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Experimental database of E110 claddings exposed to accident conditions

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ABSTRACT

An experimental database of E110 alloy has been developed on the basis of about 600 separate and combined effect tests of the Hungarian Academy of Sciences KFKI Atomic Energy Research Institute. It contains the data of oxidation, ballooning, tensile and compression tests, the results of post-test investigations, photos, figures, information concerning the test conditions and the corresponding English-language publications. The aim of this database is to give adequate information on the E110 cladding behaviour (oxidation, hydrogen uptake, mechanical performance) under accident conditions and to provide valuable experimental data for model development and code validation. This database is a part of the International Fuel Performance Experimental Database. It is accessible on-line, via the internet. This paper gives an overview of the experiments, the test facilities and conditions involved in the database. It presents the most important results and consequences and introduces the directory structure of the database.

1. Introduction

Zirconium-based alloys are widely used for nuclear reactor components such as fuel claddings, grid spacers and guide tubes. These alloys combine low neutron absorption behaviour and good mechanical and corrosion properties under operating conditions. However, rapid oxidation of the fuel cladding occurs under loss-of-coolant accident (LOCA) conditions due to the zirconium–steam reaction. The oxidation is accompanied by intensive hydrogen production. The oxidation causes the mechanical deterioration and the embrittlement of the cladding. The formed hydrogen is partly absorbed by the Zr alloy and it contributes to the cladding brittleness. In an accident situation a hydrogen-rich steam atmosphere can evolve, as well. Increased hydrogen absorption of zirconium claddings in this atmosphere may intensify the embrittlement of the material and can lead to the failure of the fuel rods under thermal and mechanical loads.

In the past decade several experimental series have been performed at the Hungarian Academy of Sciences KFKI Atomic Energy Research Institute (AEKI) with E110 (Zr, 1.0 wt.% Nb, 0.05 wt.% O, 0.01 wt.% Fe, 0.01 wt.% Hf) and Zircaloy-4 (Zr, 1.5 wt.% Sn, 0.2 wt.% Fe, 0.1 wt.% Cr, 0.1 wt.% O) claddings [1]. The aims of these experiments were to study and to compare the mechanical properties of the cladding materials and to investigate the effect of pure steam oxidation and hydrogen uptake on the mechanical performance of the claddings. The objectives have been achieved through separate effect tests with well defined conditions. Some years ago a new experimental programme started at the AEKI in order to investigate the combined effects of steam and hydrogen-contents on the mechanical properties of the E110 type cladding that is used in VVER reactors. These tests have been carried out in hydrogen-rich steam atmosphere.

The experimental data of oxidation and mechanical tests, the results of post-test investigations, figures, photos and information concerning the test conditions have been compiled in an electronic database [2]. A former version of this database that included experimental data from tests carried out in high temperature steam, was prepared at the AEKI in the EXTRA project of the EURATOM 5th Framework Programme of the European Commission [3,4]. The extension of the database was carried out in a common project with the JRC Institute of Transuranium Elements in a new test series focusing on the role of the hydrogen in the oxidizing atmosphere [5]. The latest version of the database can be found on the web site: www.nea.fr/abs/html/nea-1799.html. The database in its present form contains more than 240 oxidation tests, 170 cladding ballooning tests, 110 tensile tests and 110 ring compression tests. The collected experimental data make possible the development of new models for the simulation of E110 cladding under accident conditions. This paper gives an overview of the structure and the content of the database.

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2. Oxidation

More than 240 oxidation tests were performed at the AEKI. A high temperature tube furnace was used for the oxidation of the samples. The experimental set-up consisted of a steam generator, a three-zone furnace with temperature control system and a condensing system (Fig. 1). The outlet gas flow rate was measured by a calibrated Soap Bubble Gas Flow Meter. The steam flow was evaluated through the measured weight of the condensed water. When the temperature of the furnace and the gas flow was stable, the sample was pushed to the hot zone of the furnace. At the end of oxidation the sample was withdrawn to the cold part of the equipment. The equivalent cladding reacted (ECR) and the ZrO_2 thickness were calculated on the basis of the measured mass gain. The oxidation rate constant was also calculated for the specimens.

2.1. Test series in pure steam atmosphere

Data of seven different series of pure steam oxidation experiments were involved in the database. All the seven series aimed at the investigation of the high temperature oxidation of zirconium cladding in steam atmosphere. Some of the experiments were initiated to provide pre-oxidized samples for mechanical tests. The oxidation of the tube specimens was performed in a steam–argon mixture including 12 vol.% argon gas. The kinetics of the oxidation was studied at constant steam flow in the temperature range 500–1200 °C.

