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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
Results of forming the material and welding processes

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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.

For fabrication of the vessels and piping parts it was necessary to form the base material and calibrate the sheets or welded parts with necessary heat treatments. Additional to the technical specifications preliminary material investigations and production test of welded and unwelded material were carried out of the formed parts up to a cold work of 5 %.

Further one with respect to the material thickness of 3, 4, 5 and 10 mm of the used sheets, welding procedure test before the fabrication and welding production tests during fabrication were carried out of the base material combination sheet/sheet and sheet/forging.

Electronic beam welding was used for the welding process.

Material tests as tensile tests, charpy-V-tests, bend tests, metallographic tests, hardness tests, radiographic tests a.s.o. were carried out.
The results of the examinations confirm the specified requirements. For the material forming process an optimization was necessary after the preliminary results to get final sufficient material behaviour results.

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.

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 Chemical analysis at the ingot and the semi-finished products, Tensile Tests at RT and Designtemperature, Charpy-V-Tests at RT, Charpy-V-Tests in the temperature range between –256 °C and +150 °C, Bend test for plates, Corrosion tests, Metallographic Tests (Type of Microstructure, Cleanliness, Grain Size), Hardness Tests, Ultrasonic Tests, Dimensional Control, Roughness Tests, Visual Inspection were specified in the material test sheet.

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:

- transverse tensile test at room temperature at each end of each master strip
- transverse bend test at each of each master strip
- longitudinal + transverse tensile tests at RT and 288 °C at one 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.

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.

Test results of the sheets and forgings are presented in /6/.

4. Fabrication Process

For the bottom and the topconstruction and for the flanges of the vessels of the HNS, CNS and the beam tube No. 6 forging material was used; for the pipe parts sheet material was formed to pipes. The pipe diameter were 66 mm up to 402 mm by a sheet thickness of 3,4 and 5 mm. The transformation degree of the sheets was between 1 % and 4,8 %.

After the forming process before welding a heat treatment on 530 °C, 60 min was done.

After the welding process a calibration of the pipe parts was necessary. For the cylindrical vessel parts of the HNS, fabricated from 10 mm sheets additional heat treatment at 630 °C/ 40 min was done.

For the first forming process up to 4.8 %, the calibration process and following heat treatment material investigations for cold work of the base material between 4 %, 5 %, 6 % and for the welds between 2 and 8 % were carried out, see chapter 7.
For the welding process welding procedure tests before the fabrication and welding production tests during the fabrication were carried out, see chapter 6.
Repairs in welds were take into account for 3, 4 and 5 mm thickness.

5. Welding procedure

For welding procedure electronic beam welding (unit 30 KW) was used. The welding take place in a vacuum chamber, the process vacuum in the vacuum chamber was $< 1 \cdot 10^{-4}$ mbar. The welding parameter are recorded.
For the weld shape butt joints were selected, for the final welds beamstopper are foreseen.
No auxiliary materials are used for the welding process, the welding position for all welds are horizontal.

6. Results of welding procedure and welding production tests

The results of the welding procedure and welding production tests are listed in table 1, 2 and 3. The results of the surface hardness across the base material, heat affected zone and the weld material shows no great increase in hardness in the weld region.
The results of the tensile tests are above the specified value for the base material.

7. Material investigation

Before the forming process of the sheets preliminary material investigation up to a cold work of 5 % and a following heat treatment at 530 °C were carried out.
These tests were done for the base material and for the welds. The results are listed up in table 4 and shows that the increase of the yield strength and tensile strength is less if a heat treatment after the forming process occur.

The tensile tests across the weld shows similiar behaviour.

Additional material investigation were done for the base material with the conditions cold work 5 %, heat treatment 530 °C, calibration with cold work of 4 % and 6 %. The same procedure was done for the longitudinal weld with a cold work of 2 %, 4 %, 6 %, 8 %. The results are listed up in table 5.

A second heat treatment with 530 °C or 630 °C and a third heat treatment of 630 °C was investigated for the weld region in the production welding tests, see AP 5/9, 5/10; 5/11 and 5/12 in table 1.

8. Summary

The results of the welding procedure tests before the fabrication and the welding production tests during the fabrication fulfil the requirements of the specifications. This includes the repairs of welding and the heat treatments during the fabrication process.

The investigations of the behaviour of cold work conditions on the mechanical behaviour under radiation is running at time.

No. of the VP/AP	Wall thickness (mm)	Heat treat-ment	$R_{P0,2}$ N/mm²	$R_{P1\%}$ N/mm²	R_m N/mm²	A %	Z %	Remarks
			> 303		> 386	> 25		RT
VP 4/1 AP 5/1	3	-	429 451	469 471	508 510		60 60	RT RT
VP 4/2	10	-	362 353 680	428 421 799	541 536 911	 8,5	 8	RT RT –196 °C
AP 5/2	3	-	383 455	467 482	509 523		60 62	RT RT
AP 5/4	4	-	436 437	463 461	498 485		60 60	RT RT
AP 5/5	4	-	392 391 638	466 457 703	539 547 779		51 51 8	RT RT –196 °C
AP 5/6	4	-	401	475	537		51	RT, Beam stopper
AP 5/7	5	-	355 350	426 422	510 504	56/40	32 48	RT RT
AP 5/12	10	530 °C/ 630 °C/ 630 °C	489 227	508 243	529 295	24/25,5	55 70	RT Sheet 250 °C Sheet
	10	630 °C	436 199	454 209	559 334	27,6 35,5	51 64	RT Forging 250 °C Forging
	10	530 °C/ 630 °C/ 630 °C	488	507	530	-	51	RT Weld Sheet
	10	530 °C/ 630 °C/ 630 °C	470	489	531	-	56	RT Weld Sheet/ Forging
AP 5/9	4	530 °C/ 530 °C	331 190	417 230	511 321	27 39,5	52 67	RT 250 °C
AP 5/10	5	530 °C 530 °C	359 200	451 241	530 324	39/33 -	38 45	RT 250 °C
AP 5/11	10	530 °C/ 530 °C	472 246	480 264	540 328	21 33	58 70	RT 250 °C

