Sensitivity of Measured Steam Oxidation Kinetics to Atmospheric Control and Impurities

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Abstract

The most direct means of improving the ability of water cooled reactors to withstand excessive cladding oxidation during a loss of coolant accident is to either modify or replace zirconium cladding. It is important to understand what level of agreement is to be expected as a function of systematic differences in steam oxidation testing techniques and instrumentation among testing facilities. The present study was designed to assess the sensitivities of some of the current and proposed reactor cladding materials. Steam oxidation sensitivity of Zircaloy-2, FeCrAl and Mo to O₂ impurities in steam were examined. It was shown that the effect of O₂ impurities is negligible for the two former materials while significant in the case of Mo.

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1. Introduction

Excessive cladding oxidation is the most prominent failure mode for a light water reactor (LWR) nuclear fuel during an extended loss of cooling accident scenario.[1] The most direct means of improving the ability of water cooled reactors to withstand such an occurrence is to either modify or replace zirconium cladding with a material that exhibits improved steam oxidation resistance at elevated temperatures. It is generally accepted that a candidate cladding material must display oxidation resistance to steam at temperatures in excess of 1200°C for it to be considered accident tolerant with respect to steam oxidation.[1]

A number of worldwide research programs are currently evaluating material systems with this objective. Proposed cladding options extend from Fe-based alloys optimized for oxidation resistance and performance under LWR irradiation conditions to revolutionary composites that incorporate functionally graded coatings to mitigate oxidation at high temperature.

The oxidation resistance of a candidate cladding material is one of many metrics that a material must satisfy to be considered for commercial deployment, though it is one of the more readily tested given the focus on accident tolerant fuel cladding development. National laboratories, universities, and industrial partners all contribute to the broad base of data on the oxidation tolerance of various candidate cladding materials, each employing nonstandardized experimental equipment and testing procedures. It is important to understand what level of agreement is to be expected as a function of these systematic differences to both facilitate academic dialog and assess observed discrepancies.

Recent screening tests performed to evaluate molybdenum as a possible LWR cladding material provide an extreme example of such discrepancies. Nelson *et al.* concluded that, while Mo will oxidize rapidly in air at high temperatures, the oxidation rate is muted significantly in water vapor.[2] However, Pint *et al.* observed nearly three orders of magnitude higher oxidation under reportedly similar conditions.[3] Identification of the source of this difference in observed performance is essential to assessing the feasibility of Mo-based LWR cladding concepts. Further, the cause of such a substantial variance in what is conventionally believed to be a straightforward testing technique may have far reaching implications to the development, deployment, and ultimate success of certain accident tolerant cladding concepts.

In academic reporting, typically only the percentage of steam used is

reported, for example 10% water vapor in either air or Ar is a common test parameter for steam turbine applications. If air ingress is excluded[4] evaluation of cladding materials for LWR service with an emphasis on accident behavior instead requires focus on water alone as the oxidizing species. Therefore, research in this area tends to focus on testing under 100% steam atmospheres. However, the means used by researchers to obtain and verify the purity of steam used to perform cladding oxidation studies is largely unreported in academic literature. The absence of applicable steam oxidation standards further complicates comparison of oxidation measured among laboratories.

The present study was designed to assess the sensitivities of current and advanced reactor cladding materials to O₂ impurities in steam introduced by testing protocols or instrumentation. The aim of the work is not to provide minimum performance criteria for test instrumentation, but rather to motivate more detailed reporting of experimental parameters, (i.e. including flow rates, purge/carrier gases, system evacuation procedures, etc), since these can have an effect on the residual O₂ in the system.

2. Steam Oxidation Testing

There are several experimental variables to be considered when steam oxidation testing. The authors will briefly outline a number of these variables, but the bulk of these are not the focus of the experimental work presented; though the effects of these variables are worth consideration by a researcher when planning and reporting on steam oxidation. Many are limited by the instrumentation available to a researcher.

2.1. *Dynamic vs Static Testing*

There are two ways to monitor steam oxidation rates during testing: dynamic and static. In a dynamic oxidation test, either the sample's weight change is monitored in-situ via thermogravimetric analysis (TGA)[5] or the amount of hydrogen off-gas, the byproduct of the steam oxidation reaction, is measured using a variety of methods such as utilization of an eudiometer[6, 7] or a calibrated mass spectrometer at the exhaust[4, 8, 7]. In a static test, the sample weight is measured at the beginning and end of an oxidation test. Multiple oxidation tests are required to study oxidation dynamics via static testing. However, these two methods should produce comparable results for the same material and atmospheric conditions. Static oxidation testing is

73 widely used due to its ease of set-up and sample accommodation, and al-
74 though data collection is more cumbersome, enhanced flow uniformity and
75 relaxed limits on specimen geometry are possible.

