INTERNATIONAL NUCLEAR ENERGY RESEARCH INITIATIVE

Final Report Executive Summary

Advanced Corrosion-Resistant Zirconium Alloys for High Burnup and Generation IV Applications

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Research Objectives

The objective of this collaboration between four institutions in the US and Korea is to demonstrate a technical basis for the improvement of the corrosion resistance of zirconium-based alloys in more extreme operating environments (such as those present in severe fuel duty cycles (high burnup, boiling, aggressive chemistry) and to investigate the feasibility (from the point of view of corrosion rate) of using advanced zirconium-based alloys in a supercritical water environment. This technical basis is to be obtained through the comparison of the corrosion kinetics and the examination of the fine structure of oxide layers formed in model alloys. These model alloys are designed to isolate specific features of the microstructure thought to affect the formation of the protective oxide layer so that their effect on the corrosion rate can be studied individually. The key aspect of the program is to rationalize the differences in corrosion kinetics between alloys through the differences in the structure and evolution of the protective oxide formed on each alloy. The structural differences in the oxides were studied using advanced characterization techniques (including submicron-beam synchrotron radiation diffraction and fluorescence, cross-sectional transmission electron microscopy (TEM), transmitted light optical microscopy, and nano-indentation) to characterize both the metal and the oxide so that we can also relate these differences in oxide structure to the original microstructure of the alloy.

Results and Conclusions

A detailed study has been conducted to address the issue of susceptibility of model Zr alloys to uniform corrosion in the proposed supercritical water reactor and of the specific role of alloying elements. Thirty model alloys were corrosion-tested for periods over 400 days in 360°C water (as well as limited testing in lithiated water) and from 150 to over 300 days in 500°C supercritical

water. The corrosion results show a wide range of corrosion behavior with the data included in Appendices A, B, and C and discussed in Tasks 2 and 3.

The general agreement between the three different tests performed at 500°C was quite good. In particular, little difference was seen between the results from static and dynamic autoclave testing. The results from lower pressure steam testing agreed with the supercritical water results but showed slightly less oxide growth. Since the rankings of the alloys were preserved from test to test, it is possible the steam test could serve as a preliminary screening test for supercritical water behavior.

1. Corrosion tests for the model Zr alloys showed a wide variety of corrosion resistance depending on the alloy composition during both 360°C and at 500° testing. These differences manifested themselves in the difference in pre-transition kinetics (as characterized by values of A and n) and in the different tendencies for breakaway.
2. In comparison with other alloys being considered for the SCWR, the Zr alloys showed higher corrosion rates than austenitic alloys and lower corrosion rates than ferritic-martensitic and 12Cr steels.
3. Significant concerns exist in terms of creep rates, and other properties. However, this research indicates that proper alloying additions induce protective oxide growth in model Zr alloys, such that from the corrosion point of view, Zr alloys should be considered as possible candidate materials for application in the supercritical water reactor.

The oxides were also characterized to determine the oxide structures associated with protective and non-protective behavior. These characterization efforts are discussed in Task 4 and rationalized in terms of corrosion behavior in Task 5. Additional results are included in Appendices D, E and F. The main conclusions are:

4. Characteristic differences exist in the oxide-metal interface regions of protective and non-protective oxides. In particular, the presence of two interfacial oxide phases, a highly oriented tetragonal phase and a sub-oxide phase were associated with protective behavior uniform corrosion???
5. The overall crystallographic texture of oxides also showed clear differences between protective and non-protective oxides. The growth direction of the oxides was similar but the pole figures were considerably more anisotropic in the non-protective oxides than in the protective ones.
6. The protection mechanisms and the mechanisms of transition/breakaway were discussed and the differences between alloys are rationalized in terms of mechanistic models.

Benefits of this research

As a result of this program, it has been shown that some Zr alloys can withstand the high temperature corrosion of a supercritical water reactor. The mechanisms of alloy protection have

been studied in detail and rationalized in terms of the oxide structure and its influence on breakaway. This will allow better fuel cladding alloy design for both low and high temperature applications, based on mechanistic concepts.

As a result of this research program, collaborations were established between Penn State, KAERI U. of Michigan, and Westinghouse which proved to be very fruitful. The results of the research have so far been published in three journal publications and two conference proceedings. One more journal publication and two conference proceedings are scheduled for publication in the near term. Additionally this research supported the graduate thesis work of two Ph.D. students at Penn State, one M.Sc. student at Michigan, as well as post-doctoral researchers at both institutions. Graduate students were also supported in KAERI and Hanyang University.

Task 1: Alloy Fabrication and Characterization

This task included manufacturing model alloys to investigate the corrosion mechanism of Zr alloys. The model alloys can be categorized into various groups: Zr-Sn, Zr-Nb, Zr-Nb-Sn, Zr-Fe-Cr, Zr-Cr-Fe, Zr-Cu-Mo and a last group of reference alloys (crystal bar, sponge Zr and Zircaloy-4). Additional model alloys with higher alloying element content were manufactured by KAERI in 2004. The chemical composition of the model alloys tested in this project is summarized in Table 1.1 and 1.2.

The model alloys were prepared by arc melting. The arc-melted buttons were re-melted at least 4 times to promote chemical homogeneity. A typical arc-melted button measures about 70 mm in diameter and 10 mm in thickness, and weighs about 400 g. The arc-melted ingots were beta-solution treated at 1050°C for 30 min in a vacuum furnace, hot-rolled after pre-heating at 580 to 720°C for 10 min and cold-rolled three times to a final thickness of 0.8 mm. Between the rolling steps, the cold-rolled sheets were intermediate-annealed at a temperature of 580 to 720°C depending on the alloy system. Specimens for corrosion testing, 25 by 20 by 0.8 mm in size, were cut from the manufactured strip, mechanically ground up to 1200 grit SiC paper, and then pickled in a solution of 5 vol.% HF, 45 vol.% HNO₃ and 50 vol.% H₂O.

Table 1.1. Target chemical composition of the original model alloys.

Group	Composition (wt.%)	Process temperature
Zr-Sn	Zr-0.2Sn, Zr-0.4Sn Zr-0.8Sn, Zr-1.2Sn	580°C
Zr-Nb	Zr-0.2Nb, Zr-0.4Nb Zr-1.0Nb, Zr-1.5Nb Zr-2.5Nb	650°C
Zr-Sn-Nb	Zr-0.4Sn-0.2Nb Zr-0.4Sn-0.4Nb	650°C
Zr-Fe-Cr	Zr-0.2Fe-0.1Cr (L) Zr-0.2Fe-0.1Cr (H) Zr-0.2Fe-0.1Cr (L) Zr-0.2Fe-0.1Cr (H)	L: 580°C H: 720°C
Zr-Cr-Fe	Zr-0.5Cr Zr-0.5Cr-0.2Fe Zr-1.0Cr Zr-1.0Cr-0.2Fe	650°C
Zr-Cu-Mo	Zr-0.5Cu Zr-0.5Cu-0.5Mo Zr-1.0Cu Zr-1.0Cu-0.5Mo	580°C
Reference	Zr sponge Crystal bar Zr Zircaloy-4	580°C 580°C 720°C

Table 1.2. Target chemical composition of the additional model alloys.

Group	Composition (wt.%)	Process temperature
1.0Fe	Zr-1.0Fe	650°C
	Zr-1.0Fe-0.5Cr	650°C
0.6Fe	Zr-0.6Fe	650°C
	Zr-0.6Fe-0.3Cr	650°C
	Zr-0.6Fe-0.3Mo	650°C
	Zr-0.6Fe-0.6Nb	580°C
1.0Cr	Zr-1.0Cr-0.5Fe	650°C
	Zr-1.0Cr-0.5Mo	650°C

Grain Size of as-fabricated model alloys

The as-fabricated alloys were examined using polarized light microscopy in reflection mode to measure the average grain size. The alloys show a homogeneous predominantly equiaxed grain microstructure, as would be expected from a recrystallizing heat treatment. The measured grain sizes are reported in Table 1-. Micrographs of these alloys are shown in Appendix A.

Table 1-3: Grain size.

Alloy Designation	Composition	Grain Size (microns)	Stdev
11	Zr-0.2Fe-0.1Cr L	9.67	2.65
12	Zr-0.2Fe-0.1Cr H	14.51	3.41
41	Sponge Zr	11.27	2.03
42	Crystal bar Zr	15.37	2.83
43	Zircaloy-4	14.19	2.88

The grain size measured in the Zr-0.2Fe-0.1Cr alloys annealed at high temperature is bigger than the same sample annealed at low temperature, as expected. The largest grain size of all is observed in crystal bar Zr, which is in agreement with the fact that this is the lowest alloying element content alloy, since alloying elements tend to slow down grain growth.

Hardness of Model Alloys

To obtain a rough measure of the mechanical properties of the fabricated alloys, Vickers micro-hardness measurements were taken. Samples were cut and mounted in epoxy and tested in a LECO V-100-C1 hardness tester for a 10 second period under a 1000 g load. The results show that, with the exception of the crystal bar Zr, all alloys tested exhibited hardnesses in the range 140-170, with small variations within series. The series with higher alloying contents had slightly higher hardness values than those ones with lower alloying element content. This indicates that the alloys have similar mechanical characteristics (with the exception of crystal bar Zr).

Figure 1-1: Vickers micro-hardness for all the model alloys investigated.

Texture Measurements of as fabricated alloys

Basal diffraction pole figures of as-fabricated alloys were collected and the results are shown in Appendix F. The Kearns factors (defined as the fraction of resolved basal poles aligned along a particular macroscopic direction [1]) were calculated from the inverse pole figure of the alloys, and are shown in Table 1.4. The calculated f_N (fraction of basal poles along normal direction) ranges from 0.59 to 0.64, again with the exception of crystal bar Zr which exhibits a $f_N=0.51$. This value is similar to reported values of f_N for as-fabricated Zr components.

Table 1-4: f_N measured for the model alloys.

<i>Alloy</i>	<i>Texture index</i>
14 - Zr-0.4Fe-0.2Cr H	0.62
21 - Zr-0.5Cr	0.59
22 - Zr-0.5Cr-0.2Fe	0.62
23 - Zr-1.0Cr	0.62
24 - Zr-1.0Cr-0.2Fe	0.69
33 - Zr-1.0Cu	0.63
34 - Zr-1.0Cu-0.5Mo	0.63
41 - Sponge Zr	0.64
42- Crystal Bar Zr	0.51
43- Zircaloy-4	0.68

Thus the texture, hardness and grain size of the alloys is similar. Given that homogeneous and similar microstructures were obtained, it is possible to isolate the other variables as composition differences and investigate their impact on the corrosion behavior of the alloys, as is the objective of this study.

Characterization of second-phase particles (SPP)

The alloys prepared and used in this study and their target chemical compositions are listed in Table 1-5. The actual compositions were measured and did not vary from the target composition by more than 10% (e.g. Zr1.0Fe, could vary by $\pm 0.1\text{Fe}$, to Zr0.9Fe or Zr1.1Fe). The main groups contained alloys that form second phase particles (Zr-Fe-Cr alloys, Zr-Cr-Fe alloys, Zr-Cu-Mo) and alloys that form extensive solid solutions in alpha-Zr, (Zr-Nb, Zr-Sn and Zr-Nb-Sn) in addition to pure Zr and Zircaloy-4.

The as-fabricated model alloys were characterized using synchrotron radiation X-ray diffraction to identify second phases present. One example of such an examination is shown in Figure 1.2, where the x-ray intensity is plotted versus two theta angle acquired using an x-ray energy of 17 keV, for the examination of the alloys Zr-0.4Fe-0.2Cr and Zr-0.2Fe-0.1Cr, processed at 580°C (indicated as L for low temperature heat treatment) and 720°C (indicated H for high temperature heat treatment). The vertical scale is logarithmic to highlight in greater detail the small second phase peaks, which comprise only a small fraction of the total sample volume. The experiments were run the APS synchrotron facility where the combination of the high photon flux and the low background permits detection of small diffraction peaks. Second phase particles having

volume fractions as low as 0.1 to 0.2% can be detected, as the ones expected for the alloy systems investigated in this project. Figure **Error! Reference source not found.** illustrates these capabilities.

The alloys were characterized using synchrotron radiation X-ray as described in chapter 2 and the target precipitate structures and sizes were observed. The types of precipitates identified and their approximate size and volume fraction are also shown in Table 1-6. It is apparent from the table that synchrotron radiation allows a level of characterization of the metallic alloy microstructure that can help highlight the specific role of individual alloy features on the corrosion process.

Figure 1.2: Diffracted intensity versus two-theta angle (logarithmic scale) for two Zr-Fe-Cr alloys (Zr-0.2Fe-0.1Cr and Zr-0.4Fe-0.2Cr) processed at two different temperatures (580 and 720°C) obtained with synchrotron radiation for the purpose of identification of second phase particles phases present in the alloy.

The precipitates volume fractions were calculated assuming that all the present elements have precipitated out, which is consistent with the findings from the diffraction patterns, where the peaks heights for alloys having similar alloying contents were very close. The instrumental line broadening was measured using a diffraction pattern collected from a NIST LaB₆ standard (Standard Reference Material 660a Lanthanum Hexaboride Powder). The instrumental broadening was measured to be 0.05 degrees in 2θ at 9.5

keV. After subtracting the instrumental peak broadening, the FWHM (full width at half maximum) of the diffraction peaks was calculated as seen on Equation:

$$FWHM_{2\theta} = \sqrt{FWHM_{Observed}^2 - FWHM_{LaB_6}^2} \quad (1-1)$$

Table 1.5 : Characteristics of the model alloys.

Alloy system	Composition (wt.%)	Precipitate Characteristics		
		Crystal Structure	Size (nm)	Volume Fraction
Zr-Nb	Zr-0.2, 0.4, 1.0, 1.5, 2.5Nb	hcp Zr(Nb,Fe) ₂ , bcc β-Nb and orthor. Zr ₃ Fe	75-110	Increases as Nb content increases (0-2.3%)
Zr-Fe-Cr	Zr-0.4Fe-0.2Cr and Zr-0.2Fe-0.1Cr (two processing temperatures: 580 °C (L) and 720 °C (H))	Cubic and hex. Zr(Cr,Fe) ₂	35-107	~1%
Zr-Cr-Fe	Zr0.5Cr, Zr-0.5Cr-0.2Fe, Zr-1.0Cr, Zr-1.0Cr-0.2Fe	Cubic ZrCr ₂ and cubic Zr(Cr,Fe) ₂	40-60	~1-1.5%
Zr-Cu-Mo	Zr-0.5Cu, Zr-0.5Cu-0.5Mo Zr-1.0Cu, Zr-1.0Cu-0.5Mo	Tet. Zr ₂ Cu and cubic ZrMo ₂ when Mo present	> 100	~0.5-2%
Zr-Sn-Nb	Zr-0.4Sn-0.2Nb, 0.4Sn-0.4Nb	no precipitates	N/A	N/A
Zr-Sn	Zr-0.2, 0.4, 0.8, 1.2 Nb	no precipitates	N/A	N/A
Reference Alloys	Sponge Zr, Crystal bar Zr, Zircaloy-4	no precipitates for Zr hcp C14 Zr(Cr,Fe) ₂ for Zircaloy-4	80	~0.5 for Zircaloy-4

Similar characterization was performed for all the alloys studied. All alloys considered for this study formed second-phase precipitates with the exception of Zr-0.2Nb and Zr-0.4Nb alloys, which have a Nb concentration below the solubility limit in α-Zr. The types of second phase precipitates were: (i) of the C14 hcp Zr(Cr,Fe)₂ type in the ZrFeCr and ZrCrFe alloys (with the exception of the low temperature processed materials which exhibited cubic C15 type Laves phase precipitates of similar composition) , (ii) bcc β-Nb and hcp (Zr,Nb)Fe₂ precipitates in the Zr-Nb alloys (Fe is an impurity in these alloys at 100-600 ppm) (iii) cubic C15 ZrCu₂ and hcp C14 ZrMo₂ type in the ZrCuMo alloys and (iv) the usual precipitates in Zircaloy-4. No precipitates were detected in either sponge or crystal bar Zr, and the detection limit is estimated at <0.1%. The different processing temperatures in the ZrFeCr alloys created alloys with small and large precipitates and the two compositions gave different volume fractions.

References:

- [1] J. J. Kearns and C. R. Woods, J. Nucl. Mater., vol. 20, (1966), 241.

Task 2: Corrosion Testing at 360°C

The autoclave corrosion tests for the model alloys were performed at both KAERI and Westinghouse. The testing environments at KAERI included both 360°C pure water and 360°C water containing 70 ppm Li while testing at Westinghouse was only in 360°C pure water. The testing in pure water was performed to assess the uniform corrosion behavior of the materials while testing in lithiated water was performed as an accelerated corrosion test and to assess the sensitivity of the alloys to lithium which is added to PWR coolant for pH control.

Autoclave testing was performed in accordance with ASTM G2 (Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680°F [360°C] or in Steam at 750°F [400°C]) in which the autoclave is steamed prior to reaching the test temperature in order to reduce dissolved oxygen concentration.

Coupons were cut from the manufactured strip and measured approximately 25 mm × 20 mm × 0.8 mm in size. Duplicate samples from each alloy were tested to provide archive samples at intermediate exposure times. These samples were used for the oxide characterization task. The coupons were prepared for autoclaving by mechanically grinding the surface using 1200 grit SiC paper and then pickling in a solution of 5 vol.% HF, 45 vol.% HNO₃ and 50 vol.% H₂O followed by thorough rinsing. The coupons were then measured and weighed prior to autoclaving.

The autoclave tests were performed to generate both pre-transition and post-transition corrosion data. The pre-transition data (specimen weight gain) were collected at relatively short time intervals during the first 50 to 100 days to characterize the pre-transition corrosion kinetics. Data collection at longer term intervals of about 30 days was performed to characterize the post-transition kinetics.

Westinghouse Test Results

Westinghouse autoclave testing focused on the precipitate forming alloys as listed in Table 1.5. The materials included three alloy families (ZrFeCr, ZrCrFe, and ZrCuMo) along with reference material (Zircaloy-4 and Zr). The results for the 360°C water experiments are presented in the following series of figures, expressing measured weight gain in mg/dm² versus exposure time (days) for the model alloys. The results for the precipitate alloys show individual curves for several coupons of each alloy, whereas the particular samples archived for characterization are marked in red.

Figure 2.1: Weight gain *versus* time curves for the ZrFeCr alloy series after autoclave exposure to 360°C water at saturation pressure (18.7 MPa): a) Zr-0.2Fe-0.1Cr low temperature (580°C) anneal, b) Zr-0.2Fe-0.1Cr high temperature (720°C) anneal, c) Zr-0.4Fe-0.2Cr low temperature (580°C) anneal and d) Zr-0.4Fe-0.2Cr high temperature (720°C) anneal.

The weight gain curves for the ZrFeCr series (alloys 11-14) alloys are shown in Figure 2.1. The two alloys in this series had a constant Fe:Cr ratio = 2 (similar to Zircaloy-4) and were processed at 580°C or 720°C. The precipitate size increased with increased processing temperature and the volume fraction of precipitates increased as the Fe and Cr content increased.

Alloy 11 underwent breakaway at about 200 days of exposure. Alloy 12 shows signs of a transition or possible breakaway at around 300 days as the weight gain departs from a power law relationship. Alloys 13 and 14 had consistently the best corrosion resistance among the model alloys tested, suggesting that small additions of Fe and Cr up to 0.4 Fe (wt %) and 0.2 Cr (wt %) significantly enhance the corrosion resistance of Zr. These facts suggest that improvement in corrosion behavior is achieved with (i) an increase in the volume fraction of precipitates and (ii) an increase of precipitate size due to the fact that the same alloy (Zr-0.2Fe-0.1Cr) processed at a higher temperature survived with a protective oxide for a longer period of time (Alloy 12 versus Alloy 11).

The ZrCrFe alloys contain higher concentrations of Cr and Fe and were chosen for their potential application in high temperature environments. The precipitate chemistry in these alloys is different than the precipitate chemistry in the above ZrFeCr alloys due to the higher Cr content. Processing was performed at an intermediate temperature of 650°C.

Figure 2.2: Weight gain *versus* time curves for the ZrCrFe alloy series after autoclave exposure to 360°C water at saturation pressure (18.7 MPa): **a)** Zr-0.5Cr, **b)** Zr-0.5Cr-0.2Fe, **c)** Zr-1.0Cr and **d)** Zr-1.0Cr-0.2Fe.

Figure 2.2 shows the weight gain curves for the ZrCrFe series (alloys 21-24). The Zr-0.5Cr and Zr-1.0Cr alloys did not exhibit good corrosion resistance, but some improvement was observed with the addition of 0.2 wt % Fe. The Zr-0.5Cr alloy suffered breakaway corrosion from the beginning of the testing while breakaway in the Zr-1.0Cr was delayed to about 50 days. The addition of 0.2 wt% Fe significantly increased the corrosion resistance of both of the Zr-Cr binary alloys. Zr-1.0Cr-0.2Fe exhibited the best corrosion resistance in this series and did not show signs of breakaway through the duration of test (~ 400 days).

The third set of alloys (ZrCuMo) was selected for their potential application at high temperature. The alloys also provided different precipitates than those present in ZrFeCr and ZrCrFe. Since Cu is a more noble metal than Fe and Cr, the susceptibility of Cu

containing precipitates to oxidation is expected to be lower and may have an impact on corrosion performance.

In the ZrCuMo alloys series (Alloys 31 to 34) the better performing alloys were the Zr-Cu alloys as shown in Figure 2.3. Alloy 31 (Zr-0.5Cu) has shown protective behavior for the duration of the test, but a few coupons did deviate from that behavior and have shown breakaway behavior. Alloy 33 (Zr-1.0Cu) consistently exhibited protective behavior up to 430 days, showing signs of a transition at about 200 days of exposure. Further exposure would be necessary to assess if the oxide would or not recover from the transition at 200 days, but the corrosion rate after the transition appears to be decreasing suggesting a transition rather than breakaway.

Figure 2.3: Weight gain *versus* time curves for the ZrCuMo alloy series after autoclave exposure to 360°C water at saturation pressure (18.7 MPa): **a)** Zr-0.5Cu, **b)** Zr-0.5Cu-0.5Mo, **c)** Zr-1.0Cu and **d)** Zr-1.0Cu-0.5Mo.

Commercially pure Zr (sponge and crystal bar) and Zircaloy-4 were also tested to serve as reference materials for the model alloys. Figure 2.4 shows the weight gain curves for the three reference alloys (Alloys 41-43).

The commercially pure Zr coupons either showed non protective behavior from the beginning of the corrosion test or in a few samples showed protective behavior up to 50 days of exposure. No significant differences were seen between Crystal bar and Sponge Zr, indicating that the higher impurity level of sponge Zr (~ 600 wt. ppm Fe versus 100 wt. ppm for crystal bar) did not impact corrosion behavior. After this period, a

destabilization of the oxide growth occurred that is characterized by a rapid acceleration of corrosion. After breakaway, the oxide turns white (characteristic of a porous, non-protective oxide) and powdery. This phenomenon is termed “breakaway corrosion” and is distinguished from an oxide transition which recovers stable growth.

Figure 2.4: Weight gain *versus* time curves for the ZrCrFe alloy series after autoclave exposure to 360°C water at saturation pressure (18.7 MPa): **a)** Sponge Zr **b)** Crystal Bar Zr and **c)** Zircaloy-4.

The weight gain observed for Zircaloy-4, manufactured in the same way as the models alloys, is shown in Figure 2.4c. The Zircaloy-4 samples exhibit the classical behavior of cyclic oxide growth. After approximately 150 days there is an increase in the corrosion rate, associated with the temporary loss of protectiveness at the oxide transition, followed by the reconstruction of the protective oxide and the re-establishment of the same kinetics exhibited before the transition. The oxide growth is stable and the appearance of the oxide is black and shiny.

Figure 2.5: Weight gain *versus* time curves for the ZrNb, ZrSn and ZrNbSn alloy series after autoclave exposure to 360°C water at saturation pressure (18.7 MPa): **a)** ZrNb **b)** ZrNbSn and **c)** ZrSn.

Figure 2.5 shows the weigh gains curves for the model solid solution alloys (ZrNb, ZrSn and ZrNbSn) that were investigated in this project. The ZrSn alloys exhibit higher initial corrosion rates and an oxide transition around 50-60 days of exposure. It is also clear that some weight loss occurs after 120 days, likely due to oxide spalling. The higher Sn content alloys (0.8 and 1.2 wt/%) suffered unstable oxide growth after 280 days of exposure. The lower Sn content alloys resisted and formed protective oxide layers up to the duration of the test, about 350 days. A second transition seems to be taking place at 300 days, but further data would be necessary to access that possibility. The increase in corrosion rate at 300 days may lead to unstable growth of oxide, i.e., breakaway corrosion.

In the ZrNb system, the Zr-0.2Nb alloy showed lowest corrosion with little dependence of corrosion rate with Nb content between 0.4 and 1.5 Nb. No clearly marked transition in corrosion kinetics was observed in the ZrNb alloys.

The ZrNbSn alloys showed an oxide transition but exhibited corrosion rates that were similar to the equivalent Zr-Nb alloys of the same Nb content.

Figure 2.6 shows images of the autoclaved coupons exposed to 360°C water. As seen in the images, the oxides formed on different alloys have different characteristics. The oxide formed on alloy 14 (Zr-0.4Fe-0.2Cr, 720°C) is intact and shiny black, as were all the oxides formed on the ZrFeCr model alloys. The oxide formed on alloy 21 (Zr-0.5Cr) has a green color due to presence of Cr. Although the alloy has undergone breakaway corrosion, no spallation of the oxide occurred. Oxides formed on the remaining ZrCrFe alloys presented similar aspects, even after breakaway. The oxide formed on alloy 33 (Zr-1.5Cu) is also intact as opposed to the oxide formed on alloy 34 (Zr-1.0Cu-0.5Mo) where the oxide is in the process of losing its integrity and pieces of oxide spalling from the surface of the sample. The oxide formed on the Crystal Bar Zr is different from the ones formed on the other alloys. As shown in Figure 2.6, the oxide is initially black and then begins to turn into a white and powdery oxide with exposure time; the final oxide, which is also a breakaway oxide, is completely powdered and white.

The results suggest that the alloy composition and microstructure play a dominant role in determining corrosion behavior. Minor changes in the composition of the alloys can lead to dramatic changes in corrosion behavior, as, for example, the addition of 0.2Nb to pure Zr.

In general, the best performing alloys in water 360°C were from the ZrFeCr system and the best behaving alloy was alloy 14, Zr-0.4Fe-0.2Cr, 720°C. This is in accordance to Barberis *et al*, where they found that in addition to the second phase particle (SPP) size, their volume fraction is an important parameter. An increase in volume fraction of the SPPs would decrease the corrosion rate, whatever nature of the SPP. Alloy 14 has the highest SPP volume fraction among the ZrFeCr model alloys.

The key to good corrosion resistance is to avoid breakaway corrosion and to maintain a stable, protective oxide and oxide growth. The pre-transition portion of the weight gain curves were systematically fit to the power law of the type $W = K t^n$, where W is weight gain, K is the pre-exponential and t is time. The pre-exponential term K is a rate at which the sample gains weight per unit of time to the power of n . Characterization of the oxidation kinetics was performed based upon three factors: K , n , and time to transition.

The fit to the corrosion data was performed on the average weight gain measurement from 4-8 samples. The power law used to fit the curves provided an excellent fit to the corrosion results (R^2 values were consistently near 0.99). The weight gains of the individual samples were also fit with the same power law, and very similar values of K and n were obtained. This shows that the results are consistent from sample to sample and there is little variability in the corrosion results, more specifically in the pre-transition regime.

Figure 2.6: Zr alloy coupons autoclaved in 360°C water.

Figure 2.7: Fit of the weight gain *versus* exposure time curve to the power law of the type $W = A.t^n$, for Sponge Zr. The points represent averages of the samples 42-09, 42-10, 42-11 and 42-12 in 360°C water.

Figure 2.7 shows an example of the fit of the weigh gain curve for Zr sponge (Alloy 42). The constants for the fitted curves for the pre-transition kinetics of the model alloys in water 360°C are tabulated in Table 2.1.

The calculated values of n are almost all below 0.5, and some significantly below (~0.18), indicating in most of the alloys a significant deviation from a pure diffusion based process and consequent parabolic behavior. The reason for these departures is not clear, but could be related to factors in the oxide that hamper or impede diffusion. These could include the presence of lateral cracks which serve as obstacles to oxygen ion migration, increasing grain size within the oxide could reduce grain boundary diffusion, or the presence of stress, all of which could slow down diffusion and affect the value of n . Another possibility is related to the fact that the protective layer of the oxide is only a fraction of the measured oxide layer; if this fraction changes (increases) with thickness then kinetics lower than parabolic could be established. Finally, it is possible that slower electron transport through the oxide layer slows down oxygen ion transport.

It is important to notice that the values of n shown in Table 2.1 are characteristic of each alloy series. The ZrNb samples have an average n of 0.42, reasonably close to parabolic. In the Zr-Sn alloys, the average n is 0.32 and for ZrNbSn it is 0.41. In contrast to the precipitate forming alloys, including Zircaloy-4, the value of n is closer to 0.2.

In comparing Zircaloy-4 with the model alloys made from its individual constituents (Sn, Fe, Cr), it is possible to observe that an alloy with the same FeCr content as Zircaloy behaves quite well, and does not show an oxide transition within the time tested (Zr-0.4Fe-0.2Cr). At the same time the ZrSn alloys show a transition and subsequently, breakaway (Zr-0.8 and 1.2 Sn).

Table 2.1: Pre-transition kinetics constants for model Zr alloys test in the 360°C water.

<i>Alloy Group</i>	<i>Alloy</i>	<i>A</i>	<i>n</i>	<i>ave. A</i>	<i>ave. n</i>
Zr-Fe-Cr	Zr-0.2Fe-0.1Cr, 580°C	10.2	0.20	9.89	0.21
	Zr-0.2Fe-0.1Cr, 720°C	10.2	0.20		
	Zr-0.4Fe-0.2Cr, 580°C	9.78	0.21		
	Zr-0.4Fe-0.2Cr, 720°C	9.39	0.22		
Zr-Cr-Fe	Zr-0.5Cr	non-protective from the beginning of test		10.9	0.19
	Zr-0.5Cr-0.2Fe	11.5	0.17		
	Zr-1.0Cr	9.73	0.21		
	Zr-1.0Cr-0.2Fe	11.6	0.18		
Zr-Cu-Mo	Zr-0.5Cu	13.6	0.17	14.1	0.18
	Zr-0.5Cu-0.5Mo	13.6	0.17		
	Zr-1.0Cu	15.1	0.17		
	Zr-1.0Cu-0.5Mo	14.0	0.21		
Zr	Sponge Zr	8.50	0.20	7.56	0.22
	Crystal Bar Zr	6.61	0.24		
Zircaloy	Zircaloy-4	11.0	0.21	10.20	0.22
	Zr-4 Control	9.40	0.23		
Zr-Sn	Zr-0.2Sn	9.30	0.29	9.50	0.29
	Zr-0.4Sn	8.10	0.32		
	Zr-0.8Sn	10.0	0.27		
	Zr-1.2Sn	10.6	0.27		
Zr-Sn-Nb	Zr-0.4Sn-0.2Nb	5.92	0.43	6.52	0.41
	Zr-0.4Sn-0.4Nb	7.11	0.39		
Zr-Nb	Zr-0.2Nb	6.40	0.43	5.92	0.45
	Zr-0.4Nb	5.00	0.52		
	Zr-1.0Nb	5.90	0.45		
	Zr-1.5Nb	5.80	0.44		
	Zr-2.5Nb	6.50	0.40		

KAERI Test Results

Parallel corrosion testing was also performed at KAERI. The autoclave corrosion tests for the model alloys included testing in both 360°C pure water and in 360°C lithiated water.

Zr-Fe-Cr alloy, which was manufactured by US and transferred to ROK, was tested in 360°C water and in 360°C lithiated water using the autoclaves in KAERI. Zr-Fe-Cr alloys were designed to change the size and the volume fraction of the precipitates by varying the alloying element content and the processing temperature. Figure 2.8 shows the corrosion behavior of Zr-Fe-Cr alloys in 360°C water as well as in 360°C lithiated water. Zr-0.2Fe-0.1Cr and Zr-0.4Fe-0.2Fe which were manufactured by the higher temperature processing (720°C) showed a lower corrosion rate. However, the breakaway behavior was observed in the alloys processed at the lower temperature (580°C). Some part of the oxide, especially in the vicinity of the edges showed the evidence of the corrosion acceleration, but the remaining part of the oxide still showed a black color and an adherent behavior. Such a localized corrosion behavior seems to result from the inhomogeneity of the base alloy rather than a specific corrosion behavior. This non-uniform corrosion behavior was not in the Zr-0.4Fe-0.2Cr (580°C) material tested in the US and may be a result of variability in individual coupons. Zr-Fe-Cr alloys showed the breakaway behavior at around 150 days in lithiated water except for Zr-0.4Fe-0.2Cr(H) that maintained lower corrosion rate and a black, adherent oxide up to 450 days. The excellent behavior of Zr-0.4Fe-0.2Cr (H) was consistent with the US results.

The corrosion behavior of Zr-Cr and Zr-Cr-Fe alloys, which were manufactured in the US and transferred to ROK, was evaluated in 360°C water and in 360°C lithiated water. Figure 2.9 shows the corrosion behavior of Zr-Cr and Zr-Cr-Fe alloys. Zr-0.5Cr and Zr-1.0Cr showed a higher corrosion rate and have the brown-colored oxide. The corrosion rate of both binary Zr-Cr alloys was poor with the rate of Zr-0.5Cr being higher than that of Zr-1.0Cr. The results were similar to those observed in the US testing with poor corrosion of both binary Zr-Cr alloys. On the other hand, Fe-containing alloys showed a low corrosion rates and have a black-colored oxide up to 430 days. The corrosion rate of the two Fe-containing alloys was almost the same as each other. It was confirmed that the beneficial effect of Fe was explicit. Testing in the US also showed the beneficial effect of Fe in the Zr-1.0Cr-0.2Fe alloy. In lithiated water, however, all the alloys showed the breakaway behavior after 90 days except for Zr-0.5Cr-0.2Fe alloys where the breakaway was occurred at 140 days.

Zr-Cr and Zr-Cr-Fe was also manufactured in ROK with the same nominal composition and the same manufacturing process and tested along with US-manufactured Zr-Cr and Zr-Cr-Fe alloys. Their corrosion behavior in 360°C pure and in lithiated water was depicted in Figure 2.10. The corrosion behavior of ROK-manufactured Zr-Cr-Fe alloys was almost similar to the US-manufactured Zr-Cr-Fe alloys. Fe-containing alloys maintained low corrosion rate up to 600 days and have a black and adherent oxide. In

lithiated water, all the Zr-Cr-Fe alloys showed the breakaway behavior after 60 days. However, the weight gain increment of Fe-containing alloys was smaller when compared to Zr-Cr alloys. The beneficial effect of Fe was also confirmed to be clear even in the lithiated water.

The US-manufactured Zr-Cu-Mo alloys showed the breakaway behavior at the early stage of the corrosion test and the oxide turned white as shown in Figure 2.11. The Zr-Cu alloys survived to more than 400 days and retained a black, adherent oxide. The addition of Cu was shown to be deleterious in lithiated water: Zr-1.0Cu and Zr-1.0Cu-0.5Mo showed the breakaway behavior just after 60 days whereas the time to transition was extended to 180 days in Zr-0.5Cu and Zr-0.5Cu-0.5Mo which showed the trace of the localized corrosion on the oxide surface.

Figure 2.12 shows the corrosion test result for the ROK-manufactured Zr-Cu-Mo alloys. The corrosion resistance of ROK-manufactured Zr-Cu-Mo alloys showed the breakaway behavior except for Zr-1.0Cu alloys which have a low corrosion rate and a black oxide up to more than 600 days. The alloys with a higher Cu content showed a better corrosion resistance. The breakaway was occurred at the earlier time in the Mo-containing alloy. The results are qualitatively similar to the corrosion results from the testing in the US with the Zr-Cu binary alloys exhibiting better behavior than the Mo-bearing alloys. In lithiated water, all the Zr-Cu-Mo alloys showed the breakaway behavior at the early stage of the corrosion test.

Corrosion tests were also performed for the reference alloys: Zr sponge, crystal bar Zr and Zircaloy-4 which were supplied by the US. Figure 2.13 shows the corrosion behavior of the reference alloys in 360°C water and in 360°C lithiated water. The sponge Zr and crystal bar Zr were very susceptible to the corrosion in 360°C pure water as well as in lithiated water. Zircaloy-4 survived and showed a weight gain of 100mg/dm² at 450 days in 360°C pure water. These results are similar to the US testing with the pure Zr materials exhibiting breakaway behavior and Zircaloy-4 showing periodic growth of the oxide. However, the corrosion rate of Zircaloy-4 was increased in lithiated water and the oxide changed to brown and showed the evidence of the partial spallation.

