

# Biaxial creep test of DIN 1.4790 cladding material

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| <p><b>Summary</b></p> <p>The objective of the SAFER2028 MATFINE 2023 project Task 2.1 was to investigate the adhesion and consistency of zircaloy-based Cr-Al coated cladding material on the substrate, coupled with assessing the material's resistance to thermal creep through multiaxial stress testing. Planned activities included elevated temperature testing with internal pressure, monitored by axial and hoop strain measurements, followed by microscopy examination of coating performance.</p> <p>However, due to VTT's policy, the planned materials were excluded, shifting the focus to training new personnel on VTT's complex biaxial thermal creep equipment. The primary educational objective became instructing an individual in setting up tests, operating the equipment, and analyzing generated data. The chosen fuel cladding material was DIN 1.4790, with dedicated test samples tailored for VTT's biaxial creep test equipment.</p> <p>The testing equipment utilized the Pneumatic Loading Apparatus (PLA) concept, featuring bellows for axial load generation and flexibility in axial – hoop stress ratios. Thermal creep testing required a specialized high temperature furnace, and a strain measurement system monitoring axial and hoop strains.</p> <p>The experiment initiated at 450°C and 400 bar internal pressure, incrementally increasing to accelerate the creep rate. Unfortunately, at the 465-hour mark, a compressor valve failure led to the termination of the test. Despite this, the primary goal of training a new person for creep testing was successfully initiated. It is essential to emphasize that the VTT biaxial creep testing equipment, along with its subsystems, is intricate and demands additional training for the individual to achieve proficiency and to independently operate the system.</p> |  |
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## Preface

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## 1. Objective

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The original objective of the planned investigations for MATFINE 2023 project Task 2.1 was to examine the adhesion and consistency of the coating on the substrate material, utilizing zircaloy-based Cr-Al coated cladding material. Simultaneously, the material's resistance to thermal creep was expected to be assessed through thermal creep testing under multiaxial stress conditions. Anticipated activities included testing at elevated temperatures with internal pressure, accompanied by axial and hoop strain monitoring. Subsequent examination of the coating's performance was to be conducted through microscopy (LOM, SEM) in specific regions of the tested samples. The results obtained were expected to contribute valuable insights for modeling the mechanical degradation of advanced fuel cladding materials.

Nevertheless, the available Zry-based and Cr/Cr-Al coated materials available at VTT were excluded from these tests in adherence to VTT's policy [1]. Consequently, the focus of the task was redirected towards training a new person in the operation of VTT's biaxial thermal creep equipment. This specialized equipment [2], designed and constructed at VTT, presents a significant learning curve for novices. Therefore, the primary objective was to instruct new individual on manually arranging the test setup, operating the equipment's control systems, and analyzing the raw data generated by the system. The optimal fuel cladding material chosen for this application was the DIN 1.4790 type. Test samples of this cladding material, specifically designed for VTT's biaxial creep test equipment, have already been produced and demonstrated their reliability [3].

## 2. Materials and methods

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The subsequent section provides an overview of the characteristics and attributes of both the testing equipment employed and the material subjected to testing.

### 2.1 Testing equipment

Traditional mechanical material testing methods utilize a mobile pull rod to exert an axial load on the specimen. However, when testing in liquid or pressurized environments, the presence of pressure boundary feed-throughs becomes a drawback due to issues related to leakage and challenging control of friction forces. In the Pneumatic Loading Apparatus (PLA) testing concept developed by VTT, axial load is generated using pneumatic loading units called bellows [2]. Unlike facilities relying on moving elements over the pressure boundary, the bellows-based system only connects to the control unit through pressure lines and electrical feedback connectors. This enables the construction of test setups that are devoid of moving elements beyond the pressure boundary. Consequently, even under demanding conditions, such as delicate in-reactor testing for nuclear material, the system proves effective in testing small-sized specimens.