76 *2.2. Sample Configuration*

77 Sample configuration is likely the second most widely varied experimental
78 condition. Static testing systems can accommodate multiple samples in a
79 single test. Samples can be either hung vertically or aligned horizontally
80 in a tube furnace while steam is purged through the tube. The nature of
81 dynamic testing in a TGA requires one sample is tested at a time. Sample
82 placement on either a flat surface of hanging fixture introduces yet another
83 important variable: the flow velocity of steam across the sample surface.
84 Inherently a hanging sample will experience a more laminar flow pattern. Of
85 course sample materials can vary depending on lot and supplier, necessitating
86 independent elemental analysis to truly compare one study to the next.

87 *2.3. Steam Purity*

88 Other variables which can introduce inconsistencies in reported data from
89 one test system to another include furnace fixturing, thermocouple type,
90 placement and calibration, gas flow velocities, thermal profiles (ramp vs
91 isothermal oxidation testing), and gas purity.

92 The effect of steam purity will be the focus of this correspondence, as it
93 can lead to orders of magnitude discrepancies in measured oxidation when
94 available O_2 is the oxidation rate limiting factor. Several of the candidate
95 claddings currently under investigation are variants of materials which have
96 well understood behavior in steam and O_2 environments, e.g. Cr- and Al-
97 containing steels. As lesser-understood families of materials are explored,
98 little experience exists concerning their steam oxidation behavior and there-
99 fore more attention must be paid to the experimental testing parameters
100 which could affect the measured oxidation.

101 *2.3.1. Sources of Oxygen Impurities in Steam*

102 For materials which do not form passivating oxide layers, O_2 control
103 becomes paramount to accurate steam oxidation reporting, particularly at
104 $T < 1000^\circ\text{C}$. Oxygen impurities can originate from a number of sources.

105 2.3.1.1 Air Ingress

106 Air ingress during sample loading can be minimized using vacuum cycles prior
107 to testing provided the system is equipped with a vacuum pump. The ability
108 to evacuate a system prior to testing is necessary to efficiently minimize O₂
109 contamination. In addition, furnaces with open exhaust ports to air near
110 the sample can lead to additional air ingress during testing. In addition to
111 air ingress during sample introduction and back-flow of air through exhaust
112 ports, atmospheric oxygen leaks in gas fittings and equipment mating seals
113 are a challenge inherent to any process where purge/carrier gas purity is of
114 extreme importance.

115 2.3.1.2 Carrier and Purge Gases

116 Most steam oxidation testing systems employ carrier and purge gases to pro-
117 vide for efficient steam flow through the system. Using an air cover gas will
118 inherently increase the O₂ in the system. Even commercially purchased gases
119 can contain meaningful levels of oxygen impurities. For example, commer-
120 cially purchased ‘Ultra High Purity’ (UHP) Ar is often measured to have up
121 to 15 parts per million (ppm) O₂. For samples with strong oxygen affinities,
122 such as zirconium, molybdenum, and tungsten, this can cause oxidation in
123 an atmosphere otherwise believed to be inert.

124 2.3.1.3 Partial Pressure of O₂ in High Temperature Steam

125 In addition to system leaks and purge gas impurities, steam dissociates at
126 elevated temperatures to produce a temperature dependent amount of O₂.
127 The partial pressure of O₂ (p_{O_2}) in increasing steam pressures is plotted as a
128 function of temperature in Figure 1. The p_{O_2} in steam was estimated using
129 a law of mass action calculation. At 1 atm H₂O, the p_{O_2} corresponds to
130 approximately 0.007 ppm O₂ at 600 °C. However at 1250°C, there is over
131 120 ppm O₂ in steam. Therefore a 10 ppm O₂ leak at 600°C introduces
132 three orders of magnitude more oxygen to the system than would be present
133 in a “pure steam” test; however at T>1200°C a 10 ppm leak adds less than
134 10% O₂ to the amount already present in steam at that temperature. For
135 materials in which O₂ is the primary oxidizing reactant rather than H₂O, the
136 10 ppm leak at 600°C leads to significantly higher oxidation. This will be
137 exemplified in the results section of this correspondence.

138 In addition to O_2 dissociation causing increased p_{O_2} , O_2 has a significant
139 (several ppm) solubility in water.[9] The water reservoirs used to feed steam
140 generators are pressurized by a backing gas to encourage flow. If this backing
141 gas or reservoir atmosphere contains O_2 , the O_2 in the steam generated will
142 be elevated due to dissolved O_2 in the water source.

