ICANS-XV
15th Meeting of the International Collaboration on Advanced Neutron Sources
November 6-9, 2000-10-23 Tsukuba, Japan

23.18

Use of Zircaloy 4 material for the pressure vessels of hot and cold neutron sources and beam tubes for research reactors

Dr. Erwin Gutsmiedl — Technical University Munich, FRM-II, Germany
E-mail: ERWIN.GUTSMIEDL@Physik.TU-Muenchen.DE
Dipl.-Ing. Anton Scheuer — TÜV Rheinland/Berlin-Brandenburg e.V., Cologne, Germany

Abstract

The material Zircaloy 4 can be used for the pressure retaining walls for the cold and hot neutron sources and beam tubes. For the research reactor FRM-II of the Technical University Munich, Germany, the material Zircaloy 4 were chosen for the vessels of the cold and hot neutron source and for the beam tube No. 6.

The sheets and forgings of Zircaloy 4 were examinated in the temperature range between –256 °C and 250 °C. The thickness of the sheets are 3, 4, 5 and 10 mm, the maximum diameter of the forgings was 560 mm. This great forging diameters are not be treated in the ASTM rule B 351 for nuclear material, so a special approval with independent experts was necessary. The requirements for the material examinations were specified in a material specification and material test sheets which based on the ASTM rules B 351 and B 352 with additional restriction and additional requirements of the basic safety concept for nuclear power plants in Germany, which was take into consideration in the nuclear licensing procedure. Charpy-V samples were carried out in the temperature range between –256 °C and 150 °C to get more information on the ductile behaviour of the Zircaloy 4.
The results of the sheet examination confirm the requirements of the specifications, the results of the forging examination in the tangential testing direction are lower than specified and expected for the tensile strength. The axial and transverse values confirm the specification requirements.

For the strength calculation of the pressure retaining wall a reduced material value for the forgings has to take into consideration.

The material behaviour of Zircaloy 4 under irradiation up to a fluence of ~ $1 \cdot 10^{22}$ n/cm² was investigated. The loss of ductility was determined. As an additional criteria the variation of the fracture toughness was studies.

Fracture mechanic calculations of the material were carried out in the licensing procedure with the focus to fulfill the leak before rupture criteria of the vessel wall. The results shows a good material behaviour against specified cracks in the unirradiated and irradiated material condition.

Fatigue analysis curves were determined under consideration of the material test data and the influence of the irradiation fluence up to $1 \cdot 10^{22}$ n/cm².

1. Introduction

For the cold and hot neutron sources and the beam tube No. 6 of the research reactor FRM-II, the material Zircaloy 4 were used for the pressure retaining walls. The constructional dimensions of the components are for the

- **hot source**
  inpile part (outer vessel)
  $\varnothing_{a1}$ = 289 mm $\varnothing_{i1}$ = 283 mm s_1 = 3 mm l_1 = 709 mm

  inpile part (outer tubes)
  $\varnothing_{a2}$ = 74 mm $\varnothing_{i2}$ = 68 mm s_2 = 3 mm l_2 = 1440 mm
  $\varnothing_{a3}$ = 86 mm $\varnothing_{i3}$ = 80 mm s_3 = 3 mm l_3 = 3282 mm

  inpile part (inner vessel)
  $\varnothing_{a4}$ = 283 mm $\varnothing_{i4}$ = 277 mm s_4 = 3 mm l_4 = 720 mm

  inpile part (inner tubes)
  $\varnothing_{a5}$ = 66 mm $\varnothing_{i5}$ = 60 mm s_5 = 3 mm l_5 = 4806 mm

- **cold source (vacuum vessel)**
  bottom part
  $\varnothing_a$ = 319,5 mm $\varnothing_i$ = 311,5 mm s = 4 mm l = 503 mm

  top part
  $\varnothing_{a1}$ = 140 mm $\varnothing_i$ = 134 mm s = 3 mm l = 1476 mm
  $\varnothing_{a2}$ = 230 mm $\varnothing_i$ = 224 mm s = 3 mm l = 286 mm
  $\varnothing_{a3}$ = 402 mm $\varnothing_i$ = 394 mm s = 4 mm l = 892 mm
  $\varnothing_{a4}$ = 540 mm (flange part) l = 80 mm

- **beam tube**
  $\varnothing_{a1}$ = 265 mm $\varnothing_{i1}$ = 255 mm s = 5 mm l = 1030 mm
  $\varnothing_{a2}$ = 163 mm $\varnothing_{i2}$ = 155 mm s = 4 mm l = 1508 mm
  $\varnothing_{a3}$ = 270 mm (flange part) l = 90 mm
  $\varnothing_{a4}$ = 400 mm (flange part) l = 135 mm

For manufacturing of these parts the following semi-finished product were ordered:

sheets: 21 pieces with 3 mm thickness
sheets: 17 pieces with 4 mm thickness
sheets: 2 pieces with 5 mm thickness
sheets: 5 pieces with 10 mm thickness

forgings: 21 pieces (plates, rings, rods) with $\varnothing_a$ up to 370 for plates, $\varnothing_a$ = 550 mm , $\varnothing_i$ = 384 mm for rings and $\varnothing_a$ = 125 mm for rods

2. Material specification

The requirements for the semi-finished product were fixed in the material specification [1] and the material test sheets [2], [3]. The material specification and material test sheets based on the ASTM rules B 351-92 /5/ and B 352-92 [4]. In the chemical analysis the composition of the element hydrogen (H), carbon (C), oxygen (O) were restricted to H < 20 ppm, C $\leq$ 200 ppm, O = 900-1400 ppm.

Further the elements Pb, Ca, Cl, Na, Nb, P, S, Ti, V were measured.
The requirements for the mechanical properties of the annealed condition in the longitudinal and transverse testdirection for the tensile strength, the yield strength and elongation for the sheets are identical with the values in [4].

The requirements for the mechanical properties for the forgings based in the longitudinal direction on the values of [5]. For the transverse or tangentional direction at room temperature (RT) the values for the tensile strength, yield strength and elongation, which were not specified in [5], the same values from the longitudinal direction were used for acceptance values. At higher temperature no acceptance values were further fixed, these values shall be fixed within approval of the material with the independent experts.

For the charpy-V-test the lowest single values at RT was fixed for acceptance of $\geq$ 24 J/cm².
The following tests were specified in the material test sheet.

1. Chemical analysis at the ingot and the semi-finished products
2. Tensile Tests at RT and Designtemperature
3. Charpy-V-Tests at RT
4. Charpy-V-Tests in the temperature range between –256 °C and +150 °C
5. Bend test for plates
6. Corrosion tests
7. Metallographic Tests (Type of Microstructure, Cleanliness, Grain Size)
8. Hardness Tests
9. Ultrasonic Tests
10. Dimensional Control
11. Roughness Tests
12. Visual Inspection

These tests were part of the inspection and test manual of the manufacturer.