The PLA concept has demonstrated success in various material testing applications, including slow strain rate testing (SSRT), stress corrosion cracking (SCC), and creep-fatigue testing (CF), in both challenging simulated and real-world service environments like supercritical water (SCW), irradiative settings, hydrogen, and high-temperature corrosive pressurized gas environments [3-5]. Simultaneously, most experimental setups created for biaxial creep testing of tubes rely solely on internal pressure to exert axial and hoop stress on the specimen. In these applications, the axial-hoop stress ratio remains fixed and is not adjustable within the testing facility. Pneumatic loading units, however, offer the flexibility of providing axial load to the tubular test specimen in either the push or pull direction, allowing for testing with various axial – hoop stress ratios. The PLA concept similarly employs internal pressure to induce axial and hoop stress on the test specimen. The additional axial load system based on bellows technology has been successfully employed in various applications in the past.

The additional axial load, either in push or pull configuration, can be applied to the test specimen by exerting internal pressure of up to 200 bar on the push or pull bellows. This pressure can generate an axial force of up to 4 kN on a thin-walled tubular specimen. The internal pressure in the bellows is regulated by pneumatic servo-controlled pressure adjusting loops. These Programmable Logic Control (PLC) loops can operate with air, helium, or nitrogen gases depending on the testing environment. The internal pressure of the tubular test specimen is also regulated by the high-pressure control loop, which is powered by argon gas, with a maximum pressure level of 700 bar. To ensure and optimize the structure and functionality of the loading frame from the design stage, a comprehensive 3D model of the biaxial creep testing equipment has been created using ironCAD™ software. Figure 1 illustrates the loading frame of the biaxial creep testing device. Two distinct pneumatic loading units, or bellows, are mounted on the loading frame, serving as the supporting component for the test specimen. The axial load for the tubular test specimen is generated by pneumatic loading equipment in either a push or pull motion. Nuts are employed to secure the tubular specimen to the loading frame, with the lower fixing point being movable and the upper end being fixed.

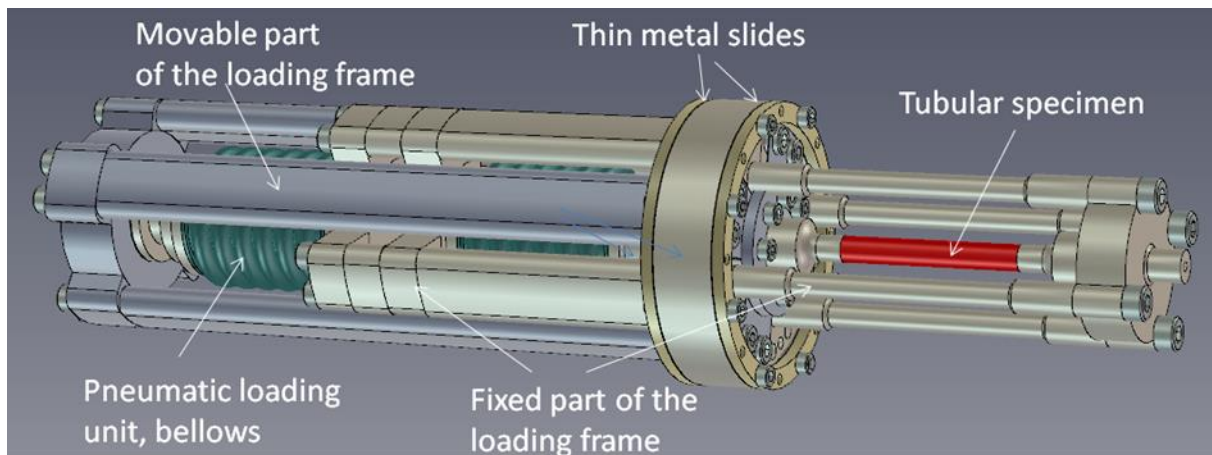


Figure 1. The biaxial testing device with the main parts

For testing thermal creep properties with the described biaxial testing equipment, a dedicated furnace designed for high temperature testing is required. The existing configuration allows testing exclusively in air atmosphere. The schematic representation of the furnace's structure is depicted in Figure 2a. The furnace temperature is adjustable through three distinct zones utilizing PID controllers.

To monitor changes in sample dimensions, the equipment utilizes a strain measurement system based on the mechanical extensometer type. The axial and hoop strains are measured at a gage length of 25 mm and the middle of the tubular specimen, respectively. Figure 2b illustrates the schematic test setup for the strain measurement system.