143 3. Experimental

144 Three materials, Molybdenum, Zircaloy-2 (zirconium alloy), and APMT
145 (Fe-22Cr-5Al-3Mo), were chosen to test and compare the oxidation results
146 among various steam oxidation testing configurations. Each of these materi-
147 als display varied oxidation kinetics, allowing the researchers to test the effect
148 of O_2 impurities on very different candidate cladding material families. Mo
149 represents one of the more novel cladding coating concepts and has been the
150 source of the previously mentioned discrepancies in measured oxidation.[2, 3]
151 Zirconium alloys are currently deployed in LWRs and have a long history of
152 steam oxidation testing. Lastly, APMT represents one of the leading families
153 of accident tolerant cladding concepts given its demonstrated resistance to
154 steam oxidation coupled with acceptable retention of mechanical properties
155 under irradiation.[10]

156 The three steam oxidation testing configurations used were a TGA (Dyn-
157 THERM LP-HT-II Instrument, Rubotherm, Bochum, Germany), custom
158 high temperature furnace (HT Furnace), and simultaneous thermal analyzer
159 (STA 449 F3, Netzsch Instruments, Selb, Germany). These systems were cho-
160 sen for comparison because they span the breadth of instrumentation used for
161 steam oxidation testing. The Rubotherm and STA F3 are both capable of dy-
162 namic testing, though the Rubotherm is not evacuated prior to testing while
163 the STA F3 employs a turbo vacuum pump for evacuation and extensive O_2
164 monitoring instrumentation. Conversely, the HT furnace is capable of static
165 testing multiple samples to 1700°C in steam at very high steam throughputs
166 (250 mL/h water) from a custom steam generator. The Rubotherm TGA
167 was used for a variety of Zircaloy-2 and APMT tests. The sample hangs
168 vertically, allowing laminar flow. A 50 mL/min UHP Ar purge was used
169 prior to steam exposure to flush residual O_2 from the system. The HT fur-
170 nace was used for several Mo tests. The vertical sample orientation ensures
171 laminar flow across the HT furnace and Rubotherm tested samples, while an
172 Al_2O_3 platform was used in the STA F3. The STA F3 was used to test all
173 three materials under varying experimental conditions and is equipped with

174 a water vapor generator (DV2ML, Astream, Germany) capable of up to 20
175 mL/h water throughput, and oxygen sensors (RapidOx OEM447, Cambridge
176 Sensotec) on both the inlet and outlet to the furnace. The HT furnace and
177 Rubotherm were not evacuated prior to the oxidation tests and were simply
178 purged with UHP Ar. The test capabilities and configurations of all three
179 systems are summarized in Table 1.

180 Several experimental conditions could be held constant to aid in the com-
181 parison. The Mo, Zircaloy-2, and APMT tested in each system came from
182 the same respective lots. All systems were allowed to equilibrate at the test
183 temperature under an Ar purge, before steam was introduced. The ramp
184 rates and dwell times were held constant between the two systems. At each
185 laboratory, the samples were weighed on bench top balances before and after
186 steam exposure. The experimental results reported in this correspondence
187 will demonstrate the effect of ppm levels of O₂ contamination in steam test-
188 ing as well as the oxidation that can occur prior to steam exposure during
189 isothermal testing when the inert ramp to temperature utilizes purge gas with
190 ppm levels of contamination. Finally the effect of testing multiple samples
191 simultaneously will be discussed.

192 4. Results & Discussion

193 The three materials used exemplify the need for accurate and thorough
194 reporting of experimental conditions. Testing each of these materials under
195 varied conditions provides data to support the occurrence of discrepancies
196 among reported oxidation rates for similar materials tested under like con-
197 ditions. In an effort to better understand the sensitivity of these materials
198 to O₂ impurities, the purge and carrier gases plumbed to the STA F3 were
199 doped with 1-100 ppm O₂ to simulate O₂ leaks characteristic of most steam
200 oxidation systems.

201 The comparative results are displayed in Table 2.

202 4.1. Molybdenum

203 A specialty gas bottle with 100 ppm O₂ in Ar was first plumbed to the
204 inlets of the STA F3 for the steam carrier gas and the protective gas. The
205 environment in the STA F3 during the 700°C isothermal exposure was 0.55
206 atm water vapor with approximately 30 ppm O₂ measured on the outlet O₂
207 sensor. The partial pressure of steam is calculated by taking into account the
208 local atmospheric pressure, (0.728 atm at Los Alamos National Laboratory),

209 and any dilution of the steam by both the carrier and protective gases. This
 210 was repeated using UHP Ar, which contained approximately 10 ppm O₂,
 211 resulting in a little over 2.5 ppm O₂ in the STA. Lastly, the gettered Ar was
 212 used as both the carrier and protective gas, while the system had a known 1
 213 ppm O₂ leak. This data is compared to that obtained in 2013 ($p_{O_2} < 10^{-12}$ ppm)
 214 in Figure 2.

