

RESULTS FROM POST-MORTEM TESTS WITH MATERIAL FROM THE OLD CORE-BOX  
OF THE HIGH FLUX REACTOR (HFR) AT PETTEN

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**ABSTRACT**

Results are reported from hardness measurements, tensile tests and fracture mechanics experiments (fatigue crack growth and fracture toughness) on 5154 aluminium specimens, fabricated from remnants of the old HFR core box. The specimen material was exposed to a maximum thermal neutron fluence of  $7.5 * 10^{26} \text{ n/m}^2$  ( $E < 0.4\text{eV}$ ).

Test results for this fluence (ratio of the thermal to fast neutron flux density is 1.17) are: hardness 63HR15N, 0.2 - yield strength 525 MPa and total elongation 2.2% strain. Material which was exposed to a lower thermal fluence of  $5.6 * 10^{26} \text{ n/m}^2$ , but with a thermal to fast neutron ratio of about 4, shows more radiation hardening : 67HR15N, 0.2 - yield strength 580 MPa and 1.5% total elongation.

Fatigue crack growth rates range from  $5 * 10^{-5} \text{ mm/cycle}$  to  $10^{-3} \text{ mm/cycle}$  for  $\Delta K$  ranging from 8 to 20  $\text{MPa}\sqrt{\text{m}}$ . The most highly exposed ( $7.5 * 10^{26} \text{ n/m}^2$ ) material shows accelerated fatigue crack growth due to unstable crack extension at  $\Delta K$  of about 15  $\text{MPa}\sqrt{\text{m}}$ . The lowermost meaningful measure of plane strain fracture toughness is 18  $\text{MPa}\sqrt{\text{m}}$ .

Except for the fracture toughness which is a factor of about 3 higher the results show reasonable agreement with the expected mechanical properties estimated in the "safe end-of-life" assessment of the old HFR vessel.

## INTRODUCTION

The HFR reactor vessel was replaced in 1984 after more than 20 years of operation because doubts had arisen over the condition of the aluminium alloy construction material (ASTM 5154). Data from destructive testing of the old vessel material are used in support of the design analysis for the new vessel which is constructed from the same material.

## EXPERIMENTAL

Material, original condition.

The construction material of the old HFR vessel was a modified British aluminium-magnesium alloy, designated BS1477 NP5/6M (modified : 3.5-3.9% Mg). The chemical analysis of the fabrication cast is given in Table 1. The composition is close to the specification for type 5154 aluminium alloy.

Table 1. Chemical composition of the vessel material (wt%)

	Mg	Si	Fe	Mn	Cu	Zn
old vessel	3.7	0.14	0.38	0.32	0.02	0.03
5154 alloy	3.1-3.9	- 0.45 -		0.10	0.10	0.20

Room temperature (300 K) tensile properties are : 0.2 - yield stress 100 MPa, UTS 250 MPa, uniform elongation 20% strain and total elongation 25% strain. The data illustrate the fully annealed "Soft" condition of the material, similar to the 5154-0 condition.

Material, end-of-life condition.

The most highly exposed segments of the old reactor vessel, the centre parts of the core box, have been selected for post-mortem testing.

The walls have been exposed to neutron radiation for a period of about  $4.24 \times 10^8$  s, ending November, 1983. The accumulated neutron fluences, calculated for the mid-centre positions, are listed in Table 2.

Table 2. Estimated neutron fluences of the core-box walls, calculated for the mid-centre positions (ref. 3).

fluence, $10^{26} \text{ n/m}^2$	Northwall	Eastwall	Southwall	Westwall
thermal ( $E < 0.414 \text{ eV}$ )	5.6	3.2	5.0	7.5
fast ( $E > 0.1 \text{ MeV}$ )	1.7	0.8	1.9	6.9
ratio thermal/fast	3.3	4.0	2.6	1.1

After the destructive removal of the old vessel, the core box walls were preserved for further segmentation and preparation of tensile specimens and compact tension specimens. Further material remains available for bend test specimens and fracture toughness specimens.

All specimens were machined from mid-thickness ( $\frac{1}{2}T$ ) material by equally reducing both surfaces, except for the tensile samples from the West wall which have been produced from quarter thickness ( $\frac{1}{4}T$ ,  $\frac{3}{4}T$ ) material.

#### Testing.

The post-mortem testing encompassed Rockwell hardness measurements, tension testing, fatigue crack growth testing and fracture resistance measurements. All tests were performed in air at room temperature.

Superficial Rockwell hardness measurements were made on the mill-cut side-surfaces of the blocks, prior to the final machining of the specimens.

