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

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**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 examined in the temperature range between  $-256\text{ }^{\circ}\text{C}$  and  $250\text{ }^{\circ}\text{C}$ . 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  $-256\text{ }^{\circ}\text{C}$  and  $150\text{ }^{\circ}\text{C}$  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  $\sim 1 \cdot 10^{22}$  n/cm<sup>2</sup> 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/cm<sup>2</sup>.

## 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)

$$\varnothing_{a1} = 289 \text{ mm} \quad \varnothing_{i1} = 283 \text{ mm} \quad s_1 = 3 \text{ mm} \quad l_1 = 709 \text{ mm}$$

inpile part (outer tubes)

$$\varnothing_{a2} = 74 \text{ mm} \quad \varnothing_{i2} = 68 \text{ mm} \quad s_2 = 3 \text{ mm} \quad l_2 = 1440 \text{ mm}$$

$$\varnothing_{a3} = 86 \text{ mm} \quad \varnothing_{i3} = 80 \text{ mm} \quad s_3 = 3 \text{ mm} \quad l_3 = 3282 \text{ mm}$$

inpile part (inner vessel)

$$\varnothing_{a4} = 283 \text{ mm} \quad \varnothing_{i4} = 277 \text{ mm} \quad s_4 = 3 \text{ mm} \quad l_4 = 720 \text{ mm}$$

inpile part (inner tubes)

$$\varnothing_{a5} = 66 \text{ mm} \quad \varnothing_{i5} = 60 \text{ mm} \quad s_5 = 3 \text{ mm} \quad l_5 = 4806 \text{ mm}$$

### - cold source (vacuum vessel)

bottom part

$$\varnothing_a = 319,5 \text{ mm} \quad \varnothing_i = 311,5 \text{ mm} \quad s = 4 \text{ mm} \quad l = 503 \text{ mm}$$

top part

$$\varnothing_{a1} = 140 \text{ mm} \quad \varnothing_i = 134 \text{ mm} \quad s = 3 \text{ mm} \quad l = 1476 \text{ mm}$$

$$\varnothing_{a2} = 230 \text{ mm} \quad \varnothing_i = 224 \text{ mm} \quad s = 3 \text{ mm} \quad l = 286 \text{ mm}$$

$$\varnothing_{a3} = 402 \text{ mm} \quad \varnothing_i = 394 \text{ mm} \quad s = 4 \text{ mm} \quad l = 892 \text{ mm}$$

$$\varnothing_{a4} = 540 \text{ mm} \quad (\text{flange part}) \quad l = 80 \text{ mm}$$

### - beam tube

$$\varnothing_{a1} = 265 \text{ mm} \quad \varnothing_{i1} = 255 \text{ mm} \quad s = 5 \text{ mm} \quad l = 1030 \text{ mm}$$

$$\varnothing_{a2} = 163 \text{ mm} \quad \varnothing_{i2} = 155 \text{ mm} \quad s = 4 \text{ mm} \quad l = 1508 \text{ mm}$$

$$\varnothing_{a3} = 270 \text{ mm} \quad (\text{flange part}) \quad l = 90 \text{ mm}$$

$$\varnothing_{a4} = 400 \text{ mm} \quad (\text{flange part}) \quad l = 135 \text{ mm}$$

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,  
 $\varnothing_a = 550 \text{ mm}$ ,  $\varnothing_i = 384 \text{ mm}$  for rings and  $\varnothing_a = 125 \text{ mm}$  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 tangential 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/cm<sup>2</sup>.

