Technical Report

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Creep testing of thick-wall copper electron beam and friction stir welds at 75, 125 and 175°C

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Abstract

Thick section copper canisters are planned to be used as a corrosion protection of nuclear waste disposal containers for long term underground deposit in Sweden. The copper canisters will have the top and possibly the bottom lid welded to the canister walls using electron beam or friction stir welding. Due to the high external hydrostatic pressure and the relatively high temperature of the waste during the first one hundred years the copper will creep. The creep process will close the manufacturing gap between the cast iron container and the copper canister. The creep ductility must be sufficient to avoid cracking of the weld and other parts of the canister.

Specimens cut from the friction stir welds and the electron beam welds have been creep tested at temperatures ranging from 75 to 175°C. Cross-weld specimens were used for both friction stir and electron beam welds. Weld metal, heat affected zone and base metal were also studied for friction stir welds. The results for the electron beam welds show that the main creep deformation is concentrated to the weld metal where the failure takes place. Weld metal and most cross-weld tests of friction stir weld material show similar creep lives and ductility as base metal tests. Ductility at rupture was found to exceed 30% for friction stir weld specimens, and the Norton power law exponent was determined to be between 30 and 50.

Contents

1 Introduction

The planned method for disposal of spent nuclear fuel in Sweden is to encapsulate the fuel and then deposit it deep in the bedrock. An inner cast iron canister is supposed to give sufficient strength to withstand the hydrostatic pressure in the deep depository. An outer canister made of copper should give corrosion protection for the required time of at least 100,000 years /1/. 30 to 50 mm thick copper is considered. The canisters will be produced either as extruded tube /2/ onto which copper lids are welded, or dorn pressed with an integral bottom onto which only the lid is welded.

The copper chosen is phosphorous doped and oxygen free (Cu-OFP). The creep properties of this material and similar alloys have been studied in several investigations /3, 4, 5, 6, 7/. To obtain sufficient creep ductility the oxygen free copper has to be doped with about 50 ppm phosphorous /8/. Work has also been performed on the recrystallisation and extrusion properties of the same chemical composition copper /9, 10/.

There is initially a gap of about 2 mm between the iron canister and the copper shell. This gap will close slowly during the first 200 years due to the hydrostatic pressure from the water on the outside of the deposited canister. This deformation should take place without cracks being formed in the copper. The estimated temperature of the spent fuel during this timeframe, ca 80–90°C, is high enough to activate creep in the copper. The gap will thus close given that the creep ductility of the material is high enough. Of special consideration are the lid welds. It is imperative that the welds have sufficient creep ductility to be able to deform without cracking. The maximum strain in the copper is 5% /1/. Translated to a multiaxial stress state it can be estimated that a uniaxial creep ductility of at least 10% is needed to avoid cracking. The copper and the welds must exhibit at least this creep ductility in testing. The temperature will decrease to the level of the surrounding bedrock, 15°C, during the first thousand years of the containment /1/.

Weld processes considered for the lid weld include electron beam and friction stir welding. The welding is complicated by the need for a process that can be remotely controlled since the material being encapsulated is highly radioactive. The aim of the work presented in this paper is to evaluate the creep properties of experimental welds made using electron beam and friction stir welding processes. An abridged version of this report has been published at the Materials Research Society’s spring meeting in San Francisco in April 2004 /11/.

2 Experimental

Material from two welds was used in this investigation. The electron beam weld had SKB internal identification number L010 and the friction stir weld the identification number CW65. A more complete report on the weld microstructure can be found in /12/. The average grain size was measured on the copper welds before testing and found to be about 2,000 µm for the electron beam welds and 75 µm for the friction stir welds. Knowing the average grain size, calculations were performed of the Petch-Hall effect. The result was that just taking the grain size into account, the room temperature yield strength is 15 MPa lower for the electron beam welds compared to the friction stir welds.

From one weld, manufactured using electron beam welding, specimens were cut in a cross weld position in such a way that both weld metal, heat affected zones and base material were included in the gauge length of the material, Figure 2-1. These specimens were creep tested at 75, 125 and 175°C. From a friction stir weld, specimens were cut in a cross-weld position similar to the one used for the electron beam weld. Since it was known that this type of welds has different microstructural properties at the top and the bottom, specimens were extracted from both of these positions. In addition, specimens were cut longitudinally along the weld metal and the heat affected zone, as well as from the unaffected base metal. Specimens from the friction stir welds were also tested at 75, 125 and 175°C.

Care was taken during the manufacture of the specimens to minimise the introduction of cold working, but no anneal was applied to the material either before or after the manufacturing step.

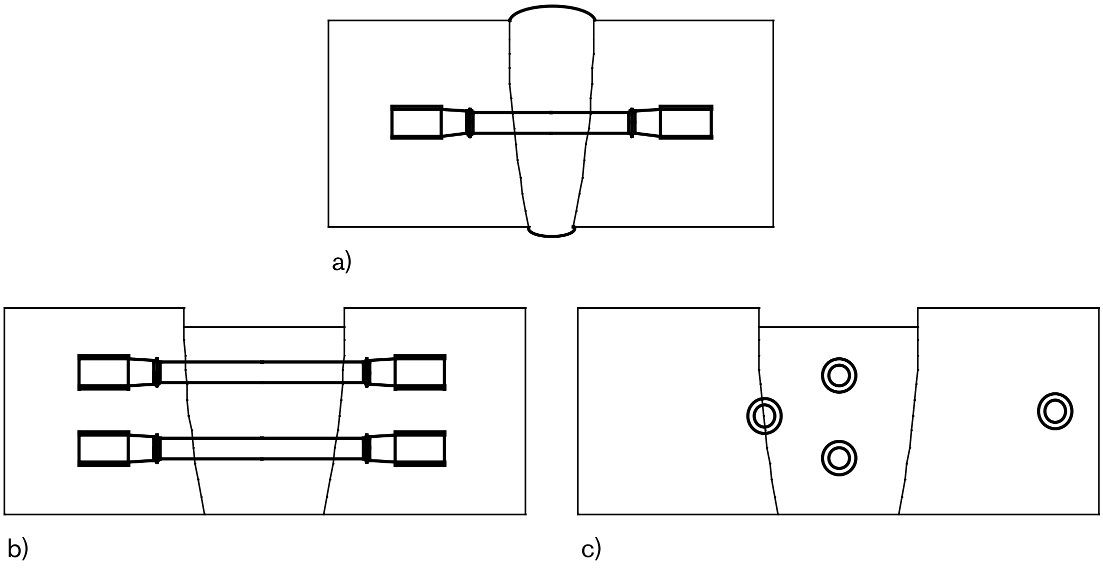


Figure 2-1. *Specimen extraction positions. a) Cross-weld positions EB, b) Cross-weld positions FSW, c) Longitudinal positions FSW.*

3 Results

3.1 Initial loading strain results

When the test is started the specimen is placed inside the furnace and heated to test temperature without any load applied. After the test temperature has been reached the specimen is allowed to soak for a few hour to guarantee that the specimen has reached a stable temperature and then the load is applied. For practical reasons the load is applied stepwise but the full load is to be applied within 3 minutes from the start of the loading. During this initial loading the strain is measured and logged, Figure 3-1.

It is shown in Figure 3-1 that the initial strain is dependent on the applied load in a linear manner. The initial strain is in most cases in excess of 6% with the exception of the parent metal specimens, which show both a lower initial strain and a less pronounced linear relationship. The reason for this is not apparent but might be connected to the amount of annealing the material is subjected to. The friction stir weld metal is annealed during the welding operation but the parent metal is in this case not annealed after the rolling operation that formed the initial tube. This could result in the parent metal being slightly harder before testing than the weld specimens and thus showing less initial strain.

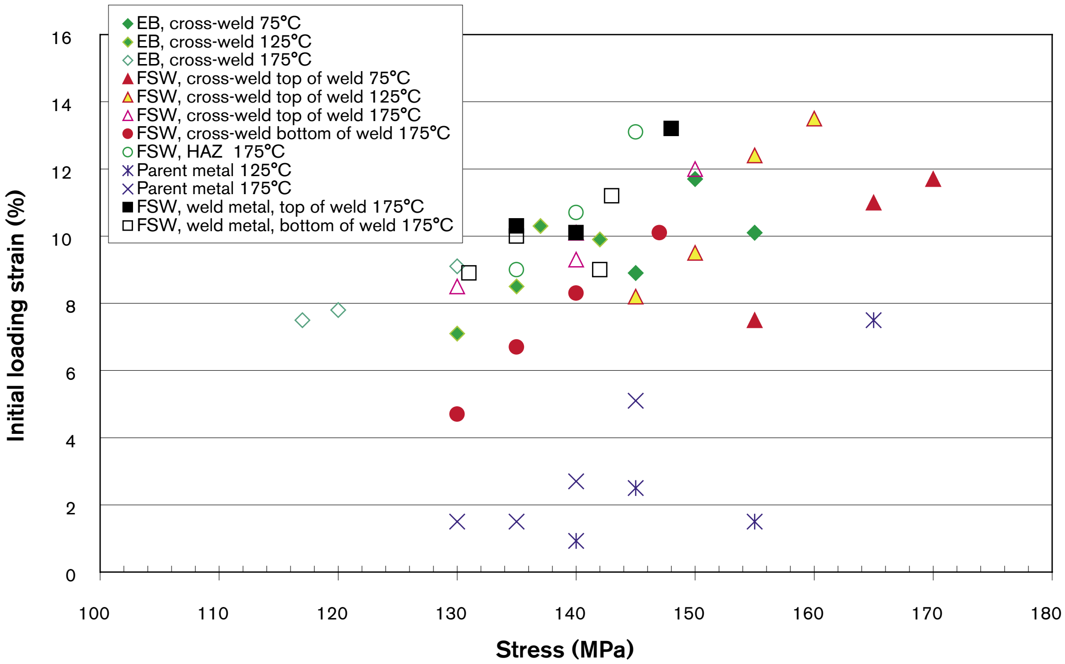


Figure 3-1. *Initial strain versus stress for all the specimens tested in this work.*

3.2 Creep test results

The results from the creep testing are given in Table 3-1. The results from the electron beam weld testing are shown in Figure 3-2 and the results for friction stir weld specimens in Figure 3-3. The electron beam welds have significantly shorter creep lives than the friction stir welds for the same applied stress. This difference is about two orders of magnitude in time at the three investigated temperatures. The results for the friction stir welds are similar for different microstructures. Cross-weld, heat affected zone, weld metal and parent metal gave about the same creep lives. The position of the rupture in the cross-weld specimens varied between the weld metal and the heat affected zone.

Table 3-1. Results from creep testing. Results in brackets are the last values from interrupted tests.