The material specification [3] and the material test sheets [4], [5] contains the requirements of the basic safety concept for nuclear power plants in Germany, which was take into consideration in the nuclear liscensing procedure. The material specification and the material test sheets were approved by the independent experts.

For the sheets the number of tests were fixed per lot, where a lot contains 5 sheets, for the forgings the lot was specified per 500 kg.

Additionally to the material test sheet examination further material testing occur within the individual expert analysis and opinion report.

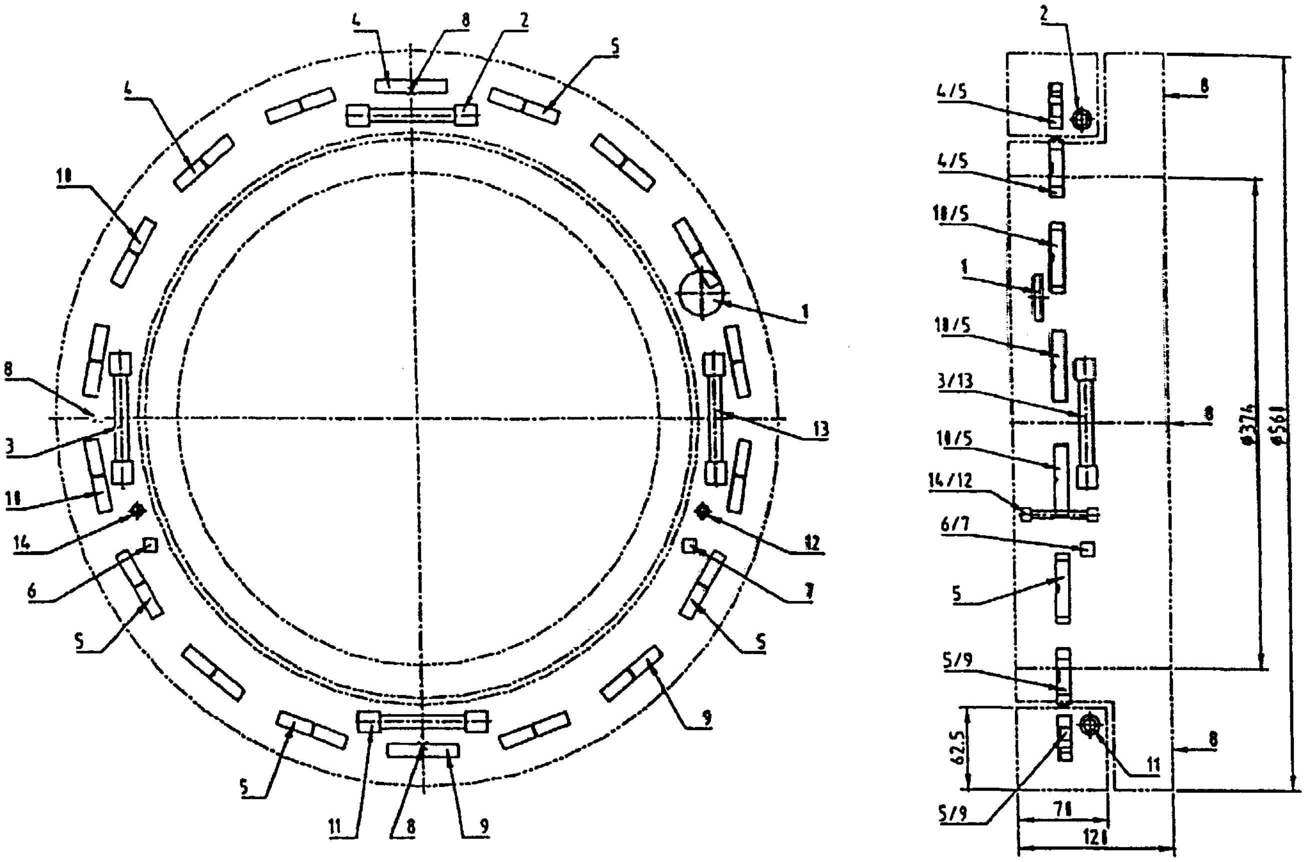
The following additional testings were carry out for the sheets:

For thickness 4 mm
- 2 transverse tensile test at room temperature at each end of each master strip
- 2 transverse bend test at each of each master strip
- 1 longitudinal + 1 transverse tensile test at 288 °C at one end of each master strip

For thickness 10 mm
- 1 longitudinal + 1 transverse tensile test at 288 °C at one end of each master strip
- 2 transverse tensile test at room temperature at each end of each master strip

Further for the 10 mm thickness Charpy-V-tests at the temperatures 150 °C, RT, -196 °C, –256 °C in the longitudinal and transverse direction were tested.

For the forgings are carry out material testings for tensile tests and Charpy-V-tests near these surface, at d/4 and d/2 of the forging diameter to check the forging ratio in the tangentional and axial direction. Further tensile and Charpy-V-tests are carry out for different testing directions and temperatures. The material testing and samples plans for forging-dimension are explained in figure 1 and 2.



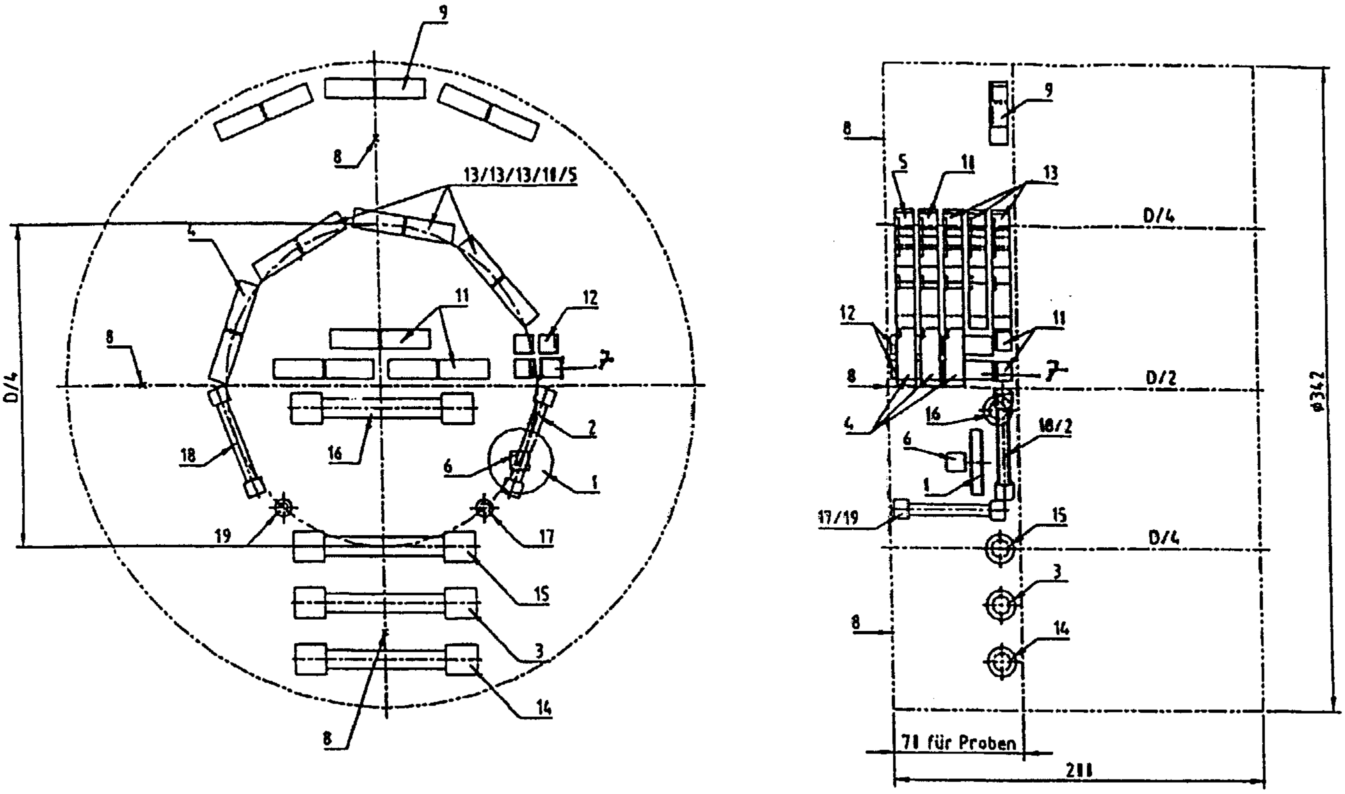
Samples corresponding material test sheet

