Contribution ID: **14** Type: **not specified**

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Tuesday 28 October 2025 10:40 (30 minutes)

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INTRODUCTION
This paper describes the materials and manufacturing processes of ATF concepts experimentally tested within the ATF-TS coordinated research project (CRP) from 2020 until 2025. Only cladding materials were tested and no fuels were provided to any participant. There are two possible development options for ATF claddings [1]:
- Improve currently used Zr-based alloys (e.g., deposition of protective coatings)
- Replace Zr-based alloys with alternative advanced materials

The CRP participants focused mainly on the improvement of traditional Zr-based materials using thin coatings on the outer surface, namely Cr-based coatings on commercial Zr-based alloys such as Optimized ZIRLOTM (Opt.ZIRLO), Zircaloy-4, and Zr-1%Nb. In addition, other innovative materials such as FeCrAl alloys and SiC/YAG composites were fabricated and provided for testing. Reference uncoated Zr-based materials were provided for parallel testing to provide a baseline for comparison with ATF materials when available. An overview of ATF materials tested is presented in Table 1.
The ATF and reference cladding materials were generously provided by the participants for testing. Fabrication processes and their optimizations were performed internally by material providers and were outside the scope of the CRP. It should also be noted that materials were provided in different geometries (plates and tubes; variations in outer diameter and length, etc.) and quantities. Some institutes provided only 1-2 cm long segments, others were able to provide tubes up to 1.5 meter long. In addition to the coated Zr-based alloys, two types of FeCrAl cladding were also tested. The first was provided by KIT and was a reference material for the QUENCH-19 test, and the second was FeCrAl provided by NPIC with different composition.
TABLE 1. SUMMARY OF ATF CLADDING MATERIALS TESTED WITHIN THE ATF-TS CRP AND THEIR PROVIDERS
Institute Country Coated Zr-based Cladding Alternative materials Notes
Czech Technical University in Prague Czech Republic Cr and CrN-Cr coated Opt. ZIRLOTM Tubes, large quantities, up to 1.5 meter long
Karlsruhe Institute of Technology Germany Cr-coated ZIRLO, M5 and Zircaloy-4 FeCrAl B136Y3 –13Cr-6.2Al-0.03Y (wt.%) Tubes, large quantities, up to 0.5 meter long
Institute of Nuclear Chemistry and Technology Poland SiC/YAG coated Zr1Nb Plates, optimization of deposition process not completed
Canadian Nuclear Laboratories Canada TiAl-coated Zircaloy-4 CANDU reactors as a focus, tubes, large quantities
Nuclear Power Institute of China China Cr-coated Zircaloy-4 FeCrAl 13Cr-4.5Al-2Mo-1Nb-0.4Ta-0.05Y (wt%)
China Nuclear Power Engineering China Cr-coated Zircaloy-4
Belarusian State University Belarus Cr-coated and plasma-treated E110 Tubes, small quantities, optimization of fabrication process not completed
Atomic Energy Organization of Iran Iran Cr and CrN/Cr coated Zr1Nb Tubes, large quantities, coatings optimized during the project
KEPCO Nuclear Fuels Korea Cr coated HANA-6 Tubes, large quantities, coatings optimized

CHROMIUM COATED ZIRCONIUM-BASED ALLOY
Pure Cr coatings with the highest level of technology readiness were the most popular among materials

providers. Six institutes provided coated Zr claddings with pure Cr coatings –CTU, KIT, CNPE, NPIC, AEOI, and KNF. The coating thickness and deposition parameters vary significantly, resulting in different microstructures and performance of the tested coatings. In addition, different Zr-substrates (commercial Zr-based cladding materials) were used by different institutes.

The Czech Technical University in Prague (CTU) developed three types of chromium coatings for Opt. ZIRLO™ and Zircaloy-4 cladding materials using the Hauzer Flexicoat 850 and 1500 machines. These coatings include a thick Cr coating (17-20 μm), a thin Cr coating (~7 μm) and a combination of CrN (approximately 6 μm) and an outer Cr coating (~12 μm). The deposition process involves cleaning, plasma cleaning, and physical vapor deposition (PVD) with specific parameters to ensure high-quality coatings. The resulting microstructure of these coatings is shown in Figure 1 [2].

