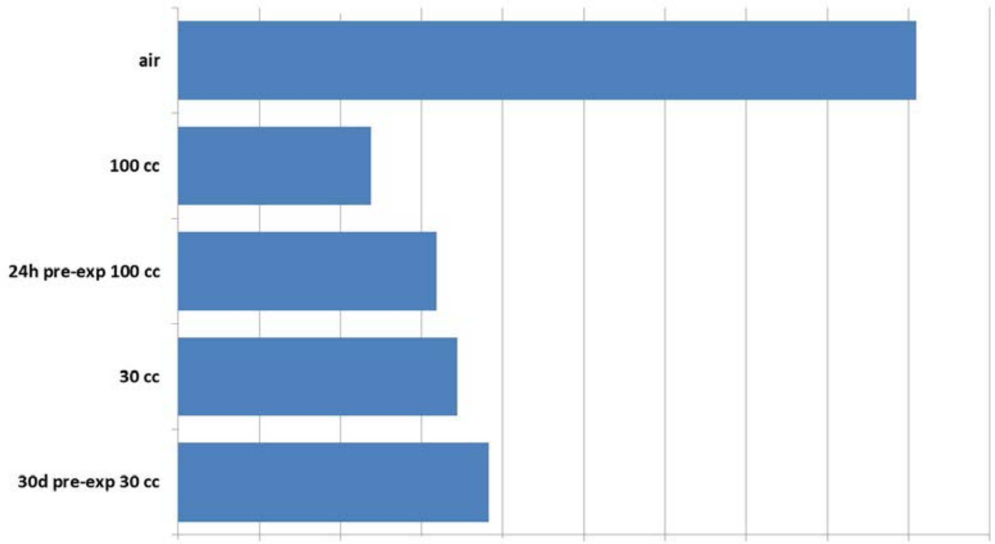
RESEARCH REPORT

VTT-R-00491-13



Collation of LTCP Test Results Obtained in Years 2008-2012

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Summary

This report collates the obtained J-R test results from Master's thesis work and the work performed in DEFSPEED and ENVIS projects so far. The effect of hydrogenated PWR primary water on the Low Temperature Crack Propagation (LTCP) susceptibility of nickel based weld metals Alloy 182, 82, 152 and 52 has been studied performing J-R tests at a slow displacement rate in simulated low temperature PWR primary water. Hydrogen contents of 0, 5, 30, 64 and 100 cm^3 H_2/kg H_2O have been used. Significant scatter in fracture toughness J_Q and tearing modulus T values was observed in all the tested materials. However, the results show clearly that Alloy 182 is the most susceptible material to LTCP of the nickel-based weld metals, whereas Alloy 52 retains its high toughness at least up to 30 cm^3 H_2/kg H_2O. Increasing hydrogen content reduces the fracture toughness of nickel-based weld metals in general.

Confidentiality	Public

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Preface

The funding of the ENVIS (Environmental influence on cracking susceptibility and ageing on nuclear materials) project by VYR (the State Nuclear Waste Management Fund), VTT (Technical Research Centre of Finland), SSM (Swedish Radiation Safety Autohority) and OECD Halden project as well as the profound support from the Support Group 6 is highly appreciated.

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Author

Contents

1. Introduction

This research report was carried out as a part of the SAFIR 2014 project ENVIS and it summarises the results obtained thus far from fracture toughness (J-R) tests performed in the years 2008-2012. The J-R tests were conducted in low temperature water (55 °C) with varying contents of hydrogen (0-100 $cm^3/kg\ H_2O$). The results for weld metals Alloy 182, Alloy 82, Alloy 152 and Alloy 52 are presented.

Low Temperature Crack Propagation (LTCP) is a hydrogen induced degradation mechanism that has been observed in laboratory conditions in nickel based weld metals Alloy 182/82 and Alloy 152/52 [1-6], and in high strength Alloy X-750 [7]. The phenomenon occurs especially in the temperature range of 50 to 150 °C in aqueous hydrogenated environments. The decrease in J_{IC} values for Alloys 82 and 52 in 54 °C water containing 150 $cm^3\ H_2/kg\ H_2O$ is reported to be an order of magnitude compared with results obtained in air tests [2-4].

An essential feature of LTCP is a transition in cracking mechanism from ductile dimple fracture to intergranular (IG) cracking, and the relation between fracture toughness of a material and cracking mechanism is normally clearly evident. According to the results of several studies [1-8], the mechanism of LTCP is hydrogen induced. Mills et al. [2] presents fractographic evidence of hydrogen embrittlement for weld metal Alloy EN82H. The fracture surface appearance of non-precharged specimens tested in hydrogenated water and hydrogen precharged specimens tested in air is remarkably similar. In addition, transgranular facets that were observed in both cases provide additional evidence that a hydrogen embrittlement mechanism is active in low temperature water. The effect of hydrogen has also been demonstrated in several studies [4, 5, 7] by varying the hydrogen content of low temperature water. These studies show that increasing hydrogen content decreases the fracture toughness of nickel based materials. However, more recently it has also been reported that even at low hydrogen contents (3-5 $cm^3\ H_2/kg\ H_2O$) the reduction in fracture toughness is significant [9]. The most significant reductions of fracture toughness have been observed in hydrogenated water at about 50 °C. The fracture toughness is restored when temperature increases to above 150 °C [2].

The microstructure has a crucial effect on the LTCP susceptibility of nickel based alloys. The morphology of the grain boundaries has an effect of hydrogen trapping [10]. Large MC-type precipitates are usually present in wrought Alloys 600 and 690. During welding, small niobium- and titanium-rich carbides are formed at the grain boundaries. These small precipitates act as hydrogen traps, and therefore they are detrimental to LTCP resistance. In multiple bead welds, recrystallization in the solidified beads causes a decrease in LTCP resistance because deformation localizes in the lower strength recrystallized areas. This induces localization of hydrogen in the material [11]. In addition, it has been suggested that the intrinsic dislocations and vacancies stored in the grain boundaries represent trapping sites for hydrogen in low temperature regimes [12]. Therefore, the diffusivity of hydrogen in nickel-based materials can be reduced along grain boundaries, which may enhance intergranular cracking.

Initiation of LTCP has been studied by Brown and Mills [13]. They discovered that intergranular LTCP does not initiate directly from an as-machined notch, but a sharp crack is required for LTCP to occur. However, once a transgranular tear is formed to the notch, it serves as a sharp crack from which LTCP initiates. In addition, a hydrogenated water environment decreases the crack initiation toughness, which is associated with a hydrogen-induced transgranular faceting mechanism. LTCP can initiate directly from a crack-like weld defect, and the crack growth behaviour is similar to that of fatigue pre-crack initiated LTCP [13].

The effect of loading rate on the LTCP susceptibility of Alloy 182 and 52 dissimilar metal weld (DMW) specimens has been studied at VTT by performing J-R tests in hydrogenated low temperature water [1,14,15,16]. The results of the J-R tests show that the fracture toughness of the studied DMW samples was practically unaffected by the environment when the loading rate was 6.7 mm/h. At a lower loading rate, 0.1 mm/h, most Alloy 182 DMW specimens showed typical features of LTCP, including interdendritic fracture and significant reduction in fracture toughness. However, the Alloy 52 DMW specimens did not show any signs of LTCP even at the slow (0.1 mm/h) loading rate.

In this report results from three different test series are presented. The first test series was performed as a Master's thesis work [14], the second within the DEFSPEED project [15] and the latest, still ongoing test series within the ENVIS project. As this report aims to be a general overview of the LTCP results, more detailed information of each test series is presented in references [14] and [15].

2. Materials and Methods

2.1 Materials

Two types of materials have been used to manufacture the SE(B) test specimens for J-R testing, i.e., pure weld metal blocks and dissimilar metal weld (DMW) mock-ups. Pure weld metal blocks were fabricated from Alloys 182, 82, 152 and 52. In DMW mock-ups the studied weld metals consist of Alloys 182, 152 and 52. In addition, some specimens of sensitized austenitic stainless steel AISI 304 were tested and the results are reported in [14].

2.1.1 Pure Weld Metal Test Blocks

The SE(B) test specimens of pure weld metals, i.e., Alloy 182, Alloy 82, Alloy 152 and Alloy 52, were cut from the samples presented in Figure 1. The samples were fabricated by welding multiple beads on cruciform section steel bars, as presented in Figure 2, using weld wires identified in
Table 1. Welding was carried out manually without post weld heat treatment (PWHT).

Figure 1. Pure weld metal test blocks of Alloy 82, Alloy 182, Alloy 52 and Alloy 152, respectively [15].