The performed experiments proved that the mass gain due to the oxidation is proportional to the square root of the oxidation time. The mass gain for unit surface area can be described as follows:

$$\frac{\Delta m}{S_A} = k \cdot t^{1/2} = A \cdot \exp\left(\frac{-Q}{RT}\right) \cdot t^{1/2} \tag{1}$$

where Δm is the mass gain (mg), S_A is the surface area (cm^2), A is the pre-exponential factor, Q is the activation energy (J), R is the gas constant = 8.314 J/(mol K), T is the temperature (K), t is the time of steam oxidation (s), k is the reaction rate constant ($\text{mg/cm}^2/\text{s}^{1/2}$).

On the basis of the test data a best estimate correlation for the oxidation rate of E110 alloy was developed [6]

$$k = 658 \cdot \exp\left(\frac{-10200}{T}\right) \tag{2}$$

The reaction rate was derived on the basis of the mass gain measurements in 122 isothermal oxidation tests. Fig. 2 represents the individual mass gain rates measured in the tests and the best fit curve as a function of the reciprocal temperature.

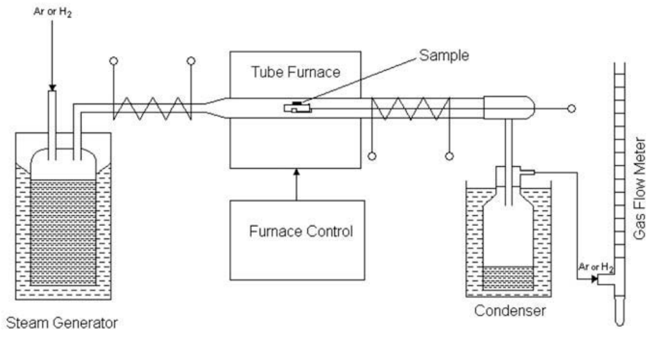


Fig. 1. Scheme of the experimental set-up for oxidation tests.

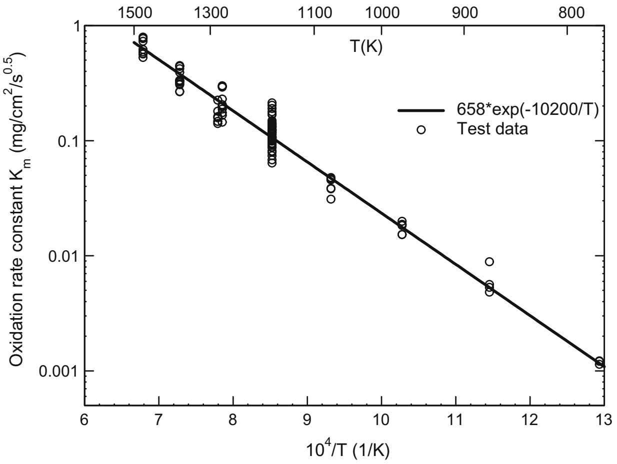


Fig. 2. Oxygen mass gain rate constant of E110 alloy as a function of the reciprocal temperature. AEKI test data and best fit line.

2.2. Test series in hydrogen-rich steam atmosphere

Four different series of hydrogen-rich steam oxidation experiments were performed in order to study the effect of the presence of hydrogen in the steam environment on the oxidation kinetics, and to provide pre-oxidized samples for ring compression, tensile and ballooning tests.

Earlier experimental studies indicated that the hydrogen content in the steam atmosphere can influence the oxidation kinetics of the zirconium alloys, but the reported results are contradictory. Chung and Thomas [7] investigated the oxidation kinetics of Zircaloy-4 claddings at a hydrogen fraction above 0.5 in the temperature range of 1200–1400 °C and observed a significant retardation of oxidation rates by the hydrogen at both small and large steam supply rates. Kim et al. [8] studied the oxidation rate of sponge-Zr in a steam–hydrogen mixture at high pressure (70 atm) in the temperature range of 350–400 °C. They observed a decrease of the oxidation rate only at very high hydrogen content close to steam-starved conditions. However, in other papers completely different results were published. Moalem and Olander [9] studied the weight gain of Zircaloy specimens at 1200 °C varying the hydrogen content in the steam between 0.5 and 0.91 mol fractions. The authors concluded that the oxidation rate is enhanced by the thermal effect of the rapid hydrogen uptake at the beginning of the oxidation.

