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IMPROVED SERVICE-LIFE OF 15-kg
REDUCTION REACTOR VESSELS

by

L. L. Nelson

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

THE DOW CHEMICAL COMPANY
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U.S. ATOMIC ENERGY COMMISSION
CONTRACT AT(29-1)-1106

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ABSTRACT

Reduction vessels used for bomb reducing batches of UF_4 to uranium metal were failing after approximately twenty-five runs. The failures were caused by progressive cracking around the inside of the top of the vessel. These vessels were centrifugally cast from Type-310 stainless steel. It was determined that the cracking was caused by sensitization from the thermal cycling to which these vessels were subjected. Sensitization resistant Type-347 stainless steel is now used for the reactors and no cracking has been evident to fluorescent penetrant inspection.

SUMMARY

Sensitization resistant Type-347 stainless steel is now used for construction of the large reactor vessels. The vessels are fabricated by centrifugal casting.

Tests showed that wrought material offered a slight advantage over the cast material as far as properties were concerned. The difficulty of fabricating a wrought vessel with a suitable flange attachment more than outweighed the advantage of a wrought structure, however, so centrifugal casting was selected as the standard fabrication method.

The pitting-type defects are now repaired by welding-in and grinding, using Type-347 stainless steel rod exclusively. Most of the vessels currently in use have had service-lives on the order of two-hundred runs, and are still in satisfactory condition.

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INTRODUCTION

Reduction bombs used to prepare 15-kg uranium buttons from the green salt (UF_4) were a problem because of short service-life. The operating cycle to which these vessels are subjected is as follows: The green salt, calcium metal for the reduction, and iodine to assist in slag formation, are charged into a magnesia liner which is packed into the reduction vessel with magnesia sand (Figure 1). The charged bomb is then purged with argon, sealed, and lowered into an induction heating well. The induction heating raises the charge temperature to 275-300°C. Then the induction heating is shut off and the reduction reaction proceeds spontaneously. The temperature of the vessels at the single thermocouple location may then rise to 600°C and in rare cases rise to 1000°C. The temperature of the reactants is in excess of 1500°C but the reaction vessel is insulated from this maximum temperature by magnesia sand and a liner.

Defects which have appeared in these vessels are of three types:

Pitting - Pitting occurs mainly in or near the lid and occasionally farther down inside the vessel. This is probably caused by impingement of slag or molten uranium on the stainless steel. It occurs infrequently and can be repaired by filling-in with weld metal.

Bulging - The vessels in rare instances have bulged in the lower area. This condition is caused by reduction runs in which the vessel is subjected to unusually high pressure and/or temperature. The bulge occurs in the maximum temperature zone of the vessels, i.e. about one-third from the bottom. This appears to be essentially a creep phenomenon and is not a severe problem since it occurs infrequently.

Cracking - After approximately twelve firings the vessels developed invisible vertical cracks on the inside, from 1 to 2 inches below the rim. The cracks were detected by fluorescent penetrant inspection. The cracks became progressively worse until the vessels had to be discarded after approximately twenty-five runs. In some of the more severe cases, the cracks had almost completely penetrated the vessel wall (Figure 7).

Of the three types of defects, the cracking was the cause for most rejections and was also the factor which limited the average service-life of the vessel to twenty-five firings. This proved expensive, since the cost of one cast-and-machined vessel was over \$900.

EXPERIMENTAL

A reactor vessel exhibiting the pitting and cracking type defects was selected for study. This vessel exhibited above-average service-life, having been used for 31 reductions before visible cracking caused it to be removed from service. The vessel was sectioned, and the following tests were performed:

Tensile tests of specimens from four locations on the vessel, at both elevated and room temperature.

The Strauss test for sensitization. This consists simply of immersing a specimen in hot HNO_3 for long periods of time to see if the metal is susceptible to attack at the grain boundaries.

Photomicrographic examination of the metallurgical structure of the vessel.

TEST RESULTS

Tensile Tests:

These tensile tests (Table I) were performed on standard 0.250 dia. by 2 in. gauge-length specimens cut from a discarded reactor. Elevated-temperature tests were performed at 700°C , which is considered the normal maximum for the operating cycle of these reactors. Handbook values for mechanical properties of wrought Type-310 stainless steel are included in Table I for comparison.

Strauss Test for Sensitization:

A sample from a discarded reactor was immersed 4-hours in hot 1:1 HNO_3 . The acid was refluxed periodically to maintain concentration. Grain boundary etching occurred, indicating that the material had been sensitized. The sample selected exhibited a pitting defect, which did not

appear to contribute to sensitization, and is considered a separate problem (Figures 5 and 6).

Photomicrographic Examination:

Complete photomicrographic examination of material from discarded reactors was performed. Photomicrographs (Figures 2-9) show the long dendritic cast structure of the vessels, the nature of the cracking defects, and the nature of the pitting defects.

