# ROUND ROBIN EXERCISE OF THE CANDIDATE ATF CLADDING MATERIALS WITHIN THE IAEA ACTOF PROJECT

Martin Ševeček, Jakub Krejčí, Adéla Chalupová, Jitka Kabátová, František Manoch, Jan Kočí, Ladislav Cvrček
*Czech Technical University in Prague/UJP Praha, Prague, Czech Republic*

Chongchong Tang, Martin Steinbrűck, Mirco Grosse
*Institute for Applied Materials, Karlsruhe Institute of Technology, Karlsruhe, Germany*

Sami Penttilä, Juha-Matti Autio, Jari Lydman, Aki Toivonen
*VTT Materials for Power Engineering, Technical Research Centre of Finland, Espoo, Finland*

Bożena Sartowska, Wojciech Starosta, Lech Waliś
*Institute of Nuclear Chemistry and Technology, Warsaw, Poland*

Zoltan Hózer, Tamás Novotny, Erzsébet Perezné-Feró, Anna Pintér Csordás, M. Horváth, Péter Szabó
*Hungarian Academy of Sciences, KFKI Atomic Energy Research Institute, Budapest, Hungary*

Claudia Giovedi
*University of São Paulo, São Paulo, Brazil*

Peng Xu
*Westinghouse Electric Company, Hopkins, SC, USA*

*The paper presents a summary of the round robin test activity organized within the IAEA ACTOF project. The test conditions and sample matrix were finalized during fall 2017 and the tests have been performed during the next 14 months. Two fundamental experimental tests related to normal operating conditions and accidental conditions of LWRs were defined: High-temperature steam oxidation and Long-term corrosion tests. Originally, four laboratories/institutes joined the round robin exercise – CTU in Prague, KIT, VTT, and INCT. Later, also MTA EK joined the activity. Zircaloy-2 substrate was provided by Westinghouse Electric Company and two institutes applied their protective coatings on its surface (CTU – pure Cr coating and INCT – ZrSi-Cr coating). KIT applied MAX phase coating on Zircaloy-4 substrate. Additionally, AISI 348 steel provided by USP, Brazil was tested. The geometry of the samples varied based on the needs of particular institutes. Long-term corrosion tests were performed by three laboratories in PWR and VVER chemistry for at least 63 cumulative days. High-temperature steam oxidation tests were performed by four institutes at three pre-defined conditions. Participants performed pre- and post-characterization of the material based on their standard procedures and available techniques. The paper summarizes motivation, plans, sample matrix, materials, testing conditions and preliminary results including lessons learnt. Full reports will be published in the ACTOF TECDOC under preparation.*

## I. INTRODUCTION

Fukushima-Daiichi accident in 2011 clearly showed that Zr-based cladding materials used in all Light Water Reactors (LWRs) worldwide negatively contribute to the progression of severe accidents. Even though they have been utilized in LWRs for decades and their performance is very good, there are, however, events when they worsen and accelerate the incidents such as Three Mile Island accident or Fukushima-Daiichi events [1], [2]. For that reason, new types of cladding materials are investigated all around the world. These new materials are generally titled as Accident Tolerant or Advanced Technology Fuels (ATF). The main objective of their development is to improve their accidental tolerance while maintaining or improving their performance in normal operating conditions and transients [3]–[5]. The development of new ATF candidates is supported also by international organizations such as OECD/NEA or International Atomic Energy Agency (IAEA). IAEA initiated in 2014 a coordinated research project (CRP) focused on ATF. The main objectives of the CRP are to:

- Support options for the development of nuclear fuel with improved tolerance in severe accident conditions
- Support modelling of new fuel designs with advanced cladding or fuel
- Acquire data through experiments on new fuel types and cladding materials to support their use for fuel with improved accident tolerance

There are many individual activities and three joint actions within the CRP titled “Analysis of Options and Experimental Examination of Fuels for Water-Cooled Reactors with Increased Accident Tolerance (ACTOF)”. One of the key joint actions planned was the round robin test (RRT) for cladding materials that are considered as Accident Tolerant or Advanced Technology Fuel (ATF) cladding candidates. The tested cladding materials include three coated cladding concepts and AISI stainless steel. There were two fundamental tests related to LWR operation defined – high-temperature steam oxidation test and long-term corrosion test in prototypical LWR conditions that focus on both accidental conditions as well as normal operating conditions.

