



ASSESSMENT OF FRACTOGRAPHIC INVESTIGATION REPORT AND APPLICABILITY OF THE MASTER CURVE METHOD

Kim Wallin

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Summary <p>The report “Shell C, Fractographic Investigation of fracture faces – Material 1.2MD07. D02-ARV-01-086-123 Rev. A” summarizes the results of the fractographic investigations of the fracture faces from mechanical destroyed unirradiated and irradiated samples of the original shell C by SEM. The objective of the report was to find evidence for bigger agglomerations of non-metallic inclusions, especially Al₂O₃-inclusions in the samples. This report has been evaluated here and the following can be concluded:</p> <ol style="list-style-type: none"> 1. The report gives only a superficial exemplary examination of different microstructural features on the fracture surfaces. No details regarding the location of the found features are given. Nor is there performed any statistical analysis regarding the distribution and frequency of features. Also, since the cleavage fracture initiation sites have not been examined, nothing can be said about the relevance of the found features with respect to cleavage initiation. 2. The report appears incomplete since the last specimens have barely been examined at all and a detailed discussion of the findings is lacking. 3. There is a clear difference in the amount of grain boundary fracture between un-irradiated and irradiated material, but the relevance of this has not been addressed. It would be imperative to also examine fracture surfaces of surveillance sets with other fluence than the single fluence examined here. 4. The possible detrimental effect of MnS inclusions, and differences in their distributions, have not been examined in any detail. This is a deficit in the report. 	
Espoo 14.02.2022 Signature	 Kim Wallin Professor Emeritus, Dr. Tech.
Contact address Heinakuja 4A, 02760, Espoo 76, FINLAND E-mail: kim.wallin@kwsolutions.fi	
<div style="text-align: right;"> <i>K_w-solutions Ltd, Tel. +358 50 511 4126, Business ID 2940190-8</i> </div>	

Preface

This report describes the work performed within the Schweizerische Energie-Stiftung order dealing with on the report “Shell C, Fractographic Investigation of fracture faces – Material 1.2MD07. D02-ARV-01-086-123 Rev. A”, the following questions are addressed:

1. Which conclusions can be drawn from the fractographic investigations?
2. Is the assessment of the fractographic investigation by Axpö that the absence of Al₂O₃ inclusions in the accelerated irradiation specimens was causally justified by slight differences in the location of sampling, especially with regard to the position in the RPV wall?
3. Can it be excluded, that the inclusion clusters of shell C contain MnS as well as Al₂O₃? If it can't be excluded, should it have been considered in the analyses of Axpö?
4. Can it be excluded, that the shell C material with inclusion clusters has experienced microscopic changes due the result of a wrong heat treatment during manufacturing and to its above-average irradiation history of more than 50 years?
5. Can it be excluded that the predominant fracture mode of shell C has changed from cleavage fracture to grain boundary fracture?
6. What is the conservatism of the Master Curve application in this case?

The contact for the work representing Schweizerische Energie-Stiftung is Fabian Lüscher.

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Author

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Appendix A

1. Background

The RPV of the Swiss Beznau 1 NPP, which consists of welded metal rings, has the so-called shell C in the area closest to the nuclear fuel. Compared to shell D, which is also close to the core, and to the shells C and D of the neighbouring NPP Beznau 2, shell C has always been more brittle. However, the last set of accelerated irradiation samples taken from the reactor in 2009 showed an increase in embrittlement of a magnitude that had not been predicted in this form.

In 2015, ultrasonic indications of cluster density were detected in Ring C, which Axpo classified as non-metallic inclusions in the form of aluminium oxide. Non-metallic inclusions are usually assessed as a material degradation. Most indications were situated in the first 50 mm of depth (measured from the wet clad surface) and in a narrow band of 25 cm height in the lower part of the ring (close to weld RN5).