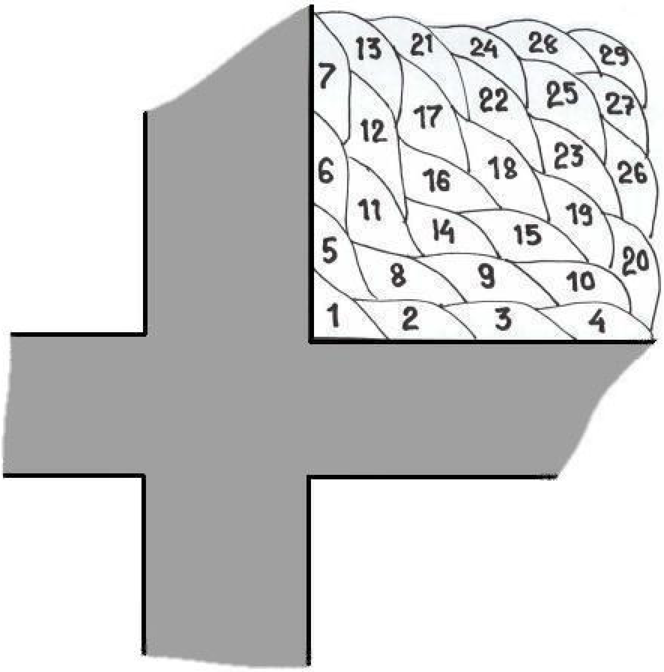

Figure 2. Welding sequence of the pure weld metal test block Alloy 182 [14].

Table 1. Chemical compositions of the weld filler metals [17].

	Alloy 182	Alloy 82	Alloy 152	Alloy 52
C	0.03	0.04	0.05	0.03
Si	0.80	0.03	0.04	0.13
Mn	6.50	2.98	3.48	0.24
P	0.01	0.00	0.00	<0.001
S	0.00	0.00	0.00	<0.001
Cr	15.70	19.94	28.74	29.20
Mo	-	-	0.01	0.03
Ni	68.00	72.60	55.20	59.28
Nb	1.80	Nb + Ta 2.47	Nb + Ta 1.54	Nb + Ta < 0.02
Ti	0.10	0.34	0.09	0.51
Fe	6.70	1.00	10.39	9.80
Al	-	-	0.06	0.72
Cu	<0.01	0.01	<0.01	0.04

2.1.2 Dissimilar Metal Weld (DMW) Mock-up Test Blocks

In the first LTCP related work [14] the tested materials were dissimilar welds that are called TU2 (Alloy 52), TV (Alloy 182) and NO (Alloy 182). The test blocks are presented in Figures 3-5.

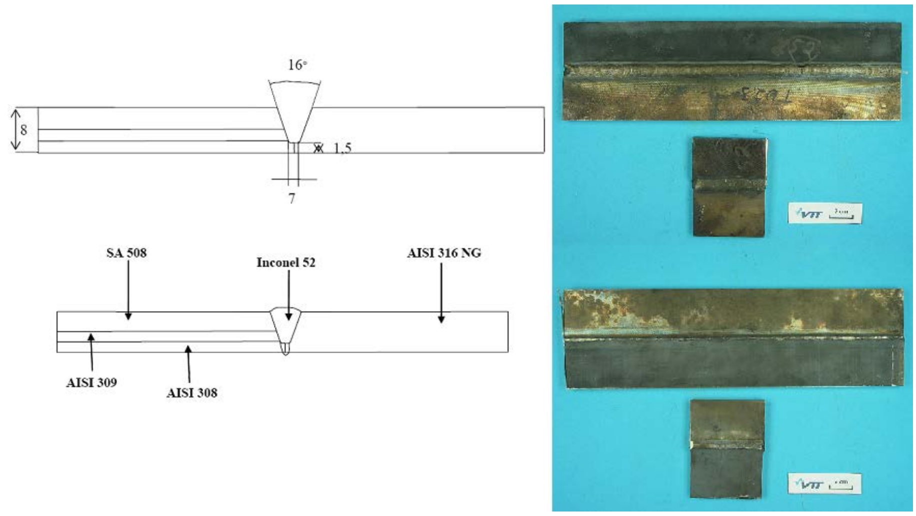

Figure 3. Test block TU2 (Alloy 52). A schematic drawing of the weld on the left, photographs taken from both sides of the actual test block on the right [14].

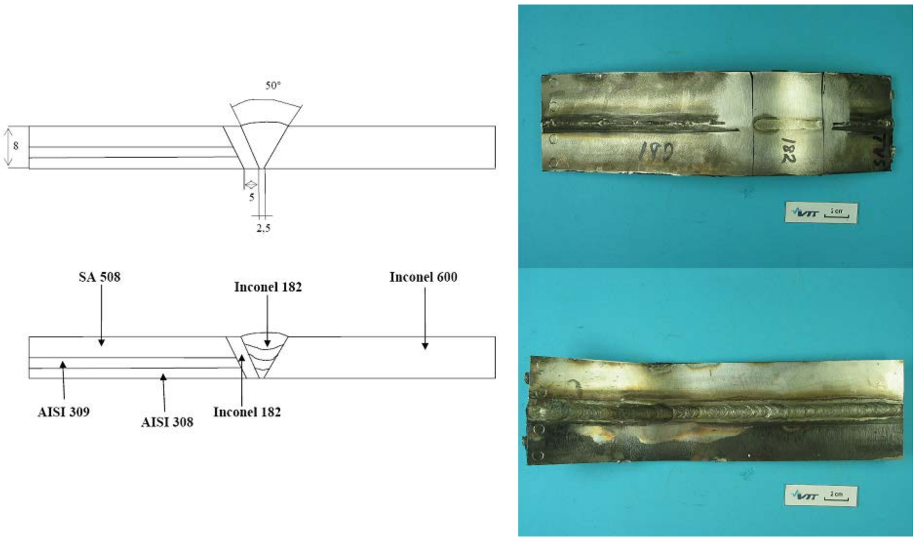

Figure 4. Test block TV (Alloy 182). A schematic drawing of the weld on the left, photographs taken from both sides of the actual test block on the right [14].

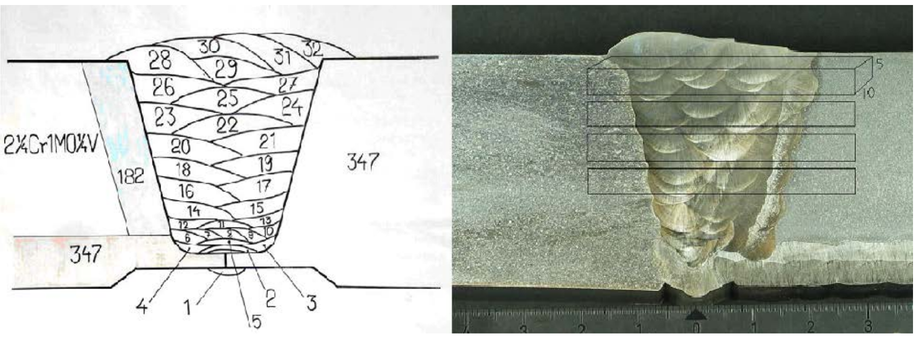

Figure 5. Test block NO (Alloy 182). A schematic drawing of the weld cross-section on the left including welding sequence and surrounding base materials. On the right a photograph taken from the actual test block cross-section with schematic drawings of the specimens (5x10x55 mm) that were cut of the block [14].

EDS analyses that were performed on cross-sections of the DMW mock-ups show that in the first mentioned DMW (TU2) intermixing between the base material and the weld metal was significant (Figure 6). The Alloy 182 DMWs were relatively close to the nominal chemical composition in the studied area, i.e., in the middle of the weld (Figure 7 and Figure 8).