Specimen	Temp [°C]	Stress [MPa]	Time [h]	Elongation [%]	Red. of area [%]	Min. creep rate [%/h]	Status
EB, cross-weld 75°C							
1-A-1	75	155	180	26.2	80.7	0.01667	
1-B-2	75	145	13,708	(4.8)	–	(0.00020)	Interrupted
1-C-3	75	150	1,688	30.9	78.1	0.00190	
EB, cross-weld 125°C							
2-120-A	125	130	9,514	25.0	66.5	0.00016	
2-100-B	125	135	2,251	27.8	66.6	0.00150	
2-170-C	125	142	39	30.3	87.5	0.09035	
2-005-D	125	137	527	30.1	86.7	0.00638	
EB, cross-weld 175°C							
3-290-B	175	130	44	28.8	84.3	0.08050	
3-310-C	174	120	1,158	28.1	71.2	0.00175	
3-230-D	175	117	1,474	24.6	64.5	0.00270	
FSW, cross-weld top of weld 175°C							
4-1-A	175	150	40	38.3	87.5	0.16780	
4-2-B	175	140	2,016	40.1	81.1	0.00128	
4-8-C	175	140	619	39.1	80.9	0.01259	
4-3-D	175	130	10,875	39.3	72.8	0.00115	
FSW, cross-weld bottom of weld 175°C							
5-2-A	175	140	635	37.8	78.6	0.01283	
5-1-B	175	130	12,787	31.7	40.5	0.00090	
5-3-C	175	135	4,606	37.8	75.2	0.00250	
5-4-D	175	147	45	35.0	85.6	0.23000	
FSW, cross-weld top of weld 125°C							
6-5-A	125	150	8,430	37.6	85.4	0.0005	
6-2-B	125	145	10,566	(12.0)	–	(0.0004)	Interrupted
6-3-C	125	155	1,150	41.5	85.7	0.0017	
6-1-D	125	160	150	42.3	88.3	0.0322	
FSW, cross-weld top of weld 75°C							
7-1-A	75	155	8,593	(6.7)	–	(0.0001)	Interrupted
7-2-B	75	165	7,122	35.1	86.8	0.0004	
7-3-C	75	170	660	36.5	86.8	0.00420	

Specimen	Temp [°C]	Stress [MPa]	Time [h]	Elongation [%]	Red. of area [%]	Min. creep rate [%/h]	Status
FSW, HAZ 175°C							
8-2-A	175	135	6,272	54.1	80.9	0.00280	
8-3-B	175	140	1,133	52.1	85.3	0.01030	
8-4-C	175	145	211	48.9	88.6	0.04699	
FSW, weld metal, top of weld 175°C							
9-2-A	175	135	10,754	52.9	79.7	0.00320	
9-3-B	175	140	2,311	52.3	75.0	0.00760	
9-4-C	175	148	90	52.6	84.1	0.19000	
FSW, weld metal, bottom of weld 175°C							
10-2-A	175	135	1,113	36.4	48.6	0.00648	
10-3-B	175	131	6,106	52.7	68.9	0.00180	
10-4-C	175	142	719	30.8	52.9	0.01081	
10-5-B	175	143	174	35.1	53.7	0.04041	
Parent metal 125°C							
11-2-A	125	140	15,818	(2.6)	–	–	Interrupted
11-3-B	125	145	11,275	(5.3)	–	–	Interrupted
11-4-C	125	155	7,148	(11.1)	–	(0.00027)	Interrupted
11-1-D	125	165	1,657	36.6	89.8	0.00102	
Parent metal 175°C							
12-2-A	175	130	20,762.6	18.3	–	(0.00031)	Interrupted
12-3-B	175	135	14,681	45.2	80.2	0.00048	
12-4-C	175	145	162	38.9	85.7	0.05427	
12-1-D	175	140	1,622	42.0	82.6	0.00592	

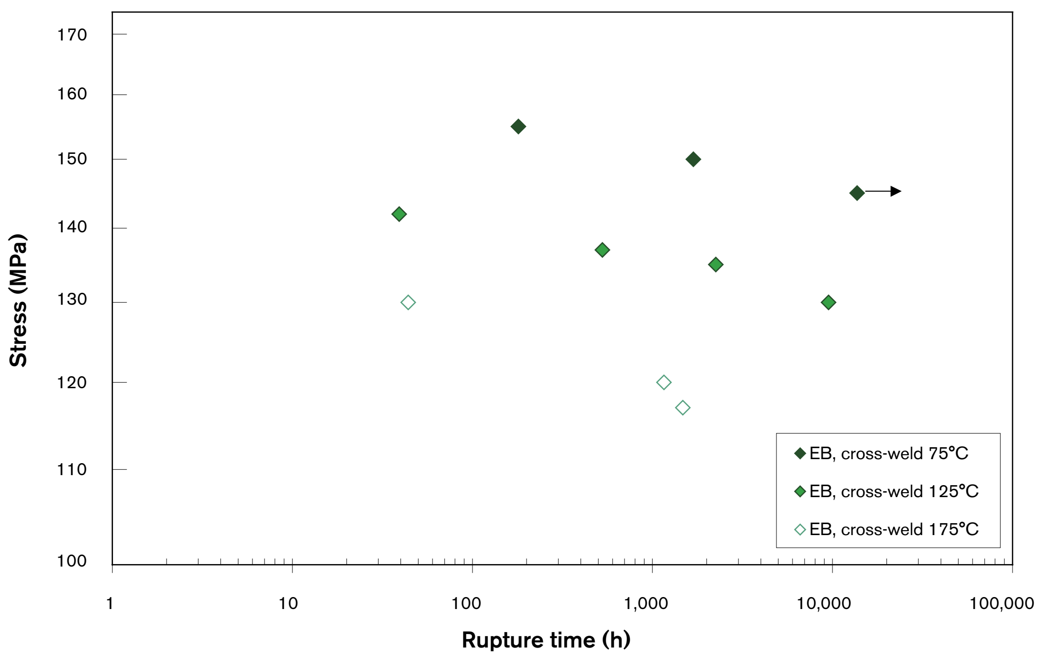


Figure 3-2. *Creep rupture time versus applied stress for electron beam weld specimens at 75, 125 and 175°C.*

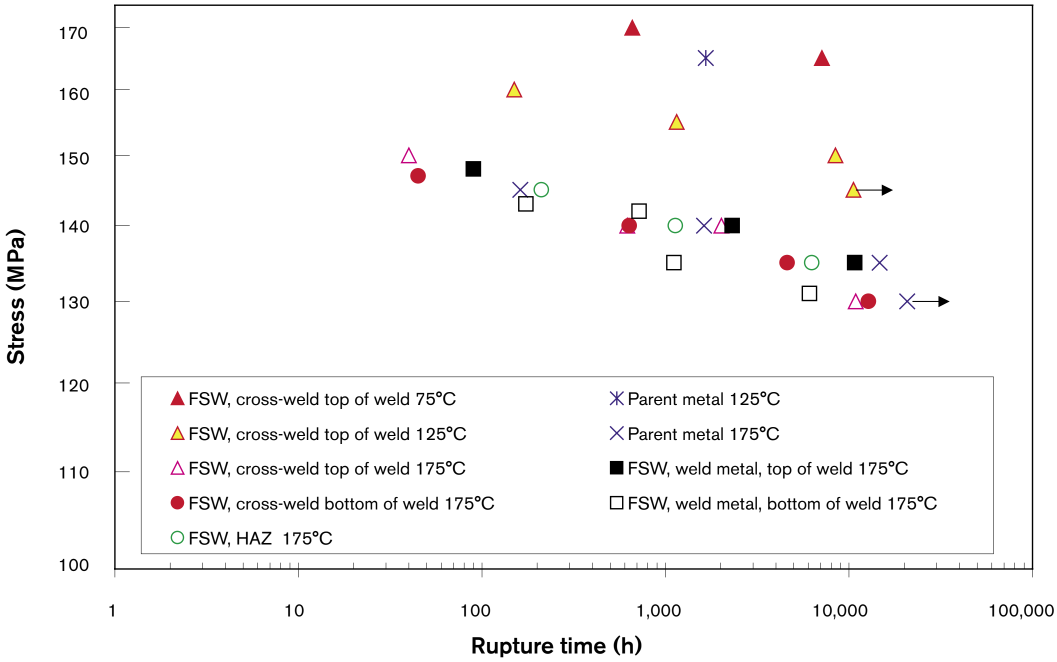


Figure 3-3. *Creep rupture time versus applied stress for friction stir weld specimens.*

In Figure 3-4 and Figure 3-5 the minimum creep rate versus stress for electron beam welds and friction stir welds are shown respectively. The Norton exponent, n, in the power law creep equation (1) was found to be quite high, between 30 and 50.

$$\dot{\varepsilon}_{min} = B\sigma^{n} \qquad (1)$$

where $\dot{\varepsilon}_{min}$ is the minimum creep rate, σ is the applied stress and B and n are constants.

Creep ductility was measured as elongation at rupture and reduction of area at rupture, Figure 3-6 and Figure 3-7. The creep elongation was found to be above 30% for all friction stir weld specimens and 20–30% for the electron beam weld specimens. Also for the area reduction the values are higher for friction stir weld specimens than for electron beam specimens. Most friction stir weld specimens exhibited area reductions of more than 60%. In all cross-weld friction stir specimens the creep deformation was almost uniform over the whole gauge length with only a slight tendency for necking in the heat affected zone.

Longitudinal weld metal taken from the bottom part of friction stir welds has a creep life that is about half of that for the other FSW specimens tested at 175°C and also shows a more brittle rupture. This can be seen in Figure 3-8 where in a) the typical appearance of the rupture in a cross-weld is shown and in b) the more brittle appearance of a rupture in the bottom of the weld metal. The area reduction measured on specimens from the bottom part of the weld was between 40 and 50%.

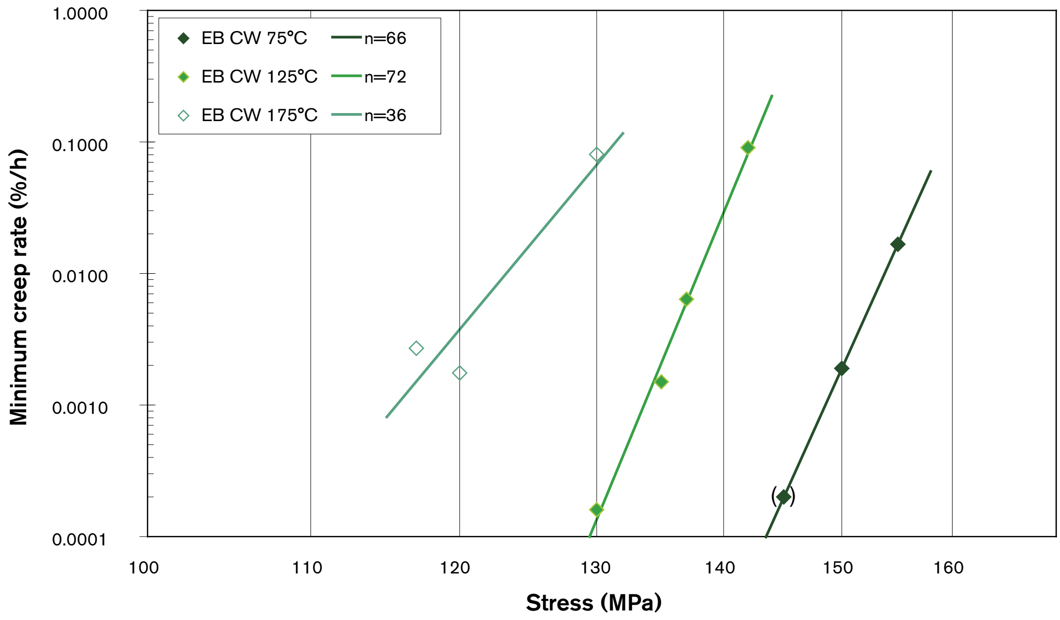


Figure 3-4. *Norton plots for the electron beam weld tests. Data points in parenthesis are estimated from interrupted tests.*