Figure 2.14 shows the corrosion behavior of Zr-Nb alloys in 360°C pure water and in 360°C lithiated water with 70 ppm Li. The corrosion rate was increased with increase of Nb content in Zr-0.2Nb and Zr-0.4Nb which are designed to investigate the solute element effects on the corrosion of Zr alloys. In the Zr-Nb alloys whose Nb content was more than the solubility in alpha-Zr, the corrosion rate was slightly decreased with increase of the Nb content. The oxide was still black and maintained to be adherent even up to 600 days regardless of alloys compositions. Zr-Nb alloys were very susceptible to the breakaway corrosion in 360°C lithiated water. The corrosion rate was accelerated just after 60 days in Zr-0.2Nb and Zr-0.4Nb. However, the time to the breakaway was extended to the longer time when Nb content was more than the solubility limit. The color of the oxide changed to brown but the oxide was not spalled after the breakaway.

Figure 2.15 shows the corrosion behavior of the Zr-Nb-Sn alloys in 360°C water and in 360°C lithiated water. Zr-0.4Sn-0.4Nb showed a lower corrosion rate when compared to Zr-0.4Sn-0.2Nb. However, in the Zr-Nb binary alloys, the Zr-0.2Nb showed a lower corrosion resistance than Zr-0.4Nb. It was thought that the corrosion mechanism of the Sn containing alloys was different from the binary alloys. Moreover, the Sn addition was very beneficial for the corrosion resistance in lithiated water. The time to the breakaway was about three times longer in Sn-containing ternary alloys.

The corrosion resistance of Zr-Sn binary alloys was similar to each other and their corrosion rate was maintained at the relatively lower level before the breakaway, as shown in Figure 2.16. After 280 days, Zr-0.8Sn and Zr-1.2Sn alloys showed the breakaway corrosion behavior. The weight gain increment of Zr-1.2Sn was two times greater than that of Zr-0.8Sn. The color of the oxide was changed to be brown in Zr-1.2Sn. However, the Zr-0.2Sn and Zr-0.4Sn alloys maintained their lower corrosion rate up to 350 days. The Zr-Sn alloys have more corrosion resistance than Zr-Nb alloys in lithiated water. Their corrosion resistance was increased with increase of Sn content in lithiated water except for Zr-1.2Sn.

Figure 2.8: Corrosion behavior of US-manufactured Zr-Fe-Cr alloys in 360°C pure water and lithiated water.

Figure 2.9: Corrosion behavior of US-manufactured Zr-Cr-Fe alloys in 360°C pure water and lithiated water.

Figure 2.10: Corrosion behavior of ROK-manufactured Zr-Cr-Fe alloys in 360°C pure water and lithiated water.

Figure 2.11: Corrosion behavior of US-manufactured Zr-Cu-Mo alloys in 360°C pure water and lithiated water.

Figure 2.12: Corrosion behavior of ROK-manufactured Zr-Cu-Mo alloys in 360°C pure water and lithiated water.

Figure 2.13: Corrosion behavior of the reference alloys in 360°C pure water and lithiated water.

Figure 2.14: Corrosion behavior of Zr-Nb alloys in 360°C pure water and lithiated water.

Figure 2.15: Corrosion behavior of Zr-Sn-Nb alloys in 360°C pure water and lithiated water.

Figure 2.16: Corrosion behavior of Zr-Sn alloys in 360°C pure water and lithiated water.

Task 3: Corrosion Tests at 500°C

High temperature autoclave testing was performed at 500°C to assess the feasibility of utilizing Zr-based alloys in supercritical water (SCW) applications. The high temperature test environments included 500°C/1500 psi steam and 500°C/3650 psi supercritical water (SCW). Testing was performed in a manner consistent with the ASTM G2. The corrosion behavior of the specimens was evaluated by measuring the weight gain as a function of the exposure time.

The corrosion test in supercritical water was performed in two types of facilities: (i) a static autoclave (closed environment in which water does not recirculate) at KAERI (designated as SCW static) and (ii) a dynamic loop system (water is constantly refreshed, and chemistry is controlled) at the University of Michigan (designated as SCW dynamic). Details of the testing at the University of Michigan are described below. The 500°C steam corrosion test was performed at Westinghouse and KAERI in static autoclaves at 10.3 MPa (1500 psi). Corrosion coupons for all testing at 500°C were comparable to those prepared for testing in 360°C environments. Coupon size was 20 mm × 25 mm and the surface was prepared by mechanical grinding followed by pickling and thorough rinsing.

A test environment of 500°C is very aggressive for Zr-based alloys as prior experience has shown that widely used alloy such as Zircaloy-4 forms very thick oxide films after a few days of exposure in 500°C steam. The results from testing of the model alloys in this program have demonstrated that some of the compositions exhibit significant improvement over Zircaloy-4. Sample weight gains for the model alloys are provided in the appendices (Appendix C). The results of the testing are discussed below following a description of the supercritical water test facility at the University of Michigan.

Corrosion Testing at 500 °C Supercritical Water Test Facility (University of Michigan)

All corrosion tests at the University of Michigan were conducted in a flowing supercritical water loop (Figure 3.1). The closed loop system operated at a flow rate of 12 ml/min. Water in a main column is continually pumped through a secondary loop where impurities are filtered out by an ion exchanger. A gas line feeds into the bottom of the main column, in which Ar gas is bubbled to deoxygenate the water to <10 ppb O₂.

The main loop of the system consisted of a pump, pre-heater, autoclave, chiller, and back-pressure regulator (BPR). Water flowed from the bottom of the main column into a high pressure liquid chromatography pump capable of flow rates to 100 ml/min and rated to 6000 psi at 24 ml/min. The water was pumped into a pre-heater, which raised the temperature of the water from room temperature to approximately 440°C. Water from the pre-heater passed through an insulated section of pipe into the autoclave (Figure 3.2). Water entered the autoclave at the bottom of the enclosure; the entrance of the outlet line was located at the top. As the autoclave filled, the corrosion coupons were immersed in water that was eventually forced out the top when the autoclave was full. The autoclave was heated by three heater bands, which brought the temperature up to

500°C. They were positioned so that temperature within the autoclave remained uniform and stable. After exiting the autoclave, water passed through a chiller where heat was exchanged with an external line of flowing cold water to bring the water temperature down to room temperature. Pressure in the autoclave was controlled by a BPR rated to 4000 psi on the outlet line. Low-pressure water on the backside of the BPR was passed through another ion exchanger and back into the top of the main column. All high-temperature areas of the loop were enclosed in a 2.54 cm thick Lexan shield to protect lab personnel and equipment from any steam leaks that could occur.

Figure 3.1: Schematic of the flowing supercritical water loop.

Figure 3.2: A diagram of the inside of the autoclave.

Corrosion coupons were hung from an internal tree by Inconel wire that hooked through the hole in each coupon and wrapped around a branch of the tree. The tree was capable of supporting up to 48 samples at a time, allowing each sample to be exposed on all surfaces except the small area within the hole touched by the wire. Two coupons of each alloy were tested simultaneously in all corrosion tests to increase the reliability of the results. Coupons of the same alloy were hung on opposite sides of the autoclave to detect any lack of uniformity in internal autoclave conditions.

Conductivity, temperature, oxygen content and pressure in the loop were measured throughout the test. Conductivity was measured in the inlet line between the main column and pump, and again in the outlet line immediately after the BPR. The ion exchange system maintained the conductivity of the inlet water at values as low as $0.06\mu\text{S}/\text{cm}$. The temperature was measured by thermocouples located in the center of the pre-heater coils, at the inlet line, attached to the external side of the autoclave, and close to the center of the inside of the autoclave. The inlet and internal thermocouples were used primarily to monitor and record the temperatures of water entering and within the autoclave. The thermocouples in the pre-heater and on the outside of the autoclave were used by temperature controllers located next to the system. These controllers regulated the current to the pre-heater and heater bands using solid state relays. The constant feedback the controllers received from the thermocouples allowed both heating systems to be maintained at stable temperatures throughout the test, and were crucial for increasing and decreasing temperature at system startup and shutdown. Dissolved oxygen content was measured by an Orbisphere Model 2713 High Sensitivity Oxygen Detector that can analyze water from either the inlet or outlet line. Two Omega pressure transducers measured pressure at the inlet to the pre-heater and on the high-pressure side of the BPR. The temperature, pressure, and conductivity data were relayed to a computer running LabView software that records new data every 10 seconds. To help ensure safety and system stability, LabView can also contact lab personnel through pagers when system parameters exceed set bounds.

The tests were conducted in accordance with the standards described in ASTM G2-88. Argon was bubbled through the main column until the dissolved oxygen content was less than 10 ppb. The loop was run in bypass mode until initial conductivity of the water was as low as possible, typically around 0.06 μ S/cm. The autoclave was then purged with argon and filled with water. The autoclave was pressurized to approximately 25.1 MPa (3650 psi) using the BPR and heated to 500°C. After each test ran for a specified amount of time, the autoclave temperature was lowered to room temperature and pressure, and subsequently drained and opened. Shortly after being recovered from the autoclave, the corrosion coupon surfaces were photographed with a digital camera to record their appearance.

500 °C Corrosion Results

The high temperature corrosion testing of the model alloys was performed in 500°C steam and in 500°C supercritical water. The alloys tested in 500°C were the same as those tested in 360°C water. Results from the multiple test facilities and test environments revealed common trends in the performance of the model alloys. Details of the weight gain measurements are provided in the appendices with the results summarized in Table 3.1.

It is apparent that alloys with no precipitate-forming alloy additions exhibit the poorest behavior at elevated temperature. These include the Zr-Sn alloys, Zr-0.2Nb, Zr-Sn-Nb alloys, and Zr (sponge and crystal bar). These alloys did not survive the high temperature for even short term exposures. Also included in this group of alloys is Zircaloy-4 which contains the solute Sn as well as small additions of precipitate-forming elements Fe and Cr. These alloys (solute alloys plus Zircaloy-4) are the first ten listed in Table 3.1.

Figure 3.3 shows results from Zr-Sn-Nb that exhibited breakaway and nodular corrosion behavior after relatively short exposure times. Zr-Sn binary alloys were very susceptible to corrosion in 500°C steam and in 500°C supercritical water. The corrosion weight gain increased to more than 2500mg/dm² just after 1 day in 500°C steam and the corroded specimen which was entirely covered with the white oxide was easily broken just by a small force when handle to measure the weight gain.

The remaining model alloys (with the possible exception of Zr-0.4Nb) are the alloys that form second phase particles (SPPs). (Note: Zr-0.4Nb likely contains SPPs due to the presence of iron impurities in the alloy). The corrosion weight gains measured for the Zr-Nb alloys are shown in Figure 3.4. The weight gains are similar for all of the alloys with more than 0.2 Nb, and the oxide appears to be protective for over 240 days in 500°C SCW, although with a reasonably high corrosion rate. The corrosion behavior of Zr-Nb alloys in 500°C steam and supercritical water was approximately the same. The exception to this group was the Zr-0.2Nb alloy which showed breakaway behavior and oxide spalling in the early stages of corrosion in both corrosion environments, showing a weight gain of 1793mg/dm² after 1 day and an oxide that was entirely white. Overall,

the corrosion rate of Zr-Nb alloys in supercritical water was twenty times higher than that in 360°C pure water. The results from KAERI are similar to those from the 500°C steam testing at Westinghouse with poor performance of Zr-0.2Nb and comparable behavior of the higher Nb-content alloys.

Figure 3.5 and Figure 3.6 show the corrosion behavior of Zr-Fe-Cr alloys in 500°C steam and supercritical water. Fe and Cr content were changed to vary the volume fraction of the precipitates. Zr-0.2Fe-0.1Cr and Zr-0.4Fe-0.2Cr alloys were processed at 580°C and 720°C to change the precipitate size. The higher alloy content Zr-0.4Fe-0.2Cr material performed consistently better in steam and SCW among the four test facilities (see Table 3.1). Some variability is noted as Zr-0.2Fe-0.1Fe showed comparable results in steam (KAERI) (Figure 3.5a) and SCW (UM) (Figure 3.6b). However, these alloys (Zr-0.2Fe-0.1Cr) performed poorly in steam (Westinghouse) and SCW (KAERI) after short times regardless of the processing temperature.

Corrosion results for Zr-Cr-Fe alloys are shown in Figure 3.7a (SCW – KAERI) and Figure 3.8 (steam – W, and SCW – UM). The beneficial effect of Fe is clear as seen by comparing the corrosion results of the binary Zr-Cr alloys and Zr-Cr-Fe (see Table 3.1). Zr-Fe-Cr ternary alloys showed better corrosion resistance than the binary Zr-Cr alloys, suggesting that Fe additions improved the corrosion resistance of Zr-Cr binary alloys. (It is possible that the comparable performance of Zr-1.0Cr in SCW (Figure 3.7a) is due to the presence of iron impurities in the material.)

Corrosion results from Zr-Cu-Mo alloys in SCW (KAERI) are shown in Figure 3.7b. These alloys were melted and rolled into strip in ROK. The binary Zr-Cu alloys showed protective behavior throughout the test but with weight gains somewhat higher than the Zr-Cr-Fe alloys. The higher Cu content alloy (Zr-1.0Cu) showed marginally better corrosion resistance than the Zr-0.5Cr alloy, in agreement with the general trend of higher alloying element content being beneficial to corrosion resistance at elevated temperatures. The presence of Mo had a deleterious impact on the corrosion resistance of the material, causing nodular corrosion and increasing the overall weight gain. The Zr-0.5Cu-0.5Mo oxide exhibited non-protective behavior from the start of the test while the Zr-1.0Cu-0.5Mo alloy showed protective behavior, but later developed nodular oxide and increased corrosion rate. Mo was also found to be detrimental to manufacturing the alloys when it was processed at 580°C as the Mo-containing alloys were susceptible to edge cracking of the strip during the cold rolling step. Results from Zr-Cu-Mo alloys that were fabricated in the US showed poorer corrosion behavior for both Zr-Cu and Zr-Cu-Mo (see Table 3.1).

Corrosion tests for the 2004 manufactured alloys (Table 1.2) were also performed in supercritical water. The additional alloys were designed to have a higher alloying content when compared to the original model alloys primarily because the results from the original model alloys indicated that precipitate-forming alloys with higher alloying content showed promise in improving the corrosion resistance in 500°C supercritical water. Results from the new model alloys are shown in Figure 3.9. The additional model alloys did not show better corrosion resistance than the best performing of the

original model alloys. However, all of the new precipitate-forming alloys exhibited better performance than the solute alloys confirming that the presence of precipitates is important in improving the high temperature corrosion performance. It is possible that there is an optimal composition to improve the corrosion resistance in 500°C supercritical water, or the lack of improvement for higher alloying contents could be related to the processing heat treatment.

Table 3.1: Summary of 500°C Corrosion Testing

Alloy	500°C Steam		500°C SCW	
	Westinghouse	KAERI	UM	KAERI
Zr-Sn Alloys				
Zr-0.2Sn	<1 day	1 day	-	1 day
Zr-0.4Sn	<1 day	1 day	-	1 day
Zr-0.8Sn	<1 day	1 day	-	1 day
Zr-1.2Sn	<1 day	1 day	-	1 day
Zr-0.2Nb Alloy				
Zr-0.2Nb	<1 day	1 day	-	1 day
Zr-Sn-Nb Alloys				
Zr-0.4Sn-0.2Nb	<1 day	30d (nodules)	-	6d
Zr-0.4Sn-0.2Nb	<1 day	30d (nodules)	-	6d
Reference Alloys				
Zr (sponge)	<1 day	-	-	1d (spall)
Zr (crystal bar)	<1 day	-	-	1d (spall)
Zircaloy-4	<1 day	-	1d (spall)	1d (2462) ¹
Zr-Nb Alloys				
Zr-0.4Nb	73d (528) ¹	30d (~350) ¹	-	240d (1487) ¹
Zr-1.0Nb	73d (495) ¹	30d (~350) ¹	-	240d (1496) ¹
Zr-1.5Nb	73d (538) ¹	30d (~350) ¹	-	240d (1434) ¹
Zr-2.5Nb	73d (552) ¹	30d (~350) ¹	-	240d (1420) ¹
Zr-Fe-Cr Alloys				
Zr-0.2Fe-0.1Cr L	<1 day	320d (~1100) ¹	-	3d (spall)
Zr-0.2Fe-0.1Cr H	<1 day	320d (~990) ¹	150d (211) ¹	3d (spall)
Zr-0.4Fe-0.2Cr L	129d (172) ¹	320d (~1200) ¹	150d (184) ¹	240d (771) ¹
Zr-0.4Fe-0.2Cr H	129d (340) ¹	320d (~1480) ¹	150d (306) ¹	240d (1220) ¹
Zr-Cr-Fe Alloys				
Zr-0.5Cr	<1 day	-	1d (4282) ¹	3d
Zr-0.5Cr-0.2Fe	76d (spall)	-	150d (204) ¹	240d (507) ¹
Zr-1.0Cr	4 d	-	-	240d (499) ¹
Zr-1.0Cr-0.2Fe	129d (222) ¹	-	150d (423) ¹	240d (344) ¹
Zr-Cu-Mo Alloys				
Zr-0.5Cu	<1 day	-	1d (3970) ¹	240d (700) ¹
Zr-0.5Cu-0.5Mo	<1 day	-	1d (spall)	3d (spall)
Zr-1.0Cu	32d (481) ¹	-	1d (550) ¹	240d (638) ¹
Zr-1.0Cu-0.5Mo	17d (221) ¹	-	1d (576) ¹	96 (spall)

¹ Number in parentheses is weight gain in mg/dm² at the specified time.

Figure 3.3: Corrosion behavior of Zr-Sn-Nb alloys in 500°C steam (KAERI) and supercritical water (KAERI).

Figure 3.4: Corrosion behavior of Zr-Nb alloys in 500°C steam (KAERI) and supercritical water (KAERI).

Figure 3.5: Corrosion behavior of Zr-Fe-Cr alloys in 500°C steam (KAERI) and supercritical water (KAERI).

Figure 3.6: Corrosion behavior of Zr-Fe-Cr alloys in 500°C steam (W) and supercritical water (UM).

Figure 3.7: Corrosion behavior of Zr-Cr-Fe and Zr-Cu-Mo alloys in 500°C supercritical water (KAERI).

Figure 3.8: Corrosion behavior of Zr-Cr-Fe in 500°C steam (W) and supercritical water (UM).

Figure 3.9: Corrosion behavior of new (precipitate-forming) model alloys in supercritical water (KAERI).

Pre-transition kinetic constants

Fitting of the kinetic curves was performed similar to that done at low temperature and the results are shown in Table 3.2. Not much difference is seen in the calculated values of A and n among the different environments for a given alloy. In general the values of A are much higher (30-35) than at 360 °C (which shows values of about 7-10). For the precipitate-based alloys the value of n is close to being cubic ($n=0.33$) and is somewhat higher than the values calculated at 360 °C, which were on the order of 0.2-0.25. In contrast the Zr-Nb alloys exhibit a considerably higher value of n , near 0.66. The ZrFeNb alloy shows an intermediate value of n , at 0.43. Similar tendencies were observed in the calculations of the values of n for low temperature corrosion of the same alloys, i.e., the lowest values of n were for the ZrFeCr and ZrCrFe alloys and the highest for the ZrNb alloys, with intermediate values for mixtures of the two. This suggests that the alloying elements have an effect on the atomic transport in the oxide layer, likely by affecting the concentrations of oxygen vacancies that are responsible for transport of oxidizing species through the layer.

Alloy	Condition	A	n	$\bar{n}$
Zr0.4Fe0.2Cr (L)	SCW dynamic	32	0.34	0.32
	500 °C steam	31	0.29	
Zr-0.4Fe-0.2Cr (H)	SCW dynamic	32	0.36	
	500 °C steam	38	0.25	
	SCW static	35	0.36	
Zr-1.0Cr-0.2Fe	SCW dynamic	36	0.35	0.33
	500 °C steam	30	0.33	
	SCW static	30	0.30	
Zr-0.5Cr-0.2Fe	SCW dynamic	33	0.36	0.33
	500 °C steam	40	0.30	
Zr0.6Fe	500 °C steam	30	0.35	0.33
Zr0.6Fe0.3Cr		26	0.33	
Zr0.6Fe0.3Mo		38	0.30	
Zr1.0Fe	500 °C steam	35	0.33	0.35
Zr1.0Fe0.5Cr		26	0.35	
Zr1.0Cr0.5Fe		24	0.35	
Zr1.0Cr0.5Mo		24	0.36	
Zr0.6Fe0.6Nb	500 °C steam	32	0.43	0.43
Zr-0.4Nb	SCW static	30	0.71	0.67
Zr-1.0Nb		31	0.67	
Zr-1.5Nb		33	0.64	
Zr-2.5Nb		33	0.65	

Table 3.2: Calculated values of A and n for the initial protective stage of the corrosion process.

Task 4: Oxide Characterization

One of the main goals of this project is to discern the microstructural differences of oxides formed in different model alloys under the same conditions. The idea is to relate these microstructure differences to the corrosion kinetics and to the stability of the oxide. To that purpose we performed a systematic characterization of the oxides using a variety of experimental techniques. These observations are summarized in this section, and illustrated by the relevant data.

As mentioned in sections 2 and 3, the model alloys in this work were tested under three different conditions: (i) in water at 360 °C, (ii) in steam at 500 °C (1500 psi) and (iii) in supercritical water at 500 °C (3500 psi). The general procedure was to archive samples at different stages in the corrosion process to be examined. For each model alloy, there were 8 coupons tested at each corrosion condition, and coupons were taken out at set intervals to typify particular stages in the corrosion process. For example, Figure 4.1 shows the weight gain versus exposure time for Zircaloy-4 samples tested in 360°C water. As mentioned in section 2, there is good reproducibility from sample to sample. The red dots indicate where samples were archived. In this case, samples were taken just before (28 and 150 days) and after (180 and 240 days) the oxide kinetic transition, after the second transition at 353 days and so on. The equivalent data for all the alloys in this study is in Appendix A.

Figure 4.1: Oxide weight gain *versus* exposure time for Zircaloy-4 in 360°C water highlighting the samples archived (red points) for oxide characterization.

The main technique used to characterize the oxide layers was synchrotron radiation diffraction and fluorescence, both using cross sectional microbeam and using frontal bulk diffraction. Basic oxide cross-sectional examinations were performed using optical and SEM. Additional characterization was performed using TEM. We summarize these observations in this section.

4.1 Optical examination and SEM examination

General aspect of oxide surfaces

As shown in Figure 2.6 which shows corrosion coupons tested in water at 360°C water (reproduced as Figure 4.2), the clearest difference between the general aspect of oxides formed during protective and non-protective behavior is their color. Generally, protective oxides tend to be black, adherent and shiny, while non-protective oxides tend to be whitish, matt and flaky. At the two extremes, the protective oxides (Zr_{0.4}Fe_{0.2}Cr (H), 14-8 and 14-9) are black and shiny while the non-protective ones are whitish and the oxide tends to flake off upon handling (crystal bar Zr, 42-2 and 42-3). The earlier stages of non-protectiveness in crystal bar Zr are whitish and irregular (samples 42-12 and 42-15), while localized corrosion is seen in sample 34. The samples 21 and 33 show intermediate behavior in the form of grayish or green oxides, indicating somewhat less protective oxides.

Oxides that are semi-protective (i.e. undergo kinetic transition but recover protective character later) show an in-between, grayish appearance. In addition, a few samples showed oxide spallation at the edges and were disregarded in the final analysis (they considered to have fabrication artifacts). It should also be noted that one of the alloys (ZrCr) showed a protective oxide that was light green in color. Finally as illustrated in section 3, a few samples underwent localized corrosion (nodular corrosion) during high temperature testing.

Figure 4.2: Visual appearance of oxide coupon samples formed on various model alloys after corrosion in water at 360°C.

Optical and SEM examinations of Cross sectional Oxides

Metallographic cross sections were prepared to examine the oxide metal interface for oxides formed in model alloys. In general, the non-protective oxides were significantly more cracked than the protective oxides, and their oxide metal interfaces were more irregular. Stable protective oxide growth was associated with an even, homogeneous uncracked oxide. The extreme example of a non-protective oxide is given by the oxides

formed in pure Zr (both sponge and crystal bar). Figure 4.3 shows an oxide formed on sponge Zr before (a) and after (b) the breakaway. The oxide formed before the breakaway is protective, uncracked and homogeneous while the oxide formed after the transition grows unevenly, with oxide “fingers” going into the metal and large scale cracking occurring.

Figure 4.3: Cross sectional scanning electron micrographs of oxides formed on various Zr alloys during corrosion in 360°C water. (a) sponge Zr, seven days exposure (pre-breakaway), (b) crystal bar Zr, 3 days exposure (breakaway), (c) Zr1.0Cu0.5Mo, 150 days exposure (pre-breakaway), (d) Zr1.0Cu0.5Mo, 240 days exposure (breakaway), (e) Zr0.5Cr 146 day exposure after breakaway and (f) Zr0.4Fe0.2Cr (H) 180 days (no breakaway seen).

Figure 4.3 (c) and (d) show alloy Zr1.0Cu0.5Mo which behaved in a protective manner and then lost protectiveness. Large scale cracking is seen after the oxide becomes non-

protective, however the oxide-metal interface is more homogeneous than in 4.3 (b). Finally 4.3 (e) shows the layers of cracks formed on Zircaloy-4 upon several kinetic transitions and Figure 4.3 (f) shows alloy $\text{Zr}_{0.4}\text{Fe}_{0.2}\text{Cr}(\text{H})$ which did not lose protectiveness for the full duration of the test.

As can be seen in Figure 4.3, in general, the oxide metal interface was less wavy and more homogeneous in protective oxides. This suggests that the mechanism of the oxide instability might be related to lateral variations in the growth rate of the oxide. Since the instabilities are very pronounced in pure Zr but diminish with the presence of a small amount of alloying elements, one natural hypothesis is that the alloying elements provide additional conductivity to the oxide layer by the presence of intermetallic precipitates. More importantly, the presence of such precipitates would make oxide conductivity be more laterally homogeneous. In the absence of such precipitates the oxide conductivity would be lower and the transport of electrons that helps close the circuit of the corrosion process will be more difficult. The remnant conductivity will then vary with whatever impurities exist in the oxide, and short circuits could exist that would cause the local corrosion rate to increase.

Zircaloy-4 exhibited marked kinetic transitions as seen in Figure 4.1, the traces of which could be seen in the form of a layer of cracks spaced parallel to the oxide metal interface, which appear with the spacing correspondent to the thickness at which transitions are observed, as seen in Figure 4.4. Other Sn containing alloys also exhibited transitions such that it may be possible to associate marked oxide kinetic transitions with the presence of Sn.

Figure 4.4: (a) Zircaloy-4 after 353 days exposure in 360°C water (two or three transitions) and (b) $\text{Zr}_{0.2}\text{Nb}$ after 60 days in 360°C water.

Similar features were observed in the oxides formed during high temperature testing. Figure 4.5 shows a sequence of cross sectional images of the oxides formed in the best performing model alloys during 500°C supercritical water testing. It is clear that these protective oxides are homogeneous and that the interfaces are smooth. Another feature that can be observed in these pictures is the diffusion layer seen just ahead of the oxide metal interface. This layer is similar to the layer observed during corrosion of zirconium alloys at lower temperature and consists of oxygen dissolved into the Zr matrix, to near the solubility level (about 30 at. %) and forming a sub-oxide phase [1]. The difference

here is that the diffusion (or sub-oxide) layers observed at lower temperature were 0.1 to 0.5 microns thick, whereas these layers are 5 to 6 microns thick. In this case the layers are thick enough that the weight gain has to be corrected for the fact that not all oxygen that goes into the metal forms Zr dioxide [2].

This observation, coupled with others regarding oxide phases present and oxide texture, suggests that the corrosion protection mechanisms are similar at 360°C and at 500°C although the kinetics are faster at the higher temperature.

Figure 4.5. Cross section images of the coupons and their weight gains following (a) 90 days and (b) 150 days of exposure in deaerated SCW at 500°C.

4.2. Overall oxide texture determination using synchrotron radiation diffraction

The oxides that grow on Zr alloys are highly textured. This is because as the oxide grows, stresses can accumulate that can be minimized by proper oxide orientation [3]. In addition there are orientation relationships that cause the oxide to grow with a preferential orientation relative to the base metal.

The experimental procedure is illustrated in Figure 4.6. A 17 keV synchrotron radiation beam was incident on the sample at a glancing angle of 3-4 degrees (4.6a). Diffraction patterns were collected on an image plate, at various values of the sample normal angle (ϕ) by rotating the sample around the normal by 5 degrees from 0 to 200 degrees. The recorded images were integrated along the diffraction rings by 5 degrees sectors, effectively varying along the angle χ (4.6b and d). Integrating along each ring we obtained partial pole figures for the peaks of interest (mostly monoclinic ZrO_2). The pole figure coverage is illustrated in Figure 4.6c, which corresponds to about 0-70 degrees χ and 0-200 degrees ϕ .

In this manner we can *simultaneously* obtain the partial pole figures for the various poles of interest. For the two-theta range we examined, pole figures can be obtained for the following poles: 110_M , 011_M , -111_M , 111_M , 200_M , 020_M , 002_M , -201_M . In the 200_M family of planes the peaks often overlap with each other and with other peaks from metal phases, so care needs to be exercised in the interpretation. Nevertheless, especially for the peaks that stand on their own, pole figures can be obtained for all peaks in the chosen two-theta range, with a single data collection.

This was done for oxides formed on the various alloys. Figure 4.7 shows a set of partial pole figures obtained by the procedure above from an examination of a protective oxide formed on ZIRLO in pure water. Figure 4.8 shows a comparison between the pole figures of three different oxides formed in ZIRLO-protective (4.8a), Zr-0.5Cu-0.5Mo (semi-protective, 4.8b) and sponge Zr (non-protective 4.8c).

Figure 4.6: (a) Schematic experimental setup (b) integration procedure, (c) pole figure coverage and (d) actual diffraction pattern from Zircaloy-4 sample 43-17, collected in the image plate having the 5° integration sectors superimposed.

Figure 4.7 Partial pole figures for various monoclinic ZrO_2 poles, as indicated, obtained from an oxide formed on ZIRLO during corrosion testing in 360°C water

Figure 4.8: Partial pole figures for various monoclinic poles as indicated, for three oxides formed during exposure to 360°C water in (a) ZIRLO (protective oxide) , (b) Zr_{0.5}Cu_{0.5}Mo (semi protective oxide), and (c) sponge Zr (non-protective).

The results show that in many ways the similarities in oxide texture for protective and non-protective oxides are greater than the differences, both at low and at high temperature. By calculating the angle between the maxima of different poles and the normal direction the oxide growth direction has been determined to be between $\bar{2}01_M$ and $\bar{4}01_M$. Previous researchers have shown that the theoretical ideal oxide growth direction (from the point of view of minimizing stresses) is the $\bar{6}01$ monoclinic oxide direction, but that the actual oxides grow in between the $\bar{4}01$ and $\bar{3}01$ directions [4], in agreement with this work.

The degree of oxide orientation in the 200_M direction was measured by calculating the resolved fraction of $[200]_M$ poles along the growth direction of the oxide, (analogously to the Kearns factor in Zr alloys [5]) for various oxides. These values were found to be similar for the various oxides studied (oxides formed on alloys 11, 22, 23, 33, 34, before and after breakaway), so this is not a distinguishing feature of protective and non-protective oxides. For both protective and non-protective oxides, the $\{\bar{1}11\}$ planes show two lobes oriented with their normals (poles) along the transverse x normal plane of the metal, and about 40-45 degrees from the oxide growth direction. The difference is that the $[\bar{1}11]_M$ and $[200]_M$ diffraction lobes exhibit about the same intensity (and are less localized) in the protective oxides and significantly skewed (one lobe stronger than the other) in the non-protective oxides.

This is also true during high temperature corrosion testing. Figure 4.9 shows three partial pole figures obtained using synchrotron radiation diffraction for two oxides formed at high temperature, one protective and one non-protective oxide. The two oxides are formed on Zr0.5Cr in 500°C steam and Zr1.0Cr0.2Fe in supercritical water. The latter forms a protective oxide while the former forms a non-protective oxide.

The figures above show that the oxides form with the growth direction aligned close to $[\bar{2}01]_M$ (and thus close to $\bar{3}01$), as the intensities of the $[\bar{2}01]_M$ pole figure are at a maximum close to the normal direction (i.e. the growth direction). The intensities of the $[\bar{1}11]_M$ poles are mostly aligned in the TN plane, about 40-45 degrees from the normal (more so for the Zr0.5Cr non-protective oxide than for the Zr1.0Cr0.2Fe protective oxide). This results from the previously determined orientation relationship $[200]_M // [0002]_{hcp}$ and $[020]_M // [10\bar{1}0]_{hcp}$ [1].

It was a common observation in the non-protective oxides that one of the $[\bar{1}11]_M$ lobes was systematically stronger than the other. This may indicate that during non-protective oxide growth, the balance between the possible variants of growth that obey the orientation relationship above is perturbed, so that colonies of grains in one orientation dominate. A corresponding imbalance is seen in the $[200]_M$ peak, which in the top left part of Figure 4.9 shows a higher intensity to the left of normal (whereas the $[\bar{1}11]_M$ is right of the normal). In contrast, in the protective oxide, the $[\bar{1}11]_M$ intensities are less concentrated in the TN plane and are more evenly distributed in the circumferential direction. Whether the unequal distribution of intensities between possible $[\bar{1}11]_M$ orientations is a cause or an effect of the oxide lack of protectiveness is open to question.

Figure 4.9: Pole Figures for the ZrO_2 monoclinic peaks indicated, and obtained as averaged over the oxide layer, in frontal geometry for two oxides, one non-protective (Zr0.5Cr) and one protective (Zr1.0Fe0.2Cr). These figures are normalized such that the level “1” corresponds to the average intensity.

4.3 Oxide structure studied using microbeam synchrotron radiation diffraction and fluorescence

To study in greater detail the phases formed, grain size, local texture (oxide orientation), and the structure of the oxide-metal interface, we utilized microbeam synchrotron radiation diffraction and fluorescence. This technique has been applied by our group to study the structure of metals and oxides[1, 2, 6-11].

Experiments were performed at the Advanced Photon Source (APS) at Argonne National Laboratory. The experimental setup is shown in Figure 4.10.

Figure 4.10: Schematic drawing showing the acquisition geometry for microbeam synchrotron radiation diffraction and fluorescence.

The beamline (2ID-D) utilized is equipped with zone plate diffraction gratings that can produce a monochromatic $0.2 \mu\text{m} \times 0.3 \mu\text{m}$ X-ray beam with a photon flux of 5×10^9 photons/s and an X-ray energy band width (dE/E) of 0.01 % [12]. The beam that was used during the experiments was focused to a size of $0.25 \mu\text{m}$ in the vertical direction (normal to the oxide-metal interface) so that it had a $2 \mu\text{m}$ footprint in the horizontal direction (parallel to the oxide-metal interface). The beam energy was 9.5 keV, corresponding to a wavelength of 0.1305 nm. Diffraction and fluorescence data were simultaneously collected from the sample as it was translated across the beam in step sizes ranging from 0.15–0.50 μm . The diffraction data were collected by a flat two-dimensional detector (CCD camera), while the fluorescence data were collected by an energy dispersive Ge (Li) detector with an energy resolution of 200 eV.

The positions of the oxide-metal and oxide-water interfaces were determined by monitoring Zr L line fluorescence counts as the sample was translated across the beam. The lower density of zirconium atoms in the oxide than in the metal results in a slight reduction in Zr L intensity as

the micro-beam is moved from the metal to the oxide. This allowed the determination of the oxide location. The distance between the oxide-metal interface and the oxide-water interface measured by this method was in good agreement, to within 0.5 μm , with optical determination of the oxide thickness. Figure 4.11 shows two of these fluorescence plots across the oxide layer.

Figure 4.11: X-ray fluorescence plots obtained as a function of distance from the oxide metal interface for oxides formed on (a) Zircaloy-4 and (b) Zr1.0Cu0.5Mo in 360°C water.