Concerning the high-pressure gas supply system, both pressure adjustment systems, namely for the bellows and the tubular specimen, are controlled by the PLC. A schematic illustration of the pressure adjustment systems is presented in Figure 3. The bellows receive pressurization from air, while the tubular specimen is pressurized using Argon. The maximum controlled pressure for the tubular specimen is set at 690 bar (with the compressor having a maximum pressure of 1500 bars).

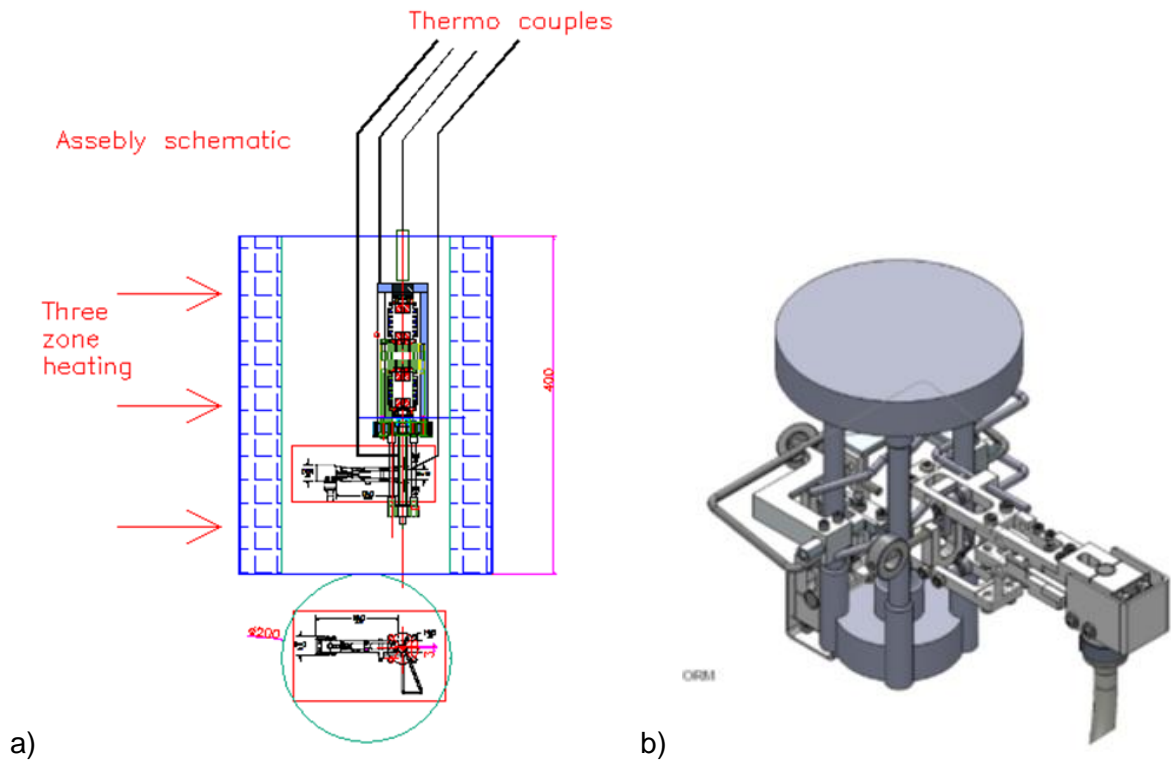
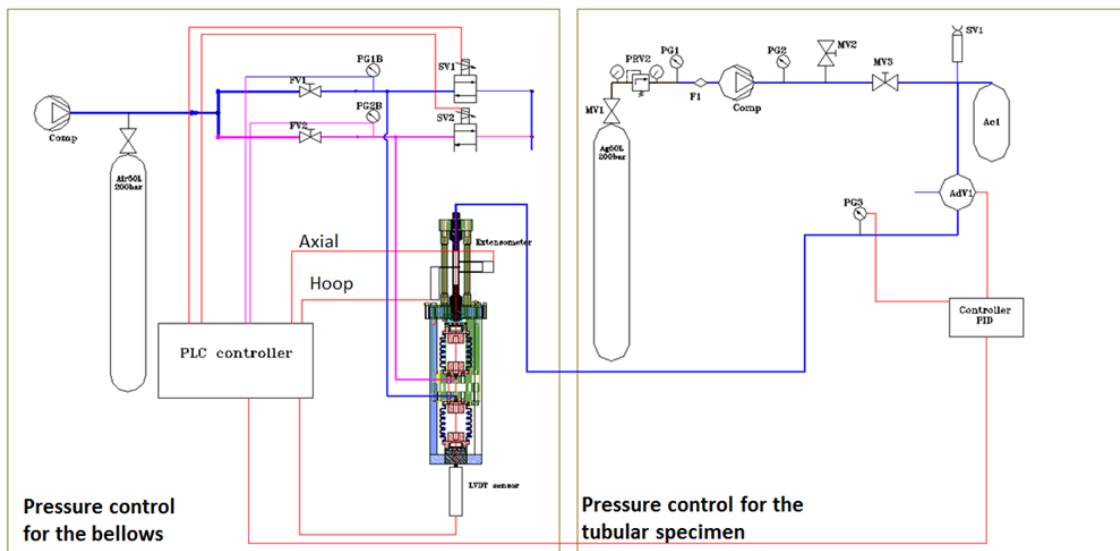


