Microstructure and damage mechanisms in thermomechanically treated Si-solution-strengthened spheroidal graphite cast iron

SUJAKHU Surendra^{1,a} and CASTAGNE Sylvie^{2,3,b *}

¹School of Mechanical and Aerospace Engineering, Nanyang Technological University (NTU),
50 Nanyang Avenue 639798 Singapore, Singapore

²Department of Mechanical Engineering, KU Leuven (WTK, KU Leuven), Celestijnenlaan 300,
B-3001 Leuven, Belgium

³Member Flanders Make, Celestijnenlaan 300 B-3001 Leuven, Belgium

^asurendra003@e.ntu.edu.sg, ^bsylvie.castagne@kuleuven.be, *corresponding author

Keywords: Ductile iron, graphite, microstructure, thermomechanical treatment, fatigue, damage

Abstract. Spheroidal Graphite Irons (SGIs) are ductile cast irons with toughness and ductility comparable to those of carbon steel. In particular, high silicon Solution Strengthened Ferritic (SSF) SGIs are developed to provide higher strength with excellent ductility suitable for structural applications. The main characteristics of these materials lie in the graphite particles inclusions whose morphology and count greatly influence the mechanical properties and more specifically the fatigue crack initiation and propagation behaviour of the SGI components. In this work, SGIs specimens have been subjected to various thermomechanical treatments in order to analyse the influence of these treatments on the microstructure of the material. Observations of degenerated forms of graphite particles alongside the spheroidal nodules in the microstructure were then used as a basis for correlation with damage mechanisms at the microscale. In static tensile testing, it was observed that the matrix-nodule interface decohesion and plastic deformation of the ferrite matrix were the dominant damage mechanisms. In separately performed fatigue crack initiation and fatigue crack propagation tests, it was confirmed that the graphite particle shape played a decisive role in crack initiation and propagation. The results of the microstructural characterization have been implemented in a computational model for further study of the influence of the microstructure on the fatigue behaviour of these materials.

Introduction

Excellent castability, machinability and affordability have made cast iron a material of choice for many applications. In general, cast irons are considered as brittle iron-carbon alloys but all types of cast irons are not brittle. In particular, ductile cast irons present a toughness and ductility comparable to that of carbon steel. Spheroidal Graphite Iron (SGI) is a type of ductile cast iron that contains spheroidal graphite nodules, providing the added benefits of higher fatigue resistance, toughness and ductility. These unique properties of SGI are attributed to the nodular graphite form, which helps to minimize the stress intensification effect around the graphite nodules and improves the mechanical and fatigue properties of the material [1, 2]. With superior mechanical and fatigue properties, SGIs are undoubtedly better when considering fracture and fatigue. Even if the graphite shape plays a significant role in determining the overall behavior of SGIs, the properties of the iron matrix are also important as most of the applied load is distributed into the matrix.

SGIs used in automobile and wind turbine parts are exposed to repeated mechanical and thermal loads. These applied loads, separately or in combination, make SGI components prone to failure. To increase failure resistance, many postprocessing treatments like heat treatment [3] and surface peening processes are applied [4, 5]. Heat treatment processes change the matrix structure to achieve desired properties [3], and surface enhancement processes like Deep Cold Rolling (DCR) usually introduce compressive residual stress by surface plastic deformation [4, 5]. Whether it is a complete change of matrix in a heat treatment process or a change of the properties of the same

matrix in a DCR process, one common point in both types of processes is that the microstructure of the material change. When it comes to fatigue and failure, it is desirable to design optimized components, to estimate safe loads and lifespan, and to replace the components at the end of their service life. The basis for this approach is the estimation of the component life at the applied load with a reasonable safety factor. However, for most component failures, whether it is by rapid fracture or steady fatigue crack growth, microstructure features are originating points for the fracture and also influence further crack growth [6]. Therefore, the analysis of the microstructural effects is of utmost importance.

In this paper, the effects of thermal cycling and DCR process on SGI microstructure and phases hardness are discussed. Microstructure characterization considerations required for SGI samples treated with similar thermal and mechanical treatment process are presented. Fatigue damage mechanisms on SGI material are briefly discussed considering the material microstructure. Based on the microstructure characterization results and damage mechanisms, multiscale FE modeling approach for eXtended Finite Element Method (XFEM) crack growth is proposed, including preliminary results and the challenges faced with this modeling approach.

Experimental

Materials. EN-GJS-500-14 SGI grade with 3.71 % silicon and chemical composition as presented in Table 1 was studied in this work. Addition of higher silicon content in the melt forms a solid solution strengthened ferritic matrix. The sample material was cast in the form of a 50 mm thick cast plate, and had the mechanical properties of Young's modulus $E = 170$ GPa, yield stress $\sigma_y = 410$ MPa, ultimate tensile strength UTS = 510 MPa and elongation = 17 %. The material was treated with inoculant and nodulizer to form spheroidal graphite, but some fraction of graphite deviated from nodular shape resulting in average nodularity by area of 74 ± 3 %.