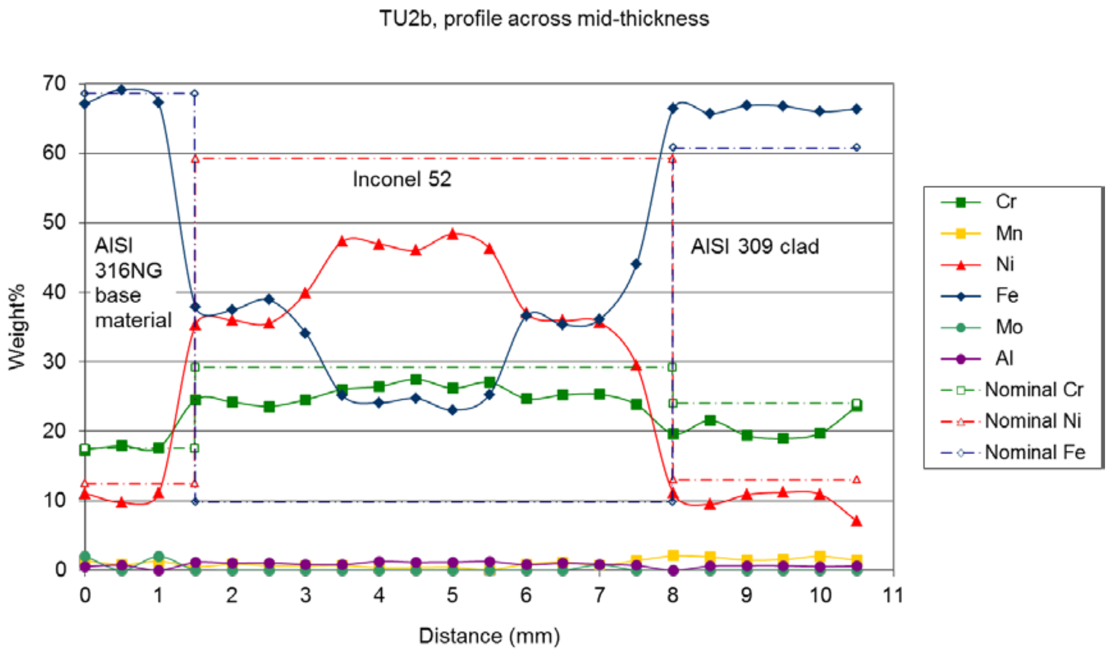

Figure 6. EDS analysis of the chemical composition profile of the dissimilar metal weld Alloy 52 (TU2) [15].

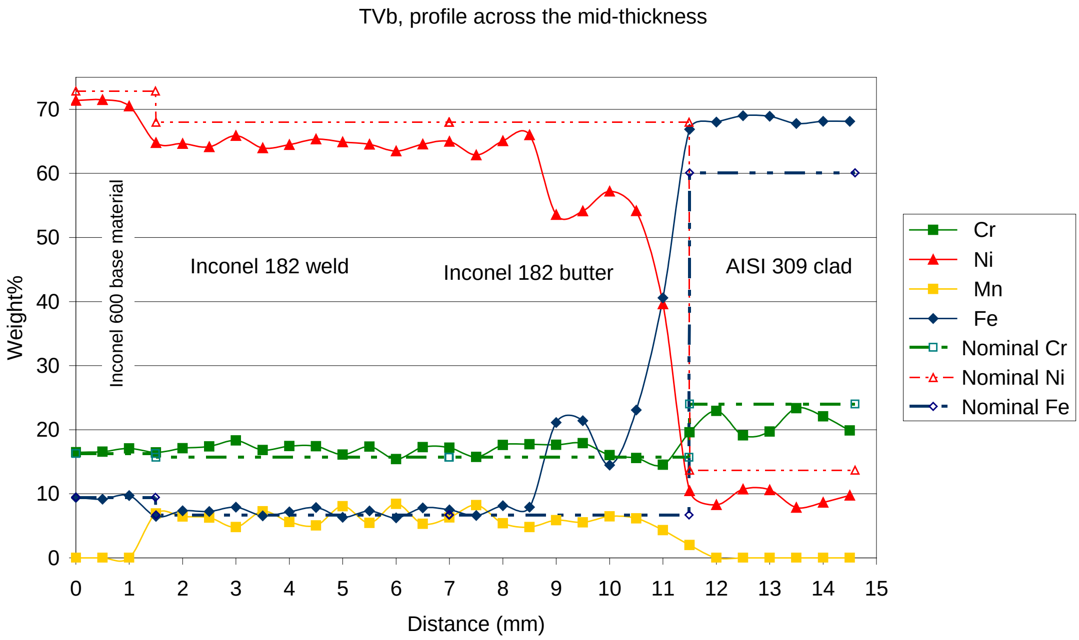

Figure 7. EDS analysis of the chemical composition profile of the dissimilar metal weld Alloy 182 (TV) [15].

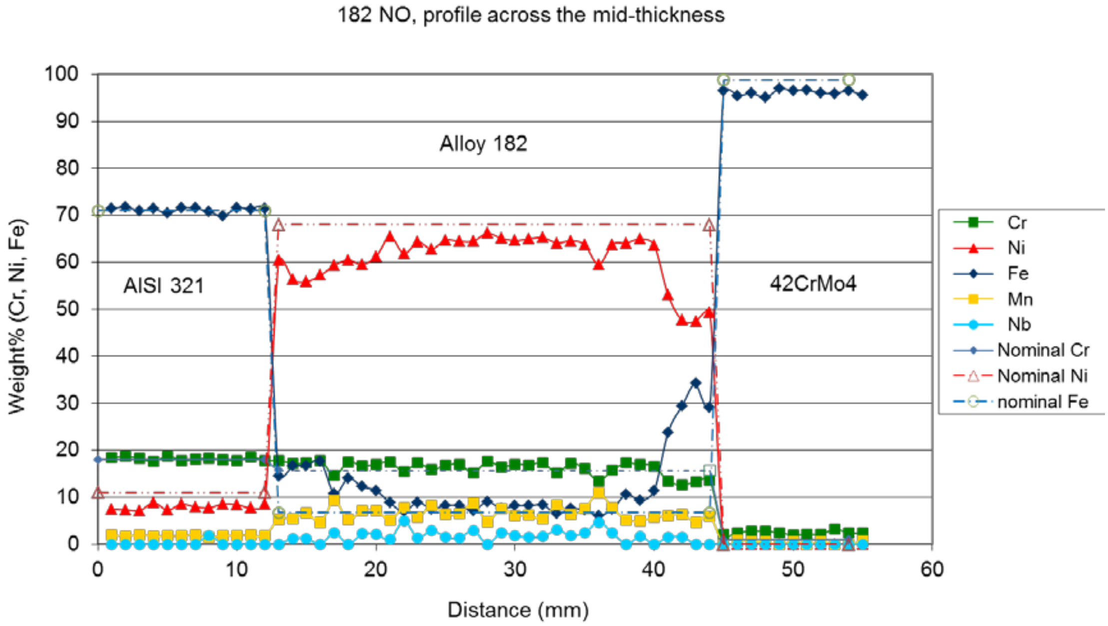

Figure 8. EDS analysis of the chemical composition profile of the dissimilar metal weld Alloy 182 (NO) [15].

The more recently tested DMW mock-up materials were manufactured using weld metals Alloy 182, Alloy 152 and Alloy 52. The cross-section images of the mock-ups are presented in Figures 9-11.

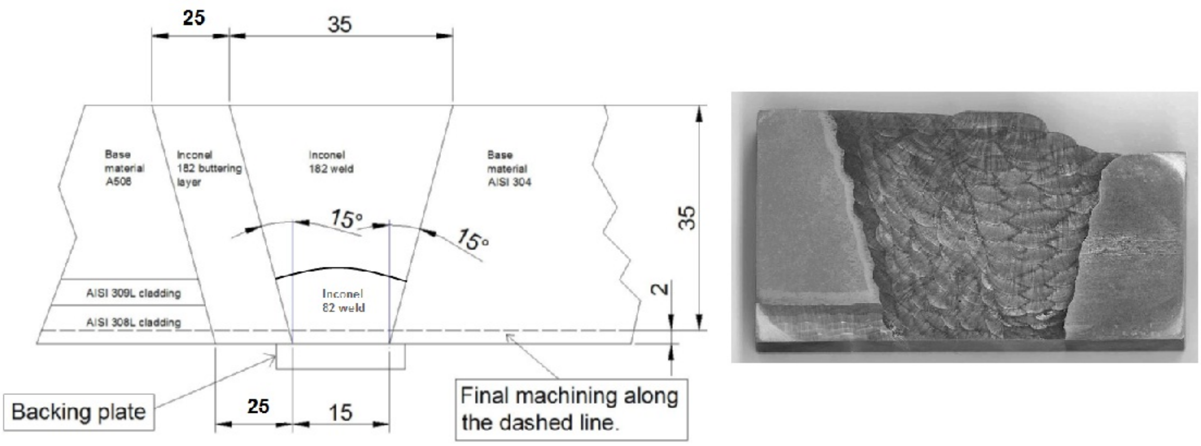

Figure 9. A schematic drawing of weld cross-section of test block BWR mock-up (Alloy 182) on the left, and a photograph taken from the actual polished and etched cross-section on the right.