1. Chemical analysis
2. Tensile test at Roomtemperature (RT)
3. Tensile test at Design temperature
4. Charpy-V test
5. Charpy-V test for toughness curve, temperatures –196 °C, -20°C, +150 °C
6. Corrosion test
7. Metallographic test
8. Hardness test 3 x

Additional Samples corresponding expert approval

9. Charpy-V test at 180°
10. Charpy-V test at –256 °C
11. Tensile test, RT
12. Tensile test, axial
13. Tensile test, tangential at 288 °C
14. Tensile test, axial at 288 °C

Figure 1: Material Testing- and Sampling Plan
Forging dimension 560 / 374 mm rd. x 120



Samples corresponding material test sheet

1. Chemical analysis
2. Tensile test at RT
3. Tensile test at DT
4. Charpy-V test at RT
5. Charpy-V test
6. Corrosion test
7. Metallographic test
8. Hardness test 3 x

Additional Samples corresponding expert approval

9. Charpy-V test, tangential at RT
10. Charpy-V test, tangential D/4 at RT
11. Charpy-V test, tangential D/2 at RT
12. Charpy-V test, axial D/4 at RT
13. Charpy-V test, tangential at –256 °C
14. Tensile test, tangential at RT
15. Tensile test, tangential D/4 at RT
16. Tensile test, tangential D/4 at RT
17. Tensile test, axial D/4 at RT
18. Tensile test, tangential at 288 °C
19. Tensile test, axial at 288 °C

Figure 2: Material Testing- and Sampling Plan
Forging dimension 342 mm rd. x 200

3. Material delivery

The production of the Zircaloy 4 were fixed from the manifacturer in a special process flow outline. This process flow outline has fixed the fabrication process from the preparing of the electrodes with the sponge, the alloying elements and/or recycled material to the ingot and slab preparation and final production of the plates and forgings.

The ingot production for the plates and the final production of the plates take place in a french company, the final production of the forgings takes place in a german company.

4. Test results of the sheets

4.1 Chemical composition

The chemical composition on finished product are within the specification.

ELEMENTS	NITROGEN	HYDROGEN	OXYGEN	THIN	IRON	CHROMIUM	IRON + CHROMIUM
Specification	≤ 80 ppm	≤ 25 ppm	900-1400 ppm	1.20 to 1.70 %	0.18 to 0.24 %	0.07 to 0.13 %	0.28 to 0.37 %
Results (3 mm) Strip Nr. 21.1 Strip Nr. 21.2	29 25	9 9	1220 1210	1.32 1.31	0.21 0.21	0.11 0.11	0.32 0.32
Results (4 mm) Strip Nr. 11.1 Strip Nr. 11.2	28 30	9 10	1210 1180	1.34 1.32	0.21 0.21	0.10 0.10	0.31 0.31
Results (5 mm) Strip Nr. 12.1 Strip Nr. 12.2	23 25	9 13	1200 1220	1.32 1.32	0.21 0.21	0.10 0.10	0.31 0.31
Results (10 mm) Strip Nr. 35.1 Strip Nr. 36.1	29 27	9 9	1190 1220	1.54 1.53	0.21 0.21	0.11 0.11	0.32 0.32

4.2 Mechanical Properties

The results of the mechanical properties for the tensile tests are shown in table 1, 2, 3, 4, 5. The results are higher than specified. The values of the uniform elongation are between 8,75 % an 13,5 %.

AT ROOM TEMPERATURE

	Ultimate Tensile (N/mm^2)		0,2 % Yield Strength (N/mm^2)		Elongation (%)		Area reduction when breaking (%)	
Direction	L	T	L	T	L	T	L	T
Specified	≥ 400	≥ 386	≥ 241	≥ 303	≥ 25	≥ 25	for information purpose	
Strip Nr. 21.1	503	478	337	429	30	31	52	62
Strip Nr. 11.1	502	479	353	427	31	30	51	60
Strip Nr. 12.2	496	469	332	411	29	31	52	58
Strip Nr. 35.1	518	495	344	451	27	32	43	56

Table 1: Mechanical properties at RT

AT 150 °C

	Ultimate Tensile (N/mm²)		0,2 % Yield Strength (N/mm²)		Elongation (%)		Area reduction when breaking (%)	
Direction	L	T	L	T	L	T	L	T
Specified	for information purpose							
Strip Nr. 21.1	358	317	235	270	41	48	64	71
Strip Nr. 11.1	360	328	226	277	42	47	62	71
Strip Nr. 12.2	376	310	213	251	44	47	63	86
Strip Nr. 35.1	378	330	232	300	37	44	51	66

Table 2: Mechanical properties at 150 °C

AT 250 °C

	Ultimate Tensile (N/mm²)		0,2 % Yield Strength (N/mm²)		Elongation (%)		Area reduction when breaking (%)	
Direction	L	T	L	T	L	T	L	T
Specified	for information purpose							
Strip Nr. 21.1	272	244	147	173	46	48	68	79
Strip Nr. 11.1	270	235	144	172	42	45	66	75
Strip Nr. 12.2	261	234	130	158	42	49	64	74
Strip Nr. 35.1	283	250	154	197	40	43	62	72

