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ORIGINAL RESEARCH ARTICLE



Effects of Static Strain Aging on Mechanical Performance of Ductile Cast Iron

Ville Björklund (), Hannu Hänninen, and Sven Bossuyt

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EN-GJS-400-15U nodular cast iron intended to be used as load-bearing element in long-term geological disposal canisters containing spent nuclear fuel in Finland and Sweden was studied for static strain aging (SSA). Tensile test specimens manufactured from the nodular cast iron were pre-strained to 1%, 2% and 3% nominal plastic strains. The pre-strained specimens were aged at different temperatures ranging from room temperature to 400 °C for varying times. The aged specimens were tested with conventional tensile testing using constant cross-head speed of 0.016 mm/s. Additionally, four specimens were studied with digital image correlation (DIC) during the tensile testing to obtain full-field strain measurements. SSA resulted in elevated pronounced yield point in all the conditions, while the as-received material showed continuous yielding behavior. SSA reduced the elongation to fracture. DIC tests showed more localized yielding behavior in the SSA specimens. Over-aging effect was observed at 400 °C where increasing prestrain did not increase the yield stress more. For 1-day aging time, the highest yield stress increment was found after aging at 200°C. The yield stress of the material was almost identical after aging in 100°C and 200°C.

Keywords DIC, ductile cast iron, spent nuclear fuel, strain aging, strain localization

1. Introduction

The spent nuclear fuel in Finland and Sweden is planned to be disposed according to the long-term geological disposal concept called KBS-3. The KBS-3 concept is based on the multi-barrier principle designed in a way that the radionuclides need to penetrate multiple distinct barriers to leak out of the system to the environment. In this concept copper canisters are used to seal the spent nuclear fuel after which the canisters are deposited about 500 m deep in the crystalline bedrock and surrounded with a layer of bentonite clay. The repository is designed to last at least 100 000 years, and it should remain intact and sealed the whole period to ensure the safe nuclear decay of the spent fuel.

The KBS-3 copper canister itself consists of two main components: the outer 50 mm thick copper shell made from oxygen-free phosphorous-doped copper (Cu-OFP) and the insert made of ductile cast iron. The purpose of the outer copper shell is to serve as ductile corrosion-resistant barrier. The insert is intended to be the main load-bearing element of the canister and is made of nodular cast iron of grade EN-GJS-400-15U with the ferritic matrix microstructure. The requirements set for the cast iron insert in ambient temperature are > 12% elongation to fracture, > 240 MPa tensile yield strength, > 370 MPa ultimate tensile strength and fracture toughness $K_{\rm IC} = 78$ MPa(m)¹/₂ (Ref 1, 2). In the final repository the canister will be subjected to different types of loads that the canister must be able to withstand without failing. The canister needs to withstand an isostatic pressure load of 45 MPa that is the sum of the maximum hydrostatic pressure and the swelling pressure of the bentonite buffer. The most critical type of loading scenario is the possible rock shear movement across the deposition hole that can subject the canister to bending. The design target for such a rock shear movement is 50 mm shear that according to the modeling studies can cause a plastic strain of up to 2.5 % in the cast iron insert (Ref 1, 2). The analyses have shown that the copper overpack will maintain its integrity under the various loading conditions as long as the cast iron insert remains intact. For this reason, it is particularly important to ensure the long-term integrity of the cast iron insert in all possible conditions in the repository. In addition to the mechanical loads, the canister will be subjected to elevated temperatures in the final repository due to the nuclear decay of the spent fuel. Although pessimistic evaluations of the heating in the canister have resulted in possible temperatures as high as 230 °C in the cast iron insert (Ref 3), the latest studies suggest that the cast iron insert will heat up to 125 °C, while the temperature of the copper overpack will remain under 100 °C during the whole deposition period (Ref 4). The elevated temperatures are only affecting the canister during the first 1000 years of the deposition, but the temperature of the canister will not drop under 0 °C during the whole estimated deposition period (Ref 2).

The plastic strain, e.g., due to rock shear, and elevated temperature in the repository conditions makes the dynamic and static strain aging effects possible on the cast iron insert. The phenomenon of strain aging first discovered by Cottrell and Bilby (Ref 5) is well known and extensively studied for

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steels. Especially static strain aging in ferritic steels has been widely used in automotive industry to increase the yield strength of the sheet material by specific treatment process, called bake hardening (Ref 6-14). However, only limited number of studies on the effects of strain aging on cast iron have been conducted (Ref 15-17), even though the regular yielding behavior of the EN-GJS-400-15U in similar castings has been studied and characterized (Ref 18). The same principles should apply to both cast irons and steels, but one major difference between cast irons and steels is the higher silicon content in cast iron, which has been demonstrated to increase the activation energy of carbon diffusion (Ref 19, 20). Another difference is the inherent non-uniformity of local strain distributions around graphite nodules (Ref 21, 22), and the possibility of superimposed residual strains from non-uniform shrinkage during the casting process (Ref 23-26). It is known that ferritic steels are susceptible to static strain aging already at low and moderate temperatures (Ref 27-30), which raises concerns regarding the possibility of static strain aging (SSA) of the cast iron insert that has mainly a ferritic microstructure. Although some studies on dynamic strain aging (DSA) (Ref 15-17), and on related phenomenon of quench aging on ductile iron (Ref 31) have been conducted, static strain aging has been largely overlooked and no publications on SSA in ductile cast iron exist. In this study, the static strain aging of the ductile cast iron in the range of conditions covering those predicted for the spent nuclear fuel repository is examined.

