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Oxidation investigation of cladding specimens for regular and accident tolerant fuel rods under LOCA conditions

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Abstract. The high-temperature oxidation tests were carried out for the regular fuel rod claddings specimens made of sponge-based zirconium alloy (E110G) and for the accident tolerant fuel (ATF) ones – pure vacuum melted molybdenum (VCPM) and niobium alloy (Nb-1%Zr). The tests were carried out under the ambient pressure \( p \approx 0.1 \text{ MPa} \) in pure water steam. The experimental data on the oxidation characteristics were obtained for E110G specimens in the temperature range \( T = 1100 \text{ – } 1500 \text{°C} \), that for VCPM and Nb-1%Zr are investigated under extended temperature-duration range (more than 1 hour). The thermal effects of molybdenum (\( Q_{SMR} \)) and niobium (\( Q_{SNR} \)) interactions with steam were defined and the derived oxidation rate constants for refractory metals were compared with the known ones. Based on the computations performed with PARAM-TG code the high-temperature oxidation characteristics of model fuel assemblies of large-scale facilities under LOCA conditions with regular and ATF claddings were compared. It was shown that Zr-steam interaction of fuel rod cladding (\( Q_{SZR} \)) is more intensive compared with VCPM and Nb-1%Zr ones under investigated conditions.

1. Introduction
After the Fukushima Dai Ichii disaster in 2011 the accident tolerant fuels (ATF) are developed throughout the world. ATF is expected to be the «nuclear fuel – construction material» system which meets the increased agrees of reactor nominal operation conditions as well as under Loss-Of-Coolant-Accidents (LOCA) and Reactivity-Initiated Accidents (RIA) conditions. To prevent the intensive steam-zirconium reaction the modifying of regular rod claddings (corrosion-resistant coating) as well as the material change (FeCrAl steels [1], SiC ceramics [2]) are examined. Long-term ATF versions foresees the modifying of UO\(_2\) composition in the fuel pellets on the dense, high thermal conducting and refractory ones (UN, U\(_3\)Si\(_2\)) [3].

Refractory metals and their alloys are perspective materials for ATF claddings usage [4]. One of the most perspective refractory material is molybdenum. Its high melting point (\( T_{\text{Mbo}} = 2623 \text{°C} \)), the modulus of elasticity (\( E_{\text{Mbo}} = 310-330 \text{ GPa} \)) and thermal conductivity coefficient (\( \lambda_{\text{Mbo}} = 95 – 125 \text{ (W/(m·K))} \)) while low thermal linear expansion coefficient (\( \alpha_{\text{Mbo}} = (8,0 – 10,0) \cdot 10^6 \text{ 1/K} \)) under comparatively low density (\( \rho_{\text{Mbo}} = 10,23 \text{ g/cm}^3 \)) allow to develop the Mo-based alloys with large specific strength [5]. For example, high strength performance and creep resistance demonstrate the alloys based on molybdenum doped with titan...
and zirconium (BM-1, IJMA-2, TZM). Although niobium represents a bit worse thermal and mechanical properties it is still in interest due to high manufacturability and low price.

Light water reactors VVER and PWR, installed with regular zirconium cores as well as refractory ATF ones must keep safety under nominal operating and accident conditions. Heat up and cool down performance of regular and advanced LWR’s under LOCA conditions take place during the heat and mass transfer to two-phase flow [6], core construction elements degradation and high-temperature oxidation of fuel rods claddings at water steam medium. For example molybdenum trioxide at atmospheric pressure and the temperature above 500 °C starts to volatile and close to melting point ($T_{m\text{MoO}_3} = 801 \, ^{\circ}\text{C}$) MoO$_3$ formation and volatility rates become almost equal [7]. The chemical reaction rate constant, thermal effects of Mo-steam and Nb-steam interactions, accompanied with hydrogen generation, at temperatures above 1200 °C and atmospheric pressure have not been investigated in details to nowadays.