Table 3: Mechanical properties at 250 °C

	Ultimate Tensile (N/mm²)	0,2 % Yield Strength (N/mm²)	Elongation (%)	Area reduction when breaking (%)
Direction	T	T	T	T
Specified	≥ 386	≥ 303	≥ 25	for information purpose
Strip 11.1 Top	474	425	31	59
Strip 11.1 Bottom	474	428	32	61
Strip 11.2 Top	473	422	32	61
Strip 11.2 Bottom	474	425	32	59
Strip 11.3 Top	462	412	33	62
Strip 11.3 Bottom	462	415	32	61
Strip 35.1 Top	494	453	30	56
Strip 35.1 Bottom	492	450	28	56
Strip 36.1 Top	499	459	30	53
Strip 36.1 Bottom	492	450	31	54

Table 4: Mechanical properties for additional testing at RT

	Ultimate Tensile (N/mm²)		0,2 % Yield Strength (N/mm²)		Elongation (%)		Area reduction when breaking (%)	
Direction	L	T	L	T	L	T	L	T
Specified	≥ 186	≥ 179	≥ 103	≥ 120	≥ 30	≥ 30	for information purpose	
Strip Nr. 11.1	253	222	126	149	43	44	67	78
Strip Nr. 11.2	247	220	138	151	43	45	66	79
Strip Nr. 11.3	244	215	128	144	43	44	68	74
Strip Nr. 36.1	248	227	131	171	36	41	60	64

Table 5: Mechanical properties for additional testing at 288 °C

4.3 Impact strength

The results of the impact strength fulfill with exceptions of two single values (strip 35.1 and 36.1) the aimed value of 24 J/cm² The impact strength decrease with lower temperature than RT and is appropriate for the material involved. For the higher temperature 150 °C the impact strength increase to a mean value of 56 J/cm² and is higher than the aimed value of 24 J/cm². All specimen shows no crystallin proportion in the temperature range between 150 °C to -256 °C.

Strip Nr.	Thickness	Specimen direction	Testtemp. °C	Impact strength	Mean value J/cm²
12.1	3	Transv.	RT	30/30/31	30
12.2	3	Transv.	RT	30/29/32	30
35.1	10	Transv.	RT	21/30/31	27
36.1	10	Transv.	RT	19/31/30	27
		Long.	RT	34/36/26	32
		Transv.	RT	25/25/25	25
		Transv.	150	41/64/64	56
		Transv.	-20	19/25/24	23
		Transv.	-196	8/11/11	10
		Transv.	-256	11/14/10	12
		Long.	-196	16/15/13	15

Table 6: Results of Charpy-V-tests

4.4 Grain Size Determination

The specification value of finer or equal to 9 ASTM E112 were fulfilled with values of

- 11,5 for 3 mm thickness,
- 11 for 4 mm thickness,
- 10,5 for 5 mm thickness,
- 9 for 10 mm thickness.

4.5 Hardness tests Rockwell B

Specification	≤ 98 HRB
Strip Nr. 21.1 (3 mm) Top Bottom	 92 – 92 – 92 93 – 94 – 94
Strip Nr. 21.2 (3 mm) Top Bottom	 92 – 93 – 93 93 – 92 – 93
Strip Nr. 21.3 (3 mm) Top Bottom	 93 – 93 – 92 95 – 93 – 94
Strip Nr. 11.1 (4 mm) Top Bottom	 91 – 92 – 91 93 – 92 – 93
Strip Nr. 11.2 (4 mm) Top Bottom	 91 – 91 – 92 93 – 92 – 93
Strip Nr. 11.3 (4 mm) Top Bottom	 90 – 92 – 92 92 – 91 – 93
Strip Nr. 12.1 (5 mm) Top Bottom	 90 – 91 – 91 93 – 93 – 92
Strip Nr. 12.2 (5 mm) Top Bottom	 91 – 91 – 92 92 – 92 – 92
Strip Nr. 35.1 (10 mm) Top Bottom	 88 – 87 – 88 87 – 89 – 89
Strip Nr. 36.1 (10 mm) Top Bottom	 90 – 89 – 89 88 – 87 – 88

4.6 Corrosion resistance

The 72-hours corrosion tests at temperature of 400 °C on pickled samples at pressure 105 bar shows a weight gains of

- 18.0, 18.7 mg/dm² (3 mm plates)
- 19.5, 18.5 mg/dm² (4 mm plates)
- 18.4, 18.3 mg/dm² (5 mm plates)
- 19.6, 19.8 mg/dm² (10 mm plates)

and fulfilled the specified gain of $\leq$ 22 mg/dm².

4.7 Bend tests

The bend tests with a radius of 3 x plate thickness for 3, 4, 5 mm strips and 5 x plate thickness for 10 mm strips (one face machined up to 8 mm) shows no evidence of cracking on the outer surfaces.

4.8 Dimensional and visual Inspections are conform the specification

4.9 Final heat treatment

The final heat treatment takes place for the 3 mm, 4 mm and 5 mm in a continuos furnace with a speed of 1 m/min (3 mm), 0,8 m/min (4 mm), 0,6 m/min (5 mm) at a temperature of 745 °C, for the 10 mm strips in a static furnace with a time of 3 to 4 hours, at a temperature of 650-700 °C.

4.10 Ultrasonic testing

The ultrasonic testing of the slabs and of the finished product are conform the specification.

5. Test results of the forgings

5.1 Chemical analysis

The check analysis of the pieces shows for H = 9 ppm, N =23 ppm and O = 1230 ppm, the other element are within the specification requirements.

5.2 Mechanical properties

The specified values for the tensile test are listed in table 7.

Temperature °C	Specimen Direction	Ultimate Tensile N/mm²	0,2 Yield Strength N/mm²	Elongation %
RT	Transverse Tangentional	413	241	14
RT	Longitudinal	413	241	14
316	Longitudinal	214	103	24
316	Transverse Tangentional	to evaluate in the approval		

<u>Table 7:</u> Specified mechanical properties for RT

For the design temperature of the cold and hot neutron source and the beam tube under consideration of a linearity between RT and 316 °C we get the following values listed in table 8 for the ultimate tensile and 0,2 yield strength.

Temperature °C	Ultimate Tensile N/mm²	0,2 Yield Strength N/mm²
130	338	190
150	325	180
180	305	166
250	258	133
288	233	115

<u>Table 8:</u> Expected mechanical properties for 130, 150, 180, 250, 288 °C

A comparison from the material test results with these values shows, that for some specimen the Ultimate Tensile Strength (UTS) for RT and higher temperature in the tangentional and/or transverse direction are lower than expected in table 8 (see also figure 3 and 4).

There is a deviation of maximum 31 N/mm² in the tensile strength at RT for the specimen No. 518-1 and 520-15, this is a variation of –7,5 % of the specified and expected value.