2. Materials and Methods

The material used for the study is spheroidal graphite cast iron of grade EN-GJS-400-15U. The specimens were manufactured from the full-scale cast iron insert, casting I73, from which a block was provided by Posiva Oy, Finland. The chemical composition of the cast iron is presented in Table 1. The chemical composition analysis provided by Posiva Oy was supplemented with the nitrogen content measurement performed by Eurofins with a LECO machine.

Two pieces of material were cut from the cast iron insert close to the insert surface for manufacturing the tensile test specimens. The material was cut from the insert according to Fig. 1(a)). The tensile test specimens were cut with electrodischarge machining (EDM) according to the geometry shown in Fig. 1(b) which is based on standard ASTM E8 (Ref 32). The EDM affected surface layer of the tensile test specimens was removed from the gauge section using mechanical grinding with 320 grit emery paper followed by surface finishing with 500 grit emery paper. For the tensile tests measured with digital image correlation the specimens were only ground with 320 grit emery paper to ensure that the paint adheres well to the specimen surface. As the grinding removed some material and because the cross-section area changes during the pre-straining, each specimen was measured before the pre-straining and before the final tensile test. The engineering stresses were calculated accordingly.

Constant extension rate tensile testing (CERT) was conducted at room temperature using an MTS Insight Electromechanical 30 kN Standard Length tensile testing machine, with cross-head speed 0.016 mm/s for loading, i.e., a nominal strain rate of 0.0005 s^{-1} which is quasi-static, and 0.1 mm/s for unloading. Strain was measured as an average strain by using an extensometer with an initial gauge length of 25 mm. For the reference behavior of the as-received material, a tensile test was conducted. Pre-straining of specimens to 1%, 2%, or 3% nominal plastic prestrain was performed at the same cross-head speeds. This range of pre-strain conditions, along with specimens of the as-received material for reference, was chosen to cover the range of strains considered plausible after a rock shear event in the repository (Ref 1, 2). Aging of pre-strained or reference specimens was carried out in air in a resistance furnace at 100 °C, 200 °C, 300 °C, or 400 °C, for 1 day or 11 days, or at RT for 84 days. Although 300 °C and 400 °C significantly exceed the temperatures expected in the repository, short aging times at these higher temperatures were included to mimic the over-aging that could occur at lower temperatures in the repository with very long aging times. After the aging treatment the specimens were removed from the furnace and left to cool down in air at room temperature for approximately 30 min to 1 h before final tensile testing them the same day, to ensure thermal equilibrium while minimizing the possibility of further aging at room temperature.

To observe full-field spatiotemporal patterns of strain localization and assess possible effects of static strain aging on strain localization, 4 additional tensile specimens were tested after aging at 100 °C for one day, using digital image correlation (DIC) (Ref 33) for the strain measurements. A stereo DIC system with 2 camera set-up was used with Correlated solutions' Vic3D v9 software. Hardware details and DIC processing parameters are compiled in Table 2. Detailed calibration information can be seen in Appendix 1 Table 4. The speckle pattern was applied with spray paint. First a thin layer of white spray paint was applied as base coat to achieve a good contrast in the speckle pattern. A black spray paint was then used to apply the speckles on top of the base coat. Figure 2 shows a representative speckle pattern, including a close-up showing typical pattern features consistent with the recommended feature size of 3-5 pixels (Ref 34), as well as one of the infrequent larger paint speckles. The pattern was removed before the aging and a new pattern applied before the final tensile test. Although it is easy to calculate the average strain that would be measured with the extensometer from the fullfield strains measured by DIC, and know after pre-straining precisely how much strain was applied to each specimen, these data are not immediately available during the test, so the prestraining was carried out to predetermined cross-head displacements that were predicted to result in approximately 1%, 2%, and 3% pre-strain, respectively.

The DIC images were recorded with 500 ms time intervals during the tensile tests. For the DIC calculation, different

Table 1 Chemical composition of cast iron grade EN-GJS-400-15U from casting I73 in wt.%

С	Si	Mn	S	Р	Ni	Cu	Mg	Fe	Ν
3.48	2.48	0.22	0.004	0.01	0.04	0.02	0.04	Bulk	0.0041



Fig. 1 (a) Location where the tensile test specimens were cut from the casting I73 marked in red. (b) Tensile test specimen geometry

Sony IMX255
4096 px x 2168 px
3.5 µm
23 px
5 px
15
2 mm
Affine
Gaussian
Optimized 8-tap
Lagrange
Normalized sum square difference (NSSD)
Single, undeformed

Table 2 Hardware and DIC settings used in Vic3D



Fig. 2 Specimen 24 speckle pattern for tensile test and DIC. The higher magnification shows a large speckle with a more typical quality pattern around the edges

numbers of pictures were used. The displacement between the images when using every captured image was 0.008 mm. For the DIC calculations around yield point and at final rupture every captured image was used for the DIC analysis. For the

pre-straining every 6th image was used as the higher interval was sufficient to see the strain fields at the end of the prestraining in the required detail. The filter type used was a Gaussian weighted filter with 90% center weight.