The tests were performed according to the ASTM standard method E 18 using the 15N scale with 150N total load applied by the diamond cone indenter and a load removal time of 8s.

Tensile tests were performed according to the ASTM standard test method E 8. The specimens were pin-loaded and tested at constant strain rate of  $5 * 10^{-4} \text{ s}^{-1}$ .

The fatigue crack growth tests were performed under constant-amplitude cyclic loading (triangular wave form) with R-ratio of 0.1 (ratio of the peak values of the applied loading cycle). The mechanical notch (crack starter) length

was 12.5 mm ( $a/W = 0.25$ ). The pre-crack extension length was 2 mm. Pre-crack fatigue loading was performed at a cyclic frequency of 20 Hz, crack growth testing was performed at 10 Hz. The target final crack length was 32 mm ( $a/W = 0.64$ ). The  $\Delta K$ -values ranged from about 8 MPa $\sqrt{m}$  to 25 MPa $\sqrt{m}$ . The tests were performed according to the ASTM test method E 647 for constant-load-amplitude fatigue crack growth rates above  $10^{-8}$  m/cycle. Crack extension was measured by means of the direct current potential drop technique.

After termination of the fatigue crack growth tests the specimens were fracture-loaded by means of displacement controlled monotonic loading until separation. The applied displacement and the corresponding load were recorded to derive a measure of the fracture toughness of the specimens. This fracture-loading was performed after having achieved a certain fatigue crack length which was less or equal to the target final fatigue crack length of 32 mm. The fracture resistance measurements were performed basically according to the ASTM E 399 standard practice for plane-strain fracture toughness ( $K_{IC}$ ) measurements, however the results were analyzed according to the standard practice ASTM B645 for fracture toughness testing of aluminium alloys.

Post-test examinations consisted of optical microscopy, fractography, transmission electron microscopy and microprobe X-ray analyses to measure the Si-content of selected samples. The silicon is primarily created by the nuclear reactions of the thermal neutrons with the aluminium. The thermal neutron exposure of the samples is calculated from the Si-measurements, taking into account the original Si-content of the non-exposed material.

## RESULTS AND DISCUSSION

In this paper the accent is placed on the results from the fracture resistance measurements. The data, obtained from twenty pre-fatigue-loaded specimens, are reported in terms of the stress-intensity factor  $K_I$ , the linear-elastic fracture parameter being used in the recent defect analyses of the new HFR vessel (1). The data are combined with related information from the other experimental measurements. The full extent of all experimental results will be reported together with the metallographic observations at an appropriate symposium next year (2).

Two versions of the Compact-Tension type specimen were originally intended to be applied for the post-mortem testing of the core-box material. The dimensions of the specimens for the fatigue crack growth experiments are 62.5 mm width and 50.0 mm height. The thickness of these specimens is 10.0 mm or 12.5 mm. Because the dimensions of specimens for fracture toughness experiments can only be justified after completion of the experiment, a specimen with dimensions of 50.0 mm width and 48.0 mm height with the conservative thickness dimension of 17.0 mm was considered to be favourable to measure valid  $K_{IC}$ -values. However, due to budgetary restrictions the fatigue crack growth specimens were also used for the fracture resistance measurements. The specimens did meet the E399 thickness (B) requirement

$$B > 2.5 \left( \frac{K_Q}{\sigma_y} \right)^2$$

with B being 10.0 mm or 12.5 mm,  $K_Q$  the conditional fracture toughness and  $\sigma_y$  the tensile yield strength. One specimen from the East wall, with the lowest thermal neutron exposure and the highest  $K_Q$ -value, should have a thickness of about 13.0 mm, if a yield stress value of 420 MPa is assumed.

The twenty specimens were almost equally divided over the four walls of the old core-box. The selected positions represent about 50 to 90 % of the maximum neutron exposure of each wall. The measured Si-content ranges from 0.55 to 1.85 weight percentages. The thermal fluences, calculated from the Si-measurements, range from  $1.8 * 10^{26} \text{ n/m}^2$  to  $7.5 * 10^{26} \text{ n/m}^2$ . The estimated values of the thermal-to-fast-neutron flux density ratio range from the minimum value of 1.0 for a specimen from the west wall to a maximum value of 6.3 for a specimen from the South wall (3).