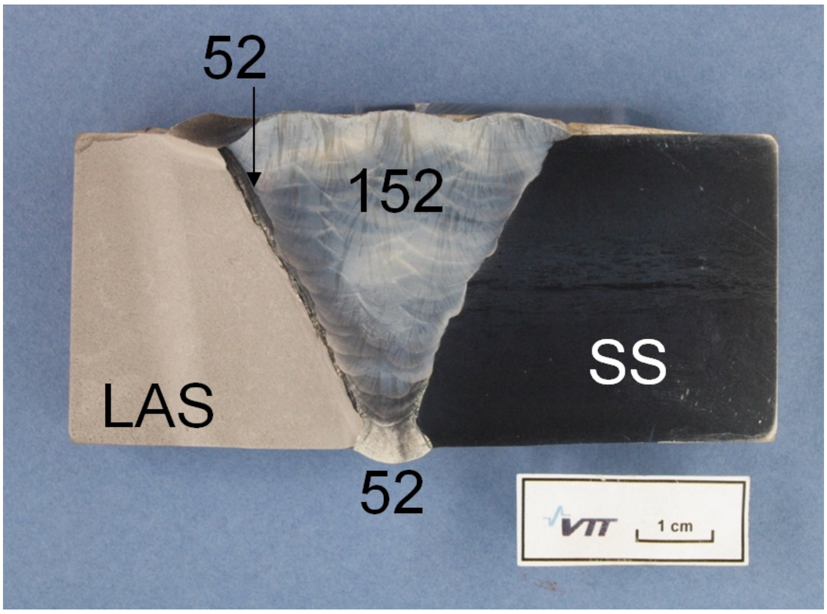

Figure 10. A photograph of KAIST mock-up (Alloy 152) test block cross-section (polished and etched) showing low alloy steel and stainless steel base materials, Alloy 52 butter and root and Alloy 152 weld.

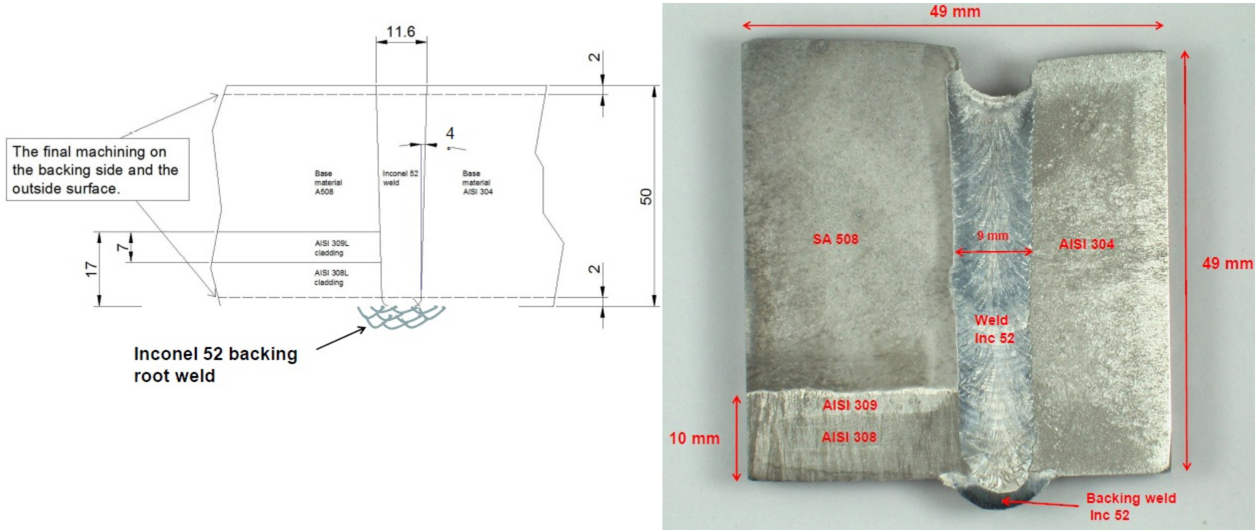

Figure 11. A schematic drawing of weld cross-section of test block Alloy 52 narrow gap weld mock-up on the left, a photograph taken from the actual polished and etched cross-section on the right.

The results of EDS analyses are presented for materials Alloy 152 (KAIST mock-up) (Figure 12) and Alloy 52 (Alloy 52 narrow gap mock-up) (Figure 13). The chemical composition of the test block is relatively close to the nominal Alloy 152 composition in case of KAIST mock-up. However, in Alloy 52 narrow gap mock-up dilution of nickel is somewhat significant (~10% lower than nominal).

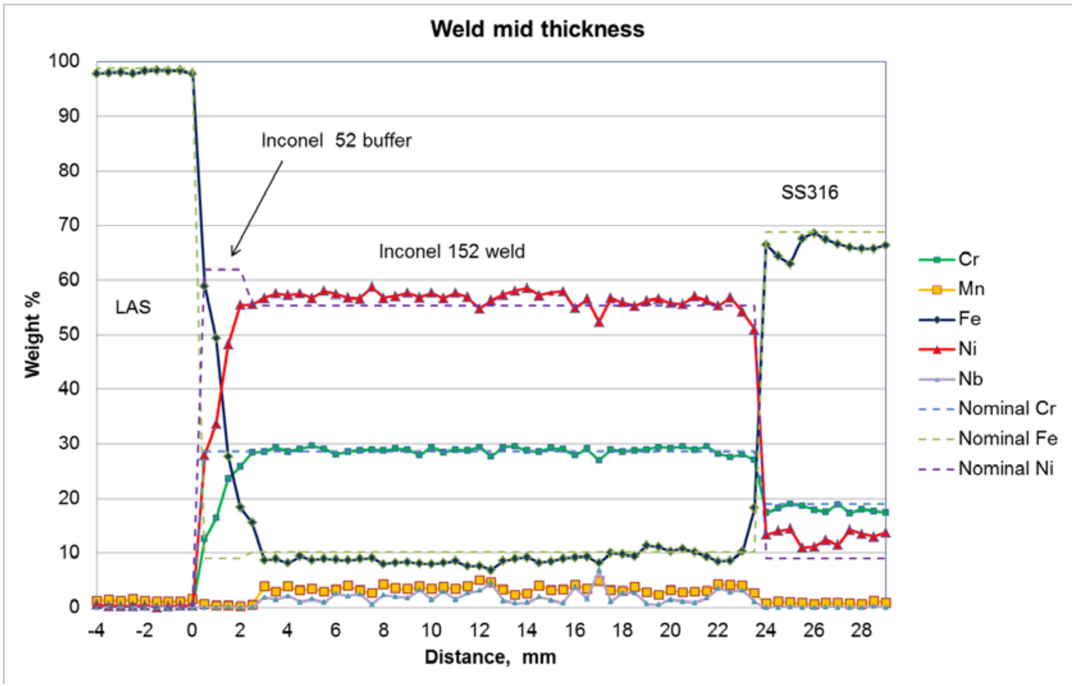

Figure 12. EDS analysis of the chemical composition profile of the DMW Alloy 152 (KAIST).

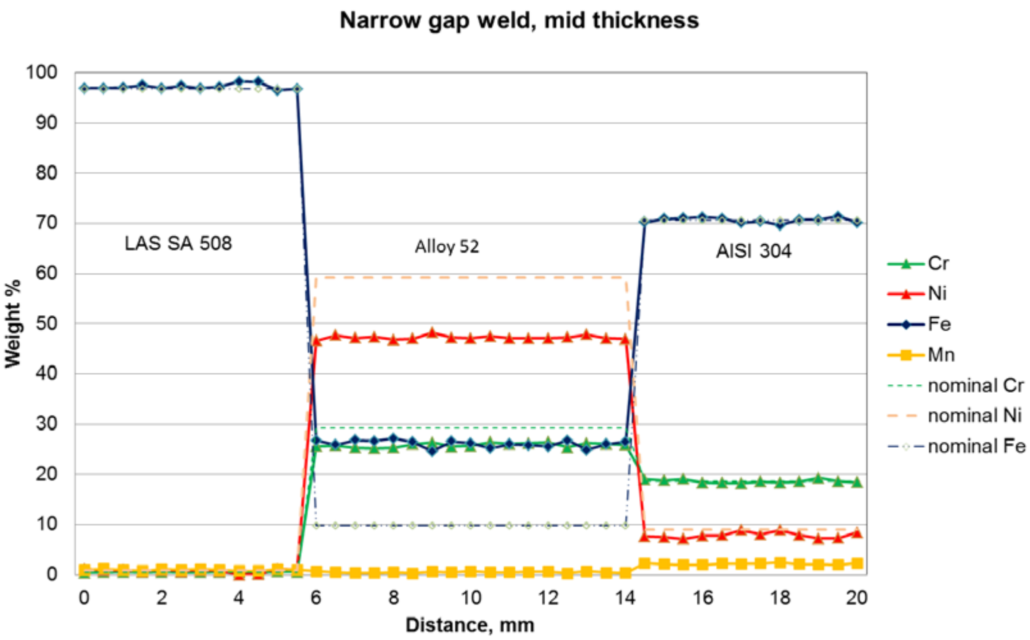

Figure 13. EDS analysis of the chemical composition profile of the DMW Alloy 52 (Alloy 52 narrow gap mock-up). The EDS analysed sample was in post weld heat treated (PWHT) condition in contrast to J-R test specimens that were tested in as-welded condition.

