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INTERACTIONS OF ZIRCALOY CLADDING WITH GALLIUM: 1998 MIDYEAR STATUS

D. F. Wilson
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ORNL/TM-13625

Metals and Ceramics Division

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Date Published: June 1998

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Prepared for the
U.S. Department of Energy
Office of Fissile Materials Disposition
GA 01 02 01 4

Prepared by
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managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-96OR22464

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ACRONYMS

EBW	electron beam welding
IC	intermetallic compound
ICF	intermetallic compound formation
LANL	Los Alamos National Laboratory
LBW	laser beam welding
LME	liquid metal embrittlement
LWR	light-water reactor
MOX	mixed oxide
ORNL	Oak Ridge National Laboratory
WG	weapons grade
wt	weight

ABSTRACT

A program has been implemented to evaluate the effect of gallium in mixed-oxide (MOX) fuel derived from weapons-grade (WG) plutonium on Zircaloy cladding performance. The objective is to demonstrate that low levels of gallium will not compromise the performance of the MOX fuel system in a light-water reactor. The graded, four-phase experimental program was designed to evaluate the performance of prototypic Zircaloy cladding materials against (1) liquid gallium (Phase I), (2) various concentrations of Ga_2O_3 (Phase II), (3) centrally heated surrogate fuel pellets with expected levels of gallium (Phase III), and (4) centrally heated prototypic MOX fuel pellets (Phase IV). This status report describes the results of a series of tests for Phases I and II.

Three types of tests are being performed: (1) corrosion, (2) liquid metal embrittlement, and (3) corrosion-mechanical. These tests will determine corrosion mechanisms, thresholds for temperature and concentration of gallium that may delineate behavioral regimes, and changes in the mechanical properties of Zircaloy.

Initial results have generally been favorable for the use of WG-MOX fuel. The MOX fuel cladding, Zircaloy, does react with gallium to form intermetallic compounds at ≥300°C; however, this reaction is limited by the mass of gallium and is therefore not expected to be significant with a low level (parts per million) of gallium in the MOX fuel. Although continued migration of gallium into the initially formed intermetallic compound can result in large stresses that may lead to distortion, this was shown to be extremely unlikely because of the low mass of gallium or gallium oxide present and expected clad temperatures below 400°C. Furthermore, no evidence for grain boundary penetration by gallium has been observed.

1. INTRODUCTION

The U.S. Department of Energy has established a dual-track approach to the disposition of plutonium arising from the dismantling of nuclear weapons. Both immobilization and reactor-based mixed-oxide (MOX) fuel technologies are being evaluated. The MOX fuel option requires assessment of the potential impact of gallium (on the order of 1 to 10 ppm), not present in conventional MOX fuel, on cladding material performance. Two previous reports[1, 2] identified several compatibility issues relating to the presence of gallium in MOX fuel and its possible reaction with fuel cladding.

Gallium initially present in weapons-grade (WG) plutonium is reduced during processing to produce MOX fuel. After blending the plutonium with uranium, only 1 to 10 ppm gallium is expected in the sintered MOX fuel. Gallium present as gallium oxide (Ga_2O_3) could be evolved as the suboxide (Ga_2O). Migration of the evolved Ga_2O and diffusion of gallium in the MOX matrix along thermal gradients could lead to locally higher concentrations of Ga_2O_3. Thus, while an extremely low concentration of gallium in MOX fuel almost ensures a lack of significant interaction of gallium with Zircaloy fuel cladding, there remains a small probability that corrosion effects will not be negligible.

A number of reaction routes are possible in this clad-WG-MOX fuel system, including:

$$\left.\begin{array}{c} Ga_X O \cdot PuO_2 \\ or \\ Ga_2O \\ or \\ Ga_2O_3 \end{array}\right\} + Zircaloy \rightarrow Ga \rightarrow \left\{\begin{array}{c} Surface\ alloy \\ or \\ Grain\ boundary\ penetration \\ or \\ Liquid\ metal\ embrittlement \end{array}\right. ,$$

and

$$Ga_2O_3 + ZrO_2 \rightarrow \left\{\begin{array}{c} No\ reaction \\ or \\ Mixed\ oxide + crack + Ga \rightarrow \end{array}\right. \left\{\begin{array}{c} Surface\ alloy \\ or \\ Grain\ boundary\ penetration \\ or \\ Liquid\ metal\ embrittlement \end{array}\right. .$$

General corrosion in the form of surface alloying from formation of intermetallic compounds between Zircaloy and gallium should be mass limited and, therefore, superficial because of the expected low ratio of gallium to the surface area or volume of the Zircaloy cladding. Although the expected concentration of gallium is low and there is very limited solubility of gallium in zirconium, especially at temperatures below 700 °C,[3] grain boundary penetration and liquid metal embrittlement (LME) are possible forms of localized corrosion that are also being considered.

One fuel system damage mechanism, pellet clad interaction, has led to some failure of the Zircaloy cladding in light-water reactors (LWRs). This has been attributed to stresses in the cladding and one or more aggressive fission products. Stress corrosion cracking by iodine[4, 5] and LME by cadmium[6, 7] have been reported, and it is known that Zircaloy can be embrittled by some low-melting metals, (e.g., mercury).[8] LME is a form of environmentally induced embrittlement that can induce cracking or loss of ductility. LME requires wetting and a tensile stress, but it does not require corrosion penetration. Experimentally, it has been demonstrated that gallium can cause embrittlement of some alloys (e.g., aluminum) at low temperatures,[9, 10] but experiments relative to LME of zirconium by gallium have been limited and inconclusive.[11]

This report describes a series of gallium-cladding compatibility tests aimed at establishing confidence that low levels of residual gallium in WG-MOX fuel do not affect its long-term compatibility with Zircaloy. In addition, to address damage mechanisms it is important to understand the potential cladding interactions and the available safety margins with respect to gallium concentration.

2. OBJECTIVE

The overall objective of this task is to demonstrate that small concentrations of gallium in MOX fuel will not compromise the performance of the fuel system in an LWR. Although possible mechanisms for gallium interaction with Zircaloy have been identified,[1, 2] no significant data exist that demonstrate whether such reactions actually occur under LWR conditions and whether these reactions have any significant effects on cladding performance. In the present program, a graded, experimental approach is being pursued to determine whether the low levels of gallium will result in fuel system damage. This approach is divided into four phases. In Phase I, laboratory tests were conducted on three prototypic Zircaloy cladding materials in liquid gallium. Three types of tests were used: (1) corrosion, (2) LME, and (3) corrosion-mechanical. LME tests consist of slow strain rate, constant extension rate tensile tests in gallium metal at low temperature (30 and 100°C). Corrosion-mechanical tests consist of first exposing the Zircaloy with gallium metal and then conducting a tensile test to evaluate corrosion product effects. Although these Phase I tests conditions (much higher relative amount of gallium compared to that in a fuel rodlet) are not representative of WG-MOX, they provide limiting boundary conditions for mechanistic studies. The test matrices for the Phase I tests are shown in the Appendix.

In Phase II, two of the previously mentioned types of tests, corrosion and corrosion-mechanical, were conducted with the three cladding materials in contact with Ga_2O_3. In each type of test, four concentrations of Ga_2O_3 in Ce_2O_3 (surrogate for plutonium dioxide) were evaluated: 100, 1, 0.2, and 0.1 weight (wt) %. Although these levels of Ga_2O_3 are all considerably higher than those possible in a sintered MOX pellet,[12] they can be used to determine the types of reactions that could occur, and, like Phase I tests, they represent a nonconservative evaluation of Ga_2O_3 effects in this screening test phase. The test matrices for Phase II tests are shown in the Appendix.

Phase III tests will expose cladding material to centrally heated surrogate fuel pellets that are being manufactured by Los Alamos National Laboratory (LANL). These sintered, annular pellets, U/Ce/Ga/O, will contain less than 0.001 wt % gallium and will have a density of more than 94%. The Texas A&M University Nuclear Science Center designed a nonpressurized test system for the centrally heated pellet tests and will perform the tests. To simulate the thermal gradients across operating reactor pellets and to test for thermally driven gallium diffusion, a small diameter (~0.18-in. outside diameter) electric heater will be inserted into the center of the surrogate pellets. The heater will operate at a temperature of about 1000°C with a linear power

of 15.7 kW/m. The experiment is configured to produce a nominal cladding-pellet interface temperature of 400°C. Six 4-in. "rodlets" will be centrally heated and two will be withdrawn periodically for evaluation (the longest time at temperature will be 5000 h).

Work related to the planned Phase IV tests has recently been terminated. If conducted, Phase IV tests would be similar to Phase III tests, but prototypic MOX fuel pellets would be used. These tests were to be performed at Oak Ridge National Laboratory (ORNL) with MOX pellets produced by LANL.

3. EXPERIMENTAL

Prototypical LWR cladding materials evaluated included Zircaloy-2, Zircaloy-4, and Zirlo. Compositional ranges for these alloys are presented in Table 1.