3. Results

3.1 Tensile Testing

All the tested specimens showed continuous yielding behavior without a yield point when pre-strained in the asreceived condition. The yielding behavior of the as-received material is repeated with little to no scatter in the pre-straining region in all studied specimens (see Appendix 2, Fig. 15). Also, specimens tested after thermal aging without pre-straining (see Appendix B Fig. 16) show the same yielding behavior as the as-received material. Differences in the elongation to fracture of different specimens are due to the natural variability in the microstructure and small casting defects sometimes causing a specimen to show more pronounced strain localization earlier, resulting in earlier rupture. Tests on specimens with smaller cross section, exacerbating this natural variability, result in greater scatter of the elongation to fracture (not shown).

The stress-strain curves of pre-strained and aged specimens are shown in Fig. 3 and 4. Stress in these figures is calculated according to the original cross section so that the stress increase seen in the figures is strictly due to strain aging and not due to a geometry change of the cross section. The stress-strain curves of specimens aged for 1 day at different temperatures are shown in Fig. 3. Every examined strain aging condition results in a pronounced yield point, with yield stress increasing as the pre-strain is increased. The highest yield stress is achieved after aging at 100°C or 200°C. After that the increasing aging temperature results in lower yield stresses. Especially, after aging at 400°C the yield point is markedly lower and less pronounced than after aging at 100°C or 200°C. Despite the pronounced yield point, most of the specimens do not show any clear yield plateau with nearly constant stress after the upper yield point.

The stress-strain curves of specimens aged longer at the lower temperatures are shown in Fig. 4. The effects of SSA seem to be similar between the aging times of 1 day and 11 days, resulting in similar yield stresses. However, specimen 5 was outlier and ruptured after the yield point without any strain hardening. A pronounced yield point is also seen in the specimens aged at room temperature for 84 days, but the yield stress values of the RT specimens are not as high as those of the specimens aged at 100 °C and 200 °C.

Figure 5 shows the increase in the yield stress due to aging (i.e., the yield stress increment), $\Delta\sigma$, as a function of the nominal pre-strain level and aging temperature. The yield stress increment, $\Delta\sigma$, is defined as the difference between the upper yield point after the aging and the flow stress at the end of the pre-straining. The error bar indicates \pm one sample standard deviation (0.54 MPa) of the stress increment between 0.4 and 0.8% proof stresses during pre-straining, as an indirect quantification of the measurement error. The engineering stresses in this case are calculated using the original cross-section area before the pre-straining, to exclude the effect of the

geometry change from pre-straining on the calculated stress. At all the aging temperatures except at 400 °C, the yield stress increment $\Delta\sigma$ increases or reaches a plateau as the pre-strain is increased. At every pre-strain level, the maximum change in the yield stress is achieved after aging at 200°C, but it is not significantly higher than that after aging at only 100°C.

Figure 6 shows the elongation at maximum force A_{gt} as a function of pre-strain and aging temperature and time. There seems to be no clear correlation for A_{gt} on pre-strain level or aging time, but the A_{gt} for almost all the aged specimens is lower than that for the reference test.

All the tensile test results are compiled in Table 3. Unlike the graphs highlighting the difference between the flow stress



Fig. 3 Stress-strain curves of the tensile test specimens aged for 1 day at various temperatures. The stress-strain curves are offset (except at 100 °C), and redundant parts are omitted to improve readability. Magnifications of the stress-strain curves around the yield point are presented on the top. Legends show the specific specimen ID



Fig. 4 Stress-strain curves of the specimens aged at 100 °C and 200 °C for 11 days and the specimens aged at RT for 84 days. Magnifications of the stress-strain curves around the yield point are presented on the top. Legends show the specific specimen ID

during pre-straining and the yielding after aging, the stresses and strains in the table are defined relative to the geometry before the final tensile test rather than the original geometry before the pre-straining, except for the measured pre-strain and the yield stress increment $\Delta\sigma$, which is defined exactly as in Fig. 5. The strains for the DIC specimens (24, 26, 28, 29) are measured with digital extensometer function over the gauge length. The results show clearly that the yield ratio (YR), which is the ratio of yield strength and ultimate tensile strength (YS/ UTS), increases markedly when the strain aging effect occurs, indicating a loss of ductility.

3.2 DIC

Figure 7 shows full-field strain measurement of the reference tensile test at the beginning of the yielding and at the time of final rupture. The as-received material begins yielding at the specimen shoulders, but the deformation very quickly propagates through the entire gauge section resulting in a rather uniform distribution of strain. Before the final rupture the strain starts to localize in the middle of the specimen which then leads to rupture. The final rupture location has no correlation with the strain localization at the beginning of yielding.



Fig. 5 Yield stress increment $\Delta \sigma$ as a function of (a) nominal pre-strain in different aging conditions, and (b) as a function of temperature for different pre-strains and aging times



Fig. 6 Elongation at maximum force as a function of (a) pre-strain level and (b) aging temperature for different pre-strains and aging times

Figure 8, 9, and 10 shows that after strain aging the initial deformation upon yielding is already more localized. In Fig. 8(b), multiple complex deformation bands nucleate when the yield point is reached but become immediately stuck either due to other intersecting bands or due to microstructural imperfections. The deformation then accumulates at these locations until the final fracture. From Fig. 8(c), it can be clearly seen that the final rupture location is highly correlated with the strain localizations at the onset of yielding. There seems to be some correlation between the strain localizations after pre-straining and the final rupture as well, but this correlation is less apparent.