Original VVER cladding specimens were oxidized at the AEKI in a controlled, mixed steam–hydrogen atmosphere under isothermal conditions between 900 and 1100 °C. The hydrogen content in the steam was fixed between 20 and 36 vol.%, because the existing equipment allowed experiments within this range (1–3 test series). The oxidation rates constants measured in pure steam and in different steam–hydrogen mixtures are presented in Fig. 3. In 20 vol.% hydrogen–steam atmosphere the following relation was derived for the oxidation rate constants versus temperature:

$$k = 117 \cdot \exp\left(\frac{-8680}{T}\right) \tag{3}$$

The fourth oxidation series were performed at higher and lower hydrogen content (65 and 5 vol.%) in the steam by minor modifications to the equipment. It resulted in a reduced reaction rate constant. The best estimate correlation in a steam–hydrogen mixture including 65 vol.% hydrogen is the following:

$$k = 1964 \cdot \exp\left(\frac{-12171}{T}\right) \tag{4}$$

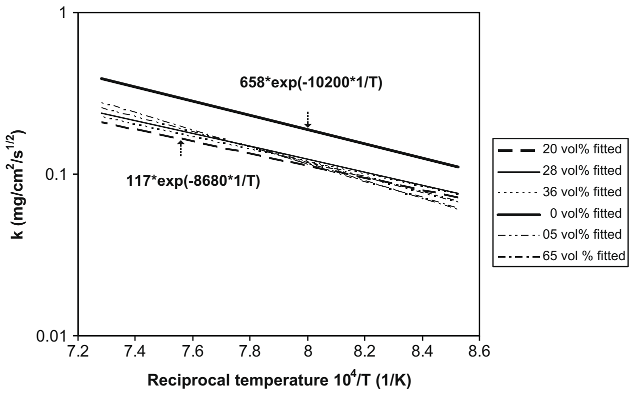


Fig. 3. Oxidation rate constants for E110 as a function of reciprocal temperature in steam–hydrogen mixture.

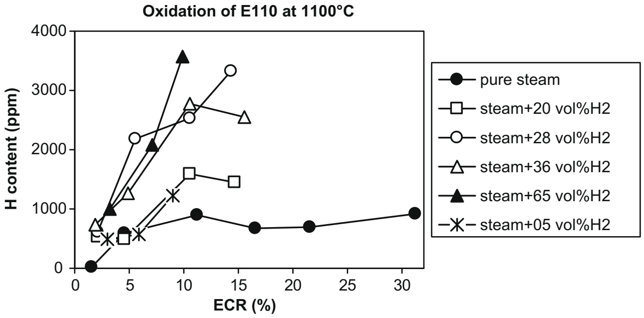


Fig. 4. Hydrogen content of E110 claddings oxidized in pure steam and in steam–hydrogen mixture at 1100 °C.

Comparing the oxidation rate constants measured in pure steam and in hydrogen-rich steam atmosphere, it can be concluded that the cladding oxidation decelerates if hydrogen is present in the steam atmosphere.

3. Hydrogen absorption

The hydrogen content of the oxidized cladding was measured by a hot extraction method after the mechanical tests. The amount of the extracted hydrogen was determined using a CHROMPACK MODEL 438A gas chromatograph. Comparing the hydrogen contents of the cladding specimens oxidized in pure steam and in hydrogen-rich steam, increased hydrogen absorption was observed in a steam–hydrogen mixture (Fig. 4). At 10% ECR four times more hydrogen was absorbed in the cladding oxidized in a steam–hydrogen mixture including 65 vol.% hydrogen than in pure steam.

4. Ballooning tests

The database comprises both single rod and rod bundle experiments. The aim of these tests was to evaluate the strength and deformation of the VVER fuel rods as well as the hazard of coolant flow blockage under LOCA conditions. The pressure and temperature histories and the residual deformations of more than 170 rods are available in the database.

4.1. 7-rod bundle tests

This test series was performed with short (150 mm) 7-rod bundles in order to investigate the possible flow blockage rate (ratio of flow cross-sections for ballooned and non-ballooned bundles) in a VVER core under LOCA conditions. The experimental facility contained a high temperature furnace with a temperature control system, a steam generator with a super heater, a pre-pressurization system to set the initial pressure of the tubes and a computerized measurement and data acquisition system.