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

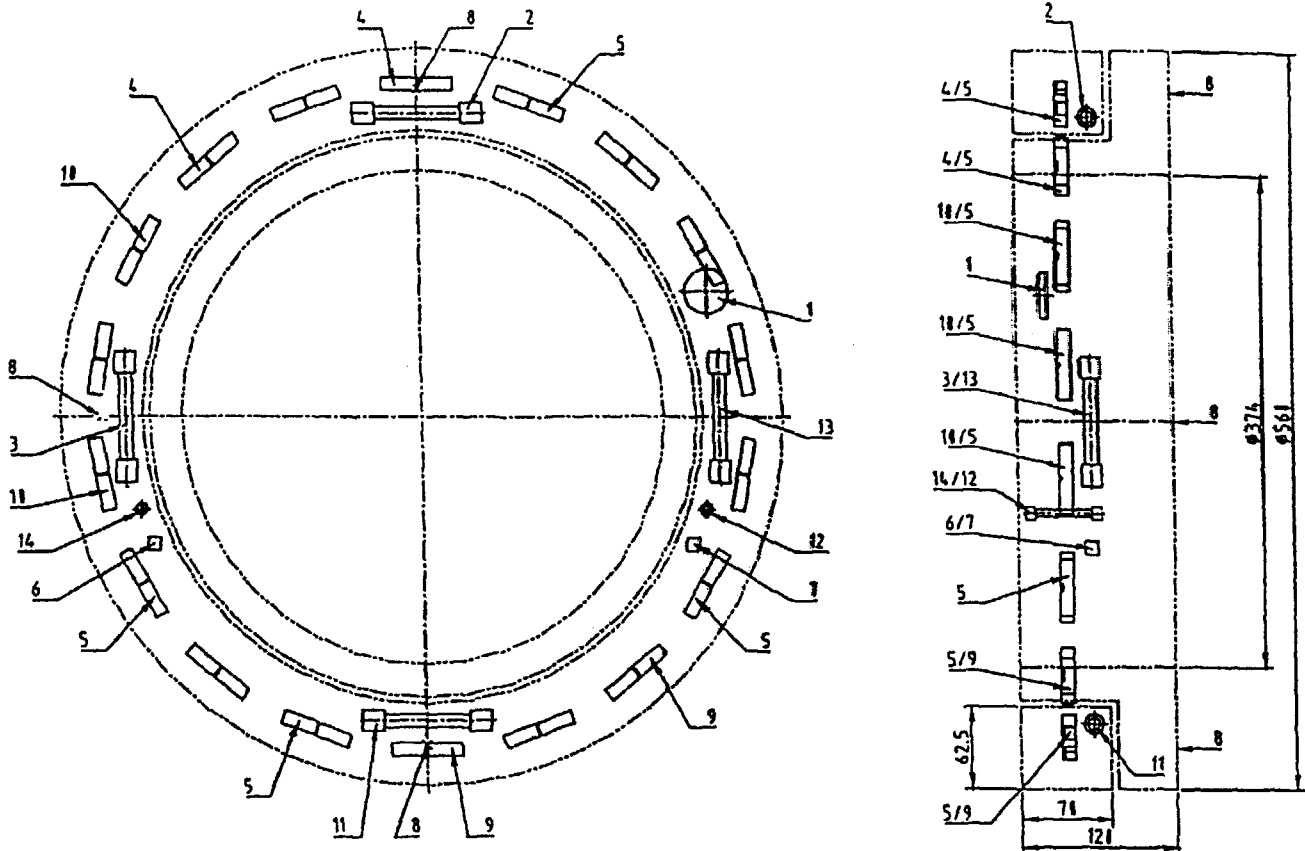
- 2 transverse tensile test at room temperature at each end of each master strip
- 2 transverse bend test at each of each master strip
- 1 longitudinal + 1 transverse tensile test at 288 °C at one end of each master strip

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 tangential 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.