Table 1. Composition of alloys based on zirconium (weight percent except where noted)

Alloy	Sn	Fe	Cr	Ni	Nb	O (ppm)
Zircaloy-2*	1.2–1.7	0.07– 0.2	0.05–0.15	0.03–0.08		1000–1400
Zircaloy-4*	1.2–1.7	0.18–0.24	0.07–0.13			1000–1400
Zirlo†	0.96–0.98	0.094–0.105	79–83 ppm		1.02–1.14	900–1200

*R. W. Cahn, P. Haasen, and E. J. Kramer, "Materials, Science, and Technology", Vol. 10b, p. 11, *Nuclear Materials*, Part 2, 1994.
†G. P. Sabol et al., "In-Reactor Corrosion Performance of Zirlo and Zircaloy-4," *Zirconium in the Nuclear Industry: Tenth International Symposium,* pp. 724-744 in *ASTM STP 1245,* ed. A. M. Garde and E. R. Bradley, American Society for Testing and Materials, Philadelphia, 1994.

Zircaloy tubing was machined into two different lengths, one for corrosion tests and another for LME or corrosion-mechanical tests, as shown in Fig. 1. End caps with and without threaded ends were machined from Zircaloy bar stock for tensile and corrosion tests, respectively. First, one end cap was welded to the specimen tube, the required quantity of reactant was then added, and the other end cap was welded in place. Because of the reactive nature of the zirconium alloys with oxygen and nitrogen, all welding was performed in either high vacuum using the electron beam welding (EBW) process or in a high-purity, helium-purged welding chamber using the laser beam welding (LBW) process. The completed test specimens for corrosion tests were encapsulated in an evacuated quartz capsule for atmospheric protection during high-temperature exposure (Fig. 1). For mechanical testing above 100°C, a chamber to provide a protective atmosphere around the test specimens was designed and fabricated. This protective chamber precludes the reaction of zirconium alloys with the ambient oxygen and nitrogen during testing.

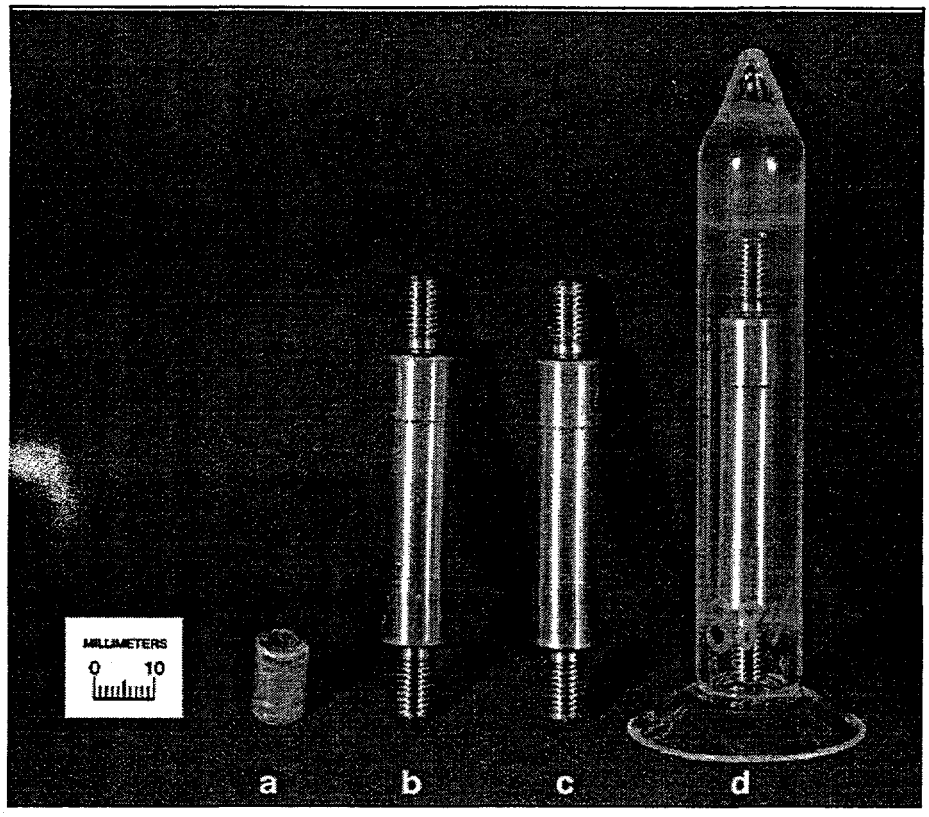


Fig. 1. Test specimens. a, corrosion; b, LME; c, corrosion-mechanical; d, encapsualted in quartz.

In addition to preliminary tests that were performed at 400 and 600°C, corrosion tests in liquid gallium were conducted at three temperatures (30, 100, and 500°C) for two exposure times (200 and 700 h); those containing Ga_2O_3 or surrogate gallium-containing oxide were also tested at three temperatures (300, 500, and 700°C) for two exposure times (6 weeks and 12 weeks). LME tests were conducted in liquid gallium at 30 and 100°C. Corrosion-mechanical tests in liquid gallium were conducted by first exposing the Zircaloy for 30 h at 300°C and then conducting a room-temperature tensile test. With Ga_2O_3, tensile tests were also conducted at room temperature and at the corrosion test temperature after exposure for 6 weeks at 300 and 500°C. The temperature range brackets the normal, operating clad temperature of 374°C.

Posttest analyses of cross sections of the clad tubes included metallography, fractography, and chemical microprobe. In the case of the corrosion tests, evidence of wall thinning, grain boundary penetration, transgranular attack, and intermetallic compound formation (ICF), when present, were documented. For the mechanical tests (LME or corrosion-mechanical), the tensile strength and ductility of specimens tested in the presence of gallium or after exposure to gallium were compared with those tested in the absence of gallium.

4. RESULTS

4.1 PHASE I

4.1.1 Corrosion Tests

Corrosion tests in liquid gallium were conducted at 30, 100, and 500°C. After exposure for 700 h at 30 and 100°C, none of the Zircaloy materials showed evidence of significant interaction with liquid gallium as indicated by the typical results shown in Fig. 2. At 500°C, all of the gallium reacted with Zircaloy to form intermetallic compounds (ICs) of the type Zr_xGa_y after 200 or 700 h. Dimensional distortion of Zircaloy capsules was observed as a result of exposure at 500°C.

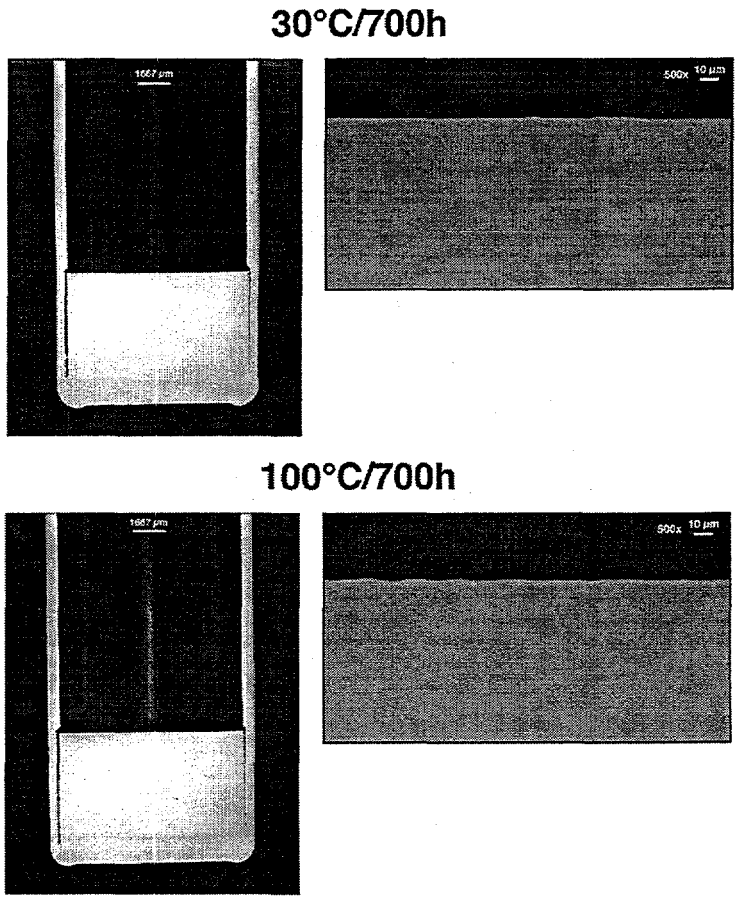
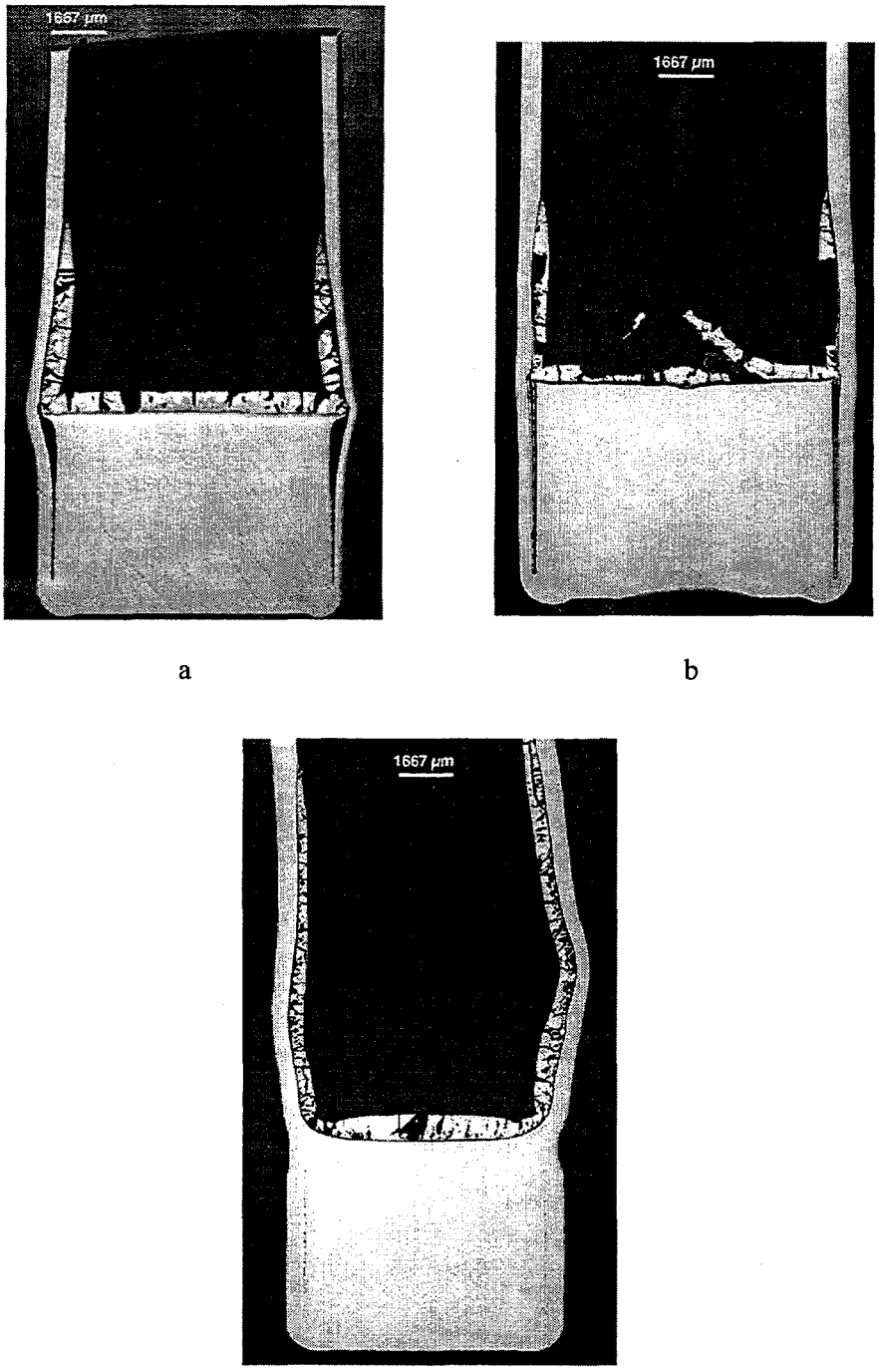