Table 1: Results of the tensile strength of the welding procedure tests and production welding tests

Strip No.	Thickness	Specimen direction	Testtemp. °C	Impact strength J/cm²	Mean value J/cm²	Remark
103 – 105 VP 4/2	10	Transv.	RT	21/21/21	21	
114 – 116 VP 4/2	10	Transv.	RT	21/20/23	21	Repair
106 – 108 VP 4/2	10	Transv.	-255	13/13/10	12	
109 – 111 VP 4/2	10	Transv.	150	41/48/48	46	
513 – 515 AP 5/11	10	Long.	RT	107/112/111	110	BM
4.1.1 - 4.1.3	10	Transv.	RT	63/63/59	62	BM
4.2.1 – 4.2.3	10	Transv.	RT	71/91/94	85	BM
4.3.1 – 4.3.3 AP 5/12	10	Transv.	RT	29/29/26	28	Weld

Table 2: Results of Charpy-V-tests (10 mm sheet) of the welding procedure test and production welding tests

Loca-tion	AP 5/12 10 mm	VP 4/1 AP 5/1 3 mm	AP 5/4 4 mm	VP 4/2 AP 5/3 10 mm	VP 4/2 AP 5/3 10 mm Repair	AP 5/2 3 mm	AP 5/5 4 mm	AP 5/6 4 mm	AP 5/7 5 mm	VP 4/1 Repair
BM	286 193 286 195	203 198	205 189	193 180 197 191 173	179 179 180 193 196	188 188	177 162	148 148	194 194	210 201
HEZ	254 190 249 204	192 183 201 202 194	194 202 203 177 182	192 183 183	191 188 181	188 193	168 165 175 178 185	175 175 165 168 168	186 181 191 186 188	197 201 201
WM	229 197 236 214	197 173	188 -	170 187 182	206 193 189	196 183	178 165	158 158	179 159	205 175 185
HEZ	241 236 241 246	191 182 197 202 201	183 192 199 201 203	177 181 197	181 187 213	181 198 189	164 219 236 195 262	206 197 208 185 197	179 186 183 193 197	210 193 193
BM	232 204 246 239	203 203	196 202	193 194 203	194 192 197	186 192	232 225	192 206	197 197	205 210

BM = Base Material
HEZ = Heat Affected Zone
WM = Weld Material

Table 3: Surface hardness across the Base Material, Heat Affected Zone and Weld Material (HV 10) of the welding tests and production welding tests

Sample No.	Cold work	Heat treat-ment	$R_{P0,2}$ N/mm²	$R_{P1\%}$ N/mm²	R_m N/mm²	A %	Z %	Remarks
Base Material (BM) 3 mm								
1	5 %	yes	442	463	509	33	61	RT, transv.
2	5 %	yes	438	464	510	31,5	59	RT, transv.
3	5 %	yes	229	249	289	46,5	75	250 °C, transv.
4	5 %	yes	371	437	528	31,5	55	RT, longit.
5	5 %	yes	373	441	532	31,5	55	RT, longit.
6	5 %	yes	196	240	335	44,0	70	250 °C, longit.
9A	5 %	no	483	498	524	26,5	61	RT
10A	5 %	no	517	519	429	22,5	60	RT
11A	5 %	no	484	518	530	24,0	58	RT
12A	5 %	no	520	518	532	27,0	60	RT
Weld Material (WM)								
3	5% BM	yes, only BM	222	248	290	-	75	250 °C, transv.
4	5% BM	yes, only BM	190	240	316	-	68	250 °C, longit.
5	5% BM	yes, only BM	422	469	516	-	52	RT, transv.
6	5% BM	yes, only BM	374	445	533	-	54	RT, longit.
10	5% BM	no	253	277	307	-	75	250 °C, longit.
11	5% BM	no	444	495	525	-	55	RT, longit.
12	5% BM	no	452	503	530	-	61	RT, longit.

BM = Base Material
WM = Weld Material

Table 4: Results of material investigation of the sheet forming of Zircaloy 4

Sample No.	Cold work	Heat treat-ment	$R_{P0,2}$ N/mm²	$R_{P1\%}$ N/mm²	R_m N/mm²	A %	Z %	Remarks
Base Material (BM) 3 mm								
1	4 %	yes	532	544	548	26	56	RT, transv.
2	4 %	yes	280	289	302	43,5	66	250 °C, transv.
3	6 %	yes	527	547	552	25	55	RT, transv.
4	6 %	yes	284	292	304	40,5	68	250 °C, transv..
Weld Material (WM)								
5	2 %BM	-	467	482	515		59	RT, transv..
6	2% BM	-	267	278	305	-	68	250 °C, transv.
7	4% BM	-	474	500	527	-	57	RT, transv.
8	4% BM	-	253	264	292	-	70	250 °C, transv.
9	6% BM	-	503	513	526	-	56	RT, transv.
10	6% BM	-	281	285	304	-	71	250 °C, transv.
11	8% BM	-	516	531	543	-	57	RT, transv.
12	8% BM	-	280	283	302	-	69	250 °C, transv.

BM = Base Material
WM = Weld Material

Table 5: Results of material investigation of cold work of the base material and weld of Zircaloy 4

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] Dr. E. Gutsmiedl, A. Scheuer
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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[7] Specification
Hot Neutron Source, KSD 8200.1

[8] Specification
Cold Neutron Source, KSD 8100.1