215 This sensitivity to O₂ is explained fairly simply using the known oxidation
 216 mechanisms of Mo.[6, 11] First Mo is oxidized to MoO₂ and MoO₃ by the
 217 existing O₂ in the system via the reactions below:

218 The rate of MoO₂ and MoO₃ formation is driven by availability of O₂ in
 219 the test atmosphere. The resulting MoO₂ is readily oxidized further to form
 220 MoO₃ via the following reaction:

Lastly, the MoO₃ is volatile in water vapor, forming molybdic acid (MoO₃-
 H₂O, also reported as MoO₂(OH)₂).[6, 11, 12]

221 Therefore, we understand the sensitivity of Mo to ppm levels of O₂ in
 222 steam as the additional formation of MoO₃, which is readily volatile in water
 223 vapor at these temperatures. It should be noted that Mo is directly oxidized
 224 by steam, however that reaction alone would not explain the discrepancies
 225 observed in this study. Furthermore, at 700°C the p_{O_2} in pure steam is <0.1
 226 ppm. Therefore a 10 ppm O₂ leak adds two orders of magnitude more O₂ to
 227 drive the formation of MoO₃, leading to more volatilization and subsequent
 228 mass loss of the sample. Similar behavior can be expected with low tempera-
 229 ture (T<1000°C) volatile hydroxide forming metals such as tungsten.[12, 13]

230 Not addressed here experimentally is the effect of flow rate/steam velocity
 231 on the oxidation of Mo. It is expected that increased flow rate will enhance
 232 the volatilization rate of MoO₃(g) and MoO₂(OH)₂(g). However Pint *et al.*
 233 report less mass loss at increased flow velocity.[3] It is hypothesized here that
 234 the decrease in mass loss at increased flow rate was due to a reduction in the

oxygen impurities that were either present in the system at the start of the experiment or introduced at the exhaust of the system during operation. The dependence of oxidation on steam velocity in systems when volatile oxides and hydroxides dominate the mass loss behavior requires further study. The flow rates used during steam oxidation testing should be reported to facilitate the comparison of results among varied studies.

4.1.1. *Effect of Testing Multiple Samples*

It should also be noted that the presence of other samples during oxidation testing can effect the oxidation results of a given sample. Since the HT furnace could accommodate up to five samples, multiple Mo foils along with Zircaloy-2 samples were oxidized simultaneously. The Mo tested along side other materials showed much less oxidation than if the one sample was solely exposed to steam (-0.88 vs -9.27 mg/cm²). This is due to a reduced local partial pressure of O₂ for each individual Mo sample. The data reported for the HT Furnace in Table 2 represents a single sample exposed to steam.

4.2. *Zircaloy-2*

Zircaloy-2 was exposed to 1000°C steam with increasing ppm levels of O₂ in the same manor as Mo in the STA F3. No difference was seen in either the total oxidation of the sample (i.e. mg/cm² calculated based upon the final measured weight gain) or in the discerned oxidation kinetics from the TGA curve. However, if O₂ is the only oxidizing agent present, increases in oxidation were witnessed by incrementally increasing the O₂ content in Ar. Figure 3 displays what is intended to be an inert isothermal test of Zircaloy-2. The oxidation test system had known oxygen impurities. The inlet p_{O₂} was 10⁻¹⁵ parts per million (ppm). The system outlet, after 3 vacuum cycles, is 1 ppm O₂, indicative of a leak in the test system. The Zircaloy-2 sample experiences 1% weight gain during the thermal profile, which ramps to 1000°C. During both the ramp and cool down, the outlet p_{O₂} signal drops by up to 10 orders of magnitude, indicating that at these temperatures during the ramp and cool down, the Zircaloy-2 getters O₂. This result also indicates that oxide formation during the ramp of of an isothermal steam test can affect the overall oxidation witnessed by the sample, since the existence of a surface oxide (a diffusion barrier) prior to steam exposure can alter the oxidation kinetics once the steam is introduced.