Fig. 2. No attack of Zircaloy after 700 h at 30 or 100°C.

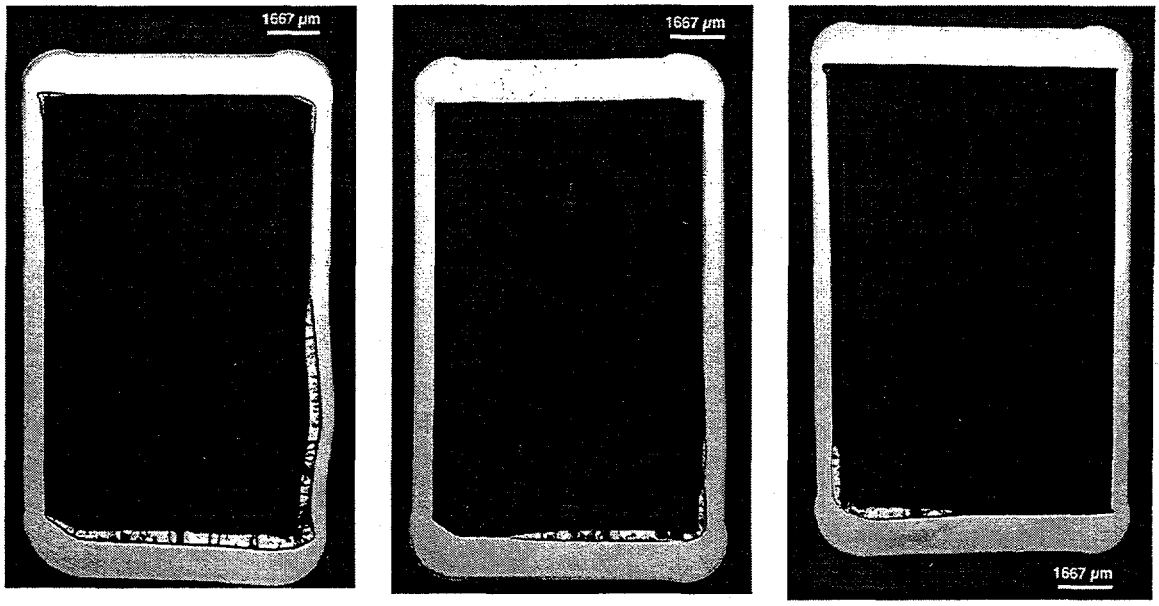
Subsequent investigation revealed that the amount of distortion was not related to the end-cap geometry or the internal gas pressure [as determined by whether EBW or LBW (Fig. 3) was used]. However, distortion was a function of the amount of gallium initially in the capsule



a b c

Fig. 3. Cross sections of Zircaloy tubes after corrosion testing with excess gallium (Ga) at 500°C for 200 h showing dimensional distortion. Distortion is independent of welding technique. a = LBW, 0.5 g Ga; b = EBW, 0.5 g Ga; c = no crevice, 0.6 g Ga.

even though there was considerable empty space remaining within the capsule at the end of the test (Fig. 4). One of the Zircaloy materials showed little or no distortion even at the highest amounts of gallium, despite the fact that a Zr_xGa_y type of IC formed, as was observed with the other Zircaloy materials. Comparisons of the corrosion product at high magnifications showed some differences in their appearance. Where no distortion occurred, the corrosion product was more porous than when distortion did occur (Fig. 5).



a b c

Fig. 4. Corrosion testing with smaller amounts of gallium (Ga) at 500°C for 200 h. a = 0.2 g Ga, shows less dimensional distortion; b = 0.09 g Ga, shows minor dimensional distortion; c = 0.05 g Ga, shows no dimensional distortion.

Further testing at temperatures below 500°C showed that, in the susceptible materials, dimensional distortion also occurred at 400 and 350°C but was not found after 200 h at 300°C. After 200 h at 300°C, there was a large amount of unreacted gallium in the capsule at the end of the test. The rate of reaction is very slow at ≤300°C, evidenced by the decreasing amount of corrosion product formed at ≤200 h (Fig. 6).

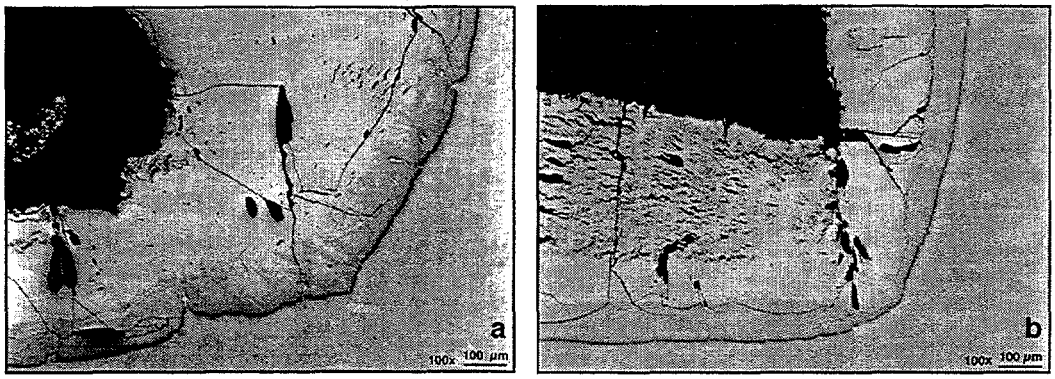


Fig. 5. Porosity in Zr_xGa_y formed at 500 °C in Zircaloy material. a, material that distorted; b, material that did not distort.