Both plots show the concentrations of the alloying elements of interest (Zr, Fe, Cu and Sn). In each case the pink squares represent the concentrations of Zr. The oxide layer is noticeable because the Zr counts diminish to a lower plateau at the place marked oxide-metal interface and to zero at the oxide-water interface. The first plot, for Zircaloy-4, shows a protective oxide, with an even Zr plateau decreasing at the water interface. The thickness measured (about 4 microns) corresponds well to the weight gain measured. It is noticeable also that the Sn profile is constant

both in the oxide and in the metal, so there is no segregation of Sn to either interface. For Fe, in Zircaloy-4 there are $\text{Zr}(\text{Cr},\text{Fe})_2$ intermetallic precipitates which can be seen in the profile (places where both Fe and Cr increase). In contrast the oxide shown in (b) is from a non-protective oxide and it shows diminution in Zr counts related to the presence of cracks. It should be noted that the epoxy used contains Cu, which shows very prominently in the plots, since the energy is just above the Cu edge. Because the epoxy gets into the cracks, high Cu counts are seen there. Further, although the alloy contains no Fe it shows an iron concentration from the autoclave. The Fe profile is augmented all through the oxide layer, indicating that it is porous and non-protective.

The close match between the fluorescence plots and the thickness of the oxide layers measured by microscopy gives confidence that the oxide locations examined in microbeam synchrotron radiation correspond to the actual values.

Processing and Integration of Diffraction Data

Diffraction patterns are acquired at each position as the beam is scanned across the oxide cross section. A typical diffraction pattern from powder zirconia is shown in Figure 4.12. The standards were used to align the beam and make a one-to-one correspondence between location in CCD and two-theta angle.

Figure 4.12: Diffraction pattern obtained from powder monoclinic ZrO_2 standard.

To obtain quantitative diffraction data, the digital data obtained from the two-dimensional detector were integrated over the elliptical sections for a fixed azimuthal angle, and the appropriate Lorentz and polarization correction factors were applied [6]. Following the integration and above corrections, a plot of diffracted intensity versus two-theta angle was obtained at each location analyzed. Figure 4.13 shows a plot of diffracted intensity versus two-theta angle for the monoclinic ZrO_2 standard shown in Figure 4.12. As shown in Table 4.1, the calculated peak positions for the monoclinic planes agree quite well with the literature values (within a few hundredths of degree).

Figure 4.13: Plot of intensity versus two theta angle corresponding to the integration of the diffraction pattern shown in Figure 4.12.

Table 4.1: Comparison of observed peak positions with literature values for powder monoclinic ZrO_2 .

<i>peaks</i>	<i>Observed (degrees 2-theta)</i>	<i>PDF 37-1484 (2-theta degrees)</i>	<i>Difference (2-theta degrees)</i>
m(110)	20.344	20.327	0.017
m(011)	20.686	20.660	0.026
m(-111)	23.802	23.797	0.005
m(111)	26.544	26.558	-0.014
m(200)	28.768	28.811	-0.043
m(020)	29.071	29.003	0.068
m(002)	29.768	29.773	-0.005
m(-201)	30.246	30.276	0.030
m(102)	32.486	32.471	0.015

It is possible then to scan the beam across the oxide layer, and by stacking the plots such as shown in Figure 4.13, to generate intensity versus two theta angle plots as a function of distance for the oxide layers generated in this study. The peaks were indexed and used to identify the various different phases present in the oxide, as discussed below. Figure 4.14 shows an example of such a plot, generated for an oxide formed on $\text{Zr}_{1.5}\text{Nb}$ in pure water.

Figure 4.14: Diffraction intensity versus two theta diffraction angle versus position in the oxide layer for an oxide formed on Zr1.5Nb, in 360°C water. The monoclinic ZrO_2 peaks are in red, the bulk ZrO_2 tetragonal 101 peak is in green, the suboxide peaks in blue and the interfacial $[002]_T$ peak in yellow.

The plot shows three distinct regions: the metal, the oxide-metal interface and the oxide. Peaks characteristic of hcp Zr are visible in the metal with very high intensities, while in the oxide peaks associated with monoclinic ZrO_2 and one peak of tetragonal ZrO_2 are observed. In the interface region, additional peaks are seen that are associated with the interfacial tetragonal phase discussed below and suboxide peaks.

In this way the diffraction and fluorescence information that can be obtained from these plots can be compared from (i) alloy to alloy, (ii) within the same alloy at different stages of the corrosion process and (iii) at 360 C and 500 C. These features are discussed in the next few sections, and in context of previous observations performed on commercial Zr alloys. The information obtained is described in the following.

4.3.1. Phases present in the bulk of the oxides and overall orientation

As also seen in the frontal diffraction texture studies, the structures of oxides were quite similar, from alloy to alloy and at the two test temperatures. However, there were some important differences that distinguish protective and non-protective oxide layers, especially at the oxide-metal interface.

For all the oxides studied, the dominant oxide phase in the bulk of the oxide (away from the oxide-metal interfaces) was monoclinic ZrO_2 , with small amounts of tetragonal phase. As seen in section 4.2, the oxide layers were heavily textured. The monoclinic oxide peak intensities were far from the random powder texture. In particular the $[200]_M$ peak intensity was almost invariably low, which is consistent with the previously determined oxide growth orientation near the $\bar{3}01_M$ direction.

The differences in oxide structure for protective and on-protective oxides were notable and consistent, especially at the oxide-metal interface. These differences will be described in the following sections. The differences in oxide structure between oxides formed at 360°C and at 500°C were mostly of degree, that is, the structures of protective oxides formed at low and high temperature were similar.

Structure of the oxide-metal interface

The oxide-metal interface is of great interest to understanding the corrosion process since this is where new oxide gets formed, and where the barrier or dense layer of the oxide is located, which controls the corrosion rate. Changes to the microstructure of the oxide-metal interface will affect the overall corrosion process.

As seen previously, the oxide-metal interface is different from the bulk of the oxide and from the metal, in various respects, especially on the protective oxides. On the metal side, a sub-oxide region is detected that extends to variable extents into the metal. This sub-oxide layer is detected by metallographic examination, especially for oxides formed at high temperature and by energy dispersive x-ray spectroscopy in the SEM. It is also detected by characteristic sub-oxide diffraction peaks observed as shoulders on the metal peaks, and which are only present in the region in the metal next to the oxide-metal interface.

In addition, the overall percentage of the tetragonal ZrO₂ phase in the oxide is higher at the oxide metal interface than in the bulk of the oxide. Other researchers have indicated that in the first stages of oxidation the first oxide observed is the tetragonal oxide later transforming to monoclinic [13], and in previous research papers we have indicated that during the advancement of the oxide in the protective regime, the new oxide is formed first as tetragonal grains which later transform to monoclinic when their size reaches 20-30 nm [1].

These observations were confirmed in the present study. Figure 4.15 shows the tetragonal fraction for various model alloys in this study, calculated according to the Garvie Nicholson formula [14, 15]:

$$f_T = \frac{I_{101_r}}{I_{111_M} + I_{101_r} + I_{\bar{1}11_M}} \quad (4.1)$$

where the I_{hkl} are the measured intensities of the corresponding tetragonal and monoclinic peaks. The calculation takes into account the oxide texture as described in [1].

Figure 4.15: Tetragonal Fraction for oxide layers formed on various model alloys as a function of distance from the oxide metal interface.

Another remarkable observation is that some of the tetragonal phase formed at the interface (as distinct from the tetragonal phase that forms in the bulk) has a strong orientation relationship both with the metal and with the subsequent monoclinic oxide that forms away from the oxide metal interface. This has been previously observed and dubbed a highly oriented tetragonal phase, and it has been suggested that it is a precursor phase to the monoclinic phase that forms subsequently in the oxide [6, 7, 16]. The presence of this phase correlates almost one to one with a protective character in the oxide, that is, the highly oriented tetragonal phase is present when the oxide is protective and it is not present when the oxide is non protective. The diffraction patterns from the interface of protective oxides exhibit diffraction intensity corresponding to this phase, as shown by arrows in Figure 4.16.

Another observation is that the $[002]_T$ peak intensities often appears with mottled contrast, indicating the presence of strain at the interface.

Away from the oxide-metal interface the orientation of the $[020]_M$ peak is more random than at the oxide metal interface, indicating that the columns lose a little of the alignment that they have in the newly formed oxide. This is shown in Figure 4.16 which shows the diffraction patterns from various oxides, taken a few microns away from the oxide-metal interface.

The diffraction patterns taken at regular intervals starting at the metal, across the oxide-metal interface until the oxide-water interface can be stacked up and plotted together as done in Fig.4.14 to show the variation of phase content and oxide orientation within the oxide layer. Figures 4.17 to 4-22 show four examples of these plots (intensity versus two theta angle versus distance to the oxide-metal interface), taken from four different oxide layers, both protective and non-protective.

	Oxide-metal interface	3 microns away
<p>Zr0.5Cr</p> <p>(90 days in 360°C water, weight gain 955.9 mg/dm²)</p>		
<p>Zr1.5Nb</p>		
<p>Zr0.2Fe0.1Cr (L)</p> <p>(415 days in 360°C water, weight gain 55.0 mg/dm²)</p>		
<p>Zr1.0Cr0.2Fe</p> <p>(150 days in 500°C supercritical water, weight gain mg/dm²)</p>		

Figure 4.16: Diffraction patterns from the oxide-metal interface, taken in cross section in the rolling-normal plane (transverse direction perpendicular to the sample surface) for samples and exposures indicated. A yellow outline shows the intensity from the [002]_T plane of the interfacial tetragonal phase.

Figure 4.17: Diffraction intensity versus two theta angle versus position in the oxide for alloy 24 ($Zr_{1.0}Cr_{0.2}Fe$) corroded in 500°C supercritical water for 150 days.

Figure 4.18: Diffraction intensity versus two theta angle versus position in the oxide for alloy $Zr_{0.4}Nb$ corroded in 500°C supercritical water for 300 days.

Figure 4.19: Diffraction intensity versus two theta angle versus position in the oxide for alloy 14 ($\text{Zr}_{0.4}\text{Fe}_{0.2}\text{Cr}$ (H)) corroded in 500°C supercritical water for 150 days.

Figure 4.20: Diffraction intensity versus two theta angle versus position in the oxide for alloy 33 ($\text{Zr}_{1.0}\text{Cu}$) corroded in 360°C water for 409 days.

Figure 4.21: Diffraction intensity versus two theta angle versus position in the oxide for alloy 21 (Zr0.5Cr) corroded in 360°C water for 90 days.

Figure 4.22: Diffraction intensity versus two theta angle versus position in the oxide for alloy 21 (Zr0.5Cr) corroded in 500°C steam.

The main differences between these series of diffraction patterns are at the oxide-metal interface. The protective oxides show the $[002]_T$ peak and the sub-oxide peaks. The $[002]_T$ peak is arrowed at the interface in the protective oxide formed on alloy Zr1.0Cu at 360 C (Figure 4.20). In the protective oxides formed at high temperature this peak is much higher as shown in Figure 4.17. The arrowed peak is very high, indicating a large volume of this phase at the interface.

Figure 4.17 shows the diffraction patterns from an oxide layer formed on alloy 24 after 150 days in supercritical water at 500°C. The oxide metal interface is at position zero, and in the negative positions, the metal peaks are indicated, with suboxide peaks indicated as “S”. In the oxide the monoclinic oxide peaks are visible. At the interface a very large peak, indicated as $[002]_T$ is shown. This peak was visible in the protective oxides seen at high temperature and absent in the oxides that were non-protective. As seen previously, the tetragonal peak is observed just ahead of the full oxide-metal interface, in the region near where the new oxide is forming. In contrast, Figure 4.18, taken from an oxide in alloy Zr0.4Nb exposed to 500°C supercritical water for 300 days shows an interface in which this peak is absent. Figure 4.19 shows an oxide layer formed on Zr0.4Fe0.2Cr (H) when exposed to 500°C supercritical water, exhibiting the a similar peak at the interface.

At lower temperature the same peak appears associated with protective oxides but is lower in intensity. Figure 4.20 shows the diffraction patterns associated with a protective oxide layer formed during exposure to 360°C water in alloy Zr1.0Cu. The peak (arrowed) is also present at the interface, but at a much lower intensity than at high temperature. Figures 4.21 and 4.22 shows two non-protective oxides generated by exposure to 360°C and 500°C respectively, in which the interfacial tetragonal phase is not present.

One possible conclusion from these observations is that the tetragonal phase formed at the interface may help align the oxide in a manner that helps minimize stresses during its growth. As such, this tetragonal phase detected at the interface would be highly aligned and may serve as a precursor phase that helps the oxide form in a more aligned fashion. It is given that to minimize stresses the oxide growth direction is near $(-301)_M$, which means that the $[020]_M$ and $[002]_M$ planes are aligned with their poles nearly normal to the oxide growth direction. It appears that a close alignment of the $[020]_M$ plane with the $10\bar{1}0_{hcp}$ plane is more favorable for stable oxide growth than a random in plane-texture of these planes, evenly distributed between the rolling and transverse directions. Because the $[002]_T$ plane is parallel to the $10\bar{1}0_{hcp}$ peak and the $[020]_M$ plane of the monoclinic phase forms parallel to the $[002]_T$ plane of the tetragonal phase, the alignment is achieved when the highly oriented tetragonal phase is present. This can also be verified in Figure 4.16 which shows that there is close alignment of the $[002]_T$ planes and the $[020]_M$ intensities along the diffraction arcs in the diffraction patterns taken close to the oxide-metal interface. This is expressed as:

$$[10\bar{1}0]_{hcp} // [002]_T // [020]_M$$

This orientation is shown schematically in Figure 4.23.

Figure 4.23: Orientation relationship of the hcp Zr metal planes and the tetragonal and monoclinic oxide planes near the oxide-metal interface in the protective oxides. R is rolling direction of the metal sheet, N is the normal direction (parallel to the oxide growth direction) and T is the transverse direction.

The interfacial tetragonal phase in the protective oxides is thus thought to induce a favorable in plane texture in which the $[020]_M$ direction of the oxide is preferentially aligned with the $10\bar{1}0_{hcp}$ planes in the metal. This alignment resulting from the interfacial tetragonal phase results in a higher ratio

$$\gamma = \frac{I_{020_M}}{I_{002_M}} \quad (4.2)$$

of diffracted intensities of the $[020]_M$ peak and the $[002]_M$ peak. This ratio was calculated for various oxides of interest and is plotted in Figure 4.24. It is clear that the ratio is higher in the protective oxides than in the non-protective oxides.

Figure 4.24: Ratio of intensities of the $[020]_M$ and $[002]_M$ oxide planes when examined in the rolling –normal sample plane (transverse direction perpendicular to surface). P indicates a protective oxide, SP semi-protective and NP non-protective.

Overall Tetragonal phase fraction level

The tetragonal phase fraction has been previously linked to corrosion behavior in Zr alloys [17, 18]. It is difficult however to separate cause and effect in this case. In previous research, we have shown that an increased tetragonal fraction did not correlate necessarily with best corrosion behavior [6]. Protective oxides of the same alloy will have higher tetragonal fraction before breakaway than after breakaway, however from alloy to alloy higher tetragonal fraction does not increase protectiveness. This trend was confirmed here. The maximum tetragonal phase fraction calculated using Equation 4.1 and corrected for texture as in [6].

The tetragonal phase fraction varied from 2% to 15-20%, and for most oxide layers, it fell between 4-8%, as seen in figure 4.25. No clear correlation was seen between protectiveness of the oxide and the overall level of tetragonal phase in the oxide.

Figure 4.24: Average tetragonal fraction for various oxides formed during low and high temperature corrosion testing.

Monoclinic Grain sizes in oxides

For a given testing temperature the grain sizes calculated from the Scherer equation are similar for the oxides studied.

For oxides formed at 360°C, the tetragonal grain sizes (calculated from the broadening of the $[101]_T$ peak) are 7-10 nm and the monoclinic grains calculated either from the broadening of either the $[-111]_M$ or $[-111]_M$ peak are about 20-25 nm, as shown in Fig.4.26. The bulk of the oxide shows a more or less even grain size whereas the only variations are near the oxide-metal interfaces the calculations have greater associated uncertainties (smaller peaks, confounding metal peaks, etc.). The monoclinic grains in the alloys that contain Sn and Nb in solid solution (Zircaloy, ZrSn, ZrNb) are possibly a little smaller (17-20 nm).

Occasionally there are variations in the grain size from point to point and these are normally associated with large single grains that show up on the diffraction pattern as bright spots. It is possible that these spots are artifacts coming from previously undetected impurity phases on the surface oxide sample, or that occasionally there are large grains in the oxide amidst the smaller ones. Regardless, the overall size of the oxide grain population is close to what is shown above. For oxides formed at 500°C the monoclinic grain sizes appear to be a little bigger (about 30-35 nm from the broadening of the $[-111]_M$ peak) and about the same size for the tetragonal as the lower temperature (10-12 nm), as shown in Fig.4.27. This is consistent with the tetragonal grains transforming to monoclinic after a certain size and not growing within the oxide.

These grain sizes are in general agreement with previous determinations of grain size in oxides in commercial alloys using the synchrotron radiation.

Figure 4.26: Average grain sizes calculated according to the Debye Scherrer formula for in various oxide layers formed during 360°C corrosion testing.

Figure 4.26: Average grain sizes calculated according to the Debye Scherrer formula for in various oxide layers formed during 500°C corrosion testing.

Microstructure characterization of the oxide by TEM

The oxide growth mechanism on Zr alloys is affected by the various factors such as microstructure, crystallographic structure, oxide texture and second phase particles embedded in oxide. TEM has been considered to be a proper technique to obtain the oxide grain size and shape, crystal structure of the grains, and the characteristics of the second phase particles. The objective of TEM observation for the oxide is to assist in the interpretation of the oxide growth mechanism of the model alloys with different alloying element in different corrosion environments.

The sample preparation of the cross-sectional oxide sample was carried out according to the following procedure. (1) Two segments with about 3 mm width were cut from the corroded samples and then glued together on the corroded surfaces. (2) This piece was inserted into a stainless steel tube with 4mm inner diameter using the G1 epoxy-type glue and then cured at 100C for 60 minutes. (3) The cured tube was sliced into the 0.3 mm thick disk by a low speed cutter. (4) The disk samples were ground from 1200 grit abrasive paper to 0.5 μm diamond cloth on both sides until the thickness was thinned to approximately 10 ± 2 μm . (5) The polished samples were glued on to the Cu grid and then finally thinned by ion milling. The prepared samples were observed using the 200 keV TEM (JEOL FX 2) in KAERI.

Figure 4.28 shows the general morphology of the two oxides on Zr-0.4Nb corroded to 90 and 300 days in 360C water, respectively. The columnar oxide grains grow in the perpendicular direction to the oxide/metal interface in the thin as well as thick oxides. Several lateral cracks were observed in the thick oxide layer, and the equiaxed grains were largely formed on the area of outer water/oxide and near the lateral cracks in the thick oxide as shown in Fig. 28 (b). It was suggested that the lateral cracks and the equiaxed grains were easily formed with increasing the oxide thickness.

Figure 4.29 shows TEM microstructures of the oxides on Zr-0.4Nb, Zr-1.5Nb and Zr-1.0Cr-0.2Fe alloys corroded to 60 days in 500C supercritical water. Since the corrosion rate in the supercritical water is much higher than that 360C water, the oxide thickness of two Nb-containing Zr alloys was thicker than 30 μm just after 60 days. At the equal corrosion time, the oxide thickness of Zr-1.5Nb alloy was thinner than that of Zr-0.4Nb alloy. Both columnar and equiaxed oxide grains were observed in the two Nb-containing Zr alloys formed. The overall oxide morphology characteristics were shown to be similar both in 360C water and 500C supercritical water. The area of columnar oxide grains of Zr-1.5Nb alloy was higher than that of Zr-0.4Nb alloy at the same corrosion time of 60 days. In both alloys, the lateral cracks in the oxides were randomly distributed in the oxide layer and the equiaxed grains were observed more frequently near the cracks. On the other hand, the oxide thickness of Zr-1.0Cr-0.2Fe alloy was very thinner than those of Nb-containing alloy at the equal exposure time. The area of columnar oxide grains of Zr-1.0Cr-0.2Fe alloy was higher than that of columnar oxide grains of Zr-1.5Nb alloy and the lateral cracks were not observed in the oxide formed on Zr-1.0Cr-0.2Fe alloy.

Figure 4.30 shows the characteristics of the oxide on Zr-1.5Nb alloy corroded in 500C supercritical water more precisely. The columnar structure grains were well developed in the inner area of the oxide and the columnar and small equiaxed grains were mixed in the outer area of the oxide. From the selected area diffraction pattern analysis, it was found that the (200) or (020) monoclinic ZrO_2 reflections were matched with the some columnar oxide grains in the oxide. In the diffraction pattern taken from inner area region of oxide, the (200) monoclinic reflections were stronger and almost well oriented. However, in the diffraction pattern taken from the middle area in oxide, the (200) monoclinic reflections are weaker and the angle between (200) and (020) monoclinic ZrO_2 are larger than in case of the inner area region. In this case, the columnar grains were transformed to the small equiaxed grains. It was assumed that columnar structure was stabilized by the well aligned oxide orientation of (200) and (020) monoclinic ZrO_2 phase.

The characteristics of the second phase particle incorporated into the oxide of Zr-1.5Nb alloy corroded for 60 days in supercritical water was shown in Figure 4.31. At the low magnification image shown in Figure 4.31 (a), lateral cracks were randomly distributed in the oxide. From the results of EDS and SAD patterns of the incorporated second phase particles in the oxide, the incorporated second phase particles in the oxide were revealed as the amorphous structure type even at the position more than 28 μm away from the metal/oxide interface. This type of incorporated second phase particles was thought to be resulted from the beta-Nb contained in Zr-1.5Nb alloy before corrosion test. Since the lateral cracks were not observed near the oxidized second phase particles, it was thought that the crack formation was not affected by the late oxidation of the incorporated beta-Nb.

From the TEM observation on the various oxides formed on model alloys, the corrosion resistance of the model alloys was closely related to the oxide morphology. The fraction of columnar grains in oxides was increased in the model alloys showing the higher corrosion resistance both in 360C water and in 500C supercritical water. The stability of the columnar grain was closely related to the texture of the oxide: The strong orientation of monoclinic (200) and (020) was well matched with the columnar grains. The role of second phase particles during the corrosion was not clear because the correlation was not obtained between the incorporated second phase particles and the oxide morphology characteristics such as lateral cracks and columnar grains.

Figure 4.28: TEM microstructures of the oxides on Zr-0.4Nb corroded up to oxide thickness of (a) 2.2 mm and (b) 4.8 mm in 360C water.

Figure 4.29: TEM microstructures of the oxides on (a) Zr-0.4Nb, (b) Zr-1.5Nb and (c) Zr-1.0Cr-0.2Fe corroded up to 60 days in 500C supercritical water.

Figure: 4.30: Oxide characteristics of Zr-1.5Nb corroded in 500C supercritical water.

(a) Low magnification bright field

(b) Precipitate in oxide

Figure 4.31: The characteristics of the second phase particle incorporated into the oxide of Zr-1.5Nb alloy corroded for 60 days in supercritical water.

Nano-indentation on the oxide

Nano-indentation test on the oxide was conducted to investigate the mechanical properties of the oxide in the model alloys more quantitatively. The objective of the nano-indentation test was to obtain the oxide property such as the nano-hardness, the elastic modulus along the oxide thickness and evaluate the oxide stability in terms of the mechanical properties of the oxide.

The model alloys tested were Zr-0.4Nb, Zr-1.5Nb, Zr-1.0Cr, and Zr-1.0Cr-0.2Fe corroded in 360°C water and in 500°C supercritical water. The specimens after autoclave testing were sectioned by the low speed cutter, mounted perpendicular to the sectioned plane, and ground with up to 4000 grit abrasive paper. In order to minimize scratches caused by the spalled oxide during grinding, the specimens were ground in parallel direction with oxide/metal interface to reduce the scratches on the oxide by the oxide debris. After grinding, the specimens were finally polished by 0.05 µm alumina cloth to reduce the surface roughness prior to the nano-indentation. Nano-indentation test was performed at the MTS nano-indenter XP model which has a displacement resolution of 0.01 nm, a maximum indentation depth of 500 µm, and a maximum load limit of 500 mN. Berkovich-type indenter was used in this study. The specimen was indented then unloaded to obtain load-displacement curve. 20 points per specimen were indented along the inclined direction to the oxide/metal interface with an interval of 50 µm. The oxide modulus and the nano-hardness were measured at each point by the load-displacement curve. As the oxide thickness of the model alloy after corrosion was too thin to measure exact value at each local point, nano-hardness and oxide modulus averaged at each local point were used in this study, except for the specimen exposed at the supercritical water in which oxide was thick enough to obtain local values.

Figure 4.32 shows the effect of Nb contents on the average nano-hardness property and the weight gain in 360°C water. Weight gain increased as the exposure time elapsed. On the other hand, little effect of Nb addition appeared in case of 360°C water corrosion. The effect of both the alloying element and the water chemistry on the oxide stability of Zr-Cr-Fe alloy is shown in Figure 4.33. Significant increase of the oxide hardness was observed when 0.2 wt% of Fe is added. Corrosion notably decreased from 277 mg/dm² in Zr-1.0Cr to 62 mg/dm² in Zr-1.0Cr-0.2Fe alloy after 390 days corrosion in 360°C water. This indicates that oxide stability was greatly increased even by small amount of Fe addition to result in the increase of corrosion resistance. In terms of environment effect, however, negligible change of the nano-hardness appeared regardless of the water chemistry such that the weight gain increased at the supercritical water compared to the 360°C water environment.

The local nano-hardness profile across the oxide thickness was obtained for the thick oxide on both Zr-0.4Nb and Zr-1.5Nb corroded in supercritical water as shown in Figure 4.34. The nano-hardness value of the metal part is approximately 3 GPa. On the other hand, oxide nano-hardness value was in the range of 8 to 14 GPa. Nano-hardness changed so abruptly that one can easily find the interface between the oxide and the metal. Although some points showed anomalous value at which the tip of the indenter is assumed to be trapped by a circumferential or lateral crack, the profiles of nano-hardness generally decrease within 10 µm then come close to the stable value. Nano-hardness value at the outer side of oxide was 8 GPa in Zr-0.4Nb alloy, whereas 11 GPa in Zr-1.5Nb alloy. Their weight gain was 582 mg/dm² in Zr-0.4Nb alloy, 483

mg/dm² in Zr-1.5Nb alloy after 60 days corrosion in supercritical water. The weight gain as well as the related oxide thickness was reduced as the Nb content increased. The slope of nano-hardness was more rapid in Zr-0.4Nb alloy than Zr-1.5Nb one. In other word, the local nano-hardness distribution across the oxide thickness was more inhomogeneous in Zr-0.4Nb than Zr-1.5Nb. Although nano-hardness value at the oxide-metal interface was similar in both alloys, the changes of nano-hardness were abrupt in Zr-0.4Nb alloys such that the nano-hardness at the outer oxide in Zr-0.4Nb was smaller than in Zr-1.5Nb alloy.

Figure 4.32: The effect of Nb content on the average nano-hardness of the oxide formed in 360C water.

Figure 4.33: The effect of Fe addition on the average nano-hardness

Figure 4.34: Local nano-hardness profile of the oxide on (a) Zr-0.4Nb and (b) Zr-1.5Nb corroded up to 60 days in supercritical water.

Electrochemical Impedance Spectroscopy on the oxide

In general, Zr oxide consisted of the porous non-protective outer layer and the impervious protective inner layer. The penetration of the electrolyte with oxygen ion through the open pore or crack of the oxide layer reduce the electrical resistance of the oxide layer and is one of the causes for the accelerated corrosion. Electrochemical impedance spectroscopy (EIS) is suitable method to evaluate these properties without destruction of the oxide. The oxides on the model alloys were analyzed by EIS to evaluate the oxide properties and to correlate with the corrosion properties of the alloys.

EIS measurement were carried out using an impedance spectrum analyzer (IM6, Zahner electric, Germany) which was connected to three-electrode electrochemical cell (K0235 Flat Cell, EG&G PAR). The EIS spectra were obtained at open-circuit potential (OCP) of the working electrode (Zr oxide) in 0.01 M H_2SO_4 + 0.1 M K_2SO_4 solution at 25°C. A platinum grid was used as a counter electrode and a saturated calomel electrode (SCE) connected to the cell by a salt bridge served as a referenced electrode. AC signal was scanned from 5×10^6 Hz to 5×10^{-4} Hz with a amplitude of 10 mV. Before the EIS measurement, the oxide was soaked in a test solution as an electrode for 0.5, 1, 2 and 3 days. Zr-0.4Nb, Zr-1.5Nb and Zr-1.0Cr-0.2Fe alloys corroded in supercritical water were selected for EIS. The thin and thick oxide were sampled at 6 day and 60 days and tested by EIS. Figure 4.35 shows Bode plots with soaking time for the thick oxide of Zr-0.4Nb corroded up to 60 days in supercritical water. Impedance and phase angle lower than 10 Hz changed significantly with soaking time which means the electrolyte penetrated into the pores or cracks of the oxide layer. However, such a tendency was not observed in the thin oxide, which means the thin oxide has more protective nature as compared to the thick oxide.

Figure 4.36 shows Bode plots of 3 day-soaked thin and thick oxide of Zr-0.4Nb corroded in supercritical water. The oxide thickness of the thin and thick oxide calculated from the weight gain was 3.8 and 38.8 μm , respectively. EIS spectrum for the thin oxide shows higher electrical resistance (impedance) and higher phase angle at low frequency (<10 Hz) when compared to the thick oxide, which means the thin oxide has a higher protectiveness than the thick oxide. From the observation on the cross-section of the oxide, the vertical crack penetrated from the surface to about 90% thickness was found along with many small pores. The thick oxide could be interpreted as a double layer structure and its low electrical resistance would be caused by the fact that the thick outer oxide has many open pore and/or crack.

Figure 4.37 shows the EIS spectra of Zr-1.5Nb and Zr-1.0Cr-0.2Fe alloys corroded in supercritical water condition for 60 days. EIS spectra of the Zr-1.0Cr-0.2Fe alloy shows higher electrical resistance and higher phase angle at low frequency (<10 Hz) than that of Zr-1.5Nb alloy. Especially, phase angle of Zr-1.0Cr-0.2Fe alloy has about 90 in a wide range of frequency ($10^{-1} < f < 10^4$ Hz) which means the oxide layer has capacitive properties and high protectiveness. Table 5 summarizes the oxide thickness, electrical resistance and specific resistance. The specific resistance of the thin oxide was orders of magnitude higher than that of thick oxide. And Zr-1.0Cr-0.2Fe alloy has higher specific resistance than that of Nb-containing Zr alloys. The corrosion rate of Zr-1.0Cr-0.2Fe alloy was very lower than Nb-containing Zr alloys in supercritical water. It implies that the specific resistance of the oxide was closely related with the corrosion rate of the alloy.

Table 4.2: Oxide thickness and specific resistance of Zr alloys corroded in supercritical water

<i>Alloy</i>	<i>Corrosion time (days)</i>	<i>Oxide thickness (μm)</i>	<i>Electrical resistance (Ω)</i>	<i>Specific resistance (Ωcm)</i>
Zr-0.4Nb (thin)	6	3.8	1.50E+09	3.95E+05
Zr-0.4Nb (thick)	60	38.8	1.30E+05	3.35E+00
Zr-1.5Nb	60	32.2	2.60E+09	8.07E+04
Zr-1.0Cr-0.2Fe	60	10.1	5.90E+10	5.84E+06

Figure 4.35: Bode plots with soaking time for the thick oxide of Zr-0.4Nb corroded up to 60 days in supercritical water.

Figure 4.36: Bode plots of 3 day-soaked (a) thin and (b) thick oxide of Zr-0.4Nb corroded in supercritical water.

(a) Zr-1.5Nb

(b) Zr-1.0Cr-0.2Fe

Figure 4.37: EIS spectra of (a) Zr-1.5Nb and (b) Zr-1.0Cr-0.2Fe alloys corroded in supercritical water condition for 60 days.

Tensile test for the model alloys

Since the maximum doses and temperatures of supercritical water reactor are similar to that of a fast reactor, thermal stability of the alloys for the cladding tube is required. So, to select the most promising candidate alloys we have to get available data on the mechanical stability. To get data on mechanical properties on which the alloys can be considered as candidate alloys for the cladding tube for the supercritical reactor, tensile tests of the model alloys were performed both at room temperature and 500°C in accordance with ASTM E8.

The thickness of the sheet specimens for the tensile test was about 1.0 mm. The specimens were machined to be about 3.0 mm in width and 12.5 mm in the nominal gage length. The tensile tests were carried out at room temperature with the strain rate 0.005(mm/mm)/min through yield stress and 0.05(mm/mm)/min after yield stress to failure. The tensile tests at 500°C had been done in 10^{-2} torr vacuum environment with the strain rate 0.05(mm/mm)/min to failure.

Figure 4.38 indicates the change of ultimate tensile strength (UTS) with the increase of Nb content. The strength of Zr-Nb alloys increase with Nb content. If the model alloys contain Nb over 1.5%, the alloys showed higher UTS than Zircaloy-4. It appears that Nb element was dissolved in the matrix to cause solid solution as well as precipitation hardening. Figure 4.39 shows the UTS and elongation of Zr-1.0X alloys. At room temperature, Zr-1.0Cu alloy has higher UTS than Zr-1.0Nb alloy and Zr-1.0Cu-0.5Mo alloy has higher UTS than Zr-1.0Cu alloy due to alloying effect on the strength. Zr-1.0Cr-0.2Fe alloy has the highest UTS among the alloys tested. At 500°C, however, the strength of the alloys does not show any trend because the hardening effect of alloying element almost disappears in the high temperature. The UTS of F/M steel is 640MPa at room temperature and 425MPa at 500°C. The UTS is higher than that of the Zr-based model alloys because the main element of F/M steel is Fe having BCC structure, and Cr which increases the strength in an elevated environment is added in the F/M steel. For Zr-based alloys, they have a limitation on alloying elements and their content since the alloying elements have an effect on the corrosion resistance and neutron absorption cross section of the Zr-based alloy for cladding to show lower strength than F/M steel.

On the other hand, it was difficult to find any trend of the change of elongation in the alloy system. But in general, addition of alloying element to the model alloys increased their strength and decreased their elongation. The total elongation of F/M steel are 20% at room temperature and 17.5% at 500°C, whereas that of the model alloys was 30~40% at room temperature and over 60% at 500°C. Thus, the model alloys has better property in ductility than in strength.

Figure 4.38: Mechanical properties of Zr-Nb alloys with the change of Nb content

Figure 4.39: Strength and elongation of Zr-1.0X alloys

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Task 5: Discussion and Modeling

In this section, we discuss the results of the corrosion testing and the oxide characterization and draw conclusions regarding the objectives of the program, namely:

- to demonstrate a technical basis for the improvement of the corrosion resistance of zirconium-based alloys in more extreme operating environments (such as those present in severe fuel duty cycles (high burnup, boiling, aggressive chemistry) and
- to investigate the feasibility (from the corrosion point of view) of using advanced zirconium-based alloys in a supercritical water environment.

This technical basis is obtained through the comparison of the corrosion kinetics and the examination of the fine structure of oxide layers formed on model alloys. These model alloys are designed to isolate specific features of the microstructure that are thought to affect the formation of the protective oxide layer. The key aspect of the program is to rationalize the differences in corrosion kinetics between alloys through the differences in the structure and evolution of the protective oxide formed on each alloy.

Overall Corrosion Performance during Corrosion Testing at 360°C

As shown in Section 2, the corrosion performance of the Zr alloys in this study varied widely, both among precipitate forming and solid solution forming alloys. Figure 5.1 shows the corrosion weight gain for a representative group of precipitate-forming Zr alloys tested in 360°C water and Figure 5.2 shows the same plot for solid solution alloys.

Figure 5.1: Weight gain vs. exposure time for precipitate-forming model alloys tested in 360 °C water.

Figure 5.2: Corrosion weight gain for Zr-Nb, Zr-Nb-Sn and Zr-Sn model alloys tested in 360 °C pure water.

Significant differences are observed in the corrosion behavior of the various alloys. These differences manifest themselves both in the tendency to exhibit breakaway behavior and in the clear separation of the value of the exponent n among the various alloys.

A few important points are listed below:

1. Several alloys, especially in the ZrFeCr system, showed protective behavior with stable oxide growth in all environments for the duration of the test.
2. Oxides formed in alloys with extensive solid solutions (especially Zr-Nb) did not show either obvious transitions or breakaway behavior.
3. The onset of breakaway was not directly related to the pre-breakaway oxide growth kinetics.
4. Very small amounts of alloying elements make a significant difference in corrosion behavior, as can be seen by comparing the behavior of alloy 12 (Zr0.2Fe0.1Cr (H)) with pure Zr.
5. As the alloying element content increases, the tendency to breakaway decreases, but the effect is not linear. A level of about 0.6% total precipitate-forming alloying element content appears to make the oxide resistant to breakaway.