One of key problems to implement Mo and Nb alloys to nuclear applications use is the high absorption cross-section of thermal neutrons ($\sigma_{\text{Mo}} = 2.4$ barns, $\sigma_{\text{Nb}} = 1.15$ barns) which is about 8-15 times larger than that of zirconium [8]. The probable way to solve this problem for molybdenum is development of two layer composite cladding. Its prototype could be of reduced thickness comparing with the regular and consisted of small Zr outer layer ($\delta_{\text{Zr}}/\delta_{\text{clad}} \approx 0.2$) and the Mo base. Such solution realizes the advantages of refractory metal to meet the ATF requirements and weaken the neutron physics penalties.

It’s known that under nominal LWR water-chemical regime Mo corrosion rate is evaluated as about 10 µm/month [9]. During three year operating period in core the loss of cladding thickness is prognosed as half of initial state. Thus the molybdenum cladding have to be guarded by different ways – two layer cladding manufacturing or metallic corrosion-resistant coating. In present study aforesaid problems were not considered and analyzed.

The scope of study is: a) experimental and computation investigation of zirconium regular rod claddings (E110G alloy) and vacuum-melted pure VCPM molybdenum specimens oxidation performance in the temperature range up to 1500 °C in water steam; b) evaluation of physico-chemical parameters – thermal effects of Mo-steam and Nb-steam reaction constants; c) benchmark performance of fuel assemblies thermal hydraulics made of regular «UO$_2$ - Zr» fuel rods and ATF rods with advanced refractory claddings under LOCA conditions.

2. Specimens and test procedure

High-temperature oxidation tests of fuel rod cladding specimens made of sponge-based zirconium alloy (E110G) and vacuum-melted pure molybdenum (VCPM) were carried out using tube fragments of 10 – 12 mm long. E110G specimens dimensions ($D \times d$) were 9.13x7.73 mm and that corresponded to VVER-1000 regular fuel rod cladding and 9.55x8.33 mm corresponded to PWR-type one. VCPM specimens were 16.9x2.5 mm and 9.9 mm in length. The chemical composition of E110G specimens contained Zr as base element, 0.9-1.1 % wt. Nb doping, 0.1 % wt. O and 0.003 % wt. H as impurities. VCPM specimens consisted of 99.92 % wt. Mo, 0.005 % wt. O and N and less than 0.03 % wt. C.

High-temperature interaction of regular zirconium fuel rod claddings and ATF refractory ones with steam were studied at GASPAR facility [10]. Oxidation tests of specimens mounted on tungsten-rhenium sheathed thermocouple BP 5/20 and covered junction 5 mm in outer diameter were carried out in ceramic tube 28 mm in OD and 2 mm wall thickness installed inside the furnace heater made of graphite (figure 1).

The study of initial surface condition and roughness of specimens forewent each test as well as their
mass measurement on analytic weight balances Shimadzu AUW 220 with 0.1 mg uncertainty error. Before the run started the inner cavity is cleaned of O₂-containing media and air by argon flow. Superheated steam with mass rate from 0.005 g/s up to 0.010 g/s with temperature 900 °C fed up with high-temperature region of the furnace. The oxidation of fuel rod claddings specimens were carried out under steady state conditions in the furnace 60 mm long isothermal region (spatial temperature gradient did not exceed 0.5 K/mm) at temperature from 800 °C up to 1500 °C. The exposure latency of high-temperature oxidation tests of fuel rod cladding specimens at 1500 °C is limited by 300 seconds. Each fuel rod cladding specimen was extracted out of the furnace isothermal region and cooled down in steam at cool rate at least 20 K/s. In some tests VCPM Mo specimens were cooled-down in isothermal region by steam with rate 4-5 K/s as the electric power supplied to furnace test section was decreased. As the high-temperature oxidation finished the specimens were weighed on analytic weight balances.