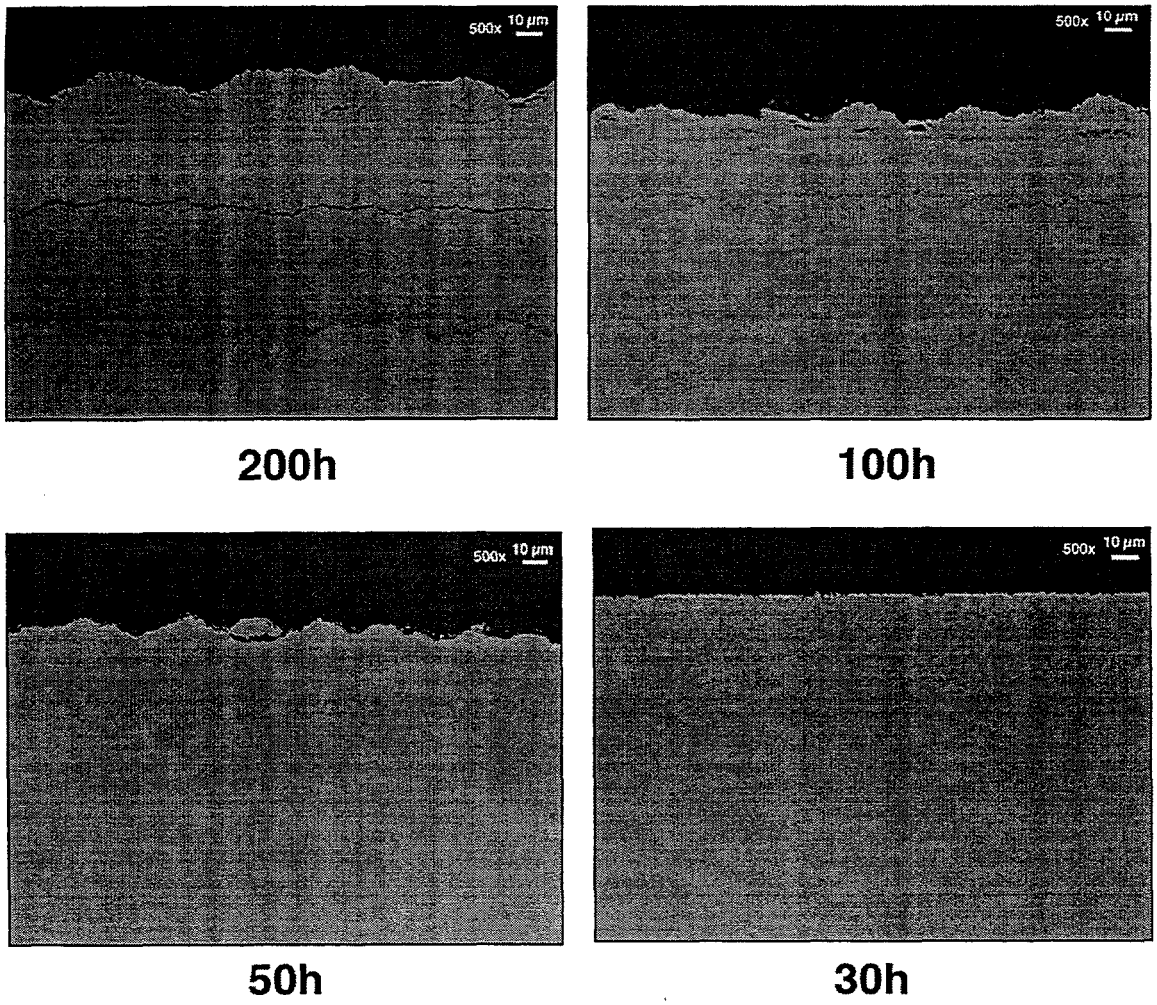


Fig. 6. At 300 °C, ICF decreases with decreasing time of exposure.

As indicated in Fig. 4, dimensional distortion at 500°C was eliminated when smaller amounts of gallium were used in the capsule. In subsequent tests at 500°C with even smaller quantities of gallium (0.025 to 0.00038 g), ICF was localized to specific regions where gallium chemically wet the capsule wall but the depth of interaction was quite shallow (Fig. 7).

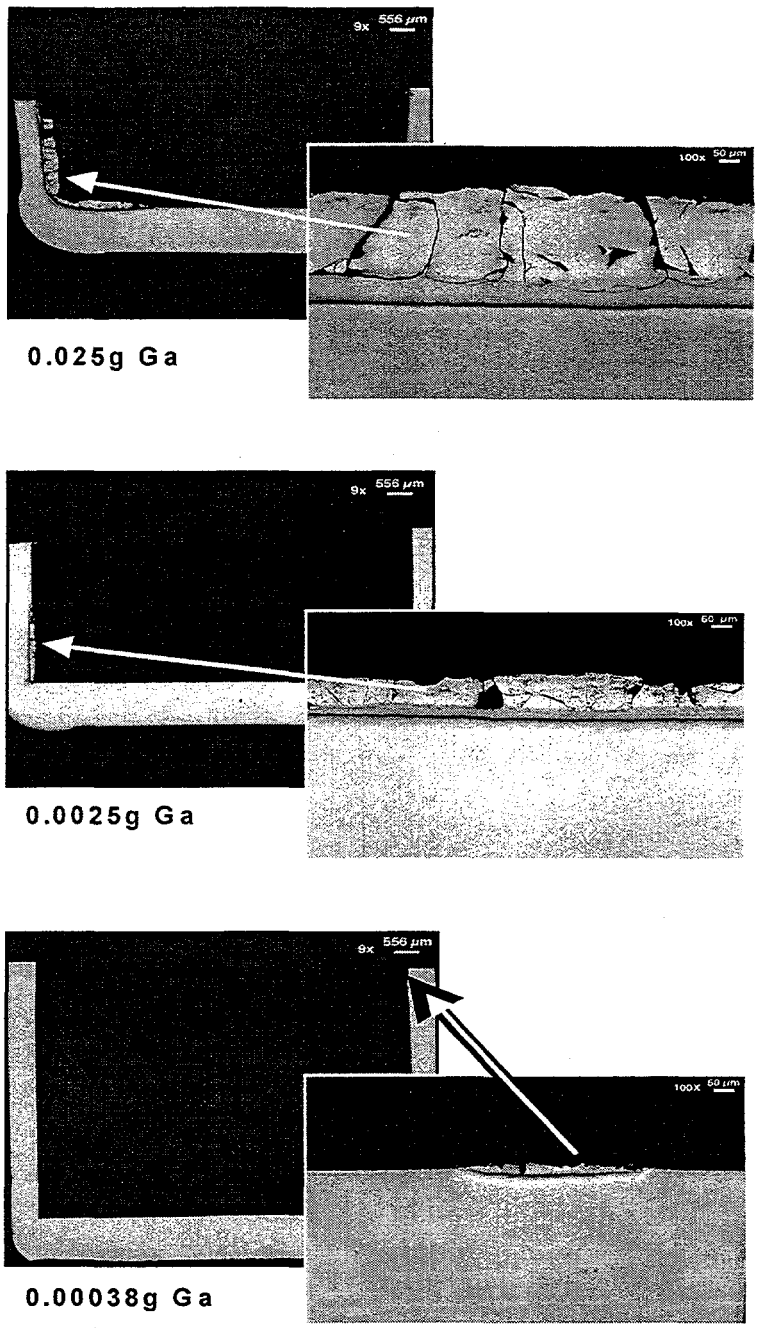


Fig. 7. ICF continues to decrease as the mass of gallium is decreased.

4.1.2 LME Tests

Slow strain rate (3.33×10^{-6}/sec), constant extension rate tensile tests were conducted in gallium at 30 and 100°C, and the results (average of three specimens per condition) are shown in Figs. 8 and 9. No differences in the tensile properties were found between the tests in gallium compared with those at the same temperature without gallium. Although there was a small decrease in strength from 100°C to 30°C, this same change occurred without gallium as well. Furthermore, the specimens all failed in a ductile manner, as shown in Fig. 10.

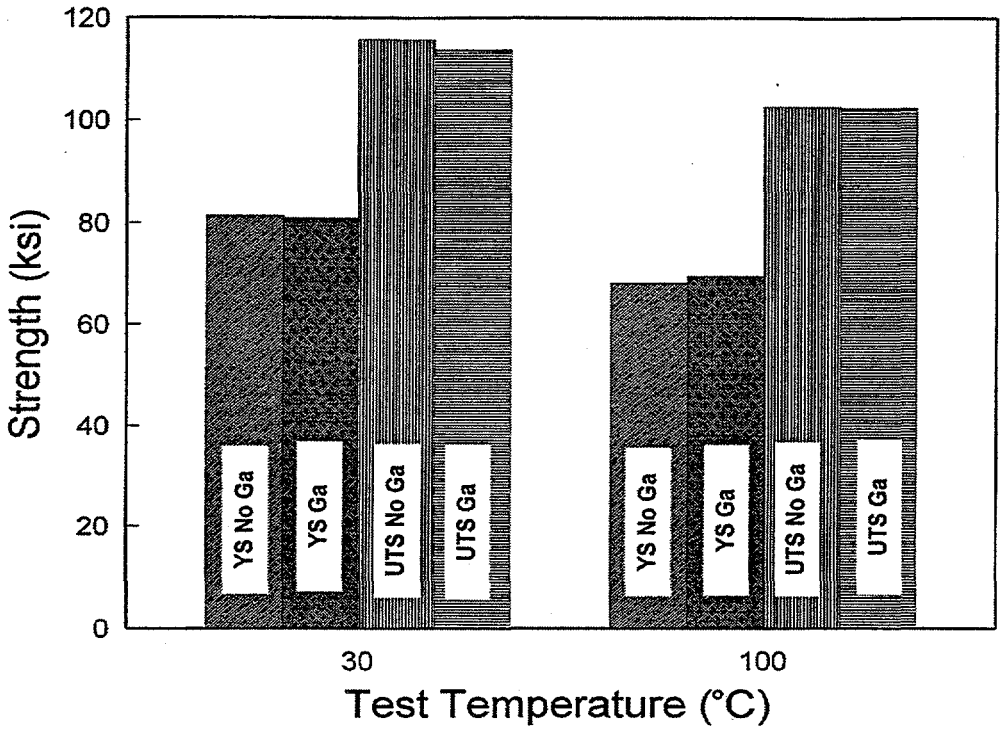


Fig. 8. No change in strength at 30 and 100°C. YS, yield strength; UTS, ultimate tensile strength.