### 1.1. RRT Objectives

There were many ATF candidate-cladding materials proposed and investigated around the world. The evaluation of the “accidental tolerance” might be, however, complex problem and can be evaluated using different parameters. Additionally, there have been standard methods for handling and testing of Zr-based alloys established, however, they might be not valid for new materials. Also, there might be new unexpected issues related to ATF materials that might arise during development and testing which are not relevant for standard cladding materials. These challenges led to the proposal of the round robin test with the following objectives:

- Test ATF cladding candidates proposed and provided by ACTOF members
- Study long-term corrosion behaviour of the ATF cladding materials in representative LWR conditions
- Study high-temperature steam resistance in LOCA-related predefined conditions
- Compare the methods, procedures and standards used in different laboratories
- Validate the currently used methods for ATF materials
- Evaluate the proposed ATF cladding materials based on the two fundamental tests performed and comparison with reference samples

The participants of the RRT are: Czech Technical University/UJP Praha (CTU), Karlsruhe Institute of Technology (KIT), Institute of Nuclear Chemistry and Technology (INCT), VTT Technical Research Centre of Finland (VTT), University of São Paulo (USP) and Hungarian Academy of Sciences Centre for Energy Research (MTA EK). The RRT can be subdivided into three sub-activities:

- ATF candidate cladding sample development and production
- Long-term corrosion test in VVER or PWR chemistry
- High-temperature oxidation

The involvement of the parties in the particular tasks of the RRT activity is summarized in Table 1.

TABLE I. Involvement of the participants of the RRT in the subtasks

| | CTU | KIT | INCT | MTA EK | VTT | USP |
|---|---|---|---|---|---|---|
| Sample production | X | X | X | | | X |
| Long-term corrosion | X | | X | | X | |
| High-temperature oxidation | X | X | | X | X | |

As can be seen in Table 1, there were four different types of ATF cladding samples produced by four institutes. Three institutes performed long-term corrosion tests in PWR or VVER chemistry and four institutes performed high-temperature oxidation testing.

### 1.2. RRT Timeline

The RRT was proposed during the second RCM meeting in June 2017. Several ACTOF participants were interested and the test matrix and involvement of the participants were finalized during fall 2017. UJP Praha and Westinghouse Electric Company provided Zr-based substrates and participant fabricated their ATF samples. The samples were transported during spring 2018 when testing started. Preliminary results were presented in November 2018 at the third RCM meeting. The results were finalized in February 2018 and summarized in a chapter of a new TECDOC under preparation. The timeline is shown in Figure 1.

Fig. 1. Timeline of the RRT joint activity.

## II. PRODUCTION OF THE ATF CANDIDATE SAMPLES

Traditional Zr-based cladding materials were used as substrates (Zry-2, Zry-4 and E110) and three different coatings were deposited on their surfaces using physical vapour deposition methods (PVD). Originally, standard Zry-2 LK3 alloy provided by Westinghouse Electric Company was chosen as the substrate for all tests with two predefined geometries. Due to time and other limitations, it was decided to use also Zry-4 substrate provided by KIT and E110 substrate provided by UJP Praha and change the sample geometry based on the requirements of particular participants (due to size limitations in furnaces, to allow for mechanical testing or single-sided oxidation etc.). The sample geometries are shown in Figure 2.

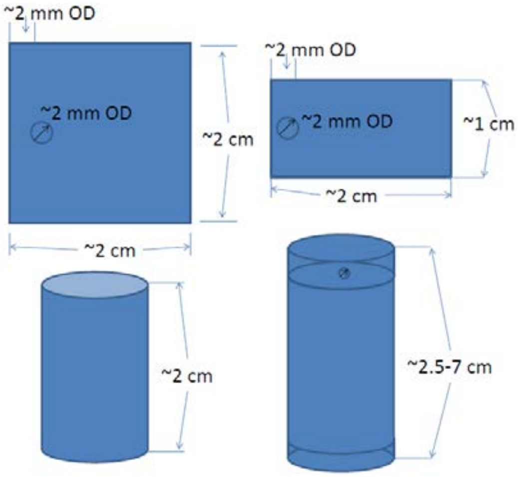

Fig. 2. The geometry of samples used in the RRT. Samples with coated as well as uncoated end caps were used.

The coatings deposited by PVD methods are: pure Cr (CTU), ZrSi-Cr (INCT) and Cr/$Cr_2AlC$/Cr (KIT). Additionally, AISI 348 stainless steel provided by USP was tested. The fabrication procedures and sample specifications are described further.

