

The Susceptibility of Spheroidal Graphite Cast Iron to Hydrogen Induced Stress Cracking

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ABSTRACT

Due to its attractive combination of cost, mechanical properties and castability, use of Spheroidal Graphite Cast Iron (SGCI) has lately, to some extent, replaced steel for use in structural and mechanical components in subsea applications.

Subsea structures are typically protected by use of sacrificial anodes attached to the host structure. Under such conditions nascent hydrogen is generated on the surface of the protected material due to the cathode reaction, hence Hydrogen Induced Stress cracking (HISC) is a constant concern for subsea components subjected to tensile stress. In this work, the SGCI's resistance to Hydrogen Embrittlement (HE) has been examined by use of Slow Strain Rate Test (SSRT) and Stepwise Constant Load (SCL) test. Since structural steel is the main competing candidate material for such subsea applications, two grades of SGCI have been compared to two structural steel grades with similar mechanical strength.

The HISC tests have been supported by fractography characterization and microstructural examination by use of Optical Light Microscope (OLM) and Scanning Electron Microscope (SEM) in combination with use of Electron Back Scattering Diffraction (EBSD) for grain size distribution measurements.

Key words: spheroidal graphite cast iron, nodular cast iron, ductile iron, hydrogen induced stress cracking, hydrogen stress cracking, hydrogen embrittlement, slow strain rate testing, stepwise constant load testing, incremental step load testing, oil & gas, subsea.

INTRODUCTION

Cast Iron with its ancient history, traced back to 6th century BCE¹, has been used for centuries to anything from manhole covers & fire hydrants to bridges. However, the development of Spheroidal Graphite Cast Iron (SGCI) or Nodular Cast Iron, in the 1940's, with resulting improvement in mechanical properties such as ductility and fracture toughness, paved the way for further growth in industrial usage of cast iron.² The material has been adopted by several industries such as automotive-, nuclear-, and wind turbine industry. During the last decade, SGCI has gained increased attention as construction material for subsea equipment in offshore oil & gas production, mainly competing with welded and bolted steel assemblies.

The attraction gained for SGCI use in subsea equipment is caused by its combination of cost, mechanical properties and castability. The good castability of the material allows for near net shape manufacturing of configurations initially consisting of assemblies joined together by fasteners or welds. Casting these initial assemblies into one part, results in a) part reduction, by combining multiple parts into one, b) elimination of fasteners and welds, c) reduced machining time, d) reduced assembly time, e) reduced waste and f) reduced transport/logistics. In an industry with extensive documentation requirements, simplification by reduction of parts and joints may contribute to significant cost reduction.

While SGCI is a rather new construction material for subsea equipment, with limited published research, carbon steel has been widely used for structural and pressure applications in subsea equipment for the last 50 years. Since carbon steels' resistance to Hydrogen in various environments is well studied, it was decided to compare SGCI's resistance to Hydrogen with carbon steel by subjecting two grades of steel and two grades of SGCI with fairly similar strength to HISC testing.

This paper presents the results of SSRT and SCL testing of the two types of construction material to evaluate the suitability of SGCI for subsea applications. Both types of materials' chemical composition, mechanical properties and microstructure were mapped as part of the test program.

EXPERIMENTAL

Characterization of Test Materials

Sample material from four commercial heats of material, two grades of SGCI: Cast Iron A (GJS-400-18-LT) and Cast Iron B (GJS-500-7) and two grades of carbon steel: Steel A (S235 JR) and Steel B (S420G2+M) were obtained for the experimental tests.

Tensile testing: The tensile properties were verified by two control tensile tests in air at ambient temperature for each test material. The tensile tests performed in a calibrated tensile test machine at a strain rate of 1.0 mm/min (in air).

Microstructure analysis: Test samples were taken from cross-sections of the fractured tensile specimens (opposite end of fracture) for microstructure characterization. The microstructure of each of the test materials was examined using both Optical Microscope (OM) at 100X magnification, Scanning Electron Microscope (SEM) at 1000X and 5000X magnification and energy dispersive X-ray spectroscopy (EDS).

Surface preparation of the samples followed ASTM¹ E3.³ The samples were cut in a Struers Labotom-5 (SiC cutting disc) and cast into an acrylic cold mounting resin. The samples were subsequently grinded (SiC paper #220) and mechanically polished with 9, 3 and 1 μ m diamond paste followed by active polishing with 0.04 μ m colloidal silica particles. Samples were rinsed in cold tap water, ethanol and dried in warm air between the polishing steps. Finally, the samples were then etched in 2 % Nital solution for 15 seconds, rinsed and dried before microstructure examination.