Each test bundle contained seven E110 cladding tubes arranged in hexagonal geometry with the pitch of 12.2 mm characterizing the VVER-440 reactor core. The cladding tubes were connected to the pre-pressurization system mounted with check valves and pressure transducers. On the opposite end the tubes were closed with Zr2%Nb plugs. The test assembly was placed in an Al_2O_3 ceramic tube with a hexagonal inner hole. Nine experiments were performed at linear temperature increase. The initial pressure of the tubes varied between 3 and 30 bars. The temperature was detected in the three different axial positions. The individual pressure history of each tube was measured on-line. Tests in argon and steam atmosphere were compared to investigate the effect of cladding oxidation on flow blockage. The findings were as follows: lower initial pressure resulted in higher failure temperature and high initial pressure resulted in larger openings. Each tube of the test bundle burst at the same axial elevation (the axial elevation shows the vertical position). The oxidation due to steam atmosphere resulted in smaller deformation compared with argon atmosphere. The typical blockage rate was 40–50% and the maximum rate was below 80% [10].

4.2. Single rod tests in pure steam atmosphere

Single rod experiments were carried out to investigate the mechanical behaviour and strength of zirconium cladding tubes and to study the effect of corrosion on the mechanical strength of E110. Accordingly, some samples were treated in steam atmosphere at 900 °C before the ballooning tests. During the ballooning tests short tube samples were investigated in isothermal conditions in the temperature range of 650–1200 °C. The specimens closed with end-plugs were placed in a quartz test tube filled with inert gas (Ar), and heated in an electrical furnace. The pressure of the inert gas in the quartz tube was kept constant at 1 bar by means of a buffer volume. After a heat-up period the samples were pressurized with argon gas. Pressurization was performed through a pipe attached to one end of the specimen. The inner pressure of the test tube was increased linearly until the burst of the sample. The temperature and pressure history was monitored on-line by a computerized data acquisition system. The residual deformation of the samples was measured after the test.

The coolant side oxidation had a significant effect on the mechanical strength of the cladding. The strength of E110 increased up to 10 μm oxide layer thickness, but decreased with further oxidation (Fig. 5). Decreasing deformation with an increasing ZrO_2 layer thickness was also observed.

4.3. Single rod tests in hydrogen-rich steam atmosphere

The experiments aimed to provide information about the effect of hydrogen-rich steam oxidation and the influence of the hydrogen content on the cladding strength and deformation under simulated LOCA conditions. Some as-received tube samples were treated in hydrogen-rich atmosphere during the ballooning tests at temperatures of 900 and 1000 °C. Different hydrogen partial pressures could be achieved by using different hydrogen–argon flow rates. After the burst tests, the residual deformation of the specimens was measured. Subsequently, some small pieces of the cladding (at the burst and close to the burst) were used for determination of the absorbed hydrogen concentration. From the experiments the followings were found: the hydrided specimens burst at higher pressure than the as-received sample. The measured

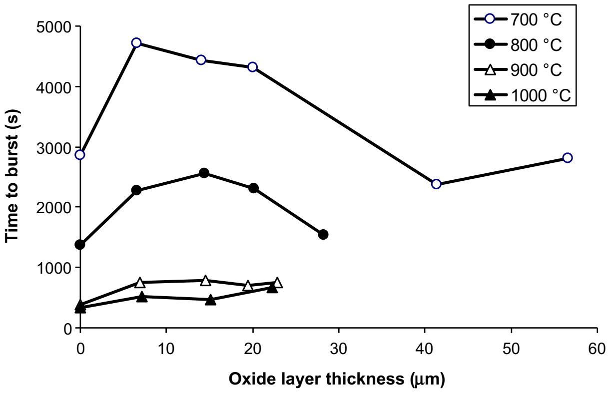


Fig. 5. Measured time to burst at constant pressurization rate in biaxial tests as a function of the ZrO_2 layer thickness on pre-oxidized E110 cladding samples.

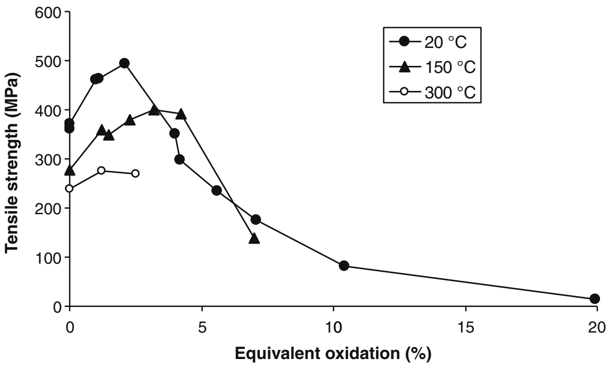


Fig. 6. Tensile strength of E110 as a function of the oxidation and the temperature.