Overall Corrosion Performance during Corrosion Testing at 500°C

The overall corrosion performance of the alloys during high temperature testing was better than expected with several alloys surviving the full exposure time. The general

agreement between the three high temperature tests was quite good. In particular little difference was seen between the results from static and dynamic autoclave testing. The results from lower pressure static testing agreed with the supercritical water results but showed slightly less oxide growth. Since the rankings of the alloys were preserved from test to test, it is possible the steam test could serve as a preliminary screening test for supercritical water behavior.

As in low temperature testing, there was wide variation in the behavior of the alloys, and compared to low temperature testing, the corrosion rates were much higher [1]. However the alloys that behaved the best in low temperature testing also behaved well in high temperature testing, i.e. the corrosion rankings were quite similar.

The results suggest that the alloy composition and microstructure are predominant in determining corrosion behavior. In general, the best alloys were from the ZrFeCr system (which also behaved the best in a 360°C water corrosion test). Higher alloying content (at least above 0.6%) improved corrosion resistance. The key to good corrosion resistance is to avoid breakaway corrosion and to maintain a stable, protective oxide. The onset of breakaway often occurs after long exposure times, which indicates that short exposure tests may be inadequate for determining corrosion behavior at high temperature.

Figure 5.3 shows a comparison of the weight gains for some of the model Zr alloys from this study and for ferritic-martensitic (F-M) and austenitic alloys, also under consideration for the SCWR, tested in supercritical water containing 25 ppb oxygen [2]. It is apparent that the corrosion rates for the best Zr alloys are higher than those seen for austenitics and lower than those seen for ferritic-martensitic alloys. However austenitic alloys exhibit extensive cracking in SCW [3]. In addition, it appears that the oxide growth in the Zr alloys is more stable than in the F-M and austenitic alloys (less incidence of weight loss or sudden weight changes). Figure 5.3 also illustrates that the current Zr-based model alloys have been tested to much longer times than the Fe-based alloys currently under consideration. It should be noted that although the results for the F-M and austenitic alloys are for tests conducted with 25 ppb oxygen, the corresponding results for tests performed in deaerated supercritical water (<10 ppb oxygen) show very similar behavior.

Overall, the oxidation rate of the best Zr alloy at 500°C is lower than that of the best F-M alloy by about a factor of 2. While significant concerns exist in terms of mechanical properties of Zr alloys at SCW temperatures, this research indicates that proper alloying additions induce protective oxide growth in model Zr alloys. From the corrosion point of view, Zr alloys should be considered as possible candidate materials for application in the supercritical water reactor.

Figure 5.3: Weight gain versus exposure time for some of the zirconium based model alloys tested in this program under different autoclave conditions (SCW or steam), as indicated. Also included are corrosion results from ferritic-martensitic and austenitic alloys from other programs [2, 3].

Corrosion Data Parameters

The weight gain results in the model alloys and the reference standard alloys can be analyzed in terms of three characteristics, (which can possibly be condensed to two), illustrated schematically in Figure 5.4:

- (i) **the pre-transition kinetics:** (red section of curve in Figure 5.4): if an oxide establishes a stable rate of growth with a protective black oxide, then the oxidation kinetics can be described by a power law of the type described in section 2.

$$w = At^n$$

where w is the weight gain in mg/dm^2 , t is the exposure time, and A and n are constants, which are characteristic of the alloy.

- (ii) **oxide transition:** it is often observed that the corrosion rate undergoes a change during oxidation, coincidentally with other changes that indicate that the oxide has lost protectiveness. In good behaving oxides, the protective layer reconstitutes itself

after the transition (blue curve) and this process repeats. The oxide thickness required to arrive at transition can be measured and serve as a measure of corrosion behavior. Some oxides do not undergo a well-marked transition and exhibit only slight slope changes (purple curve). Finally, the oxide can also become unstable at the transition and undergo loss of protectiveness (breakaway) which is our third characteristic of the corrosion process.

- (iii) **loss of protectiveness in oxide** (breakaway). Breakaway is defined here as very rapid oxide growth that continues indefinitely. The breakaway is interpreted as a transition in which the oxide reconstruction process is imperfect and does not re-constitute the protective layer. Breakaway can occur right at the beginning of the corrosion process or after some time (orange curves). It can also show only a moderate acceleration of weight gain such as in the green curve. In the green curve, the pre-transition (red) kinetics are not recovered, but they are somewhat slower and more controlled than the orange.

Figure 5.4: Schematic curves for illustrating different corrosion kinetics.

There are then three characteristics of the corrosion kinetics that define corrosion behavior. Items (ii) and (iii) can be considered jointly if we believe that (iii) is a transition type (ii) gone wrong. Alloying elements can influence corrosion by acting on these three different parameters and according to our hypothesis, something in the alloy microstructure causes a difference in the way the oxide grows. That is, a certain alloy microstructure implies a certain oxide microstructure, which implies a certain corrosion behavior. This work was started by specifying certain metal microstructures to investigate their role on corrosion.

Comparison of Corrosion Kinetic Behavior of Model Alloys

The model alloys were designed to investigate and isolate the effects of specific microstructures of both precipitates and solid solution elements on the corrosion process.

For precipitate forming alloys, the following effects of the microstructure on corrosion were considered:

- (a) **effect of precipitate size**. The Alloy 11-14 (ZrFeCr) series was prepared with the intent of comparing precipitate size while keeping volume fraction constant and vice-versa. Alloys 11 and 13 have small precipitates while Alloys 12 and 14 have large precipitates.
- (b) **effect of precipitate volume fraction**. In the same way, Alloys 11 and 12 (Zr_{0.2}Fe_{0.1}Cr) have a low volume fraction while 13 and 14 (Zr_{0.4}Fe_{0.2}Cr) have a high volume fraction. Furthermore, some of the newer alloys with higher alloying content have even larger precipitate volume fractions. Comparisons can also be made between Zr_{0.2}Nb, Zr_{1.0}Nb, and Zr_{1.5} Nb, with volume fraction increasing from 0.0 for Zr_{0.2}Nb to higher volume fractions for the remaining alloys.
- (c) **effect of precipitate type**: the comparison of the Nb alloys, high Cr alloys (alloys 21 – 24), Cu-Mo alloy (alloys 31 – 34)s and especially the newer alloys provide a variety of precipitate types.
- (d) **effect of “nobility” of precipitate alloying element**. Cu is much more noble than Cr or Fe, so comparison of alloys 31 - 34 (ZrCuMo) with Cr and Cr-Fe alloys (21 - 24) should yield this information
- (e) **effect of solute content**: On the solid solution side, the interest was just to examine the effect of valence on possibly creating or eliminating oxygen vacancies. => compare Nb <0.4 with Sn at various concentrations.

The ZrFeCr alloy corrosion results are quite reproducible, and show low rates of corrosion and no breakaway. The oxide thickness up until about 200 days in 360°C water is quite similar for the four alloys, i.e., no significant effect was observed as a result of the different precipitates sizes and volume fractions on the corrosion rate. Between 200 and 250 days, alloy 11 (Zr_{0.2}Fe_{0.1}Cr with low temperature processing) exhibits what appears to be an oxide transition, the only transition observed for the four ZrFeCr alloys.

The possible reasons for the protective behavior exhibited by these alloys (ZrFeCr) when compared to sponge Zr or crystal bar (alloys 41 and 42) are discussed below. The difference between the two alloy series (11 to 14 and 41 to 42) can be ascribed to the higher alloying element content in the ZrFe Cr alloys. The ZrFeCr alloys should have similar matrix transition element alloying contents as the solubility of Fe and Cr is in the 100's of ppm range. There are then two main differences between sponge Zr and the ZrFeCr alloys: (i) the different matrix composition (Fe/Cr in ZrFeCr vs. Fe in sponge Zr) and (ii) the presence of intermetallic precipitates in ZrFeCr.

Given the fact that no significant differences in corrosion behavior were observed between sponge and crystal bar Zr, and these exhibit significant differences in Fe content (of the level expected to be observed in the matrix of ZrFeCr alloys), it is unlikely that the difference in compositions plays a major role. The conclusion is that the precipitates play a specific role in avoiding breakaway, but not on the pre-transition kinetics as these are very similar for ZrFeCr and pure Zr. This is interesting, since there

were little differences observed for different precipitate volume fraction or different precipitates sizes. It is possible that the volume fraction of precipitates was sufficient in all of the ZrFeCr alloys to avoid breakaway. The fact that the lowest alloying element content/lowest heat treatment alloy exhibits the earliest transition indicates that differences in precipitate distribution could play a role in stabilizing the oxide layer against breakaway.

The ZrCrFe alloys in contrast, show higher rates of corrosion than ZrFeCr (except for Zr_{1.0}Cr_{0.2}Fe, which exhibits similar behavior as the ZrFeCr alloys). There is a marked difference in this case between the alloys of this group, with the corrosion rate and tendency to breakaway decreasing with both alloying content and presence of Fe.

The differences between these alloys and the ZrFeCr alloys are: (i) the precipitate crystal structures, (ii) precipitate volume fraction, and (iii) overall composition (reflected in precipitates composition). Since the differences between alloys 11 and 13 (cubic C15 precipitates) compared to 12 and 14 (hexagonal C14 precipitates) are not significant, it is not likely that (i) has a major effect on corrosion. Similar arguments as made for the ZrFeCr alloys lead us to place item (ii) as a possible second order effect, especially since a notable improvement was not observed for the newer higher alloying element content alloys. One hypothesis is that the presence of Fe stabilizes the precipitates against oxidation after incorporation in the oxide layer. Previous studies have shown that in order to oxidize precipitates containing Cr and Fe, it was necessary to reject the Fe into a metallic bcc Fe agglomerate which hampers the oxidation of Zr(Cr,Fe)₂ precipitates, compared to ZrCr₂. Such greater precipitate stability could allow them to play the role of preferential conduction paths for a longer time in the oxide layer and thus maintaining an orderly growth process.

In the ZrCuMo alloys, the corrosion behavior depends on the presence or absence of Mo. When Mo is present, the corrosion behavior is worse and its absence from ZrCu results in behavior similar to the better ZrFeCr alloys. ZrCuMo shows nodular corrosion, exhibits breakaway behavior and more scatter between samples. Thus, ZrCu behaves well and we do not understand why Mo has a negative effect on corrosion. In terms of precipitates, ZrCuMo has some additional small precipitates (it is the only alloy that has a bimodal precipitate distribution). We should note that the precipitates observed in ZrCu are of the type bct Zr₂Cu, and yet its behavior is similar to that of ZrFeCr, indicating that the precipitate crystal structure may not be important to the pre-transition kinetics.

The ZrNb alloys show stable oxide growth, without too much variation with Nb content, and protective behavior, without marked transitions. However, the oxide growth rate for these alloys is higher than the ZrFeCr alloys, with values of n around 0.4-0.45. In contrast to the ZrFeCr alloys, which have $n \sim 0.2$ and very straight lines up to 250 days, the ZrNb alloys show small “wiggles” in the curves, which could be interpreted as mild transitions. It is interesting to note that the ZrNb alloys with more than 0.4Nb have large number of beta Nb (and possibly ZrNbFe) precipitates in the matrix. Thus, we

have two series of alloys with large numbers of precipitates with two different transition behaviors concerning pre-transition kinetics and transition behavior.

Weight Gain Characteristic Parameters

The three measurable characteristics that we have selected for studying corrosion behavior are now discussed.

Pre-transition kinetics

The average weight gain from the short term autoclave tests (many measurements in the early stages of the corrosion experiment) were fit to a power law equation with the results shown in Tables 2.1 and 3.2. (Weight gains from individual coupons were also fit to the equation and yielded similar results.) The values of A and n are illustrated in Figures 5.5 and 5.6.

Figure 5.5 shows the values of n as calculated by fitting the corrosion curves in the protective regime, for both 360°C and 500°C corrosion testing. Only the alloys that exhibit sufficient protective behavior to allow a fit of the early part of the curve are shown. The vertical axis is used simply to visually separate the alloys and regimes studied. For each alloy group, various alloys and various samples are combined. The average for each group indicated in the legend is shown by a red dot. Cubic and parabolic behavior is indicated.

The results show that the values of n are in general higher during high temperature testing. The Zr-Nb alloys exhibit supra parabolic behavior ($n=0.67$), while the protective alloys in the ZrFeCr system show nearly cubic behavior ($n=0.33$), and a ZrFe-Nb alloy shows an intermediate value of $n=0.43$. At lower temperature, the Nb alloys still show the highest value of n (around 0.45), which is near parabolic behavior. The Zr-Sn-Nb and Zr-Sn alloys show lower values of n (0.41 and 0.29 respectively). The precipitate-forming alloys, including Zircaloy-4, ZrCrFe, ZrFeCr, and ZrCuMo, all show values of n quite close to those of pure Zr, that is, $n \sim 0.23$. Figure 5.6 shows the values of A for low and high temperature testing of the model alloys. Again, the value of A in high temperature (500°C) testing are a factor 3 higher than those obtained in low temperature (360°C) tests.

In addition to the differentiation between the alloy groups, it is interesting to note that the measurements are very consistent, even considering small variations from sample to sample. This suggests that it is the overall chemical composition of the alloy that determines the value of n . Although not shown in the Figures, the values of n for Sn and Nb alloys in lithiated water are very similar to the values in pure water.

The fact that crystal bar and sponge Zr and Zircaloy-4 exhibit values of n close to those of ZrCrFe alloys indicates that the precipitates have a primary role in avoiding oxide growth instabilities rather than in determining pre-transition kinetics. It is also interesting to note that there is a clear distinction between the n values in the solid

solution forming alloys (0.3-0.5) from those in the precipitate forming ones (0.15-0.25). The conclusion here is that the pre-transition kinetics are primarily determined by the alloying elements in solid solution. Because the precipitate forming alloys have very low amounts of alloying elements in solid solution, their oxide conductivity is similar to that of Zr and likely controlled by impurities. The solid solution alloys have kinetics that are dependent on the amount and the type of solute element.

Figure 5.5: Values of n for the different alloys and environments. The vertical axis has no meaning and is simply for displaying the values separately. In the upper part of the plot, the values for 500°C SCW and steam and in the bottom are the values for 360°C testing.

Figure 5.6: Values of A for the different alloys and environments. The vertical axis has no meaning and is simply for displaying the values separately. In the upper part of the plot, the values for 500°C SCW and steam and in the bottom are the values for 360°C testing.

Deviation from Parabolic Kinetics

In oxide growth limited by oxygen transport in a protective layer, the growth kinetics are parabolic ($n=0.5$). After an initial transient (as the film is established), the rate limiting step (RLS) should be some form of transport through oxide, (rather than interfacial reaction) because the oxide growth rate is observed to decrease continuously as the protective oxide thickness increases. Further evidence of this is given by the lack of dependence of corrosion rate on oxygen pressure. If it is accepted that transport through the oxide is the RLS and if this rate limiting step achieved by simple diffusion through a homogeneous medium, with fixed boundary conditions, then the parabolic law is obtained.

Since the deviations from parabolic law are all towards lower values of n (lower corrosion rates) a diffusion controlled process with sub-parabolic kinetics implies some form of slowed down or “hampered” diffusion. It is interesting to discuss the mechanisms of departure from parabolic law, as this allows one to discuss the ways in which the different alloys can influence corrosion behavior.

The first question is whether diffusion occurs through the bulk of the oxide or through the grain boundaries. Cox and co-workers [4] have shown that there is a double profile of oxygen, one very close to the surface and one extending far into the oxide. Using a diffusion equation solution, Cox et al. derived an activation energy for the far profile which was approximately 1-1.4 eV. They compared this with other measurements of

vacancy diffusion in zirconia and found that in bulk zirconia the activation energies are much higher (> 3 eV). They interpreted this as indicating that the diffusion occurred by a grain boundary diffusion mechanism. A simple calculation of the oxygen flux necessary to sustain the advancement of the oxide layer shows that the activation energy of 1-1.4 eV is that responsible for the layer advance.

However, the measurement in bulk zirconia was in stoichiometric ZrO_2 , and thus the activation energy includes both migration and formation energy. In the sub-stoichiometric zirconia layers, the vacancies are already present and thus the activation energy is constituted only of the migration energy, which could then be equated with the activation energy measured by Cox et al. The exact migration mechanism could be either bulk or grain boundary diffusion and both diffusion mechanisms are considered in the following.

Another question is the identity of the migrating species. Because the oxide is sub-stoichiometric, the oxygen vacancy is the defect considered to be the transporting species. One question arises as to what types of defects are formed in the growing oxide layer, i.e. how is charge compensated. If a vacancy gradient exists, as assumed by the parabolic model, then the number of electrons in the oxide needs to have a similar gradient to allow for charge compensation.

Mechanisms for Deviation from Parabolic Law.

Various ideas have been invoked in the literature to explain the deviation from parabolic behavior:

1. **Grain size gradient** [5]. This mechanism assumes grain boundary diffusion. As the grains grow, there is less area for oxygen to travel through. If the increase in grain size is linear with oxide thickness, a cubic law is obtained. With other dependences on thickness, other values of n would appear. In favor of this hypothesis is that (i) the grain size is observed to increase in the better performing commercial alloys, (ii) the grain size variation necessary for this increase would have to be substantial, (if the corrosion rate decreases by a factor of 2 so should the grain boundary density, and thus the grain size decrease by this factor with distance from the oxide metal interface). Such a large change is not seen.
2. **Grain boundary structure:** A more subtle grain boundary effect could be possible if the grain boundary structure changed with oxide thickness, so that it is more or less permeable to diffusion. No obvious reason exists for such a continuous change, however.
3. **Cracking:** Presence of lateral cracks, the density of which increases with oxide thickness. In this mechanism, diffusion occurs through the bulk of the oxide. As stresses develop, the density of lateral cracks increases. Because the vacancies cannot go through the cracks, the oxygen flow is diminished. Again a linear increase would cause the cubic law, and other functions lead to other values of n . (pro: we observe lateral cracks frequently in the oxides; con: oxides that crack more easily

would have lower corrosion rates, which is contrary to observation. Also, the cracking observed in the oxide occurs at or after the transition and not throughout the oxide)

4. **Stress effect on diffusion.** Stresses accumulate in the oxide as it grows, so that the lattice is “squeezed” and migration energy increases, decreasing the flow of oxygen. In favor of this idea is that compressive stresses are known to exist in the oxide layer during corrosion, but against is that a simple calculation shows that the elastic energy calculated by assuming the maximum possible stress in the elastic regime (σ_y) is small (on the order of 0.01 eV/atom) compared to the migration energy (~ 1 eV/atom).
5. **Thickness of barrier layer:** It is well known from different measurements (lithium uptake[6], and Electrochemical Impedance Spectroscopy (EIS) as in the present work) that in the pre-transition regime there is a double layer of oxide, one more protective (often called the barrier layer) and one more porous outer layer. If we denote $X = \text{Protective/Total oxide thickness ratio}$ and X does not change in the pre-transition regime, the parabolic regime is again found. If it is assumed that X changes as the oxide grows, then again almost any kinetics could be found. In that case, the $W = At^n$ curve would have a factor X that could create a new effective n . For the mechanism to yield $n < 0.5$ X would have to increase with thickness. Such a layer has been observed, but it is not obvious why X would change with thickness.
6. **Electron transport control.** In this case, we would assume that vacancy migration adjusts itself to the availability of electrons to allow for charge compensation. Fromhold [7] gives an expression for the deviation from parabolic law in terms of an exponential for a very simple case. If this is the rate limiting step, or if it alternates with vacancy diffusion as RLS, then could explain different kinetics. In favor of this idea is that this can provide a link with precipitate and precipitate distribution; but is it possible to accumulate space charge across oxide?
7. **Role of suboxide:** It is clear that a significant amount of oxygen is absorbed by the metal ahead of the advancing oxide front, as has been demonstrated in this study. Clearly, the more oxygen is lost to the metal, the slower will the oxide front advance. Such slowing down will be proportional to the length of the sub-oxide region. The model would predict that this oxygen-rich region would increase with time and that slower moving oxides would have a greater oxygen rich region. The measurements ahead of the oxide front show more oxygen in the fast moving oxides, but this is the profile associated with precipitation of oxides ahead of the front (>0.3). It is not clear how far the 0.3 plateau extends into the metal. Oxygen is in fact lost to the metal and different alloys have different profiles, but the amount of oxygen lost appears relatively small to account for the slowing down.

Mechanism number 1 can be eliminated based on observations. If the grain increase is not enough, it cannot work. Mechanisms numbers 2 or 3 appear unlikely on simple experimental and observation grounds.

If we now compare the different behavior of the classes of model alloys, we can discuss further these models. Since Zr-Nb has near-parabolic kinetics, it could be possible to study microstructure differences between these oxides and those in the precipitate alloys.

It is noted that pure Zr, in the instances when it has low corrosion rate at the beginning, (i.e. when the weight gain does not increase suddenly right at the start) has n in the range about 0.2-0.25, which is the same as that of the ZrFeCr, ZrCrFe, alloys. This, it seems reasonable to assume that the presence of precipitates does not influence the kinetics of pre-transition.

Since Sn also has a higher n than 0.2, it seems reasonable to assume that alloying elements in solid solution control pre-transition behavior. It is also interesting to note that of the ZrNb alloys, the lowest corrosion rate was seen in the only alloy with a composition below the solubility limit (Zr0.2Nb). This again points to the role of solid solution elements on the pre-transition behavior. We also note that the corrosion rate of Zr0.2Nb was similar to that of Zr0.4Sn0.2Nb; note also that Zr0.4Sn0.4Nb behaves like Zr0.4Nb, which suggest specifically that Nb content controls pre-transition kinetics. One possible hypothesis is that in the absence of solid solution elements to change the valence of the oxide and create vacancies, the oxide grows very slowly in the ZrFeCr, Zr, etc. So Sn and Nb would create vacancies in ZrO₂ and allow the transport through the oxide to be performed in a faster, but perhaps more orderly, manner. The difference in behavior of Zr and precipitates alloys would be due to the latter being able to avoid instabilities that would lead to the orange curve (Figure 5.4). This would explain why the precipitate elements have lower n , but would not explain why Zircaloy also had $n \sim 0.2$ (since Zircaloy contains Sn).

In summary, it appears that the main link between alloy microstructure and composition with pre-transition corrosion rate stems from the amount of solute in solid solution, and from the valence of this element. The evidence above would appear to indicate that the overall chemistry of the solid solution controls the pre-transition behavior, such that elements such as Nb and Sn increase the transport properties of the oxide. For the precipitate forming elements, the amount in solid solution is very small, and the pre transition rate is likely controlled by impurities or by the small amount of alloying element content.

One key question is: beyond determining mechanisms, is it important to know why the kinetics are not parabolic? It would appear not, since the greatest influence on the overall corrosion rate appears to be the thickness at first transition and the ability to avoid corrosion breakaway. In this case the precipitates formed appear to enhance oxide stability, as discussed below.

Oxide Transition

The oxide transition is defined as the thickness at which the corrosion weight gain shows a sudden increase, as shown in Figure 5.1. The thicknesses at transition can be quite different for the various alloys [8]. The transition is normally interpreted as a more or less “sudden” event that causes the oxide to lose its protective character and allows water to penetrate the oxide layer, possibly down to the metal region.

As the oxide grows, the metal is transformed into zirconia, which entails an increase in volume of approximately 1.56. The great majority of this volume increase is accommodated in the vertical direction as 1 micron of oxide results in approximately 1.56 microns of oxide. It is difficult however, to account exactly for this volume increase, as the error in the measurement of oxide thickness is not much smaller than 5%, and a few % difference in the accommodation of these strains would lead to large stresses. It is known in fact that stresses accumulate in the oxide layer and also that stresses are higher in alloys that show earlier transition (Zircaloy-4) compared to later transition (M5)[9, 10]. It is also likely that a stress gradient exists in the oxide layer, with higher stresses near the oxide-metal interface. Typically, stresses on the order of 2 GPa have been measured in the oxide layers. It is possible that the accumulation of the stresses differs among the different alloys, due to different “phase transformation tensors” (relationship between orientation of the film during transformation of Zr into ZrO_2).

It should be noted that transitions in the classical sense are not seen in some of the alloys. In the set of alloys studied, Zircaloy 4 exhibited a clear transition, as did the Zr-Sn alloys. The ZrFeCr alloys have in general not yet exhibited a clear transition, (although alloy 11 may be starting). None of the ZrNb alloys have yet shown a marked transition. So, preliminarily, we associate a marked transition with the presence of Sn. Some of the oxides however have exhibited breakaway after long exposures.

Mechanisms of oxide transition

1. Stress accumulation breaks oxide. In this case, it is assumed that the lateral stresses that accumulate reach a critical point and break the oxide. If vertical cracks are created, water has access to the oxide-metal interface and the corrosion rate increases. Although it is known that in-plane compressive stresses accumulate, which would tend to cause lateral cracks. The presence of cracks in and of itself, would cause little effect on the permeability of this oxide layer. To cause the oxide to lose protectiveness it would be necessary to create vertical cracks. This could happen for example by one of the processes shown in Figure 5.7: (a) shearing a block of the oxide, (b) blistering or (c) surface instabilities) that would allow a compressive stress to cause vertical cracks. However there are reasons to question the three processes illustrated below on either theoretical or experimental grounds: no blistering is seen during corrosion at small oxide thicknesses, no vertical cracks are seen in general and no cracks are seen associated with the ends of lateral cracks; finally surface instability (which predicts a shear stress as result of the propagation of an interface instability) only allows for about 200 Mpa as a maximum of the stresses it can induce, which is small compared to the stresses accumulated.

Thus, although stresses accumulate in oxides and stress accumulation may be different among alloys, it is difficult to see how the stresses that are present in growing oxides would cause transition.

Figure 5.7 Stress accumulation leading to oxide cracking.

2. Porosity increases and percolates; During oxide advancement, new grains are constantly created, with varying interfaces. At these interfaces, small grain boundary mismatches can create little “wormholes” at grain boundaries, which then allow access of water once the porosity is interlinked. Conceivably, the density of these wormholes (or pores) could increase with oxide thickness as a result of preferential attack at grain boundaries. If the porosity increases with oxide thickness then at some critical pore density a continuous path will be established from the oxide-metal to the oxide- water interface, a condition known as *percolation*. Cox and co-workers have measured oxide porosity, and others such as Godlewski and Pecheur, as well as Barberis [6, 11-13] have found that the external layer at least is porous. However the porosity exists in the outer layer, not in the barrier layer, which controls diffusion. In addition, there is no obvious reason for the porosity to continuously increase with continued oxide growth. Thus, because of the above, it is also not clear how this mechanism could cause transition or breakaway.

Figure 5.8 The percolation mechanism for the transition.

3. Mixed mode transition [14]: This mechanism combines a gradual process (porosity development) with a sudden process (cracking) and is based on the fact that if porosity exists, percolation can be reached with lateral cracking. In this idea, porosity is developed during the formation of the grains and remains constant thereafter, although at different stages of the oxide growth process we could have different densities of pores being created at the O/M interface. The crucial step is that while this constant porosity does not change with thickness at a given position, the onset of lateral cracks could push that porosity over the percolation condition.

Figure 5.9: The mixed mode mechanism for oxide transition/breakaway.

If this mechanism works, then oxides that have larger grains and with less grain to grain mismatch should exhibit less tendency to breakaway. This is in fact the case with the commercial alloys we have studied. The grain size measured with synchrotron radiation is however similar for the alloys studied. However the differences observed in texture formation cause differences in oxide behavior, such that an orderly process of formation of well aligned oxide grains leads to better resistance to breakaway.

This then poses the question as to what in the alloy causes orderly well-oriented oxide growth with large grains. It could be argued that the slowness of growth allows for oxide to rearrange itself which would allow better aligned growth. But, in fact some of the oxides with low n are more susceptible to breakaway (pure Zr for example), which suggest slow and *stable* oxide growth is needed. If the precipitates and alloying elements in the oxide layer allow for equalization of current around the surface (metal grain to metal grain or possibly causing instabilities in oxide growth to be flattened), then this will help delay the transition.

In summary, the mixed mode transition model seems to be a good working hypothesis for the transition mechanism.

Breakaway Susceptibility

The most obvious difference in the behavior of the alloys comes from their different breakaway behavior. Some alloys exhibit protective behavior until very long exposure times, while others show non-protective behavior right from the beginning. The key questions to ask are what in the oxide re-growth process causes breakaway and what causes the difference in breakaway susceptibility between the various alloys.

These can be two separate but related questions. One relates to what goes wrong in the transition to breakaway and the other relates to the consequences of this event.

There are various interesting points about the breakaway susceptibility. Some alloys, such as pure Zr, go for some time before they undergo breakaway. That is, they form a black protective oxide, with similar kinetics as other protective oxides and then undergo breakaway. Thus the process of forming a protective oxide appears to be irreversibly disturbed. In some way the oxide reaches a bifurcation and takes the road of fast kinetics

never to return. Once breakaway occurs it appears to be irreversible. This implies that the oxide formed causes the kinetics to be fast. Presumably, if the oxide was removed and the process restarted, the new oxide would have a chance to reform correctly. Finally, very small differences in composition make large differences in corrosion behavior.

Microstructural Features of Protective Oxides

In section 4, the microstructural features of a protective oxide compared to those of a non-protective oxide were discussed, and are summarized here.

1. The oxide-metal interface in the protective oxides showed the presence of a sub-oxide phase, which is an ordered solid solution of oxygen in Zr ahead of the oxide front. This layer was a factor of 10 thicker in the oxides formed in 500°C testing than in the oxides formed in 360°C testing.
2. Also at the oxide-metal interface, the following strong orientation relationship was seen $[10\bar{1}0]_{hcp} // [002]_T // [020]_M$. The key to establishing the strong relationship between $[020]_M$ and $[10\bar{1}0]_{hcp}$ planes is the presence at the interface of the $[002]_T$ precursor phase. This highly oriented tetragonal phase forms ahead of the oxide layer and is present in the protective oxides and absent in the non-protective ones.
3. As a result of this, the ratio of the intensities of the $[020]_M$ to $[002]_M$ peaks was higher in the bulk of the protective oxides than in the non-protective oxides.
4. In addition, differences were seen in the thickness-averaged crystallographic texture of the protective and non-protective oxides. It appears that a more orderly growth and well oriented oxides decrease the tendency for corrosion breakaway.

Following the model developed for normal oxide growth we can then try to explain breakaway. Since at transition small tetragonal equiaxed grains form which later transform to monoclinic, possibly the critical point is the proper transformation of tetragonal into monoclinic. Comparing Zr alloy corrosion behavior with Hf, Hf is quite a bit more protective than Zr. One important difference in the two oxide systems is the fact that Hf forms only one phase, and Zr forms two. It is possible that the transition from one phase to the other causes the instabilities we observed. When that transformation occurs properly, i.e. when enough grains exist of the proper orientation or when the highly oriented tetragonal phase forms and leads to new aligned oxide then breakaway is averted. It is possible that the volume increase from tetragonal to monoclinic plays a role (although it has to be better evaluated).

The main conclusions as to relating the microstructure of the alloys and of the oxides with the corrosion rate are:

1. The microstructure in the protective oxides described above, provides a better alignment and likely induces less stress accumulation, and thus delays the onset of breakaway or loss of protectiveness.
2. This microstructure associated with protective oxides is present during both 360°C and 500°C testing and in the same alloys. Thus, the protection mechanism is likely the same at both temperatures.
3. The presence of precipitates is seen as crucial at avoiding breakaway, by allowing a steady transport of electrons through the oxide, (since the precipitates are not oxidized in the barrier layer), thus avoiding large lateral differences in corrosion rate, and enhanced oxide breakup.

The proposed mechanism for loss of protectiveness, (a combination of cracking and porosity percolation) can likely explain the experimental results.

Conclusions

A detailed study has been conducted to address the issue of susceptibility of Zr alloys to uniform corrosion in the proposed supercritical water reactor and of the specific role of alloying elements. Thirty model alloys were corrosion-tested for periods over 400 days in 360°C water (as well as limited testing in lithiated water), from 150 to over 300 days in 500°C supercritical water and a wide range of corrosion behavior was observed. The general agreement between the three high temperature tests was quite good. In particular little difference was seen between the results from static and dynamic autoclave testing. The results from lower pressure static testing agreed with the supercritical water results but showed slightly less oxide growth. Since the rankings of the alloys were preserved from test to test, it is possible the steam test could serve as a preliminary screening test for supercritical water behavior.

1. Corrosion tests for the model Zr alloys showed a wide variety of corrosion resistance depending on the alloy composition during both 360°C and at 500° testing. These differences manifested themselves in the different values of A and n obtained for the different alloys and in the different tendencies for breakaway.
2. In comparison with other alloys being considered for the SCWR, the Zr alloys showed higher corrosion rates than austenitic alloys, and lower corrosion rates than ferritic-martensitic and 12Cr steels.
3. Significant concerns exist in terms of creep rates, and other properties, but this research indicates that proper alloying additions induce protective oxide growth in model Zr alloys, such that from the corrosion point of view they should be considered as possible candidate materials for application in the supercritical water reactor.

The oxides were also characterized to determine the oxide structures associated with protective and non-protective behavior and the conclusions are:

4. Characteristic differences exist in the oxide-metal interface regions of protective as compared to non-protective oxides. In particular the presence of two interfacial oxide phases, a highly oriented tetragonal phase and a sub-oxide phase was associated with protective behavior uniform corrosion.
5. The overall crystallographic texture of oxides also showed clear differences between protective and non-protective oxides. The growth direction of the oxides was similar but the distribution of the lobes was considerably more anisotropic in the non-protective oxides than in the protective ones.
6. The protection mechanisms and the mechanisms of transition/breakaway were discussed and the differences between alloys are rationalized in terms of mechanistic models.