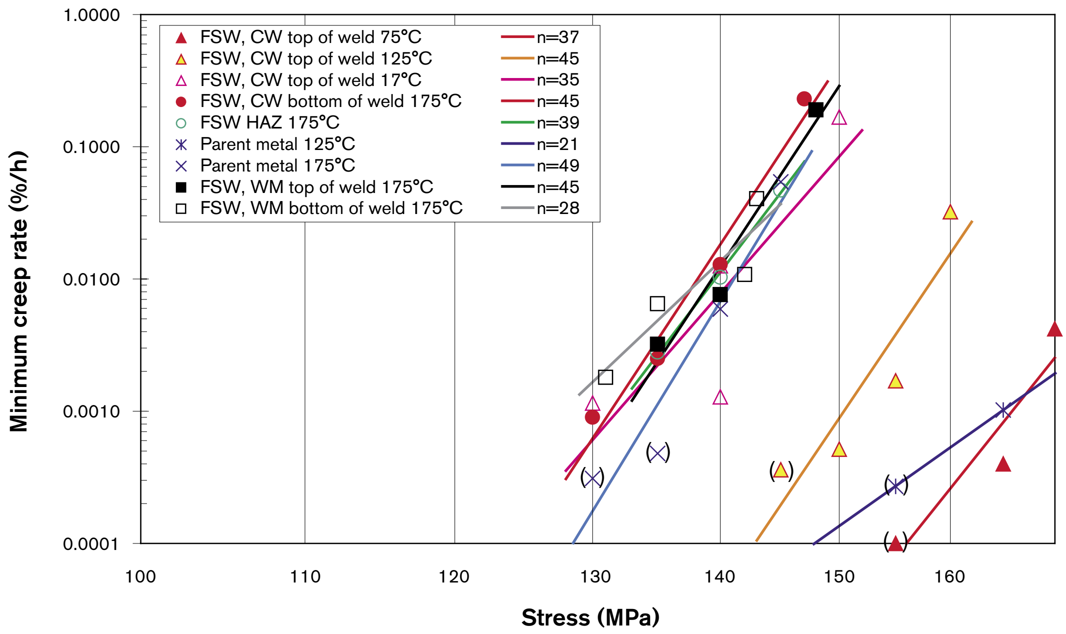


Figure 3-5. *Norton plots for the friction stir weld tests. Data points in parenthesis are estimated from interrupted tests.*

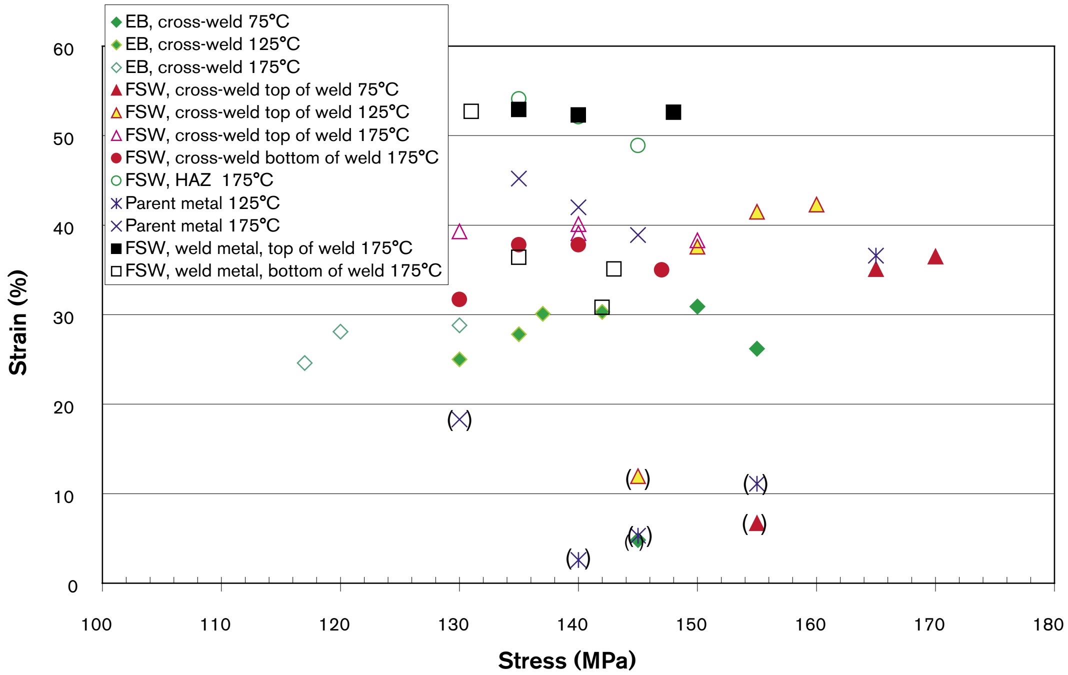


***Figure 3-6.** Elongation at rupture for both electron beam and friction stir weld tests. Data points in parenthesis are the last values from interrupted tests.*

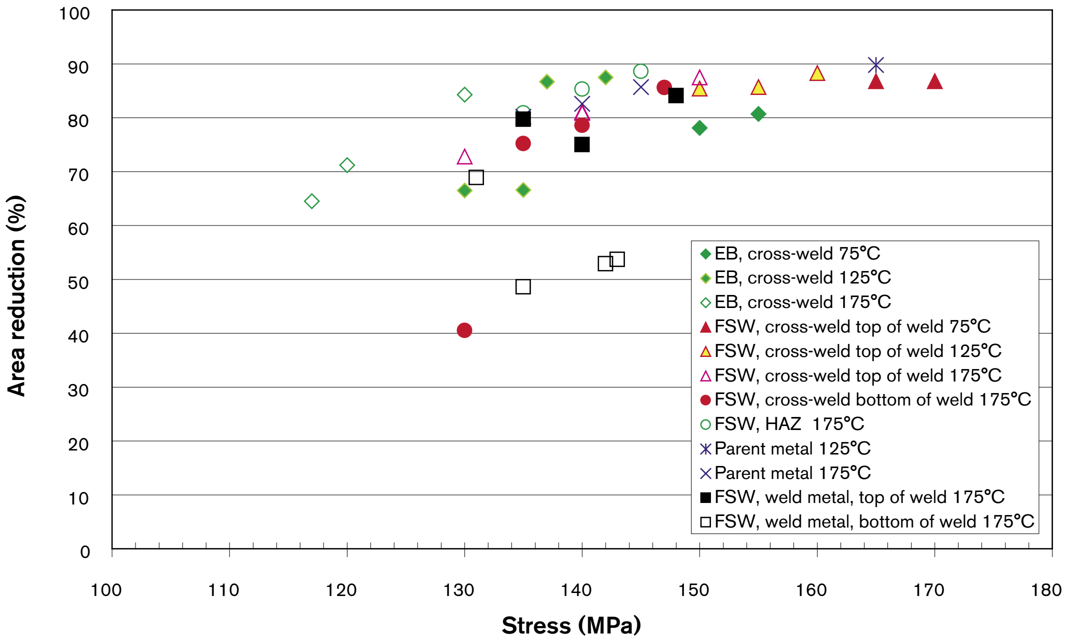
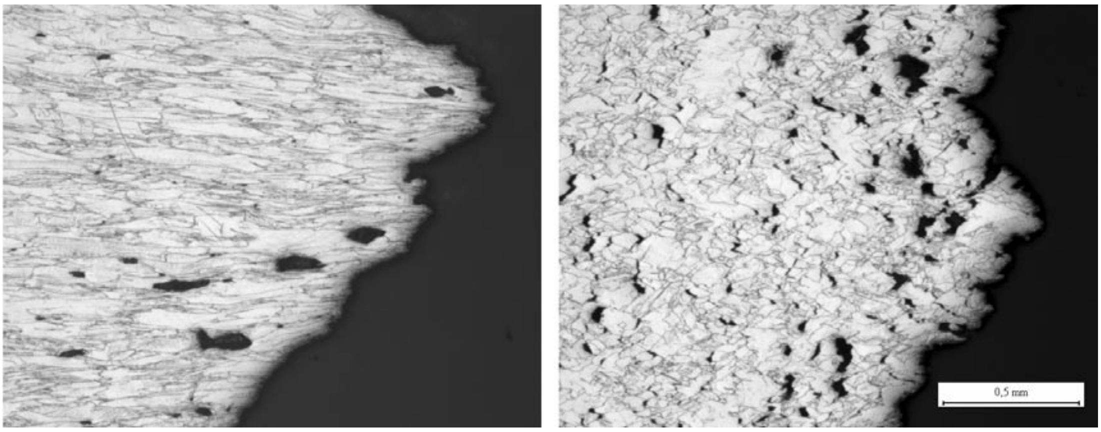


***Figure 3-7.** Reduction of area at rupture for both electron beam and friction stir weld tests.*



a) *b)*

Figure 3-8. *Micrographs of the microstructure close to the rupture. a) Typical cross weld, b) weld metal from the bottom of the weld.*

An interesting structure can be seen in Figure 3-9 where the fusion line of an electron beam weld specimen is shown. In these specimens all deformation was localised to the weld metal and the heat affected zone end the parent metal did not show any deformation. During welding of these welds the grains present in the parent metal before welding served as nucleation sites for columnar grains growing into the weld pool on cooling of the weld. This is exemplified by the continuation of sub-grain twins into the weld metal at a different angle than in the parent metal.

After testing the weld metal in the electron beam welds etches to a very dark shade even if a light etch is applied, Figure 3-10a. If the dark regions are studied at a higher magnification it can be seen that the dark shade is caused by closely spaced lines where the etching has cut more deeply into the material, Figure 3-10b. The resulting structure looks remarkably like a corrugated metal plate or a thumbprint, and if viewed from a distance the area appears dark. The reason for this is unknown but it can be noted that it only appears where the material has been heavily strained, and also only in the electron beam weld metal. The most distinguishing feature of this weld metal is the very large grains found here. Columnar grains with a length of 2 mm or more are not uncommon. A possible explanations is therefore that the lines in reality are bands of high dislocation density material that etch more easily than the surrounding material. It is well known that a great number of slip-planes are formed when large single crystals of copper are deformed. The columnar weld metal grains can in this context be viewed as single crystals. This theory would also explain the wavy bands that can be seen in Figure 3-9. The dark bands are actually regions where this corrugated surface is even more pronounced. One can see that the bands always traverse the columnar grains perpendiculary, consistent with the view that they are caused by etched-out slip-planes.

A complete metallographical study of the creep specimens can be found in Appendix A.