3. Methods

The fracture toughness tests were performed using pneumatic servo-controlled loading device and the crack growth was measured using the potential drop (PD) method. The J-R – curves were calculated according to the standard test method for measurement of fracture toughness, ASTM E 1820 – 01.

3.1 Specimens

The specimens were cut from the test blocks presented in Figure 1 (pure weld metal test blocks Alloy 182, 82, 152 and 52), Figures 3-5 (TU2 mock-up Alloy 52, TV mock-up Alloy 182 and NO mock-up Alloy 182) and Figures 9-11 (BWR mock-up Alloy 182, KAIST mock-up Alloy 152 and Aalto narrow gap weld mock-up Alloy 52). All the specimens were machined, fatigue pre-cracked to nominal 0.5 a/W and 20 % side grooved. The specimens were size 10 x 10 x 55 mm, except for specimens tested in reference [14] that were 5 x 10 x 55 mm. 1 x 1 mm slits for PD leads were machined to each specimen in both ends of the specimen and on both sides of the notch; the distance of the leads from the crack plane was 2 mm. The following specimen orientations have been used: T-S (TV and TU2) and T-L (NO) in [14], L-S in [15] and T-S in tests performed within the ENVIS project. The orientations are explained in Figure 14.The different orientations were used in order to get a sufficient amount of specimens from the available test blocks for the test series performed in the Master's Thesis and in the DEFSPEED project.

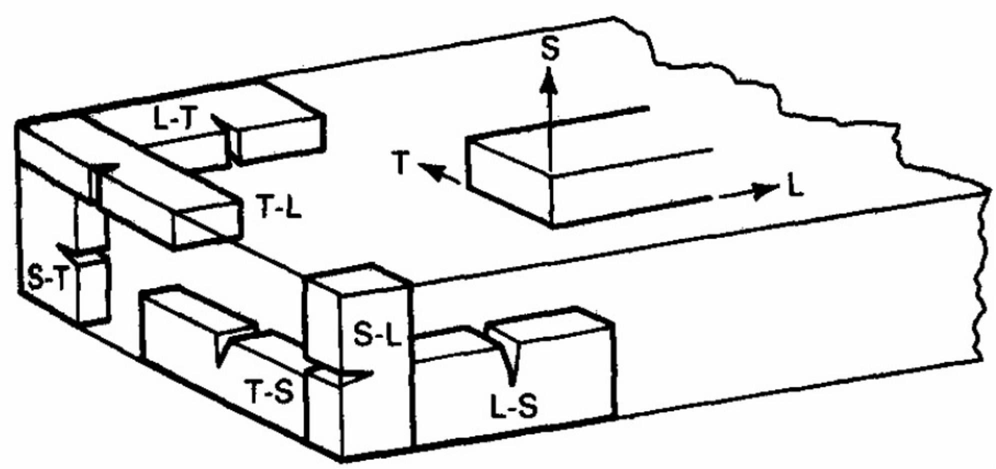

Figure 14. Schematic picture showing specimen orientations longitudinal (L), width (T) and short transverse (S). Longitudinal direction (L) corresponds to welding direction.

3.2 Environments and loading

Fracture toughness tests were performed using a pneumatic servo-controlled loading device. The tests were conducted in room temperature air and in an autoclave in hydrogenated water. The specimens were assembled to a specimen holder, and the clearance between the specimen and the bellows was originally set to be approximately 0.01 mm. The PD wires of the specimens were welded to the wires of the PD measurement equipment.

The water temperature in the autoclave tests was 55 °C, and in a few tests a 24h or 30 days pre-exposure to high temperature (300 °C) water was employed prior to loading of the specimens at 55 °C. Boric acid H_3BO_3 (200 ppm) and lithium hydroxide LiOH (2.1 ppm) was added in the water in all the tests.

4. Results

Results of J-R tests performed during the Master's thesis work, DEFSPEED and ENVIS projects are presented below. All results obtained thus far are collated in Table 2.Tearing moduli are calculated at crack growth Δa=0.5 mm, so it was performed only for the test results where Δa reached the value 0.5 mm. Average fracture toughness results for each material are presented in Figures 15-18. Fracture toughness results are also presented for each material in Figures 19-22.

*Table 2. A summary of J-R tests performed in Master's Thesis [14], DEFSPEED [15] and ENVIS thus far. Loading rate in the tests was 0.1 mm/h except for a few specimens marked with *, where the loading rate was 6.7 mm/h. Boric acid (200 ppm) and lithium hydroxide (2.1 ppm) was added in the water in all tests. Tearing moduli are calculated at crack growth Δa=0.5 mm.*

Material	Mock-up	Specimen	J_Q	Hydrogen /kg water	Tearing modulus
Alloy 182	TV	C1.1*	249	Air	199
Alloy 182	TV	C2.1	265	Air	163
Alloy 182	TV	C3.1	87	64 cc	336
Alloy 182	TV	C4.1	193	64 cc	198
Alloy 182	TV	C5.1*	236	100 cc	283
Alloy 182	TV	C6.1	232	100 cc	293
Alloy 182	TV	C7.1	93	100 cc	106
Alloy 182	NO	D1.1*	207	Air	285
Alloy 182	NO	D2.1	114	Air	104
Alloy 182	NO	D3.1*	156	100 cc	244
Alloy 182	NO	D4.1	40	100 cc	73
Alloy 182	Pure weld metal	A1	182	Air	212
Alloy 182	Pure weld metal	A8	84	30 cc	177
Alloy 182	Pure weld metal	A9	78	30 cc	124
Alloy 182	Pure weld metal	A2	53	100 cc	105
Alloy 182	Pure weld metal	A3	42	100 cc	98
Alloy 182	Pure weld metal	A5	59	24h pre-exp 100 cc	151
Alloy 182	Pure weld metal	A6	63	24h pre-exp 100 cc	138
Alloy 182	Pure weld metal	A7	69	24h pre-exp 100 cc	63
Alloy 182	Aalto	A11	40	30 cc	98
Alloy 182	Aalto	A12	73	30 cc	160
Alloy 182	Aalto	A13	60	30d pre-exp 30 cc	218
Alloy 182	Aalto	A14	93	30d pre-exp 30 cc	193
Alloy 82	Pure weld metal	B1	>300	Air	-
Alloy 82	Pure weld metal	B8	252	30 cc	391
Alloy 82	Pure weld metal	B9	102	30 cc	217

Table 2 continues.

Material	Mock-up	Specimen	J_Q	Hydrogen /kg water	Tearing modulus
Alloy 82	Pure weld metal	B2	100	100 cc	112
Alloy 82	Pure weld metal	B3	57	100 cc	280
Alloy 82	Pure weld metal	B4	155	100 cc	95
Alloy 82	Pure weld metal	B5	47	24h pre-exp 100 cc	173
Alloy 82	Pure weld metal	B6	70	24h pre-exp 100 cc	155
Alloy 82	Pure weld metal	B7	100	24h pre-exp 100 cc	203
Alloy 152	Pure weld metal	C1	304	Air	-
Alloy 152	Pure weld metal	C8	108	30 cc	190
Alloy 152	Pure weld metal	C9	151	30 cc	-
Alloy 152	Pure weld metal	C2	124	100 cc	284
Alloy 152	Pure weld metal	C3	113	100 cc	270
Alloy 152	Pure weld metal	C5	260	24h pre-exp 100 cc	-
Alloy 152	Pure weld metal	C6	94	24h pre-exp 100 cc	151
Alloy 152	Pure weld metal	C7	141	24h pre-exp 100 cc	196
Alloy 152	KAIST	C10	196	Air	293
Alloy 152	KAIST	C11	197	Air	325
Alloy 152	KAIST	C12	192	0 cc	416
Alloy 152	KAIST	C13	109	0 cc	229
Alloy 152	KAIST	C14	174	5 cc	280
Alloy 152	KAIST	C15	123	5 cc	234
Alloy 152	KAIST	C16	165	5 cc	282
Alloy 152	KAIST	C17	96	30 cc	249
Alloy 152	KAIST	C18	169	30 cc	258
Alloy 152	KAIST	C19	128	30 cc	252
Alloy 152	KAIST	C20	149	30 cc	262
Alloy 152	KAIST	C21	189	30d pre-exp 30 cc	71
Alloy 152	KAIST	C22	169	30d pre-exp 30 cc	187
Alloy 52	Pure weld metal	D1	>300	Air	-
Alloy 52	Pure weld metal	D8	>300	30 cc	-
Alloy 52	Pure weld metal	D9	>300	30 cc	-
Alloy 52	Pure weld metal	D2	54	100 cc	200
Alloy 52	Pure weld metal	D3	47	100 cc	256
Alloy 52	Pure weld metal	D4	85	100 cc	135
Alloy 52	Pure weld metal	D6	35	24h pre-exp 100 cc	221
Alloy 52	Pure weld metal	D5	127	24h pre-exp 100 cc	385
Alloy 52	Pure weld metal	D7	48	24h pre-exp 100 cc	121
Alloy 52	Aalto NGW	D14	>300	30 cc	-
Alloy 52	Aalto NGW	D13	>300	30 cc	-
Alloy 52	TU2	B5.1	>300	100 cc	-
Alloy 52	TU2	B4.1*	>300	100 cc	-
Alloy 52	TU2	B3.1	>300	64 cc	-
Alloy 52	TU2	B2.1	>300	Air	-
Alloy 52	TU2	B1.1*	>300	Air	-