Table 1: Chemical composition of EN-GJS-500-14 (%wt.)

C	Si	Cu	Mn	P	S	Mg	Sb	Ceq	Fe
3.2	3.71	0.053	0.182	0.009	0.006	0.042	0.0058	4.15	balance

Thermomechanical treatments. SGIs used for automobile engines suffer major thermal cycle during engine startup and shutdown. The critical components like crankshaft are deep cold rolled to increase their fatigue resistance. It is believed that thermal cycles and DCR process could influence microstructure integrity in SGI materials. In this work, the influence of such process on SGI microstructure is investigated based on separately performed thermal cycling and DCR treatment on as-cast SGI. The treated samples were metallographically polished and studied under an optical microscope (OM) and scanning electron microscope (SEM). The change in phase hardness was also studied using Agilent Nano indenter G200.

Considering the maximum operating temperature of a diesel engine (600 °C), the maximum temperature of thermal cycling was selected as 600 °C. The sample block was heated from room temperature (RT) to 600 °C at 10 °C/min, held for 20 minutes at the maximum temperature and cooled inside the furnace to minimize oxidation and residual stresses. As the maximum temperature of the thermal process was below the austenitization temperature of 727 °C, no change in the matrix phase was anticipated. However, the decomposition of retained cementite into graphite particles and ferrite might occur at the maximum temperature. The most susceptible area in the microstructure was the graphite-ferrite interface due to the mismatch in their thermal expansion behavior and weak nature of the interface bond.

The experimental set-up for the DCR experiments has been described in details in a previous paper [7]. Roller size and rolling pressure have shown major influence on DCR effects, so two levels of each of these parameters were used to study the effect of these parameters on the SGI material during this process. Percentage overlap and feed rate of 400 % and 1000 mm/min were used respectively. ECOROLL tools HG6 (ball size = 6.35 mm) and HG13 (ball size = 12.7 mm)

were used in three-axis CNC machine to perform DCR treatment on as-cast sample block after surface milling and metallographic grinding process, which allowed for the observation of the sample after DCR without additional grinding and with minimum polishing. Rolling pressures of 100 bar and 200 bar were used in the DCR experiments. The main concern in applying DCR treatment on SGI is that the matrix continuity is broken by the presence of graphite particles in the microstructure. Unlike the iron matrix, graphite particles are brittle and could not undergo plastic deformation causing discontinuous matrix flow on the surface. Such discontinuities in the surface layer could significantly affect surface strain hardening and residual stress state.

Thermal cycling. The as-cast EN-GJS-500-14 SGI after 16 thermal cycles (TC-16), 45 thermal cycles (TC-45) and 100 thermal cycles (TC-100) were studied. The reason to perform the analysis after the first 16 cycles is that it was reported that during thermal cycling, the volume change due to local graphitization and filling of already existing vacancies in the graphite particles underwent a change in mechanism [8]. The SEM study after 16 thermal cycles did not show any change in the interface state. The graphite particles were intact in the matrix as in the as-cast SGI. It seemed that the interface strength was not affected during the initial 16 thermal cycles. The only change that might have occurred during the initial 16 thermal cycles was graphitizing of the carbon to fill the existing vacancies and pores in the amorphous graphite particles.

The sample after 45 thermal cycles was studied under the SEM and the results are shown in Fig. 1. Decohesion of some of the graphite nodules from the ferrite matrix could be seen in the SEM images (arrows indicating areas of decohesion). Due to the continuously changing temperature in thermal cycling, thermal stresses were accumulated at the interface ultimately resulting into decohesion of the graphite particles. However, it should be noted that decohesion did not cause complete disintegration of the graphite nodules, only partial decohesion was observed. Further, study of TC-100 sample showed similar interface decohesion. No clear sign of complete decohesion of graphite particles was observed, instead, partial decohesion of interface were observed in a relatively higher number of graphite particles. The average nanoindentation hardness of graphite and ferrite matrix were not influenced by the thermal cycling treatment. The average hardness of 20 indentations on graphite was measured to be 0.64 GPa, 0.62 GPa, 0.67 GPa and 0.68 GPa for as-cast, TC-16, TC-45 and TC-100 samples respectively. Similar hardness result for the ferrite matrix was observed to be 4.87 GPa, 4.84 GPa, 4.71 GPa and 4.85 GPa for as-cast, TC-16, TC-45 and TC-100 samples respectively. The observed results suggested that even though diffusion of carbon was reported and interface decohesion was observed, no significant change on the phase hardness was noticed. Thus, the interface state is the major information that needs to be reported in the characterization of thermal cycled SGI materials.