¹ American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.

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Imaging & image processing: The optical micrographs were taken at 100X magnification using bright field illumination in a Zeiss Axio Vert.A1 inverted microscope. Images were post processed with Zen Core² software.

For the SGCI grades, graphite form and distribution were evaluated according to ASTM A247⁴ and ASTM E2567⁵ by use of digital image processing (DIP) with the software iSolution DT⁶. Nodularity, nodule count, graphite type and size were calculated. The nodularity of the graphite is calculated from the shape factor (SF)⁷ (see Figure 1) which is set to 0.6 for the graphite nodules⁵ and minimum graphite length was set to 10 µm. The graphite type is classified from I to VII and graphite size from 1 to 8 in accordance with ASTM A247⁴.

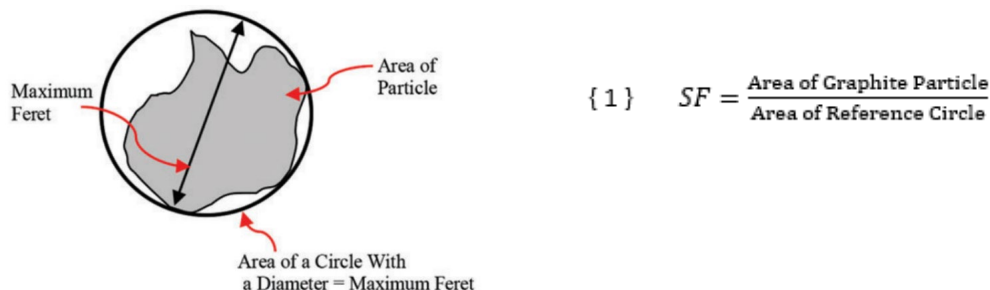


Figure 1: Schematic illustration of maximum feret (a) used to calculate the shape factor SF (b).

HISC Testing

Two types of HISC testing were performed, termed SSRT and SCL testing, respectively. The test methodology consists of comparing tensile specimens tested in hydrogen charging environment as described compared against control specimens tested in air.

Hydrogen Charging: Tensile specimens were pre-charged to obtain hydrogen concentration equilibrium in the test samples prior to testing. Pre-charging was conducted in a 3.5 %wt. NaCl solution at room temperature where test specimens were polarized to -1050 mV_{Ag/AgCl} for 72 hours (+/- 0.5 h). After pre-charging, samples were quickly rinsed in ethanol and dried in warm air before storing at - 19 °C (+/- 1°C) to minimize hydrogen diffusion out.

During the SSRT/SCL testing the specimens were submerged in a 3,5% NaCl solution polarized to -1050 mV_{Ag/AgCl} and hydrogen charged in the test jig for 60 min (+/- 10 min) to restore hydrogen equilibrium before commencing the test under the same hydrogen charging conditions.

Slow Strain Rate Testing (SSRT): Tensile specimens were pulled to fracture during a slow strain rate test in a 50 KN SSRT machine. The tests were conducted on hydrogen pre-charged sample under hydrogen charging conditions (test environment) as described and compared to baseline test in air (control environment).

The testing was executed according to recommended practice ASTM G129⁸ and NACE TM0198⁹. The strain rate was 1.0 µm/min (6.67 x10⁻⁷ s⁻¹). Tests were performed at room temperature (21 °C +/- 1°C).

Stepwise Constant Load (SCL) Testing: SCL test determines the particular load at fracture and follows a proposed test method by Johnsen et al.¹⁰ Tests were performed at room temperature (21 °C +/- 1°C) at a strain rate of 1.0 µm/s (3.94x10⁻⁵ s⁻¹). The pre-charged specimens were loaded until 95% of average ultimate tensile strength (UTS) under Hydrogen charging in 3,5% wt. NaCl solution as described above, where the UTS is determined by control specimens pulled in air (average of two tests). This load was kept for seven days and then subsequently increased with 2 % in load every 24 hour until fracture occurred. Two parallels of each material grade were tested.

² www.zeiss.com

Figure 2 illustrates the tensile specimens used for SSRT and SCL Testing. Tensile specimen gauge length for SSRT was grinded with a SiC #600 grinding foil to reduce surface grooves from machining. Specimens were rinsed in distilled water and ethanol (ultrasonic bath), dried in warm air, and stored in exicator before commencement of SSRT testing. Five measurements of the gauge diameter using a caliper with 0.01 mm were averaged and used for the calculations. After SSRT and SCL testing, fractured specimens were rinsed and stored in the same manner as above prior to further examinations.