Figure 3 shows the variation range of the test results to the specified UTS of 413 Mpa in the tangentional direction. Figure 2 shows the deviation at the test temperatures of 150 °C, 250 °C and 288 °C. The line in figure 2 is the linearity between the two specified points of 413 Mpa at RT and 214 Mpa at 316 °C.

The results of the mechanical properties for the tensile tests are shown in table 9.

Specimen Nr.	Temp. °C	Specimen Direction	Ultimate Tensile	0,2 % Yield Strength	Elongation	Area Reduction	Umform Elongation
516-2	RT	Tang.	404	292	18	39	11.3
516-11	RT	Tang.	389	300	21	36	12.0
516-12	RT	Transv.	498	400	24	48	11.7
516-3	150	Tang.	383	296	23	38	
516-13	288	Tang.	211	134	47	57	
516-14	288	Axial	264	164	37	70	
517-2	RT	Tang.	427	322	18	38	11
517-3	150	Tang.	359	248	25	47	
518-1	RT	Tang.	382	296	24	38	12.6
518-2	150	Tang.	301	208	34	48	
519-1	RT	Tang.	395	317	25	47	11.1
520-2	RT	Tang.	408	323	23	45	11.3
520-14	RT	Tang.	394	305	30	41	11.7
520-15	RT	Tang.	382	299	26	42	13.9
520-16	RT	Tang.	385	299	26	41	12.2
520-16	RT	Axial	564	407	29	51	22.2
520-3	150	Tang.	298	213	31	51	
520-18	288	Tang.	194	125	43	55	
520-19	288	Axial	279	164	37	67	
521-1	RT	Tang.	397	311	28	46	12.9
521-2	150	Tang.	288	210	36	47	
522-1	RT	Tang.	415	328	24	44	11.8
522-2	250	Tang.	239	161	38	58	
523-1	RT	Tang.	416	329	26	43	13.0
524-1	RT	Tang.	410	322	24	45	10.2
524-2	250	Tang.	222	145	50	55	
525-1	RT	Tang.	409	325	23	44	11.9
526-2	RT	Transv.	436	369	12	46	5.5
526-10	RT	Axial	466	295	21	32	12.7
526-12	RT	Transv.	465	405	16	41	8.1
526-3	250	Transv.	255	182	28	59	
526-11	288	Axial	222	118	50	62	
527-1	RT	Tang.	414	316	29	41	14.3
527-2	250	Tang.	232	145	41	57	
528-1	RT	Tang.	427	321	23	41	11.7
528-2	250	Tang.	236	146	39	58	
529-1	RT	Tang.	434	347	17	41	9.9
529-2	250	Tang.	237	159	33	51	
530-1	RT	Tang.	396	313	21	42	10.8
530-2	250	Tang.	227	154	46	57	
531-1	RT	Tang.	455	355	19	41	9.8
531-2	130	Tang.	346	252	32	47	
532-1	RT	Tang.	446	352	19	40	10.2
533-1	RT	Tang.	470	384	28	49	9.0
533-2	130	Tang.	375	282	36	54	
534-1	RT	Tang.	480	381	24	44	9.8
534-2	180	Tang.	339	239	36	50	

<u>**Table 9:**</u> Material test results for RT, 130, 180, 250, 288 °C

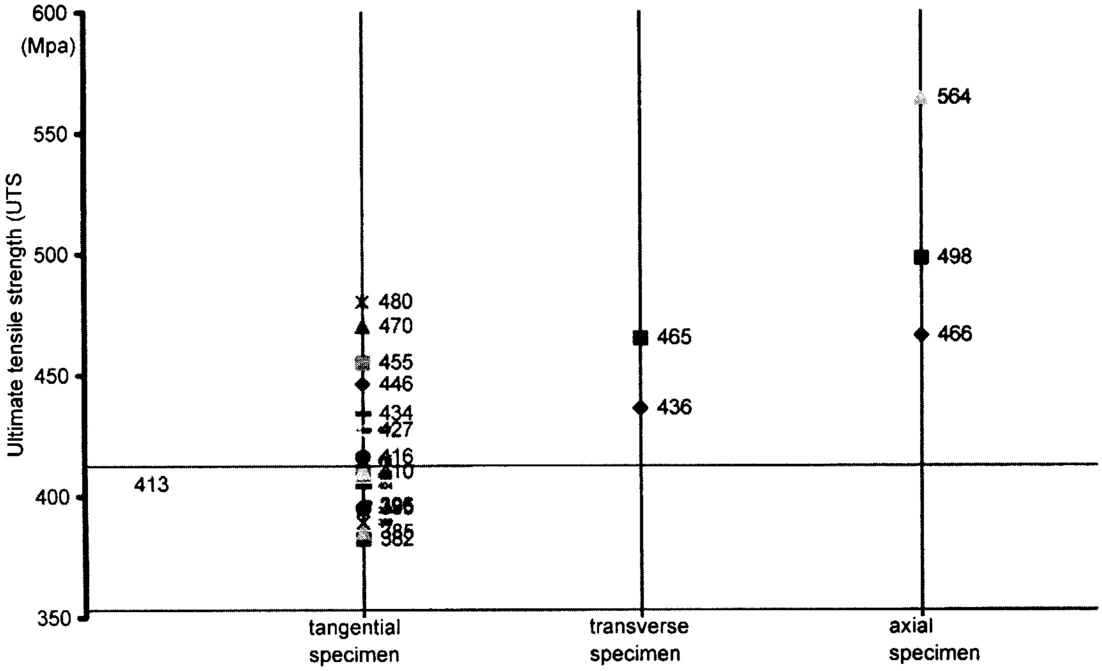


Figure 3: Ultimate tensile test (UTS) at RT for the tangential, transverse and axial direction of forgings