Figure 2. The biaxial testing device with the extensometers placed into furnace in a) and location of axial and transverse extensometers in the test setup in b)



Comp = Air compressor  
 FV1 = flowing valve 1  
 FV2 = flowing valve 2  
 PG1B = bellows 1 pressure  
 PG2B = bellows 2 pressure  
 SV1 = servo valve 1  
 SV2 = servo valve 2

PRV2 = reducing valve  
 PG1 = Pressure gauge  
 F1 = Filter  
 Comp = Ar compressor  
 PG2 = pressure gauge  
 MV2 = vent valve  
 SV1 = stop valve  
 Ac1 = accumulator  
 Adv1 = pressure adjuster  
 PG3 = high pressure gauge

Figure 3. The high-pressure gas supply system



## 2.2 Tested material and sample

The tubular specimen subjected to creep testing was fabricated from titanium-stabilized austenitic stainless steel of DIN 1.4970 type, featuring a 24% degree of cold work. The tubular section of the sample had an outer diameter of 6.55 mm and an inner diameter of 5.65 mm, with a nominal wall thickness of 0.45 mm. The chemical composition of the tested material is detailed in Table 1. It should be noted that the selection of this cladding material is primarily intended for future GEN IV reactors. However, it was chosen for this specific test due to the availability of pre-manufactured biaxial creep test samples exclusively in this material grade. Consequently, the educational objectives of the task could be effectively met using this specific test sample.

*Table 1. The chemical composition of the tested DIN 1.4970 material*

| Element           | C     | Ni    | Cr    | Mo   | Ti   | Si   | Mn   | Nb | Sn | Zr | Fe   |
|-------------------|-------|-------|-------|------|------|------|------|----|----|----|------|
| DIN 1.4970 (wt.%) | 0.096 | 15.05 | 15.06 | 1.21 | 0.44 | 0.57 | 1.86 | -  | -  | -  | Bal. |

Figure 4 illustrates the primary geometry and dimensions of the tubular biaxial creep specimen. The specimen's overall length is 123.6 mm, with a fixed length of 85 mm and an actual cladding tube part measuring 45 mm. The cladding tube section is securely inserted into the end plugs through mechanical means, and the entire specimen is affixed to the loading frame using M8 nuts at both ends.



*Figure 4. The mechanically plugged biaxial creep testing specimen*

## 3. Results

The experimental design involved initiating the test at 450°C under an internal pressure of 400 bar, resulting in 251 MPa hoop stress, 116 MPa axial stress, and 234 MPa von Mises stress. The initial expectation was that, under these conditions, the specimen would not manifest substantial creep. Therefore, the aim was to incrementally elevate both pressure and temperature. This approach served a dual purpose: to expedite the creep rate and, concurrently, to enhance the educational aspects of the experiment.