Oxygen impurities in steam do not prove to be the source of discrepancy in the case of Zircaloy-2. It is worth noting that historically the reported

271 oxidation of Zircaloy at 1100°C tends to vary significantly from study to
272 study. Table 3 gives provides data from several studies performed on this
273 material over the last 50 years. Several other experimental factors could lead
274 to the broadly varied oxidation results reported, including but not limited
275 to temperature control, steam flow velocity, and ramp vs isothermal testing.
276 These factors are not explored further in the present work, but again should
277 be kept in mind as potential variables that may contribute to difference
278 among experimental observations for future families of materials.

279 4.3. APMT

280 Since APMT is known to oxidize very little up to 1200°C due to the
281 formation of a passive oxide layer, we do not expect trace amounts of O₂
282 to affect the oxidation level of the cladding material. This hypothesis was
283 confirmed by adding incremental ppm levels of O₂ to steam in the STA F3,
284 as was done with Mo and Zy-2, and no changes in either the total oxidation
285 oxygen levels above 100 ppm O₂ in steam will be present when testing at
286 temperatures above 1200°C. Therefore impurities of a reasonable level (below
287 100 ppm) should have little effect on materials not prone to oxidation below
288 1200°C, such as several FeCrAl alloys.

289 5. Conclusions

290 Steam oxidation testing of three materials, APMT, Zircaloy-2, and molyb-
291 denum was conducted by isolating system configuration and steam purity as
292 experimental variables. For materials which exhibit diffusion limited oxida-
293 tion kinetics, little sensitivity to O₂ contamination was observed. Further-
294 more, APMT and Zircaloy-2 displayed no sensitivity to 1-100 ppm levels of
295 O₂ contamination in steam, while Mo oxidation showed a clear dependence
296 at temperatures well below 1200°C.

297 The reported 9.29 mg/cm² mass loss in the HT furnace in just 1 hour
298 would lead most researchers to disqualify Mo as a candidate cladding ma-
299 terial. However, the 0.25 mg/cm² mass loss observed in the STA F3 could
300 imply that only a slightly reducing water chemistry in an LWR would be
301 necessary to make this a viable cladding candidate. The reporting of test
302 conditions, particularly the control of O₂, is increasingly important to the
303 qualification of a lesser-studied candidate cladding materials such as Mo.

304 6. Acknowledgments

305 This work was supported by the U.S. Department of Energy, Office of
306 Nuclear Energy Fuel Cycle Research and Development program.

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Table 1: Steam oxidation test systems' configuration and capabilities

System	Measurement Type	Atmosphere Control	Sample Orientation	Temperature Limit	Maximum Steam Flow Rate	Sample Capacity
HT Furnace	Static	Purge Gas	hanging	1700°C	250 mL/hr	5
Rubotherm	Dynamic	Purge Gas	hanging	1550°C	5 mL/hr	1
STA F3	Dynamic	Pump/Purge & O ₂ monitoring	platform	1250°C	20 mL/hr	1

Table 2: Single Sample Comparison between Steam Oxidation Test Systems

Sample	Temperature (°C)	Duration (hours)	Atmosphere	STA F3 (mg/cm ²)	HT Furnace (mg/cm ²)	Rubotherm (mg/cm ²)
Zircaloy-2	1100	1	100% Steam	33		25.77
Mo Foil	700	4	100% Steam	-0.25	-9.27 -0.88*	
Mo Foil	700	4	Steam + 1 ppm O ₂	-0.3	—	—
Mo Foil	700	4	Steam + 10 ppm O ₂	-0.35	—	—
Mo Foil	700	4	Steam + 100 ppm O ₂	-0.38	—	—
APMT	1200	4	100% Steam	0.29		0.33

*Tested with multiple samples

Table 3: 1100°C, 1 h, Steam Oxidation of Zirc. Alloys as predicted by previous studies

Authors	mg/cm ²	year	Alloy	Reference
Steinbrück	40	2011	Zry-4	[14]
Moalem-Olander	29	1991	Zry-4	[5]
Pawel-Cathart	17	1979	Zry-4	[15]
Urbanic-Heidrick	74	1978	Zry-4	[16]
Baker-Just	71	1962	Zirconium	[17]

Figure 1: Plot of the partial pressure of O₂ in 0.25, 0.5, 0.75, and 1.0 atm H₂O as calculated using the Law of Mass Action.

Figure 2: Mo steam oxidation at 700°C with incremental amounts of O_2 added to the carrier and protective gases.

Figure 3: Plotted are the outlet and inlet pO_2 sensor readings for a “gettered Ar” experiment. Outlet O_2 sensor indicates that the sample “getters” O_2 at certain temperature ranges. The TGA curve for the Zircaloy-2 sample is plotted on the second y-axis in Mass %, and the thermal profile is plotted on the third y-axis.