### 2.1. Cr PVD coated Zr-based alloys

Zr-based samples were Cr coated in two batches using unbalanced magnetron sputtering (UBM) in the Hauzer Flexicoat 850 industrial system. It is a multi-purpose device flexible for numerous applications. Depending on the type, it can utilize a combination of different deposition technologies such as sputtering, HIPIMS, PACVD and other. For the purposes of this work, UBM was used. It is a special type of sputtering with extended and increased plasma density using coils in an unbalanced closed magnetic field. The images of the system, deposition chamber and rotating sample holders with Cr target are shown in Figure 3. More details about the methods used and coating parameters can be found in [6]–[10].

The coatings were deposited in a metal mode using the gas flow 90 sccm Ar (99.999 %) for about 18 hours. The thickness of the coatings was measured with a Calotest (CSM, Switzerland) and was found to be 28 ± 0.1 µm in the first batch and 16 ± 0.1 µm in the second batch.

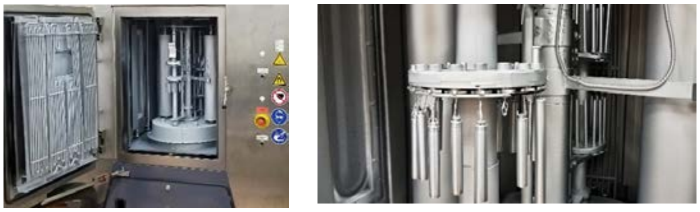

Fig. 3. Images of the Hauser Flexicoat 850 at CTU in Prague. Deposition chamber (left); Rotating sample holders with Cr target in the back (right).

### 2.2. ZrSi-Cr PVD coated Zry-2

ZrSi-Cr coated samples were produced at the Institute for Sustainable Technologies ITS (Radom, Poland). Balzers system shown in Figure 4 was used. The system contains three magnetron plasma sources, three power and control panels. It allows to deposit materials from separate targets and to obtain multi-elemental coatings [11].

Two magnetrons with separate, flat, circular targets were used. One was made of $ZrSi_2$ and the second of pure Cr. The resulting composition of the coatings as confirmed by EDS is - $Zr_{40}Si_{24}Cr_{36}$ and its thickness are about 2.5 microns.

Fig. 4. Balzers system facility: a) general view, b) position of flat, circular magnetrons, c) chamber overview

### 2.3. Cr/$Cr_2AlC$/Cr (MAX phase) PVD coated Zry-4

A research group at KIT investigates the deposition of different MAX phases on Zircaloy-4 by a PVD. Pre-tests have shown that the $Cr_2AlC$ MAX phases are the most promising with respect to their high-temperature oxidation behaviour. The coatings were synthesized via a two-step process, i.e. first deposited by magnetron sputtering and subsequently thermally annealed in pure argon. The as-deposited coatings were deposited using laboratory PVD equipment; three high-purity cylindrical elemental plates of chromium, graphite and aluminium are used as targets for magnetron sputtering process. Figure 5 shows the schematic representation of the design of the as-deposited coatings on Zircaloy-4 substrates and the arrangement of the targets and substrates during deposition.

After deposition, the coated Zircaloy-4 specimens were ex-situ annealed in pure argon using a commercial thermal balance (NETZSCH STA-449 F3 Jupiter) to facilitate the growth of MAX phases by the solid reaction of the nanoscale elemental multilayers. The isothermal holding time was 10 min at annealing temperature 550°C. $Cr_2AlC$ MAX phase was successfully obtained while no significant interdiffusion between the Cr and $Cr_2AlC$ layer has been confirmed at such conditions. [12], [13]

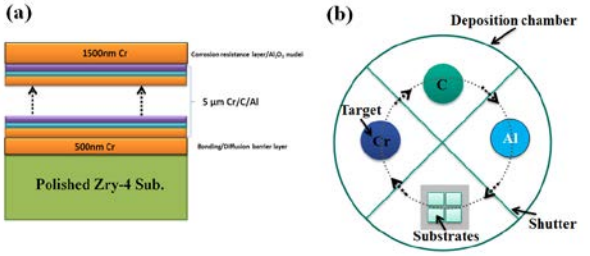

Fig. 5. Schematic representation of (a) the design of the as-deposited coatings on Zircaloy-4 substrates, and (b) arrangement of the targets and substrates during deposition.