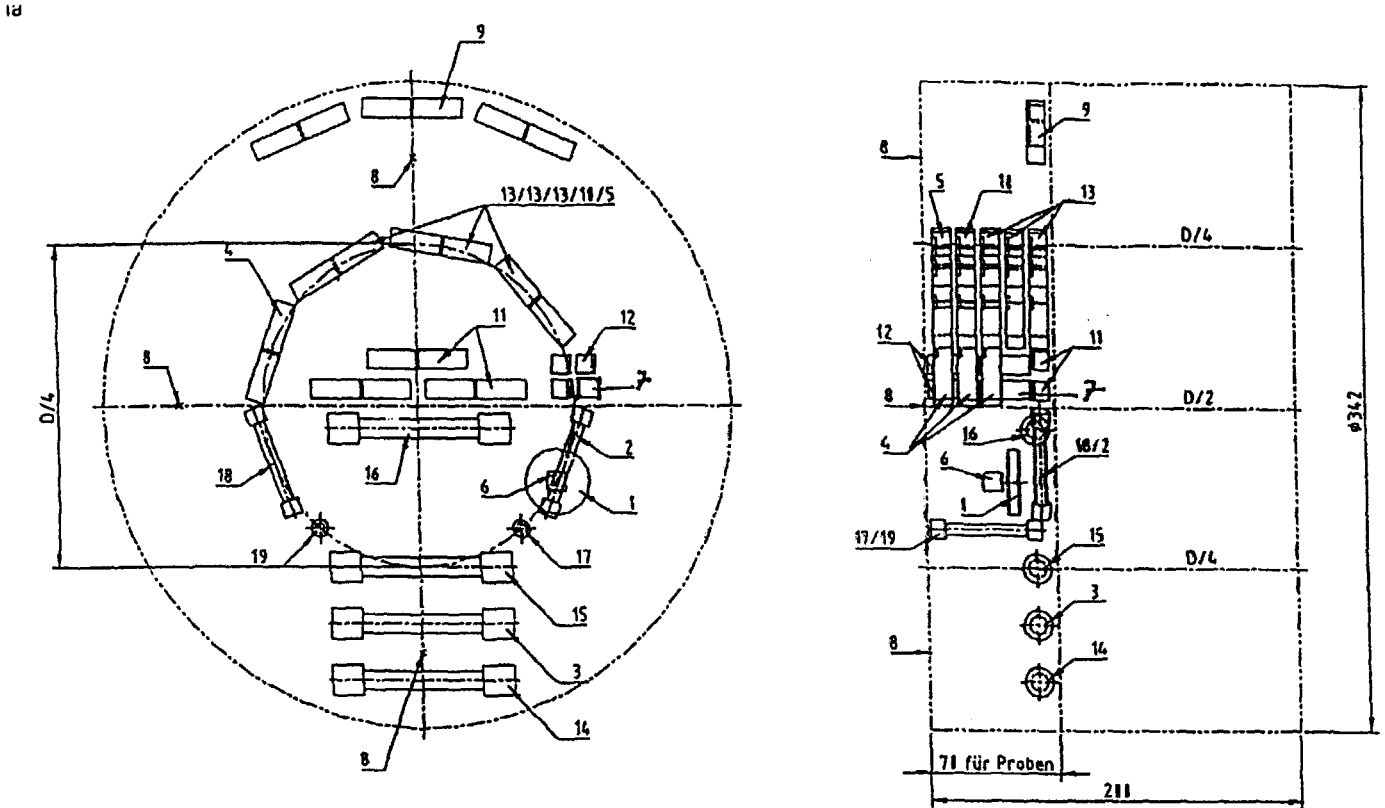
**Samples corresponding material test sheet**

- 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  $-196^{\circ}\text{C}$ ,  $-20^{\circ}\text{C}$ ,  $+150^{\circ}\text{C}$
- 6. Corrosion test
- 7. Metallographic test
- 8. Hardness test 3 x

**Additional Samples corresponding expert approval**

- 9. Charpy-V test at  $180^{\circ}$
- 10. Charpy-V test at  $-256^{\circ}\text{C}$
- 11. Tensile test, RT
- 12. Tensile test, axial
- 13. Tensile test, tangential at  $288^{\circ}\text{C}$
- 14. Tensile test, axial at  $288^{\circ}\text{C}$

**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 manufacturer 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)	29	9	1220	1.32	0.21	0.11	0.32
Strip Nr. 21.1	25	9	1210	1.31	0.21	0.11	0.32
Strip Nr. 21.2							
Results (4 mm)	28	9	1210	1.34	0.21	0.10	0.31
Strip Nr. 11.1	30	10	1180	1.32	0.21	0.10	0.31
Strip Nr. 11.2							
Results (5 mm)	23	9	1200	1.32	0.21	0.10	0.31
Strip Nr. 12.1	25	13	1220	1.32	0.21	0.10	0.31
Strip Nr. 12.2							
Results (10 mm)	29	9	1190	1.54	0.21	0.11	0.32
Strip Nr. 35.1	27	9	1220	1.53	0.21	0.11	0.32
Strip Nr. 36.1							

### 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

Direction	Ultimate Tensile (N/mm <sup>2</sup> )		0,2 % Yield Strength (N/mm <sup>2</sup> )		Elongation (%)		Area reduction when breaking (%)	
	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

Direction	Ultimate Tensile (N/mm <sup>2</sup> )		0,2 % Yield Strength (N/mm <sup>2</sup> )		Elongation (%)		Area reduction when breaking (%)	
	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

Direction	Ultimate Tensile (N/mm <sup>2</sup> )		0,2 % Yield Strength (N/mm <sup>2</sup> )		Elongation (%)		Area reduction when breaking (%)	
	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