An RPV found to have such inclusions can continue to operate in normal power mode. However, if the reactor material is exposed to the spontaneously occurring loads of an accident with loss of coolant due to the injection of cold replacement coolant, thermal shock with loss of integrity of the reactor pressure vessel can occur. Such a loss of integrity would have massive consequences for the whole of Western Europe, not only economically.

The NPP operator must exclude this loss of integrity, which cannot be controlled by safety equipment, by means of the pressurized thermal shock (PTS) evaluation. The probability of such a severe accident must be orders of magnitude smaller than $10^5/a$.

A Fractographic Investigation Report issued by AREVA summarizes the results of the fractographic investigations of the fracture faces from mechanically destroyed unirradiated and irradiated samples of the original shell C by SEM. The objective of the report was to find evidence for bigger agglomerations of non-metallic inclusions, especially Al_2O_3 -inclusions in the samples.

In fact, however, other structures were found on the fracture surfaces:

- MnS inclusions: The found MnS inclusions are linear and oriented in the direction of crack propagation. Their lengths vary between $< 50 \mu m$ and approx. $700 \mu m$ and they are located on both the fatigue crack face and the cleavage fracture face.
- Al_2O_3 inclusions: Al_2O_3 inclusions are present both as isolated oxide particles and as groups of oxides arranged in a linear way and oriented in direction of crack propagation. Most oxides in the plane of crack propagation are located within the fatigue crack face. The size of the oxide clusters lies between $85 \mu m$ and $250 \mu m$, the average size of an individual oxide particle is about $10 \mu m$.
- Intergranular features: Mainly the fatigue crack face of eight samples is characterized by surface structures that resemble intergranular cracks. The exposed grain boundaries and triple junctions are rather smooth. Needle like precipitates are to be seen on the grain boundaries. An analysis of these precipitates by EDX was not possible due to their small size and the dose rate of the sample.
- Ductile areas: Ductile areas characterized by dimples can be found on three samples. Sample C28 and C8 feature rather big area with a maximum length of approx. $2 mm$ and $400 \mu m$, respectively, whereas sample C27L shows several smaller ductile areas within the cleavage fracture.
- The fractographic investigations of the irradiated samples showed mainly manganese sulphide inclusions, intergranular characteristics as well as ductile areas with dimples. Clusters of Al_2O_3 inclusions were only found in the unirradiated samples.

2. Brittle fracture toughness (The Master Curve)

Brittle fracture is the most critical failure mechanism, as it may cause catastrophic failure at unexpectedly low stress levels and its occurrence is not related to the mechanical strength of the material. Brittle fracture can be divided into two different mechanisms: trans-granular cleavage fracture and stress controlled grain boundary fracture. Out of the two mechanisms, cleavage fracture is more common and is usually referred to when the term brittle fracture is used. Grain boundary fracture is more difficult to characterize because it can be either brittle stress controlled or ductile strain controlled. In the case of 100% grain boundary fracture it is almost impossible to distinguish the mechanism from the fracture surface appearance. When the grain boundary fracture is less than 100%, the fracture mechanism may be estimated based on the appearance of the trans-granular fracture surface appearance close to the assumed fracture initiation region. If the trans-granular fracture is cleavage or so called quasi-cleavage, the grain boundary most likely is also stress controlled and it can be treated as brittle fracture. Otherwise, it is likely, but not necessary, strain controlled and it can usually be treated as ductile fracture. Caution in the decision should be practised. One good help in making the decision is the fact that brittle grain boundary fracture toughness increases with temperature, whereas ductile grain boundary fracture is temperature insensitive near ambient temperatures. Brittle fracture toughness is preferably characterized with the elastic-plastic fracture toughness K_{JC} . The material is not a continuum. It contains grains, inclusions, segregations, precipitates and dislocations (Figure 1). This causes brittle cleavage fracture initiation to be strongly dependent on the local fracture resistance of the material.