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APPENDIX – A

Water 360°C Corrosion Experiments

Water 680F autoclaves (Westinghouse)

Alloys 11-14 - ZrFeCr

updated T554 and T556

**Alloy
11**

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
11-1	Zr-0.2Fe-0.1Cr	10.8	19.2	23.9	27.5	30.1	33.0	36.1	38.3
11-2	Zr-0.2Fe-0.1Cr	11.1	19.4	24.3	28.2	31.9	33.7	36.8	38.9
11-3	Zr-0.2Fe-0.1Cr	9.8	16.9	20.4	23.5	26.3	27.9	31.2	33.9
11-4	Zr-0.2Fe-0.1Cr	12.9	22.0	26.2	30.8	33.7	36.2	38.9	41.0
11-7	Zr-0.2Fe-0.1Cr	11.3	21.0						
11-8	Zr-0.2Fe-0.1Cr	12.3	21.4						

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
11-5	Zr-0.2Fe-0.1Cr	11.0	20.1	25.2	28.8	32.0	35.2	38.0	42.5
11-6	Zr-0.2Fe-0.1Cr	9.8	17.9	22.3	25.3	28.8	32.2	34.5	39.8

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2	55.3
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
11-9	Zr-0.2Fe-0.1Cr	10.9	13.4	15.7	17.6	19.5	20.4	22.4	23.9
11-10	Zr-0.2Fe-0.1Cr	9.6	11.8	14.2	16.5	18.1	19.3	21.0	22.7
11-11	Zr-0.2Fe-0.1Cr	10.0	12.7	15.0	17.2	18.4	20.0	21.9	24.0
11-12	Zr-0.2Fe-0.1Cr	10.3	13.0	15.3					

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0	242
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
11-13	Zr-0.2Fe-0.1Cr	18.6	21.9	24.7	27.9	30.2	35.7	43.1	52.2
11-14	Zr-0.2Fe-0.1Cr	19.4	23.5	26.5	29.8	32.9	38.6	45.2	52.6
T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0	83.1
	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
11-15	Zr-0.2Fe-0.1Cr	7.7	10.5	12.8	15.1	18.2	20.3	22.6	26.2
T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0	83.1
	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
11-16	Zr-0.2Fe-0.1Cr	6.9	9.3	11.6	13.9	16.7	18.7	20.1	23.2

Alloy 11

Alloy 11 (cont-1)

T554		240.0	270.0	300.0	330.0	353.0	381.0	409.0	437.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(18)
11-1	Zr-0.2Fe-0.1Cr	44.0	54.1	63.1	73.7	85.2	102.5	124.9	
11-2	Zr-0.2Fe-0.1Cr	43.2	53.0	62.2	71.4	81.3	98.6	120.7	145.1
11-3	Zr-0.2Fe-0.1Cr	40.8							
11-4	Zr-0.2Fe-0.1Cr								
11-7	Zr-0.2Fe-0.1Cr								
11-8	Zr-0.2Fe-0.1Cr								
T564		240	270.0	300.0	323.0	351.0	379.0	407	435.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)	Gain(17)
11-5	Zr-0.2Fe-0.1Cr	50.5	60.3	68.3	77.6	93.1	114.7	138.1	163.1
11-6	Zr-0.2Fe-0.1Cr	49.1	58.3	69.4	81.3	100.6	124.0	148.9	174.1
T556		69.3	83.3	113.3	140.4	173.3	203.3	233.3	263.3
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
11-9	Zr-0.2Fe-0.1Cr	26.1	28.3	32.0	35.5	37.9			
11-10	Zr-0.2Fe-0.1Cr	24.5	26.2	30.0	33.9	36.5	39.9	45.0	54.6
11-11	Zr-0.2Fe-0.1Cr								
11-12	Zr-0.2Fe-0.1Cr								
T562		272	295.0	323.0	351.0	379.0	407	435.0	
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	Gain(16)	

11-13	Zr-0.2Fe-0.1Cr	59.4	66.4	79.4	96.6	116.7	138.5849	162.0	
11-14	Zr-0.2Fe-0.1Cr	59.0	64.5	74.7	89.4	107.6	127.57968	149.8	
T563		116.0	146.0	176	206.0	236.0	259	287	315.0
	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
11-15	Zr-0.2Fe-0.1Cr	29.0	32.7	36.5	41.6	47.2	52.8	59.5	67.2
T563		116.0	146.0	176	206.0	236.0	259	287	315.0
	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
11-16	Zr-0.2Fe-0.1Cr	25.7	28.3	31.6	37.7	44.4	50.7	57.3	67.7

Alloy 11

Alloy 11 (cont-2)

T554		465.0	493.0
Sample	Alloy	Gain(19)	Gain(20)
11-1	Zr-0.2Fe-0.1Cr		
11-2	Zr-0.2Fe-0.1Cr	172.3	
11-3	Zr-0.2Fe-0.1Cr		
11-4	Zr-0.2Fe-0.1Cr		
11-7	Zr-0.2Fe-0.1Cr		
11-8	Zr-0.2Fe-0.1Cr		

T564		463
Sample	Alloy	Gain(18)
11-5	Zr-0.2Fe-0.1Cr	188.7
11-6	Zr-0.2Fe-0.1Cr	199.8

T556		293.3	316.3	344.3	372.3	400.3	428.3	456.25
Sample	Alloy	Gain(17)	Gain(18)	Gain(19)	Gain(20)	Gain(22)	Gain(23)	Gain(24)
11-9	Zr-0.2Fe-0.1Cr							
11-10	Zr-0.2Fe-0.1Cr	63.1	70.1	83.2	101.8	123.9	147.3	171.8
11-11	Zr-0.2Fe-0.1Cr							
11-12	Zr-0.2Fe-0.1Cr							

T562	
Sample	Alloy

11-13 Zr-0.2Fe-0.1Cr
 11-14 Zr-0.2Fe-0.1Cr

T563 **321.4**
Alloy **Gain(17)**

11-15 Zr-0.2Fe-0.1Cr 69.7

T563 **343.0** **371.0** **399.0**
Alloy **Gain(18)** **Gain(19)** **Gain(20)**
 11-16 Zr-0.2Fe-0.1Cr 83.5 102.2 123.3

Alloy 11	Summary and final results		
Archive	Days	Wt Gain	Test
11-1	409.0	124.9	T554
11-2	465.0	172.3	T554
11-3	240.0	40.8	T554
11-4	210.0	41.0	T554
11-5			T564
11-6			T564
11-7	28.0	21.0	T554
11-8	28.0	21.4	T554
11-9	173.3	37.9	T556
11-10			T556
11-11	55.3	24.0	T556
11-12	7.0	15.3	T556
11-13			T562
11-14			T562
11-15	321.4	69.7	T563
11-16			T563

Alloy
12

Archived

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)	210.0 Gain(9)	240.0 Gain(10)
12-2	Zr-0.2Fe-0.1Cr	12.9	21.3	25.4	29.0	31.1	32.9	35.1	36.8	38.2
12-4	Zr-0.2Fe-0.1Cr	14.4	23.0							
12-5	Zr-0.2Fe-0.1Cr	12.6	22.2							
12-8	Zr-0.2Fe-0.1Cr	12.4	21.2	25.2	28.9	31.2	33.0	34.6	36.7	

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)	210.0 Gain(9)	240.0 Gain(10)
12-1	Zr-0.2Fe-0.1Cr	11.8	20.8	25.1	28.7	30.7	32.8	34.3	36.2	37.8

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)	210.0 Gain(9)	240.0 Gain(10)
12-7	Zr-0.2Fe-0.1Cr	12.6	21.3	25.4	29.1	31.2	33.1	34.6	36.6	37.9

T564 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	60.0 Gain(3)	90.0 Gain(4)	120.0 Gain(5)	147.1 Gain(6)	180.0 Gain(7)	210.0 Gain(8)	240 Gain(9)
12-3	Zr-0.2Fe-0.1Cr	12.5	20.9	25.5	28.2	31.0	32.7	34.9	36.0	37.8

12-6	Zr-0.2Fe-0.1Cr	12.1	20.7	25.4	28.3	30.7	32.6	34.9	36.0	37.7
T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2	55.3	69.3
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
12-9	Zr-0.2Fe-0.1Cr	10.2	13.0	15.2	17.4	18.9	20.4	22.5	24.1	26.0
12-10	Zr-0.2Fe-0.1Cr	10.2	12.7	14.8	17.2	19.0	20.0	22.1	24.2	25.8
12-11	Zr-0.2Fe-0.1Cr	10.5	13.0	15.1	17.6	19.4	20.6	22.7	24.5	
12-12	Zr-0.2Fe-0.1Cr	10.3	12.8	14.8						
T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0	242	272
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
12-13	Zr-0.2Fe-0.1Cr	18.6	22.4	25.1	27.5	29.4	31.4	32.8	34.6	36.4
T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0	242	272
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
12-14	Zr-0.2Fe-0.1Cr	18.8	22.9	25.4	27.4	29.9	31.2	33.1	34.6	36.0
T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0	83.1	116.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
12-15	Zr-0.2Fe-0.1Cr	7.3	9.5	11.9	14.5	17.4	19.6	21.0	24.0	26.7
12-16	Zr-0.2Fe-0.1Cr	6.7	8.9	10.8	13.2	16.1	18.0	19.9	22.5	25.7

Alloy 12 (cont - 1)

		270.0	300.0	330.0	353.0	381.0	409.0	437.0
		Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(18)
12-2	Zr-0.2Fe-0.1Cr	39.9	41.7	43.8	45.8	48.1	51.6	55.9
12-4	Zr-0.2Fe-0.1Cr							
12-5	Zr-0.2Fe-0.1Cr							
12-8	Zr-0.2Fe-0.1Cr							
T554		270.0	300.0	330.0	353.0	381.0	409	415.4
Sample	Alloy	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(17)
12-1	Zr-0.2Fe-0.1Cr	39.6	41.9	44.2	46.6	49.7	53.8	55.0
T554		270.0	300.0	323.0	351.0	379.0	407.0	435.0
Sample	Alloy	Gain(11)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(18)	Gain(19)
12-7	Zr-0.2Fe-0.1Cr	39.4	41.1	42.9	44.9	47.8	51.5	56.1
T564		270.0	300.0	323	351.0	379	407	435.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)	Gain(17)
12-3	Zr-0.2Fe-0.1Cr	39.4	41.3	42.8	45.3	48.0	51.7	56.2
12-6	Zr-0.2Fe-0.1Cr	39.8	41.6	43.3	45.6	48.4	52.2	57.0

T556		83.3	113.3	140.4	173.3	203.3	233.3	263.3
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
12-9	Zr-0.2Fe-0.1Cr	27.4	29.9	31.7	33.8			
12-10	Zr-0.2Fe-0.1Cr	27.0	29.5	31.3	33.5	34.8	36.3	37.9
12-11	Zr-0.2Fe-0.1Cr							
12-12	Zr-0.2Fe-0.1Cr							

T562		295.0	323.0	351.0	379.0	407	435.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	Gain(16)
12-13	Zr-0.2Fe-0.1Cr	37.9	40.6	43.0	46.0	49.2	52.4

T562		295.0	323.0	351.0	357.4	379.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)
12-14	Zr-0.2Fe-0.1Cr	37.4	39.4	42.1	43.8	

T563		146.0	176	206.0	236.0	259	287	315.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
12-15	Zr-0.2Fe-0.1Cr	29.1	30.2	31.8	32.7	33.9	35.8	38.2
12-16	Zr-0.2Fe-0.1Cr	27.7	28.7	30.0	31.0	32.3	33.8	36.3

Alloy 12 (cont - 2)

		465.0	493.0
		Gain(19)	Gain(20)
12-2	Zr-0.2Fe-0.1Cr	60.8	
12-4	Zr-0.2Fe-0.1Cr		
12-5	Zr-0.2Fe-0.1Cr		
12-8	Zr-0.2Fe-0.1Cr		

T554		437.0
Sample	Alloy	Gain(18)
12-1	Zr-0.2Fe-0.1Cr	

T554		463.0
Sample	Alloy	Gain(20)
12-7	Zr-0.2Fe-0.1Cr	61.0

T564		463
Sample	Alloy	Gain(18)
12-3	Zr-0.2Fe-0.1Cr	60.7
12-6	Zr-0.2Fe-0.1Cr	61.8

T556	293.3	316.3	344.3
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Sample	Alloy	Gain(17)	Gain(18)	Gain(19)
12-9	Zr-0.2Fe-0.1Cr			
12-10	Zr-0.2Fe-0.1Cr	39.3	40.5	42.2
12-11	Zr-0.2Fe-0.1Cr			
12-12	Zr-0.2Fe-0.1Cr			

T562

Sample	Alloy
12-13	Zr-0.2Fe-0.1Cr

T562

Sample	Alloy
12-14	Zr-0.2Fe-0.1Cr

T563

		343.0	371.0	399.0
Sample	Alloy	Gain(18)	Gain(19)	Gain(20)
12-15	Zr-0.2Fe-0.1Cr	41.4	44.8	48.5
12-16	Zr-0.2Fe-0.1Cr	39.6	43.3	47.5

Alloy 12 (cont - 3)

Summary and final results

Archive	Days	Wt Gain	Test
12-1	415.4	55.0	T554
12-2	465.0	60.8	T554
12-3			T564
12-4	28.0	23.0	T554
12-5	28.0	22.2	T554
12-6			T564
12-7			T554
12-8	210.0	36.7	T554
12-9	173.3	33.8	T556
12-10			T556
12-11	55.3	24.5	T556
12-12	7.0	14.8	T556
12-13			T562
12-14	357.4	43.8	T562
12-15			T563
12-16			T563

Alloy 13

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
13-2	Zr-0.4Fe-0.2Cr	12.4	21.2	25.6	29.1	31.7	34.1	36.0
13-3	Zr-0.4Fe-0.2Cr	9.8	17.2	20.8	24.3	26.4	28.9	31.2
13-4	Zr-0.4Fe-0.2Cr	11.3	19.0	24.0	28.4	30.5	33.3	35.0
13-7	Zr-0.4Fe-0.2Cr	9.9	17.1					
13-8	Zr-0.4Fe-0.2Cr	9.8	17.7					

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
13-1	Zr-0.4Fe-0.2Cr	11.4	20.1	24.9	28.4	31.2	33.3	35.6

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
13-5	Zr-0.4Fe-0.2Cr	11.0	19.3	23.9	27.0	29.9	32.3	34.4
13-6	Zr-0.4Fe-0.2Cr	10.8	19.2	23.7	26.9	29.8	32.2	34.5

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
13-9	Zr-0.4Fe-0.2Cr	10.1	12.6	15.0	17.1	18.8	20.2	21.9
13-10	Zr-0.4Fe-0.2Cr	9.8	12.4	14.7	17.0	18.7	19.8	21.7
13-11	Zr-0.4Fe-0.2Cr	9.7	12.4	14.6	17.2	18.9	20.0	22.1
13-12	Zr-0.4Fe-0.2Cr	9.9	12.1	14.4				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
13-13	Zr-0.4Fe-0.2Cr	15.9	19.0	21.5	24.0	26.2	28.2	30.3
13-14	Zr-0.4Fe-0.2Cr	17.4	21.5	25.0	27.8	30.2	32.1	34.1

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
13-15	Zr-0.4Fe-0.2Cr	6.7	9.0	11.1	13.3	15.8	17.9	19.5

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
13-16	Zr-0.4Fe-0.2Cr	6.8	9.0	11.1	13.1	16.3	18.5	19.9

Alloy 13 (cont - 1)

T554		240.0	270.0	300.0	330.0	353.0	381.0	409.0	437.0
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(18)
13-2	Zr-0.4Fe-0.2Cr	39.6	41.4	43.1	44.3	45.6	47.0	48.5	50.0
13-3	Zr-0.4Fe-0.2Cr	34.6	36.5	37.8	39.0	40.2	41.3	42.7	43.9
13-4	Zr-0.4Fe-0.2Cr								
13-7	Zr-0.4Fe-0.2Cr								
13-8	Zr-0.4Fe-0.2Cr								

T554		240.0	270.0	300.0	330.0	353.0	381.0	409	415.4
Sample	Alloy	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(17)
13-1	Zr-0.4Fe-0.2Cr	39.2	41.1	42.9	44.2	45.2	46.5	47.8	49.5

T564		240	270.0	300.0	323	351.0	379	407	435.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)	Gain(17)
13-5	Zr-0.4Fe-0.2Cr	38.2	40.1	41.4	42.4	43.8	45.0	46.5	47.5
13-6	Zr-0.4Fe-0.2Cr	38.1	39.9	41.1	42.4	43.8	45.0	46.3	47.6

T556		69.3	83.3	113.3	140.4	173.3	203.3	233.3	263.3
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
13-9	Zr-0.4Fe-0.2Cr	25.2	26.8	29.6	32.8	34.6			
13-10	Zr-0.4Fe-0.2Cr	24.9	26.6	29.2	31.9	34.4	36.4	38.2	40.1
13-11	Zr-0.4Fe-0.2Cr								
13-12	Zr-0.4Fe-0.2Cr								

T562		272	295.0	323.0	351.0	379.0	407	435.0	
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	Gain(16)	
13-13	Zr-0.4Fe-0.2Cr	33.2	35.2	35.7	37.1	38.5	39.6	40.7	
13-14	Zr-0.4Fe-0.2Cr	37.0	38.1	39.6	40.9	42.0	43.3	44.3	

T563		116.0	146.0	176	206.0	236.0	259	287	315.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
13-15	Zr-0.4Fe-0.2Cr	25.1	27.1	29.4	31.0	32.3	33.6	35.0	36.3

T563		116.0	146.0	176	206.0	236.0	259	287	315.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
13-16	Zr-0.4Fe-0.2Cr	25.6	27.7	29.7	31.1	32.7	33.8	35.5	36.9

Alloy 13 (cont – 2)

T554		465.0	493.0				
Sample	Alloy	Gain(19)	Gain(20)				
13-2	Zr-0.4Fe-0.2Cr	51.5	53.4				
13-3	Zr-0.4Fe-0.2Cr	45.0	46.1				
13-4	Zr-0.4Fe-0.2Cr						
13-7	Zr-0.4Fe-0.2Cr						
13-8	Zr-0.4Fe-0.2Cr						

T554		437.0					
Sample	Alloy	Gain(18)					
13-1	Zr-0.4Fe-0.2Cr						

T564		463
Sample	Alloy	Gain(18)
13-5	Zr-0.4Fe-0.2Cr	48.7
13-6	Zr-0.4Fe-0.2Cr	48.7

T556		293.3	316.3	344.3	372.3	400.3	428.3	456.25
Sample	Alloy	Gain(17)	Gain(18)	Gain(19)	Gain(20)	Gain(22)	Gain(23)	Gain(24)
13-9	Zr-0.4Fe-0.2Cr							
13-10	Zr-0.4Fe-0.2Cr	41.3	42.5	44.0	45.4	46.7	48.0	48.9
13-11	Zr-0.4Fe-0.2Cr							
13-12	Zr-0.4Fe-0.2Cr							

T562	
Sample	Alloy
13-13	Zr-0.4Fe-0.2Cr
13-14	Zr-0.4Fe-0.2Cr

T563		343.0	371.0	399.0
Sample	Alloy	Gain(18)	Gain(19)	Gain(20)
13-15	Zr-0.4Fe-0.2Cr	37.8	39.0	40.1

T563		321.4					
Sample	Alloy	Gain(17)					
13-16	Zr-0.4Fe-0.2Cr	51.6					

Alloy 13 (cont - 3) Summary and final results

Archive	Days	Wt Gain	Test
13-1	415.4	49.5	T554
13-2			T554
13-3			T554
13-4	210.0	37.0	T554
13-5			T564
13-6			T564
13-7	28.0	17.1	T554
13-8	28.0	17.7	T554
13-9	173.3	34.6	T556
13-10			T556
13-11	55.3	23.7	T556
13-12	7.0	14.4	T556
13-13			T562
13-14			T562
13-15			T563
13-16	321.4	51.6	T563

**Alloy
14**

Archived

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)	210.0 Gain(9)
14-1	Zr-0.4Fe-0.2Cr	9.3	17.2	21.4	24.9	27.2	29.7	31.3	33.4
14-2	Zr-0.4Fe-0.2Cr	11.3	20.2	24.5	28.1	30.6	32.9	34.7	36.7
14-3	Zr-0.4Fe-0.2Cr	10.5	19.2	23.5	26.9	29.4	32.1	33.5	35.4
14-7	Zr-0.4Fe-0.2Cr	12.7	21.2						
14-8	Zr-0.4Fe-0.2Cr	11.9	21.1						
T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(7)	147.1 Gain(8)	180.0 Gain(9)	
14-4	Zr-0.4Fe-0.2Cr	10.5	19.6	23.9	27.3	30.2	32.0	34.4	
T564 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	60.0 Gain(3)	90.0 Gain(4)	120.0 Gain(5)	147.1 Gain(6)	180.0 Gain(7)	210.0 Gain(8)
14-5	Zr-0.4Fe-0.2Cr	10.4	19.9	24.3	27.4	29.9	32.3	34.5	36.3
14-6	Zr-0.4Fe-0.2Cr	10.2	18.6	23.5	26.5	29.2	31.1	33.2	35.1
T556 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	21.0 Gain(5)	28.0 Gain(6)	41.2 Gain(7)	55.3 Gain(8)
14-9	Zr-0.4Fe-0.2Cr	10.3	12.8	15.0	17.5	19.3	20.8	22.7	24.5
14-10	Zr-0.4Fe-0.2Cr	9.0	11.2	13.6	16.1	17.8	19.4	21.3	23.2
14-11	Zr-0.4Fe-0.2Cr	9.3	11.9	14.0	16.5	18.3	19.7	21.9	24.0
14-12	Zr-0.4Fe-0.2Cr	9.5	12.1	14.5					
T562 Sample	Alloy	32.0 Gain(1)	62.0 Gain(2)	92.0 Gain(3)	119.1 Gain(4)	152.0 Gain(5)	182.0 Gain(6)	212.0 Gain(7)	242 Gain(8)
14-13	Zr-0.4Fe-0.2Cr	20.1	24.3	27.2	29.9	32.1	33.8	35.9	37.6
14-14	Zr-0.4Fe-0.2Cr	18.2	21.7	25.1	27.8	30.1	32.0	33.8	35.2
T563 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	28.0 Gain(5)	42.0 Gain(6)	56.0 Gain(7)	83.1 Gain(8)
14-15	Zr-0.4Fe-0.2Cr	7.5	9.6	11.9	14.6	18.1	20.3	22.0	25.2
14-16	Zr-0.4Fe-0.2Cr	7.8	10.3	12.7	15.3	18.7	21.0	22.9	26.1

**Alloy
14**

Alloy 14 (cont -1)

T554 Sample	Alloy	240.0 Gain(10)	270.0 Gain(11)	300.0 Gain(12)	330.0 Gain(13)	353.0 Gain(14)	381.0 Gain(15)	409.0 Gain(16)	437.0 Gain(18)
14-1	Zr-0.4Fe-0.2Cr	34.9	36.7	38.2	39.0	40.1	41.5	43.2	44.9
14-2	Zr-0.4Fe-0.2Cr	38.4	39.9	41.2	42.2	43.3			
14-3	Zr-0.4Fe-0.2Cr	36.9	38.5	39.8	40.9	41.9	43.0	44.4	
14-7	Zr-0.4Fe-0.2Cr								
14-8	Zr-0.4Fe-0.2Cr								

T554 Sample	Alloy								
14-4	Zr-0.4Fe-0.2Cr								

T564 Sample	Alloy	240 Gain(9)	270.0 Gain(10)	300.0 Gain(11)	323 Gain(12)	351.0 Gain(13)	379 Gain(14)	407 Gain(16)	435.0 Gain(17)
14-5	Zr-0.4Fe-0.2Cr	38.0	39.3	40.5	41.4	42.7	44.0	45.4	46.3
14-6	Zr-0.4Fe-0.2Cr	36.6	38.3	39.4	39.9	41.2	42.4	43.7	44.9

T556 Sample	Alloy	69.3 Gain(9)	83.3 Gain(10)	113.3 Gain(11)	140.4 Gain(12)	173.3 Gain(13)	203.3 Gain(14)	233.3 Gain(15)	263.3 Gain(16)
14-9	Zr-0.4Fe-0.2Cr	26.2	27.7	30.4	33.2	35.2			
14-10	Zr-0.4Fe-0.2Cr	25.0	26.3	29.3	31.1	33.2	35.0	36.6	38.0
14-11	Zr-0.4Fe-0.2Cr								
14-12	Zr-0.4Fe-0.2Cr								

T562 Sample	Alloy	272 Gain(9)	295.0 Gain(10)	323.0 Gain(11)	351.0 Gain(12)	379.0 Gain(14)	407 Gain(15)	435.0 Gain(16)	
14-13	Zr-0.4Fe-0.2Cr	38.8	39.5	41.0	42.3	43.6	44.8	45.9	
14-14	Zr-0.4Fe-0.2Cr	36.2	37.3	38.4	39.7	40.8	42.1	43.0	

T563 Sample	Alloy	116.0 Gain(9)	146.0 Gain(10)	176 Gain(11)	206.0 Gain(12)	236.0 Gain(13)	259 Gain(14)	287 Gain(15)	315.0 Gain(16)
14-15	Zr-0.4Fe-0.2Cr	28.3	30.2	32.2	33.6	35.0	36.0	37.1	38.3
14-16	Zr-0.4Fe-0.2Cr	29.1	31.2	32.9	35.0	35.9	37.0	38.3	39.6

Alloy 14 (cont -2)

T554 Sample	Alloy	465.0 Gain(19)	493.0 Gain(20)
14-1	Zr-0.4Fe-0.2Cr	46.6	47.9
14-2	Zr-0.4Fe-0.2Cr		
14-3	Zr-0.4Fe-0.2Cr		
14-7	Zr-0.4Fe-0.2Cr		
14-8	Zr-0.4Fe-0.2Cr		

T554 Sample	Alloy		
14-4	Zr-0.4Fe-0.2Cr		

T564 Sample	Alloy	463 Gain(18)
14-5	Zr-0.4Fe-0.2Cr	47.7
14-6	Zr-0.4Fe-0.2Cr	46.1

T556 Sample	Alloy	293.3 Gain(17)	316.3 Gain(18)	344.3 Gain(19)
14-9	Zr-0.4Fe-0.2Cr			
14-10	Zr-0.4Fe-0.2Cr	39.1	40.0	41.2
14-11	Zr-0.4Fe-0.2Cr			
14-12	Zr-0.4Fe-0.2Cr			

T562 Sample	Alloy
14-13	Zr-0.4Fe-0.2Cr
14-14	Zr-0.4Fe-0.2Cr

T563 Sample	Alloy	343.0 Gain(18)	371.0 Gain(19)	399.0 Gain(20)
14-15	Zr-0.4Fe-0.2Cr	39.7	41.1	42.2
14-16	Zr-0.4Fe-0.2Cr	40.8	42.1	43.4

Alloy 14 (cont -3) Summary and final results

Archive	Days	Wt Gain	Test
14-1			T554
14-2	353.0	43.3	T554
14-3	409.0	44.4	T554
14-4	180.0	34.4	T554
14-5			T564
14-6			T564
14-7	28.0	21.2	T554
14-8	28.0	21.1	T554
14-9	173.3	35.2	T556
14-10			T556
14-11	55.3	24.0	T556
14-12	7.0	14.5	T556
14-13			T562
14-14			T562
14-15			T563
14-16			T563

Alloys 21-24 ZrCrFe

Alloy 21

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
21-1	Zr-0.5Cr	7.2	171.2	502.4	955.9				
21-2	Zr-0.5Cr	6.8	20.1	283.2	736.2				
21-3	Zr-0.5Cr	6.4	14.3	269.7	725.6				
21-4	Zr-0.5Cr	7.9	14.6	237.8	701.0				
21-7	Zr-0.5Cr	7.5	14.4						
21-8	Zr-0.5Cr	11.0	223.5						

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
21-5	Zr-0.5Cr	9.7	177.5	589.1					
21-6	Zr-0.5Cr	7.1	25.2	343.8					

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2	55.3
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
21-9	Zr-0.5Cr	10.7	17.9	50.8	138.7	225.6	326.2	509.9	
21-10	Zr-0.5Cr	11.0	22.3	68.0	157.4	243.8			
21-11	Zr-0.5Cr	11.0	21.6	66.5	155.0				
21-12	Zr-0.5Cr	10.7	17.9	53.0					

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0	242
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
21-13	Zr-0.5Cr	14.4	222.2						
21-14	Zr-0.5Cr	14.5	271.1						

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0	83.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
21-15	Zr-0.5Cr	5.7	7.1	8.7	10.7	46.7			
21-16	Zr-0.5Cr	5.6	7.2	8.6	10.8	91.3	242.2		

Alloy 21**Summary and final results**

Archive	Days	Wt Gain	Test
21-1	90.0	955.9	T554
21-2	90.0	736.2	T554
21-3	90.0	725.6	T554
21-4	90.0	701.0	T554
21-5	60.0	589.1	T564
21-6	60.0	343.8	T564
21-7	28.0	14.4	T554
21-8	28.0	223.5	T554
21-9	41.2	509.9	T556
21-10	21.0	243.8	T556
21-11	14.0	155.0	T556
21-12	7.0	53.0	T556
21-13	62.0	222.2	T562
21-14	62.0	271.1	T562
21-15	28.0	46.7	T563
21-16	42.0	242.2	T563

T554		3.0	28.0	56.0	86.0	116.0	143.1	176.0	
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(6)	Gain(7)	Gain(8)	Gain(9)	
22-1	Zr-0.5Cr-0.2Fe	7.5	14.0	19.1	22.2	76.9	156.6	234.3	
22-2	Zr-0.5Cr-0.2Fe	8.3	14.5	19.4	23.1	90.6	172.8	247.2	

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
22-3	Zr-0.5Cr-0.2Fe	8.6	16.0	25.8	64.3	131.2	218.8	297.6	373.9
22-4	Zr-0.5Cr-0.2Fe	8.5	15.0	21.3	52.7				
22-7	Zr-0.5Cr-0.2Fe	7.9	14.3						
22-8	Zr-0.5Cr-0.2Fe	8.4	15.3						

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0	210.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
22-5	Zr-0.5Cr-0.2Fe	8.2	15.2	19.9	23.8	96.7	184.0	263.0	329.9
22-6	Zr-0.5Cr-0.2Fe	9.2	17.4	23.8	27.0	101.5			

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2	55.3
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
22-9	Zr-0.5Cr-0.2Fe	12.1	14.5	16.4	18.3	19.6	20.6	22.0	23.7
22-10	Zr-0.5Cr-0.2Fe	11.4	14.0	15.9	18.2	19.6	21.1	22.9	25.3
22-11	Zr-0.5Cr-0.2Fe	11.6	14.0	15.7	17.8	19.3	20.3	21.9	24.0
22-12	Zr-0.5Cr-0.2Fe	11.1	13.4	15.5					

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0	242
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
22-13	Zr-0.5Cr-0.2Fe	16.9	21.4	81.9	158.4	238.1	307.0		
22-14	Zr-0.5Cr-0.2Fe	14.9	19.7	72.1	145.7	218.5	283.4	371.4	

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0	83.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
22-15	Zr-0.5Cr-0.2Fe	5.8	7.4	9.1	10.8	13.4	15.6	18.4	36.3
22-16	Zr-0.5Cr-0.2Fe	6.4	8.3	10.4	12.9	17.0	30.3		

Alloy 22 (cont - 1)

T554

Sample	Alloy
22-1	Zr-0.5Cr-0.2Fe
22-2	Zr-0.5Cr-0.2Fe

T554

Sample	Alloy	240.0 Gain(10)	270.0 Gain(11)
22-3	Zr-0.5Cr-0.2Fe		
22-4	Zr-0.5Cr-0.2Fe		
22-7	Zr-0.5Cr-0.2Fe		
22-8	Zr-0.5Cr-0.2Fe		

T564

Sample	Alloy	240 Gain(9)	270.0 Gain(10)
22-5	Zr-0.5Cr-0.2Fe	413.2	
22-6	Zr-0.5Cr-0.2Fe		

T556

Sample	Alloy	69.3 Gain(9)	83.3 Gain(10)
22-9	Zr-0.5Cr-0.2Fe	39.2	86.4
22-10	Zr-0.5Cr-0.2Fe	60.2	105.9
22-11	Zr-0.5Cr-0.2Fe		
22-12	Zr-0.5Cr-0.2Fe		

T562

Sample	Alloy	272 Gain(9)	295.0 Gain(10)
22-13	Zr-0.5Cr-0.2Fe		
22-14	Zr-0.5Cr-0.2Fe		

T563

Sample	Alloy	116.0 Gain(9)	146.0 Gain(10)
22-15	Zr-0.5Cr-0.2Fe	55.0	
22-16	Zr-0.5Cr-0.2Fe		

Summary and final results			
Archive	Days	Wt Gain	Test
22-1	176.0	234.3	T554
22-2	176.0	247.2	T554
22-3	210.0	373.9	T554
22-4	90.0	52.7	T554
22-5	240.0	413.2	T564
22-6	120.0	101.5	T564
22-7	28.0	14.3	T554
22-8	28.0	15.3	T554
22-9	83.3	86.4	T556
22-10	83.3	105.9	T556
22-11	55.3	24.0	T556
22-12	7.0	15.5	T556
22-13	182.0	307.0	T562
22-14	212.0	371.4	T562
22-15	116.0	55.0	T563
22-16	42.0	30.3	T563

**Alloy
23**

Archived

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)
23-1	Zr-1.0Cr	10.0	60.9	231.7	606.8			
23-2	Zr-1.0Cr	7.2	13.6	42.1	309.5			
23-3	Zr-1.0Cr	7.3	13.7	51.7	322.7			
23-4	Zr-1.0Cr	9.5	17.3	119.0	449.8			
23-7	Zr-1.0Cr	8.8	17.0					
23-8	Zr-1.0Cr	8.9	15.9					

T564 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	60.0 Gain(3)	90.0 Gain(4)	120.0 Gain(5)	147.1 Gain(6)	180.0 Gain(7)
23-5	Zr-1.0Cr	9.0	17.3	131.9				
23-6	Zr-1.0Cr	7.7	14.6	19.0	202.8			

T556 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	21.0 Gain(5)	28.0 Gain(6)	41.2 Gain(7)
23-9	Zr-1.0Cr	11.0	13.2	15.6	17.4	18.8	19.9	21.5
23-10	Zr-1.0Cr	10.2	12.9	15.3	17.5	19.3	20.3	22.3
23-11	Zr-1.0Cr	8.8	11.6	13.3	15.5	17.2	18.1	20.6
23-12	Zr-1.0Cr	8.7	11.8	14.2				

T562 Sample	Alloy	32.0 Gain(1)	62.0 Gain(2)	92.0 Gain(3)	119.1 Gain(4)	152.0 Gain(5)	182.0 Gain(6)	212.0 Gain(7)
23-13	Zr-1.0Cr	14.9	46.0					
23-14	Zr-1.0Cr	16.9	27.1	227.4				

T563 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	28.0 Gain(5)	42.0 Gain(6)	56.0 Gain(7)
23-15	Zr-1.0Cr	5.5	7.2	8.9	10.7	13.2	15.5	21.9
23-16	Zr-1.0Cr	5.5	7.2	8.9	10.9	13.5	15.3	18.4

Alloy 23**Alloy 23 (cont.)**

T554		210.0	240.0	270.0	
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	
23-1	Zr-1.0Cr				
23-2	Zr-1.0Cr				
23-3	Zr-1.0Cr				
23-4	Zr-1.0Cr				
23-7	Zr-1.0Cr				
23-8	Zr-1.0Cr				

T564		210.0	240	270.0	300.0
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)
23-5	Zr-1.0Cr				
23-6	Zr-1.0Cr				

T556		55.3	69.3	83.3	113.3
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)
23-9	Zr-1.0Cr	23.4	106.7	265.2	
23-10	Zr-1.0Cr	78.2	229.1	394.5	
23-11	Zr-1.0Cr	41.9			
23-12	Zr-1.0Cr				

T562		242	272	295.0	323.0
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)
23-13	Zr-1.0Cr				
23-14	Zr-1.0Cr				

T563		83.1	116.0	146.0	176
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)
23-15	Zr-1.0Cr	146.9	271.8	430.6	
23-16	Zr-1.0Cr	132.4	256.6	422.4	635.1

Alloy 23**Summary and final results**

Archive	Days	Wt Gain	Test
23-1	90.0	606.8	T554
23-2	90.0	309.5	T554
23-3	90.0	322.7	T554
23-4	90.0	449.8	T554
23-5	60.0	131.9	T564
23-6	90.0	202.8	T564
23-7	28.0	17.0	T554
23-8	28.0	15.9	T554
23-9	83.3	265.2	T556
23-10	83.3	394.5	T556
23-11	55.3	41.9	T556
23-12	7.0	14.2	T556
23-13	62.0	46.0	T562
23-14	92.0	227.4	T562
23-15	146.0	430.6	T563
23-16	176.0	635.1	T563

Alloy 24

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
24-1	Zr-1.0Cr-0.2Fe	10.7	18.8	22.4	26.1	28.1	31.5	
24-2	Zr-1.0Cr-0.2Fe	10.2	18.2	21.6	24.9	26.6	30.7	42.4
24-4	Zr-1.0Cr-0.2Fe	10.4	18.2	21.8	25.3	27.1	31.6	44.1
24-7	Zr-1.0Cr-0.2Fe	11.7	19.8					
24-8	Zr-1.0Cr-0.2Fe	10.3	18.1					

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
24-3	Zr-1.0Cr-0.2Fe	10.8	18.5	22.3	25.3	27.8	33.9	47.7

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
24-5	Zr-1.0Cr-0.2Fe	10.2	17.9	21.9	24.6	26.9	28.9	31.2
24-6	Zr-1.0Cr-0.2Fe	12.0	20.0	24.4	27.0	29.0		

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
24-9	Zr-1.0Cr-0.2Fe	11.5	14.4	16.6	18.5	20.0	21.7	23.3
24-10	Zr-1.0Cr-0.2Fe	11.2	13.9	15.7	18.1	19.4	20.9	22.4
24-11	Zr-1.0Cr-0.2Fe	12.7	15.3	17.1	19.0	20.5	21.9	23.5
24-12	Zr-1.0Cr-0.2Fe	11.3	13.8	15.9				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
24-13	Zr-1.0Cr-0.2Fe	18.4	22.1	24.4	26.6	29.0	39.9	
24-14	Zr-1.0Cr-0.2Fe	15.9	19.8	22.5	24.5	26.4	36.2	53.1

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
24-15	Zr-1.0Cr-0.2Fe	6.3	8.3	10.1	12.2	15.0	16.8	18.7

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
24-16	Zr-1.0Cr-0.2Fe	6.6	8.3	10.3	12.4	15.2	17.0	18.7

**Alloy
24 (cont - 1.)**

T554 Sample	Alloy	210.0 Gain(9)	240.0 Gain(10)	270.0 Gain(11)	300.0 Gain(12)	330.0 Gain(13)	353.0 Gain(14)	381.0 Gain(15)	409.0 Gain(16)
24-1	Zr-1.0Cr-0.2Fe								
24-2	Zr-1.0Cr-0.2Fe	52.5	58.3	67.6					
24-4	Zr-1.0Cr-0.2Fe	54.0							
24-7	Zr-1.0Cr-0.2Fe								
24-8	Zr-1.0Cr-0.2Fe								
T554 Sample	Alloy	210.0 Gain(9)	240.0 Gain(10)	270.0 Gain(11)	300.0 Gain(12)	330.0 Gain(13)	353.0 Gain(14)	381.0 Gain(15)	409.0 Gain(16)
24-3	Zr-1.0Cr-0.2Fe	55.9	59.8	65.3	72.8	85.0	99.3	119.0	140.8
T564 Sample	Alloy	210.0 Gain(8)	240 Gain(9)	270.0 Gain(10)	300.0 Gain(11)	323 Gain(12)	351.0 Gain(13)	379 Gain(14)	407 Gain(16)
24-5	Zr-1.0Cr-0.2Fe	41.6	56.7	70.7	87.3	100.9	120.1	142.7	167.3
24-6	Zr-1.0Cr-0.2Fe								
T556 Sample	Alloy	55.3 Gain(8)	69.3 Gain(9)	83.3 Gain(10)	113.3 Gain(11)	140.4 Gain(12)	173.3 Gain(13)	203.3 Gain(14)	233.3 Gain(15)
24-9	Zr-1.0Cr-0.2Fe	24.9	26.3	27.5	29.7	32.6	35.0		
24-10	Zr-1.0Cr-0.2Fe	24.2	25.4	26.6	29.5	33.9	43.0		
24-11	Zr-1.0Cr-0.2Fe	24.5							
24-12	Zr-1.0Cr-0.2Fe								
T562 Sample	Alloy	242 Gain(8)	272 Gain(9)	295.0 Gain(10)	323.0 Gain(11)	351.0 Gain(12)	379.0 Gain(14)	407 Gain(15)	435.0 Gain(16)
24-13	Zr-1.0Cr-0.2Fe								
24-14	Zr-1.0Cr-0.2Fe	73.1	95.7	114.0	138.9	166.1	194.2	222.1968156	249.9
T563 Sample	Alloy	83.1 Gain(8)	116.0 Gain(9)	146.0 Gain(10)	176 Gain(11)	206.0 Gain(12)	236.0 Gain(13)	259 Gain(14)	287 Gain(15)
24-15	Zr-1.0Cr-0.2Fe	20.9	23.8	25.3					
T563 Sample	Alloy	83.1 Gain(8)	116.0 Gain(9)	146.0 Gain(10)	176 Gain(11)	206.0 Gain(12)	229.0 Gain(14)	257.0 Gain(15)	285.0 Gain(16)
24-16	Zr-1.0Cr-0.2Fe	21.3	23.6	25.6	28.6	44.3	58.5	78.6	101.8

Alloy 24 (cont - 2)

T554 Sample	Alloy	437.0 Gain(18)	465.0 Gain(19)
24-1	Zr-1.0Cr-0.2Fe		
24-2	Zr-1.0Cr-0.2Fe		
24-4	Zr-1.0Cr-0.2Fe		
24-7	Zr-1.0Cr-0.2Fe		
24-8	Zr-1.0Cr-0.2Fe		

T554 Sample	Alloy	415.4 Gain(17)	437.0 Gain(18)
24-3	Zr-1.0Cr-0.2Fe	145.6	

T564 Sample	Alloy	435.0 Gain(17)	463 Gain(18)
24-5	Zr-1.0Cr-0.2Fe	192.9	217.3
24-6	Zr-1.0Cr-0.2Fe		

T556 Sample	Alloy	263.3 Gain(16)	293.3 Gain(17)
24-9	Zr-1.0Cr-0.2Fe		
24-10	Zr-1.0Cr-0.2Fe		
24-11	Zr-1.0Cr-0.2Fe		
24-12	Zr-1.0Cr-0.2Fe		

T562 Sample	Alloy		
24-13	Zr-1.0Cr-0.2Fe		
24-14	Zr-1.0Cr-0.2Fe		

T563 Sample	Alloy	315.0 Gain(16)	343.0 Gain(18)
24-15	Zr-1.0Cr-0.2Fe		

**T563
Sample Alloy**

30 days behind

Alloy 24		Summary and final results	
Archive	Days	Wt Gain	Test
24-1	150.0	31.5	T554
24-2	270.0	67.6	T554
24-3	415.4	145.6	T554
24-4	210.0	54.0	T554
24-5			T564
24-6	120.0	29.0	T564
24-7	28.0	19.8	T554
24-8	28.0	18.1	T554
24-9	173.3	35.0	T556
24-10	173.3	43.0	T556
24-11	55.3	24.5	T556
24-12	7.0	15.9	T556
24-13	182.0	39.9	T562
24-14			T562
24-15	146.0	25.3	T563
24-16	285.0	101.8	T563

Alloys 31-34 - ZrCuMo

Alloy 31**Archived**

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
31-1	Zr-0.5Cu	16.8	101.9	212.9	331.1	428.6		
31-2	Zr-0.5Cu	14.1	84.0	190.9	307.6			
31-3	Zr-0.5Cu	14.6	32.6	86.5	192.7			
31-4	Zr-0.5Cu	14.0	26.3	64.7	172.1	264.8		
31-7	Zr-0.5Cu	15.7	50.1					
31-8	Zr-0.5Cu	14.7	52.8					

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
31-5	Zr-0.5Cu	20.1	96.7	204.7	296.5			
31-6	Zr-0.5Cu	16.0	69.2	166.4	258.3			

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
31-9	Zr-0.5Cu	13.3	15.8	18.0	20.4	22.0	23.8	25.6
31-10	Zr-0.5Cu	14.2	16.9	19.0	21.5	23.2	24.7	26.7
31-11	Zr-0.5Cu	13.6	16.0	18.1	20.6	22.3	23.8	25.8
31-12	Zr-0.5Cu	14.3	16.9	19.0				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
31-13	Zr-0.5Cu	23.2	27.4	31.8	53.1	146.5		
31-14	Zr-0.5Cu	22.7	26.4	30.2	33.6	37.1	111.2	

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
31-15	Zr-0.5Cu	13.6	16.6	19.6	22.7	28.4	37.2	51.4
31-16	Zr-0.5Cu	12.5	15.8	20.5	26.3	44.6	69.6	

Alloy 31 (cont.)