Before initiating the actual test, the temperature was gradually increased to 450°C a day in advance. This intentional preheating was performed to ensure full temperature saturation, minimizing strain extensometer drifting. The test commenced with a systematic rise in internal pressure to 400 bar at a rate of 0.6 bar/s. Consequently, this process induced approximately 0.1% initial hoop strain and around 0.05% initial axial strain. After 405 hours, the internal pressure was raised to 450 bar, resulting in 283 MPa hoop stress, 131 MPa axial stress, and 263 MPa von Mises stress, aimed at accelerating the creep rate. Unfortunately, at the 465-

hour mark, an essential valve in the compressor broke, leading to the termination of the test. Plans are in place to resume the test after repairing the compressor system.

Figure 5a illustrates the temperature (measured with two independent thermocouples) and the test sample's internal pressure over time, while Figure 5b displays the hoop and axial strain as a function of time. It's important to note that data cleaning resulted in the removal of approximately 0.024% fluctuation in hoop strain and 0.012% in axial strain from the dataset.

The individual undergoing training on the VTT biaxial creep testing equipment engaged in system control maneuvers and data processing. It is essential to emphasize that the VTT biaxial creep testing equipment, along with its subsystems, is intricate and demands additional training for the individual to achieve proficiency and to independently operate the system.

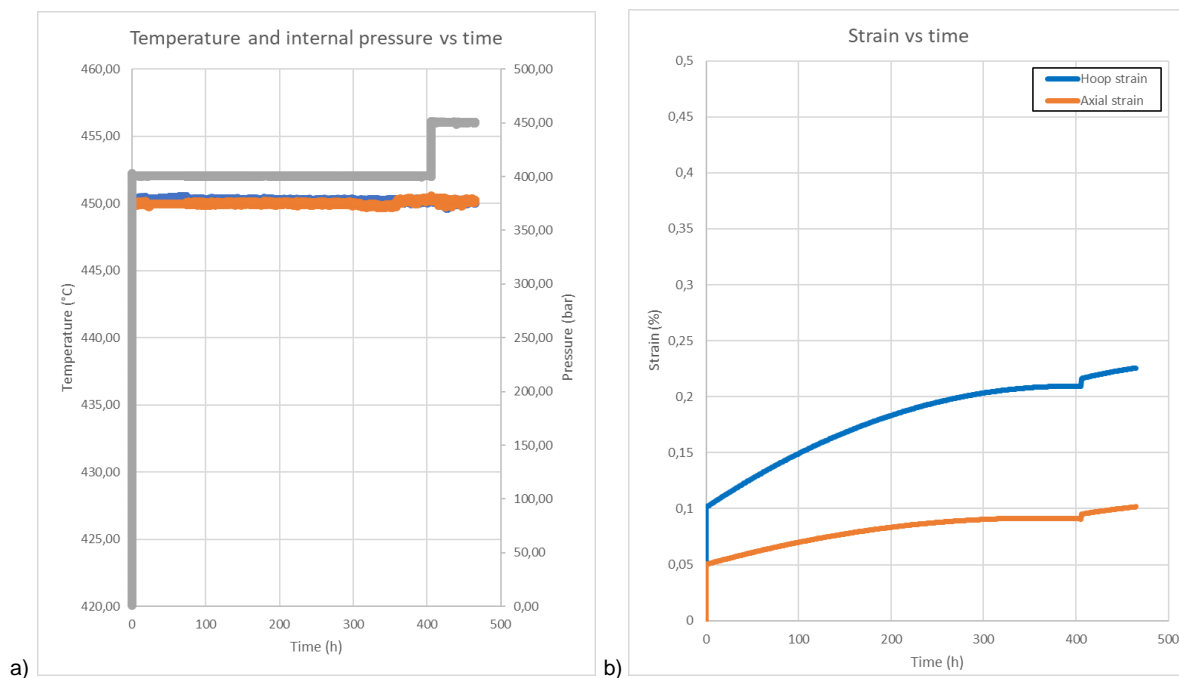


Figure 5. The temperature (measured with two thermocouples independent of each other) and test sample internal pressure vs time in a), and hoop and axial strain vs time in b)

## 4. Summary

The objective of the SAFER2028 MATFINE 2023 project Task 2.1 was to investigate the adhesion and consistency of zircaloy-based Cr-Al coated cladding material on the substrate, coupled with assessing the material's resistance to thermal creep through multiaxial stress testing. Planned activities included elevated temperature testing with internal pressure, monitored by axial and hoop strain measurements, followed by microscopy examination of coating performance.

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