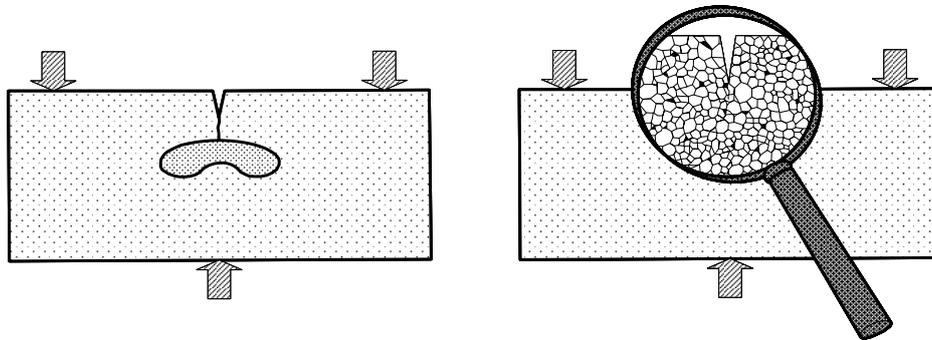


Figure 1 Difference between a continuum mechanics material definition and a real microstructure.

The different possible mechanisms of cleavage fracture and stress controlled grain boundary fracture initiation are qualitatively rather well known. Primarily the initiation is a critical stress controlled process, where stresses and strains acting on the material produce a local failure, which develops into a dynamically propagating cleavage crack. The local “initiators” may be precipitates, inclusions or grain boundaries, acting alone or in combination. An example of a typical cleavage fracture initiation process is presented schematically in Figure 2. The first step involves the cracking of a precipitate or inclusion (sometimes, it may also be a grain boundary or grain triple point). The second step consists of the carbide size micro-crack propagating into the surrounding matrix and the third step consists of the grain-size crack propagating into neighbouring grains. The two first steps are mainly affected by the particle size and the local stress and strain at the initiation site. The third step, however, is also affected by the stress gradients in the vicinity of the initiation site, since the third step covers a larger material volume.

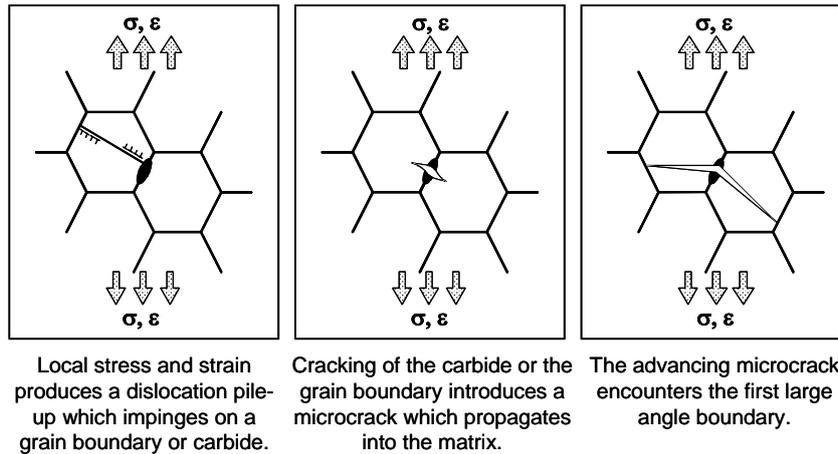


Figure 2 Schematic presentation of the necessary steps for cleavage fracture initiation.

Depending on loading geometry, temperature, loading rate and material, different steps are more likely to be most critical. For structural steels at lower shelf temperatures and ceramics, in the case of cracks where the stress distribution is very steep, steps II and III are more difficult than initiation and they tend to control the fracture toughness. At higher temperatures, where the steepness of the stress distribution is smaller, propagation becomes easier in relation to initiation and step I becomes more and more dominant for the fracture process. The temperature region where step I dominates is usually referred to as the transition region. On the fracture surface of a specimen with a fatigue crack this is usually seen as a difference in the number of initiation sites visible on the fracture surface. At lower shelf temperatures, numerous initiation sites are visible, whereas at higher temperatures, corresponding to the transition region, only one or two initiation sites are seen. The initiation sites can usually be traced on the fracture surface by analysing so called river patterns as shown in Figure 3.