T554		210.0	240.0	270.0					
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)					
31-1	Zr-0.5Cu								
31-2	Zr-0.5Cu								
31-3	Zr-0.5Cu								
31-4	Zr-0.5Cu								
31-7	Zr-0.5Cu								
31-8	Zr-0.5Cu								
T564		210.0	240	270.0	300.0	323	351.0	379	407
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)
31-5	Zr-0.5Cu								
31-6	Zr-0.5Cu								
T556		55.3	69.3	83.3	113.3	140.4	173.3	203.3	233.3
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
31-9	Zr-0.5Cu	27.6	29.5	30.6	33.4	35.5	37.9		
31-10	Zr-0.5Cu	28.9	30.3	31.7	34.5	36.8	38.9	40.6	43.1
31-11	Zr-0.5Cu	28.1							
31-12	Zr-0.5Cu								
T562		242	272	295.0	323.0	351.0	379.0	407	
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	
31-13	Zr-0.5Cu								
31-14	Zr-0.5Cu								
T563		83.1	116.0	146.0	176	206.0	236.0	259	287
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
31-15	Zr-0.5Cu								
31-16	Zr-0.5Cu								

Alloy 31		Summary and final results	
Archive	Days	Wt Gain	Test
31-1	120.0	428.6	T554
31-2	90.0	307.6	T554
31-3	90.0	192.7	T554
31-4	120.0	264.8	T554
31-5	90.0	296.5	T564
31-6	90.0	258.3	T564
31-7	28.0	50.1	T554
31-8	28.0	52.8	T554
31-9	173.3	37.9	T556
31-10	233.3	43.1	T556
31-11	55.3	28.1	T556
31-12	7.0	19.0	T556
31-13	152.0	146.5	T562
31-14	182.0	111.2	T562
31-15	56.0	51.4	T563
31-16	42.0	69.6	T563

Alloy
32

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
32-1	Zr-0.5Cu-0.5Mo	23.6	146.1	286.5	445.1	566.4		
32-2	Zr-0.5Cu-0.5Mo	17.5	134.0	263.5	399.1	500.7		
32-3	Zr-0.5Cu-0.5Mo	23.3	153.9	286.2	435.8			
32-4	Zr-0.5Cu-0.5Mo	23.6	125.0	250.5	396.5			
32-7	Zr-0.5Cu-0.5Mo	23.3	149.5					
32-8	Zr-0.5Cu-0.5Mo	21.7	147.5					

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
32-5	Zr-0.5Cu-0.5Mo	22.5	157.5	299.3				
32-6	Zr-0.5Cu-0.5Mo	22.3	143.6	284.6	402.1			

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
32-9	Zr-0.5Cu-0.5Mo	14.6	17.7	20.3	23.4	25.7	27.7	30.2
32-10	Zr-0.5Cu-0.5Mo	13.8	17.0	19.6	22.7	24.8	26.2	29.0
32-11	Zr-0.5Cu-0.5Mo	13.4	16.7	20.2	24.1	27.0	29.3	32.9
32-12	Zr-0.5Cu-0.5Mo	13.7	17.2	20.2				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
32-13	Zr-0.5Cu-0.5Mo	73.1	129.4					
32-14	Zr-0.5Cu-0.5Mo	23.8	28.1	31.8	36.1	105.9		

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
32-15	Zr-0.5Cu-0.5Mo	12.4	17.8	29.6	47.3	84.1	128.2	
32-16	Zr-0.5Cu-0.5Mo	11.8	16.7	27.8	46.8	88.2	140.2	

Alloy 32**(cont.)**

T554		240.0	270.0					
Sample	Alloy	Gain(10)	Gain(11)					
32-1	Zr-0.5Cu-0.5Mo							
32-2	Zr-0.5Cu-0.5Mo							
32-3	Zr-0.5Cu-0.5Mo							
32-4	Zr-0.5Cu-0.5Mo							
32-7	Zr-0.5Cu-0.5Mo							
32-8	Zr-0.5Cu-0.5Mo							

T564		240	270.0	300.0	323	351.0	379	407
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)
32-5	Zr-0.5Cu-0.5Mo							
32-6	Zr-0.5Cu-0.5Mo							

T556		69.3	83.3	113.3	140.4	173.3	203.3	233.3
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
32-9	Zr-0.5Cu-0.5Mo	34.4	36.2	39.3	41.7	56.4	110.7	198.3
32-10	Zr-0.5Cu-0.5Mo	33.6	35.2	38.4	40.8	60.7	126.9	
32-11	Zr-0.5Cu-0.5Mo							
32-12	Zr-0.5Cu-0.5Mo							

T562		272	295.0	323.0	351.0	379.0	407	
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	
32-13	Zr-0.5Cu-0.5Mo							
32-14	Zr-0.5Cu-0.5Mo							

T563		116.0	146.0	176	206.0	236.0	259	287
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
32-15	Zr-0.5Cu-0.5Mo							
32-16	Zr-0.5Cu-0.5Mo							

Alloy 32		Summary and final results	
Archive	Days	Wt Gain	Test
32-1	120.0	566.4	T554
32-2	120.0	500.7	T554
32-3	90.0	435.8	T554
32-4	90.0	396.5	T554
32-5	60.0	299.3	T564
32-6	90.0	402.1	T564
32-7	28.0	149.5	T554
32-8	28.0	147.5	T554
32-9	233.3	198.3	T556
32-10	203.3	126.9	T556
32-11	55.3	36.6	T556
32-12	7.0	20.2	T556
32-13	62.0	129.4	T562
32-14	152.0	105.9	T562
32-15	42.0	128.2	T563
32-16	42.0	140.2	T563

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
33-1	Zr-1.0Cu	17.7	27.3	33.6	39.4	42.2	45.8	49.5
33-2	Zr-1.0Cu	23.8	33.6	38.4	43.5	46.7	50.0	54.0
33-3	Zr-1.0Cu	20.7	30.0	35.3	39.7	42.9	45.9	49.2
33-4	Zr-1.0Cu	17.4	26.5	31.5	36.4			
33-7	Zr-1.0Cu	18.4	27.7					
33-8	Zr-1.0Cu	17.4	26.7					

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
33-5	Zr-1.0Cu	18.5	27.6	33.0				
33-6	Zr-1.0Cu	18.1	26.9	32.9	36.1	38.4	41.2	44.1

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
33-9	Zr-1.0Cu	15.3	18.0	20.7	23.3	25.5	27.2	29.6
33-10	Zr-1.0Cu	15.0	17.8	20.3	23.2	25.3	26.9	29.2
33-11	Zr-1.0Cu	14.9	17.9	20.5	23.4	25.4	27.3	29.6
33-12	Zr-1.0Cu	16.0	19.3	22.0				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
33-13	Zr-1.0Cu	25.9	30.5	34.1	37.5	40.1	43.7	48.1
33-14	Zr-1.0Cu	26.1	30.1	33.7	37.1	39.8	44.0	49.2

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
33-15	Zr-1.0Cu	13.7	16.2	19.6	22.3	25.8	28.3	30.8
33-16	Zr-1.0Cu	13.9	16.7	20.0	22.9	26.6	29.2	31.0

Alloy 33 (cont-1)

T554		210.0	240.0	270.0	300.0	330.0	353.0	381.0	409
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)
33-1	Zr-1.0Cu	53.3	64.6	73.0	82.8	89.8	94.0	99.1	103.6
33-2	Zr-1.0Cu	58.0	68.4	77.5					
33-3	Zr-1.0Cu	53.1							
33-4	Zr-1.0Cu								
33-7	Zr-1.0Cu								
33-8	Zr-1.0Cu								

T564		210.0	240	270.0	300.0	323	351.0	379	407
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(16)
33-5	Zr-1.0Cu								
33-6	Zr-1.0Cu	45.9	48.5	53.2	59.0	64.8	73.2	84.2	95.1

T556		55.3	69.3	83.3	113.3	140.4	173.3	203.3	233.3
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
33-9	Zr-1.0Cu	31.8	33.5	35.1	38.1	40.3	42.9	44.7	47.0
33-10	Zr-1.0Cu	31.3	33.3	34.8	37.9	40.4	42.4		
33-11	Zr-1.0Cu	31.6							
33-12	Zr-1.0Cu								

T562		242	272	295.0	323.0	351.0	379.0	407	435.0
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)	Gain(16)
33-13	Zr-1.0Cu	61.0	71.1	78.1	85.7	93.1	99.5	106.6	
33-14	Zr-1.0Cu	65.7	76.2	83.2	90.1	96.8	103.5	111.4	117.6

T563		83.1	116.0	146.0	176	206.0	236.0	259	287
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
33-15	Zr-1.0Cu	34.4	37.7	39.9	43.0	49.2	56.9	65.1	76.1
33-16	Zr-1.0Cu	35.2	38.3	40.8	44.5	51.0	59.6	69.1	81.6

Alloy 33 (cont-2)

T554
Sample

Alloy

33-1	Zr-1.0Cu								
33-2	Zr-1.0Cu								
33-3	Zr-1.0Cu								
33-4	Zr-1.0Cu								
33-7	Zr-1.0Cu								
33-8	Zr-1.0Cu								

T564
Sample

Alloy

435.0
Gain(17)

463
Gain(18)

33-5	Zr-1.0Cu								
33-6	Zr-1.0Cu	105.9	113.9						

T556
Sample

Alloy

263.3
Gain(16)

293.3
Gain(17)

316.3
Gain(18)

344.3
Gain(19)

372.3
Gain(20)

400.3
Gain(22)

428.3
Gain(23)

456.25
Gain(24)

33-9	Zr-1.0Cu	49.6	52.3	54.6	57.8	61.9	67.1	74.2	80.8
33-10	Zr-1.0Cu								
33-11	Zr-1.0Cu								
33-12	Zr-1.0Cu								

T562
Sample

Alloy

33-13	Zr-1.0Cu
33-14	Zr-1.0Cu

T563
Sample

Alloy

315.0
Gain(16)

343.0
Gain(18)

371.0
Gain(19)

399.0
Gain(20)

33-15	Zr-1.0Cu	88.5	99.7	110.8	120.6
33-16	Zr-1.0Cu	93.6	105.2	116.7	126.6

Alloy 33		Summary and final results	
Archive	Days	Wt Gain	Test
33-1	409.0	103.6	T554
33-2	270.0	77.5	T554
33-3	210.0	53.1	T554
33-4	90.0	36.4	T554
33-5	60.0	33.0	T564
33-6			T564
33-7	28.0	27.7	T554
33-8	28.0	26.7	T554
33-9			T556
33-10	173.3	42.4	T556
33-11	55.3	31.6	T556
33-12	7.0	22.0	T556
33-13	407.0	106.6	T562
33-14			T562
33-15			T563
33-16			T563

Alloy 34

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
34-1	Zr-1.0Cu-0.5Mo	17.9	34.3	51.5	73.7	92.7		
34-2	Zr-1.0Cu-0.5Mo	18.0	28.8	34.9	42.1	47.7	68.7	
34-3	Zr-1.0Cu-0.5Mo	17.6	28.1	35.6	45.6	53.9	74.9	108.3
34-4	Zr-1.0Cu-0.5Mo	18.1	28.6	34.9	42.0	47.4	66.1	96.3
34-7	Zr-1.0Cu-0.5Mo	18.1	29.0					
34-8	Zr-1.0Cu-0.5Mo	19.0	29.2					

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
34-5	Zr-1.0Cu-0.5Mo	18.3	29.3	35.8	40.7	48.2	68.2	92.9
34-6	Zr-1.0Cu-0.5Mo	17.3	29.7	42.0	54.9	74.6	99.5	147.8

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
34-9	Zr-1.0Cu-0.5Mo	14.1	17.5	20.9	24.0	26.6	28.4	31.4
34-10	Zr-1.0Cu-0.5Mo	14.6	17.9	21.2	24.6	27.1	29.2	32.0
34-11	Zr-1.0Cu-0.5Mo	14.4	17.8	20.9	24.3	26.5	28.9	31.6
34-12	Zr-1.0Cu-0.5Mo	13.9	17.4	20.3				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
34-13	Zr-1.0Cu-0.5Mo	24.8	29.5	33.7	38.1	49.1	74.2	
34-14	Zr-1.0Cu-0.5Mo	24.7	29.3	33.3	37.1	47.2	73.1	107.1

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
34-15	Zr-1.0Cu-0.5Mo	11.9	14.8	17.8	21.1	25.4	29.1	34.0
34-16	Zr-1.0Cu-0.5Mo	12.2	15.5	18.4	21.8	26.0	30.1	34.6

Alloy 34**Alloy 34 (cont.)**

T554		210.0	240.0	270.0				
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)				
34-1	Zr-1.0Cu-0.5Mo							
34-2	Zr-1.0Cu-0.5Mo							
34-3	Zr-1.0Cu-0.5Mo	168.5						
34-4	Zr-1.0Cu-0.5Mo	145.0	710.9					
34-7	Zr-1.0Cu-0.5Mo							
34-8	Zr-1.0Cu-0.5Mo							

T564		210.0	240	270.0	300.0	323	351.0	379
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)
34-5	Zr-1.0Cu-0.5Mo							
34-6	Zr-1.0Cu-0.5Mo	633.7						

T556		55.3	69.3	83.3	113.3	140.4	173.3	203.3
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)
34-9	Zr-1.0Cu-0.5Mo	33.9	36.4	38.5	43.0	51.7	105.6	355.1
34-10	Zr-1.0Cu-0.5Mo	34.7	34.7	36.9	41.3	45.1	57.1	
34-11	Zr-1.0Cu-0.5Mo	34.0						
34-12	Zr-1.0Cu-0.5Mo							

T562		242	272	295.0	323.0	351.0	379.0	407
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(14)	Gain(15)
34-13	Zr-1.0Cu-0.5Mo							
34-14	Zr-1.0Cu-0.5Mo							

T563		83.1	116.0	146.0	176	206.0	236.0	259
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)
34-15	Zr-1.0Cu-0.5Mo	49.6	73.4	145.9				
34-16	Zr-1.0Cu-0.5Mo	49.9	77.7	140.8	227.5			

Alloy 34		Summary and final results	
Archive	Days	Wt Gain	Test
34-1	120.0	92.7	T554
34-2	150.0	68.7	T554
34-3	210.0	168.5	T554
34-4	240.0	710.9	T554
34-5	180.0	92.9	T564
34-6	210.0	633.7	T564
34-7	28.0	29.0	T554
34-8	28.0	29.2	T554
34-9	203.3	355.1	T556
34-10	173.3	57.1	T556
34-11	55.3	34.0	T556
34-12	7.0	20.3	T556
34-13	182.0	74.2	T562
34-14	212.0	107.1	T562
34-15	146.0	145.9	T563
34-16	176.0	227.5	T563

Alloys 41-43 and Zircaloy-4 control Reference alloys

**Alloy
41**

Archived

T554 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	56.0 Gain(3)	90.0 Gain(5)	120.0 Gain(6)	150.0 Gain(7)	177.1 Gain(8)	210.0 Gain(9)	240.0 Gain(10)
41-1	Sponge Zr	21.7								
41-2	Sponge Zr	21.3								
41-3	Sponge Zr	19.3								
41-4	Sponge Zr	12.0								
41-7	Sponge Zr	20.6								
41-8	Sponge Zr	24.2								

T564 Sample	Alloy	3.0 Gain(1)	28.0 Gain(2)	60.0 Gain(3)	90.0 Gain(4)	120.0 Gain(5)	147.1 Gain(6)	180.0 Gain(7)	210.0 Gain(8)	240 Gain(9)
41-5	Sponge Zr	21.1								
41-6	Sponge Zr	21.9								

T556 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	21.0 Gain(5)	28.0 Gain(6)	41.2 Gain(7)	55.3 Gain(8)	69.3 Gain(9)
41-9	Sponge Zr	7.1	9.5	11.4	13.2	14.8	16.1	17.8	19.0	51.6
41-10	Sponge Zr	9.5	12.0	14.1	16.3					
41-11	Sponge Zr	9.1	11.8	13.8	68.9					
41-12	Sponge Zr	9.0	11.7	15.1						

T562 Sample	Alloy	32.0 Gain(1)	62.0 Gain(2)	92.0 Gain(3)	119.1 Gain(4)	152.0 Gain(5)	182.0 Gain(6)	212.0 Gain(7)	242 Gain(8)	272 Gain(9)
41-13	Sponge Zr	-1351.7								
41-14	Sponge Zr	-1483.9								

T563 Sample	Alloy	1.0 Gain(1)	3.0 Gain(2)	7.0 Gain(3)	14.0 Gain(4)	28.0 Gain(5)	42.0 Gain(6)	56.0 Gain(7)	83.1 Gain(8)	116.0 Gain(9)
41-15	Sponge Zr	5.4	27.3							
41-16	Sponge Zr	5.5	30.0	-8.5						

Alloy 41		Summary and final results	
Archive	Days	Wt Gain	Test
41-1	3.0	21.7	T554
41-2	3.0	21.3	T554
41-3	3.0	19.3	T554
41-4	3.0	12.0	T554
41-5	3.0	21.1	T564
41-6	3.0	21.9	T564
41-7	3.0	20.6	T554
41-8	3.0	24.2	T554
41-9	69.3	51.6	T556
41-10	14.0	16.3	T556
41-11	14.0	68.9	T556
41-12	7.0	15.1	T556
41-13	32.0	-1351.7	T562
41-14	32.0	-1483.9	T562
41-15	3.0	27.3	T563
41-16	7.0	-8.5	T563

Alloy 43**Archived**

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
43-1	Zircaloy-4	16.8	25.3	30.2	33.9	36.4	42.8	59.4
43-2	Zircaloy-4	10.7	19.2	23.9	28.3	30.7	36.5	54.0
43-3	Zircaloy-4	16.4	25.0	29.2	33.2	35.5	38.9	56.9
43-4	Zircaloy-4	11.0	19.2	23.9	27.8	30.4	34.4	
43-7	Zircaloy-4	11.0	19.5					
43-8	Zircaloy-4	11.2	20.5					

T564		3.0	28.0	60.0	90.0	120.0	147.1	180.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
43-5	Zircaloy-4	10.4	18.9	24.5	27.7	30.4	36.8	54.6
43-6	Zircaloy-4	16.4	25.1	30.2	33.1	35.7	39.9	58.9

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
43-9	Zircaloy-4	11.5	13.7	16.2	18.8	20.6	22.3	24.4
43-10	Zircaloy-4	11.8	14.3	16.7	19.4	21.2	22.7	24.9
43-11	Zircaloy-4	11.2	13.4	16.0	18.4	20.4	22.2	24.1
43-12	Zircaloy-4	10.8	12.9	15.5				

T562		32.0	62.0	92.0	119.1	152.0	182.0	212.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
43-13	Zircaloy-4	20.1	24.6	28.3	30.7	43.3	55.2	60.8
43-14	Zircaloy-4	19.5	23.7	27.3	29.7	43.8	54.2	59.6

T563		1.0	3.0	7.0	14.0	28.0	42.0	56.0
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
43-15	Zircaloy-4	8.4	11.3	13.7	16.6	19.7	22.5	24.4
43-16	Zircaloy-4	9.0	11.8	14.1	16.8	20.0	22.7	24.6

Alloy 43**Alloy 43
(cont)**

T554 Sample	Alloy	210.0 Gain(9)	240.0 Gain(10)	270.0 Gain(11)	300.0 Gain(12)	330.0 Gain(13)	353.0 Gain(14)	381.0 Gain(15)
43-1	Zircaloy-4	66.1	69.9	75.5	93.0	99.8	104.0	
43-2	Zircaloy-4	60.5	64.7	69.8	85.9	95.4	98.6	103.1
43-3	Zircaloy-4	64.0	68.1					
43-4	Zircaloy-4							
43-7	Zircaloy-4							
43-8	Zircaloy-4							
T564 Sample	Alloy	210.0 Gain(8)	240 Gain(9)	270.0 Gain(10)	300.0 Gain(11)	323 Gain(12)	351.0 Gain(13)	379.0 Gain(14)
43-5	Zircaloy-4	59.3	63.1	67.6	83.4	90.1	95.1	100.1
43-6	Zircaloy-4							
T556 Sample	Alloy	55.3 Gain(8)	69.3 Gain(9)	83.3 Gain(10)	113.3 Gain(11)	146.1 Gain(13)	176.1 Gain(14)	206.1 Gain(15)
43-9	Zircaloy-4	26.6	28.3	29.7	32.6	35.6	53.6	60.8
43-10	Zircaloy-4	27.1	28.8	29.9	33.0	36.1	53.9	
43-11	Zircaloy-4	26.2						
43-12	Zircaloy-4							
T562 Sample	Alloy	242 Gain(8)	272 Gain(9)	295.0 Gain(10)	323.0 Gain(11)	351.0 Gain(12)	379.0 Gain(14)	407.0 Gain(15)
43-13	Zircaloy-4	63.8	69.8	83.1	90.9	96.3	101.4	114.2
43-14	Zircaloy-4	62.5	70.2	83.3	89.9	95.3	100.8	115.2
T563 Sample	Alloy	83.1 Gain(8)	116.0 Gain(9)	146.0 Gain(10)	176 Gain(11)	206.0 Gain(12)	236.0 Gain(13)	259 Gain(14)
43-15	Zircaloy-4	28.1	31.2					
43-16	Zircaloy-4	28.5	31.4	35.8	55.1	60.5	64.3	67.1

Alloy 43**Alloy 43
(cont)**

T554 Sample	Alloy	409.0 Gain(16)	437.0 Gain(18)	465.0 Gain(19)	493.0 Gain(20)
43-1	Zircaloy-4				
43-2	Zircaloy-4	113.5	126.4	133.6	138.9
43-3	Zircaloy-4				
43-4	Zircaloy-4				
43-7	Zircaloy-4				
43-8	Zircaloy-4				

T564 Sample	Alloy	407 Gain(16)	435.0 Gain(17)	463 Gain(18)
43-5	Zircaloy-4	107.2	122.0	128.8
43-6	Zircaloy-4			

T556 Sample	Alloy	236.1 Gain(16)	266.1 Gain(17)	289.1 Gain(18)	317.1 Gain(19)	345.1 Gain(20)	373.1 Gain(22)	27.1 days behind 401.1 Gain(23)	429.1 Gain(24)
43-9	Zircaloy-4	64.6	68.4	73.0	89.8	96.6	101.0	106.6	121.5
43-10	Zircaloy-4								
43-11	Zircaloy-4								
43-12	Zircaloy-4								

T562 Sample	Alloy	435.0 Gain(16)
43-13	Zircaloy-4	123.8
43-14	Zircaloy-4	123.5

43-16 is 28
days
behind/not
in test 41-
121/122

T563 Sample	Alloy	287 Gain(15)	315.0 Gain(16)	343.0 Gain(19)	371.0 Gain(20)
43-15	Zircaloy-4				
43-16	Zircaloy-4	77.0	90.4	95.5	100.6

Alloy 43		Summary and final results	
Archive	Days	Wt Gain	Test
43-1	353.0	104.0	T554
43-2			T554
43-3	240.0	68.1	T554
43-4	150.0	34.4	T554
43-5			T564
43-6	180.0	58.9	T564
43-7	28.0	19.5	T554
43-8	28.0	20.5	T554
43-9			T556
43-10	176.1	53.9	T556
43-11	55.3	26.2	T556
43-12	7.0	15.5	T556
43-13			T562
43-14			T562
43-15	116.0	31.2	T563
43-16			T563

Zircaloy-4

Archived

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
T554-1	Zr-4 95-9075?	10.6	23.0	30.7	38.1	43.6	60.2	69.5
T554-2	Zr-4 95-9075?	10.2	23.1	30.8	38.1	43.9	60.2	69.2
T554-4	Zr-4 95-9075?	17.9	26.3	32.3	38.0	43.3	63.7	
T554-5	Zr-4 95-9075?	10.4	22.8	29.7	36.2	48.1	62.2	70.2
T554-6	Zr-4 95-9075?	10.6	22.7	30.0	36.7	47.5	62.3	70.1
T554-7	Zr-4 95-9075?	10.5	22.7	29.9	36.2	47.9	62.5	
T554-8	Zr-4 95-9075?	10.3	22.3	29.3	36.2			

T554		3.0	28.0	56.0	90.0	120.0	150.0	177.1
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(5)	Gain(6)	Gain(7)	Gain(8)
T554-3	Zr-4 95-9075?	12.6	24.6	31.8	38.6	44.2	62.4	70.7

T556		1.0	3.0	7.0	14.0	21.0	28.0	41.2
Sample	Alloy	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)
T556-1	Zr-4 95-7051	10.1	12.2	14.7	17.4	19.1	20.8	22.9
T556-2	Zr-4 95-7051	9.2	11.8	14.1	16.7	18.4	20.0	22.0
T556-3	Zr-4 95-7051	9.5	11.9	14.1	16.9	18.6	20.1	22.3
T556-4	Zr-4 95-7051	9.6	12.1	14.5				

Zircaloy-4 (cont- 1)

T554		210.0	240.0	270.0	300.0	330.0	353.0	381.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
T554-1	Zr-4 95-9075?	77.9	88.7	102.7	110.2	119.1	129.4	138.6
T554-2	Zr-4 95-9075?	78.0	88.6	102.9	110.6	119.3	129.6	138.6
T554-4	Zr-4 95-9075?							
T554-5	Zr-4 95-9075?	79.3	92.8	105.3	112.9	123.7	134.4	142.3
T554-6	Zr-4 95-9075?	79.4	92.6	105.3	112.7	123.2	134.3	142.4
T554-7	Zr-4 95-9075?							
T554-8	Zr-4 95-9075?							

T554		210.0	240.0	270.0	300.0	330.0	353.0	381.0
Sample	Alloy	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)
T554-3	Zr-4 95-9075?	79.9	91.4	105.0	112.4	121.6	133.4	141.9

T556		55.3	69.3	83.3	113.3	140.4	173.3	203.3
Sample	Alloy	Gain(8)	Gain(9)	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)
T556-1	Zr-4 95-7051	24.8	26.4	27.9	30.7	33.1	50.9	57.0
T556-2	Zr-4 95-7051	24.3	25.7	27.1	29.7	32.1	50.0	
T556-3	Zr-4 95-7051	24.3						
T556-4	Zr-4 95-7051							

Zircaloy-4 (cont-2)

T554 Sample	409.0 Gain(16)	437.0 Gain(18)	465.0 Gain(19)	493.0 Gain(20)					
T554-1	146.0	156.8	166.9	175.0					
T554-2	146.5	157.0	167.2	175.3					
T554-4									
T554-5	150.9	163.7	173.4	180.6					
T554-6	150.8	163.3	173.2	180.6					
T554-7									
T554-8									
T554 Sample	409 Gain(16)	415.4 Gain(17)	437.0 Gain(18)						
T554-3	149.6	151.6							
T556 Sample	233.3 Gain(15)	263.3 Gain(16)	293.3 Gain(17)	316.3 Gain(18)	344.3 Gain(19)	372.3 Gain(20)	400.3 Gain(22)	428.3 Gain(23)	456.25 Gain(24)
T556-1	61.7	64.9	70.1	85.7	92.2	97.3	101.2	114.2	125.0114579
T556-2									
T556-3									
T556-4									

Alloy 43		Summary and final results	
Archive	Days	Wt Gain	
T554-1			pickled
T554-2			pickled
T554-3	415.4	151.6	pickled
T554-4	150.0	63.7	pickled
T554-5			cleaned
T554-6			cleaned
T554-7	150.0	62.5	cleaned
T554-8	90.0	36.2	cleaned
T556-1			pickled
T556-2	173.3	50.0	pickled
T556-3	55.3	24.3	pickled
T556-4	7.0	14.5	pickled

T565 autoclave (Westinghouse) - 680F Water I-NERI Corrosion Coupons

Sample	1.0	3.0	7.0	14.0	28.0	42.0
	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)
KR11-3	7.3	12.3	24.5	53.8	111.0	
KR11-4	7.5	13.8	31.6	68.9	154.3	
KR12-3	7.8	11.8	21.0	50.2	130.6	
KR12-4	7.4	12.4	31.8	41.6		
KR13-3	8.5	11.7	15.3	20.7	64.0	
KR13-4	8.2	11.9	16.6	27.2	95.2	174.7
KR14-3	8.6	11.6	15.0	21.6	98.8	
KR14-4	9.1	13.9	21.6	55.7	169.3	284.7

Samples

KR21-0.2 Nb

KR22-0.4 Nb

days	1.0	3.0	7.0	14.0	28.0	42.0	56.0	59.0	69.0	79.0	97.0	125.0	153.0
KR21-3	7.8	10.6	12.8	15.3	18.7	20.4	22.7	23.3	24.6	25.7	27.4	30.1	32.7
KR21-4	7.8	10.6	12.4	14.9	17.9	20.2	22.1	23.0	24.0	25.2	27.0	29.5	31.8
KR22-3	7.9	10.5	13.3	16.5	21.2	24.7	27.9	28.2	30.4	31.9	34.9	38.5	42.2
KK22-4	7.0	9.6	12.6	15.8	20.2	23.8	26.9	27.6	29.8	31.1	34.1	38.0	41.6

days	211.0	241.0	264.0	292.0	320.0	326.4	348.0	376.0	404.0
KR21-3	36.7	38.4	40.5	45.7	57.6	59.6	64.1	68.1	71.0
KR21-4	35.8	37.9	39.6	42.1	50.9	53.9			
KR22-3	49.8	53.4	59.8	65.5	71.7	73.1	76.7	81.4	85.2
KK22-4	48.9	52.0	56.3	60.6	66.5	68.6			

Samples
KR31-0.4Sn-0.2Nb

KR32-0.4Sn-0.4Nb

	1.0	3.0	7.0	14.0	28.0	42.0	56.0	59.0	69.0	79.0	97.0	125.0	153.0
KR31-3	8.1	10.3	13.0	15.2	18.7	20.8	23.0	23.1	24.1	25.6	27.2	29.6	31.6
KR31-4	8.6	11.0	13.3	15.9	19.0	21.3	23.2	23.6	25.1	25.9	27.8	30.5	32.3
KR32-3	8.9	11.2	13.6	16.2	19.5	21.4	23.4	23.5	25.0	25.9	27.6	29.8	32.5
KR32-4	8.9	11.3	13.4	16.2	19.0	21.3	23.3	23.6	24.8	26.0	27.9	30.3	32.4

	181.0	211.0	241.0	264.0	292.0	320.0	326.4	348.0	376.0	404.0
KR31-3	33.7	36.0	37.7	40.3	46.7	58.1	59.5	63.1	66.6	69.4
KR31-4	34.3	36.5	38.4	41.1	51.2	59.9	61.0			
KR32-3	34.0	36.2	38.4	40.3	43.3	56.1	57.7	61.8	65.5	68.3
KR32-4	34.1	36.2	38.2	40.4	47.4	58.5	59.4			

Samples
KR41-1.0Nb

KR42-1.5Nb

KR43-2.5Nb

	1.0	3.0	7.0	14.0	28.0	42.0	56.0	59.0	69.0	79.0	97.0	125.0	153.0
KR41-3	7.2	9.9	12.9	16.7	22.4	26.3	29.7	30.7	33.2	34.9	38.7	43.1	47.4
KR41-4	8.7	11.8	14.7	18.3	23.2	27.6	30.6	31.6	34.3	35.9	39.3	43.8	48.0
KR42-3	8.3	11.0	13.6	17.2	21.9	25.7	28.9	29.9	32.0	33.7	37.0	40.7	44.8
KR42-4	8.1	10.2	13.2	17.0	21.7	25.8	28.9	29.7	32.2	33.6	36.8	40.8	44.7
KR43-1	8.2	10.0	13.0	16.4	20.8	24.6	28.2	28.6	30.8	32.4	35.6	39.3	43.0
KR43-2	8.2	10.4	13.4	16.2	21.1	24.9	27.9	28.8	30.9	32.9	35.7	39.5	43.2