### 2.4. AISI 348 SS

USP produced austenitic stainless steel AISI 348 samples within the RRT joint activity. There were two different geometries of austenitic stainless steel (AISI 348) samples supplied: tubes and plates. The composition and properties of both materials were the same [14]:

- Chemical composition: Fe-balance, C-0.055%, Mn-1.70%, P-0.017%, S-0.003%, Si-0.41%, Cr-17.5%, Ni-11%, Nb-0.85%, N-0.0018%, Co-0.021%, Ta<0.005%, B-0.0008%
- Micro-hardness: 150 - 200 HV
- Surface roughness: ≤ 0.7 µm Ra
- Mechanical properties at 20°C: tensile strength - 640 MPa, yield strength - 330 MPa, elongation of a 50 mm specimen - 47%
- Mechanical properties at 370°C: tensile strength - 455 MPa, yield strength - 260 MPa, elongation of a 50 mm specimen - 26%
- Non-metallic inclusions (ASTM E45, Table 1 [15]): ≤1
- Niobium carbides evaluation (ASTM E407 [16]): continuous non-stabilized areas < than 0.05 mm of equivalent diameter
- Intergranular corrosion (ASTM A262, Practice A [17]): material does not present intergranular cracks (magnification in the range 5x to 20x).

Plate samples were prepared from AISI 348 bars with a diameter of 22 mm. After machining, each plate sample was polished, identified and cleaned. Tubular samples were prepared by cutting long tubes of AISI 348 tubes with a diameter of 9.8 mm and a thickness of 0.6 mm.

## III. EXPERIMENTAL

### 3.1. Long-term corrosion

The long-term corrosion tests were done in two prototypical LWR chemistries – PWR (VTT, INCT) and VVER (CTU) at 360°C. The period of 21 days was defined with a goal of three minimal periods (63 cumulative days). Destructive testing was performed after 63 days but if there was enough material available, the tests continued for a longer time. The long-term corrosion tests performed by the participants are summarized in Table 2.

TABLE II. Long-term corrosion tests organized within the RRT performed by individual participants

| | **CTU – Czech Republic** | **VTT - Finland** | **INCT - Poland** |
|---|---|---|---|
| **Cladding material** | VVER chemistry - static | PWR chemistry - flowing | PWR chemistry - static |
| **AISI348** | 164 days | 63 days | 63 days |
| **Zry-2/Zry-2** | 164 days | 63 days | 63 days |
| **Cr coated Zry-2** | 164 days | 63 days | 63 days |
| **MAX phase coated Zry-4** | 147 days | 63 days | 63 days |
| **ZrSi-Cr coated Zry-2** | 101 days | 16.5 days | 63 days |

The experimental procedures vary depending on the available setups and standard procedures used at the institutes. For example, CTU/UJP uses 360°C, 19.4 MPa, 4 dm3 static autoclave, VTT used standard operational PWR parameters at 360°C with recirculation loop and online chemistry control whereas INCT used 360°C, 19.5 MPa, 1 $dm^3$ static autoclave.

The samples were characterized before, during and after testing by non-destructive as well as destructive methods. The characterization techniques include visual inspections, XRD, EDS, SEM, metallography, weight changes and hydrogen and oxygen pickup measurements. The change in water chemistry during testing was also analyzed.

### 3.2. High-temperature oxidation

Four participants performed high-temperature (HT) oxidation tests. The three required predefined conditions are:

1) Flowing steam 1100°C, 60 minutes
2) Flowing steam 1200°C, 30 minutes
3) Flowing steam 1300°C, 5 minutes

These parameters were defined based on estimates taking into account the standard DBA fuel safety criteria for Zr-based cladding materials such as 17% ECR limit calculated using the Baker-Just correlation [18]. Additionally, previous experience with testing of ATF cladding materials was used to estimate potential additional margins provided by the near-term ATF concepts tested and to determine reasonably long testing periods for all samples [13]. Some of the participants decided to perform more tests if larger quantities of samples were available. The summary of tested samples by particular institutes is shown in Tab. 3.