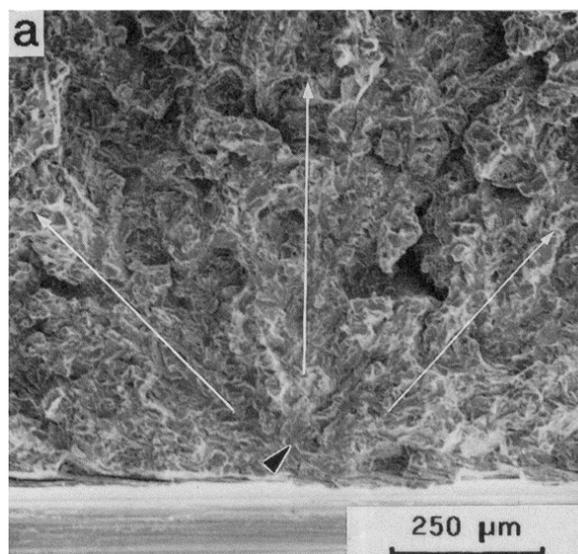


Figure 3. River patterns indicating the location of cleavage fracture initiation.

The fracture surface appearance is an effective tool in the decision if the material is on the lower shelf or in the transition region. The region has an implication on the macroscopic statistical behaviour of cleavage fracture and is therefore also affecting the application of the fracture toughness test results.

Because, as shown in Figure 1, no materials are fully uniform on a microscale, cleavage fracture initiation is a statistical event, which has implications upon the macroscopic nature of brittle fracture. A statistical model is thus needed to describe the probability of cleavage fracture.

Besides scatter, the transition region relates to statistical specimen size effects. The size effects have the consequence that fracture toughness data obtained from small laboratory specimens do not directly describe the fracture behaviour of real flawed structures. Besides statistical size effects, the transition region fracture toughness is also sensitive to constraint differences. Intensive research has been conducted in the last decade to master these problems. Different approaches have been developed and proposed, one of the most promising being the Master Curve (MC) method. It provides a description for the fracture toughness scatter, size effect and temperature dependence both for the transition region as well as the lower shelf.

The MC enables a complete characterization of a material's brittle fracture toughness based on only a few small size specimens. The method combines a theoretical description of the scatter, a statistical size effect and an empirically found temperature dependence of fracture toughness. The fracture toughness in the brittle fracture regime is thus described with only one parameter, the transition temperature T_0 (Figure 4).

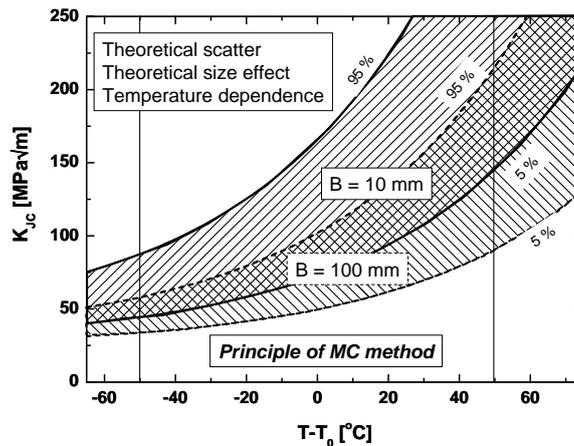


Figure 4. Principle of the basic MC approach.

The MC method is applicable for ferritic structural steels and has been standardised by the American Society for Testing and Materials, ASTM E1921-98. It is the first fracture toughness testing standard that gives advice on the use of the test result.

3. AREVA report

The examination in this report focusses on the fractographic work presented in the AREVA report. “Shell C, Fractographic Investigation of fracture faces – Material 1.2MD07. D02-ARV-01-086-123 Rev. A.” AXPO 2015.