In Fig. 9(b) at the beginning of the yielding around the yield point, a deformation band resembling typical Lüders band can be seen. The deformation band nucleates at the bottom shoulder of the tensile specimen and propagates upwards. However, the band has a diffuse front and no clear border between the yielded and un-yielded material is observed. The band gets stuck before the middle of the gauge section, not propagating through the whole gauge section. The final fracture in this specimen occurs at the location where the propagation of the Lüders band ended. The strains in the upper half of the gauge section seem to develop more uniformly, similar to the non-strain-aged specimen 26. Comparing Fig. 9(a), (b), and (c) some of the strain localizations during the final tensile test are already clearly seen after the pre-straining. The correlation is much more apparent than with specimen 24.

Figure 10 shows a more prominent localization of deformation than seen in the other strain-aged specimens. One major

Nominal pre- strain (%)	Actual pre- strain, %	Specimen ID	Aging temperature, °C	Aging time, days	Yield stress, MPa	Ultimate tensile strength, MPa	Yield ratio, YR	Yield stress increment Δσ, MPa	Elongation at maximum force Agt, %
0 (as-re-		1			264	373	0.708		12.5
ceived)		26	-		264	371	0.712		10.4
0		30	100	1	267	375	0.711		12.0
		31	200	1	265	364	0.728		8.5
1	1.007	2	25	84	311	364	0.854	21.2	8.4
	0.763	24	100	1	321	383	0.838	29.2	11.1
	0.996	12		1	323	373	0.866	27.6	8.0
	1.000	5		11	322	322	1.000	28.3	0.2
	1.002	16	200	1	323	367	0.880	30.0	7.1
	0.998	8		11	321	367	0.875	28.5	7.1
	1.003	11	300	1	318	365	0.871	24.5	6.2
	1.006	20	400	1	304	370	0.822	13.1	9.6
2	1.976	3	25	84	336	376	0.894	26.2	10.1
	1.531	28	100	1	340	387	0.879	30.6	10.8
	1.983	13		1	344	364	0.945	29.9	5.5
	1.980	6		11	344	379	0.908	29.2	7.4
	1.977	17	200	1	345	383	0.901	31.8	9.2
	1.979	9		11	342	374	0.914	30.4	6.9
	1.983	14	300	1	337	371	0.908	25.3	6.3
	1.980	21	400	1	317	365	0.868	9.2	6.4
3	2.965	4	25	84	353	377	0.936	26.0	7.6
	2.887	29	100	1	366	390	0.938	32.9	9.8
	2.956	15		1	364	383	0.950	32.2	7.0
	2.964	7		11	363	376	0.965	31.0	5.4
	2.959	18	200	1	363	370	0.981	32.8	4.5
	2.966	10		11	362	380	0.953	31.8	5.5
	2.965	19	300	1	352	374	0.941	25.1	5.4
	2.957	22	400	1	331	354	0.935	4.5	3.8



Table 3Summary of the tensile test results

Fig. 7 Specimen 26 full-field strain measurements: (a) around yield point, (b) during yielding and (c) at final rupture

deformation band nucleates in the middle of the gauge section, without propagating any further. This localization causes most of the deformation to accumulate in the middle region, where also the final fracture occurs. The prominent strain localization is visible in the DIC measurements during the whole tensile test and the strain does not distribute evenly through the gauge



Fig. 8 Full-field strain measurements of specimen 24: (a) after pre-straining, (b) around yield point after strain aging, (c) during yielding and d) at final rupture



Fig. 9 Full-field strain measurements of specimen 28: (a) after pre-straining, (b) around yield point after strain aging, (c) during yielding and d) at final rupture

section after the beginning of yielding. When comparing Fig 10(a), (b), and (c) the strain localization is clearly present already after the pre-straining and the correlation is evident.

Figure 11 shows the spatiotemporal pattern of the evolution of the distribution of strain rate along the gauge length of the specimen, overlaid with the engineering stress as a function of time. The color scale indicates the instantaneous local strain rate, averaged across the width of the gauge section. In such plots, the propagation of a Lüders band appears as a diagonal bright line, with slope proportional to the propagation speed of the band. These plots confirm that these specimens do not exhibit clear Lüders bands that propagate through the whole gauge section. Only specimen 28 shows a propagating deformation band at the beginning of yielding, and its propagation is arrested at the location of final fracture.

3.3 Microstructure and Fractography

Samples from specimens 1 and 11 were cut, and micrographs were taken with an optical microscope to investigate the graphite nodule size and distribution between specimens tested in this study. Figure 12(a) shows a microstructure of specimen 1 and Fig. 12(b) a microstructure of specimen 11 after etching in 2% Nital solution. Specimen 1 is one of the specimens that is furthest away from the casting surface and specimen 11 is one of the specimens closest to the surface. It is evident that the



Fig. 10 Full-field strain measurements of specimen 29: (a) after pre-straining, (b) around yield point after strain aging, (c) during yielding and (d) at final rupture

average size of the graphite nodules is larger closer to the casting surface. However, it seems that the casting microstructure becomes more heterogeneous when moving inwards from the outer surface, even though the average nodule size becomes smaller. The change in the microstructure can be explained by strong convections in the melt during the filling process and the closer proximity of the steel channels when getting toward the center which causes the cooling rate to be higher as demonstrated by Tadesse and Fredriksson (Ref 35). A higher cooling rate is known to cause finer microstructural features, i.e., grain size and graphite nodule size, in ductile cast iron (Ref 36, 37).