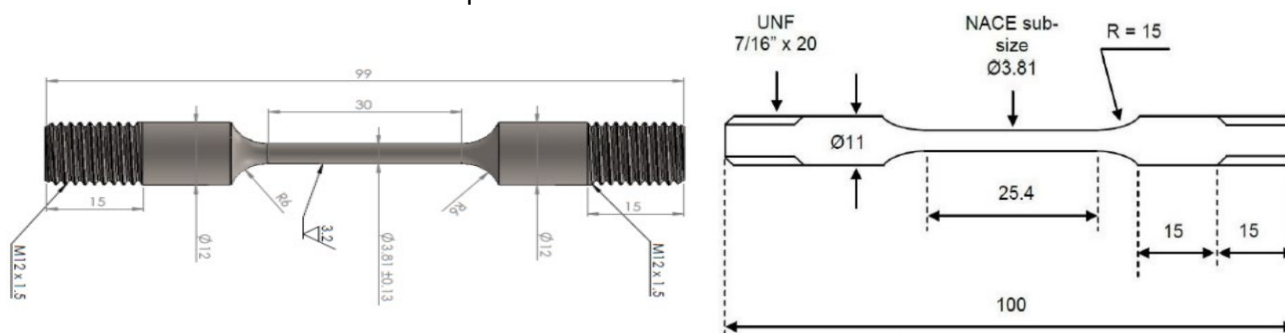


Figure 2: Tensile specimen used for the SSRT testing (left) and the SCL testing (right). Dimensions in mm.

RESULTS

Chemical Composition: The chemical composition from manufacturers ladle analysis, are presented in Table 1.

Table 1
Chemical composition of the materials.

Standard & Grade	Charge	Composition (wt%)																				
		C	Fe	Si	Mn	P	S	N	Al	Mg	Cu	Cr	Ni	V	Ti	Nb	B	Mo	As	Sn	Pb	Ca
EN 10025 S235JR	54136	0,13	Bal.	0,19	0,85	0,012	0,004	0,003	0,043	-	0,04	0,02	0,017	0,001	0,001	0,003	0,0005	0,001	-	-	-	-
EN 10225 S420G2+M	72423	0,09	Bal.	0,29	1,62	0,018	0,001	0,005	0,043	-	0,02	0,06	0,04	0,005	0,015	0,04	0,0003	0,01	0,002	0,003	0,001	0,002
EN 1563 GJS-400-18-LT	53899	3,8	Bal.	2,04	0,23	0,043	0,004	-	-	0,05	0,02	-	0,036	-	-	-	-	-	-	-	-	-
EN 1563 GJS-500-7	53954	3,66	Bal.	2,58	0,39	0,024	0,005	-	-	0,047	0,09	-	0,04	-	-	-	-	-	-	-	-	-

Mechanical Properties: The stress strain curves for the S235JR and GJS-400-18-LT are presented in Figure 3 while the stress-strain curves for S420G2+M and GJS-500-7 are presented in Figure 4. Comparison between the manufacturers certified tensile properties and the control results are presented in Table 2.