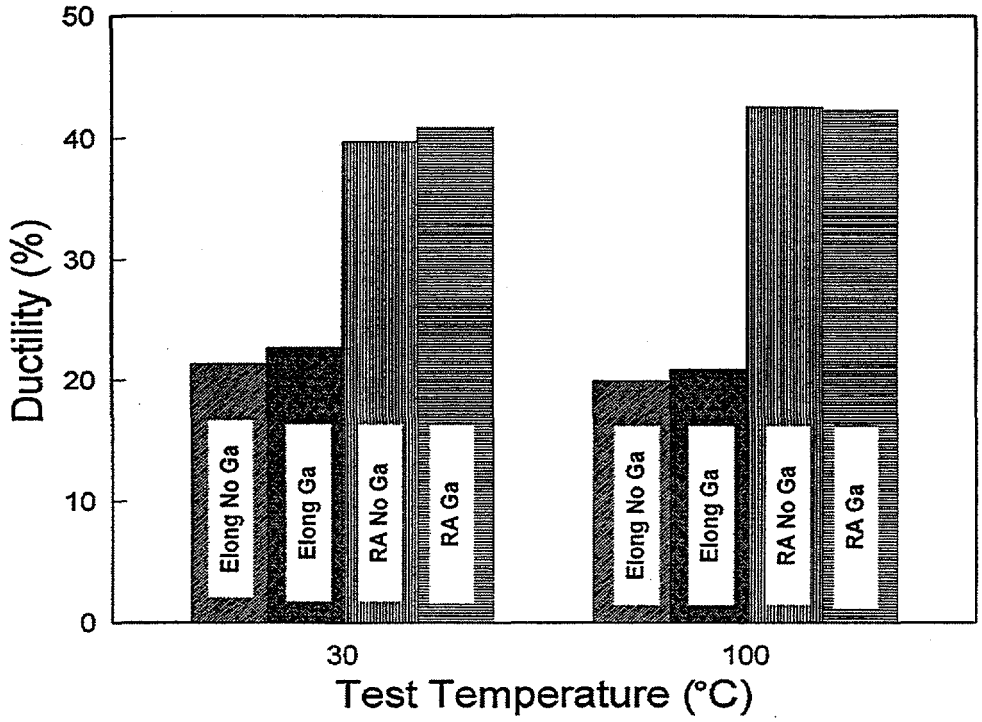


Fig. 9. No significant difference in ductility at 30 and 100°C. Elong, elongation; RA, reduction in cross sectional area.

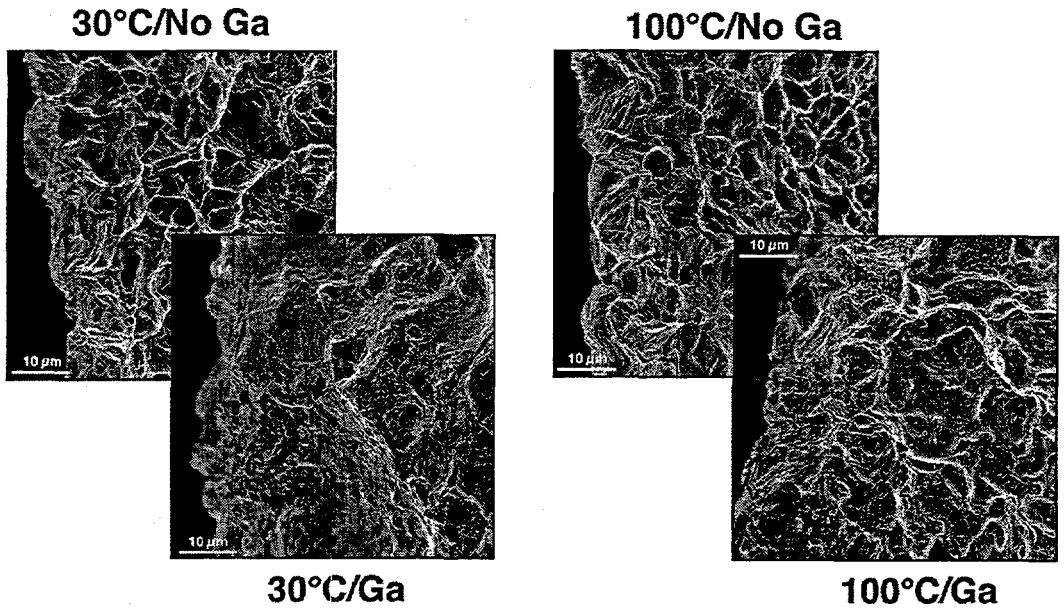


Fig. 10. Ductile failure mode at 30 and 100°C with and without gallium.

4.1.3 Corrosion-Mechanical Tests

The purpose of these tests was to obtain some measure of the effect of ICF on the mechanical properties of Zircaloy. The Zr_xGa_y IC is quite hard and brittle; therefore, significant ICF would be expected to markedly affect the mechanical properties of the Zircaloy. Furthermore, as reported previously, distortion of Zircaloy at 500°C became a complicating factor. Based on prior data from the corrosion tests, exposure of the Zircaloy to gallium for 30 h at 300°C was ultimately selected as the condition for the corrosion portion of the corrosion-mechanical tests. Under these conditions, most of the gallium did not react with the Zircaloy; however, a thin, intermittent area of ICF was present along the gage length of the Zircaloy capsule specimen. Tensile properties were then measured at room temperature and at 300°C. The results (average of three specimens per material, per condition) are shown in Figs. 11 and 12 and indicate very little, if any, effect of the formation of small amounts of ICF on the mechanical properties of Zircaloy. In addition, examination of the fracture surfaces did not indicate any significant difference in the mode of failure (Fig. 13).

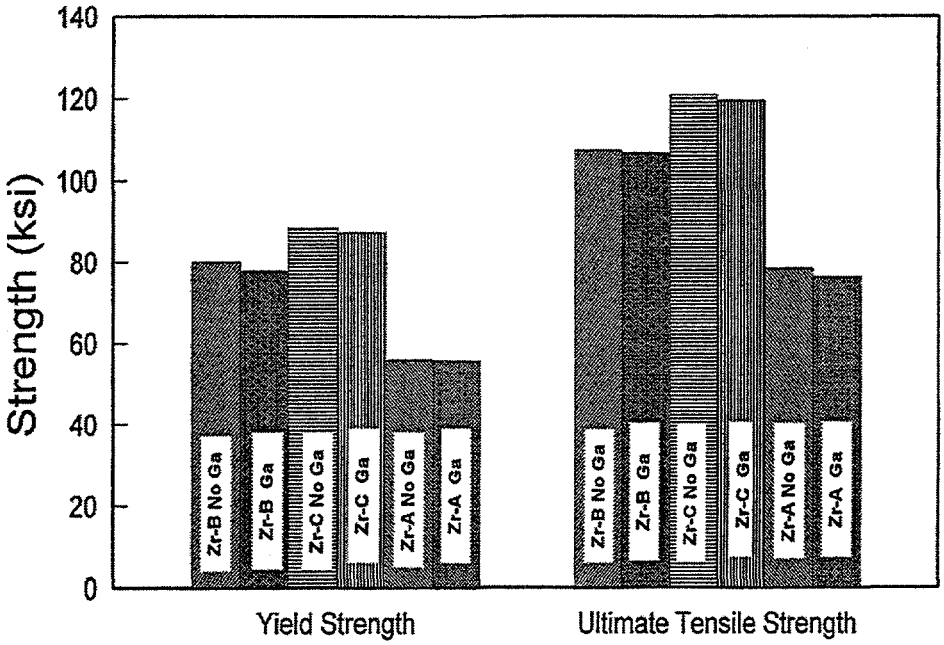


Fig. 11. No change in strength after reaction with gallium for 30 h at 300°C.

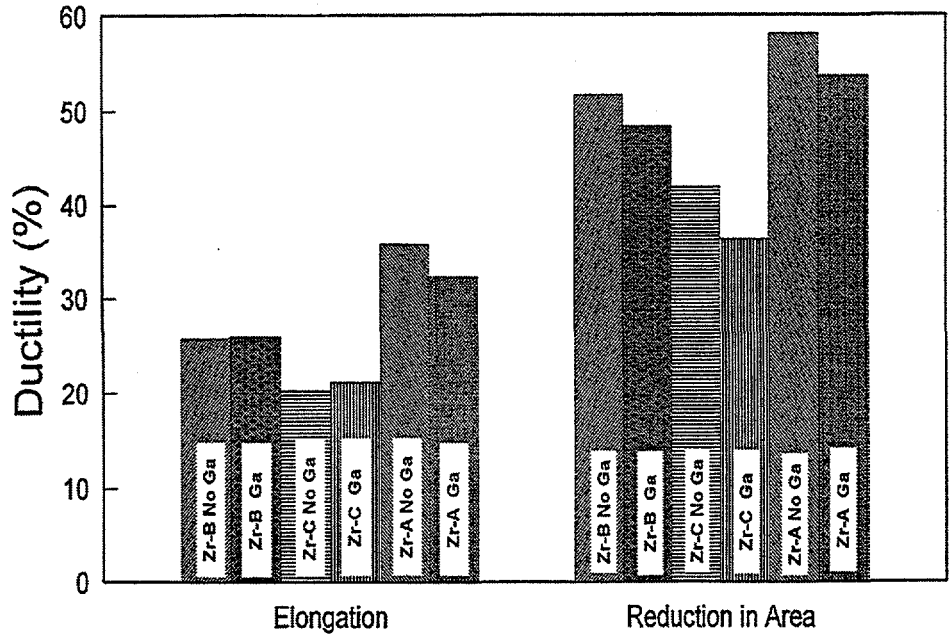


Fig. 12. No significant change in ductility after exposure to gallium for 30 h at 300°C.