Based on the micrographs shown in Fig. 12(a) and (b), the volume fraction of graphite is about 12.8 % and 11.7 %, respectively, and the nodule count is around 47.1 nodules/mm² and 22.3, respectively. The ferrite grain size is about 83 μ m. The nodularity is close to 100% of types V and VI graphite according to standard EN ISO 945-1 (Ref 38). The volume fractions are calculated using MATLAB Image Segmenter by using the "Auto Cluster" option on the gray scale version of the image with the additional changes of opening the mask by 2 pixels and eroding the mask by 1 pixel. The obvious shrinkage cavities and pores were manually excluded from the calculation. The nodule count is obtained by calculating the amount of white areas in the resulting binarized picture divided by the area of the picture in mm². The grain size is estimated using the line intercept method.

The fracture surfaces of some specimens were examined with scanning electron microscope (SEM). The examined specimens 5 and 22 are the specimens that showed the lowest elongation values and specimen 1 is the reference sample without strain aging. Figure 13(a) and (b) shows segments of the fracture surface of specimen 1, c) and d) shows segments of the fracture surface of specimen 5 that broke before yielding, and e) and f) shows segments of fracture surface can be seen. Additionally, there is an atypical feature in the ferritic matrix (circled) that resembles a small pearlite colony, but could also be just a stretched dimple pattern.

All the studied fracture surfaces show some casting defects and impurities to varying degrees, sufficient to explain the large scatter in ductility of these specimens and even larger scatter in ductility of smaller specimens. A ductile dimpled fracture pattern is observed in all examined specimens (see Fig. 13(b), (d), and (f)), and no ductile to brittle transition is observed. Even specimen 5 that broke before yielding shows a fully ductile fracture surface. Figure 14(a) and (b) shows some of the casting defects found in specimen 5. Figure 14(a) shows a larger defective segment of specimen 5 with slag and impurities and b) shows a higher magnification of a pore/cavity with impurities on the surface. The premature rupture of specimens 5 and 22 was found to be due to various casting defects and impurities in the cast that caused more pronounced and earlier strain localization, so that a smaller volume of material contributed to the ductility of the whole specimen.

4. Discussion

This study demonstrates that, despite the presence of graphite nodules, static strain aging occurs in the ferrite matrix of the ductile cast iron much as it does in steels. In steels, diffusing interstitial atoms cluster around dislocations in socalled Cottrell atmospheres, which results in a locking effect increasing the stress needed for the dislocations to move (Ref 27, 28, 39). Due to the graphite nodules, it is to be expected that the distribution of local strains during pre-straining is much more heterogeneous in cast iron than in steels, so that the overall stress-strain curve is a convolution of the strain aging behavior of the matrix having different pre-strain levels due to the distribution of local strains. It is therefore unsurprising that the Lüders plateau after yielding (Ref 40, 41) is not so clearly defined in the stress-strain curves of this material, but it is clear that this material exhibits the pronounced yield point after strain aging. Casting defects and the heterogeneous nature of the studied cast iron, as affected by the uneven cooling of the cast,



Fig. 11 Spatiotemporal pattern of the evolution of the distribution of strain rates along the gauge length and engineering stress in the gauge section for specimens (a) 26 (reference), (b) 24 (1% pre-strain), (c) 28 (2% pre-strain) and (d) 29 (3% pre-strain). The strain rate at each position Y is the calculated mean strain rate along the X-axis of the specimen



Fig. 12 (a) Micrograph from specimen 1 showing a large scatter in graphite nodule size and presence of casting defects. (b) Micrograph from specimen 11 showing the graphite nodule size and distribution close to the surface of the casting

markedly affect the details of the deformation leading up to the final failure, resulting in large scatter in, for example, the elongation to fracture. The large scatter in the elongation to fracture has previously been demonstrated by Minnebo et al. for the EN-GJS-40015U material and similar castings (Ref 18). However, even in the presence of casting defects and the

variation in the microstructure, the yielding behavior is consistent.

The yield stress increment upon strain aging, measured for this study and shown in Fig. 5, is also consistent. To quantify the measurement uncertainty, an indirect approach is necessary because—rather than repeating measurements of the same



Fig. 13 (a) Shows fracture surface of specimen 1 and (b) a different segment of the same fracture surface with higher magnification. (c) Shows fracture surface of specimen 22 and (d) a higher magnification segment of same fracture surface showing dimpled pattern and atypical feature resembling a small pearlite colony or stretched dimple pattern (circled). (e) Shows a general view of the fracture surface of specimen 5 with (f) showing a higher magnification segment of the same fracture surface with dimple pattern and some flatter surfaces

aging treatment with different specimens—the aging treatments were varied to obtain more varied information from the measurements. However, for the pre-straining part that all of the experiments have in common, comparable measurements can be extracted from the stress–strain curves of the 22 specimens shown in Fig. B 1. It can be observed that the experimental scatter in absolute stress levels at comparable deformation is larger than the experimental scatter in stress increments between comparable deformations, because variability of the microstructure between specimens contributed to variability of absolute stress levels but not stress increments. Compared to the stress increment $\Delta\sigma$ due to aging shown in Fig. 5, the variability shown in Fig. B 1 does not include the contribution of reproducibility of the aging heat treatment on



Fig. 14 (a) Shows defective segment of the fracture surface of specimen 5 with slag and impurities. (b) Shows a higher magnification of pore/cavity with impurities on the surface

the experimental scatter in the data shown in the figure. In that sense, the error bar in Fig. 5 is an underestimate. But the variability in the strain hardening stress increment does include a contribution from the variability of the microstructure to the extent and uniformity of plastic deformation which is not present in the stress increment purely due to aging. In that sense, the error bar in Fig. 5 is an overestimate. That variability in the strain hardening stress is found to be larger immediately after yielding than later, so the error bar is calculated from the second half of the common part of the pre-straining stressstrain curves. Comparing the trendlines of the effect of prestrain on $\Delta\sigma$ between two sets of specimens aged separately for 1 day at 100 °C, and measured with DIC or with a conventional extensometer, respectively, the excess and neglected contributions to the error bar appear to be similar in magnitude, because the scatter within each series is smaller than estimated from those error bars but the offset between them is closer in magnitude to the estimated error.