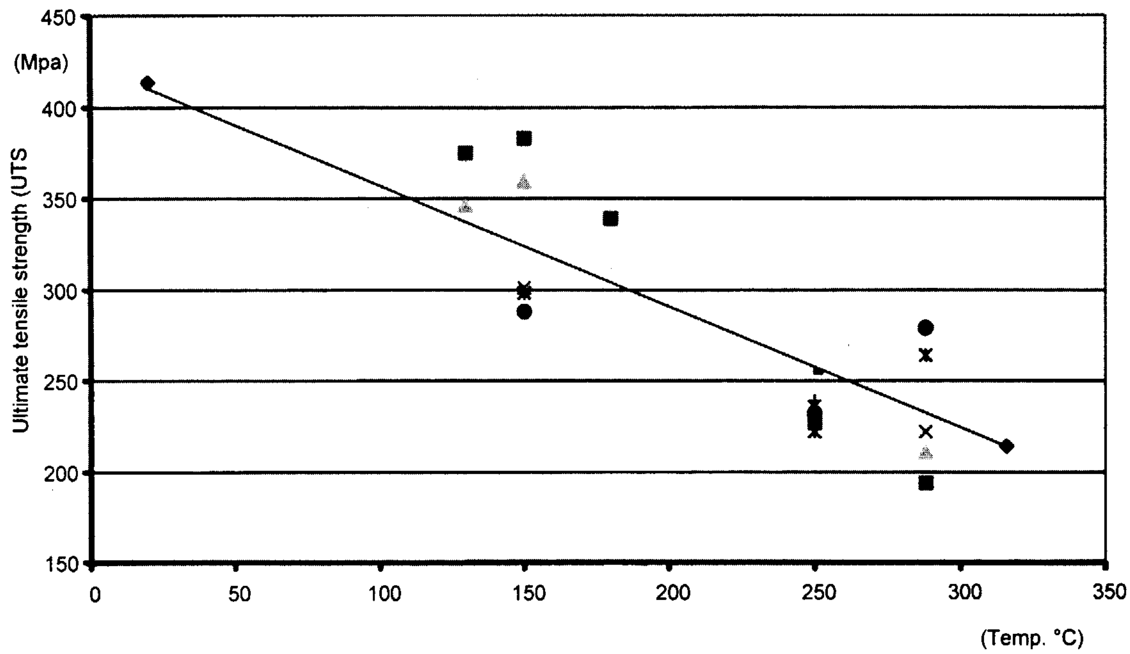


Figure 4: Ultimate tensile strength (UTS) of Zircaloy 4 forgings versus temperature

5.3 Impact strength

The results of the impact strength fulfill the aimed value of 24 J/cm² for RT.

The Charpy-V-tests near the surface, at d/4 and d/2 of the forging diameter shows no variation in the impact strength and confirm a good forging ratio. The transition curve of the impact strength from 150 °C to –256 °C is presented in figure 5 and shows at low temperature a constant toughness behaviour.
All specimen has no crystallin proportion in the temperature range between 150 °C to –256 °C.

Specimen Nr.	Temperature °C	Specimen direction	Impact Strength J/cm²
516-4	RT	Tang.	48/38/38
516-5	150	Tang.	45/43/45
516-5	–20	Tang.	33/33/31
516-5	–196	Tang.	24/19/18
516-9	RT	Tang.	35/38/35
516-10	–256	Tang.	16/15/13
517-4	RT	Tang.	25/38/30
518-3	RT	Tang.	50/60/40
520-4	RT	Tang.	35/33/33
520-5	150	Tang.	43/41/40
520-5	–20	Tang.	26/29/26
520-5	–196	Tang.	10/13/10
520-9	RT	Tang. (surface)	38/38/35
520-10	RT	Tang. (D/4)	30/35/35
520-11	RT	Tang. (D/2)	35/30/33
520-12	RT	Axial (D/4)	55/43/60
520-13	–256	Tang. (D/4)	14/18/15
521-3	RT	Tang.	33/35/33
522-3	RT	Tang.	38/40/38
524-3	RT	Tang.	38/35/40
526-4	RT	Transv.	53/50/43
526-8	RT	Transv.	112/135/88
526-9	RT	Long.	38/54/50
527-3	RT	Tang.	35/40/35
528-3	RT	Tang.	33/35/33
529-3	RT	Tang.	30/50/50
530-3	RT	Tang.	35/35/33
531-3	RT	Tang.	33/40/35
533-3	RT	Tang.	33/40/35

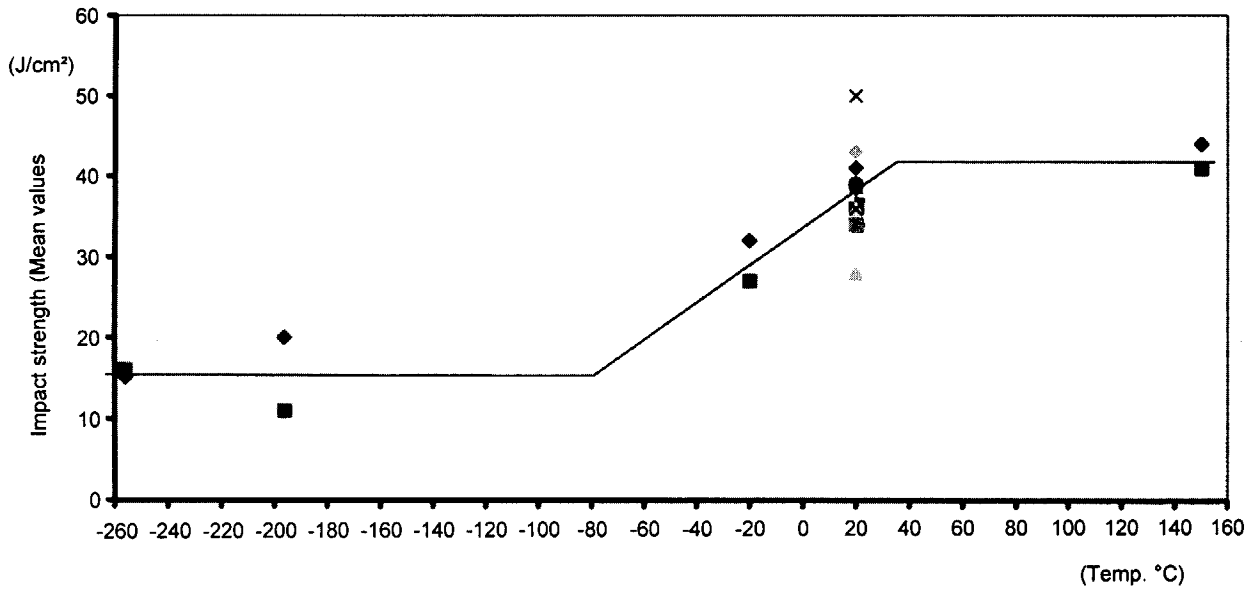


Figure 5: Transition curve of the impact strength for forgings

5.4 Grain Size Determination

The grain size determination are between 6 and 8 (ASTM-values).

5.5 Hardness Test

The hardness measured with Vickers 3 are between 157 and 204.

5.6 Corrosion tests

The corrosion tests shows a loss of weight of 17.7/14.9/15.8/17.6/14.7/15/14.1/14.9 mg/dm² < 22 mg/dm² allowable.

5.7 Dimensional and visual inspection are conform the specification

5.8 Final heat treatment

The final heat treatment takes place in a vacuum furnace at a temperature of 720 °C $\pm$10 °C and a time of 4.5 or 7 hours, depending of the thickness.

5.9 Ultrasonic testing

The ultrasonic testing of the forgings, based on the german guideline SEP 1921, was acceptable and shows no indications.

6. Strength calculation

For the strength calculation of the pressure retaining parts the UTS at RT is used to form the allowable value of Sm (Sm = Min ($UTS_{RT}/3$; 0,2 YST/1,5). So the deviation of –7,5 % has to take in consideration for calculating the Sm-value for the forging parts of the components.