A review of the fractographic work presented in the AREVA report is given in Appendix A. The AREVA report looks at fracture surfaces from broken mechanical testing samples from the unirradiated and irradiated state of Beznau 1 RPV shell C. It looks at both Charpy-V, SE(B), C(T) and WOL specimen samples. The fracture surfaces were examined by means of scanning electron microscopy. Samples from the lower and upper toughness scatter

bands were reportedly selected for the examination. A total of 19 broken specimens were examined. The AREVA report examined the overall fracture surfaces to identify specific details like, Al₂O₃ and MnS inclusions, ductile tearing, and grain boundary fracture. For all 19 specimens a single primary cleavage fracture initiation site can be identified. This verifies that the specimens failed by a so-called weakest link type of mechanism as assumed in the Master Curve method. Unfortunately, the AREVA report does not contain location information about the identified Al₂O₃ and MnS inclusions, ductile tearing, and grain boundary fractures. Also, the AREVA report has not examined the initiation sites, to see what has triggered the cleavage fracture. It is therefore not possible to link the fractography directly to the measured fracture toughness. The objective of the report was to find evidence for bigger agglomerations of non-metallic inclusions, especially Al₂O₃-inclusions in the samples and this explains partly why the initiation sites have not been examined. However, the lacking examination of the initiation sites and **especially** the lacking location information of the found inclusions is a weakness of the report. It is worth noting that the irradiated specimens all refer to surveillance set T, with a fluence of $6.04 \cdot 10^{19}$ [cm²].

The areas of ductile fracture found in the two Charpy-V specimens (C28 and C27L) and the WOL specimen C8 are all secondary events due to deviations in the cleavage fracture planes, caused by large surface roughness connected to relatively high fracture toughness and impact energy.

The fractography in the AREVA report basically only contains qualitative information about the existence of different inclusions and potentially weakened grain boundaries. A couple of conclusions regarding the fractography can be made:

- 1) The irradiated specimens show more grain boundary fracture than the un-irradiated specimens. This indicates that the irradiation fluence and irradiation temperature have caused segregation of P or S to the grain boundaries. The fraction of grain boundary fracture on the fracture surfaces is still quite small but, with time, grain boundary fracture may become significant for the fracture toughness. It would be beneficial to perform fractography also on surveillance samples with lower fluence to obtain information on the kinetics of the segregation process, to enable an estimate of the critical fluence where grain boundary fracture becomes significant for the fracture toughness.
- 2) The irradiated specimens have been taken from a region without Al₂O₃ inclusions, contrary to the unirradiated specimens. All samples have been extracted from a section below the ring C, used for qualification tests. Figure 1 shows a schematic estimate of the locations of Al₂O₃ inclusions in the ring and the qualification cut out. The irradiated samples correspond to the 1/4 or 3/4 location in the thickness. This could be a region outside the Al₂O₃ inclusions, and their location corresponds to the irradiation surveillance program position. The extraction position of the unirradiated specimens correspond more to the centre location in the thickness, and based on Figure 1, they should lie in the region of Al₂O₃ inclusions. This centre location segregation in the qualification ring may also explain the higher Master Curve T₀ values shown by the 25 mm C(T) specimen taken exactly from the centre location of the qualification cut out.