Natural scatter for the elongation at maximum force seems to be high and no clear correlations or trends can be seen in Fig. 6. Some estimation of the scatter in the A_{gt} value can be made from the two as-received datapoints (ID: 1, 26) or if we assume based on Fig. B 2 that aging without pre-strain does not affect the material, we may consider those datapoints of the as-received specimens as well (ID:30, 31). However, the most realistic estimation of the scatter can be obtained from the study of Minnebo et al., where they did multiple ordinary tensile tests for similar castings (Ref 18) and found that the elongation at maximum force can vary by a factor of 5 between different specimens from the same casting. Therefore, it is difficult to conclude more from the scatter in the elongations observed in this study, than a general reduction in the elongation with aging.

Most of the yield stress increase occurs already after aging at 100°C for 1 day and little or no further strengthening occurs at longer times or higher temperatures. Some minor effect of strain aging may still occur when increasing the aging temperature from 100 to 200 °C, but aging at 300 and 400 °C results in over-aging as evidenced by the smaller yield stress increments. Waterschoot et al. have proposed that strain aging likely occurs in multiple distinct stages (Ref 42). The first stage is the Cottrell atmosphere phase where pinning of the dislocations by interstitial atoms causes the yield strength to increase. In cold-rolled dual phase steel

with 88% ferrite and 12% martensite, there was a strain aging plateau after 1000 minutes at 50 °C indicating saturation of Cottrell atmospheres. The aging plateau reached was much faster for samples that had higher solute carbon content in the ferrite. Some further aging occurs due to precipitation and carbon clustering. It is likely that also in ductile cast iron the Cottrell atmospheres saturate already after 1 day aging at 100 °C, so further changes after 11 days aging would be due to the other effects such as precipitation hardening and carbon clustering. That is consistent with the observation that the yield point does not change significantly with further aging, but the lower yield stress is slightly increased. In contrast with some steels, where the yield stress increment decreases with increasing pre-strain (Ref 20, 29), in this study on cast iron that was only observed for over-aged specimens. In dual phase steel, Soliman et al. (Ref 43) found that the bake hardening response, i.e., the yield stress increment, depended on the aging temperature and aging time. It increased in the Cottrell atmosphere formation stage and decreased in the later carbide precipitation stage as the pre-strain was increased.

The major differences between steels and the ductile cast iron are the overall carbon and silicon content. As the carbon solubility in ferrite is low, most of the carbon is in the graphite nodules. Nevertheless, the limited carbon content in the ferrite is probably enough for the observed strain aging effects. The higher silicon content of cast iron compared to steels is known to affect the strain aging process by increasing the activation energy of the carbon diffusion. The dislocation locking process and strain aging should be slower with higher silicon contents. This was demonstrated by Seraj et al. (Ref 20) using manganese-silicon steel with about 1.1 wt.% of silicon which had significantly higher activation energy for aging and lower aging susceptibility than a low-carbon steel with 0.03 wt.% of silicon. The effect of the high silicon content in ductile cast iron can also explain why over-aging is observed at higher temperatures in these materials than is typically found for different types of steels (Ref 14, 20, 29). The effect of silicon appears, however, to be more critical regarding dynamic strain aging, where the speed of the dislocation locking directly determines whether serrated yielding occurs at specific combinations of temperature and strain rate.

Another element that needs to be considered is nitrogen, which-similarly to carbon-is small enough to appear as interstitial atom, with a higher solubility in ferrite than that of carbon. In steels the yield point phenomenon and the extent of strain aging are directly related to the amount of carbon and nitrogen in solution in the ferrite. Nitrogen is more sensitive element in strain aging than carbon and already low concentrations of 0.001-0.002 wt.% nitrogen can cause a severe strain aging effect (Ref 27). The measured nitrogen content in the casting I73 is 0.0041 wt.% which is enough for severe strain aging to occur even in the absence of carbon. However, it is not clear how critical the nitrogen content is for the strain aging sensitivity since cast iron already has more than enough interstitial carbon atoms for severe strain aging to occur. Testing alloys with different amounts of nitrogen in the solution would be necessary to evaluate its effects on the static strain aging of cast iron.

Despite the observed hardening and ductility loss of the material due to static strain aging the fracture mechanism does not change. All the examined fracture surfaces showed ductile dimpled fracture despite the strain aging. Even the specimen that fractured before yielding showed the ductile fracture mode. Some flatter facets that could be interpreted as quasi-cleavage were identified. However, these facets were mostly related to casting defects and do not resemble typical cleavage patterns that have been observed in this material (Ref 44). Ductile dimpled fracture was clearly the dominant fracture mode in all the inspected specimens.

