2 will continue as a very limited supplementary investigation.

V. CONCLUSIONS

At this time, no conclusions as to the susceptibility of Zr-2.5Nb or Zr-2.5Nb-0.5Cu to static fatigue failure can be advanced. In addition, no positive statement can be offered on the effect of oriented hydrides on sensitivity of Zircaloy-2 to this phenomenon. Work is proceeding toward these objectives.

VI. LOGBOOKS AND CONTRIBUTING PERSONNEL

The information contained in this report is recorded in ARF Logbooks Nos. C-13020 and C-13021.

Personnel contributing to this work are the following:

L. J. Adamski	-	Project Technician
F. C. Holtz	-	Group Leader
D. Weinstein	-	Project Engineer

Respectfully submitted,

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