TABLE III. High-temperature oxidation tests performed by individual participants

| **ATF cladding material** | **CTU** | **VTT** | **KIT** | **MTA EK** |
|---|---|---|---|---|
| | Steam oxidation - 1100,60'; 1200,30'; 1300°C,5' | Steam oxidation - 1100,60'; 1200,30'; 1300°C,5' | Steam oxidation - 1100,60'; 1200,30'; 1300°C,5' | Steam oxidation - 1100,60'; 1100,180'; 1200,30';1200,45'; 1200°C,60' |
| AISI348 | X | X | X | - |
| Zry-4 ref. | - | X | X | - |
| Cr coated Zry-2 | X | X | X | - |
| MAX phase coated Zry-4 | X | X | X | - |
| ZrSi-Cr coated Zry-2 | - | - | X | - |
| E110 ref. | X | - | - | X |
| Cr coated E110 | X | - | - | X |

The experimental procedures vary depending on the experimental setup used. For example, the heating rates, steam flow rates, quench parameters, temperature measurements or sample holders are setup-dependent which might slightly affect the results. In addition, different experimental setups are able to measure different parameters such as hydrogen production or online weight changes. For example, the only KIT was able to measure hydrogen generation and on the other hand, only CTU and MTA EK performed post-quench mechanical testing.

The samples were characterized before and after testing. The characterization techniques include visual inspections, XRD, SEM, microhardness, metallography, ring compression tests, hydrogen production, weight changes or hydrogen absorption measurements.

## IV. RESULTS

### 4.1. Long-term corrosion

The results of the long-term corrosion tests show the expected corrosion behaviour of Zr-based alloys (Zry-2, E110) in both VVER and PWR chemistry. This confirms that applied setups and procedures are valid and acceptable and that they can be used also for ATF material testing. Additionally, the data measured on Zr-based alloys serve as a baseline for comparison with ATF candidate materials and their evaluation.

The results measured by all participants summarized in Figure 5 indicate drastic weight loss in the case of the MAX phase coated samples suggesting dissolutions and spallation of the coating for both VVER and PWR chemistry. The weight loss/spallation is slightly slower in VVER chemistry but still extreme. Since significant spallation was observed for this sample, the weight change is not fully representative parameter. It should be noted that residuals of coating system remained on the Zr substrate even after the drastic weight loss. This suggests that some parts of the coating system are stable in LWR conditions.

ZrSi-Cr coating shows comparable corrosion kinetics as uncoated reference samples. It should be also noted that with the more complex coating systems, both weight loss and gain were observed during the tests. This was the case for ZrSi-Cr coating as well as the MAX phase coating. In the case of ZrSi-Cr coating, a high concentration of Si-based ions was detected in the autoclave after the tests. This confirms that weight gain evaluation standardly used for Zr-based alloys might not be a suitable parameter for advanced materials. Additionally, the ZrSi-Cr PVD coated samples show very high hydrogen pickup.

Both, AISI 348 and Cr coated samples show extremely low weight gains. However, hydrogen concentration inside the Cr-coated Zr-based alloy is comparable to uncoated material suggesting higher H-pickup fraction of the coated sample. Figure 6 shows the weight gains of the tested samples tested up to 164 days.

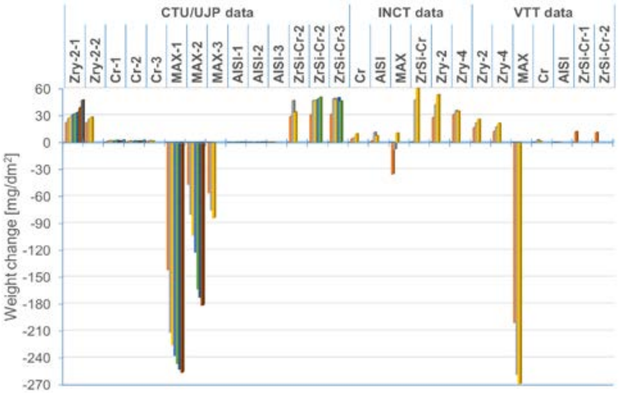

Fig. 6. Summary of all WGs measured by the three institutes involved up to 164 days as shown in Table II.

The detailed kinetics of Cr-coated samples and AISI are shown in Figure 7 in comparison with reference uncoated samples. Both ATF candidate materials show extremely low weight gains when the stable oxide is formed on their surface. Interestingly, it can be seen that Zry-2 shows higher weight gain in VVER chemistry. A transition in Zry-2 kinetics can be seen around 125 days.