All the strain-aged specimens show a remarkable increase in the yield ratio (YR). YR is a measure of ductility and the material's ability to strain harden when plastically deformed (Ref 45). In general, a good engineering practice is not to use a material with yield ratio over 0.8, as the material behavior becomes unpredictable when the yield strength is close to the ultimate tensile strength of the material. Especially, the design codes for offshore industry still use the yield ratio as a design requirement. Earlier, the offshore industry had YR limits as low as 0.7, but the requirements have relaxed to some extent as some modern materials have shown sufficient ductility even with yield ratios above 0.8. However, the YR of 0.8 is still deemed as an appropriate safety standard (Ref 46). ISO 19902 states different yield ratio limits based on the application; 0.8 for chords, 0.9 for braces and 0.75 for connector materials (Ref 46). The YR of the studied ductile cast iron in the reference tensile test is about 0.7, but strain aging with all the prestrains and aging temperatures raises the YR over 0.8. For prestrain level of 2%, the YR raises over 0.9, and for pre-strain level of 3%, the YR becomes even higher close to 1. This may cause unpredictable and sudden fracture in the material especially with the presence of casting defects as was seen in the case of specimen 5. Even though the specimens show a moderate residual ductility after the strain aging in most cases, the material properties are clearly affected by the strain aging and the high YR should be considered in the design.

The static strain aging lowers the elongation to fracture of the material even when the amount of pre-strain is included in the total elongation. The DIC results in this study show that this is because the strains localize differently in the strain-aged specimens than in the as-received material. The as-received material shows almost immediate start of uniform elongation through the whole gauge length when the specimen begins yielding. To the extent that there are any deformation bands, they are diffuse and do not propagate. The observed strain localization in as-received material may simply show the heterogeneity of the distribution of graphite nodules and casting defects. Deformation bands in strain-aged specimens have more in common with the canonical concept of Lüders bands, since the strain localization is accompanied by a sharp yield point in the stress-strain curve. However, the interaction of the heterogeneous distribution of graphite nodules and casting defects with the Lüders effect results in a more complex morphology of deformation bands that only partly propagate like typical Lüders bands. In all the strain-aged specimens, the strain first localizes at some specific site/sites along the gauge length, while the rest of the gauge length remains undeformed.

Of the strain aged specimens, specimen 28 with 2% pre-strain shows the most typical Lüders deformation, as the deformation band nucleated at the specimen shoulder at one end of the gauge length and propagated toward the other end of the specimen. However, this deformation band has a diffuse front rather than having a distinct front between the yielded and un-yielded material. The band seems to arrest at around one-third of the gauge length and the upper half of the specimen yields similarly to the as-received material with strain localization patterns that are not well described as deformation bands. The strain field in specimen 29, with 3% pre-strain, shows immediate localization of strain in the regions where the deformation was highest during the pre-straining. This is somewhat counterintuitive: if the strain aging behavior observed macroscopically may be extrapolated uniformly to the local behavior, then the locations with the highest pre-strain would have the highest yield point after aging, and yield last. This kind of clear immediate strain localization was not seen in the specimens that are pre-strained for 1% and 2%. It may be due to a change in the local strain aging behavior after very high local pre-strains, to local damage initiated during pre-straining, to stress concentrations at casting defects or at graphite nodules, or to some combination of these.

The spatial distribution of strains in relation to the spatial distribution of the graphite nodules in ductile cast iron was investigated by Kasvayee et al. (Ref 21) Microscopic shear bands were found to form between graphite particles/nodules. It is possible that some of the banding behavior seen in the DIC calculations may be at least partly due to similar localization of strain between graphite nodules and the static strain aging is likely amplifying this banding behavior further.

The studied pre-strain levels are in line with the possible strains that the canister insert may be imposed to in the repository conditions due to a rock shear movement. The higher pre-strains of 2% and 3% are pessimistic and not likely to occur in practice. Based on the mechanistic understanding of the phenomenon, it is expected that static strain aging does not occur without prior yielding in the cast iron insert. This means that the canister needs to be subjected to two rock shear movements that cause yielding, or there would need to be as yet unexamined situations that cause plastic deformation in the cast iron insert, for static strain aging to have an impact on the mechanical properties of the cast iron insert. All the strain-aged specimens except one (specimen 5) fulfilled the minimum ductility requirement of 3% plastic strain (Ref 4). This means that even if two rock shear movements would occur for the same canister, the canister may survive without failing, despite the static strain aging occurring after the first rock shear movement. However, as demonstrated in this study, it is possible that the material breaks before yielding after SSA has occurred. This may occur due to the contribution of casting defects and impurities in the cast together with the static strain aging effect.

In this study, it was demonstrated that even a small plastic deformation of 1% causes marked static strain aging effect on the ductile cast iron at low temperatures and with short aging times. Static strain aging occurs readily already at room temperature for the studied material after the plastic deformation. These findings confirm that SSA may occur in the repository conditions and the

effects of strain aging on the ductile cast iron insert should be considered further. It is also important to consider the conjoint effects of the other possible embrittlement mechanisms relevant for the repository conditions, such as hydrogen embrittlement, irradiation embrittlement and creep, that may affect synergistically with strain aging and cause the cast iron insert to become brittle. The effects of static strain aging and hydrogen embrittlement seem to be different, as they affect the material through different mechanisms. Unlike static strain aging, introducing hydrogen to ductile cast iron does not cause the yield point phenomenon to occur. It has been demonstrated that the fracture of ductile cast iron occurs by brittle cleavage mode when loaded with hydrogen (Ref 44, 47) unlike the SSA specimens. However, these phenomena manifest similarly reducing the total elongation to fracture of the material.