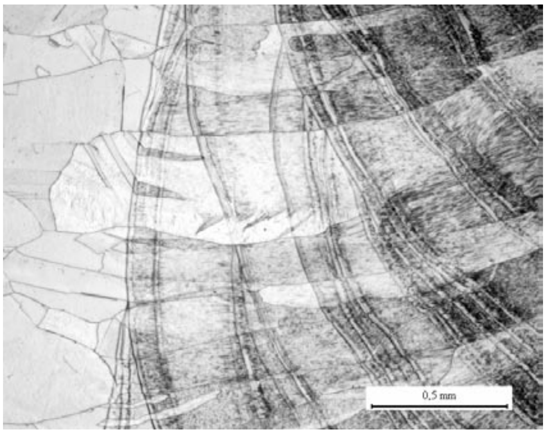
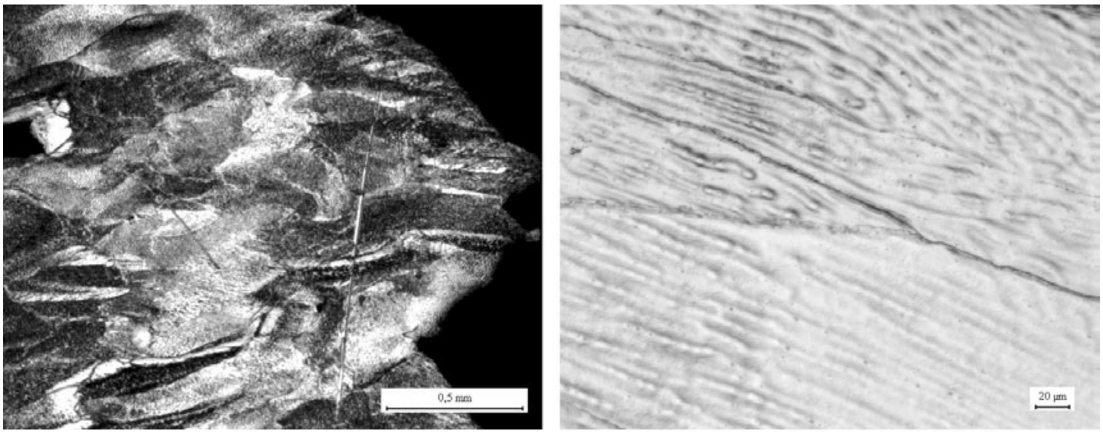


Figure 3-9. *Fusion line and deformed weld metal from an electron beam cross-weld specimen. Note the abrupt change of direction for the twins in the large grain at the left part of the picture.*



a) *b)*

Figure 3-10. *a) Dark etching weld metal in an electron beam cross-weld specimen after testing, 25×. b) Close-up of bands that possibly consists of material with a high density of dislocations that etch more easily than the surrounding matrix.*

4 Discussion and conclusions

The creep tests reported in this work show that at least 10% creep ductility has been obtained in both the friction stir welds and the electron beam welds. The ductility was measured at temperatures from 75°C to 175°C. According to the criteria set down in /1/ this is sufficient ductility. However in the case of electron beam welds all deformation is localised to the weld metal and if the weld is subjected to large strains localised necking and rupture cannot be ruled out. Friction stir welds do not exhibit this strain behaviour and the more even area reduction over the whole cross weld specimen should provide better protection against cracking in service.

An additional reason why the electron beam welds show a lower creep strength than the friction stir welds is the difference in grain size which is about 2,000 and 75 µm, respectively. Taking into account the low test temperatures, the Petch-Hall effect gives a contribution to the strength. At static loading at room temperatures this effect gives a difference of 15 MPa which is of the same order as the difference in rupture strength between the microstructures. Also previous testing reported in /8/ has shown that the grain size has a large effect on the creep life .

The similarity of the creep response of the parent metal when compared to both cross-weld and weld metal specimens at 175°C is noteworthy for friction stir welds. Apart from the specimens cut from the weld nugget there is almost no difference between parent metal results and weld results. The likely explanation is that the variation in microstructure between parent metal and weld metal is small. Creep strain is accumulated in the parent metal part as well as in the weld metal, and necking is taking place either in the weld metal or in the parent metal.

The initial loading strain was in most cases measured as over 6% during a short time-period of less than 3 minutes. This shows that the copper canister and the welds have short time ductility when the canister is still warm. Short time ductility is important if the canister is subjected to an earthquake or similar. An earthquake can mean that the bedrock around the canister shifts and applies a sudden shear load on the copper. The timeframe for an earthquake is in the order of minutes and the initial loading strain in a creep test could therefore reasonably be viewed as an earthquake type load. The short time ductility is higher the higher the applied load.

5 Acknowledgments

The authors wish to thank Svensk Kärnbränslehantering AB (the Swedish Nuclear Fuel and Waste Management Co) for funding this work and supplying the test material.

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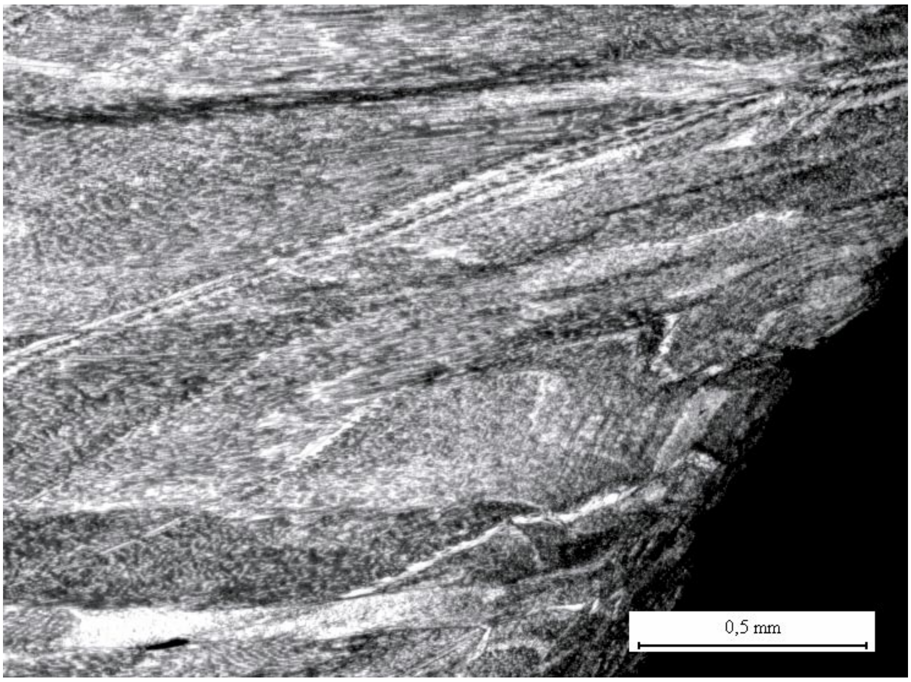
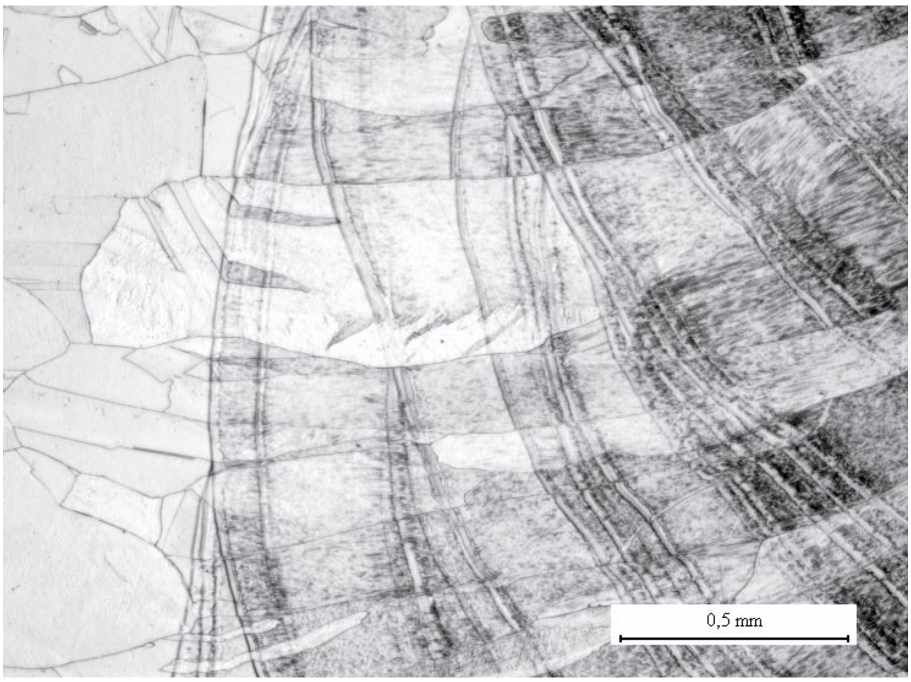
Appendix A

Light optical microscopy (LOM)

(The stress direction is always horizontal in the images.)

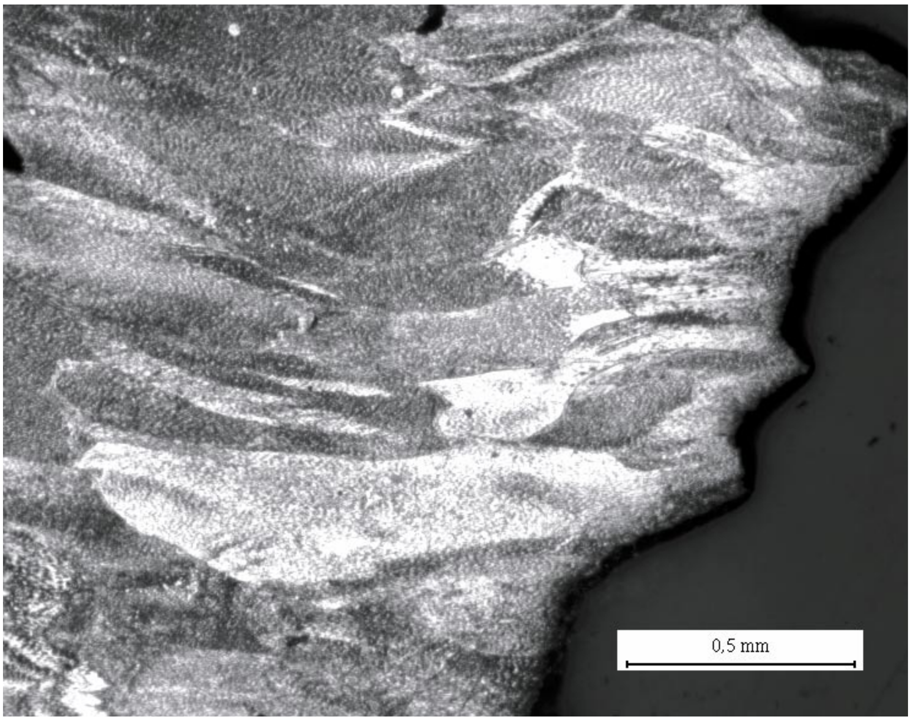


a) Magnification 25x



b) Magnification 25x

Figure A-1. Electron beam cross-weld creep test. Test data was 75°C/150 MPa and the rupture time 1,688 hours. a) Deformed grains at rupture, b) deformed grains at fusion line with evidence of recrystallisation across the fusion line.