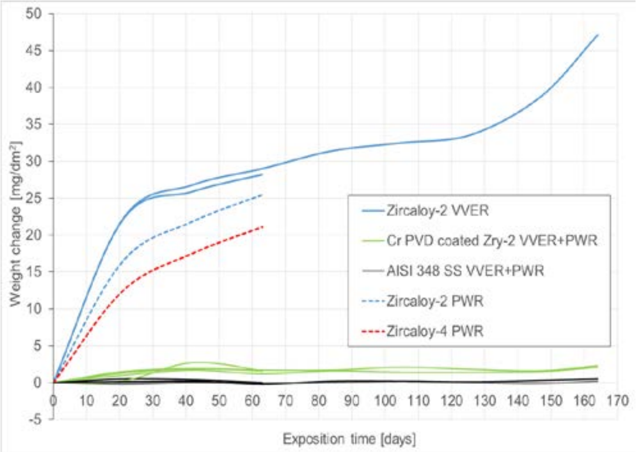

Fig. 7. Corrosion kinetics of two reference Zr-based alloys in comparison with two ATF candidates.

The highlights of the post-test characterization of samples tested in long-term corrosion tests are:

- MAX phase – drastic spallation and dissolution, but some residuals of the coating system remain (Figure 8)

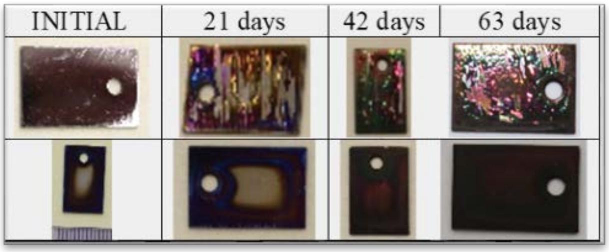

Fig. 8. Visual evaluation of the MAX phase coated sample

- ZrSi-Cr – slightly higher weight gains compared to uncoated samples; dissolution of silica and extremely high hydrogen pickup (Table 4)
- Cr – very low and stable weight gains; stable and strong oxide layer; comparable H-concentration to uncoated material
- AISI 348 – low weight gain but the Fe-rich oxide layer is very fragile

TABLE IV. Evaluation of hydrogen concentration inside the samples after 63 days

| | **Uncoated Zry-2** | **Cr PVD coated Zry-2** | **MAX phase PVD coated Zry-4** | **AISI348** | **ZrSi-Cr PVD coated Zry-2** |
|---|---|---|---|---|---|
| 63 days: H, ppm | 14.2 | 15.0 | 30.4 | 1.3; 1.4 | 191.8; 191.2 |

### 4.2. Long-term corrosion

The weight gains of the oxidized samples as a function of calculated weight gains using Cathcart-Pawel correlation [14] as measured at UJP Praha is shown in Figure 9. The results show a reduction of the weight gains for all of the tested samples in comparison with uncoated Zr-based cladding materials.

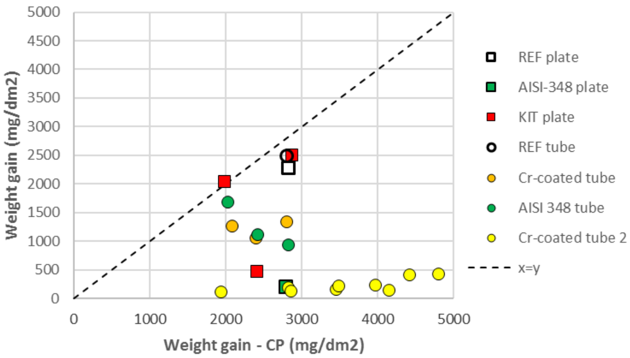

Fig. 9. Experimentally measured weight gains in relation with calculated weight gain using Cathcart-Pawel correlation (valid for most Zr-based alloys).

Additionally, the weight gains are directly linked with the oxidation kinetics for Zr-based alloys and the weight gain reduction suggests lower hydrogen production and limitations of the exothermic HT reaction with steam as well. These are the main required properties of ATF cladding materials.

However, the weight gain evaluation was established for standard Zr-based alloys to simplify their evaluation. Nevertheless, it does not fully cover the cladding performance during accidental conditions. The oxidation testing needs to be accompanied by mechanical testing to quantify the accidental tolerance. Similarly, to long-term corrosion tests, some of the samples showed extensive oxide and material spallation (e.g. AISI 348). For that reason, the weight change is a summation of oxidation weight gains and spallation (weight loss). The weight gain is from this perspective not a representative parameter for oxidation kinetics evaluation.