At shut-down of the old HFR reactor vessel in 1983, the maximum thermal neutron fluence at the center of the West wall has been estimated at  $7.5 * 10^{26} \text{ n/m}^2$  (4). From the Si-measurements this maximum thermal fluence is calculated to be  $8.3 * 10^{26} \text{ n/m}^2$  for material from the mid-center positions of the West wall. The underestimation of about 10 % is quite reasonable having regard for the uncertainties in the original calculations which had to take into account neutron history over a period of more than 20 years. Using the maximum thermal fluence of  $8.3 * 10^{26} \text{ n/m}^2$  as a reference, the twenty specimens cover a range of 20 % to 90 % of the maximum thermal neutron exposure.

Hardness measurements and tension tests showed irradiation hardening and an associated reduction of ductility. The Superficial Rockwell hardness number increased by a maximum of about 35 points  $HR_{15N}$ . The measured hardness numbers ranged from 51 to 67  $HR_{15N}$ .

The tension tests showed yield strength values ranging from 380 MPa to 590 MPa with corresponding Ultimate Tensile Strength (UTS) data ranging from 430 MPa to 610 MPa and total elongation values of 4.1 % strain to 1.5 % strain respectively. The data for the West wall (thermal to fast flux ratio between 1.0 and 1.5) fit very well with data from accelerated irradiations in the period 1970-1981 (same ratio of about 1.0) for the surveillance program of the old HFR vessel, reported by Lybrink (5).

In addition to the hardening effect from the thermal neutrons, the thermal-to-fast neutron flux density ratio plays a role. This effect is demonstrated in Table 3 where data are listed for West wall and North wall locations. There is an equal Si-content of about 1.37 weight percentage (thermal neutron fluence of  $5.5 * 10^{26} \text{ n/m}^2$ ) but with thermal to fast flux ratios of 1.0 and 4.8 respectively.

Table 3. Effect of neutron flux density ratio (th/f-ratio) on radiation hardening

Specimen location	Si-wt-%	th/f-ratio	hardness $HR_{15N}$	yield strength MPa	UTS MPa	uniform % elongation	total %
West wall	1.36	1.0	62.6	506	526	1.2	1.8
North wall	1.38	4.8	66.6	573	592	0.5	1.5

The influence is clear for the hardness and the tensile strength values. Due to the low ductility values, the thermal-to-fast flux ratio effect is less obvious on total elongation but uniform elongation is clearly affected. Increased strengthening with a higher thermal-to-fast flux ratio has been reported previously by Farrell (6). The measured effect is larger than predicted by Lybrink in ref. 4.

The results from the fracture toughness experiments are listed in Table 4. The table gives the maximum failure load ( $P_m$ ), the pop-in load ( $P_{pop}$ ), the load corresponding to an effective crack extension of 2 percent (load at 5 % deviation from linearity, ( $P_5$ )) and the conditional fracture toughness load ( $P_Q$ ) together with the calculated value of the conditional fracture toughness ( $K_{IQ}$ ). Further the qualification of the  $K_{IQ}$ -values according to E399 or B645 is given in this table.

Table 4. Results from fracture toughness experiments

No.*	$P_m$ KN	$P_{pop}$ KN	$P_5$ KN	$P_Q$ KN	$P_m/P_Q$ ratio	$K_{IQ}$ MPa $\sqrt{m}$	Qualification **
N11	4.10	3.95	3.95	3.95	1.04	25.3	M
N12	4.50	4.30	4.30	4.30	1.05	22.0	V
N13	4.30	4.20	4.20	4.20	1.02	24.9	M
N14	10.75	9.80	9.70	9.80	1.10	25.3	invalid : a/w = 0.31
N15	3.90	3.70	3.70	3.70	1.05	23.9	M
N18	5.55	5.25	5.20	5.25	1.06	24.0	V
E22	4.70	4.25	4.20	4.25	1.10	27.0	M
E23	5.45	4.55	4.50	4.55	1.20	28.9	M
E25	4.85	4.35	4.40	4.40	1.10	27.8	M
E27	6.15	4.75	4.75	4.75	1.29	30.3	invalid : B<13 mm, $P_m/P_Q=1.29$
S28	3.60	3.50	3.50	3.50	1.03	23.0	M
S30	3.80	3.65	3.60	3.65	1.04	24.2	M
S31	3.7est.	-	3.65	3.65	1.01	24.3	M
S33	4.30	4.05	4.00	4.05	1.06	26.6	M
S34	11.55	10.55	10.45	10.55	1.09	26.5	invalid : a/w = 0.30
S2-4	6.80	6.60	6.65	6.65	1.02	24.4	V
W3	5.35	5.30	5.30	5.30	1.01	22.3	M
W4	5.25	5.20	5.20	5.20	1.01	17.7	M
W7	3.85	3.85	-	3.85	1.0	16.5	invalid, $K_{fat}/K_Q = 1.1$
W10	6.10	5.85	5.80	5.85	1.04	25.7	M