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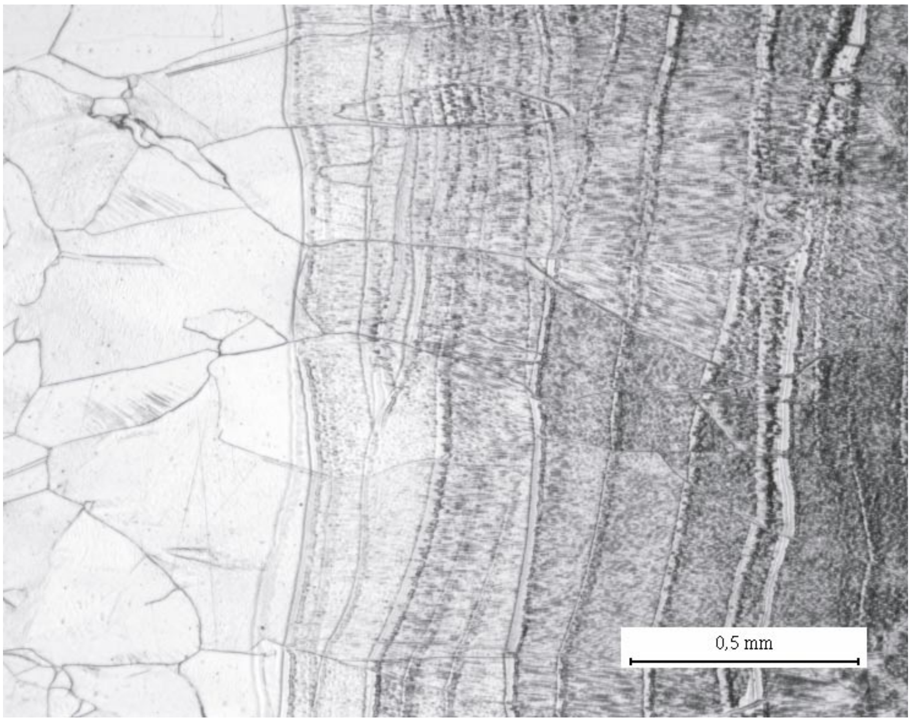
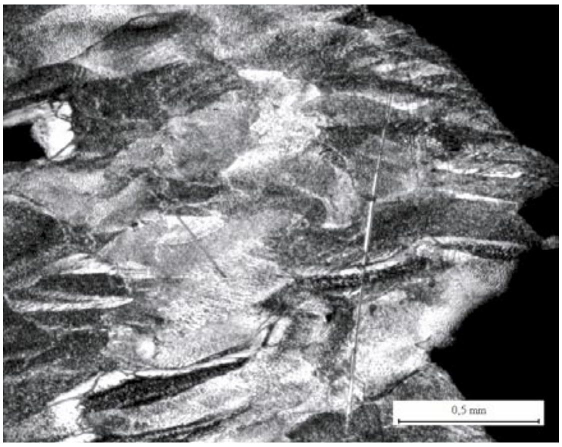


b) Magnification 25x

Figure A-2. Electron beam cross-weld creep test. Test data was 125°C/130 MPa and the rupture time 9,514 hours. a) Deformed grains at rupture, b) deformed grains at fusion line with evidence of recrystallisation across the fusion line.



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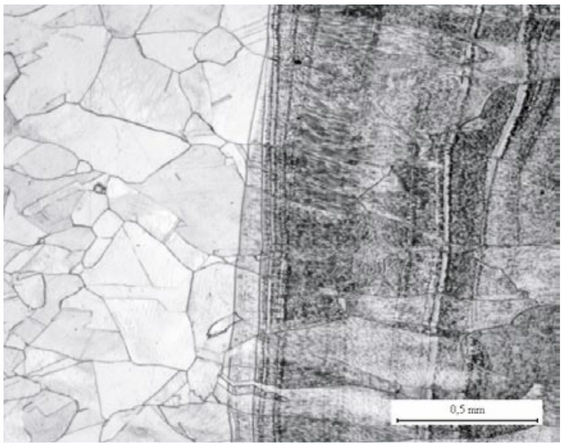

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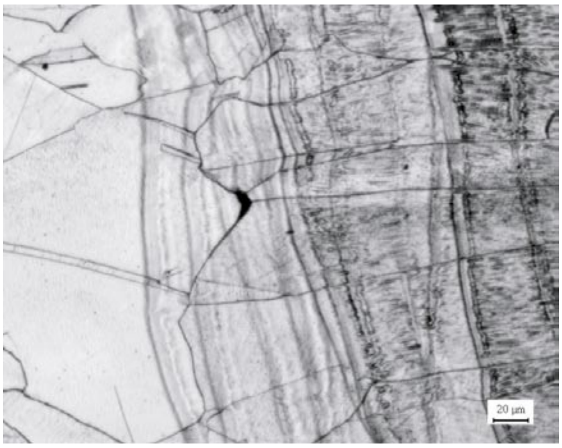
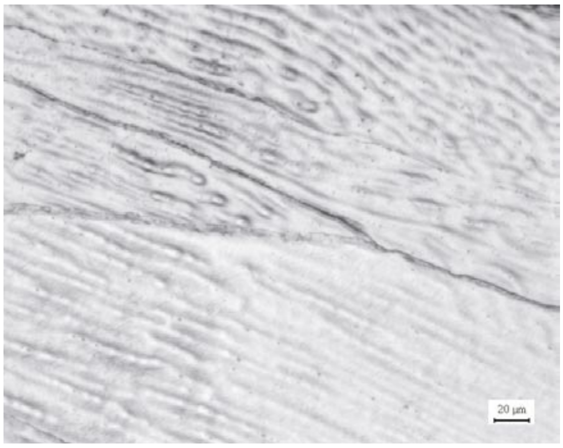
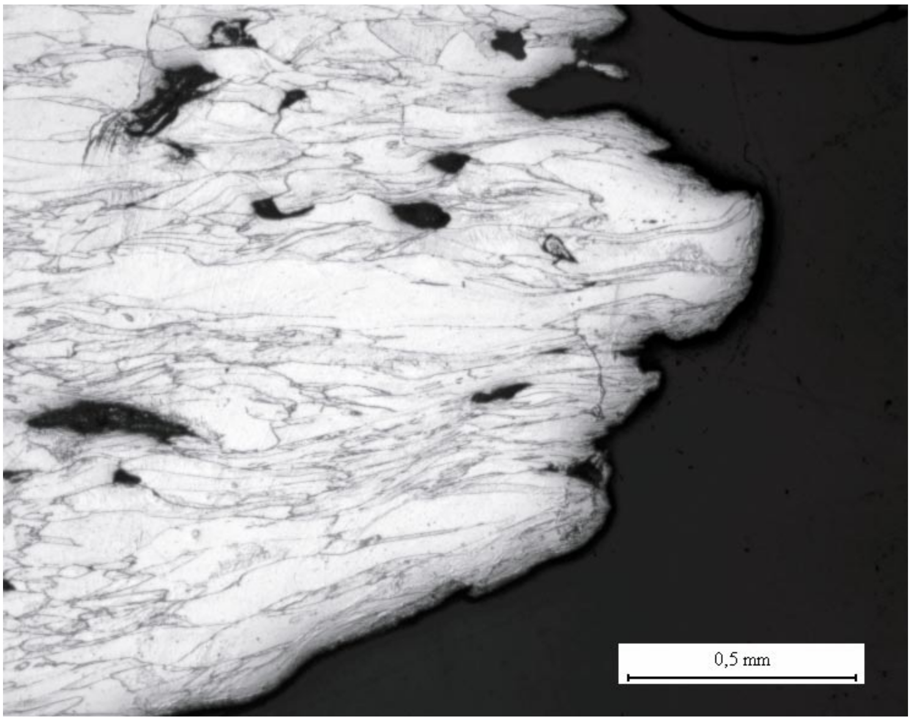


c) Magnification 200x

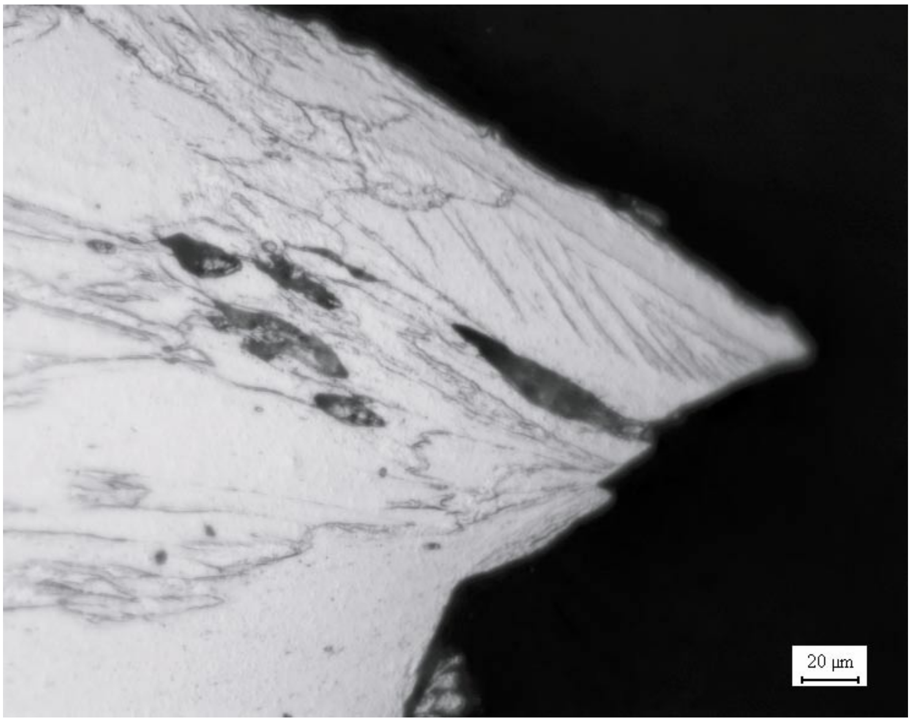


d) Magnification 200x

Figure A-3. Electron beam cross-weld creep test. Test data was 175°C/117 MPa and the rupture time 1,474 hours. a) Deformed grains at rupture with a crack visible, b) deformed grains at fusion line with evidence of recrystallisation across the grain boundary, c) cracks at the fusion boundary with evidence of strain etching, d) close-up of etch bands possibly associated with excessive straining of the matrix.