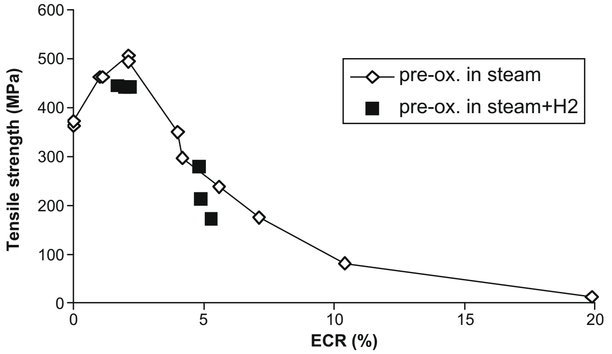


Fig. 7. Tensile strength of E110 as a function of the oxidation at room temperature.

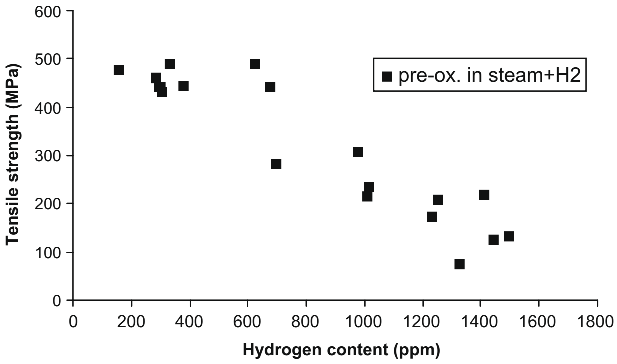


Fig. 8. Tensile strength of E110 as a function of the hydrogen content at room temperature.

hydrogen concentration around the burst was not different from the concentration at the burst. On the other hand a relationship between the hydrogen content and the cladding strength was not observed.

5. Tensile tests

The database contains results from a series of tests in pure and in hydrogen-rich steam. About 110 tensile tests were carried out with tube and sheet specimens using an INSTRON 1195 universal testing machine. The velocity of the crosshead moving was 0.5–2 mm/min. Digital data acquisition provided the recording of the load–displacement curves.

5.1. Test series with samples oxidized in pure steam atmosphere

The primary objective of the tensile tests was to investigate the effect of temperature and oxidation on the strength of E110 alloy. Most of the tests were carried out in the temperature range of 20–350 °C. The effect of oxidation on the mechanical strength was studied through the testing of pre-oxidized specimens. Specimens with and without annealing as well as with and without pre-oxidation, were compared. The annealing of the specimens was performed at 580 or 700 °C for several hours. Pre-oxidation of the specimens was performed in steam atmosphere at 900 °C for different times between 100 and 1600 s. Both the temperature and the steam flow rate were constant during the oxidation. Since the ends of the tube specimens were plugged, only the outer surfaces were oxidized.

The annealing did not influence the strength and the strain of the specimens considerably. On the other hand the pre-oxidation of the specimens in steam atmosphere had a significant effect. Tensile test and ballooning experiments indicated similar effects of oxidation on cladding strength and deformation. Below an ECR of about 5%, the tensile strength increases with oxidation up to a definite maximum and decreases with further oxidation. This phenomenon is clearly seen in Fig. 6, and moreover in Figs. 5 and 7, together representing the test data from three different experiments.

5.2. Test series with samples oxidized in hydrogen-rich steam atmospheres

The aim of these tests was to investigate the effect of hydrogen-rich steam oxidation and the hydrogen content on the strength of E110 alloy. The pre-oxidation of the samples was performed from 900 to 1100 °C in hydrogen-rich steam. The tensile tests were performed at room temperature. The effect of the oxidation was consistent with the results of earlier tensile tests with cladding specimens pre-oxidized in pure steam at 900 °C [4]. However, the tensile strength is slightly lower in a steam–hydrogen mixture than in pure steam (Fig. 7). This means that the fuel cladding is less loadable presumably, due to the higher hydrogen content of the cladding oxidized in hydrogen-rich steam. (The hydrogen content was not measured after the earlier tensile tests.)

On the basis of the experiments the mechanical deterioration of the cladding was observed above 600 wppm hydrogen content (Fig. 8).