Direction	Ultimate Tensile (N/mm <sup>2</sup> )	0,2 % Yield Strength (N/mm <sup>2</sup> )	Elongation (%)	Area reduction when breaking (%)
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

Direction	Ultimate Tensile (N/mm <sup>2</sup> )		0,2 % Yield Strength (N/mm <sup>2</sup> )		Elongation (%)		Area reduction when breaking (%)	
	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/cm<sup>2</sup>. 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/cm<sup>2</sup> and is higher than the aimed value of 24 J/cm<sup>2</sup>. 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/cm <sup>2</sup>
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	$\leq 98$ 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/dm<sup>2</sup> (3 mm plates)
- 19.5, 18.5 mg/dm<sup>2</sup> (4 mm plates)
- 18.4, 18.3 mg/dm<sup>2</sup> (5 mm plates)
- 19.6, 19.8 mg/dm<sup>2</sup> (10 mm plates)

and fulfilled the specified gain of  $\leq 22$  mg/dm<sup>2</sup>.

#### **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 continuous 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/mm <sup>2</sup>	0,2 Yield Strength N/mm <sup>2</sup>	Elongation %
RT	Transverse Tangential	413	241	14
RT	Longitudinal	413	241	14
316	Longitudinal	214	103	24
316	Transverse Tangential	to evaluate in the approval		

**Table 7:** 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/mm <sup>2</sup>	0,2 Yield Strength N/mm <sup>2</sup>
130	338	190
150	325	180
180	305	166
250	258	133
288	233	115

**Table 8:** 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 tangential 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/mm<sup>2</sup> 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 tangential 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	

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



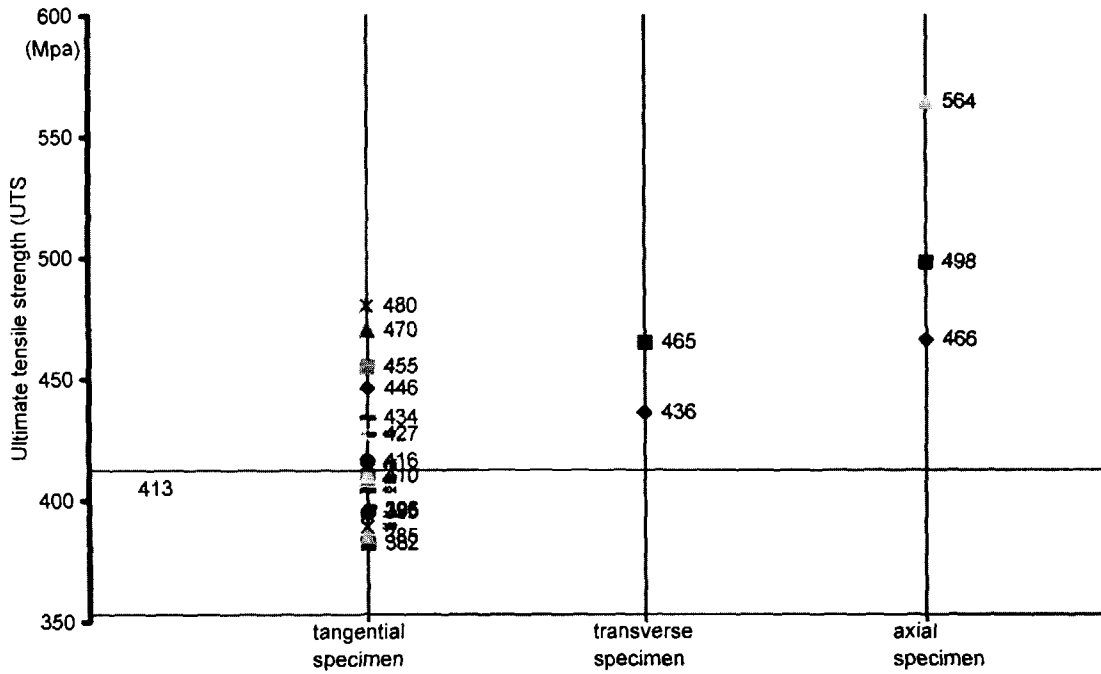


Figure 3: Ultimate tensile test (UTS) at RT for the tangential, transverse and axial direction of forgings