Karlsruhe Institute of Technology (KIT) applied Cr coatings to Zircaloy-4, M5 and ZIRLO cladding tubes using magnetron sputtering. The process included ion etching with argon ions and coating at a maximum temperature of 270℃. Two coating thicknesses were used: 7.5 μm and 10.5 μm. Quality control was performed using destructive testing and adhesion tests according to German standards.

China Nuclear Power Engineering Co., Ltd. (CNPE) provided two types of Cr-coated cladding: a Zr-Sn-Nb alloy coated with magnetron sputtering and a Zry-4 alloy coated with arc ion plating. The coating microstructure analyses showed smooth surfaces and uniform chromium distribution for the magnetron sputtered samples, whereas the arc ion plated samples showed small droplets and thinner coatings.

The China Nuclear Power Institute (NPIC) used multi-arc ion plating to coat Zircaloy-4 cladding tubes. The process involved mechanical polishing, ultrasonic cleaning, and deposition with specific parameters to reduce droplet formation and improve the density of the coating. The resulting coatings exhibited fiber or columnar crystal morphology.

The Atomic Energy Organization of Iran (AEOI) prepared several batches of Cr-coated Zr-1%Nb samples using cathodic arc evaporation. The process included ultrasonic cleaning, argon ion sputtering, and deposition with specific parameters. The coatings showed good adhesion and uniform thickness, with a clear boundary between the Cr coating and the substrate. The deposition chamber used for coating preparation and resulting coating microstructure are shown in Figure 2.

And KEPCO Nuclear Fuels (KNF) used Arc Ion Plating (AIP) to coat the HANA-6 cladding tubes. The process involved plasma etching, Cr ion deposition, and post-coating inspections to ensure quality. The coated cladding materials were provided to various laboratories for detailed testing.

FIG. 1. Cr coatings produced by CTU on Opt. ZIRLO –SEM/EBSD analysis of the microstructure

(A)

(B) (C)

FIG. 2. (A) Arc-PVD chamber used by AEOI to deposit Cr coatings; (B) SEM-BSE image of the as-deposited Cr coating; (C) Fracture surface of Cr-coated Zr-1%Nb cladding tube

ZIRCONIUM-BASED ALLOYS WITH ALTERNATIVE COATINGS

Pure Cr is the most advanced solution for coated Zr-based claddings, but there are still several drawbacks such as Cr-Zr eutectic reaction at about 1330 ° C, Cr-enhanced embrittlement of Zr, low ductility of Cr, etc. Therefore, alternative coating designs and materials have been proposed and are being optimized. Four alternative coated cladding materials were fabricated within ATF-TS by CTU, AEOI, CNL, and INCT. The alternative coatings are CrN/Cr, TiAl, and SiC/YAG.

CTU developed CrN/Cr coated Opt. ZIRLO to enhance the corrosion resistance and reduce the interdiffusion of Zr-Cr. The CrN interlayer was chosen on the basis of its ability to reduce the kinetics of eutectic formation and improve high-temperature steam oxidation resistance. The deposition process involved reactive magnetron sputtering for the CrN layer, followed by a Cr top layer. The resulting microstructure showed a submicrometer Cr layer to improve adhesion, with a grain size and texture similar to that of pure Cr coatings.

AEOI prepared Zr-1%Nb tubular samples with CrN and multilayer Cr/CrN coatings. The coating process involved physical vapor deposition, with pre-treatment including ultrasonic cleaning and argon ion sputtering. The coatings were characterized by SEM and GI-XRD, revealing the presence of Cr, CrN, and Cr2N phases. The Cr2N phase formed due to nitrogen gas flow manipulation during the coating process. The coating parameters for both multilayer Cr/CrN and single layer CrN are shown in Table 2.

TAB.2. COATING PARAMETERS USED FOR CRN AND MULTILAYER COATING BY AEOI

Parameter	Coating thickness (μm)	Number of targets	Bias voltage (V)	Substrate temperature (℃)	Ar gas pressure (sccm)	Periodic injection of Nitrogen gas (min)	Coating duration (h)
CrN Coating	7–10	2	100	300 ± 2	15	~55.0*	~7.0
multilayer Coating	7–10	2	100	300 ± 2	15	~4.0	~8.5

* To control shedding and decrease residual stress of CrN coating

CNL provided Ti/Al coated Zircaloy-4 tubes with different outer diameters relevant to CANDU reactors for high-temperature oxidation and burst tests. The coating was applied by magnetron sputtering deposition, with a target thickness of 10 μm and a composition of 45-50 at.% Ti to 55-50 at.% Al. The resulting coatings showed good adhesion and uniform thickness, with some variations due to the deposition rate. The resulting microstructure observed by SEM and XRD pattern are shown in Figure 3.