3. High-temperature oxidation tests results
The oxidation tests of regular E110 sponge-based fuel rod claddings were carried out in the temperature range 1100 – 1500 °C. Oxidation of ATF VCPM specimens exposed up to 3600 seconds at maximal temperature in steam was performed at 1200 °C and 1400 °C at atmospheric pressure. As it was established previously the temperature of E110G fuel rod claddings specimens exceeded that of furnace and this effect is determined by steam-zirconium reaction heat release [11]:

\[ Zr + 2H_2O = ZrO_2 + 2H_2 + Q_{SZR} \]  

(1)

In figure 2 the oxidation regimes of E110G alloy (a) and molybdenum VCPM (b) rod claddings specimens under the high-temperature oxidation test conditions at GASPAR facility are shown.

\[ q_t \] – time-dependent heat generation in the furnace test section during cool-down phase

Figure 2. High-temperature oxidation regimes of fuel rod claddings specimens in steam:

a) - E110G alloy, b) – VCPM Mo
Additional heat up of E110G alloy specimen in the furnace test section at 1200 °C due to Zr-steam reaction \( \Delta T_{gZr} \) \( (Q_{SZR} = 6.28 \text{ MJ/kg Zr}) \) had the value of 25 – 30 °C under permanent steam supply conditions (figure 2a). The evident temperature rise \( \Delta T_{gMo} \) due to Mo-steam reaction was not observed in the temperature range from 900 °C to 1400 °C (figure 2b). The detailed study showed \( \Delta T_{gMo} \) was close to 8-10 °C at furnace test section 1200 °C. That value is in range of BP5/20 thermocouple
measurement error. Thus molybdenum oxidation is less intensive compared with that of E110G alloy in the temperature range 1200 °C – 1400 °C.

The results of specific weight gain measurements for the zirconium E110G alloy and molybdenum VCPM specimens oxidized at 1200 °C and 1400 °C are shown in figure 3. The exposure latency of VCPM specimens more than 3600 seconds caused by additional oxidation process during slow cool-down phase. The weight gain during this phase is also took into consideration indeed. The dependency of specific weight gain change on the exposure latency for E110G specimens agrees with the well-known concepts [12]. The mass change of molybdenum specimens is significantly less than that of zirconium specimens and the monotonous decrease is observed. As it can be seen the oxidation kinetics of E110G alloy is governed by parabolic law [13] at 1200 °C–1500 °C and exposure times up to 500 seconds. At the same time VCPM weight change is ruled by linear law [14] at 1200 °C–1400 °C and exposure times up to 5000 seconds. So the oxidation process of both metals has qualitative difference. As for regular zirconium alloy the ZrO2 layer formation which as being the diffusion barrier limits the steam supply to pure Zr. As molybdenum oxidizes the unsteady oxide MoO2 forms and in the presence of unlimited steam supply reduces to volatile MoO3 and is entrained with steam from specimen surface.

4. The evaluation of Mo-steam and Nb-steam reactions rate constants

When molybdenum reacts with water steam flow the gaseous (MoO)n, MoO2(OH) are formed and the metal surface becomes covered with MoO2 film. Assuming the reaction products to be the ones of first order let us form the Mo-steam interaction equation [7]

\[ Mo + 3H_2O = MoO_3 + 3H_2 + Q_{SMR} \quad (2) \]

The value of «Mo-H2O» reaction rate constant \( K_{Mo} \) is 31,9·10^-6 kg Mo/(m²·s) and 10,21·10^-3 kg Mo/(m²·s) at 1130 °C and 1700 °C respectively according to [11]. Let us assume the dependency of reaction rate constant on temperature is governed by Arrhenius equation at fixed absolute medium pressure

\[ K_p = A \cdot \exp\left[-E_A/(R \cdot T)\right] \quad (3) \]

where \( R \) – is universal gas constant 8.31 J/(mole·K), \( T \) – temperature, K. The activation energy \( E_A \) of chemical reaction and empiric coefficient \( A \) are determined by solving two-equation system at \( T_1 = 1130 \) °C and \( T_2 = 1700 \) °C:

\[ E_A = -R \cdot \frac{T_1 \cdot T_2}{T_1 - T_2} \cdot \ln\left(\frac{K_{p2}}{K_{p1}}\right) \quad (4), \quad A = K_{p2} \cdot \left(\frac{K_{p1}}{K_{p2}}\right)^{(T/T_1 - T_2)} \quad (5), \]