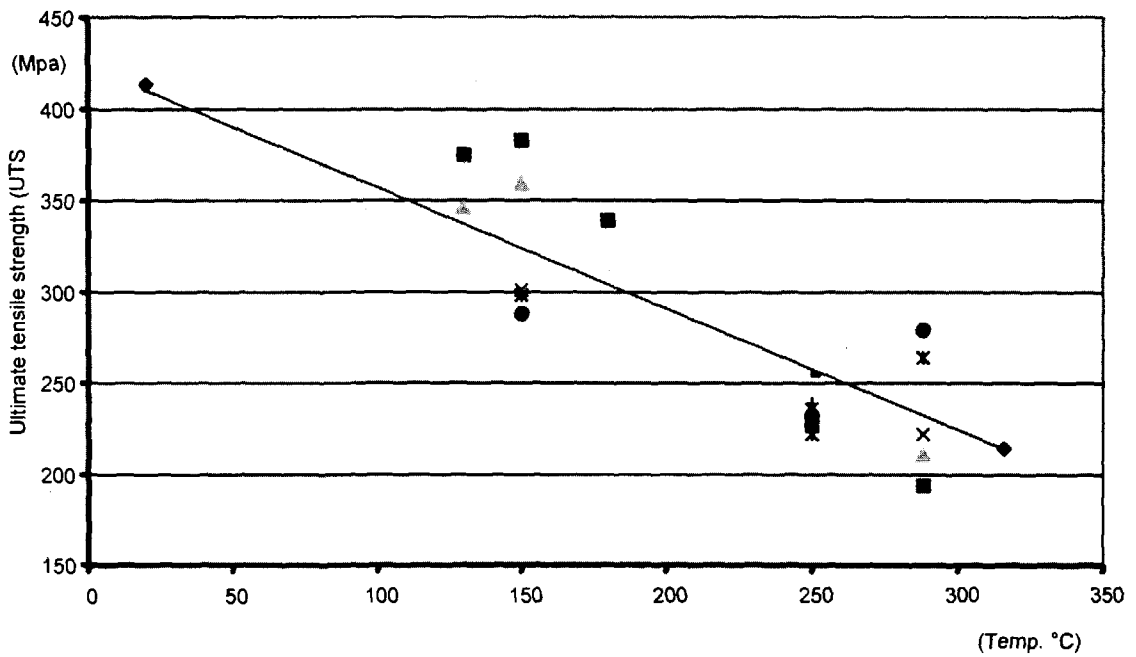


Figure 4: Ultimate tensile strength (UTS) of Zircaloy 4 forgings versus temperature