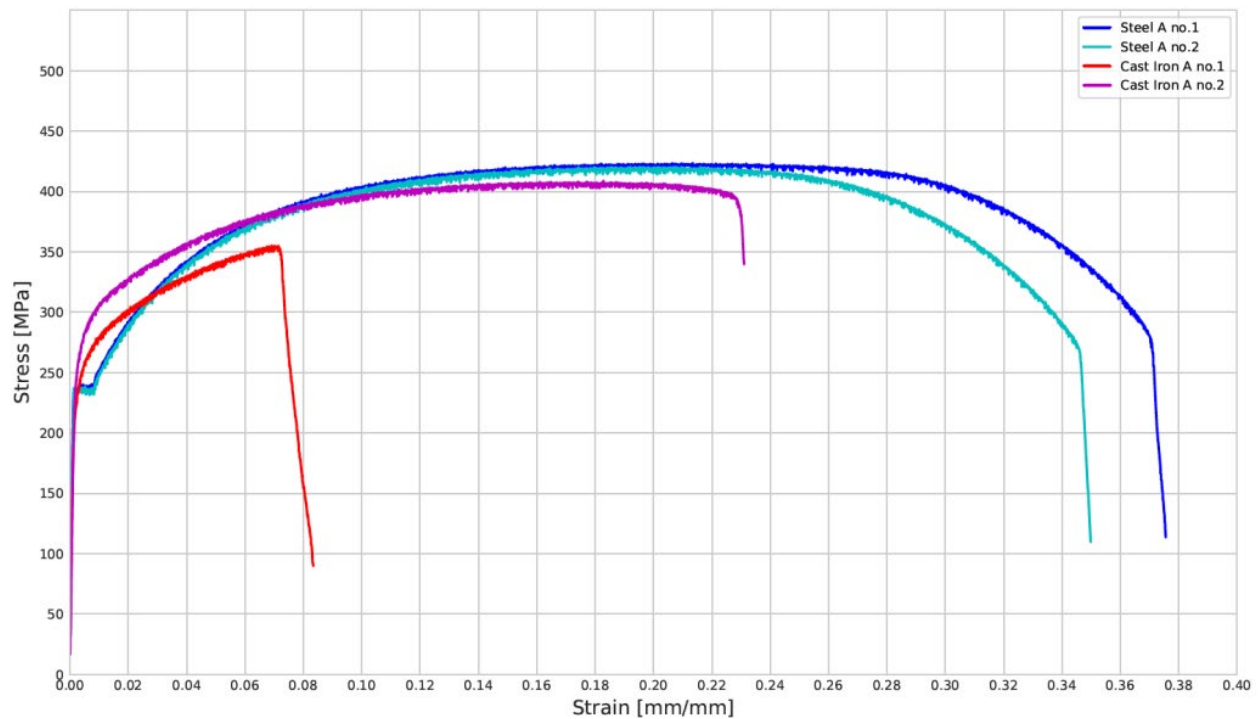


Figure 3: Stress-strain curves in air for Steel A (S235JR) and Cast Iron A (GJS-400-18-LT). Strain rate 1.0 mm/min.

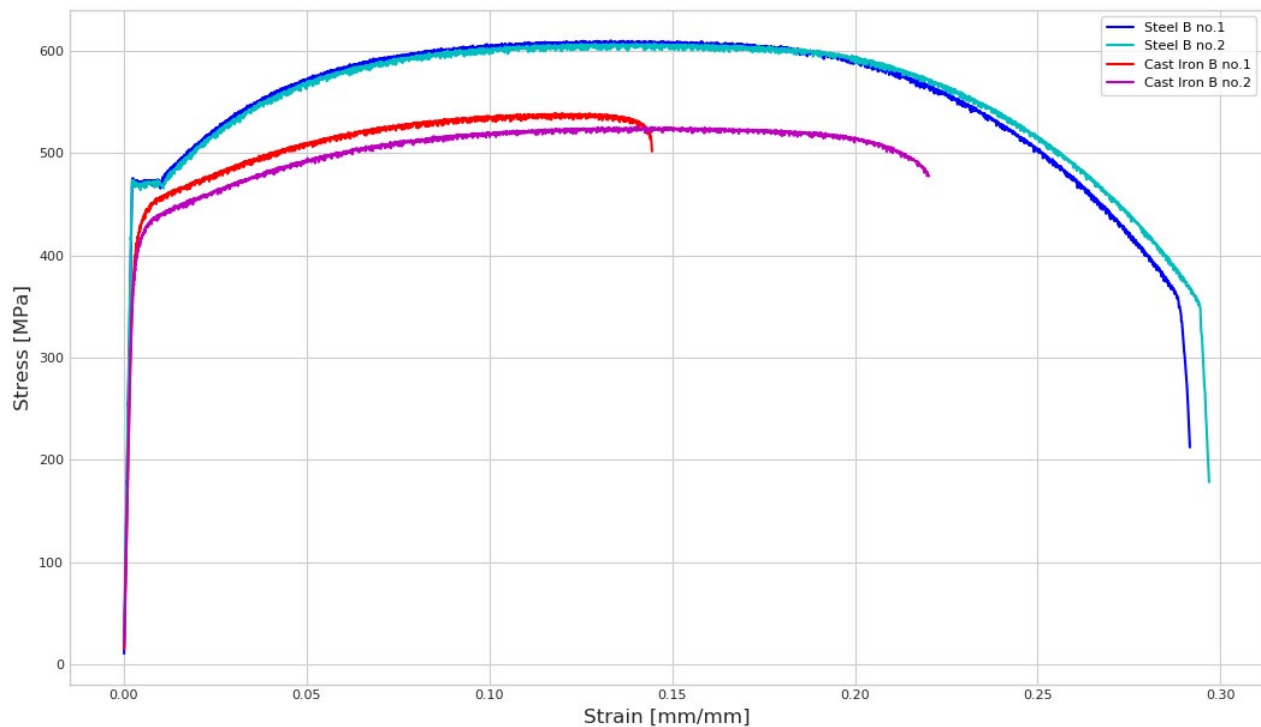


Figure 4: Stress-strain curves in air for Steel B (S420G2+M) and Cast Iron B (GJS-500-7). Strain rate 1.0 mm/min.