The dependency of Mo-steam reaction rate constant on temperature is

\[ K_{Mo} = 1.498 \cdot 10^4 \cdot \exp\left[-2.329 \cdot 10^5/(R \cdot T)\right] \quad (6) \]

\[ Q_{SMR} = E_A / \mu_{Mo} \quad (7) \]
According to (6) the thermal effect of molybdenum interaction with steam equals to \( Q_{SMR} = 2.43 \cdot 10^6 \text{ J/kg} \) and it is 2.6 times less than that of Zr-steam reaction \( Q_{SZR} = 6.28 \cdot 10^6 \text{ J/kg} \).

In [14] the oxidation parameters study of molybdenum specimens in steam in the temperature range \((600-1200) \text{ °C}\) was carried out. Data processing of experimental points on specific weight gain versus exposure time in steam allowed to determine the Arrhenius type chemical constant rate correlation

\[
K_{pMo}^{\text{Nel}} = 0.947 \cdot 10^3 \cdot \exp\left[-2.084 \cdot 10^5/(R \cdot T)\right]
\]  
\( (8) \)

The dependency of specific weight gain change on the exposure time at 1200 °C (figure 4) showed the data [14] and the author’s obtained results in present study are in good agreement. The obtained experimental data analysis and processing on the VCPM weight change during high-temperature steam oxidation at different temperatures allowed to define the Arrhenius equation parameters more accurately:

\[
K_{pMo}^{\text{auth}} = 0.651 \cdot 10^3 \cdot \exp\left[-2.192 \cdot 10^5/(R \cdot T)\right]
\]  
\( (9) \)

These ones were compared with the known data (figure 4). The chemical constant rate values predicted by (8) were extrapolated up to 2000 °C assuming the monotonous trend of line. As it can be seen the molybdenum volatilization rate in steam is strong-dependent on temperature and increases to 50-60 times in the range 1200 °C – 1600 °C. Due to such rapid dependence the accurate measurement of specimen temperature is very important. Moreover the obtained experimental data on the constant rate shows the decreased oxidation intensity compared with known ones up to 7 times. Probably these disagreements could be explained by three main reasons. The unprecized temperature measurements of specimen and steam supplied. Impurities composition in test specimens and the mass transfer conditions differs from test to test. Besides the form and orientation of specimens in horizontal laminar steam flow and vertical transient flow should be adjusted. The way of cool-down also contributes to determination of weight gain.

The similar way the constant rate on Nb-steam reaction was derived by processing of experimental array data points [15]

\[
K_{pNb} = 12.33 \cdot \exp\left[-1.221 \cdot 10^5/(R \cdot T)\right]
\]  
\( (10) \)

The thermal effect of niobium-steam reaction \( Q_{SNR} \) has value of 1.32 MJ/kg. Comparison of computation results shows that constant rate for molybdenum is one degree less than that of niobium at 1600 °C in steam.

5. Numerical simulation of Mo specimen oxidation in steam applied to GASPAR test section

The numerical simulation of aforesaid problem was performed in 2D steady-state axial-symmetric formulation using Ansys Fluent v.14 [16]. Oxidation process took place in test section represented by
tube 60 mm in length with inner diameter 28 mm. The specimen 9.9 mm in length had 16.9 mm outer
diameter and 2.5 mm wall thickness. The outer diameter of thermocouple sheath was 5 mm. The gravity
force was considered to be negligible. The steam flow was represented by laminar flow. The Mo-steam
interaction took place on the boundary surfaces of specimen and the first reaction rate was assumed.
Arrhenius equation parameters were given as $A = 0.651 \times 10^3$ kg/(m$^2$·s) and $E_A = 2.192 \times 10^5$ J/(mole·K).
Finite rate laminar was used so the rate constant depended on the Arrhenius equation only and did not
depend on flow mixing. The interdiffusion of mixture components is calculated using molecular-kinetic
theory. Thermal conductivity and viscosity of mixture were calculated according to the ideal gas
approach.