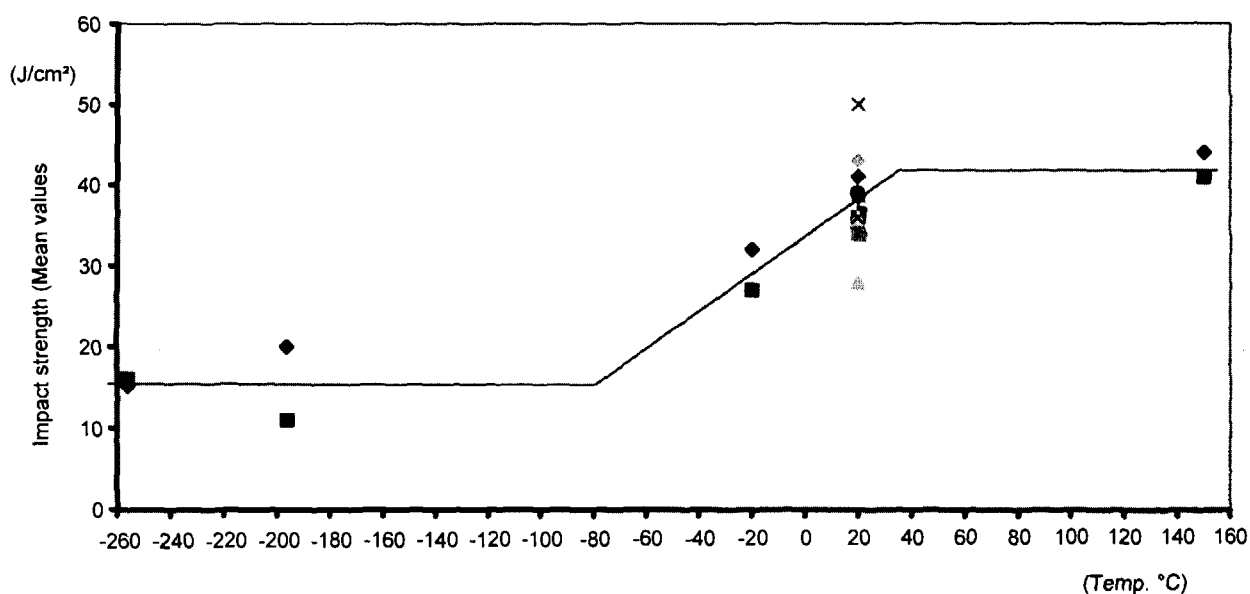
### 5.3 Impact strength

The results of the impact strength fulfill the aimed value of 24 J/cm<sup>2</sup> 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/cm <sup>2</sup>
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/dm<sup>2</sup> < 22 mg/dm<sup>2</sup> 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  $S_m$  ( $S_m = \text{Min} (UTS_{RT}/3; 0,2 YST/1,5)$ ). So the deviation of  $-7,5\%$  has to take in consideration for calculating the  $S_m$ -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/cm<sup>2</sup> (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  $\sim 5\%$  and a percentage elongation before reduction of  $\sim 1\%$ . As an additional criteria the fracture toughness of the Zircaloy-4 was studied up to the fluence of  $1 \cdot 10^{22}$  n/cm<sup>2</sup>. These data were taken 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  $-20\text{ }^\circ\text{C}$  to  $150\text{ }^\circ\text{C}$ . For the fracture toughness value  $K_{IC}$  of irradiated material (fluence of  $1,5 \cdot 10^{22}$  n/cm<sup>2</sup>) the following values were used for the annealed material condition [6], [7]:

$$K_{IC} = 45 \text{ MPa } \sqrt{\text{m}} \text{ for } 100\text{ }^\circ\text{C}$$

$$K_{IC} = 25 \text{ MPa } \sqrt{\text{m}} \text{ for } -20\text{ }^\circ\text{C to } -180\text{ }^\circ\text{C}$$

For the weld lower values were used

$$K_{IC} = 30 \text{ MPa } \sqrt{\text{m}} \text{ for } 100\text{ }^\circ\text{C}$$

$$K_{IC} = 20 \text{ MPa } \sqrt{\text{m}} \text{ for } -20\text{ }^\circ\text{C to } -180\text{ }^\circ\text{C}$$

The fracture mechanic calculation shows with a postulated crack at the surface of 0,4 mm deepness and 20 mm length and the stress level 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.