6. Ring compression tests

About 110 radial compression tests are included in the database. The compression tests were performed at room temperature,

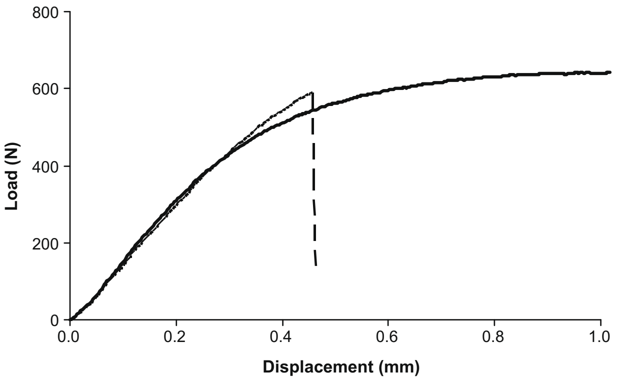


Fig. 9. Load–displacement curves recorded during ring compression tests of E110.

using the same universal testing machine used for the tensile tests. The load–displacement curves were recorded. The specific energy at failure was used for the evaluation of the cladding ductility since it can be easily determined as the quotient of the integral of the force–displacement curve and the ring height

$$E_s = \frac{1}{L}\int_0^{U_C} F(U)dU \tag{5}$$

where E_s is the specific energy, F is the compression force, U is the displacement, U_c is the displacement at first ring crack, L is the ring height.

6.1. Test series with samples oxidized in pure steam atmosphere

The objectives of the compression tests were to characterize the embrittlement process and to investigate the ductile–brittle transition of the cladding. Fig. 9 shows typical load–displacement curves of ring compression tests for ductile and brittle E110 specimens. The ductile behaviour is characterized by a plateau typical of plastic deformation. The evaluation of the experiments indicated ductile characteristics only above 50 mJ/mm specific energy [11,12]. Hence, this specific energy was chosen as a boundary value for the categorization of the ring specimens to “ductile” or “brittle”. On the basis of this categorization the ductility limit was expressed in terms of oxidation time as a function of the temperature [13]:

$$\tau = 2 \times 10^{-4} \exp\left(\frac{17500}{T}\right) \tag{6}$$

This function of the ductility limit gives the maximum oxidation time τ (s) for which the cladding remains ductile at temperature T (K).

6.2. Test series with samples oxidized in hydrogen-rich steam atmosphere

Fig. 10 illustrates the specific energy at failure versus the hydrogen content of the specimens. The filled symbols indicate the ductile specimens. Considering the 50 mJ/mm limit, the ductile–brittle transition of the cladding takes place at a hydrogen content of about 500 wppm.

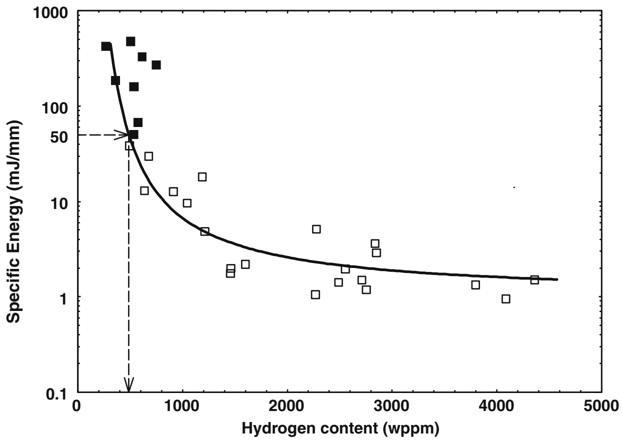


Fig. 10. Specific energy at failure as a function of hydrogen content of the samples.

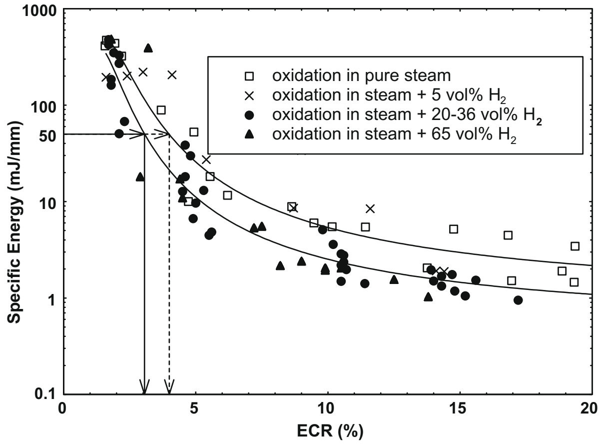


Fig. 11. Specific energy at failure versus the measured oxidation ratio.