Steam with 0.010 g/s mass rate was supplied to the inlet cross-section of test section. Inlet steam
temperature was given as 1200 °C and 1400 °C. Adiabatic conditions were considered on the inner
surface of test section and thermocouple sheath.

The 2D distributions of steam-H$_2$ flow temperature and MoO$_3$ mass fraction are presented at figures
5a, 5b. As it can be seen the final mass fractions of MoO$_3$ does not exceed with 2.2 % and its peak value is
on the inner surface of specimen. Heat generation of SMR rises up almost to one grade and its amount is
3.1 mW and 26 mW respectively.

![Temperature Distribution](image1)

**Figure 5.** Oxidation characteristics distributions close to the
specimen heated up to 1400 °C: a) temperature of steam-H$_2$ flow,
b) MoO$_3$ mass fraction in steam-H$_2$ flow

The computation results of Mo specimen volatility showed the values $14.4 \times 10^{-6}$ kg/(m$^2$·s) at 1200 °C
and $118 \times 10^{-6}$ kg/(m$^2$·s) at 1400 °C respectively. That is in quite satisfactory agreement with
the experimental data obtained using gravimetric technique (figure 6).
In figure 6 the comparison of obtained experimental data on specific oxidation weight change and the well-known ones are presented. As it was previously mentioned the difference reaches to 7 times, determined by temperature-dependent trend lines. The results of numerical finite-element code computations are in good agreement with the author’s obtained data.

6. Thermal hydraulics performance of regular Zr and ATF-Mo fuel assemblies

Regular model FA consisted of 19 fuel rod simulators fixed with 6 spacer grids \[17\]. The core modeling zone 1275 mm in length consisted of fuel rods with UO$_2$ pellets and E110G alloy claddings surrounded by Zr shroud, ZrO$_2$ thermal insulation and stainless steel case. The core simulator was cooled down by ambient air. The heat up of model FA was realized by direct current flow in refractory Ta heaters implemented in the pellet slug of fuel rod simulators. Model ATF fuel assembly is the same construction but Zr claddings were replaced by VCPM Mo ones.

The numerical simulation of model FA heat up under LOCA conditions was performed using PARAM-TG thermal hydraulics code \[17\]. Computation 2D unsteady model took into consideration physico-chemical processes of metal interaction with steam as well as that of radiation heat transfer, axial thermal conductivity and linear thermal expansion. The thermal physics properties of given substances were considered to be temperature-dependent. The spatial distribution of heat release was considered to be inhomogenous due to temperature-dependent resistance of electric heaters.

Top and bottom boundaries of computation region assumed to be adiabatic, heat flux in the center of model FA symmetry was equal to zero and natural convective heat transfer considered to be on the outer surface of core simulator case. The initial condition was set as homogeneous temperature of core simulator equal to 300 °C. The model FA was heated up in pure steam.

Mo-steam oxidation kinetics of ATF fuel rods was took into account according to equation (9).

Assuming the unlimited supply of steam to zirconium surface high-temperature oxidation kinetics of E110G alloy is governed by parabolic law:

\[ \frac{d(\Delta m/S)}{dt} = K_{pZr}, \]  

(11)

where \( \Delta m \) – fuel rod cladding weight gain, kg, \( S \) – initial surface area, m$^2$, \( t \) – time, s. Zr-steam reaction rate constant can be calculated using Baker-Just correlation \[18\]:

\[ K_{pZr} = 3.33 \cdot 10^7 \cdot \exp\left[-1.905 \cdot 10^5/(R \cdot T)\right], \]  

(12)

where \( K_p \) is expressed in \((\text{mg Zr})^2/(\text{cm}^4 \cdot \text{s})\). As temperature exceeds with 1520 °C zirconium oxidation rate is changed according to \[19\]

\[ K_{pZr} = 87.9 \cdot 10^4 \cdot \exp\left[-1.381 \cdot 10^5/(R \cdot T)\right], \]  

(13)
where $K_p$ is expressed in (mg Zr)$^2/(cm^1\cdot s)$.