Deep Cold Rolling. The average nanoindentation hardness of the graphite particles and ferrite matrix after DCR treatment is presented in Table 2. The average hardness of graphite particles was observed to decline after DCR treatment, mostly due to damage of the amorphous graphite phase during the process. The ferrite hardness was observed to increase after cold rolling. Strain hardening due to plastic deformation of the ferrite matrix is the reason for such increase in hardness after DCR process. For the same ball size, if the hardness was compared at different rolling pressures, higher

Fig. 1: Graphite-ferrite interface decohesion after 45 cycles (RT-600 °C) b) magnified view of image a).

pressure showed higher hardness illustrating a positive effect for the rolling pressure. Similarly, comparing hardness at the same rolling pressure for different ball sizes, higher hardness was observed for larger roller ball illustrating a positive effect for the ball size as well.

Table 2: Average nanoindentation hardness and standard deviation (all hardness in GPa)

Phase		As-cast	HG6		HG13	
			100 bar	200 bar	100 bar	200 bar
Ferrite	Average	4.87	5.1	5.45	5.82	6.19
	Std.	0.37	0.37	0.25	0.26	0.29
Graphite	Average	0.68	0.39	0.35	0.56	0.38
	Std.	0.23	0.23	0.17	0.21	0.19

SEM studies of deep cold rolled samples were carried out to examine the influence of DCR on SGI microstructure. The microstructure images for the cold rolled sample (HG13 tool at 200 bar pressure) are presented in Fig. 2. Observation of the cold rolled surfaces (Fig. 2 (a)) showed that the surface microstructure mostly appeared with elongated graphite shapes and plastically deformed ferrite matrix. Microcracks were also observed around some of the graphite particles. It was difficult to measure the graphite nodularity because many surface graphite particles were partially covered by the plastic flow of the ferrite matrix. The samples were subsequently polished using 3 μm and 1 μm diamond suspensions for around 5 minutes in each stage for another SEM study. It allowed clearer observation of the surface microstructure (Fig.2 (b)), exposing graphite particles underlying surface cracks. These surface cracks seemed to connect close graphite particles. It was also noticed that many of the elongated shaped graphite particles were due to partial exposure of subsurface graphite particles very close to the surface. To expose actual morphology of the subsurface graphite particles and characteristics of the observed surface cracks, the sample was further ground for around 5 minutes and 3 minutes using SiC papers P1000 and P2000 respectively followed by polishing as stated earlier. The graphite particles mostly appeared nodular revealing that graphite morphology characterization after DCR could be misleading due to partial exposure of the graphite particles (Fig. 3 (a)) and surface microcracks between close by graphite particles. The surface cracks observed on the rolled samples were mostly limited on the surface, and no sign of deep matrix cracks was found. Further, EBSD analysis was performed on the sample to estimate the influence of plastic deformation on grain orientations. In the IPF map (Fig. 3 (b)), gradient of the crystal orientation could be clearly visualized within a grain. The local crystal misorientations within grains were due to the severe plastic deformation of the ferrite matrix. It could be noticed that the gradient of crystal orientations is significant around the graphite particles.

Fatigue analysis. In static tensile testing, it was observed that the matrix-nodule interface decohesion and plastic deformation of the ferrite matrix were the dominant damage mechanisms. Fatigue damage was investigated by separately performed Fatigue Crack Initiation (FCI) and Fatigue Crack Propagation (FCP) tests [9, 10]. FCI tests exhibited substantial influence of graphite nodules shape on the early crack initiation as most of the fatigue microcracks were initiated from the

Fig. 2: Microstructure study on cold rolled specimen (HG13 at 200 bar pressure) a) after DCR process and b) after additional polishing.

compacted and irregular graphite nodules; either by internal graphite cracking or by decohesion or by a combination of decohesion and internal cracking. Spheroidal graphite nodules, on the other hand, did not initiate microcracks at the time of crack initiation from the degenerated graphite particles, but they showed matrix-nodules interface decohesion and some of the larger spheroidal nodules showed circumferential internal cracks. A quantitative study of graphite nodules damage is reported elsewhere [10]. The fracture surface study of the FCI specimen revealed the presence of large degenerated graphite nodules, signifying the influence of such degenerated graphite nodules as defects on the ferrite matrix. FCP tests were performed on miniature compact-tension (CT) specimens to investigate stable fatigue crack propagation. Spheroidal graphite-matrix decohesion inducing a crack tip blunting effect was the most frequent damage mechanism during the stable crack propagation in the SGI microstructure. The compacted and irregular graphite nodules were mostly fractured when present in front of the crack tip. Crack branching was observed either by the main crack kinking towards nearby graphite nodules or by secondary cracks growth toward the main crack.