(A)
(B)
FIG. 3. (A) TiAl cross-section with gaps between coating and the substrate for the 13.08 mm OD Tube due to Surface Roughness; (B) XRD Pattern from a 5.3 mm OD Zr-4 Tube, Coated with Ti/Al.

The Institute of Nuclear Chemistry and Technology (INCT) developed SiC/YAG composite coatings for Zr-1%Nb substrates using suspension plasma spraying (SPS). The coatings aimed to improve corrosion resistance and high-temperature stability. The SiC grains were coated with YAG using a sol-gel method, and the resulting powders were sprayed onto the substrates. The coatings were characterized by SEM and EDS, revealing a fine-grained structure with good adhesion. However, the initial deposits showed some delamination, indicating the need for further optimization. The SEM observations of the outer surface and cross section are shown in Figure 4.
(a) (b) (c) (d)
FIG. 4. Plasma sprayed coatings on Zr-1%Nb plate, spray distance 80 mm, SEM (SE) image, a) argon, b) argon-hydrogen and cross-section of the coatings sprayed by argon-hydrogen plasma SEM (BSE) image with magnification: c) 1 000, d) 4 000

FECRAL
KIT provided nuclear-grade FeCrAl alloys, specifically B136Y3 (Figure 5), originally provided by Oak Ridge National Laboratory (ORNL) and previously used for the QUENCH-19 bundle test [3]. These alloys were fabricated in tubular shapes with an outer diameter of 9.52 mm and a wall thickness of 381 μm. NPIC on the other hand developed a FeCrAl alloy with a composition of 13Cr-4.5Al-2Mo-1Nb-0.4Ta-0.05Y (wt%). This alloy was designed to improve the adhesion of the oxide protective layer and strengthen the Fe-bcc matrix. The alloy underwent homogenization, solution treatment, hot extrusion, and cold pilger rolling to achieve the final cladding tube dimensions of 9.5 mm outer diameter and 0.38 mm wall thickness. The processing flow included vacuum induction melting, homogenization, red feeding forging, extrusion, warm rolling, cold rolling, and recrystallization annealing.
Nuclear-grade FeCrAl alloys B136Y3 were provided by ORNL in tubular shapes. The tubes had an outer diameter (OD) of 9.52 ±0.04 mm; the wall thickness was 381 ±9 μm. Fig. 2.24 shows an optical micrograph after etching with a solution of glycerin of 45 ml, HNO3 of 15 ml and HCl of 30 ml. The grains are fairly uniformly distributed with a grain size of 80 ~ 130 μm. The chemical composition (Febal Cr13 Al6.2 Y0.03 C0.01) was provided by ORNL.