	181.0	211.0	241.0	264.0	292.0	320.0	326.4	348.0	376.0	404.0		
KR41-3	51.4	56.2	60.0	64.1	68.4	73.5	75.0	79.2	83.9	88.1		
KR41-4	52.3	56.8	60.8	64.6	68.8	73.4	74.6					
KR42-3	48.2	52.4	56.1	62.2	68.6	74.4	75.4	80.3	85.2	89.6		
KR42-4	48.7	52.8	57.5	63.0	69.2	74.3	75.5					
KR43-1	46.5	50.2	53.2	56.4	62.8	71.4	73.6	79.5	84.8	89.1		
KR43-2	46.8	50.4	53.8	56.8	63.3	71.3	73.3					

T643 autoclave (Westinghouse) - 680F Water I-NERI Corrosion Coupons

Alloy	Sample
Zr-1.0Fe	N101-1
Zr-1.0Fe	N101-2
Zr-1.0Fe	N101-3
Zr-1.0Fe	N101-4
Zr-1.0Fe	N101-5

Alloy	Sample	Gain (1)	Gain (2)	Gain (3)	Gain (4)	Gain (5)	Gain (6)	Gain (7)	Gain (8)	Gain 9	Gain 10	Gain 11	Gain 12
	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-1.0Fe	N101-1	6.7	7.9	11.2	14.5	15.7	18.8	21.3	24.8	27.9	30.4	32.7	34.8
Zr-1.0Fe	N101-2	6.4	7.8	11.4	14.8	16.1	19.3	21.4	25.3	28.7	30.9	33.7	36.0
Zr-1.0Fe	N101-3	6.5	8.6	11.2	15.1	16.5	19.9	22.4	26.3	29.5	33.3	34.6	36.8
Zr-1.0Fe	N101-4	6.0	8.0	11.0	14.6	15.8	19.4	22.0	25.7	29.3	31.6	34.5	36.3
Zr-1.0Fe	N101-5	6.0	7.6	11.3	14.8	16.4	19.7	22.0	26.0	28.9	31.9	34.4	36.3

Alloy	
Zr-1.0Fe-0.5Cr	N102-1
Zr-1.0Fe-0.5Cr	N102-2
Zr-1.0Fe-0.5Cr	N102-3
Zr-1.0Fe-0.5Cr	N102-4
Zr-1.0Fe-0.5Cr	N102-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-1.0Fe-0.5Cr	N102-1	6.3	7.7	10.0	12.1	13.1	15.3	16.7	19.1	21.2	22.5	23.9	25.1
Zr-1.0Fe-0.5Cr	N102-2	6.1	7.7	9.8	12.3	13.1	15.3	16.9	19.3	21.2	22.7	23.9	25.1
Zr-1.0Fe-0.5Cr	N102-3	6.0	7.5	10.2	12.0	13.1	15.4	16.9	19.2	21.2	22.6	24.1	25.2
Zr-1.0Fe-0.5Cr	N102-4	6.6	7.9	10.3	12.3	13.5	15.6	17.4	19.8	22.0	23.4	24.6	25.5
Zr-1.0Fe-0.5Cr	N102-5	6.0	7.2	9.9	12.0	13.2	15.2	16.9	19.1	21.4	22.5	24.0	24.9

Alloy	
Zr-0.6Fe	N201-1
Zr-0.6Fe	N201-2
Zr-0.6Fe	N201-3
Zr-0.6Fe	N201-4
Zr-0.6Fe	N201-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-0.6Fe	N201-1	6.0	7.2	10.1	12.5	13.7	16.0	17.8	20.8	23.4	25.2	27.0	28.2
Zr-0.6Fe	N201-2	5.0	6.4	9.1	11.8	12.7	15.3	17.2	20.1	22.7	24.5	26.3	27.7
Zr-0.6Fe	N201-3	5.5	6.9	9.6	12.0	13.2	15.1	17.7	20.4	22.8	24.7	26.5	27.9
Zr-0.6Fe	N201-4	6.2	7.8	10.3	13.6	14.7	17.3	19.4	22.4	25.0	27.0	28.6	30.5
Zr-0.6Fe	N201-5	5.6	7.7	10.1	13.1	13.9	16.8	18.7	21.5	23.8	25.8	27.6	29.3

Alloy	
Zr-0.6Fe-0.3Cr	N202-1
Zr-0.6Fe-0.3Cr	N202-2
Zr-0.6Fe-0.3Cr	N202-3
Zr-0.6Fe-0.3Cr	N202-4
Zr-0.6Fe-0.3Cr	N202-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-0.6Fe-0.3Cr	N202-1	6.3	8.2	9.9	12.0	13.0	14.8	16.2	19.0	20.5	21.9	22.9	24.1
Zr-0.6Fe-0.3Cr	N202-2	5.9	7.2	9.7	12.0	12.9	15.2	16.4	18.8	20.1	21.7	22.7	24.1
Zr-0.6Fe-0.3Cr	N202-3	6.0	7.3	9.8	11.7	12.7	14.9	16.5	18.3	20.5	21.6	22.5	23.7
Zr-0.6Fe-0.3Cr	N202-4	6.1	7.5	9.6	12.3	12.9	15.2	16.6	18.6	20.0	21.7	22.4	24.0
Zr-0.6Fe-0.3Cr	N202-5	6.5	7.8	10.4	12.4	13.3	15.3	16.5	18.8	20.1	22.2	22.7	23.7

Alloy	
Zr-0.6Fe-0.3Mo	N203-1
Zr-0.6Fe-0.3Mo	N203-2
Zr-0.6Fe-0.3Mo	N203-3
Zr-0.6Fe-0.3Mo	N203-4
Zr-0.6Fe-0.3Mo	N203-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-0.6Fe-0.3Mo	N203-1	7.8	9.9	12.9	16.4	17.7	20.7	23.0	26.4	29.5	31.8	34.2	36.0
Zr-0.6Fe-0.3Mo	N203-2	6.6	9.0	12.5	15.6	16.5	19.9	21.7	25.4	28.7	30.9	33.1	34.9
Zr-0.6Fe-0.3Mo	N203-3	6.9	10.1	13.2	16.6	17.8	20.8	23.1	26.8	29.8	32.4	34.6	36.5
Zr-0.6Fe-0.3Mo	N203-4	7.0	9.3	12.2	15.2	16.5	19.7	21.8	25.5	28.3	31.5	33.1	35.0
Zr-0.6Fe-0.3Mo	N203-5	7.3	9.6	13.0	16.1	17.2	20.8	23.0	26.7	29.8	32.5	34.6	36.7

Alloy	
Zr-0.6Fe-0.6Nb	N204-1
Zr-0.6Fe-0.6Nb	N204-2
Zr-0.6Fe-0.6Nb	N204-3
Zr-0.6Fe-0.6Nb	N204-4
Zr-0.6Fe-0.6Nb	N204-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-0.6Fe-0.6Nb	N204-1	6.4	7.8	9.9	12.7	13.4	16.8	19.0	22.4	25.3	28.4	30.8	33.1
Zr-0.6Fe-0.6Nb	N204-2	6.1	7.6	10.1	12.5	13.6	16.3	18.9	22.4	25.3	28.1	30.5	32.4
Zr-0.6Fe-0.6Nb	N204-3	6.5	8.4	10.4	12.8	13.9	16.9	18.8	22.9	26.3	28.8	31.3	33.4
Zr-0.6Fe-0.6Nb	N204-4	6.4	7.8	9.9	12.5	13.5	16.5	18.5	22.4	25.3	28.3	30.5	32.5
Zr-0.6Fe-0.6Nb	N204-5	6.6	7.9	10.2	12.7	13.6	16.3	18.4	22.3	25.5	28.2	30.2	32.6

Alloy	
Zr-1.0Cr-0.5Fe	N301-1
Zr-1.0Cr-0.5Fe	N301-2
Zr-1.0Cr-0.5Fe	N301-3
Zr-1.0Cr-0.5Fe	N301-4
Zr-1.0Cr-0.5Fe	N301-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-1.0Cr-0.5Fe	N301-1	6.6	8.3	10.1	12.0	12.8	14.5	16.2	18.4	19.7	21.4	22.4	23.6
Zr-1.0Cr-0.5Fe	N301-2	6.1	8.2	10.0	11.9	12.5	14.8	16.2	18.5	20.2	21.4	22.6	23.5
Zr-1.0Cr-0.5Fe	N301-3	6.4	8.0	10.1	12.0	12.5	14.5	15.9	17.9	19.7	21.3	22.4	23.5
Zr-1.0Cr-0.5Fe	N301-4	6.8	8.3	10.3	12.1	12.6	14.9	16.2	18.4	20.2	21.3	22.3	23.6
Zr-1.0Cr-0.5Fe	N301-5	5.0	6.0	7.7	9.6	10.5	12.5	13.6	15.7	17.4	18.6	19.6	20.5

Alloy	
Zr-1.0Cr-0.5Mo	N302-1
Zr-1.0Cr-0.5Mo	N302-2
Zr-1.0Cr-0.5Mo	N302-3
Zr-1.0Cr-0.5Mo	N302-4
Zr-1.0Cr-0.5Mo	N302-5

	days	0.5	1.0	3.1	7.1	9.3	16.3	23.3	37.3	51.3	65.3	79.3	93.3
Zr-1.0Cr-0.5Mo	N302-1	6.3	7.7	9.9	11.7	12.5	15.0	16.5	20.2	23.0	25.1	27.4	30.2
Zr-1.0Cr-0.5Mo	N302-2	5.4	6.8	8.7	10.6	11.2	13.6	15.3	17.8	20.5	22.8	25.3	28.8
Zr-1.0Cr-0.5Mo	N302-3	5.7	6.7	8.4	10.8	12.0	14.3	16.1	19.4	22.3	24.4	26.5	29.8
Zr-1.0Cr-0.5Mo	N302-4	5.2	6.2	7.9	9.7	10.3	12.5	14.0	16.7	19.1	21.0	23.1	25.8
Zr-1.0Cr-0.5Mo	N302-5	5.8	6.8	8.5	10.6	11.7	13.9	15.8	18.5	20.8	23.6	25.4	27.9

T679 autoclave (Westinghouse) - 680F Water INERI Model Alloys – short duration experiment

Alloy	Sample	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)
	days	0.5	1	2	4	7	14
Zr-0.2Fe-0.1Cr L	11-39	5.4	6.5	7.7	9.2	10.8	12.8
Zr-0.2Fe-0.1Cr L	11-40	5.6	6.8	8.2	9.5	11.1	13.2
Zr-0.2Fe-0.1Cr L	11-41	5.5	6.5	7.8	9.3	10.6	12.8
Zr-0.2Fe-0.1Cr H	12-39	5.6	6.8	8.1	9.5	11.0	12.9
Zr-0.2Fe-0.1Cr H	12-40	5.6	7.1	8.3	9.6	11.1	13.4
Zr-0.2Fe-0.1Cr H	12-41	5.5	6.5	7.8	9.2	10.3	12.6
Zr-0.4Fe-0.2Cr L	13-39	6.2	7.3	8.4	10.0	11.4	13.6
Zr-0.4Fe-0.2Cr L	13-40	5.6	6.8	7.9	9.5	10.7	13.0
Zr-0.4Fe-0.2Cr L	13-41	5.9	7.1	8.6	10.0	11.2	13.6
Zr-0.4Fe-0.2Cr H	14-39	7.0	8.3	9.8	12.4	13.1	15.5
Zr-0.4Fe-0.2Cr H	14-40	6.3	7.8	9.4	11.2	12.7	15.0
Zr-0.4Fe-0.2Cr H	14-41	6.5	7.7	9.5	10.8	12.4	14.7

Alloy	Sample	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)
	days	0.5	1	2	4	7	14
Zr-0.5Cr	21-39	4.6	5.5	6.1	7.6	8.5	10.1
Zr-0.5Cr	21-40	4.3	5.2	5.8	7.3	8.4	10.2
Zr-0.5Cr	21-41	4.2	5.2	6.0	7.2	8.2	10.2
Zr-0.5Cr-0.2Fe	22-39	5.1	5.9	7.1	8.3	9.2	11.2
Zr-0.5Cr-0.2Fe	22-40	4.7	5.2	6.7	8.0	9.4	10.9
Zr-0.5Cr-0.2Fe	22-41	4.7	5.8	6.6	8.0	9.3	10.9
Zr-1.0Cr	23-39	4.1	5.3	6.3	7.4	8.3	10.0
Zr-1.0Cr	23-40	4.4	5.2	6.5	7.4	8.4	10.1
Zr-1.0Cr	23-41	4.6	5.0	6.0	7.3	8.5	9.7
Zr-1.0Cr-0.2Fe	24-39	5.2	6.6	7.8	9.2	10.4	12.6
Zr-1.0Cr-0.2Fe	24-40	4.9	6.1	7.4	8.9	10.1	12.2
Zr-1.0Cr-0.2Fe	24-41	5.1	6.5	7.5	9.1	10.5	12.8

Alloy	Sample days	Gain(1) 0.5	Gain(2) 1	Gain(3) 2	Gain(4) 4	Gain(5) 7	Gain(6) 14
Zr-0.5Cu	31-39	12.5	16.8	21.7	27.2	29.5	32.8
Zr-0.5Cu	31-40	12.5	16.8	22.4	28.0	30.3	35.2
Zr-0.5Cu	31-41	12.6	17.1	22.9	28.5	31.6	40.0
Zr-0.5Cu-0.5Mo	32-39	10.5	13.1	17.4	22.0	23.8	26.0
Zr-0.5Cu-0.5Mo	32-40	10.9	13.1	17.5	22.2	24.1	27.0
Zr-0.5Cu-0.5Mo	32-41	10.8	12.7	16.5	20.8	22.6	25.3
Zr-1.0Cu	33-39	11.9	13.8	15.7	18.5	20.0	22.8
Zr-1.0Cu	33-40	11.8	13.8	15.7	18.1	20.5	22.7
Zr-1.0Cu	33-41	12.5	14.3	16.1	18.8	20.9	23.5
Zr-1.0Cu-0.5Mo	34-39	10.6	12.7	14.1	17.5	19.3	21.7
Zr-1.0Cu-0.5Mo	34-40	10.9	12.7	14.5	17.4	19.7	21.7
Zr-1.0Cu-0.5Mo	34-41	10.7	12.1	13.9	16.7	18.8	21.1

Alloy	Sample days	Gain(1) 0.5	Gain(2) 1	Gain(3) 2	Gain(4) 4	Gain(5) 7	Gain(6) 14
Sponge Zr	41-39	6.0	12.6	32.0	44.7	60.3	-150.8
Sponge Zr	41-40	5.9	12.9	34.3	46.8	46.0	-225.4
Sponge Zr	41-41	5.8	13.0	35.3	0.0	0.0	0.0
Crystal bar Zr	42-39	5.0	8.5	31.3	30.9	24.7	-17.7
Crystal bar Zr	42-40	4.9	9.7	32.4	27.5	16.5	-35.4
Crystal bar Zr	42-41	4.4	8.4	29.7	0.0	0.0	0.0
Zircaloy-4	43-39	8.4	9.9	11.2	13.2	14.9	17.5
Zircaloy-5	43-40	8.3	9.7	11.2	13.4	14.8	17.7
Zircaloy-6	43-41	8.4	9.9	11.4	13.4	15.1	17.9

T600 autoclave (Westinghouse) - 680F Water INERI Model Alloys - short duration experiment

T600	Auto.	47-103
680F Water	Cycle	103
INERI Model		
Alloys	Hours	6
	Total	6

Sample	Area	Wght(0)	Wght(1)	Gain(1)	
11-23	0.1059	1.99054	1.99123	6.5	mg/dm2
12-23	0.1071	2.14094	2.14150	5.2	mg/dm2
13-23	0.1070	2.08448	2.08504	5.2	mg/dm2
14-23	0.1092	2.15665	2.15720	5.0	mg/dm2
21-23	0.1043	1.86720	1.86759	3.7	mg/dm2
22-23	0.1051	2.01791	2.01836	4.3	mg/dm2
23-23	0.1056	1.94132	1.94172	3.8	mg/dm2
24-23	0.1052	2.06873	2.06923	4.8	mg/dm2
31-23	0.1046	1.48123	1.48220	9.3	mg/dm2
32-23	0.1058	1.99870	1.99968	9.3	mg/dm2
33-23	0.1053	1.94022	1.94123	9.6	mg/dm2
34-23	0.1060	2.06472	2.06571	9.3	mg/dm2
41-23	0.1039	1.82424	1.82466	4.0	mg/dm2
42-23	0.1072	2.13254	2.13313	5.5	mg/dm2
43-23	0.1038	1.95147	1.95233	8.3	mg/dm2
KR11-7	0.1041	2.20544	2.20582	3.7	mg/dm2
KR12-7	0.1017	1.46231	1.46270	3.8	mg/dm2
KR13-7	0.1040	2.12389	2.12438	4.7	mg/dm2
KR14-7	0.1044	2.34492	2.34537	4.3	mg/dm2
KR21-7	0.1008	1.29912	1.29948	3.6	mg/dm2
KR22-7	0.1044	2.16663	2.16702	3.7	mg/dm2
KR31-7	0.1026	1.77295	1.77341	4.5	mg/dm2
KR32-7	0.1040	2.10853	2.10906	5.1	mg/dm2
KR41-7	0.1030	2.22604	2.22644	3.9	mg/dm2
KR42-7	0.1007	1.35867	1.35908	4.1	mg/dm2
KR43-7	0.1054	2.37133	2.37174	3.9	mg/dm2
N101-1	0.0790	1.70479	1.70520	5.2	mg/dm2
N102-1	0.0790	1.58090	1.58153	8.0	mg/dm2
N201-1	0.0792	1.66188	1.66233	5.7	mg/dm2
N202-1	0.0791	1.66664	1.66696	4.0	mg/dm2
N203-1	0.0776	1.38047	1.38082	4.5	mg/dm2
N204-1	0.0780	1.42671	1.42706	4.5	mg/dm2
N301-1	0.0796	1.90234	1.90265	3.9	mg/dm2
N302-1	0.0813	2.02809	2.02855	5.7	mg/dm2

T601 autoclave (Westinghouse) - 680F Water INERI Model Alloys - short duration experiment

T601	Auto.	47-104
680F Water	Cycle	104
INERI Model		
Alloys	Hours	2.5
	Total	2.5

Sample	Area	Wght(0)	Wght(1)	Gain(1)	
11-24	0.1060	1.79277	1.79321	4.2	mg/dm2
12-24	0.1118	2.23592	2.23636	3.9	mg/dm2
13-24	0.1051	1.99364	1.99405	3.9	mg/dm2
14-24	0.1084	2.15502	2.15546	4.1	mg/dm2
21-24	0.1035	1.74208	1.74235	2.6	mg/dm2
22-24	0.1073	2.13840	2.13873	3.1	mg/dm2
23-24	0.1060	2.06421	2.06454	3.1	mg/dm2
24-24	0.1034	1.63160	1.63199	3.8	mg/dm2
31-24	0.1063	1.93100	1.93159	5.5	mg/dm2
32-24	0.1054	1.89715	1.89775	5.7	mg/dm2
33-24	0.1066	1.97125	1.97187	5.8	mg/dm2
34-24	0.1079	2.16719	2.16787	6.3	mg/dm2
41-24	0.1052	2.09213	2.09259	4.4	mg/dm2
42-24	0.1046	2.12667	2.12710	4.1	mg/dm2
43-24	0.1050	2.05363	2.05433	6.7	mg/dm2
KR11-9	0.1031	1.66656	1.66691	3.4	mg/dm2
KR12-8	0.1041	2.10373	2.10412	3.7	mg/dm2
KR13-8	0.1037	1.77060	1.77099	3.8	mg/dm2
KR14-8	0.1049	2.26556	2.26609	5.1	mg/dm2
KR21-8	0.1018	1.68996	1.69026	2.9	mg/dm2
KR22-8	0.1020	1.63781	1.63819	3.7	mg/dm2
KR31-8	0.1030	2.08028	2.08077	4.8	mg/dm2
KR32-8	0.1035	1.92474	1.92521	4.5	mg/dm2
KR41-8	0.1025	2.12965	2.13008	4.2	mg/dm2
KR42-8	0.1039	2.23568	2.23611	4.1	mg/dm2
KR43-8	0.1053	2.47016	2.47059	4.1	mg/dm2
N101-2	0.0773	1.46408	1.46440	4.1	mg/dm2
N102-2	0.0793	1.60863	1.60897	4.3	mg/dm2
N201-2	0.0782	1.48470	1.48500	3.8	mg/dm2
N202-2	0.0793	1.76189	1.76220	3.9	mg/dm2
N203-2	0.0783	1.48033	1.48067	4.3	mg/dm2
N204-2	0.0789	1.63621	1.63656	4.4	mg/dm2
N301-2	0.0803	1.81140	1.81168	3.5	mg/dm2
N302-2	0.0805	1.91208	1.91235	3.4	mg/dm2

T602 autoclave (Westinghouse) - 680F Water INERI Model Alloys - short duration experiment

T602	Auto.	54-131
680F Water	Cycle	131
INERI Model		
Alloys	Hours	24
	Total	24

Sample	Area	Wght(0)	Wght(1)	Gain(1)	
11-25	0.1078	2.05408	2.05490	7.6	mg/dm2
12-25	0.1066	2.12805	2.12886	7.6	mg/dm2
13-25	0.1053	2.01367	2.01442	7.1	mg/dm2
14-25	0.1061	2.06460	2.06541	7.6	mg/dm2
21-25	0.1063	2.17564	2.17615	4.8	mg/dm2
22-25	0.1056	2.09721	2.09777	5.3	mg/dm2
23-25	0.1070	2.08035	2.08094	5.5	mg/dm2
24-25	0.1032	1.70617	1.70708	8.8	mg/dm2
31-25	0.1060	1.81129	1.81270	13.3	mg/dm2
32-25	0.1027	1.67822	1.67947	12.2	mg/dm2
33-25	0.1043	1.93105	1.93235	12.5	mg/dm2
34-25	0.1052	1.87497	1.87631	12.7	mg/dm2
41-25	0.1049	2.11002	2.11162	15.3	mg/dm2
42-25	0.1047	2.05414	2.05581	16.0	mg/dm2
43-25	0.1045	1.95562	1.95655	8.9	mg/dm2
KR11-8	0.1048	2.16341	2.16401	5.7	mg/dm2
KR12-9	0.1024	1.56470	1.56547	7.5	mg/dm2
KR13-9	0.1047	1.98172	1.98234	5.9	mg/dm2
KR14-9	0.1045	2.31991	2.32071	7.7	mg/dm2
KR21-9	0.1010	1.72599	1.72648	4.9	mg/dm2
KR22-9	0.1011	1.40625	1.40680	5.4	mg/dm2
KR31-9	0.1038	2.12759	2.12830	6.8	mg/dm2
KR32-9	0.1035	1.93393	1.93466	7.1	mg/dm2
KR41-9	0.1038	2.25166	2.25230	6.2	mg/dm2
KR42-9	0.1006	1.34358	1.34422	6.4	mg/dm2
KR43-9	0.1047	2.12283	2.12352	6.6	mg/dm2
N101-3	0.0777	1.46739	1.46807	8.8	mg/dm2
N102-3	0.0793	1.74299	1.74373	9.3	mg/dm2
N201-3	0.0790	1.62431	1.62484	6.7	mg/dm2
N202-3	0.0798	1.88267	1.88327	7.5	mg/dm2
N203-3	0.0801	1.79903	1.80023	15.0	mg/dm2
N204-3	0.0798	1.67152	1.67201	6.1	mg/dm2
N301-3	0.0810	1.92398	1.92446	5.9	mg/dm2
N302-3	0.0806	1.79847	1.80094	30.6	mg/dm2

T603 autoclave (Westinghouse) - 680F Water INERI Model Alloys - short duration experiment

T603	Auto.	58-82
680F Water	Cycle	82
INERI Model		
Alloys	Days	3.0
	Total	3.0

Sample	Area v-rjc	Wght(0) v-rjc	Wght(1) v-rjc	Gain(1)	
11-26	0.1068	2.03820	2.03925	9.8	mg/dm2
12-26	0.1071	2.11096	2.11209	10.6	mg/dm2
13-26	0.1062	2.06959	2.07056	9.1	mg/dm2
14-26	0.1057	2.06014	2.06125	10.5	mg/dm2
21-26	0.1083	2.22951	2.23019	6.3	mg/dm2
22-26	0.1056	2.04288	2.04363	7.1	mg/dm2
23-26	0.1061	1.91516	1.91597	7.6	mg/dm2
24-26	0.1046	1.91054	1.91138	8.0	mg/dm2
31-26	0.1055	1.81882	1.82125	23.0	mg/dm2
32-26	0.1040	1.79499	1.79734	22.6	mg/dm2
33-26	0.1072	2.03325	2.03521	18.3	mg/dm2
34-26	0.1069	1.98817	1.98995	16.7	mg/dm2
41-26	0.1031	1.86979	1.87495	50.1	mg/dm2
42-26	0.1054	2.06907	2.07526	58.7	mg/dm2
43-26	0.1013	1.76282	1.76396	11.3	mg/dm2

KR11-10	0.1020	2.11368	2.11499	12.8	mg/dm2	estimated area (need sample dimensions)
KR12-10	0.1020	1.74249	1.74342	9.1	mg/dm2	estimated area (need sample dimensions)
KR13-10	0.1020	1.68634	1.68728	9.2	mg/dm2	estimated area (need sample dimensions)
KR14-10	0.1020	2.27850	2.27950	9.8	mg/dm2	estimated area (need sample dimensions)
KR21-10	0.1020	1.51981	1.52049	6.7	mg/dm2	estimated area (need sample dimensions)
KR22-10	0.1020	2.02667	2.02738	7.0	mg/dm2	estimated area (need sample dimensions)
KR31-10	0.1020	2.10285	2.10358	7.2	mg/dm2	estimated area (need sample dimensions)
KR32-10	0.1020	1.86566	1.86662	9.4	mg/dm2	estimated area (need sample dimensions)
KR41-10	0.1020	2.05675	2.05761	8.4	mg/dm2	estimated area (need sample dimensions)
KR42-10	0.1020	1.57680	1.57773	9.1	mg/dm2	estimated area (need sample dimensions)
KR43-10	0.1020	2.31241	2.31329	8.6	mg/dm2	estimated area (need sample dimensions)
N101-4	0.0800	1.41545	1.41746	25.1	mg/dm2	estimated area (need sample dimensions)
N102-4	0.0800	1.53332	1.53540	26.0	mg/dm2	estimated area (need sample dimensions)
N201-4	0.0800	1.70436	1.70513	9.6	mg/dm2	estimated area (need sample dimensions)
N202-4	0.0800	1.60675	1.60753	9.8	mg/dm2	estimated area (need sample dimensions)
N203-4	0.0800	1.60687	1.60781	11.7	mg/dm2	estimated area (need sample dimensions)
N204-4	0.0800	1.73140	1.73215	9.4	mg/dm2	estimated area (need sample dimensions)
N301-4	0.0800	1.68892	1.69153	32.6	mg/dm2	estimated area (need sample dimensions)
N302-4	0.0800	1.94506	1.94597	11.4	mg/dm2	estimated area (need sample dimensions)

ROK Experiments - 680F Water

INERI Model Alloys

days	30	60	90	120	150	180	210	240	270	300	330	360	390	420	450	486
Zr0.2Nb	19.4	24.7	27.8	30.1	34.4	35.4	37.0	42.2	48.3	58.9	63.7	69.1	73.3	76.0	79.4	84.2
Zr0.4Nb	21.3	29.1	34.7	39.9	47.6	51.2	54.1	59.8	65.0	72.1	75.1	81.2	86.1	90.4	94.9	101.2
Zr1.0Nb	22.9	30.5	38.2	44.5	52.6	56.8	61.4	67.1	71.7	79.9	83.3	89.6	95.2	101.0	105.7	113.0
Zr1.5Nb	22.5	30.3	36.8	42.7	49.2	53.5	57.5	63.1	67.9	75.3	78.7	84.9	90.6	96.6	100.4	107.2
Zr1.5Nb	21.2	28.2	34.1	39.1	46.3	51.3	54.7	59.4	65.2	72.4	75.7	81.4	86.5	91.5	95.0	101.6

days	516	546	576	606	
Zr0.2Nb	85.4	90.9	92.1	95.3	mg/dm2
Zr0.4Nb	104.0	111.1	114.4	120.4	mg/dm2
Zr1.0Nb	116.4	123.6	126.7	131.9	mg/dm2
Zr1.5Nb	110.6	117.7	120.2	125.0	mg/dm2
Zr1.5Nb	105.3	110.3	114.3	118.2	mg/dm2

days	3	9	14	21	28	42	56	70	100	130	160	190	226	256
Zr0.2Sn	13.1	16.4	21.8	23.1	24.8	25.7	28.2	31.5	38.1	39.4	39.9	41.2	41.0	42.7
Zr0.4Sn	13.7	16.3	21.8	23.4	25.3	26.1	27.6	32.0	36.7	38.8	39.4	39.0	40.9	42.5
Zr0.8Sn	14.5	17.5	23.3	24.6	26.5	27.7	29.2	34.5	39.9	40.4	41.4	40.0	44.4	49.0
Zr1.2Sn	14.3	18.2	23.9	25.3	27.3	28.2	30.0	35.8	43.7	42.3	43.7	42.5	47.0	54.2

days	286	316	346	
Zr0.2Sn	43.4	46.5	48.9	mg/dm ²
Zr0.4Sn	43.2	48.8	53.3	mg/dm ²
Zr0.8Sn	59.1	173.8	273.3	mg/dm ²
Zr1.2Sn	74.3	295.4	489.2	mg/dm ²

days	30	60	90	120	150	180	210	240	270	300	330	360	390	420	450
Zr0.4Sn0.2Nb	22.1	29.5	35.1	39.4	46.4	49.8	53.6	59.9	64.5	72.0	75.7	81.4	86.3	90.9	95.4
Zr0.4Sn0.4Nb	20.5	24.3	28.2	29.4	33.1	33.8	34.9	39.2	44.0	52.0	58.4	66.0	70.5	72.7	74.5

days	486	516	546	576	606	
Zr0.4Sn0.2Nb	101.9	105.6	112.4	114.7	121.3	mg/dm2
Zr0.4Sn0.4Nb	79.0	79.6	84.3	84.4	87.0	mg/dm2

ROK Experiments - 680F Lithiated water

INERI Model Alloys

days	30	60	90	120	150	180	210	
Zr0.2Nb	25.6	41.6	587.8	1827.7				mg/dm2
Zr0.4Nb	32.5	44.1	204.7	spall	--	--	--	mg/dm2
Zr1.0Nb	35.0	43.1	46.7	49.2	54.0	114.4	spall	mg/dm2
Zr1.5Nb	33.6	42.1	45.8	47.9	52.9	56.7	spall	mg/dm2
Zr2.5Nb	34.9	41.3	43.5	45.2	49.5	53.2	444.9	mg/dm2

days	30	60	90	120	150	180	210	240	mg/dm2
Zr0.4Sn0.2Nb	28.9	38.8	39.9	41.7	44.6	48.2	78.0	spall	mg/dm2
Zr0.4Sn0.4Nb	35.7	44.3	47.1	51.1	54.5	60.3	634.6	spall	mg/dm2

days	30	60	90	120	150	180	210	mg/dm2
Zr0.2Sn	22.1	29.7	49.3	98.1	493.1	732.9		mg/dm2
Zr0.4Sn	21.7	31.3	84.5	171.3	596.1	973.7		mg/dm2
Zr0.8Sn	22.6	32.7	36.6	43.9	110.5	198.4	280.7	mg/dm2
Zr1.2Sn	23.0	32.9	131.4	210.5	485.5	734.2	956.2	mg/dm2

500°C Steam (Westinghouse)

ZrFeCr Alloys

Alloy	Sample days	Gain(1) 0.25	Gain(2) 0.50	Gain(3) 0.75	Gain(4) 2.00	Gain(5) 3.25	Gain(6) 3.75	Gain(7) 4.25	Gain(8) 7.25	Gain(9) 8.25	Gain(10) 9.25
Zr-0.2Fe-0.1Cr	11-17	44.3	48.7	50.1	38.7	34.5	25.9				
Zr-0.2Fe-0.1Cr	11-18	59.1									
Zr-0.2Fe-0.1Cr	12-17	169.7									
Zr-0.2Fe-0.1Cr	12-18	364.0									
Zr-0.4Fe-0.2Cr	13-17	20.8	24.6	28.2	36.6	42.6	44.4	45.7	54.0	56.2	58.1
Zr-0.4Fe-0.2Cr	13-18	21.0	24.8	28.6	36.9	42.8	44.5	46.1	54.4	56.7	59.3
Zr-0.4Fe-0.2Cr	14-17	27.7	31.4	33.2	42.4	49.9	50.9	52.4	60.7	63.0	65.1
Zr-0.4Fe-0.2Cr	14-18	27.3	31.4	35.6	45.1	51.9	53.1	54.9	64.6	65.8	68.2

Alloy	Sample days	Gain(11) 12.25	Gain(12) 17.25	Gain(13) 22.25	Gain(14) 27.25	Gain(15) 32.25	Gain(16) 37.25	Gain(17) 42.25	Gain(18) 47.25
Zr-0.2Fe-0.1Cr	11-17								
Zr-0.2Fe-0.1Cr	11-18								
Zr-0.2Fe-0.1Cr	12-17								
Zr-0.2Fe-0.1Cr	12-18								
Zr-0.4Fe-0.2Cr	13-17	64.2	68.5	74.1	83.8	90.0	96.0	101.8	108.2
Zr-0.4Fe-0.2Cr	13-18	65.3	69.8	75.3	84.4	91.1	96.7	102.6	109.0
Zr-0.4Fe-0.2Cr	14-17	70.7	75.7	81.3	91.2	90.5	98.3	106.3	115.5
Zr-0.4Fe-0.2Cr	14-18	74.6	79.7	84.5	94.1	101.2	108.4	114.4	121.7

ZrFeCr Alloys (conti.)

Alloy	Sample days	Gain(20)	Gain(22)	Gain(23)	Gain(24)	Gain(25)	Gain(26)	Gain(27)	Gain(28)	Gain(29)
		57.25	67.25	69.33	71.25	76.25	81.25	86.25	96.25	97.25
Zr-0.2Fe-0.1Cr	11-17									
Zr-0.2Fe-0.1Cr	11-18									
Zr-0.2Fe-0.1Cr	12-17									
Zr-0.2Fe-0.1Cr	12-18									
Zr-0.4Fe-0.2Cr	13-17	117.7	127.3	128.8	130.1	134.5	138.0	141.4	148.2	148.9
Zr-0.4Fe-0.2Cr	13-18	118.2	127.4	129.1	130.3	134.6				
Zr-0.4Fe-0.2Cr	14-17	131.7	150.3	154.0	157.0	168.0	179.4	195.6	214.8	220.7
Zr-0.4Fe-0.2Cr	14-18	130.4	36.7							

Alloy	Sample days	Gain(30)	Gain(31)	Gain(32)	Gain(33)	Gain(34)	
		100.25	105.25	110.25	115.25	129.3	mg/dm2
Zr-0.2Fe-0.1Cr	11-17						mg/dm2
Zr-0.2Fe-0.1Cr	11-18						mg/dm2
Zr-0.2Fe-0.1Cr	12-17						mg/dm2
Zr-0.2Fe-0.1Cr	12-18						mg/dm2
Zr-0.4Fe-0.2Cr	13-17	151.0	153.7	157.1	161.2	172.2	mg/dm2
Zr-0.4Fe-0.2Cr	13-18						mg/dm2
Zr-0.4Fe-0.2Cr	14-17	229.5	240.5	255.1	285.1	339.9	mg/dm2
Zr-0.4Fe-0.2Cr	14-18						mg/dm2

ZrCrFe Alloys (Steam, W)

Alloy	Sample	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	Gain(7)	Gain(8)	Gain(9)
	days	0.25	0.50	0.75	2.00	3.25	3.75	4.25	7.25	8.25
Zr-0.5Cr	21-17	786.3	853.6							
Zr-0.5Cr	21-18	723.9								
Zr-0.5Cr-0.2Fe	22-17	27.6	32.1	36.6	46.3	52.5	54.7	56.5	64.7	67.2
Zr-0.5Cr-0.2Fe	22-18	29.5	34.1	39.0	48.4	55.2	56.9	58.9	67.6	69.8
Zr-1.0Cr	23-17	31.5	37.0	44.1	81.1	109.3	110.9	101.6	65.9	
Zr-1.0Cr	23-18	34.8	40.4	45.1	58.0	67.9	65.7	69.6	65.3	70.7
Zr-1.0Cr-0.2Fe	24-17	21.0	23.4	28.0	37.4	43.9	45.5	48.0	57.8	59.4
Zr-1.0Cr-0.2Fe	24-18	20.2	23.6	28.2	38.0	45.8	47.6	49.7	58.9	61.1

Alloy	Sample	Gain(10)	Gain(11)	Gain(12)	Gain(13)	Gain(14)	Gain(15)	Gain(16)	Gain(17)
	days	9.25	12.25	17.25	22.25	27.25	32.25	37.25	42.25
Zr-0.5Cr	21-17								
Zr-0.5Cr	21-18								
Zr-0.5Cr-0.2Fe	22-17	70.4	76.7	81.5	86.5	95.4	102.9	108.9	114.7
Zr-0.5Cr-0.2Fe	22-18	72.5	78.8	83.6	88.8	96.8	103.7	109.4	115.1
Zr-1.0Cr	23-17								
Zr-1.0Cr	23-18	45.6	40.9						
Zr-1.0Cr-0.2Fe	24-17	63.4	70.6	75.6					
Zr-1.0Cr-0.2Fe	24-18	64.1	71.4	76.7	81.8	90.1	97.9	103.8	110.1

ZrCrFe Alloys (conti.)