When evaluating the oxidation kinetics based on the hydrogen production measured during the tests, the results differ. The results of integral hydrogen production during the three predefined tests are shown in Figure 8. It can be seen that for example, the Cr-coated samples produce very low hydrogen that was suggested also by the low values of weight gains shown in Figure 7. On the other hand, the AISI 348 samples produce more hydrogen that uncoated Zr-based alloys but its weight gain is low.

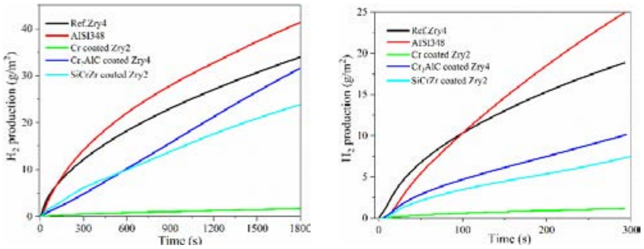

Fig. 10. Integral hydrogen production for 1200 °C, 30min and 1300 °C, 5min steam oxidation tests.

The tests show that weight gain is not a fully representative parameter and that it should not be solely used as a parameter to evaluate "accidental tolerance". The mechanical testing also showed that the ductile-to-brittle transition of coated materials can occur with lower ECR values than for Zr-based alloys. It does not mean that e.g. Cr coating does not enhance the safety, but it requires alternative criteria to determine DBT.

The highlights of the post-test characterization of samples tested in long-term corrosion tests are:

- AISI 348 – two different Fe-rich oxide layers found; non-protective and not fully dense. It leads to acceleration of the kinetics and higher H-production.
- MAX phase – not adherent close to edges, spallation; but in the central area protective effect is obvious.
- ZrSi-Cr – slightly reduces oxidation and H-production but oxygen diffuses through the coating and forms oxides underneath
- Cr – very low weight gains and H-production. Inter-diffusion of coating and substrate.

## V. CONCLUSIONS

An experimental program has been carried out with four types of ATF candidate cladding samples within the IAEA ACTOF CRP project. Four institutes produced their ATF candidate samples – Cr PVD coated Zr alloys, MAX phase PVD coated Zr alloys, ZrSi-Cr PVD coated Zr alloys and AISI 348 SS. Two fundamental tests were defined – high-temperature oxidation and long-term corrosion. Three participants performed long-term corrosion testing for minimal 63 days period in PWR or VVER chemistry. Four participants performed oxidation tests in steam and air atmosphere between 1100 °C and 1300 °C including characterization and mechanical testing.

When evaluating the performance of tested samples, it can be seen that ZrSi-Cr coated samples show higher weight gains and extremely high hydrogen pickup during long-term corrosion test compared to uncoated samples. Spallation and dissolution of MAX phase coated samples was observed in PWR as well as VVER chemistry during long-term corrosion tests. These two concepts are therefore not feasible for further development from the perspective of normal operation. AISI 348 SS, as well as Cr, coated samples show low weight gains and acceptable corrosion performance in both VVER and PWR chemistries. The hydrogen pickup of Cr-coated samples should be, however, further studied.

In the accidental conditions, the AISI 348 performs much worse than Cr coated samples and also worse than uncoated reference samples at extreme severe accidental conditions. AISI 348 does not form protective oxides at HT and shows rapid oxidation with excessive hydrogen production. It can be concluded that only Cr-coated cladding satisfies the requirements of ATF cladding materials when evaluating the four studied ATF concepts. Additionally, it was found that standard methods (e.g. weight gain evaluation) might not be fully representative for the evaluation and testing of new materials that differ from Zr-based alloys. The testing and industry standards related to handling, cleaning as well as testing are not fully developed and established for these new materials. Based on the results, it can be expected the new issues and phenomena not observed with Zr-based alloys might appear in future.

It should be noted that not all activities originally planned were finished due to the limited time of the ACTOF project but a new round-robin test will be initiated as part of future IAEA CRP projects. The activity was initiated during the second half of the ACTOF project and there were several delays caused by transport and regulatory issues, unavailability of required materials or funding. Despite the minor problems encountered during the RRT activity, there was a high number of valuable data produced by all participants. The RRT activity can be considered as very successful from the perspective of the involved parties as well as the community. The full report presented in the ACTOF TECDOC will include all the details about the production, testing methods and results that were produced within this RRT activity.

## ACKNOWLEDGMENTS

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