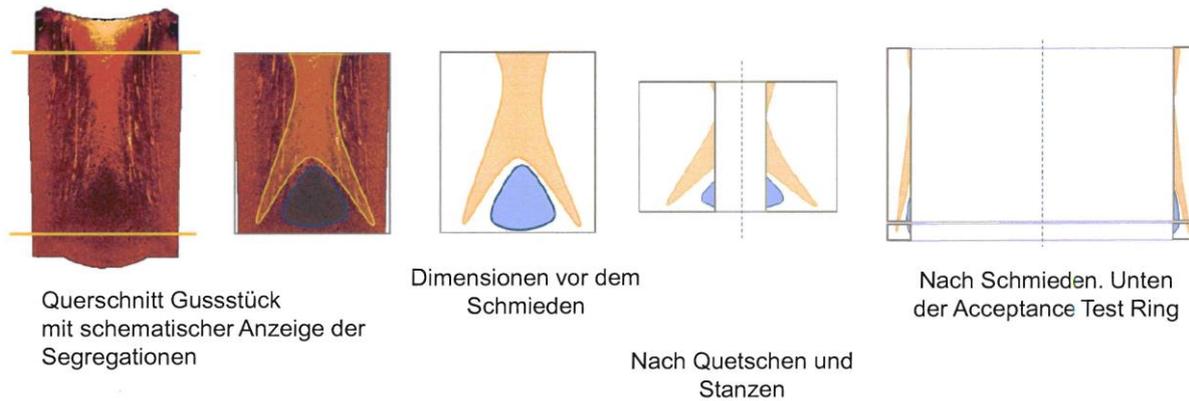


Figure 1. Schematic estimate of locations of Al_2O_3 inclusions, from AXPO interim report.

- 3) All specimens contain MnS inclusions. This is quite normal since one reason for Mn addition in the steel is to remove solute sulphur, which otherwise might segregate to the grain boundaries. MnS inclusions are not generally considered to be excessively detrimental to cleavage fracture, but there have been reported cases where cleavage fracture initiation has been triggered by a MnS, often combined to a brittle precipitate. *See e.g. Thesis by Abhijit Ghosh, entitled "Effect of Microstructure and Crystallographic Texture on Impact Toughness in Low Carbon Ferritic Steel", submitted to the Indian Institute of Technology, Kharagpur in 2016.* Ductile fracture is more affected by MnS inclusions since they act as nucleation sites for voids and ductile fracture is a continuing process of void nucleation, growth and coalescence. The amount of MnS inclusions show a variation from specimen to specimen. Especially the 25 mm C(T) specimens B1C.3, B1C.5 and B1C.7 show many MnS inclusions close to the specimen back surface. Even though not necessarily significant for the fracture toughness of these specimens, a specimen with a crack front located in a region with many MnS inclusions might show decreased fracture toughness. It is unclear if any other specimens cover the same material region. This strengthens the need for specimen extraction diagrams and metallography of the cut out material.
- 4) The examined specimens do not represent from a metallurgical perspective a macroscopically homogeneous material. Based solely on the fractography report it is not possible to address the representativity of the examined fracture surfaces. Since the initiation sites have not been studied in any detail, it is not possible to rule out that inclusions or grain boundary fracture may have played a role in the cleavage fracture initiation event.

4. Answers

These are the answers to the questions made regarding the fractography report. It should be emphasized that the answers relate almost solely to the information in the fractography report. With more information the answers may change, or be strengthened.

1. Which conclusions can be drawn from the fractographic investigations?

The fractography presented in the report is not very detailed. It does not try to identify the structure corresponding to the cleavage initiation site. It only provides an overall description of features observed on the fracture surfaces. It can be concluded that a low ductile tearing resistance is not a factor in the fracture toughness values. Neither does grain boundary fracture play a pronounced role, even though cleavage initiation interacting with grain boundary fracture cannot be ruled out. The same is true for the MnS inclusions. The possible effect of regions with many MnS inclusions on the fracture toughness should be examined. Based on the fractography, the role of Al_2O_3 inclusions on the cleavage fracture initiation is

minimal, even though some effect cannot be ruled out, since the initiation sites have not been investigated.

2. Is the assessment of the fractographic investigation by Axpo that the absence of Al_2O_3 inclusions in the accelerated irradiation specimens was causally justified by slight differences in the location of sampling, especially regarding the position in the RPV wall?