Alloy	Sample days	Gain(18)	Gain(19)	Gain(20)	Gain(22)	Gain(23)	Gain(24)	Gain(25)
		47.25	52.25	57.25	67.25	69.33	71.25	76.25
Zr-0.5Cr	21-17							
Zr-0.5Cr	21-18							
Zr-0.5Cr-0.2Fe	22-17	120.0	123.6	118.1	72.1			
Zr-0.5Cr-0.2Fe	22-18	120.6	125.8	130.6	139.2	140.5	142.0	145.8
Zr-1.0Cr	23-17							
Zr-1.0Cr	23-18							
Zr-1.0Cr-0.2Fe	24-17							
Zr-1.0Cr-0.2Fe	24-18	116.2	122.3	127.9	138.7	138.7	140.6	146.0

Alloy	Sample days	Gain(26)	Gain(27)	Gain(28)	Gain(29)	Gain(30)	Gain(31)	Gain(32)
		81.25	86.25	96.25	97.25	100.25	105.25	110.25
Zr-0.5Cr	21-17							
Zr-0.5Cr	21-18							
Zr-0.5Cr-0.2Fe	22-17							
Zr-0.5Cr-0.2Fe	22-18	140.8	124.6	28.0	16.8	14.8	11.5	11.3
Zr-1.0Cr	23-17							
Zr-1.0Cr	23-18							
Zr-1.0Cr-0.2Fe	24-17							
Zr-1.0Cr-0.2Fe	24-18	150.9	155.6	164.1	165.2	167.6	175.9	185.5

Alloy	Sample days	Gain(33)	Gain(34)	
		115.25	129.3	
Zr-0.5Cr	21-17			mg/dm2
Zr-0.5Cr	21-18			mg/dm2
Zr-0.5Cr-0.2Fe	22-17			mg/dm2
Zr-0.5Cr-0.2Fe	22-18	14.1	24.7	mg/dm2
Zr-1.0Cr	23-17			mg/dm2
Zr-1.0Cr	23-18			mg/dm2
Zr-1.0Cr-0.2Fe	24-17			mg/dm2
Zr-1.0Cr-0.2Fe	24-18	199.2	221.7	mg/dm2

ZrCuMo Alloys (Steam, W)

Alloy	Sample days	Gain(1) 0.25	Gain(2) 0.50	Gain(3) 0.75	Gain(4) 2.00	Gain(5) 3.25	Gain(6) 3.75	Gain(7) 4.25	Gain(8) 7.25	Gain(9) 8.25
Zr-0.5Cu	31-17	1321.4								
Zr-0.5Cu	31-18	1216.2								
Zr-0.5Cu-0.5Mo	32-17	656.4	668.3							
Zr-0.5Cu-0.5Mo	32-18	668.9								
Zr-1.0Cu	33-17	72.6	77.4	87.0	116.6	141.5	150.1	159.5	211.3	211.3
Zr-1.0Cu	33-18	83.4								
Zr-1.0Cu-0.5Mo	34-17	81.9	86.9	92.9	114.8	133.5	138.3	144.6	173.3	173.2
Zr-1.0Cu-0.5Mo	34-18	86.1								

Alloy	Sample days	Gain(10) 9.25	Gain(11) 12.25	Gain(12) 17.25	Gain(13) 22.25	Gain(14) 27.25	Gain(15) 32.25	
Zr-0.5Cu	31-17							mg/dm2
Zr-0.5Cu	31-18							mg/dm2
Zr-0.5Cu-0.5Mo	32-17							mg/dm2
Zr-0.5Cu-0.5Mo	32-18							mg/dm2
Zr-1.0Cu	33-17	234.0	279.3	320.2	363.4	422.5	480.7	mg/dm2
Zr-1.0Cu	33-18							mg/dm2
Zr-1.0Cu-0.5Mo	34-17	183.3	204.4	221.4				mg/dm2
Zr-1.0Cu-0.5Mo	34-18							mg/dm2

Reference Alloys (Steam, W)

Alloy	Sample	Gain(1) 0.25	Gain(2) 0.50	Gain(3) 0.75
Sponge Zr	41-17	1897.5		mg/dm ²
Sponge Zr	41-18	1791.6		mg/dm ²
Crystal Bar Zr	42-17	2043.5		mg/dm ²
Crystal Bar Zr	42-18	2097.0		mg/dm ²
Zircaloy-4	43-17	539.2	690.0	mg/dm ²
Zircaloy-4	43-18	631.3		mg/dm ²
Zr-4 95-7051	T558-1	653.9	941.2	mg/dm ²
Zr-4 95-7051	T558-2	820.7		mg/dm ²
Zr-4 95-7051	T558-3	514.9		mg/dm ²

ZrSn Alloys (Steam, W)

Alloy	Sample days	Gain(1) 0.5	Gain(2) 1.0
Zr-0.2Sn	KR11-5	1689.4	mg/dm2
Zr-0.2Sn	KR11-6	1662.8	mg/dm2
Zr-0.4Sn	KR12-5	1969.8	mg/dm2
Zr-0.4Sn	KR12-6	1793.6	mg/dm2
Zr-0.8Sn	KR13-5	1705.8	mg/dm2
Zr-0.8Sn	KR13-6	1750.1	mg/dm2
Zr-1.2Sn	KR14-5	1758.1	mg/dm2
Zr-1.2Sn	KR14-6	1908.5	mg/dm2

ZrNb Alloys (< 0.5Nb) (Steam, W)

Alloy	Sample days	Gain(1) 0.5	Gain(2) 1.0	Gain(3) 4.0	Gain(4) 5.0	Gain(5) 6.0	Gain(6) 9.0	Gain(7) 14.0	
Zr-0.2Nb	KR21-5	1754.6							mg/dm2
Zr-0.2Nb	KR21-6	1575.7							mg/dm2
Zr-0.4Nb	KR22-5	31.9	29.2	34.2					mg/dm2
Zr-0.4Nb	KK22-6	25.5	32.7	57.7	62.6	69.0	84.0	96.2	mg/dm2

Alloy	Sample days	Gain(8) 19.0	Gain(9) 24.0	Gain(10) 29.0	Gain(11) 34.0	Gain(12) 39.0	Gain(13) 44.0	
Zr-0.2Nb	KR21-5							mg/dm2
Zr-0.2Nb	KR21-6							mg/dm2
Zr-0.4Nb	KR22-5							mg/dm2
Zr-0.4Nb	KK22-6	115.2	154.1	187.3	222.0	264.3	302.6	mg/dm2

Alloy	Sample days	Gain(14) 49.0	Gain(15) 54.0	Gain(17) 64.0	Gain(18) 66.1	Gain(19) 68.0	Gain(20) 73.0	
Zr-0.2Nb	KR21-5							mg/dm2
Zr-0.2Nb	KR21-6							mg/dm2
Zr-0.4Nb	KR22-5							mg/dm2
Zr-0.4Nb	KK22-6	344.1	381.5	465.9	485.0	496.2	528.4	mg/dm2

ZrSnNb Alloys (Steam, W)

Alloy	Sample days	Gain(1)	Gain(2)
		0.5	1.0
Zr-0.4Sn-0.2Nb	KR31-5	916.4	mg/dm2
Zr-0.4Sn-0.2Nb	KR31-6	966.4	mg/dm2
Zr-0.4Sn-0.4Nb	KR32-5	625.2	mg/dm2
Zr-0.4Sn-0.4Nb	KR32-6	645.2	mg/dm2

ZrNb Alloys (>0.5 Nb) (Steam, W)

Alloy	Sample days	Gain(1) 0.5	Gain(2) 1.0	Gain(3) 4.0	Gain(4) 5.0	Gain(5) 6.0	Gain(6) 9.0	Gain(7) 14.0	
Zr-1.0Nb	KR41-5	23.5	33.6	68.2	75.4	83.6	105.2	123.5	mg/dm2
Zr-1.0Nb	KR41-6	22.2	32.1	67.7	74.7	82.7	102.8	122.4	mg/dm2
Zr-1.5Nb	KR42-5	23.2	31.8	60.5	67.3	75.3	94.6	114.1	mg/dm2
Zr-1.5Nb	KR42-6	22.7	31.0	61.6	68.2	75.8	93.3	112.1	mg/dm2
Zr-2.5Nb	KR43-5	22.8	32.3	64.3	70.9	78.6	97.4	116.2	mg/dm2
Zr-2.5Nb	KR43-6	22.8	31.9	64.9	71.5	79.3	97.3	116.0	mg/dm2

Alloy	Sample days	Gain(8) 19.0	Gain(9) 24.0	Gain(10) 29.0	Gain(11) 34.0	Gain(12) 39.0	Gain(13) 44.0	Gain(14) 49.0	
Zr-1.0Nb	KR41-5	145.1	179.9	208.9	233.4	264.8	295.2	325.3	mg/dm2
Zr-1.0Nb	KR41-6	144.9	181.9	216.7	245.0	278.8	312.6	345.5	mg/dm2
Zr-1.5Nb	KR42-5	135.9	175.4	203.4	234.4	270.0	304.8	338.8	mg/dm2
Zr-1.5Nb	KR42-6	134.7	179.6	213.1	247.2	287.5	322.9	363.9	mg/dm2
Zr-2.5Nb	KR43-5	137.9	174.1	215.1	253.7	289.2	325.2	361.7	mg/dm2
Zr-2.5Nb	KR43-6	137.6	175.3	218.0	256.8	291.6	331.2	370.3	mg/dm2

ZrNb Alloys (>0.5 Nb) (conti.)

Alloy	Sample days	Gain(14)	Gain(15)	Gain(17)	Gain(18)	Gain(19)	Gain(20)	
		49.0	54.0	64.0	66.1	68.0	73.0	
Zr-1.0Nb	KR41-5	325.3	352.8	409.7				mg/dm ²
Zr-1.0Nb	KR41-6	345.5	377.2	441.9	456.0	466.3	495.3	mg/dm ²
Zr-1.5Nb	KR42-5	338.8	373.1	442.1				mg/dm ²
Zr-1.5Nb	KR42-6	363.9	398.7	475.3	497.2	508.9	538.1	mg/dm ²
Zr-2.5Nb	KR43-5	361.7	398.0	477.0				mg/dm ²
Zr-2.5Nb	KR43-6	370.3	409.3	490.1	507.9	518.6	552.2	mg/dm ²

Zr-1.0Fe Alloy (Steam, W)

Alloy	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-1.0Fe	N101-6	33.8	44.1	59.2	74.0	85.9		mg/dm2
Zr-1.0Fe	N101-7	38.0						mg/dm2
Zr-1.0Fe	N101-8	35.2	45.2	59.9	75.2	86.7	97.7	mg/dm2
Zr-1.0Fe	N101-9	34.5	44.4	59.2	74.0	85.5	97.5	mg/dm2
Zr-1.0Fe	N101-10	34.8	44.7	60.0	75.0	87.1	97.8	mg/dm2

Zr-1.0Fe-0.5Cr Alloy (Steam, W)

Alloy	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-1.0Fe-0.5Cr	N102-6	26.9						mg/dm2
Zr-1.0Fe-0.5Cr	N102-7	25.2	33.2	44.7	57.1	66.2		mg/dm2
Zr-1.0Fe-0.5Cr	N102-8	25.8	33.7	45.6	58.1	67.0	77.9	mg/dm2
Zr-1.0Fe-0.5Cr	N102-9	24.4	31.9	44.2	56.1	65.4	76.4	mg/dm2
Zr-1.0Fe-0.5Cr	N102-10	26.1	33.5	45.9	58.6	69.0	82.0	mg/dm2

Zr-0.6Fe Alloy (Steam, W)

Alloy	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-0.6Fe	N201-6	33.8						mg/dm2
Zr-0.6Fe	N201-7	34.4	44.9	58.0	71.8	82.1		mg/dm2
Zr-0.6Fe	N201-8	34.3	45.1	58.5	73.4	84.0	94.2	mg/dm2
Zr-0.6Fe	N201-9	35.2	45.5	59.3	74.2	84.4	94.5	mg/dm2
Zr-0.6Fe	N201-10	35.0	45.0	58.8	73.1	83.6	93.6	mg/dm2

Zr-0.6Fe-0.3Cr Alloy (Steam, W)

Alloy	Sample days	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	
		1.0	2.0	5.0	10.0	15.0	20.0	
Zr-0.6Fe-0.3Cr	N202-6	26.1						mg/dm2
Zr-0.6Fe-0.3Cr	N202-7	26.4	33.2	43.4	54.5	63.8		mg/dm2
Zr-0.6Fe-0.3Cr	N202-8	26.1	32.9	43.4	55.1	64.4	74.8	mg/dm2
Zr-0.6Fe-0.3Cr	N202-9	25.4	32.3	42.5	54.2	63.4	73.4	mg/dm2
Zr-0.6Fe-0.3Cr	N202-10	26.1	32.9	43.7	55.3	64.5	74.4	mg/dm2

Zr-0.6Fe-0.3Mo Alloy (Steam, W)

Alloy	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-0.6Fe-0.3Mo	N203-6	37.7						mg/dm2
Zr-0.6Fe-0.3Mo	N203-7	39.2	48.1	62.0				mg/dm2
Zr-0.6Fe-0.3Mo	N203-8	38.6	47.8	61.4				mg/dm2
Zr-0.6Fe-0.3Mo	N203-9	32.5	41.7	56.2	71.4	82.9	93.8	mg/dm2
Zr-0.6Fe-0.3Mo	N203-10	42.9	52.2	66.4	80.6	90.9	101.4	mg/dm2

Zr-0.6Fe-0.6Nb Alloy (Steam, W)

Alloy	Sample days	Gain(1)	Gain(2)	Gain(3)	Gain(4)	Gain(5)	Gain(6)	
		1.0	2.0	5.0	10.0	15.0	20.0	
Zr-0.6Fe-0.6Nb	N204-6	30.6						mg/dm2
Zr-0.6Fe-0.6Nb	N204-7	32.4	45.0	66.7	87.8	107.6		mg/dm2
Zr-0.6Fe-0.6Nb	N204-8	30.9	41.8	62.0	82.0	103.9	143.0	mg/dm2
Zr-0.6Fe-0.6Nb	N204-9	31.5	42.2	62.2	80.9	100.0	135.9	mg/dm2
Zr-0.6Fe-0.6Nb	N204-10	32.8	44.6	65.8	87.6	109.2	145.8	mg/dm2

Zr-1.0Cr-0.5Fe Alloy (Steam, W)

Alloy	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-1.0Cr-0.5Fe	N301-6	22.9	30.3					mg/dm2
Zr-1.0Cr-0.5Fe	N301-7	23.3	30.6	42.6	53.0	60.9		mg/dm2
Zr-1.0Cr-0.5Fe	N301-8	23.6	30.8	41.9	53.1	61.5	72.8	mg/dm2
Zr-1.0Cr-0.5Fe	N301-9	23.8	30.7	42.9	53.8	61.8	73.2	mg/dm2
Zr-1.0Cr-0.5Fe	N301-10	23.4	30.4	41.7	52.4	60.9	72.6	mg/dm2

Zr-1.0Cr-0.5Mo Alloy (Steam, W)

	Sample days	Gain(1) 1.0	Gain(2) 2.0	Gain(3) 5.0	Gain(4) 10.0	Gain(5) 15.0	Gain(6) 20.0	
Zr-1.0Cr-0.5Mo	N302-6	23.7						mg/dm2
Zr-1.0Cr-0.5Mo	N302-7	24.2	31.1	42.9	55.4	64.9		mg/dm2
Zr-1.0Cr-0.5Mo	N302-8	24.6	31.4	42.2	55.3	65.2	76.6	mg/dm2
Zr-1.0Cr-0.5Mo	N302-9	25.1	32.0	43.4	55.5	65.2	76.7	mg/dm2
Zr-1.0Cr-0.5Mo	N302-10	24.2						mg/dm2

SCW - Static autoclave - KAERI

ZrNb Alloys (SCW, KAERI)

days	0	1	3	6	9	12	15	18	21	24	30
Zr0.2Nb	0	spall									
Zr0.4Nb	0.0	32.4	56.8	99.6	139.5	171.8	212.1	242.8	265.7	300.1	329.7
Zr1.0Nb	0.0	35.0	58.6	100.2	135.9	166.2	205.3	236.5	263.0	292.7	325.9
Zr1.5Nb	0.0	35.5	61.1	98.1	132.1	161.3	193.8	220.0	243.7	264.8	293.4
Zr2.5Nb	0.0	36.0	60.0	97.3	131.8	162.7	201.4	231.9	258.3	285.1	317.6

days	36	42	48	60	66	72	78	84	96	108	120	132
Zr0.2Nb												
Zr0.4Nb	395.0	419.5	477.6	576.6	585.3	635.3	670.2	719.6	788.0	850.0	895.2	968.6
Zr1.0Nb	386.8	410.5	460.9	551.0	571.5	608.5	659.8	698.2	759.7	828.9	877.8	941.2
Zr1.5Nb	352.0	366.4	414.5	483.0	508.4	556.2	593.7	633.4	685.7	752.4	799.6	863.4
Zr2.5Nb	384.0	400.3	450.6	534.7	553.3	576.2	627.7	678.0	733.2	799.0	840.9	898.0

days	144	156	168	180	192	204	216	228	240	
Zr0.2Nb										
Zr0.4Nb	1032.9	1092.6	1159.5	1222.0	1286.7	1338.6	1384.0	1435.4	1486.8	mg/dm2
Zr1.0Nb	1010.1	1075.9	1146.4	1213.9	1283.2	1336.6	1390.2	1439.2	1496.2	mg/dm2
Zr1.5Nb	936.0	1001.2	1066.7	1137.8	1207.6	1268.2	1312.8	1373.2	1434.5	mg/dm2
Zr2.5Nb	964.2	1020.2	1076.3	1137.7	1201.3	1254.6	1301.7	1359.6	1419.9	mg/dm2

ZrNbSn Alloys (SCW, KAERI)

days	0	1	3	6	
Zr0.4Sn0.2Nb	0	58.67437	75.24797	spall	mg/dm2
Zr0.4Sn0.4Nb	0	40.35063	57.87763	spall	mg/dm2

ZrSn Alloys (SCW, KAERI)

days	0	1	
Zr0.2Sn	0	spall	mg/dm2
Zr0.4Sn	0	spall	mg/dm2
Zr0.8Sn	0	spall	mg/dm2
Zr1.2Sn	0	spall	mg/dm2

ZrFeCr Alloys (SCW, KAERI)

days	0	1	3	6	9	12	15	18	21	24	30	36	42
Zr0.2Fe0.1Cr(L)	0	28.6	spall										
Zr0.2Fe0.1Cr(H)	0	38.4	spall										
Zr0.4Fe0.2Cr(L)	0	28.9	48.6	71.0	78.1	87.1	94.8	100.0	106.4	112.4	117.0	127.9	130.5
Zr0.4Fe0.2Cr(H)	0	29.5	50.0	77.7	85.7	94.8	103.9	113.9	127.9	151.1	190.6	235.3	268.2

days	48	60	66	72	78	84	96	108	120	132	144	156
Zr0.2Fe0.1Cr(L)												
Zr0.2Fe0.1Cr(H)												
Zr0.4Fe0.2Cr(L)	153.8	183.8	193.9	236.6	270.0	319.1	358.4	398.2	465.2	483.1	501.0	531.4
Zr0.4Fe0.2Cr(H)	323.9	384.0	401.6	461.3	508.3	563.2	649.4	711.7	782.7	794.8	826.0	878.4

days	168	180	192	204	216	228	240	
Zr0.2Fe0.1Cr(L)								mg/dm ²
Zr0.2Fe0.1Cr(H)								mg/dm ²
Zr0.4Fe0.2Cr(L)	560.8	594.4	624.2	678.9	697.5	736.2	770.6	mg/dm ²
Zr0.4Fe0.2Cr(H)	941.7	1004.2	1053.0	1115.1	1131.1	1187.8	1219.5	mg/dm ²

ZrCrFe Alloys (SCW, KAERI)

days	0	1	3	6	9	12	15	18	21	24	30	36	42
Zr0.5Cr	0	53.7	1011.4										
Zr0.5Cr0.2Fe	0	28.8	44.3	55.6	66.6	77.9	88.9	94.7	101.4	108.7	113.0	123.3	124.3
Zr1.0Cr	0	30.1	46.4	57.2	67.0	76.6	85.7	90.5	96.7	106.3	114.3	130.0	133.7
Zr1.0Cr0.2Fe	0	28.9	44.6	56.2	67.5	79.3	90.3	95.7	102.0	109.5	112.1	125.4	123.1

days	48	60	66	72	78	84	96	108	120	132	144	156	168
Zr0.5Cr													
Zr0.5Cr0.2Fe	142.1	166.4	166.7	175.3	187.3	210.6	248.0	282.9	309.5	328.8	356.5	392.9	421.8
Zr1.0Cr	155.9	191.8	196.1	206.3	224.2	253.5	294.3	332.6	354.7	384.2	401.9	417.7	437.7
Zr1.0Cr0.2Fe	133.9	153.3	154.5	156.3	160.7	170.0	180.2	199.3	208.0	220.2	231.8	246.2	259.0

days	180	192	204	216	228	240	
Zr0.5Cr							mg/dm2
Zr0.5Cr0.2Fe	450.8	470.4	494.6	501.9	513.5	506.7	mg/dm2
Zr1.0Cr	454.9	469.0	475.9	484.4	491.1	499.0	mg/dm2
Zr1.0Cr0.2Fe	273.5	284.8	301.3	309.6	324.5	343.6	mg/dm2

ZrCuMo Alloys (SCW, KAERI)

days	0	1	3	6	9	12	15	18	21	24	30	36
Zr0.5Cu	0	51.6	79.6	89.0	98.4	109.7	120.4	125.4	131.4	139.6	143.5	164.9
Zr0.5Cu0.5Mo	0	847.8	spall									
Zr1.0Cu	0	35.2	55.2	64.2	74.9	86.7	99.6	108.4	117.0	125.2	131.8	143.9
Zr1.0Cu0.5Mo	0	41.3	63.1	72.5	86.1	100.2	115.2	128.1	140.1	147.1	150.3	173.0

days	42	48	60	66	72	78	84	96	108	120	132
Zr0.5Cu	164.1	219.0	260.7	263.8	264.6	334.5	367.8	395.6	459.5	504.7	553.8
Zr0.5Cu0.5Mo											
Zr1.0Cu	145.8	189.4	241.8	242.8	254.3	284.2	340.3	369.7	414.6	467.5	491.3
Zr1.0Cu0.5Mo	199.2	290.4	393.3	409.8	432.6	509.3	579.5	spall			

[illegible]

Reference Alloys (SCW, KAERI)

days	0	1	
Zr sponge	0	spall	mg/dm2
Crystal bar	0	spall	mg/dm2
Zircaloy-4	0	2461.7	mg/dm2

New Alloys (SCW, KAERI)

days	0	1	3	6	12	18	24	30	36	48	60	72	84
Zr1.0Fe	0	56.4	106.4	123.8	135.2	143.4	157.1	161.6	176.0	202.6	263.3		
Zr1.0Fe0.5Cr	0	49.6	96.1	105.9	109.0	116.1	118.8	121.2	156.4	166.0	190.7	237.1	260.8
Zr0.6Fe	0	56.2	106.8	124.4	133.9	140.8	171.3	168.3	245.3	277.6	370.7	442.1	474.3
Zr0.6Fe0.3Cr	0	49.1	100.0	105.7	114.0	120.7	142.1	153.2	234.8	288.0	338.3	428.9	443.3
Zr0.6Fe0.3Mo	0	61.9	116.1	122.9	127.0	136.3	159.5	166.1	222.2	257.8	317.3	330.3	351.7
Zr0.6Fe0.6Nb	0	98.7	193.4	256.9	347.8	377.1	443.1	540.7	579.1	686.0	781.2	886.8	1039.3
Zr1.0Cr0.5Fe	0	51.3	93.4	122.0	74.8	80.1	85.0	85.0	81.3	88.9	102.9	108.2	111.0
Zr1.0Cr0.5Mo	0	45.8	89.9	94.8	99.5	108.3	127.1	147.6	174.9	202.7	232.1	268.3	295.9

days	96	108	120	132	144	156	180	192	
Zr1.0Fe	274.0	342.1	391.6	463.9	492.3	564.0	656.5	662.3	mg/dm ²
Zr1.0Fe0.5Cr	271.5	290.7	309.0	332.7	349.9	379.8	404.1	416.2	mg/dm ²
Zr0.6Fe	570.6	652.7	694.6	758.3	784.9	859.5	939.4	975.5	mg/dm ²
Zr0.6Fe0.3Cr	451.2	485.9	535.5	598.9	644.4	711.2	785.5	819.2	mg/dm ²
Zr0.6Fe0.3Mo	489.4	619.4	698.1	758.7	784.9	831.4	863.7	882.8	mg/dm ²
Zr0.6Fe0.6Nb	1135.0	1216.2	1304.5	1413.5	1488.1	1572.0	1715.9	1799.2	mg/dm ²
Zr1.0Cr0.5Fe	115.0	125.9	129.4	140.0	150.0	164.1	171.7	175.7	(Partially spalled) mg/dm ²
Zr1.0Cr0.5Mo	315.6	338.0	360.9	380.9	399.4	417.3	445.4	466.9	mg/dm ²

SCW - Dynamic Autoclave - UM

Experimental alloys tested in this study.

Alloy system	Alloys and composition (wt%)
Zr-Fe-Cr	Zr-0.2Fe-0.1Cr
	Zr-0.4Fe-0.2Cr
Zr-Cr-Fe	Zr-0.5Cr
	Zr-0.5Cr-0.2Fe
	Zr-1.0Cr
	Zr-1.0Cr-0.2Fe
Zr-Cu-Mo	Zr-0.5Cu
	Zr-0.5Cu-0.5Mo
	Zr-1.0Cu
	Zr-1.0Cu-0.5Mo

Average weight gain (for two samples) of tested alloys after exposure for 24 hours.

Alloy system	Alloy	Ave. weight gain, 24 hr. (mg/dm ²)
Zr-Fe-Cr	Zr-0.2Fe-0.1Cr (720°)	27.9
	Zr-0.4Fe-0.2Cr (580°)	25.5
	Zr-0.4Fe-0.2Cr (720°)	29.5
Zr-Cr-Fe	Zr-0.5Cr	4282.2
	Zr-0.5Cr-0.2Fe	32.0
	Zr-1.0Cr-0.2Fe	38.0
Zr-Cu-Mo	Zr-0.5Cu	3966.5
	Zr-0.5Cu-0.5Mo	-1669.2 (spalling)
	Zr-1.0Cu	550.3
	Zr-1.0Cu-0.5Mo	576
Reference	Zircaloy-4	disintegrated

ZrFeCr and ZrCrFe Alloys (SCR, UM)

Alloy	1	2	3	6	9	15	21	30	60	90	120	150	days
Zr-0.2Fe-0.1Cr (H) - alloy 12	31.7	0	46.9	57.7	65	77.8	86.5	98.1	130.3	157.4	181.8	210.7	mg/dm2
Zr-0.4Fe-0.2Cr (L) - alloy 13	26.2	0	42.3	51.7	59.4	73.2	81.1	91.8	118.1	144.6	163.6	183.5	mg/dm2
Zr-0.4Fe-0.2Cr (H) - alloy 14	31.7	0	48.8	60.6	68.8	84.4	94.4	108	146.1	197.8	242.7	305.8	mg/dm2
Zr-0.5Cr-0.2Fe - alloy 22	33.2	42.9	47.1	58.1	66.1	78.8	88.2	100.5	125.2	157.9	179.8	204.1	mg/dm2
Zr-1.0Cr-0.2Fe - alloy 24	38.1	47.2	53.8	64.8	73.8	91.1	105.6	122.9	168.1	260.8	341.6	422.7	mg/dm2

APPENDIX - D

Alloys and samples examined in cross-section with the microbeam

Alloys Examined with Microbeam Synchrotron Radiation - water samples

<i>Alloys Examined</i>	<i>Sample designation</i>	<i>Exposure time (days)</i>	<i>Weight Gain (mg/dm²)</i>	<i>Oxide thickness (μm)</i>
Zr-0.2Fe-0.1Cr (L)	11-4	210	41	2.73
Zr-0.2Fe-0.1Cr (L)	11-1T-2 and 11-1L-2	409	124.9	8.3
Zr-0.2Fe-0.1Cr (L)	12-1	415.5	55	3.7
Zr-0.4Fe-0.2Cr (H)	14-2	354	43.4	2.89
Zr-0.5Cr	21-4	90	701	46.73
Zr-0.5Cr	21-3	90	725.6	48.37
Zr-0.5Cr	21-12	28	53	3.53
Zr-0.5Cr-0.2Fe	22-9	83.3	86.4	5.76
Zr-0.5Cr-0.2Fe	22-15	116	55	3.67
Zr-1.0Cr-0.2Fe	23-15	146	430.6	28.71
Zr-1.0Cr-0.2Fe	24-2	270	67.6	4.51
Zr-1.0Cu	33-10	173.3	42.4	2.83
Zr-1.0Cu	33-1L-2	409	103.6	6.9
Zr-1Cu-0.5Mo	34-3	210	168.5	11.23
Reference Sponge Zr	41-9	69.3	51.6	3.44
Reference Crystal Bar Zr	42-10	69.3	91.2	6.08
Reference Zircaloy-4	43-10	176.1	53.9	3.59
Zr-0.2Sn	WL11-7	60	115.48	7.70
Zr-1.2Sn	WL14-8	60	196.2	13.08
Zr-0.2Nb	WL21-7	616	105.46	7.03
Zr-1.5Nb	WL42-1	646	129.98	8.67
Zr-0.4Nb-0.4Sn	WL32-7	616	89.68	5.98

The figures are a collection of diffraction patterns, showing diffracted intensities (in arbitrary units) versus two-theta diffraction angle as a function of position in the cross sectional samples, with the μ-beam translating from the metal, across the metal/oxide interface and inward the oxide layer, to the oxide/water interface.

3d-plots summarizing the microbeam experiments for the water 360°C samples

11-4

11-1T and 11-1L

a)

b)

12-1

14-2

21-4

21-3

21-12

22-9

22-15

23-15

24-2

33-10

33-1L-2

34-3

41-9

42-10

43-10

Zr-0.2Sn

Zr-1.2Sn

Zr-0.2Nb

Zr-1.5Nb rolling and transverse directions

a)

b)

Zr-0.4Nb-0.4Sn

Alloys Examined with Microbeam Synchrotron Radiation - high temperature and commercial alloys

<i>Alloys Examined</i>	<i>Sample designation</i>	<i>Exposure time (days)</i>	<i>environment</i>	<i>Weight Gain (mg/dm²)</i>	<i>Oxide thickness (μm)</i>
Zr-0.2Fe-0.1Cr (H)	12	60	500°C at 3500 psi SCW	130.3	8.80
Zr-0.4Fe-0.2Cr (L)	13	60	500°C at 3500 psi SCW	118.1	7.98
Zr-0.4Fe-0.2Cr (H)	14	60	500°C at 3500 psi SCW	146.1	9.87
Zr-0.5Cr-0.2Fe	22	60	500°C at 3500 psi SCW	125.2	8.46
Zr-1.0Cr-0.2Fe	24	60	500°C at 3500 psi SCW	168.1	11.36
Zr-0.4Nb	SC22-4	300	500°C at 3500 psi SCW	1718.5	114.6
Zr-1.5Nb	SC42-6	252	500°C at 3500 psi SCW	1466.12	97.7
Zr-0.2Fe-0.1Cr (L)	11-17	3.75 d	500°C at 1500 psi steam	Not measured	Not measured
Zr-0.4Fe-0.2Cr (H)	14-18T-2	n/a	500°C at 1500 psi steam	n/a	8.0
Zr-0.5Cr	21-18	6 hrs	500°C at 1500 psi steam	724	48.27
Zr-1Cr-0.2Fe	24-17	17.3	500°C at 1500 psi steam	75.6	5.04
Zr-1.5Nb	ST42-2 (2 samples)	30	500°C at 1500 psi steam	332.86	22.2
Zr-2.5Nb tube (commercial)	C36 other direction	784	360°C water	190.5	12.7
ZIRLO tube (commercial)	E26 other direction	784	360°C water	272.2	18.15
ZIRLO sheet (commercial)	T-349 other direction	665	360°C water	245.4	16.4

Zr-0.2Fe-0.1Cr (H) - SCW

Zr-0.4Fe-0.2Cr (L) - SCW

Zr-0.4Fe-0.2Cr (H) - SCW

Zr-0.4Nb- SCW

Zr-1.5Nb- SCW

Zr-0.2Fe-0.1Cr (L) – 500°C Steam

Zr-0.4Fe-0.2Cr (H) - 500°C Steam

Zr-0.5Cr - 500°C Steam

Zr-1.5Nb - 500°C Steam

ST42-2Long
Zr-1.5Nb

ST-42-2rolling
Zr-1.5Nb

Zr-2.5Nb tube (commercial) - 500°C Steam

ZIRLO tube (commercial) - 500°C Steam

ZIRLO sheet (commercial) - 500°C Steam

T-349-Other Direction
ZIRLO

APPENDIX - E

Pole Figures from Model Alloy Oxides in Frontal Geometry

1BM Pole Figures

360°C Water

11-9: $\theta=3$, 360°C Water, 2.5 μm

← RD

TD

TD

← RD

RD

21-4: 360°C Water, 46.73 μm

← TD

← TD

RD

TD

←

TD

23-15: 360°C Water, 28.71 μm

TD

23-15: Repeat, 360°C Water, 28.71 μm

← TD

TD

← RD

TD

RD

TD

RD

TD

← TD

RD

RD

TD

KR4310 (110)M

KR4310 (011)M

KR4310 (-111)M

KR4310 (111)M

KR4310 (111)M

KR4310 (-201)M

TD

RD

← RD

RD

RD

500°C Steam

← RD

TD

TD

RD

RD

← TD

23-17: 500°C Steam, spall

E -60

TD

TD

← RD

43-17: 500°C Steam, 0.5 dd, 46 μm

RD

RD

500°C SCW

RD

RD

RD

RD

RD

550°C Air

RD

← RD

RD

Appendix F

Characterization of As-Fabricated Model Alloys

Figure F-1: Optical micrographs of sponge Zr, alloy 41 **a)** 200x and **b)** 500x.

Figure F-2: Optical micrographs of crystal bar Zr, alloy 42 **a)** 200x and **b)** 500x.

Figure F-3: Optical micrographs of alloy 43, Zircaloy-4 **a)** 200x and **b)** 500x.

Figure F-4: Optical micrographs of **a)** alloy 11, Zr-0.2Fe-0.1Cr annealed at 580°C 200x and **b)** alloy 12, Zr-0.2Fe-0.1Cr annealed at 720°C 200x.

Basal Pole Figures for Model Alloys

The pole figures are shown in Figure F-5

Figure F-5: Zr HCP (0002) pole figures for the ZrFeCr and ZrCrFe alloys (SRD = Sheet Rolling Direction, STD = Sheet Transverse Direction, SND = Sheet Normal Direction).

Figure F-6: Zr HCP (0002) pole figures for the ZrCu, ZrCuMo, Sponge Zr and Zircaloy-4 (SRD = Sheet Rolling Direction, STD = Sheet Transverse Direction, SND = Sheet Normal Direction).

Figure F-7: Zr HCP (0002) pole figures for the ZrSn and ZrNb alloys (SRD = Sheet Rolling Direction, STD = Sheet Transverse Direction, SND = Sheet Normal Direction).