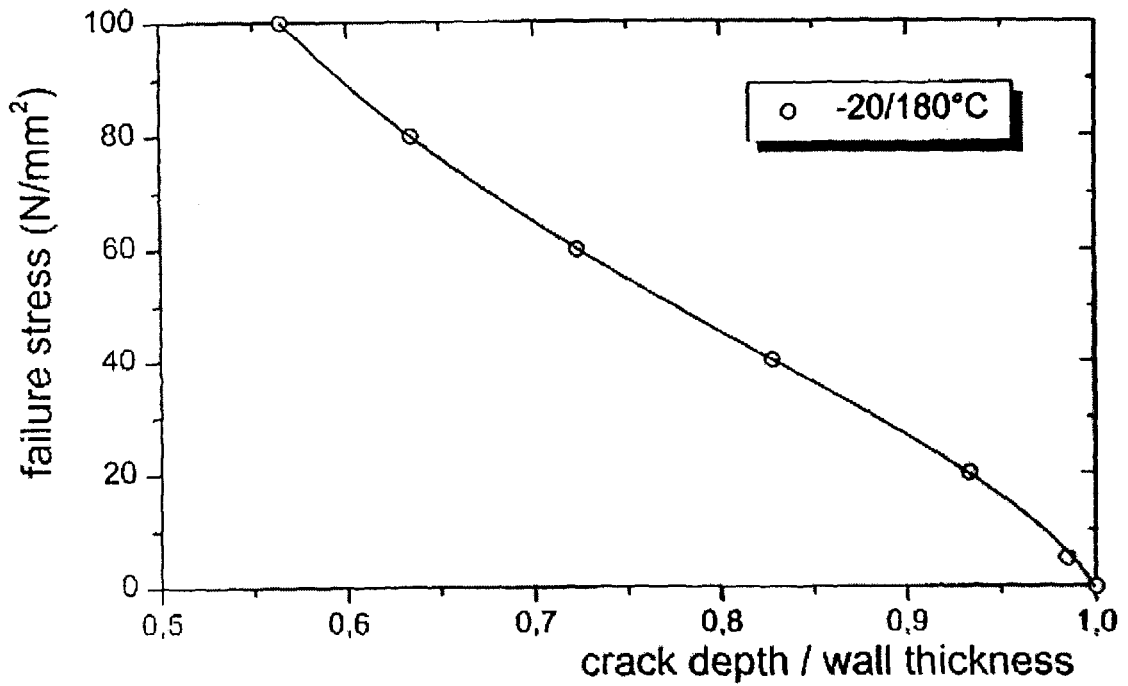


Figure 6: Failure stresses of vacuum vessel of COLD NEUTRON SOURCE (length of surface crack 20 mm, wall thickness 4 mm)