The effect of strain rate during the strain aging was not considered in this study. As a result, there remains concern regarding the dynamic strain aging of the cast iron insert. In the repository conditions, the canister can be subjected to a wide variety of different strain rates due to the long time-span and different types of rock movements. This could result in conditions where dynamic strain aging occurs already at low and moderate temperatures. There have been some studies regarding the dynamic strain aging of ductile cast iron, but the strain rate dependency has not been determined yet, and whether the DSA occurs already at low temperatures below 125 °C when the strain rate is low enough has not been demonstrated. Another aspect to consider is that the behavior of the ductile cast iron was studied here only in uniaxial tensile loading. The possible loading conditions of the cast iron insert in the repository include compressive and multiaxial loading, and considering the marked asymmetry of the deformation behavior of ductile cast iron under compressive and tensile loading the strain aging effects should also be studied under different loading conditions.

5. Conclusions

Dog-bone-shaped tensile specimens of ductile cast iron were pre-strained to 1%, 2% and 3% plastic strain and then aged at varying temperatures ranging from RT to 400 °C. The study demonstrated that SSA occurs in all the tested conditions for the ductile cast iron. SSA already occurs after 1% plastic strain and aging at RT. SSA manifests as a pronounced elevated yield point as well as reduced elongation to fracture. The dislocation locking by interstitial solutes seems to already saturate after aging at 100 °C for 1 day. Considering the yield stress increment some further aging may occur at 200 °C but the effect is small. Aging at higher temperatures of 300 °C and 400 °C results in an over-aging, leading to a lower increase in the yield stress compared to the lower temperatures and a less pronounced and sharp yield point. The DIC study demonstrates that SSA changes the strain localization in the material. The as-received material yields uniformly throughout the whole gauge section, whereas the SSA specimens show a fast nucleation of complex deformation bands which in most cases do not propagate through the gauge section leading to higher strain localizations and reduced elongation to fracture.

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Author Contributions

Conceptualization, funding acquisition and methodology were done by V.B., H.H. and S.B.; software, V.B.; formal analysis, investigation, writing—original draft preparation, visualization and data curation were done by V.B.; writing—review and editing and project administration were done by H.H., S.B.; supervision was done by S.B. All authors have read and agreed to the published version of the manuscript.

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Data Availability

The research data are available from the corresponding author upon reasonable request.

Conflict of interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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Appendix 1

DIC Camera Parameters

During the tensile tests, images for DIC were recorded at 500 ms time intervals, corresponding to 0.008 mm cross-head displacement.

See Table 4.

Table 4 Camera parameters after calibra	tion. Two values of the same pa	rameter refer to cameral and ca	amera2, respectively	
Specimen ID: Parameter	26	24	28	29
Image center X [px]	1078.4/1088.18	1077.45/1072.12	1077.25/1071.42	1078.76/1068.16
Image center Y [px]	2061.27/2127.59	2046.88/2115.62	2046.07/2114.07	2045.22/2112.78
Focal length in X [px]	16573.9/16578.7	16535.4/16532.9	16537/16534.8	16540.1/16536.4
Focal length in Y [px]	16573.9/16578.9	16535.4/16532.9	16537/16534.8	16540.1/16536.4
kappa 1	-0.112345/-0.0867573	- 0.106448/ $-$ 0.0902324	- 0.105139/ $-$ 0.0898123	- 0.12959/ $-$ 0.0889555
kappa 2	2.56854/1.61638	1.43499/0.922843	1.35275/0.891792	1.13369/0.739622
Average image scale [px/mm]	48.6393/49.2306	48.2728/48.7747	48.3216/48.8243	48.2863/48.7923
Min image scale [px/mm]	46.4795/46.829	46.5514/46.8031	46.5517/46.8035	46.356/46.8024
Max image scale [px/mm]	51.1151/51.7308	50.8453/51.3373	50.8452/51.3369	50.8447/51.3363
Average FOV [mm × mm]	$84.21 \times 44.57 / 83.20 \times 44.04$	$84.85 \times 44.91 / 83.98 \times 44.45$	$84.77 \times 44.87 / 83.89 \times 44.40$	$84.83 \times 44.90 / 83.95 \times 44.43$
Rotation angle around optical axis Alpha [deg]	0.191493	0.190616	0.187828	0.185675
Stereo rotation angle Beta [deg]	36.4158	36.4706	36.4714	36.4819
Tilt angle Gamma [deg]	-0.1565	-0.183669	-0.187254	-0.190619
Translation X [mm]	-200.572	-200.098	-200.111	-200.109
Translation Y [mm]	-0.31647	-0.247938	-0.23661	-0.228348
Translation Z [mm]	64.8264	64.5965	64.6142	64.6128
Baseline/distance between cameras [mm]	210.789	210.266	210.284	210.282

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Appendix 2

Additional Stress–Strain Curves

See Figs. 15 and 16.



Fig. 15 Stress-strain curves of the tensile specimens during the pre-straining part showing repeatable yielding behavior up to 3% pre-strain in all tested specimens



Fig. 16 Stress-strain curves of non-pre-strained specimens aged for 1 day at 100 °C and 200 °C

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