TABLE I
MECHANICAL PROPERTIES OF SAMPLES FROM
CENTRIFUGALLY CAST TYPE-310 SS REACTOR VESSEL

<u>Sample Location</u>	<u>Test Temp. °C</u>	<u>Ultimate Tensile Strength (psi)</u>	<u>Yield Strength (psi)</u>	<u>% Elongation</u>
20 in. from bottom	700	60,400	23,600	38
12 in. from bottom	700	59,900	20,400	40
7 in. from bottom	700	58,200	19,600	37
3 in. from bottom	700	58,600	24,200	35
20 in. from bottom	20	86,500	44,400	37
Wrought Type-310 SS	20	100,000	45,000	--
Wrought Type-310 SS	700	50,500	--	--

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APPENDIX

ILLUSTRATIONS

OUTSIDE DIMENSIONS OF REACTOR BODY:
9" DIA. X 23" HIGH

Figure 1. SCHEMATIC OF 15-kg URANIUM REDUCTION REACTOR (NOT TO SCALE)

Figure 2. TRANSVERSE CROSS SECTION OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL VESSEL. As etched, 16X (approximately one-half total wall section). This discloses long radial dendrites intersecting at irregular grain boundaries. Since the dendrites are long and radially oriented, the grain boundaries in many cases form rather direct paths through the vessel wall. It is along these boundaries that sensitization and cracking occurs, leading to failure of the reactor vessels (Figure 3). The photographs shown in this report, unless otherwise stated, show about one-half of the wall thickness of the vessel, with what was formerly the vessel ID at the bottom of the picture

Figure 3. TRANSVERSE CROSS SECTION OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL VESSEL. As polished, 20X (vessel ID at bottom). This discloses the cracks, due to corrosion and stress in sensitized grain boundaries, just as they begin to occur. Further subjection of this particular vessel to high pressure and hot halides would have caused these cracks to propagate rapidly. The corrosion of the sensitized boundaries creates a weakened area which begins to crack under stress when the vessel is subjected to operating pressure

Figure 4. TRANSVERSE CROSS SECTION OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL WHICH FAILED. As etched, 20X, wall section. This shows how cracks can propagate along grain boundaries once sensitization, followed by stress and corrosion, causes initial stress-riser cracks to form. The different dendrite orientations on each side of the crack are readily apparent, illustrating how the crack coincides with what was formerly a grain boundary

Figure 5. VIEW OF SAMPLE FROM INSIDE SURFACE OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL. Specimen as removed from 1:1 HNO_3 Strauss test bath, 12X. This surface, formerly the inside surface of a reactor vessel, appeared crack-free before the Strauss test was applied. The large dark pit at upper left was the only defect apparent, and was the reason for selection of this particular sample. The test bath has preferentially attacked the sensitized grain boundaries, resulting in deep crevices at these locations. The crevices appear as cracks in this picture.

Figure 6. TRANSVERSE CROSS SECTION OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL. As etched, 16X (vessel ID at bottom). This shows a section with pitting-type defects. Some embryo cracking is also evident in this specimen. It appears that the pitting does not affect the cracking tendencies except for accelerated corrosion of sensitized grain boundaries near the point of pitting. (Note heavily corroded boundary just to left of large pit.) Such pits obviously do not effect the strength of the vessel wall as much as the cracks, since they reduce the effective wall thickness by only a few percent, and leave relatively sound adjacent metal, as this photograph shows. The cracking defect, however, weakens the vessel wall throughout its thickness, and could lead to catastrophic failure (Figure 7).

Figure 7. TRANSVERSE SECTION OF CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL. As polished, 12X (vessel ID at bottom). This shows a severely cracked section of the vessel. The dark globular defect to left of center appears to be a casting defect. This probably acted as a stress riser, and may also have provided a channel to allow corrosive materials to permeate the vessel wall more deeply than usual. This set of defects was interconnected before the specimen was ground-down to reveal a plane where the cracks had achieved maximum penetration. The irregular hole at lower right developed when small grains, their boundaries attached by sensitization, were removed completely from the matrix. The beginning of a similar condition may be observed in Figure 6.

Figure 8. WROUGHT AND ANNEALED TYPE-310 STAINLESS STEEL FROM ROLL-FORMED REACTOR VESSEL. As etched, 100X. This photomicrograph illustrates the superior structure of wrought material. Even at 100X size, as compared to 12-20X size for the pictures of cast structures, the grain size appears finer here. The wrought material is thus more uniform, and does not possess macroscopic grain boundaries as is the case with cast material.

Figure 9A. PHOTOGRAPH OF BROKEN END OF TENSILE SPECIMEN FROM CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL, 8.5X. Test conducted at room temperature. This shows how the material deformed with the long dendritic grains sliding past one another. Rupture also occurs between the grains, as shown in Figures 9B and 9C.

Figure 9B. SAME SAMPLE AS IN FIGURE 9A, 8.5X. This shows the broken end of the tensile specimen. Several dendrites which parted away from those adjacent, during fracture, are discernible. Note that these dendrites extend clear across the diameter of this specimen, which was 0.250 in. before the test. The unclear focus is due to the great variation in relief across the surface viewed.

Figure 9C. PHOTOGRAPH OF BROKEN END OF TENSILE SPECIMEN FROM CENTRIFUGALLY CAST TYPE-310 STAINLESS STEEL REACTOR VESSEL, 8.5X. Test conducted at 700°C. This shows how the interdendritic mode of fracture is retained at 700°C. However, there is some fracture through the dendrites as evidenced on the right edge of this specimen. It is possible that at elevated temperature the tendency of the material to fracture between grains is reduced (Table I).