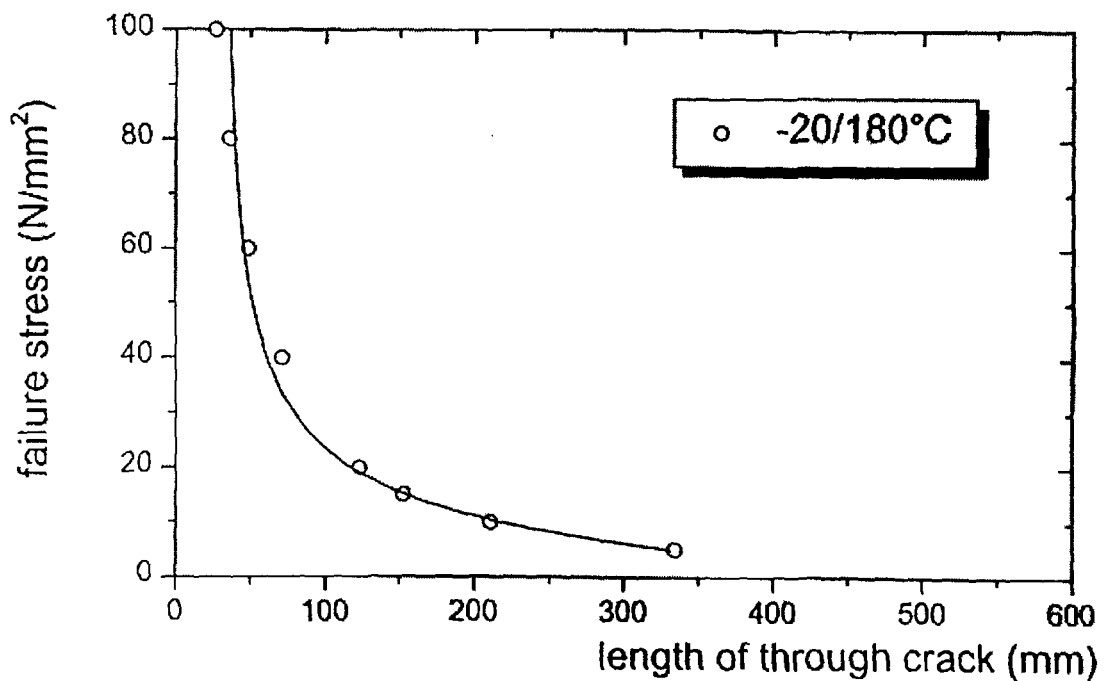


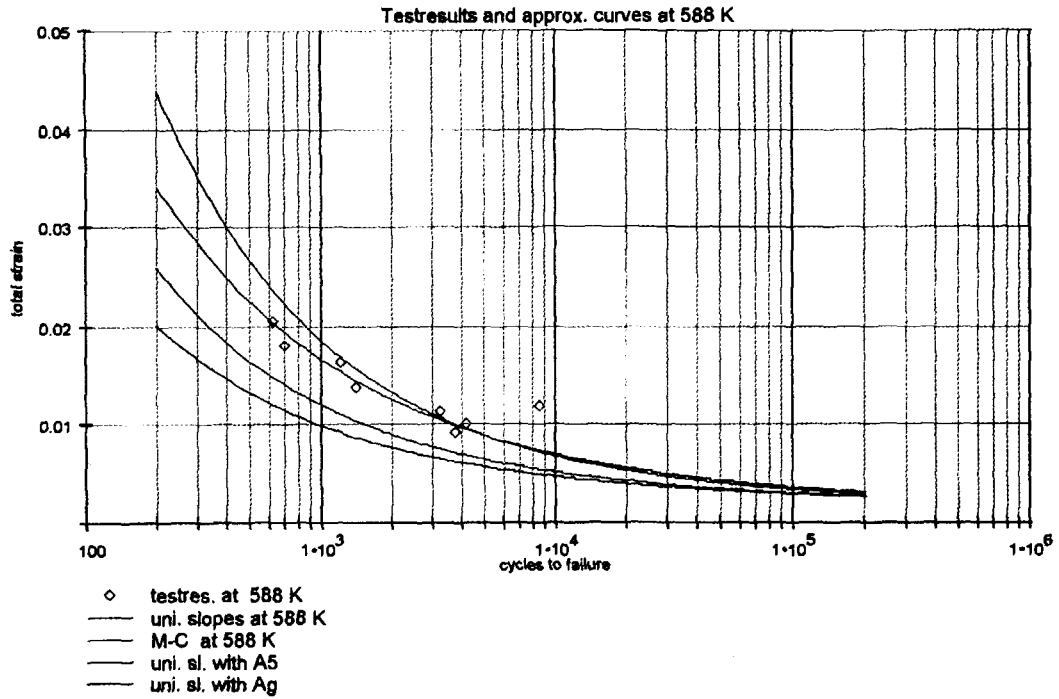
Figure 7: Critical length of through crack of vacuum vessel of COLD NEUTRON SOURCE (wall thickness 4 mm, welding)

## 9. Fatigue curves for Zircaloy 4

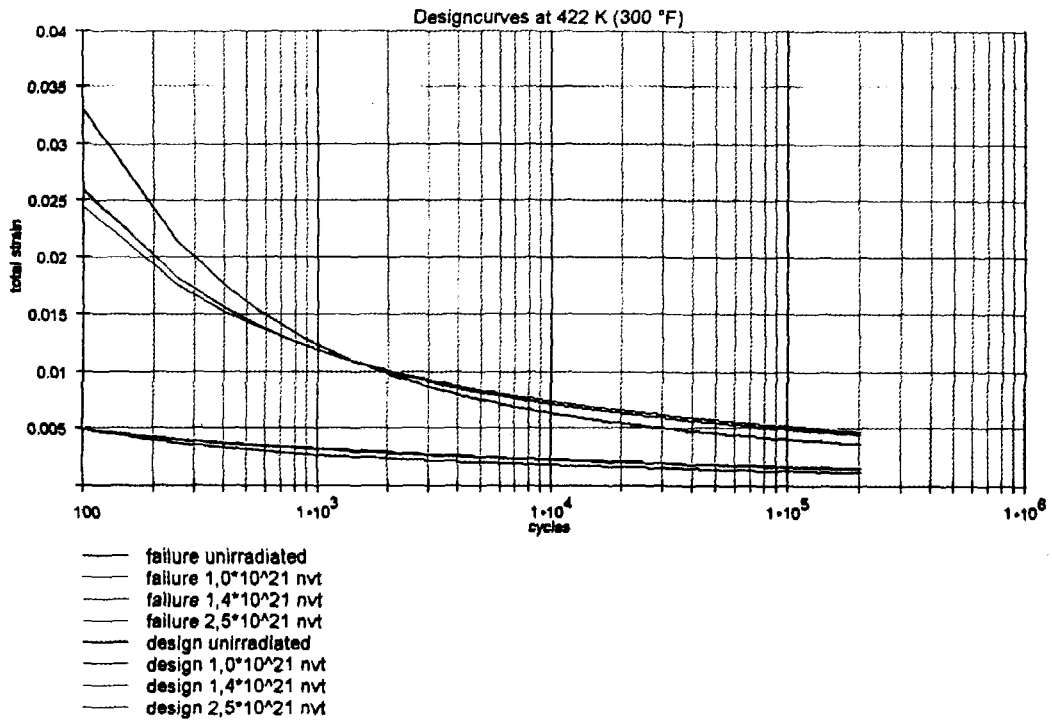
Low cycle fatigue design curves for the unirradiated material Zircaloy 4 were calculated with the method of the Universal Slope of Manson. The material test results were taken into consideration. On the basis of these results the expected fatigue design curves for irradiated Zircaloy-4 were evaluated, see figure 8, 9. These low cycle fatigue design curves are the basis 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/cm<sup>2</sup>.



**Figure 8**



**Figure 9**

## References

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