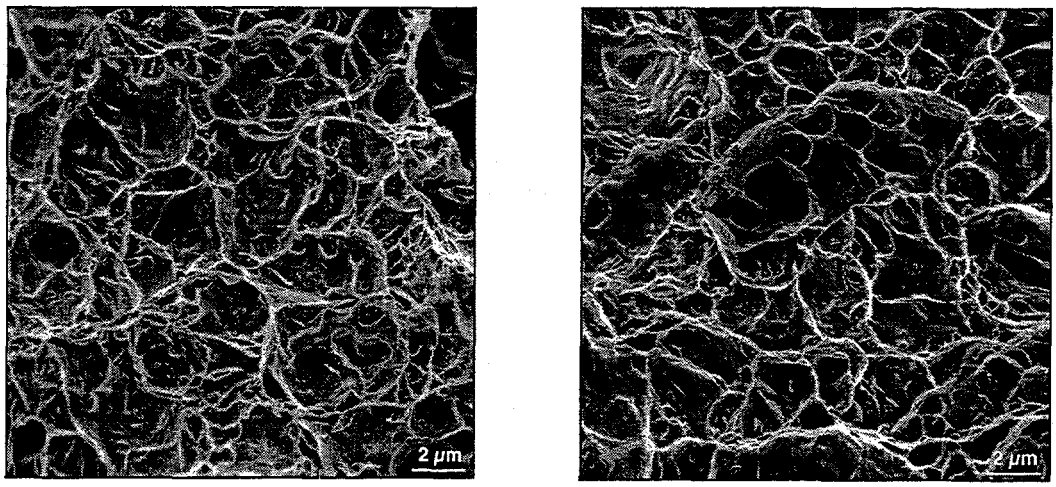


Fig. 13. Ductile fracture observed after 30 h at 300°C without and with gallium. Specimen mechanically tested at room temperature.

4.2 PHASE II

4.2.1 Corrosion Tests

Corrosion tests in Ga_2O_3 powder were conducted at 300, 500, and 700°C for 6 and 12 weeks. Ce_2O_3 powder was mechanically blended with Ga_2O_3 powder to produce mixtures that contained 100, 1, 0.2, and 0.1 wt % Ga_2O_3 to assess the effect of concentration of gallium present during the exposures.

Before initiating these tests, several surrogate oxides (for PuO_2) were tested at 500 or 700°C with Zircaloy: ZrO_2, CaO, and Ce_2O_3, as well as Ga_2O_3. In all cases, as would also occur with PuO_2,[1] an oxide layer was found on the inner surface of the Zircaloy that was qualitatively identified as ZrO_2 (Fig. 14).

In the matrix tests, there were significant differences among results at the three temperatures. At 300°C, no significant reaction was noted except for very slight surface roughening, even after the 12-week exposures (Fig. 15). At 500°C, the oxide layer mentioned previously was observed in all of the tests (Fig. 16). The thickness of the oxide layer was independent of Ga_2O_3 concentration and did not appreciably increase with time between 6 and 12 weeks. A distinguishable zone can be noted below the oxide layer. Although this zone was larger for the tests with 100% Ga_2O_3, there was no appreciable difference among 1, 0.2, and 0.1% Ga_2O_3 exposures.

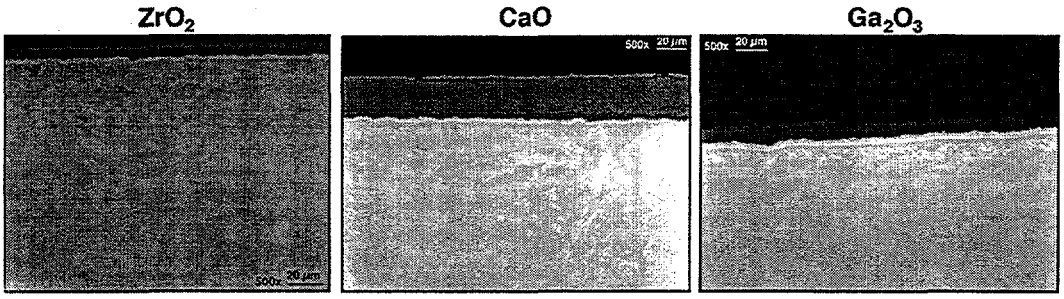


Fig. 14. Zircaloy reacts with oxides at 500 °C to form zirconium oxide. Zirconium oxide formation is not limited to reaction of Zircaloy with Ga_2O_3.

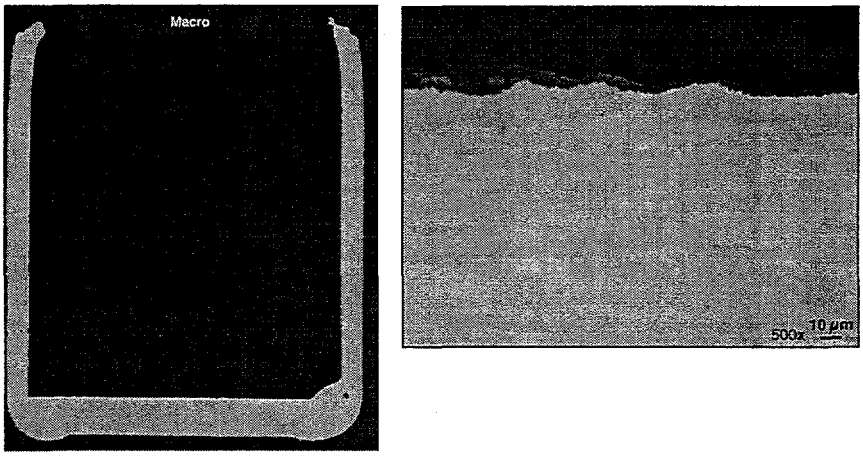


Fig. 15. No significant reaction of Zircaloy with Ga_2O_3 after 12 weeks at 300 °C.

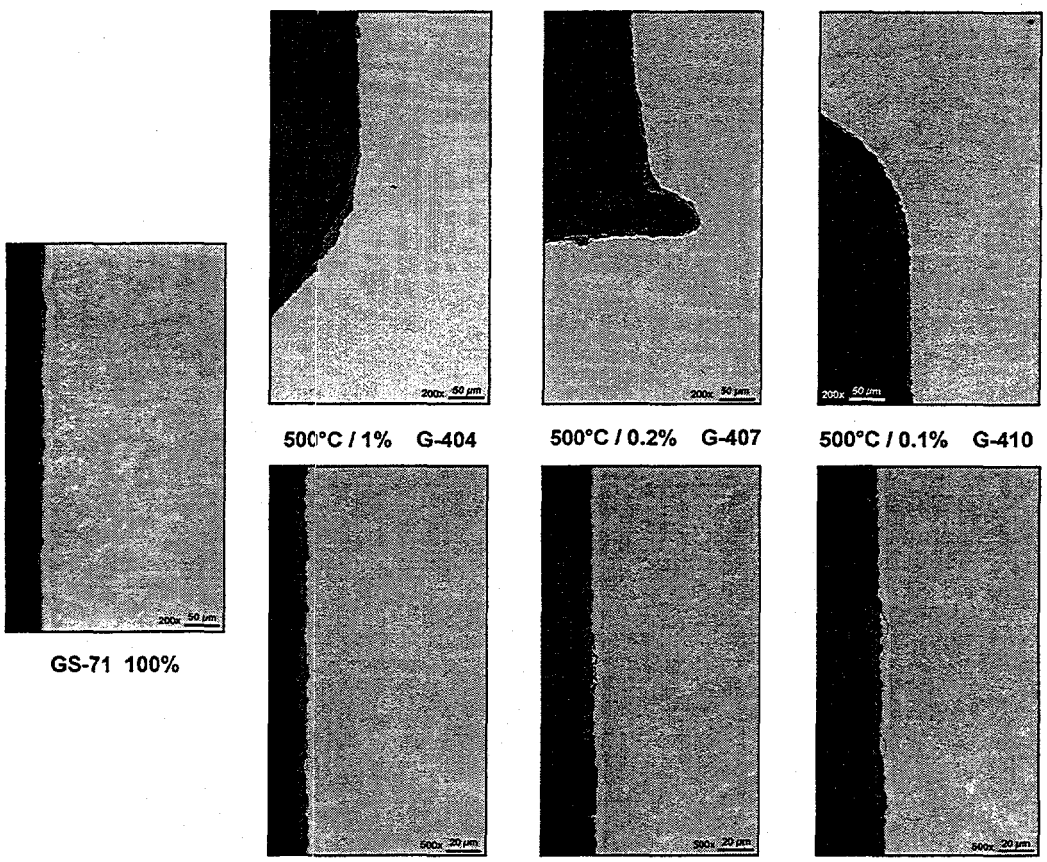


Fig. 16. Oxide layer present at 500°C for all ratios of Ga_2O_3 to cerium oxide.

At 700°C, there was formation of an oxide layer on the inner surface of the Zircaloy and an identifiable gallium-rich zone below the oxide layer. The thickness of both the oxide and gallium-rich zone increased with time, but the gallium-rich zone was visible only with 100% Ga_2O_3 (Fig. 17). Another feature not seen at 500°C is cracking of the Zircaloy. Generally, testing at 700°C resulted in a somewhat uniform distortion of the Zircaloy tubing, and the cracks may be associated with an oxygen-enriched zone.