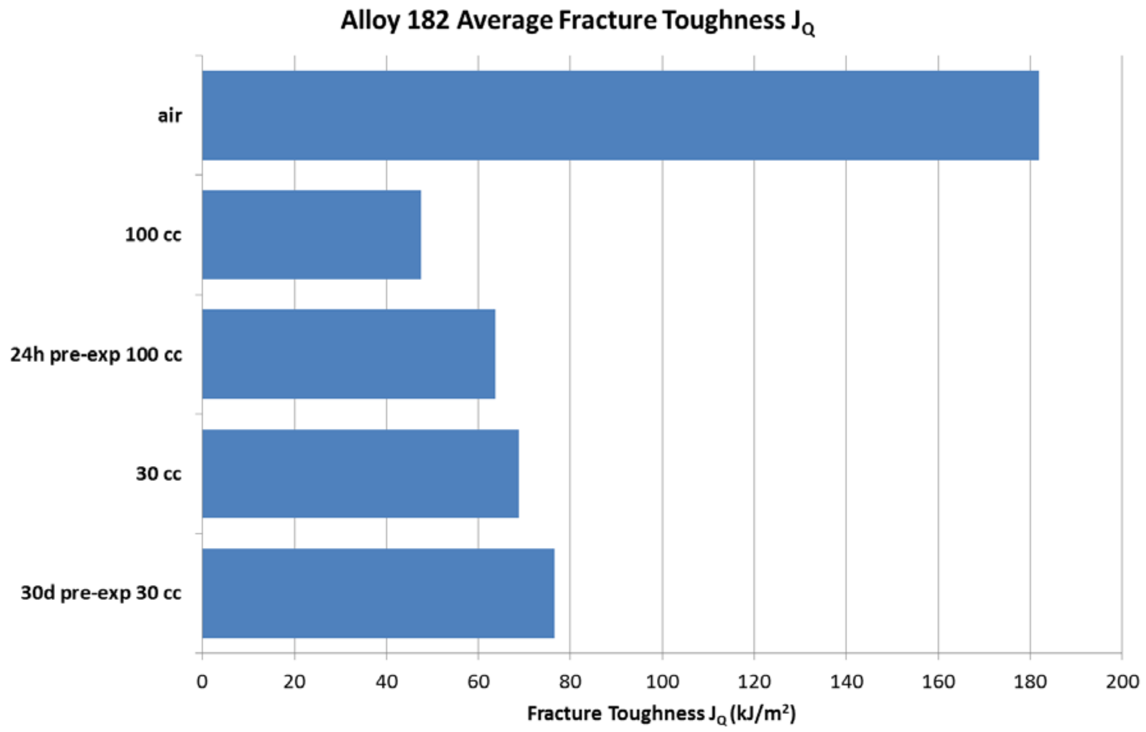

Figure 15. Average fracture toughness (J_Q) values for Alloy 182 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O, with and without high temperature (300 ˚C) pre-exposure.

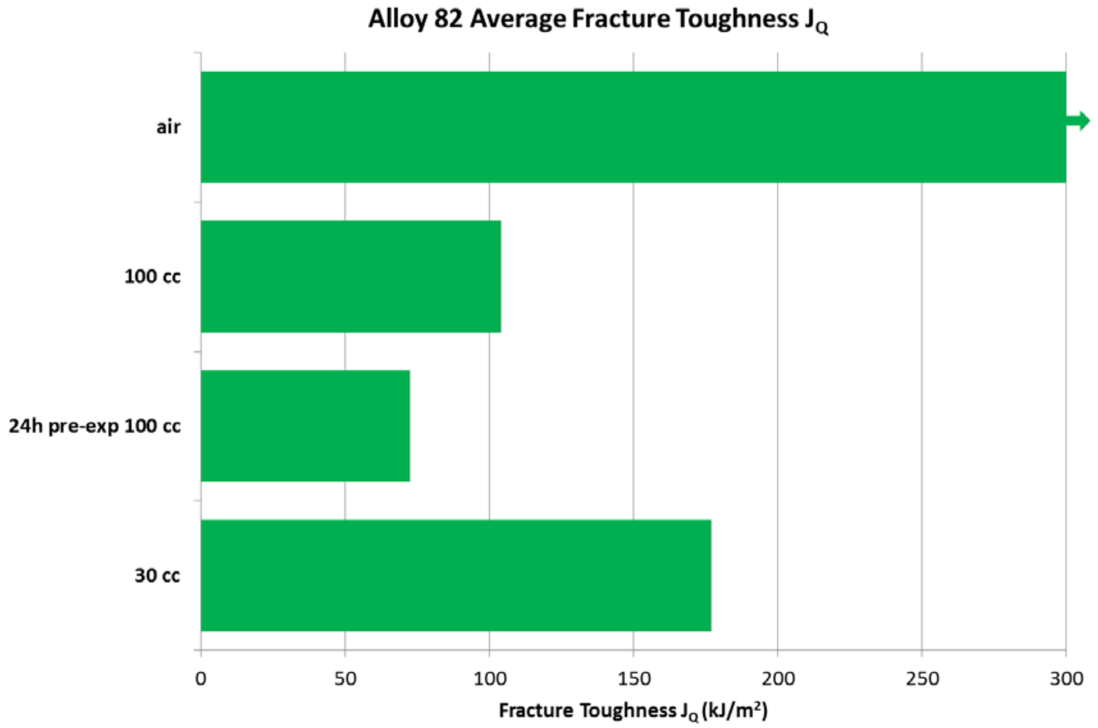

Figure 16. Average fracture toughness (J_Q) values for Alloy 82 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O and with high temperature (300 ˚C) pre-exposure.

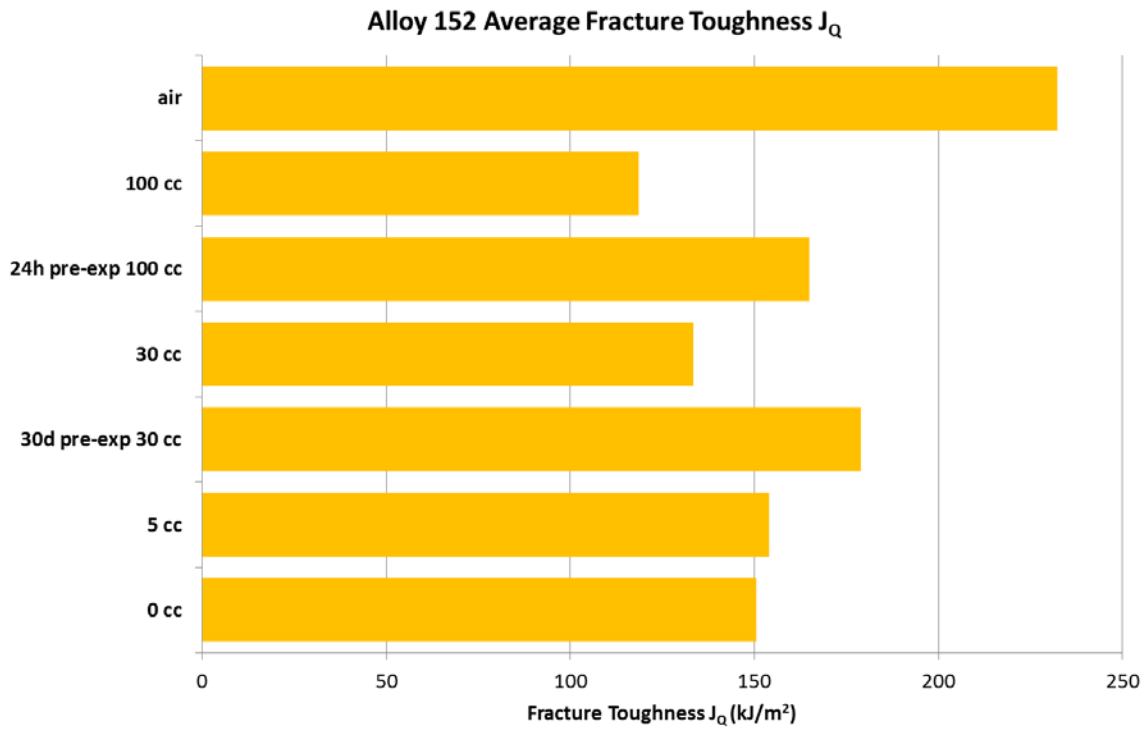

Figure 17. Average fracture toughness (J_Q) values for Alloy 152 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with various hydrogen contents and with high temperature (300 ˚C) pre-exposure.