Fig. 11 represents the specific energy at failure as a function of the measured ECR. The open symbols indicate the specimens oxidized in pure steam. The ductility limit is at an ECR of about 4–5% oxidation level. These results are compared with recent data for specimens oxidized in a steam–hydrogen mixture of 20–65 vol.% H_2, represented by the filled symbols. The figure clearly shows that, due to more intense hydrogen absorption, the embrittlement of the cladding takes place at a lower oxidation level in a steam–hydrogen mixture than in pure steam.

However, the ductility limit remains valid in a steam–hydrogen mixture, because below this line there are no brittle specimens (Fig. 12). In other words, the cladding does not become brittle earlier in a hydrogen-rich steam atmosphere, since the deceleration of the oxidation compensates the negative effect of enhanced hydrogen absorption.

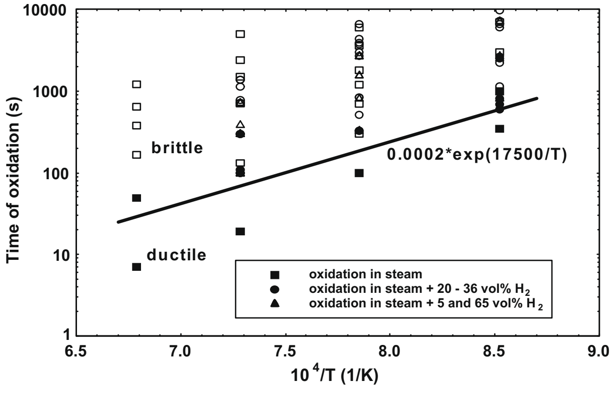


Fig. 12. Time of oxidation versus the reciprocal of the oxidation temperature.