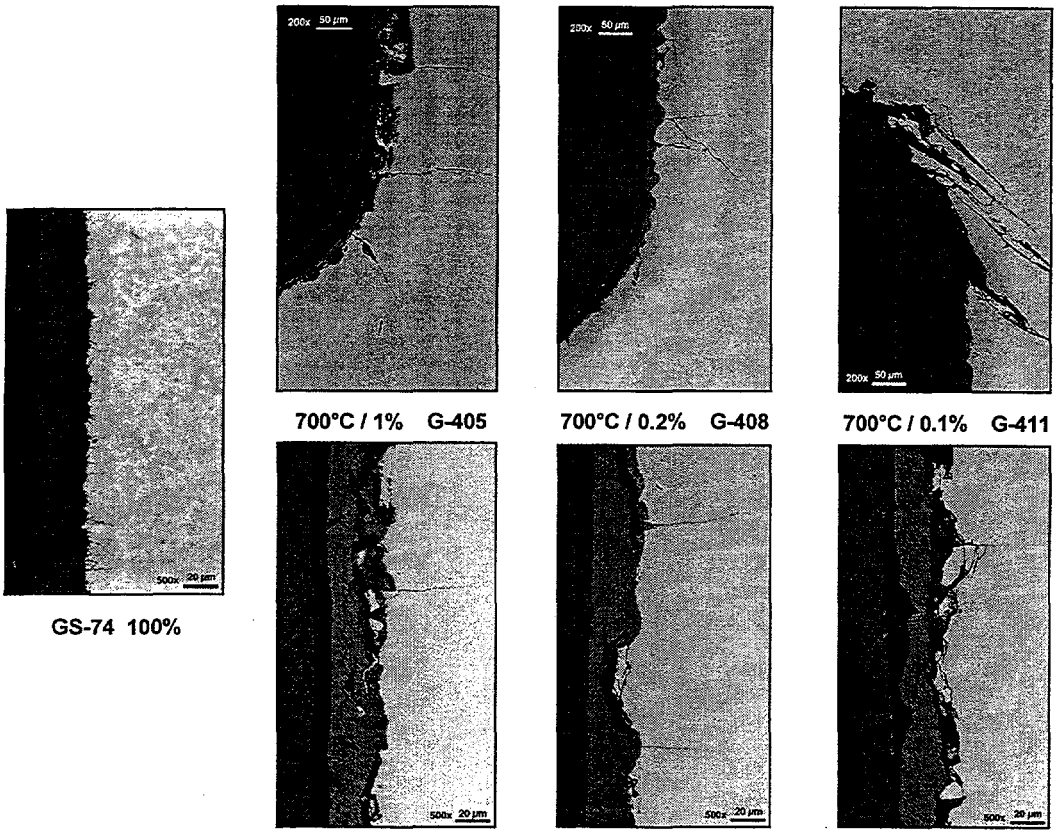


Fig. 17. Zr_xGa_y identified below the zirconium oxide layer after exposure to 100% Ga_2O_3 at 700°C.

4.2.2 Corrosion-Mechanical Tests

This portion of the testing program is ongoing, and results thus far are available only for corrosion testing at 300 and 500°C with one of the Zircaloy materials. As shown in Figs. 18 and 19, there was no change in the room temperature tensile properties (three specimens per condition) of Zircaloy after exposure at 300°C, as would have been predicted based on the corrosion test results reported previously. At 500°C, a decrease in both room temperature yield and ultimate tensile strength was measured for all Ga_2O_3 concentrations, with a corresponding increase in ductility as measured by elongation and reduction in area. Control tests are in progress to determine whether these changes are a result of heat treatment at 500°C instead of the test environment. Mechanical testing after exposure at 700°C was not possible because of distortion during the 700°C exposure.

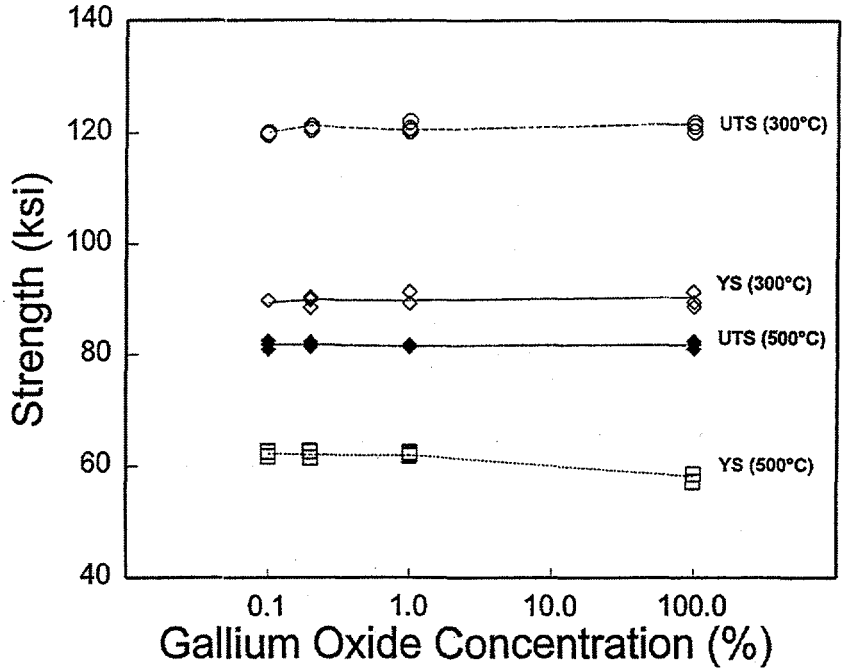


Fig. 18. No significant change in strength after exposure to Ga_2O_3 in cerium oxide (6 weeks at 300 and 500°C). YS, yield strength; UTS, ultimate tensile strength.

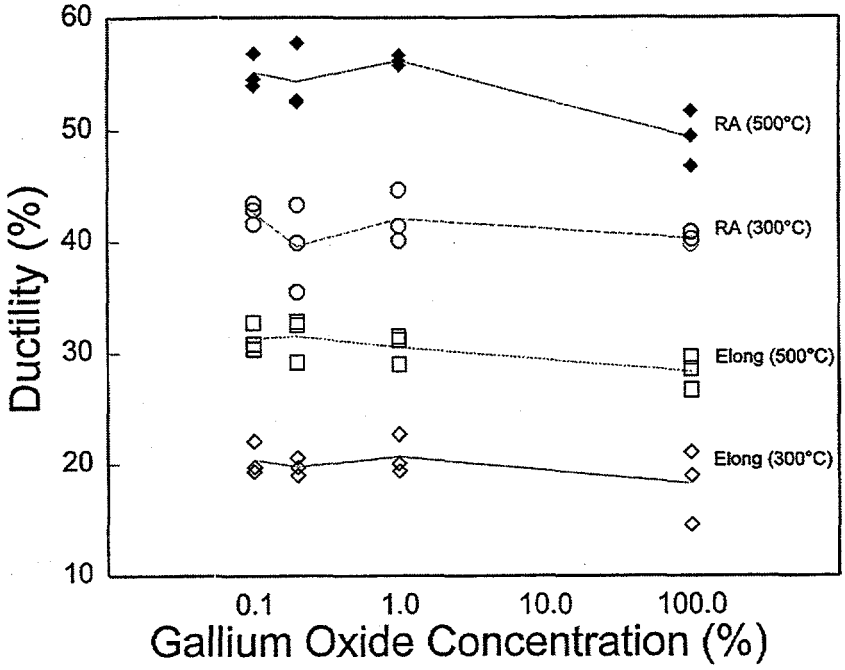


Fig. 19. No significant change in ductility after exposure to Ga_2O_3 in cerium oxide (6 weeks at 300 and 500°C). Elong, elongation; RA, reduction in cross sectional area.

5. CONCLUSIONS

5.1 PHASE I

Corrosion testing revealed that liquid gallium reacts with Zircaloy to form an IC of the form Zr_xGa_y. The extent of reaction is a function of temperature, but more importantly, is strongly dependent on the amount of gallium involved. Although distortion of Zircaloy can be a by-product of ICF at >300°C, distortion at 500°C did not occur at ratios of weight of gallium to surface area of Zircaloy that were 10^4 to 10^5 higher than expected with WG-MOX fuel in an LWR fuel pin. Mechanical property testing after corrosion exposure to form a thin IC layer on the gage section of Zircaloy specimens further confirmed there should be little effect of small quantities of gallium in MOX fuel on the Zircaloy cladding.

No localized corrosion effects have been found. In the corrosion tests (up to 500°C), there was no evidence of grain boundary attack in addition to ICF. In the LME tests at 30 and 100°C, there was no change in the tensile properties when Zircaloy was tested in liquid gallium and the fracture remained ductile.

5.2 PHASE II

Results of testing in Ga_2O_3 or Ce_2O_3-Ga_2O_3 mixtures have not indicated any significant effects at 300°C. At 500°C, an oxide layer formed on the Zircaloy and room temperature tensile tests showed a slight loss in strength with a corresponding increase in ductility. The oxide layer formed during exposure of Zircaloy to several other oxide powders, making it uncertain whether any mechanical effects are related to the presence of gallium. Further testing and analysis are in progress.

At 700°C, which is considerably above LWR operating temperatures, there is evidence of reduction of Ga_2O_3 by Zircaloy and formation of a gallium-rich corrosion product below the ubiquitous oxide layer. However, this gallium-rich zone has not yet been affirmed in tests with <100% Ga_2O_3. Testing and analysis on this issue are still in progress.