7. Zircaloy 4 behaviour under irradiation

The material behaviour of Zircaloy 4 under irradiation up to a fluence of $1 \cdot 10^{22}$ n/cm² (fast neutrons) with respect to variation of the tensile properties was investigated. The study shows a loss of ductility and an elongation after fracture of ~ 5 % and a percentage elongation before reduction of ~ 1 %. As an additional criteria the fracture toughness of the Zircaloy-4 was studies up to the fluence of $1 \cdot 10^{22}$ n/cm². These data were take over for fracture mechanic calculations.

8. Fracture mechanic calculations

Fracture mechanic calculations of the material were carried out for the operation and emergency conditions temperature between –20 °C to 150 °C. For the fracture toughness value K_{IC} of irradiated material (fluence of $1,5 \cdot 10^{22}$ n/cm²) the following values were used for the annealed material condition [6], [7]:

K_{IC} = 45 MPa $\sqrt{m}$ for 100 °C
K_{IC} = 25 Mpa $\sqrt{m}$ for –20 °C to –180 °C

For the weld lower values were used

K_{IC} = 30 MPa $\sqrt{m}$ for 100 °C
K_{IC} = 20 Mpa $\sqrt{m}$ for –20 °C to –180 °C

The fracture mechanic calculation shows with a postulated crack at the surface of 0,4 mm deepness and 20 mm length and the stresslevel a safety value between the crack length to the critical crack length of 6-8,5 for the design and operational condition and 2-4 for emergency conditions. So the required leak before rupture criteria in the design of the vessels of the cold and hot neutron source and the beam tube No. 6 in the licensing procedure was fulfilled.
Fig. 6 shows the wall break through stress and fig. 7 the critical length of through crack of the vacuum vessel of a cold Neutron source.

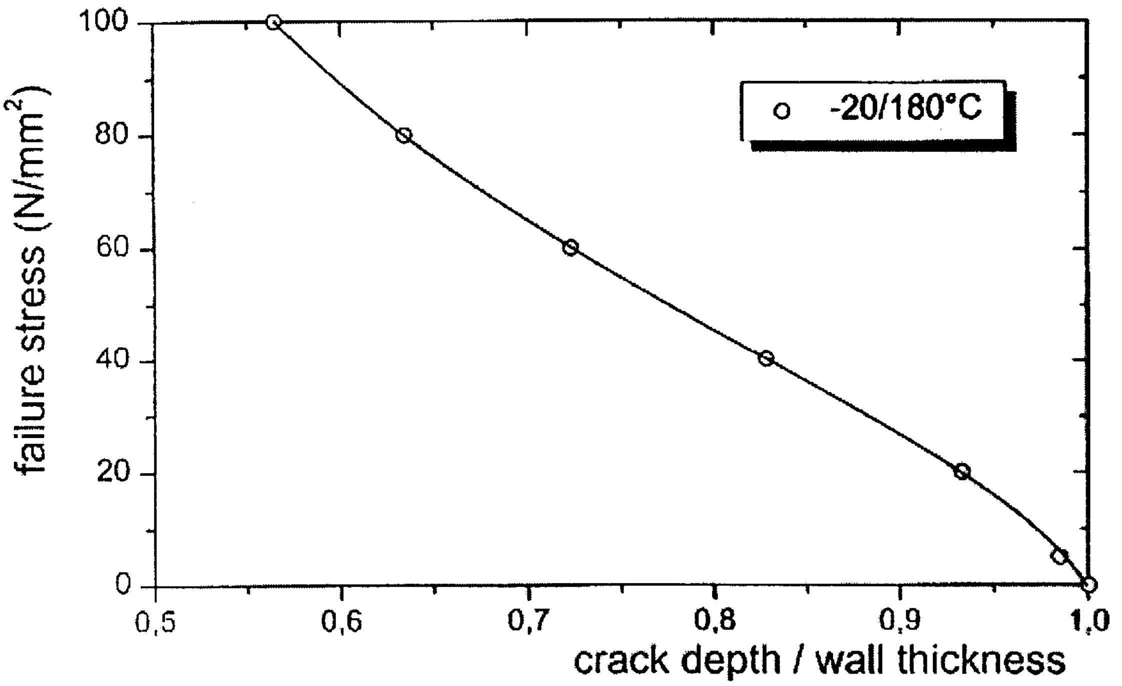


Figure 6: Failure stresses of vacuum vessel of COLD NEUTRON SOURCE (length of surface crack 20 mm, wall thickness 4 mm)

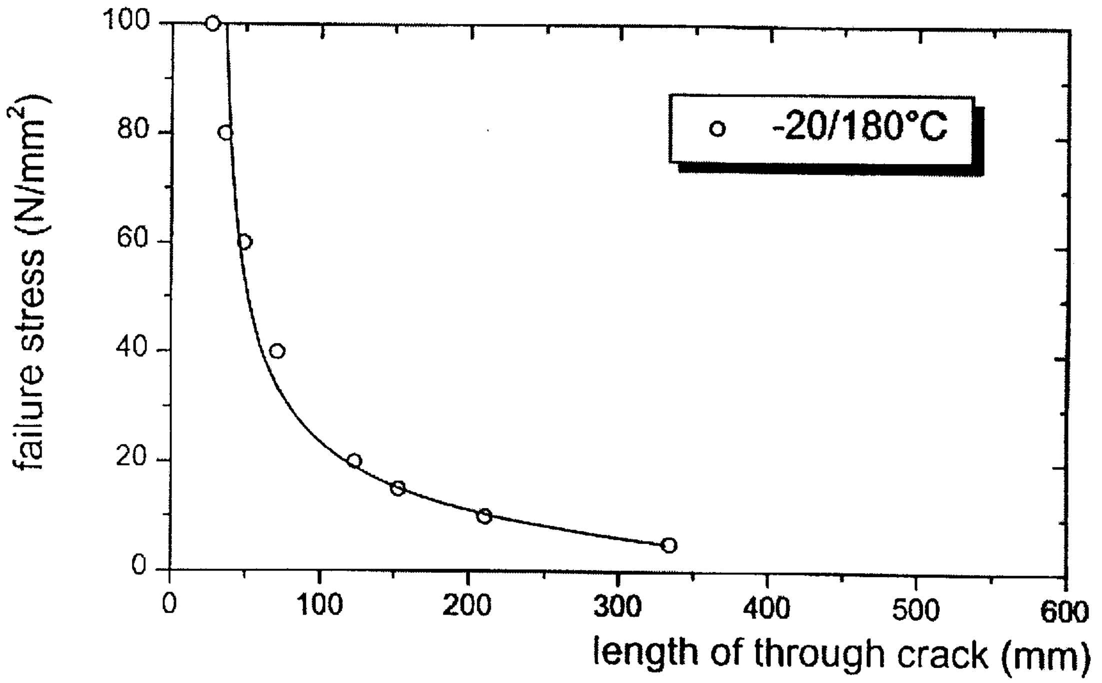


Figure 7: Critical length of through crack of vacuum vessel of COLD NEUTRON SOURCE (wall thickness 4 mm, welding)

9. Fatigue curves for Zircaloy 4

Low cycle fatigue design curves for the unirradiated material Zircaloy 4 were calculated with the method of the Universal Slope of Manson. The material test results were take into consideration. On the base of this results the expected fatigue design curves for irradiated Zircaloy-4 was evaluated, see figure 8, 9. These low cycle fatigue design curves are the base for the fatigue analysis of the vessels of the hot and cold neutron sources and the beam tube No. 6 of FRM-II.