All samples have been extracted from a section below the ring C, used for qualification tests. As noted above, based on Figure 1, the irradiated samples correspond to the $\frac{1}{4}$ or $\frac{3}{4}$ location in the thickness. This could be a region outside the Al_2O_3 inclusions, whereas the extraction position of the unirradiated specimens correspond more to the centre location in the thickness, and based on Figure 1, they should lie in the region of Al_2O_3 inclusions. In the report “Axpo technical report KKB530D0220, Rev. 2, dated 21 October 2016. Assessment of aluminium oxide inclusions on irradiated material properties of Beznau surveillance Material” a specimen from another surveillance set was investigated by metallography. The specimen C7-B154 originates from surveillance capsule S with a fluence of $1.07 \cdot 10^{19}$ [cm^2] and it contains both Al_2O_3 and MnS inclusions. Since the different surveillance sets correspond to different locations in the qualification cut out, some sets may contain Al_2O_3 inclusions, and some may not. This makes the Axpo conclusion regarding slight differences in the location of sampling plausible.

3. Can it be excluded, that the inclusion clusters of shell C contain MnS as well as Al_2O_3 ? If it can't be excluded, should it have been considered in the analyses of Axpo?

It is possible that the inclusion clusters of shell C contain MnS as well as Al_2O_3 since it is possible that Al_2O_3 inclusions solidify in the vicinity of a MnS inclusion. Their difference lies in their size and number and it is unclear if a cluster containing both MnS as well as Al_2O_3 inclusions is any worse than a single MnS inclusion. Based on the “ENSI Review of the Axpo Power AG Safety Case for the Reactor Pressure Vessel of the Beznau NPP Unit 1”, MnS seems to have been, at least to some extent, considered in the analyses. In the review it is stated that “Axpo performed metallographic examinations on unirradiated and irradiated specimens of the acceptance test Shell C material. The examination was performed on 12 broken C(T)-25 mm specimens. The same inclusion types, shapes, and orientations were found in all examined specimens. Mainly three different types of non-metallic inclusions were observed: elongated MnS inclusions, elongated Al_2O_3 inclusions, and a combination of MnS inclusions, Ca and fine globular Al_2O_3 inclusions. All specimens showed a homogeneous distribution of inclusions and the average inclusion content was in the range of 0.13 % to 0.45 % volume fraction. The sulphide inclusion lines have a maximum length of 0.93 mm and the alumina inclusion lines of 0.90 mm.” More details should be found in Axpo technical report “KKB530D0159, Rev. C, dated 29 November 2017: Summary of material properties of Beznau 1 RPV used for assessment of UT indications” if available. This report claims to contain metallographic examinations on unirradiated and irradiated specimens of the acceptance test Shell C material, for 25 mm C(T) specimens. Both the ENSI Review and the Axpo report must have a typographical error since no irradiated 25 mm C(T) specimens should exist.

4. Can it be excluded, that the shell C material with inclusion clusters has experienced microscopic changes due the result of a wrong heat treatment during manufacturing and to its above-average irradiation history of more than 50 years?

The Al_2O_3 inclusions are not affected by heat treatment, they are formed already in the melt steel and are chemically inactive with the metal matrix. It can therefore be excluded that the inclusions would experience changes during the operation of the plant.

5. Can it be excluded that the predominant fracture mode of shell C has changed from cleavage fracture to grain boundary fracture?

Provided that the fluence of the irradiated specimens is representative or conservative with respect to the pressure vessel, based on the fractography showing only limited spots of grain boundary fracture, it can be excluded that the predominant fracture mode of shell C has changed from cleavage fracture to grain boundary fracture. This statement is strictly only valid for the material region that the irradiated specimens represent. Since there are, in the report, no fractography, or detailed chemistry with regard to P and S, of irradiated material representing the region with Al₂O₃ inclusions, nothing can be said about that region's propensity to grain boundary fracture. Specimens from other surveillance sets should also undergo fractography to get a more detailed picture of the grain boundary fracture kinetics.