The computation results of regular and ATF fuel rods heat-up phase during LOCA are shown in figure 7a, 7b. As the power rate was proposed identical the cladding temperature and metal-steam reaction parameters were significantly different. Thus the influence of Zr-steam reaction starts to become apparent in about 2000 seconds while maximal cladding temperature reaches to ~1100 °C. The development of Mo-steam reaction becomes visible when rods have heated up to 1400 °C and that requires 3000 seconds more ($Q_{SZR}$ and $Q_{SMR}$ in figure 7). It can be seen the slope of Zr cladding oxidation rate curve ($\Delta \delta$) is changed while reaching temperature 1520 °C (equation 12). The more intensive oxidation kinetics governs the losses of pure zirconium in E110G alloy (equation 13). At the same time molybdenum oxidation kinetics is increased monotonous with temperature rise. After ~6500 seconds of heat-up phase complete oxidation of E110G alloy claddings with the ZrO$_2$ layer formation take place in the hottest zone and maximal cladding temperature of FA-Zr reaches to 1706 °C. Additional ~100 µm thickness layer is due to linear thermal expansion and material change ($\rho_{Zr} = 6.53$ g/cm$^3$, $\rho_{ZrO_2} = 5.72$ g/cm$^3$). It’s noticeable that local chemical power is equal to half of input electric one and the $Q_{SZR}$ is 20 % of total. The FA-Mo heats up to 1600 °C only after 8500 seconds while and the chemical power is that of 4-5 % of total power. The effective thickness layer of volatile MoO$_3$ does not exceed with 95 µm. Thus the FA-Mo shows better performance in LOCA conditions in comparison with FA-Zr and is the interest of further R&Ds.

![Figure 7](image_url)

**Figure 7.** Comparison of 19 rod regular and ATF model fuel assemblies under LOCA heat-up phase: a) – FA-Zr, b) – FA-Mo
Conslusion

1. The experimental oxidation investigation of regular and ATF fuel rod claddings specimens manufactured with zirconium E110G sponge-based alloy and molybdenum VCPM was carried out at GASPAR facility in the temperature range from 1200 °C to 1400 °C and exposure latencies up to 5000 seconds. The experimental results demonstrated higher Mo oxidation resistance (up to one grade) in comparison with the E110G alloy specimens.

2. Based on the author’s experimental data obtained in the temperature range 1200 °C - 1400 °C the oxidation constant rate for VCPM Mo fuel rod claddings specimens was developed. It was found the thermal effect of Mo-steam reaction is 2.6 times less than that of Zr-steam. The defined by experimental data processing thermal effect of niobium-steam reaction has value of 1.32 MJ/kg. Comparison of computation results shows that constant rate for Mo is one degree less than that of Nb at 1600 °C.

3. The comparison of Mo VCPM specific volatility values with the well-known data of past studies showed the difference reaching up to 7 times so the more accurate evaluation is required.

4. The pure molybdenum-steam oxidation model was developed and the computations of high-temperature oxidation of VCPM claddings specimens tested at GASPAR facility were executed using ANSYS package. The comparison of numerical simulation results and experimental data on Mo volatility showed the satisfactorial agreement.

5. The benchmark performance computations of model fuel assemblies manufactured with E110G alloy (FA-Zr) and ATF molybdenum VCPM (FA-Mo) claddings under LOCA heat-up phase were performed using PARAM-TG code. It was found that in identical terms FA-Zr fuel rod claddings reached 1700 °C, had become completely oxidized in the hottest zone. While the cladding thickness of FA-Mo ones decreased only by 16 %.

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