Even though testing and evaluation are still in progress, based on the results obtained thus far, it does not appear that 1 to 10 ppm gallium in MOX fuel will have any measurable effect on the properties of Zircaloy cladding.

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APPENDIX

The following tables summarize the status of the work performed. Non-vendor-supplied materials are listed with their trade names. Vendor-supplied materials are listed with generic names.

Table A.1. Additional compatibility tests of zirconium/Zircaloy with gallium or Ga_2O_3

Material	Test environment	Temp. (°C)	Time (h)	Comments
Zr	Ga	400	100	Intermetallic compound formation (ICF)
Zr	Ga	600	134	ICF
Zircaloy-2	Ga	400	100	ICF
Zircaloy-2	Ga	600	100	ICF
Zircaloy-2	Ga_2O_3	400	200	Thin ZrO_2 layer visible at 1000X
Zircaloy-2	Ga_2O_3	600	200	Slightly thicker ZrO_2 layer visible at 1000X
Zircaloy-2	Ga_2O_3	500	672	ZrO_2 layer plus Ga within alloy
Zircaloy-2	Ga_2O_3	500	2016	ZrO_2 layer plus Ga within alloy; no change in thickness (compared with 4 weeks)
Zircaloy-4	Ga_2O_3	700	672	Much thicker ZrO_2 layer (compared with 500°C) plus ICF below oxide; cracks in ZrO_2
Zircaloy-4	Ga_2O_3	700	2016	4 mil ZrO_2 layer plus 3 mils of ICF below oxide
Zircaloy-B (vendor)	Ga	500	200	Electron beam welding (EBW) (vacuum); ICF; very slight distortion
Zircaloy-C (vendor)	Ga	500	200	EBW (vacuum); ICF; distortion
Zircaloy-B (vendor)	Ga	500	200	Thin end cap (0.50 in.); ICF; very slight distortion
Zircaloy-B (vendor)	Ga	500	200	Thick end cap (0.2 in.) with circumferential laser beam welding (LBW) of end cap to seal crevice; ICF; distortion
Zircaloy-C (vendor)	Ga	500	200	Thin end cap; ICF; distortion
Zircaloy-C (vendor)	Ga	500	200	Thick end cap with LBW; ICF; distortion
Zircaloy-C (vendor)	Ga	500	200	~650 ppm Ga in ZrO_2 powder
Zircaloy-C (vendor)	Ga	500	200	~650 ppm Ga at bottom of capsule covered with ZrO_2 powder
Zircaloy-4	Ga_2O_3	700	672	~1800 ppm Ga_2O_3 blended with ZrO_2; interaction to <0.5 mil
Zircaloy-C (vendor)	ZrO_2	500	200	ZrO_2 as surrogate

Table A.1. Additional compatibility tests of zirconium/Zircaloy with gallium or Ga_2O_3

Material	Test environment	Temp. (°C)	Time (h)	Comments
Zircaloy-C (vendor)	CaO	500	200	CaO as surrogate
Zircaloy-B (vendor)	Ce_2O_3	500	200	Ce_2O_3 as surrogate
Zircaloy-B (vendor)	Ga	500	200	3 g Ga; evaluation of W insert geometry for LME & C-M tests
Zircaloy-C (vendor)	Ga	500	200	3 g Ga; evaluation of W insert geometry for LME & C-M tests
Zircaloy-C (vendor)	—			No insert; evaluation of specimen geometry for LME & C-M tests
Zircaloy-C (vendor)	Ga			Large insert; evaluation of W insert for LME & C-M tests
Zircaloy-C (vendor)	Ga			No insert; evaluation of specimen geometry for LME & C-M tests
Zircaloy-C (vendor)	Ga			Small insert; evaluation of W insert for LME and C-M tests
Zircaloy-C (vendor)	Ga	300	200	Determine acceptable, f(T), (no swelling & very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-C (vendor)	Ga	400	200	Determine acceptable, f(T), (no swelling & very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-C (vendor)	Ga	350	200	Determine acceptable, f(T), (no swelling & very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-B (vendor)	Ga	300	100	Determine acceptable, f(t), (no swelling & and very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-B (vendor)	Ga	300	25	Determine acceptable, f(t), (no swelling & very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-B (vendor)	Ga	300	50	Determine acceptable, f(t), (no swelling & very thin corrosion layer) corrosion conditions for C-M tests
Zircaloy-A (vendor)	Ga	500	200	Comparison of distortion, f(material)
Zircaloy-B (vendor)	Ga	500	200	Comparison of distortion, f(material)
Zircaloy-C (vendor)	Ga	500	200	Comparison of distortion, f(material)
Zircaloy-A (vendor)	Ga/Cd	500	200	Comparison of distortion, f(material)
Zircaloy-B (vendor)	Ga/Cd	500	200	Comparison of distortion, f(material)
Zircaloy-C (vendor)	Ga/Cd	500	200	Comparison of distortion, f(material)
Zircaloy-A (vendor)	Ga	500	200	25 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-B (vendor)	Ga	500	200	25 mg Ga; demonstrate amount of attack with decreasing Ga mass

Table A.1. Additional compatibility tests of zirconium/Zircaloy with gallium or Ga_2O_3

Material	Test environment	Temp. (°C)	Time (h)	Comments
Zircaloy-C (vendor)	Ga	500	200	25 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-A (vendor)	Ga	500	200	2.5 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-B (vendor)	Ga	500	200	2.5 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-C (vendor)	Ga	500	200	2.5 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-A (vendor)	Ga	500	200	0.25 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-B (vendor)	Ga	500	200	0.25 mg Ga; demonstrate amount of attack with decreasing Ga mass
Zircaloy-C (vendor)	Ga	500	200	0.25 mg Ga; demonstrate amount of attack with decreasing Ga mass

Table A.2. Status of corrosion tests of Zircaloy with gallium metal

Material	Time (h)	Temperature (°C) 30	100	500	500*	Comment
Zircaloy-A (vendor)	200			✔	✔	No attack at 30 or 100°C; Intermetallic compound formation (ICF) at 500°C
	700	✔	✔	✔	✔	
Zircaloy-B (vendor)	200			✔	✔	ICF; dimensional distortion
	700	✔	✔	✔	✔	No attack at 30 or 100°C; ICF at 500°C but no increase over 200 h; dimensional distortion
Zircaloy-C (vendor)	200			✔	✔	ICF; dimensional distortion
	700	✔	✔	✔	✔	No attack at 30 or 100°C; ICF at 500°C but no increase over 200 h; dimensional distortion at 500°C

* With cadmium.
✔ — Completed test.

Table A.3. Status of corrosion tests of Zircaloy with Ga_2O_3

Materials	Ga_2O_3 (wt %)	Time (weeks)	Temperature (°C) 300	500	700	Comment
Zircaloy-A (vendor)	100	6	✔	✔	✔	
	100	12	✔	✔	✔	
	1	6	✔	✔	✔	
	0.2	6	✔	✔	✔	
	0.1	6	✔	✔	✔	

Table A.3. Status of corrosion tests of Zircaloy with Ga_2O_3

Materials	Ga_2O_3 (wt %)	Time (weeks)	Temperature (°C) 300	500	700	Comment
Zircaloy-B (vendor)	100	6	✔	✔	✔	No interaction at 300°C; ZrO_2 at 500 and 700°C; intermetallic compound formation (ICF) at 700°C
	100	12	✔	✔	✔	Same as for 6 weeks except greater depth of interaction at 700°C
	1	6	✔	✔	✔	
	0.2	6	✔	✔	✔	
	0.1	6	✔	✔	✔	
Zircaloy-C (vendor)	100	6	✔	✔	✔	No interaction at 300°C; ZrO_2 at 500 and 700°C; ICF at 700°C
	100	12	✔	✔	✔	Same as for 6 weeks except greater depth of interaction at 700°C
	1	6	✔	✔	✔	
	0.2	6	✔	✔	✔	
	0.1	6	✔	✔	✔	

✔ — Completed.

Table A.4. Status of mechanical tests of Zircaloy with gallium metal

Material	Type	Temperature (°C) 30	100	300	300*	Comment
Zircaloy-A (vendor)	LME	✔	✔			
	CM			✔	x	
Zircaloy-B (vendor)	LME	✔	✔			
	CM			✔	x	
Zircaloy-C (vendor)	LME	✔	✔			
	CM			✔	x	Fracture surface exhibited nonductile features

* With cadmium.
✔ — Completed test.
LME — Constant extension rate tensile (CERT) test at temperature (triplicate tests).
CM — CERT test at room temperature after 200 h at temperature (triplicate tests).

Table A.5. Status of corrosion-mechanical* tests of Zircaloy with Ga_2O_3

Materials	Ga_2O_3 (wt %)	Temperature (°C) 300	500	700	Comment
Zircaloy-A (vendor)	100	x	x	x	
	1	x	x	x	
	0.2	x	x	x	
	0.1	x	x	x	
Zircaloy-B (vendor)	100	x	x	x	
	1	x	x	x	
	0.2	x	x	x	
	0.1	x	x	x	
Zircaloy-C (vendor)	100	x	x	x	
	1	x	x	x	Room temperature tensile testing completed for preexposure at 300 and 500°C
	0.2	x	x	x	
	0.1	x	x	x	

x — Scheduled.

✔ — Completed.

* Constant extension rate tensile test at room temperature after 6 weeks at temperature (triplicate tests).