6. What is the conservatism of the Master Curve application in this case?

Important for the application of the Master Curve is that the fracture is controlled by a weakest link type mechanism. In this case all specimens show one primary initiation site, thus confirming a weakest link type mechanism. It should of course be emphasised that the Master Curve describes only the test results used in the application of the Master Curve. For macroscopically uniform materials the result describes the whole material conservatively, provided that statistical, experimental, and metallurgical uncertainties are accounted for. If the material in ring C differs considerably from the material used for the Master Curve, the result may be conservative, or not, depending on situation. Based solely on the fractography report the question cannot be answered. There are two sets of irradiated SE(B) specimens corresponding to the fluence $6 \cdot 10^{19}$. The first set was manufactured from broken WOL specimens and the resulting T_0 value agrees well with the surveillance CVN results. The T_0 for the second set, manufactured from broken surveillance CVN specimens, is not available.

Following is a list of reports that should contain complementary information to help in the answering of the questions:

AREVA technical document KKB530D0135, Rev A, dated 25 March 2013. Ermittlung von Bruchmechanikkennwerten an unbestrahlten Proben für Beznau-1: Fertigung, Prüfung und Auswertung von C(T)10-Proben aus dem Werkstoff 1.2MD07.

Axpo technical document KKB530D0126, Rev A, dated 11 July 2012. Ermittlung von Bruchmechanikkennwerten an unbestrahlten Proben für Beznau-1: Fertigung, Prüfung und Auswertung von SE(B)-Proben aus dem Werkstoff 1.2MD07.

Axpo inspection report KKB530D0195, Rev. A, dated 27 October 2016. Metallographic examination of Beznau 1 RDB Shell C material, Material: 1.2MD07.

Axpo technical report KKB530D0275, Rev. A, dated 23 February 2017. Conservatism of reference temperature determined with SE(B) specimens from Beznau 1 surveillance capsule T.

Axpo technical report KKB530D0250, Rev. NA, dated 01 February 2017. Assessment of aluminium oxide inclusions on irradiated material properties Beznau surveillance material – 2nd Phase.

Axpo technical report AN-530-MB12028, Rev. 0, dated 22 January 2013. Summary of Surveillance Material Testing Beznau Unit 1+2 for ORNL review.

Axpo technical report KKB580D0389, Rev. A, dated 27 October 2016. Metallographic examination of Beznau 1 RDB Shell B material, Material: 1.2MD07.

Axpo technical report KKB580D0379, Rev. A, dated 27 October 2016. Metallographic examination of irradiated WOL25X specimens, Material: 1.2MD07.

Axpo technical report English Translation of KKB580D0378, Rev. NA, dated 18 August 2016.

Axpo technical report KKB530D0233, Rev. D, dated 28 November 2017. Heat treatment, sampling, manufacturing, measuring and photo documentation of tensile specimens with diameter 5 mm and 12,5 mm, notched tensile, C(T) 12.5 and C(T)25 specimens.

Axpo technical report KKB530D0225, Rev. NA, dated 21 April 2016. Axpo RPV surveillance material – location of the Charpy specimens in the wall thickness.

5. Conclusions

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1. The report gives only a superficial exemplary examination of different microstructural features on the fracture surfaces. No details regarding the location of the found features are given. Nor is there performed any statistical analysis regarding the distribution and frequency of features. Also, since the cleavage fracture initiation sites have not been examined, nothing can be said about the relevance of the found features with respect to cleavage initiation.
2. The report appears incomplete since the last specimens have barely been examined at all and a detailed discussion of the findings is lacking.
3. There is a clear difference in the amount of grain boundary fracture between un-irradiated and irradiated material, but the relevance of this has not been addressed. It would be imperative to also examine fracture surfaces of surveillance sets with other fluence than the single fluence examined here.
4. The possible detrimental effect of MnS inclusions, and differences in their distributions, have not been examined in any detail. This is a deficit in the report.