10. Summary

The results of the material testing, the irradiation investigations and the fracture mechanics calculations confirm the use of Zircaloy-4 for vessel material of hot and cold neutron sources and beam tubes for a life time of 30 years and a fluence of $1 \cdot 10^{22}$ n/cm².

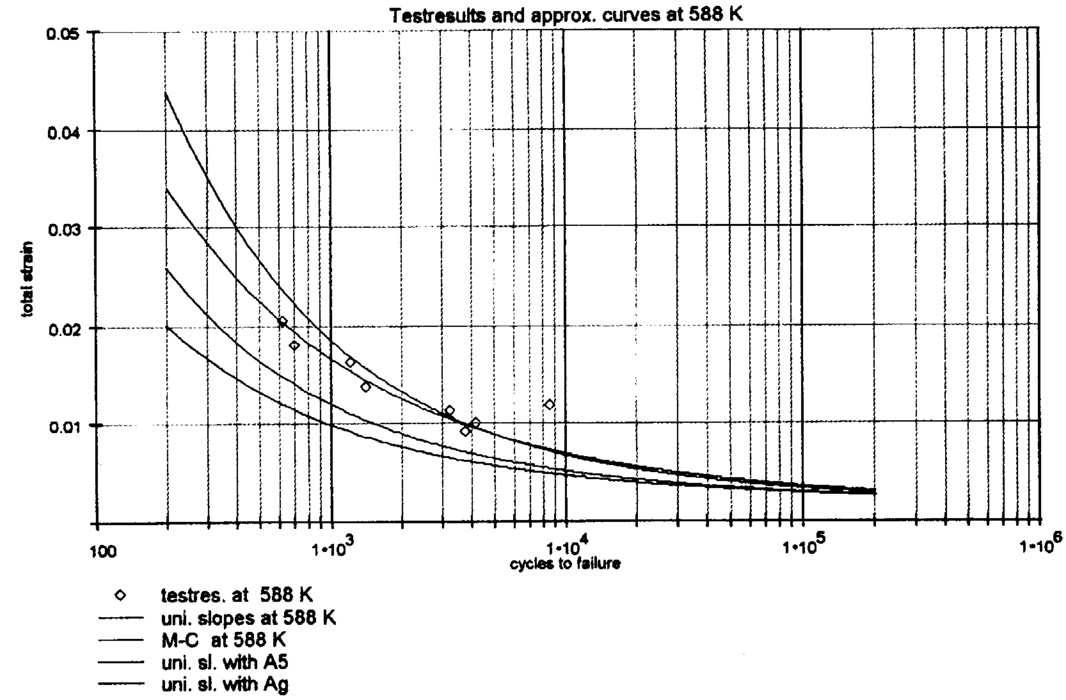


Figure 8

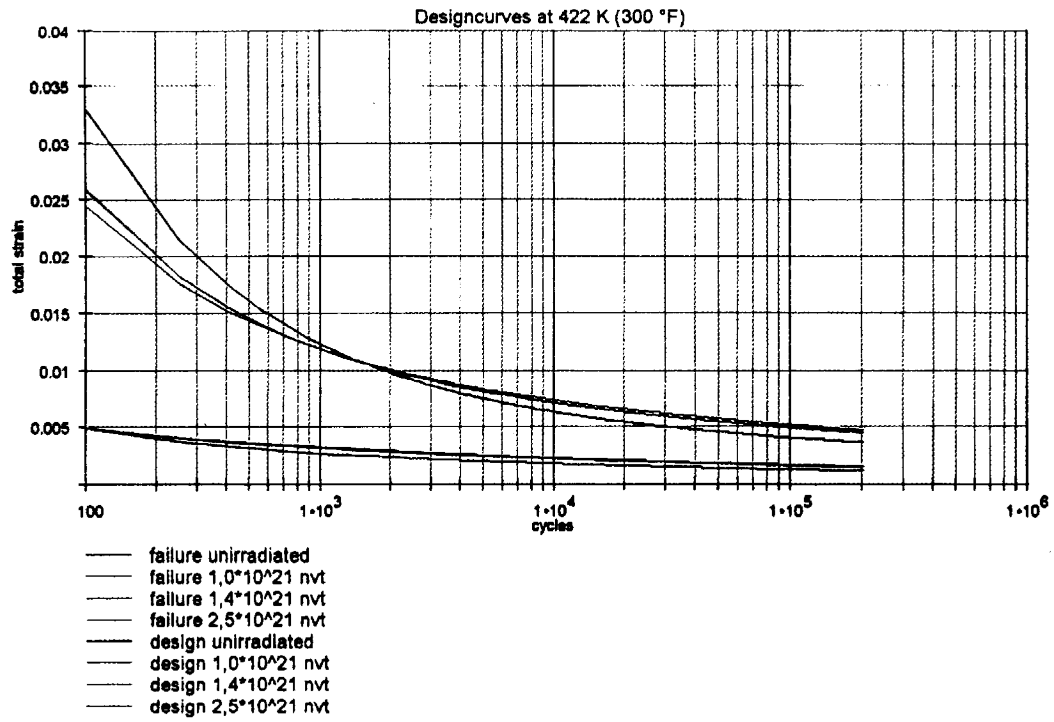


Figure 9

References

[1] Material specification
WSD 8200.1

[2] Dr. E. Gutsmiedl, A. Scheuer
Material test sheets for Zircaloy 4 plates
WBD 8200.1, Rev. 1 of 19.08.1998 with amendment of 30.09.1998

[3] Dr. E. Gutsmiedl, A. Scheuer
Material test sheets for Zircaloy 4 forgings
WBD 8200.3, Rev. 1 of 19.08.1998 with amendment of 30.09.1998

[4] ASTM B 352-92
Standard specification for Zirconium and Zirconium Alloy Sheets, Strip and Plate for Nuclear Application

/5/ ASTM B 351-92
Standard specification for Hot-Rolled and Cold finished Zirconium and Zirconium Alloy Bars, Rod and Wire for Nuclear Application

[6] R.G. Hoagland, A.L. Bement, R.G. Rowe
Application of Fracture Mechanics in Evaluation Initiation and Propagation of Brittle Fracture in Reactor Structural Components

[7] P.H. Kreyns, W.F. Bourgeois, C.J. White, P.L.Charpenter, B.F. Kammenzind and D.G. Franklin
Embrittlement of Reactor Core Materials