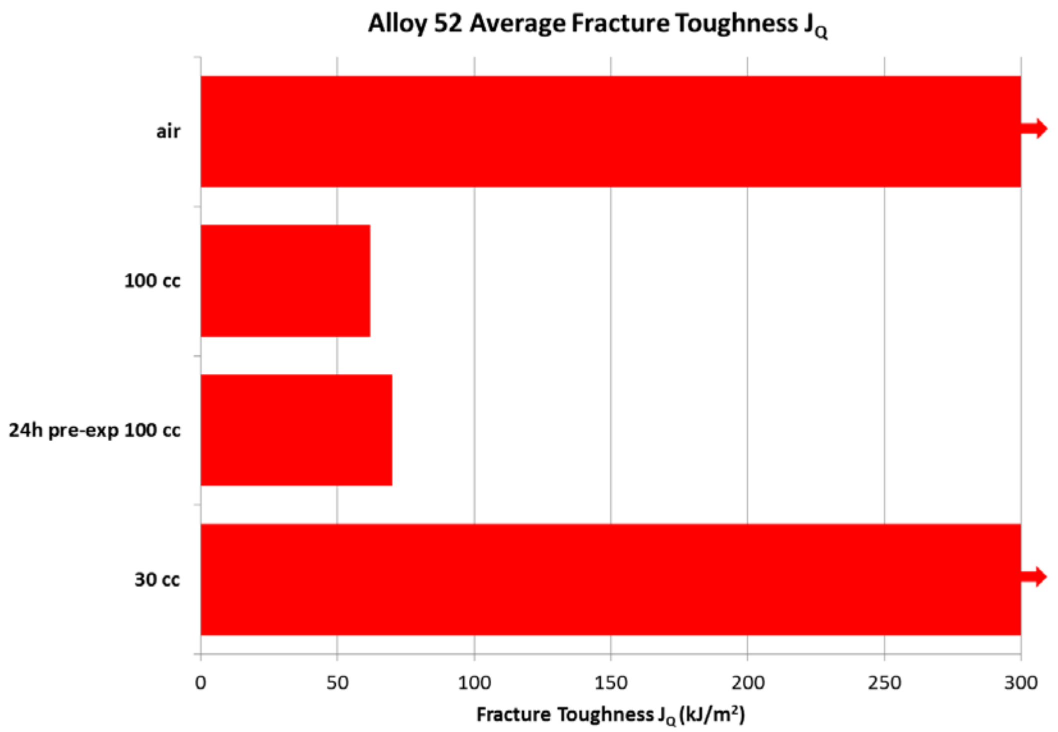

Figure 18. Average fracture toughness (J_Q) values for Alloy 52 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O and with high temperature (300 ˚C) pre-exposure.

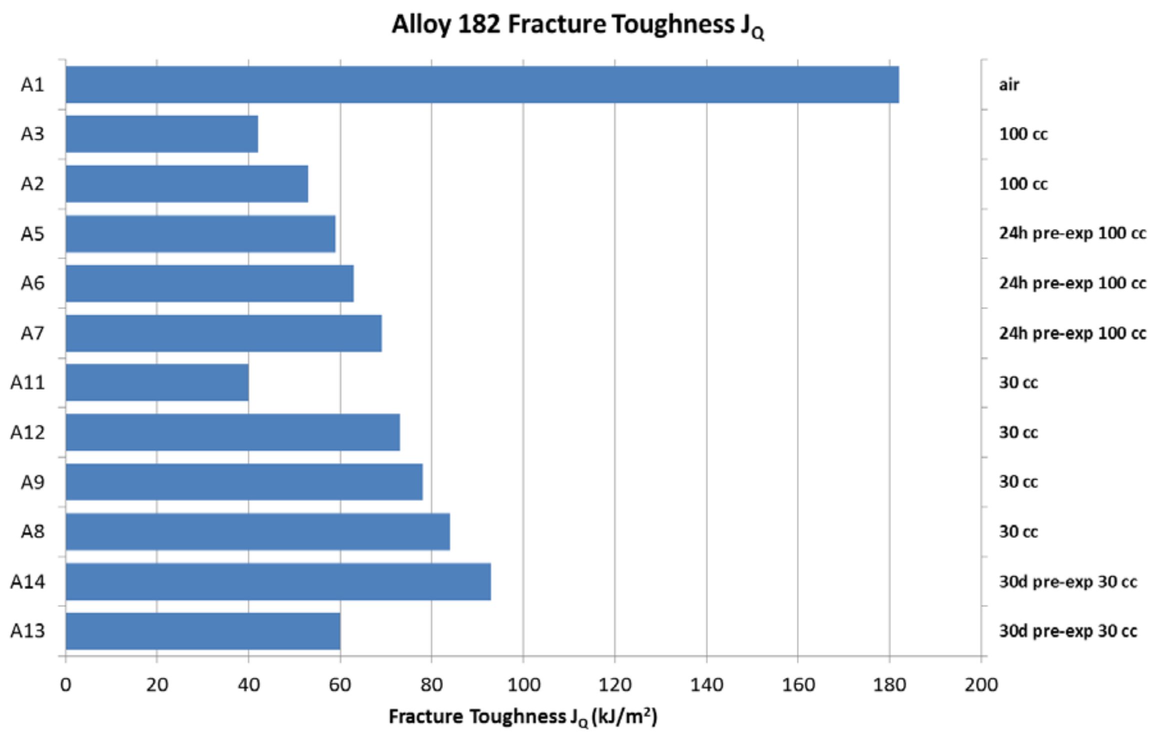

Figure 19. Fracture toughness (J_Q) values for Alloy 182 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O, with and without high temperature (300 ˚C) pre-exposure.

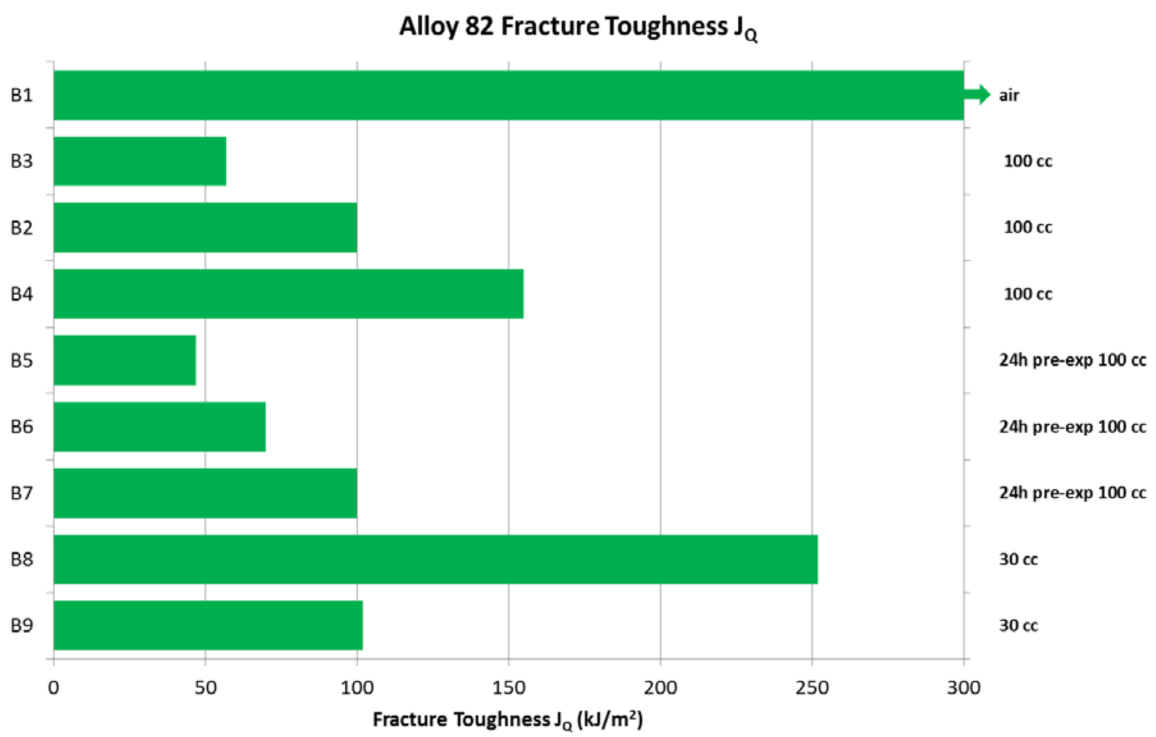

Figure 20. Fracture toughness (J_Q) values for Alloy 82 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O and with high temperature (300 ˚C) pre-exposure.