FIG. 5. Microstructure of the FeCrAl B136Y received

NPIC provided FeCrAl with chemical composition - 13Cr-4.5Al-2Mo-1Nb-0.4Ta-0.05Y (wt%). The addition of Y, Mo, Nb, and Ta aims to improve the adhesion of the oxide protective layer and strengthen the Fe-bcc matrix. The FeCrAl alloy was subjected to homogenization and solution treatment, followed by hot extrusion and cold pilger rolling with a thickness reduction of about 40% for four times to obtain the cladding tube. The FeCrAl ingot billet prepared by HIP (hot isostatic pressed) was then subjected to homogenization and solution treatment. Cold pilgered cladding tube was obtained by hot extrusion and pilger rolling with a thickness reduction of ~40% for four times and followed by annealing at 800℃ for 30 min to reduce stress and defects and prevent premature cracking during rolling. The final geometry of the cold pilgered thin-wall tube are 9.5 mm in outer diameter and 0.38 mm in wall thickness.
On the basis of high-temperature compression experiments on FeCrAl alloy, the influence of deformation parameters (deformation temperature, strain rate) on high-temperature deformation behaviours was studied. The Arrhenius type model was used to establish the constitutive equation and thermal processing diagram of the FeCrAl alloy, and the influence of thermal deformation parameters on the effect of the material microstructure. By studying the recrystallization behavior, texture evolution, Laves phase precipitation, and its interaction with grain boundaries during the processing and heat treatment of FeCrAl alloy cladding pipes, the quality of FeCrAl pipe processing and microstructure was well controlled. In order to improve the service performance of the pipe, the introduction of kink strips effectively improved the strengthening and toughening effect of the alloy, the red forging process was used to optimize the hot processing technology of the tube blank, and the warm and cold rolling process was used to optimize the pipe rolling technology, successfully overcoming the problem of containing Laves phases. In order to solve the problems of high hardness and difficult processing of FeCrAl alloy, a method combining a warm/cold deformation process is used to achieve fine control of the structure and properties of FeCrAl cladding material and realize batch production processing of full-size defect-free FeCrAl alloy thin-walled cladding tubes.
The processing flow is as follows:
Vacuum induction melting ⟶ homogenization ⟶ red feeding forging ⟶ extrusion ⟶ warm rolling (three passes) ⟶ cold rolling (seven passes) ⟶ recrystallization annealing ⟶ final product.
(a) (b)
FIG. 6. Microstructure of FeCrAl (a) after pilger rolling and (b) after annealing
SUMMARY

This paper provides a detailed description of the ATF fuel cladding materials manufactured and tested within the ATF-TS CRP. The primary materials include chromium-coated zirconium-based alloys, chromium-nitrogen-/chromium-coated zirconium-based alloys, and FeCrAl alloys.

Chromium coatings were applied to zirconium-based alloys mainly to improve their oxidation resistance and mechanical properties. Various deposition techniques, such as magnetron sputtering and arc ion plating, are used to create thick and thin Cr coatings, as well as CrN/Cr multilayer coatings. These coatings are characterized by their uniform thickness, good adhesion, and enhanced high-temperature performance.

CrN/Cr coatings are developed to reduce the interdiffusion of Zr-Cr and improve corrosion resistance. The deposition process involves reactive magnetron sputtering for the CrN layer, followed by a Cr top layer. These coatings exhibit a fine-grained structure and good adhesion, making them suitable for high-temperature steam oxidation testing.

FeCrAl alloys were designed to enhance the high-temperature oxidation resistance and mechanical properties of the nuclear fuel cladding. These alloys undergo processes such as homogenization, hot extrusion, and cold pile rolling to achieve the desired microstructure and mechanical properties. The addition of elements such as Y, Mo, Nb, and Ta improves the adhesion of the oxide protective layer and strengthens the Fe-bcc matrix.

Based on the experience of ATF-TS fabrication, the following recommendations can be made:

- Optimization of Coating Processes: More research should focus on optimizing the deposition parameters for chromium and CrN/Cr coatings to enhance their performance under reactor conditions. This includes refining the thickness, composition, and microstructure of the coatings to achieve better adhesion and corrosion resistance.
- Development of New Materials: Exploring new materials and composite coatings can provide additional benefits in terms of corrosion resistance and high-temperature stability. The integration of these materials into existing cladding technologies should be investigated to improve overall performance.
- Testing and Validation: Extensive testing and validation of the coated cladding materials under simulated reactor conditions are essential. This includes high-temperature oxidation tests, mechanical property evaluations, and long-term durability assessments to ensure coatings can withstand the harsh environments of nuclear reactors.
- Collaboration and Knowledge Sharing: Continued collaboration between research institutions, universities, and industry partners is crucial to advance the development of advanced coatings for nuclear fuel cladding. The sharing of knowledge and best practices can accelerate the adoption of these technologies and improve their effectiveness.

By addressing these recommendations and focusing on future research directions, the development of advanced coatings for the cladding of nuclear fuel can significantly enhance the safety and efficiency of nuclear reactors, contributing to the sustainable and reliable generation of nuclear energy.

ACKNOWLEDGEMENTS

The activities presented in the paper were conducted within the framework of the IAEA ATF-TS project, and we would like to acknowledge the contribution of many coworkers, students, and advisors who help to fabricate, optimize, and share the ATF and reference cladding materials.

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Session Classification: 1

Track Classification: Advances in ATF Materials and Manufacturing