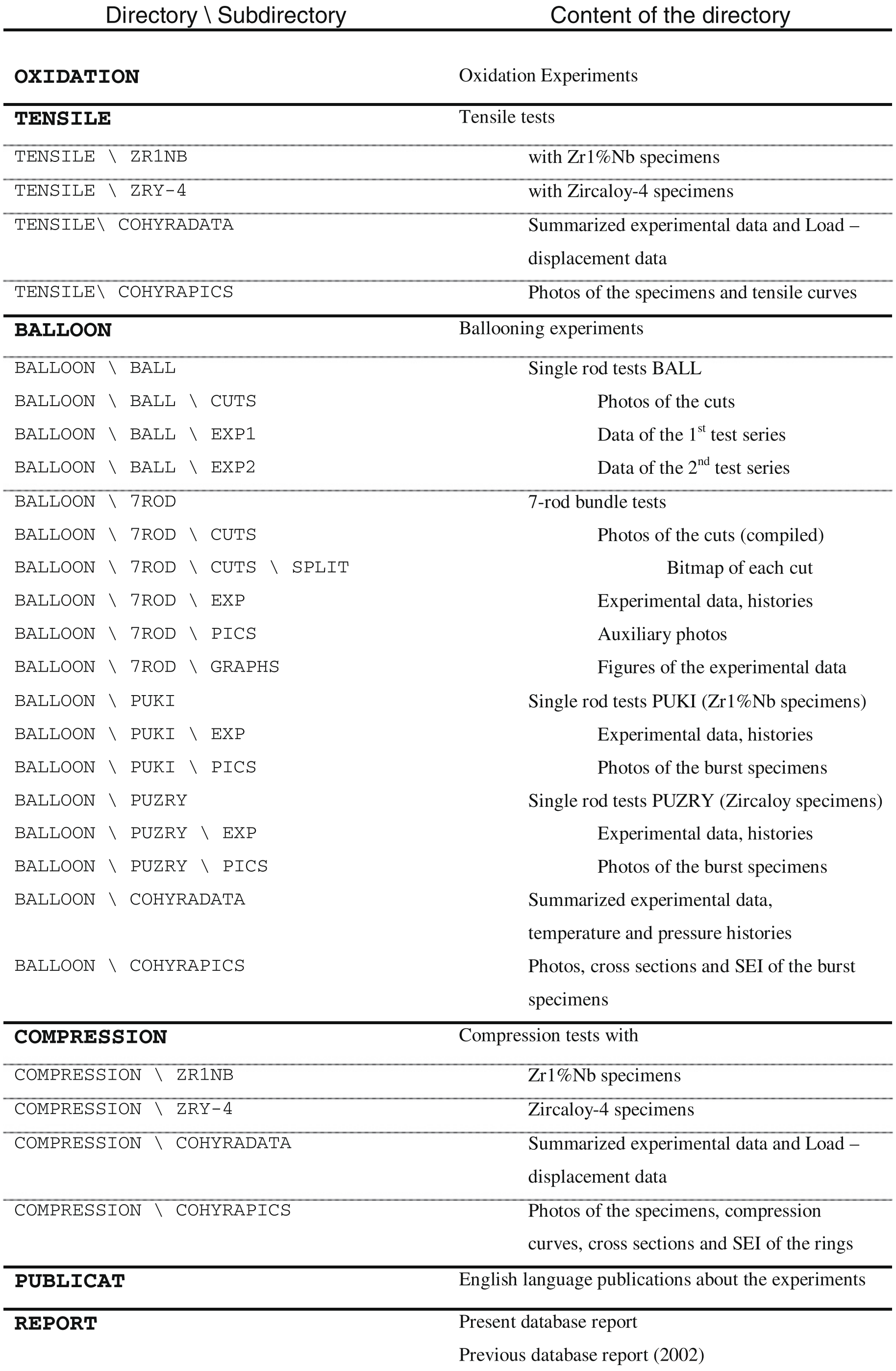
7. Structure of the database

The data and experimental conditions are stored in four main directories for oxidation, ballooning, and the tensile and compression tests. Two additional directories contain the experimental technical reports and the corresponding English-language publications. The results of post-test examinations (visual observations, metallographic analysis, SEM analysis) are also included in the database. All the experimental data are stored in formatted ASCII files (∗.prn) to support computerized processing independently of the applied operation system. The publications, figures and photos are collected in pdf, jpeg or bmp formats. Fig. 13 shows the directory structure of the database.

8. Postscript

This database is a comprehensive compilation of the separate and combined effect tests performed at the AEKI on E110 alloy. It contains about 600 tests and covers a wide range of accident conditions. The experimental database provides detailed information on the VVER cladding behaviour under accident conditions and ensures a solid background for model development. The International Fuel Performance Experimental Database includes this database making it available via the internet. It is open to be populated with data from further experiments.

The AEKI experimental data has already been used in several international projects for the development and validation of



Directory \ Subdirectory	Content of the directory
OXIDATION	Oxidation Experiments
TENSILE	Tensile tests
TENSILE \ ZR1NB	with Zr1%Nb specimens
TENSILE \ ZRY-4	with Zircaloy-4 specimens
TENSILE\ COHYRADATA	Summarized experimental data and Load – displacement data
TENSILE\ COHYRAPICS	Photos of the specimens and tensile curves
BALLOON	Ballooning experiments
BALLOON \ BALL	Single rod tests BALL
BALLOON \ BALL \ CUTS	Photos of the cuts
BALLOON \ BALL \ EXP1	Data of the 1st test series
BALLOON \ BALL \ EXP2	Data of the 2nd test series
BALLOON \ 7ROD	7-rod bundle tests
BALLOON \ 7ROD \ CUTS	Photos of the cuts (compiled)
BALLOON \ 7ROD \ CUTS \ SPLIT	Bitmap of each cut
BALLOON \ 7ROD \ EXP	Experimental data, histories
BALLOON \ 7ROD \ PICS	Auxiliary photos
BALLOON \ 7ROD \ GRAPHS	Figures of the experimental data
BALLOON \ PUKI	Single rod tests PUKI (Zr1%Nb specimens)
BALLOON \ PUKI \ EXP	Experimental data, histories
BALLOON \ PUKI \ PICS	Photos of the burst specimens
BALLOON \ PUZRY	Single rod tests PUZRY (Zircaloy specimens)
BALLOON \ PUZRY \ EXP	Experimental data, histories
BALLOON \ PUZRY \ PICS	Photos of the burst specimens
BALLOON \ COHYRADATA	Summarized experimental data, temperature and pressure histories
BALLOON \ COHYRAPICS	Photos, cross sections and SEI of the burst specimens
COMPRESSION	Compression tests with
COMPRESSION \ ZR1NB	Zr1%Nb specimens
COMPRESSION \ ZRY-4	Zircaloy-4 specimens
COMPRESSION \ COHYRADATA	Summarized experimental data and Load – displacement data
COMPRESSION \ COHYRAPICS	Photos of the specimens, compression curves, cross sections and SEI of the rings
PUBLICAT	English language publications about the experiments
REPORT	Present database report Previous database report (2002)

Fig. 13. Directories of the database and their contents.

transient fuel behaviour codes and severe accident codes (TRANSURANUS, FRAP-T6, FRAPTRAN, MELCOR) [14,15].

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