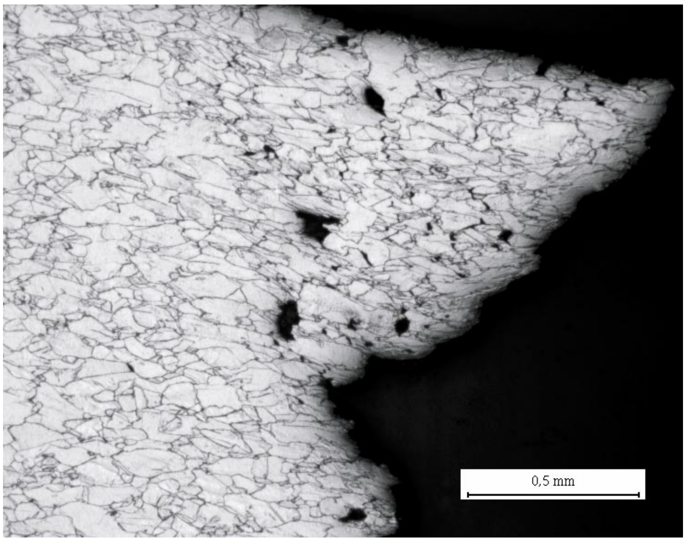


a) Magnification 25x

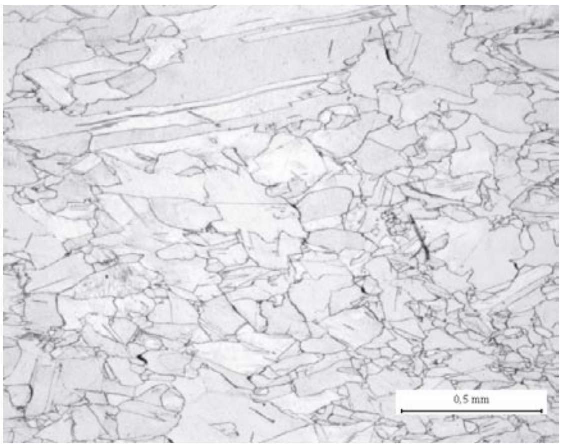


b) Magnification 200x

Figure A-4. Friction stir cross-weld specimens creep test. The specimens were extracted from the top of the weld. Test data was 175°C/130 MPa and the rupture time 10,875 hours. The specimens were extracted from the top of the weld. a) Deformed grains at rupture with microcracks and large cavities, b) close-up of a similar structure.



a) Magnification 25x



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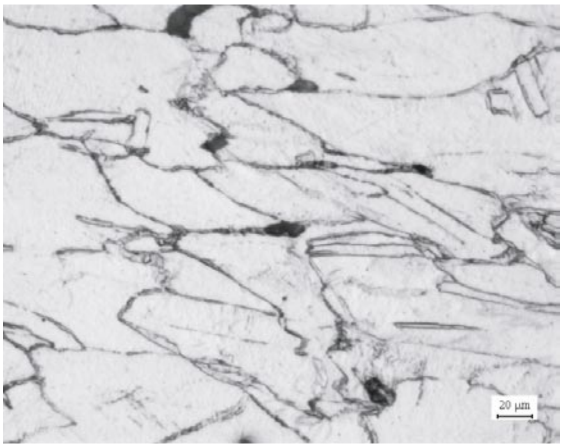

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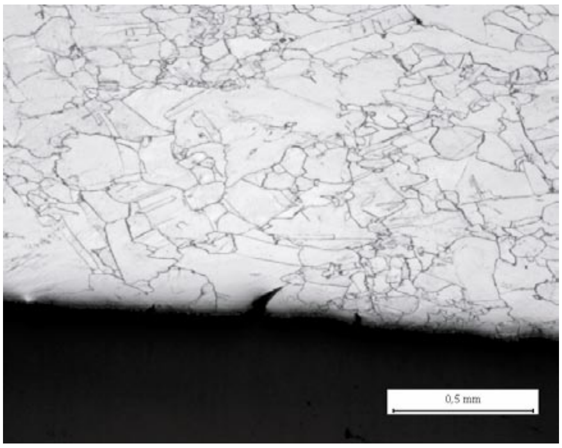
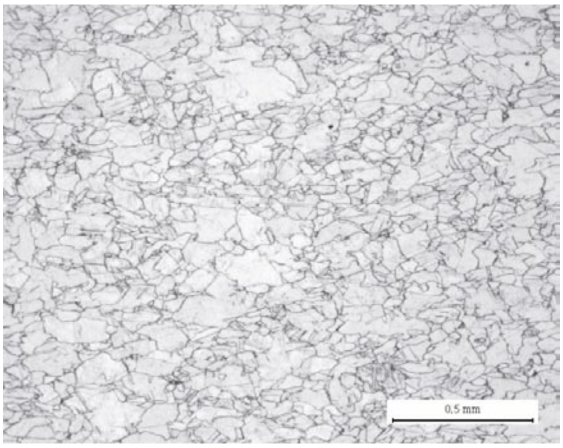
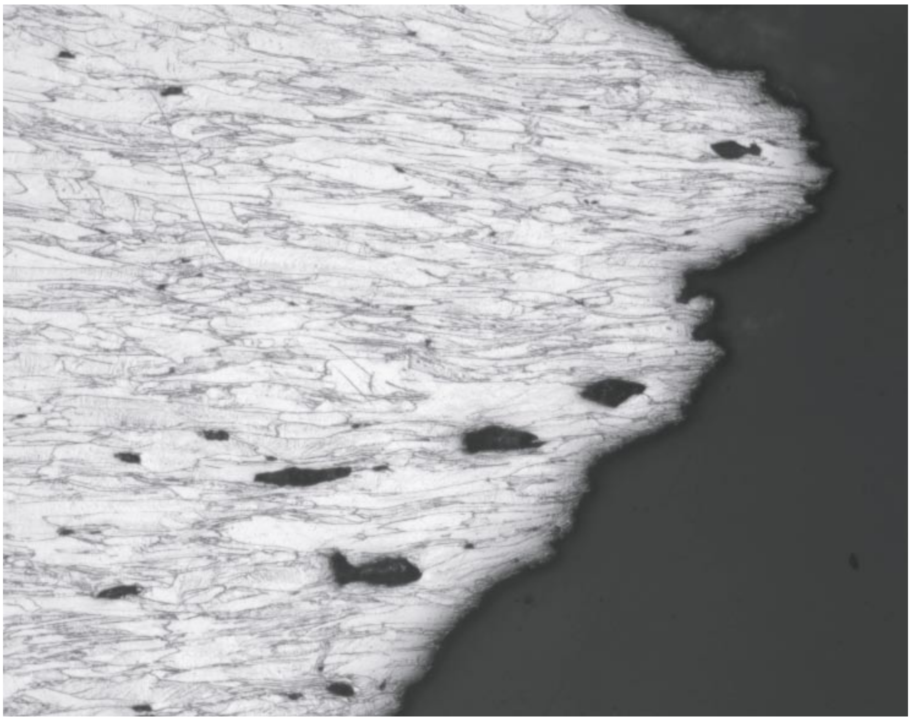


d) Magnification 25x

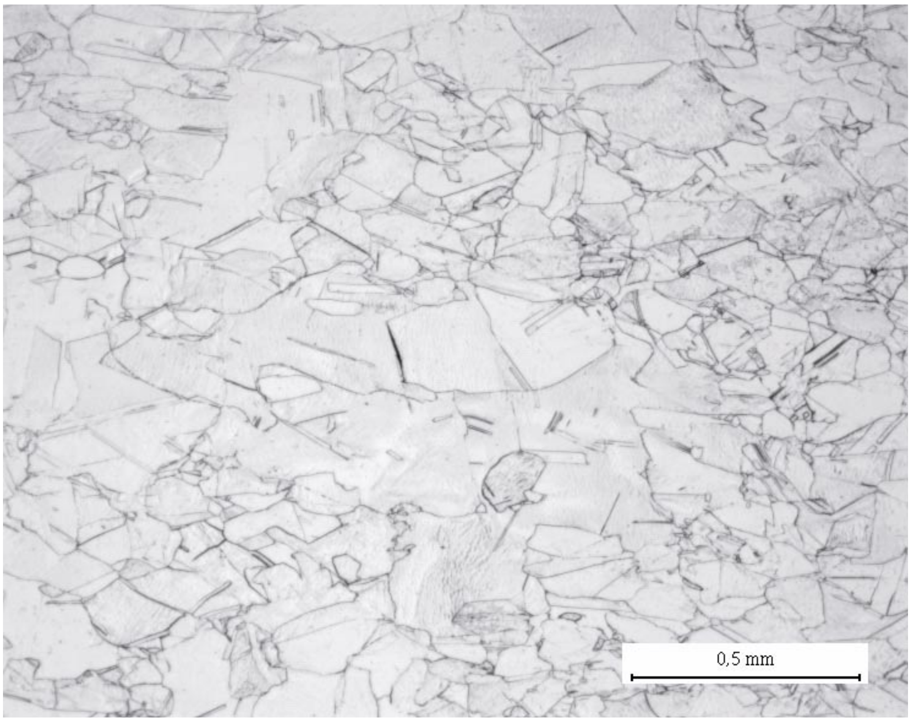


e) Magnification 25x

Figure A-5. Friction stir cross-weld specimens creep test. The specimens were extracted from the bottom of the weld. Test data was 175°C/135 MPa and the rupture time was hours. a) Slightly deformed grains at rupture with fine grain size and a large number of cavities, b) microcracks at the transition from fine grained to coarse grained structure, c) cavities, d) a surface microcrack, e) fine grained structure without cavities or microcracks ca 5 mm from the rupture.