Modelling

Methodology. The SGI microstructure simulation was developed starting from an homogenized representative volume element (RVE) model to a multiscale model. A three-graphite RVE model (Fig. 4 (a)) was formulated, in which graphite particles size, distance between them and graphite-to-ferrite proportion were estimated from average microstructural characterization results. The graphite phase was assumed purely elastic and a power-law hardening elastic-plastic behavior was used for the ferrite matrix. A surface-based cohesive approach was used to represent the graphite-ferrite interface, allowing decohesion of graphite particles, and the interface properties were approximated by comparing the overall stress-strain in the simulation to the experimental curve. XFEM based (extended finite element method) crack initiation and propagation was studied in the RVE model, and implemented in a multiscale FE model of the CT specimen (Fig. 4 (b)) in ABAQUS. The microstructure submodel was developed with the image-based finite element software OOF2.

Fig. 4: a) Representative Volume Element (RVE) model and b) Multiscale FE model of CT specimen (colored figure: online version).

Fig. 3: Microstructure after additional grinding and polishing (HG13 at 200 bar) a) SEM image and b) Inverse Pole Figure (IPF) map (colors: see online).

Results. In the RVE model, higher stress and strain bands were observed connecting larger graphite particles, and crack initiated at one of the high strain concentration regions at the graphite-ferrite interface. The cohesive interface enabled graphite particles to be modeled as bound particles until critical stress was reached, which was later modeled as partially decohesed particles. Implementing the simulation approach to a multiscale model showed inhomogeneous microstructure strains comparable to the digital image correlation (DIC) strain maps. XFEM available in ABAQUS was explored to simulate crack initiation and propagation. The simulation suffered convergence problems. One major limitation in the current XFEM formulation was its inability to initiate multiple cracks even if the initiation criterion had been reached at multiple points. Also, convergence was stopped just before the crack at the notch was about to connect the decohesed graphite particles.

Conclusions

Solution strengthened SGI cast iron was thermal cycled and deep cold rolled to study the effects of such thermal and mechanical treatments on the SGI microstructure. For thermal cycling below austenitization temperature, partial decohesion of the graphite-ferrite interface was noticed due to different thermal expansion behavior of two phases. Surface matrix plastic deformation seemed to influence graphite particles characterization, and surface cracks were observed on the cold rolled sample. The fatigue damage study showed dominant influence of graphite particle shape, elongated graphite particles mostly initiating early cracks. Multiscale FE model well represented inhomogeneous microstructure stresses and strains. However, the simulation could not converge up to full crack propagation with the current model but crack initiation could well be evaluated.

Acknowledgments

The authors acknowledge Profs. Jarfors and Salomonsson from Jönköping University, Sweden for providing the EN-GJS-500-14 material used in this work and advising on the modeling, as well as Prof. Sakaguchi from Tokyo Institute of Technology, Japan for his support with fatigue testing.

References

- [1] C. Labrecque and M. Gagne, Review ductile iron: fifty years of continuous development, *Can. Metall. Q.* 73 (1998) 343-378.
- [2] D.F. Socie and J. Fash, Fatigue behavior and crack development in cast irons, *Trans. Am. Foundrymen's Soc.* 90 (1982) 385-392.
- [3] I.A. Chakrabarty, Heat treatment of cast irons, in: M.S.J. Hashmi, *Comprehensive Materials Finishing*, Elsevier Inc., 2017, pp. 246-287.
- [4] G. Cevik and R. Gurbuz, Evaluation of fatigue performance of a fillet rolled diesel engine crankshaft, *Eng. Fail. Anal.* 27 (2013) 250-261.
- [5] I. Altenberger, Deep rolling—the past, the present and the future, *Shot Peening*, (2005) 144-155.
- [6] *ASM Handbook, Fatigue and Fracture*, ASM International. Vol. 19 (1996).
- [7] A. Lim, S. Castagne and C.C. Wong, Effect of deep cold rolling on residual stress distributions between the treated and untreated regions on Ti-6Al-4V alloy, *J. Manuf. Sc.* 138 (2016) 111005, 8 pages.
- [8] T. Matsushita, E. Ghassemali, A.G. Saro, L. Elmquist, A.E.W. Jarfors, On the thermal expansion and density of CGI and SGI cast irons, *Metals*. 5 (2015) 1000-1019.
- [9] S. Sujaku, S. Castagne, M. Sakaguchi, K.A. Kasvayee, E. Ghassemali, A.E.W. Jarfors, W. Wang, On the fatigue damage micromechanisms in Si-solution-strengthened spheroidal graphite cast iron, *Fatigue Fract. Eng. Mater. Struct.* 41 (2018) 635-641.
- [10] S. Sujaku, S. Castagne, M. Sakaguchi, K.A. Kasvayee, E. Ghassemali, A.E.W. Jarfors, W. Wang, Micromechanisms investigation on fatigue crack initiation and propagation in high silicon spheroidal graphite cast iron, 8th Low Cycle Fatigue Conference. (2017) 276-281.