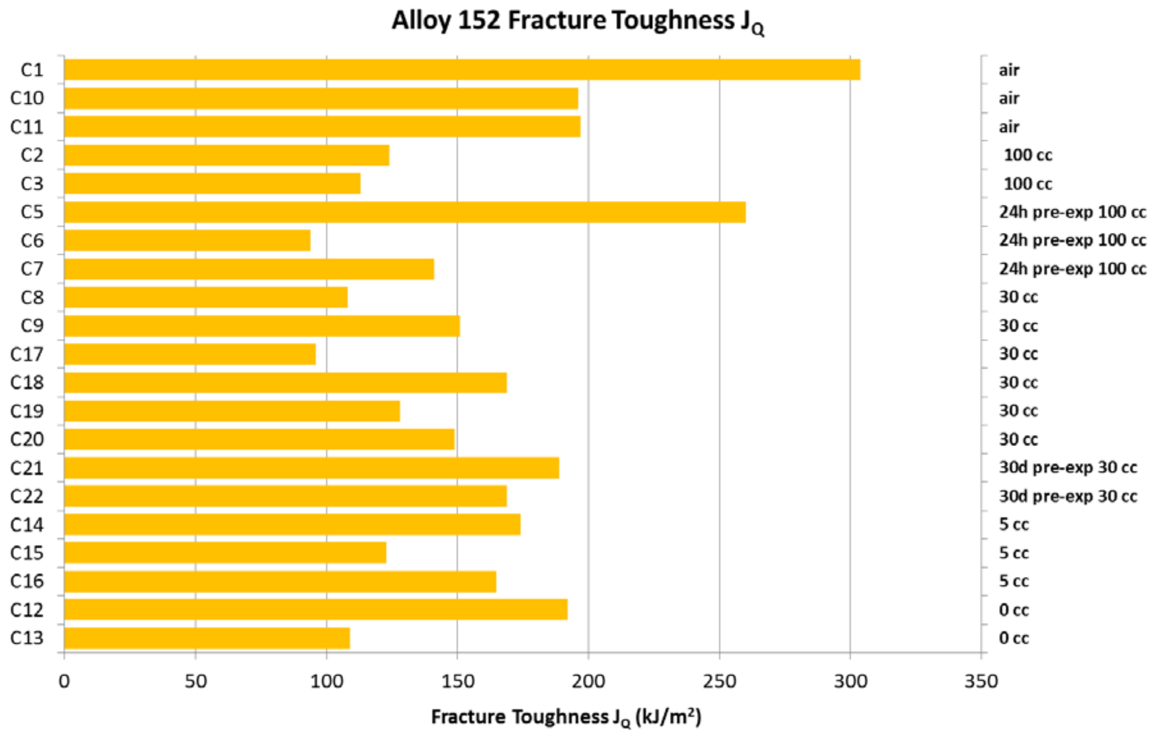

Figure 21. Fracture toughness (J_Q) values for Alloy 152 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with various hydrogen contents and with high temperature (300 ˚C) pre-exposure.

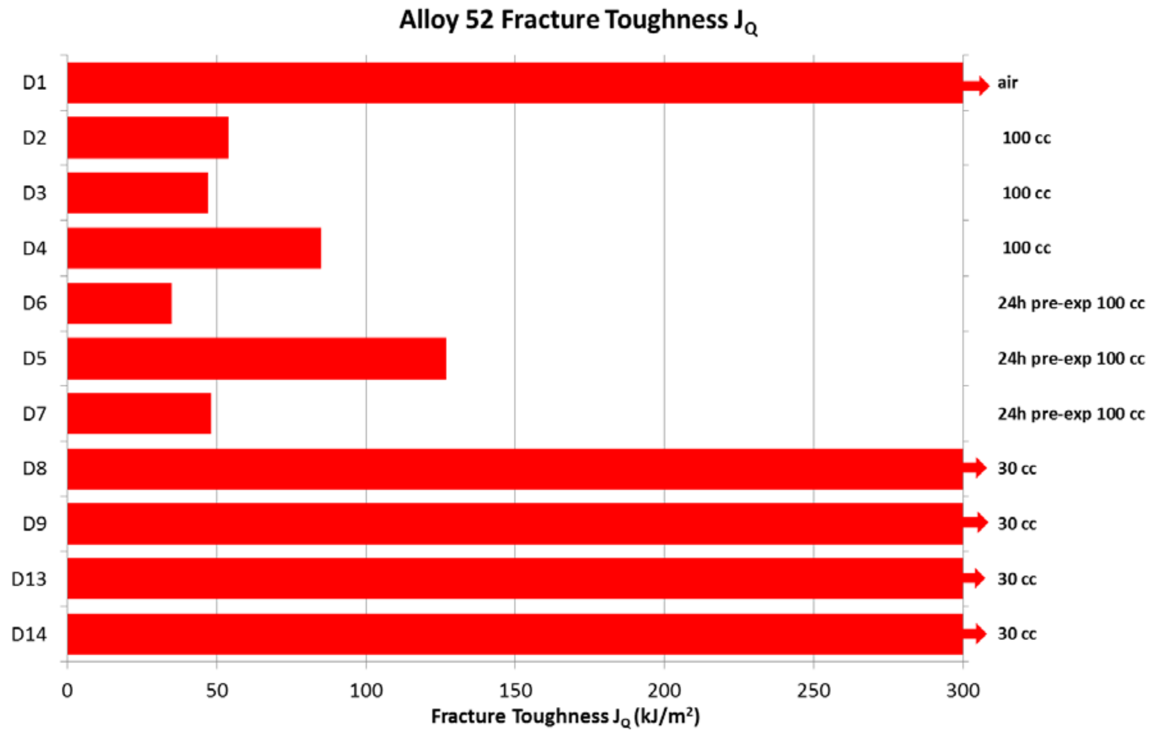

Figure 22. Fracture toughness (J_Q) values for Alloy 52 tested in 55 ˚C water (200 ppm H_3BO_3 and 2.1 ppm LiOH) with hydrogen contents of 100 and 30 cm^3 H_2/kg H_2O and with high temperature (300 ˚C) pre-exposure.

5. Discussion and Conclusions

The J-R test results for Alloys 182, 82, 152 and 52 show that the fracture toughness (J_Q) values decrease due to hydrogenated low temperature water in all the tested materials. A general observation is that Alloy 182 is the most susceptible material to LTCP of the nickel-based weld metals, whereas Alloy 52 retains its high toughness at least up to 30 cm^3 H_2/kg H_2O. Alloy 152 seems to have the highest J_Q values in very high hydrogen contents (100 cm^3 H_2/kg H_2O) and it has the smallest relative reduction of J_Q.

This study shows that increasing hydrogen content reduces the fracture toughness of nickel-based weld metals Alloy 182, 82, 152 and 52. Figures showing average fracture toughness values (18-21) illustrate this phenomenon. In addition, the results for Alloys 182, 152 and 52 indicate that pre-exposure to high temperature water slightly increases the J_Q values when compared to the results obtained in corresponding environment without pre-exposure. However, Alloy 82 behaved in an opposite manner, and the reason for that remains unclear. The diverging behaviour of Alloy 82 may possibly be explained by scattering.

A total of 69 J-R test results are collated in this report. The number of environments and different specimen orientations together with scattering J_Q values make it somewhat difficult to draw straightforward conclusions on every test series. Especially in the Master's thesis work where the specimen size was small (5 x 10 x 55 mm) the scattering was significant. A weld is always inhomogeneous regarding its mechanical and microstructural properties, which plays a crucial role in hydrogen induced cracking phenomena. However, the considerably extensive amount of test data justifies general comparisons between the tested weld metals and applied hydrogen contents.

The work continues with further tests and more detailed investigations on the role of the microstructures on the LTCP behaviour.

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