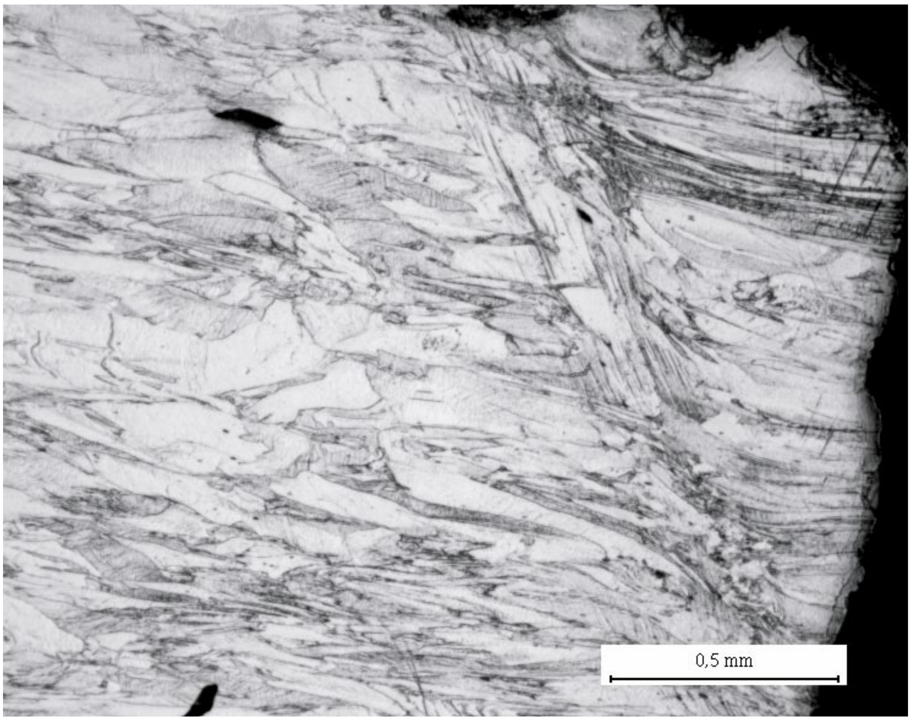


a) Magnification 25x

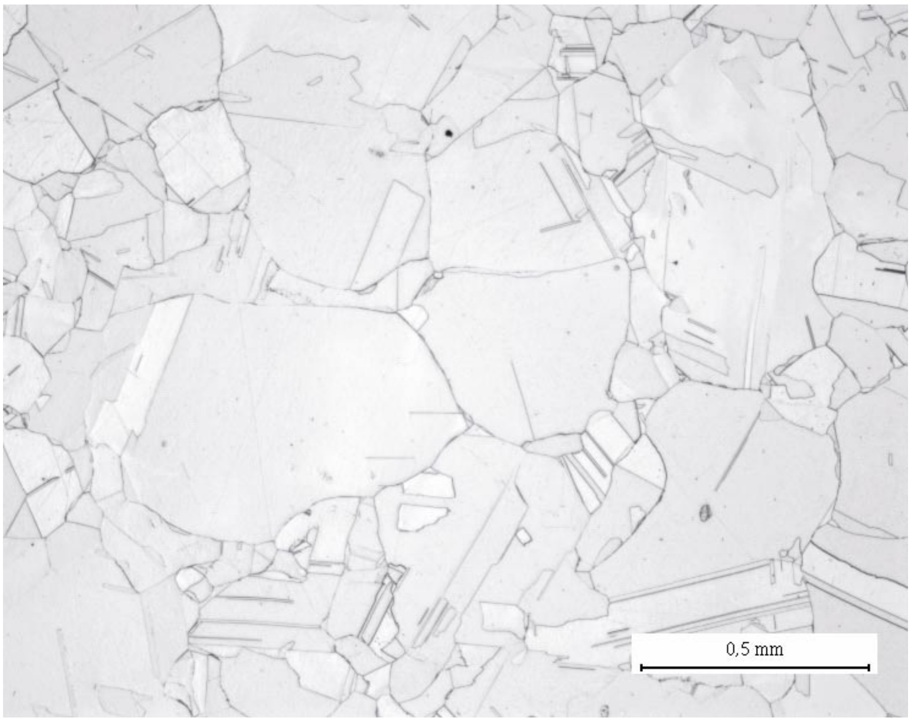


b) Magnification 25x

Figure A-6. Friction stir cross-weld specimens creep test. The specimens were extracted from the top of the weld. Test data was 125°C/159 MPa and the rupture time 8,439 hours. The specimens were extracted from the top of the weld. a) Deformed grains and cavities at rupture. b) This specimen had a second necking starting to develop on the other side of the gauge length from the rupture. In this micrographs some microcracks can be seen and possibly some deformation of the grains themselves.

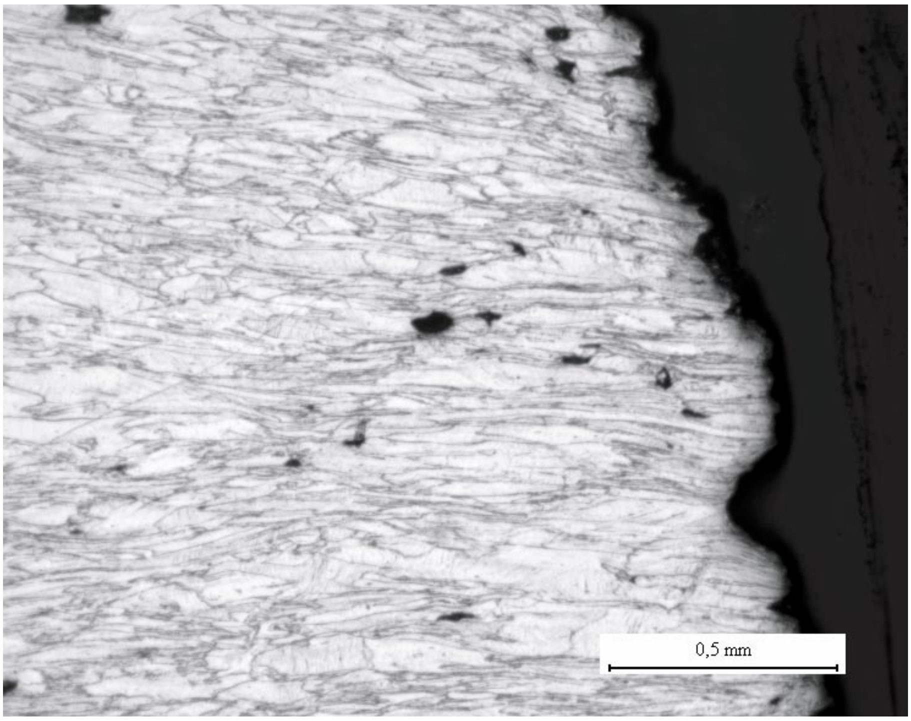


a) Magnification 25x

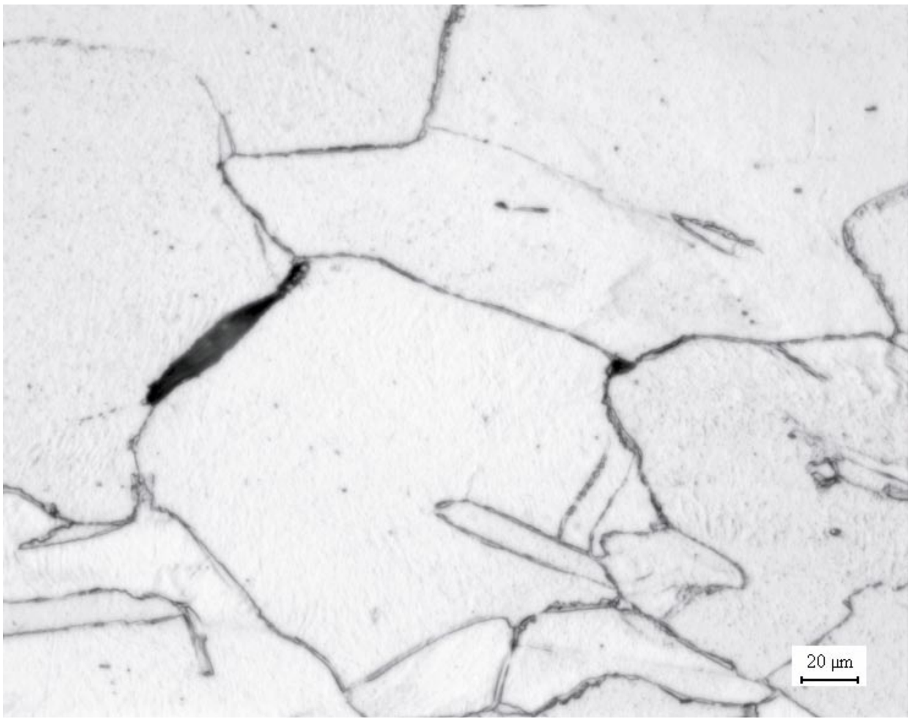


b) Magnification 25x

Figure A-7. Friction stir cross-weld specimens creep test. The specimens were extracted from the top of the weld. Test data was 75°C/170 MPa and the rupture time 660 hours. a) Heavy deformation but few cavities at the rupture, b) typical appearance of unstrained material.

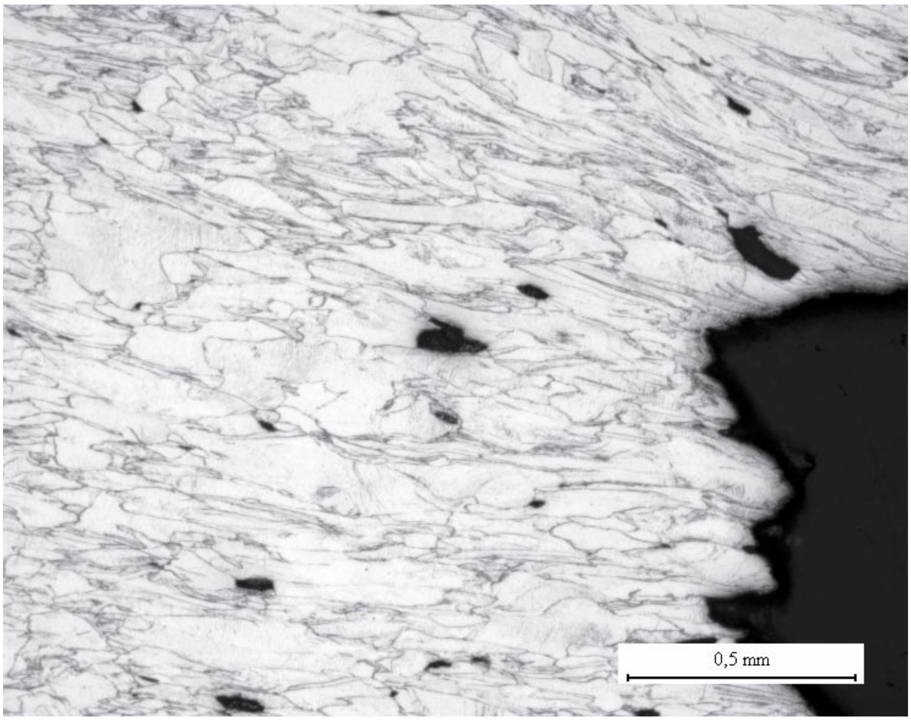


a) Magnification 25x



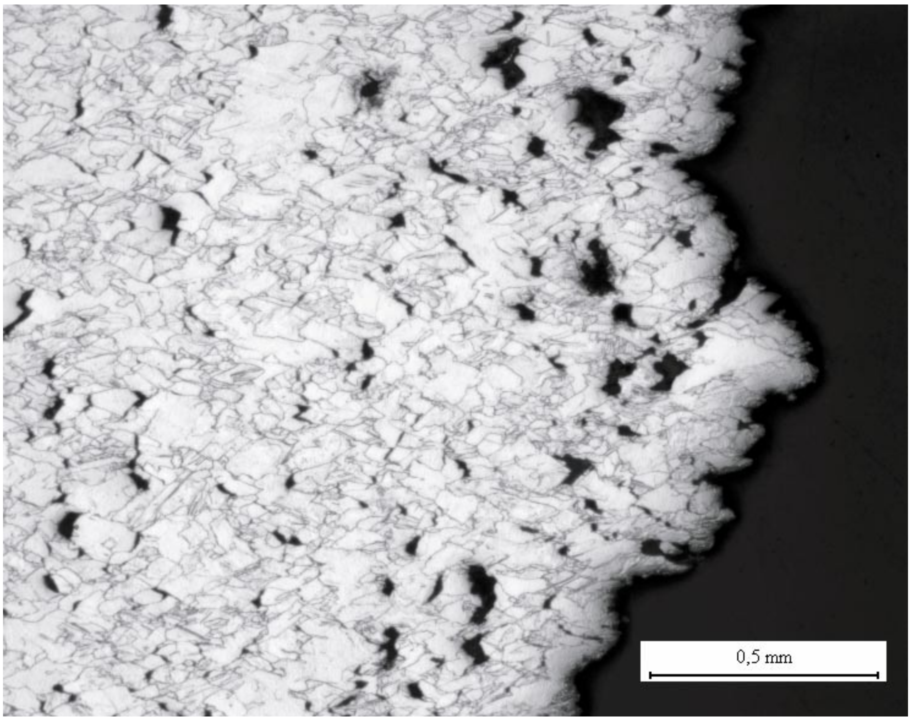
b) Magnification 25x

Figure A-8. Friction stir weld metal creep test. The specimens were extracted from the heat affected zone. Test data was 175°C/140 MPa and the rupture time 1,133 hours. a) Deformed grains at rupture, b) microcracks in the gauge length.