\* N = North wall, E = East wall, S = South wall, W = West wall

\*\* V = valid  $K_{IC}$  according to ASTM E399

M = meaningful " $K_{IC}$ " according to ASTM B645

est = estimated due to early interruption of test record



The  $K_Q$ -values range from 16.5 MPa $\sqrt{m}$  to 30.3 MPa $\sqrt{m}$  with Si-content ranging from 0.54 wt-percentage to 1.85 wt-percentage (thermal fluence  $1.8 * 10^{26}$  n/m<sup>2</sup> to  $7.5 * 10^{26}$  n/m<sup>2</sup>). The data showed a consistent trend of decreasing  $K_Q$ -values with increasing Si-content, an effect of the thermal-to-fast flux ratio could not be identified.

Most of the specimens did not fulfil the E399 validity requirement on pre-crack length. The ASTM standard practice B645 for fracture toughness testing of aluminium alloys augments the basic test method E399 in the areas of the requirements for valid test results in terms of specimen size and fatigue pre-cracking conditions. If the E399 crack length requirement is not met, B645 requires  $0.4 < a/w < 0.6$  to classify the conditional fracture toughness  $K_Q$  as a meaningful measure of critical fracture toughness (" $K_{IC}$ "). This meaningful measure of the critical fracture toughness is considered to be within 5 to 10 percent of the value of the critical stress intensity factor  $K_{IC}$  which would have been obtained if all criteria had been met. The specimens approached the B645 requirement. Two specimens had a very short crack length of about  $a/w = 0.3$ , nevertheless the  $K_{IQ}$  values of 25.4 MPa $\sqrt{m}$  and 26.5 MPa $\sqrt{m}$  fall within the range of the meaningful critical toughness values according B645.

Two experiments did not fulfil the E399 load ratio requirement  $P_m/P_Q \leq 1.10$ . One did meet the relaxed B645 requirement  $P_m/P_Q \leq 1.2$ , the  $P_m/P_Q$ -ratio of the other was 1.29. The latter is the test with the specimen which did also not fulfil the thickness requirement. The lowest  $K_Q$ -value of 16.5 MPa $\sqrt{m}$  is obtained from an experiment which did not meet the final fatigue loading condition due to the high stress intensity factor range ( $\Delta K$ ) during the final fatigue loading. The  $K$ -ratio ( $K_{max}/K_Q$ ) was about 1. This specimen showed crack growth instability during the final fatigue crack growth.  $K_Q$  is obtained from the crack growth data at instability.

Three results give valid  $K_{IC}$  values ranging from 22.0 MPa $\sqrt{m}$  to 24.4 MPa $\sqrt{m}$ . Four results are judged invalid, although  $K_Q$ -values fit with the trend and the band-width of the other results. The remaining 13 results are considered to give meaningful " $K_{IC}$ " values, ranging from 17.7 to 28.9 MPa $\sqrt{m}$ . The lowermost meaningful " $K_{IC}$ " value of 17.7 MPa $\sqrt{m}$  is about 3 times the value of 6 MPa $\sqrt{m}$  which was estimated at shut-down of the old reactor vessel on the basis of the surveillance tensile test results (7). This factor of 3 implies critical crack length values 9 times greater than in the "fitness-for-purpose" assessments of the new HFR vessel compared with the crack length values based on the  $K_{IC}$ -value of 6 MPa $\sqrt{m}$ .

## SUMMARY AND CONCLUSIONS

After more than 20 years neutron expose of the HFR core-box walls :

- o the Rockwell Superficial hardness number increased by up to 35 points  
 $HR_{15N}$
- o the 0.2-yield strength increased by up to 500 Mpa
- o the ductility is reduced to a minimum value of about 1.5 % total elongation with 0.6 % uniform elongation
- o the irradiation effect on the fatigue crack growth rate is minor for applied  $\Delta K$ -ranges between 8 MPa $\sqrt{m}$  to 15 MPa $\sqrt{m}$
- o the lowermost meaningful measure of the critical plane-strain fracture toughness " $K_{IC}$ " is 17.7 MPa $\sqrt{m}$
- o a maximum of 1.9 weight percentage Si is created by reactions of the thermal neutrons with the aluminium (max. thermal fluence of  $8.3 * 10^{26}$  n/m<sup>2</sup>,  $E < 0.414$  eV)
- o the greatest hardening is observed for the North wall
- o the thermal to fast neutron flux density ratio has to be taken into account for neutron damage predictions of aluminium-magnesium alloys (5000 series)

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