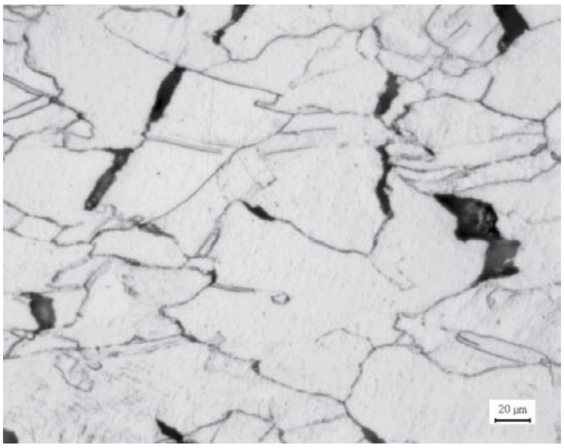


a) Magnification 25x

Figure A-9. Friction stir weld metal creep test. The specimens were extracted from the top of the weld. Test data was 175°C/140 MPa and the rupture time 9,311 hours. a) Deformed grains at rupture.



a) Magnification 25x



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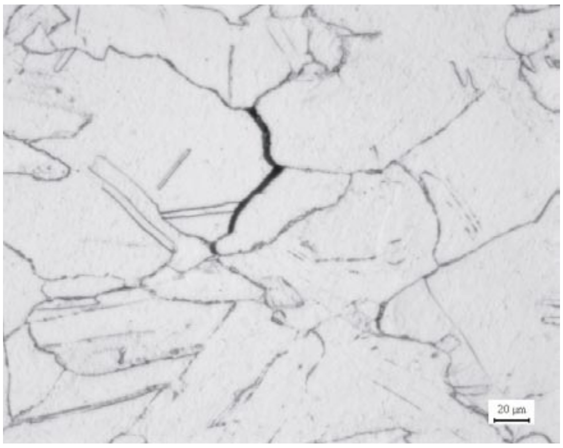
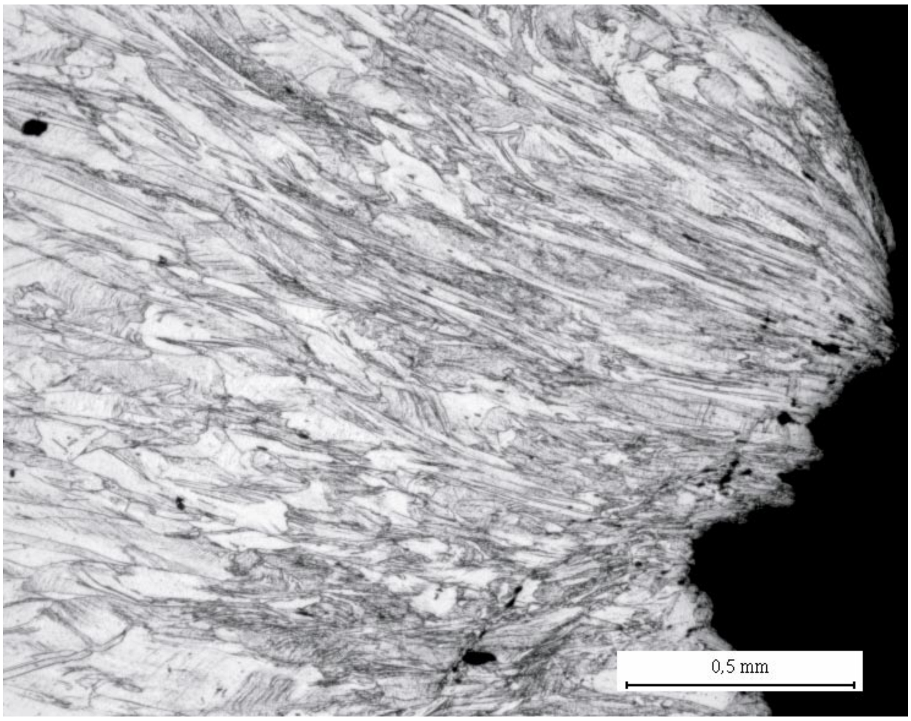
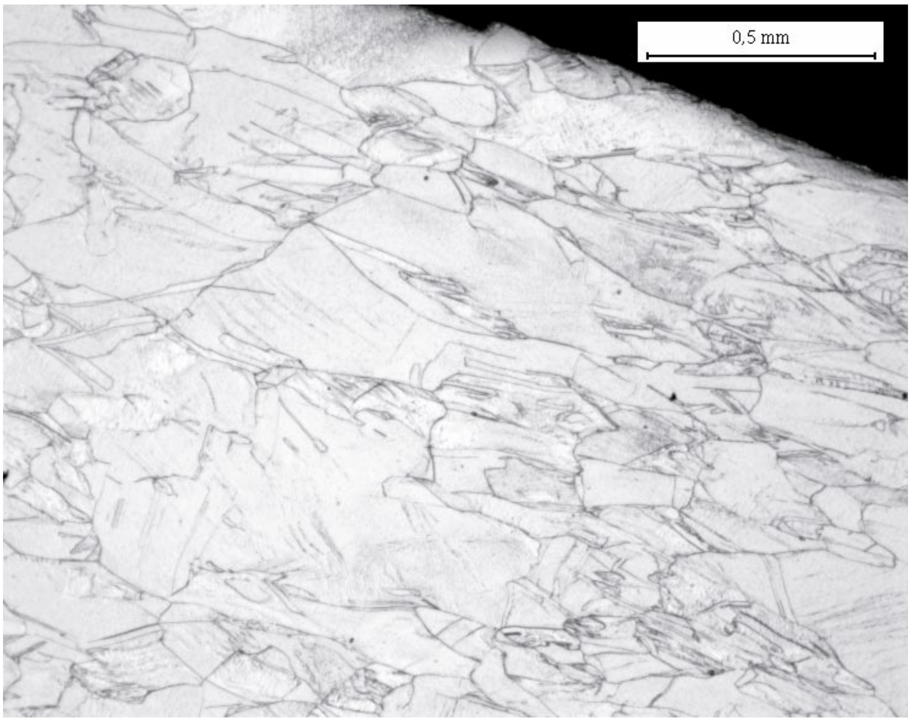


c) Magnification 200x

Figure A-10. Friction stir weld metal creep test. The specimens were extracted from the bottom of the weld. Test data was 175°C/131 MPa and the rupture time 6,106 hours. a) Slightly deformed grains at rupture with lots of microcracks, b) lots of microcracks about 5 mm from the rupture, c) microcracks about 25 mm from the rupture.

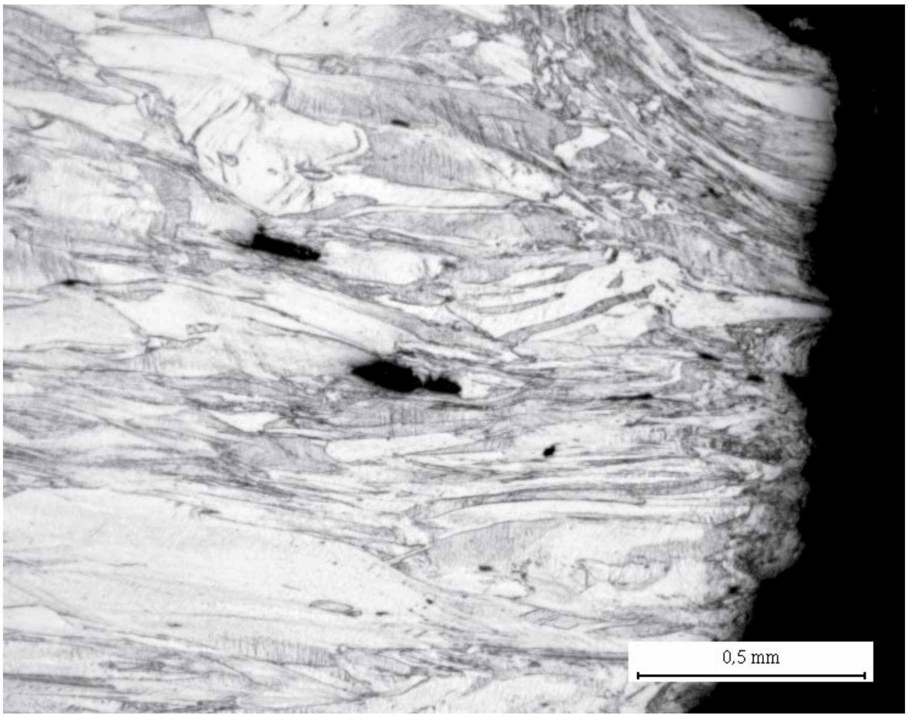


a) Magnification 25x

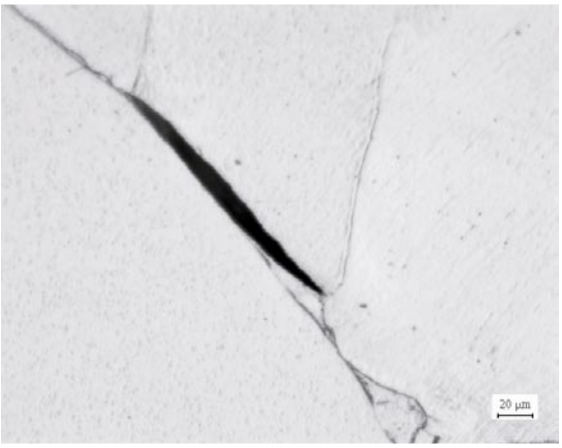


b) Magnification 25x

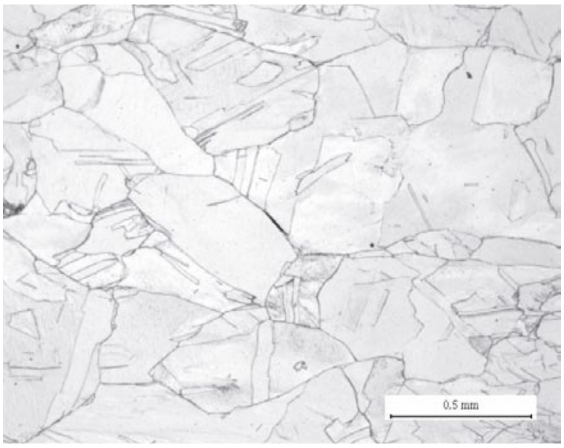
Figure A-11. Parent metal creep test. Test data was 125°C/165 MPa and the rupture time 1,657 hours. a) Deformed grains at rupture, b) deformed grains in the gauge length about 15 mm from the rupture.



a) Magnification 25x



b) Magnification 200x



c) Magnification 25x

Figure A-12. Parent metal creep test. Test data was 175°C/145 MPa and the rupture time 162 hours. a) Heavily deformed grains at rupture with strain structures perpendicular to the strain direction, b) microcrack, c) microcrack in the gauge length.

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