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# Microstructural strain mapping during in-situ cyclic testing of ductile iron

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ABSTRACT

This paper focuses on local strain distribution in the microstructure of high silicon ductile iron during cyclic loading. In-situ cyclic test was performed on compact-tension (CT) samples inside the scanning electron microscope (SEM) to record the whole deformation and obtain micrographs for microstructural strain measurement by means of digital image correlation (DIC) technique. Focused ion beam (FIB) milling was used to generate speckle patterns necessary for DIC measurement. The equivalent Von Mises strain distribution was measured in the microstructure at the maximum applied load. The results revealed a heterogeneous strain distribution at the microstructural level with higher strain gradients close to the notch of the CT sample and accumulated strain bands between graphite particles. Local strain ahead of the early initiated micro-cracks was quantitatively measured, showing high strain localization, which decreased by moving away from the micro-crack tip. It could be observed that the peak of strain in the field of view was not necessarily located ahead of the micro-cracks tip which could be because of the (i) strain relaxation due to the presence of other micro-cracks and/or (ii) presence of subsurface microstructural features such as graphite particles that influenced the strain concentration on the surface.

## 1. Introduction

The influence of the microstructure constitutes on the variation of the mechanical properties is one of the main subjects in the design process of the cast components that are produced from ductile iron [1]. Ductile iron has been widely used in many industrial applications (e.g. gearboxes, crankshafts) mainly due to its relatively low cost and excellent castability [2]. Ductile iron is a multiphase material containing graphite particles as secondary phases (7–15 vol%) in the matrix. High silicon ductile iron is an alloy of ductile iron with the silicon content around 4 wt% [3]. The metal matrix of this material is mainly ferritic. This alloy is being used in many engineering applications because of its excellent machinability and higher fatigue strength [4,5]. Ductile iron shows specific behavior during loading and fracture (e.g. steady macroscopic crack propagation despite the local cleavage fractures), which is due to microstructural inhomogeneity caused by the graphite particles as a secondary phase [6,7].

An understanding of the yielding and cracking behavior at the micro-scale is crucial for designing more durable materials [8,9]. Crack initiation and propagation in the microstructure of ductile iron has been well investigated by using in-situ testing [6]. Depending on the loading conditions, different micromechanisms are responsible for the failure. Under monotonic tension loading, voids form due to graphite-matrix decohesion with subsequent micro-crack initiation and growth inside and around the graphite particles [10,11]. This occurs at the stress levels close to the macroscopic yield point, in which a kink occurs in the tensile stress-strain response [12]. Eventually, macrocracks form because of coalescence of micro-cracks that link the adjacent voids [13].

Zybell et al. [14] recorded the crack propagation during cyclic loading by performing an in-situ fatigue test under an optical imaging system. During the cyclic loading, early micro-cracks originated from the voids that were formed from decohesion of the degenerated graphite particles and also from the shrinkage porosities. Micro-cracks propagate by partial debonding of most of the spheroidal graphite particles along the crack path to failure [15]. The iron matrix microstructure (ferritic, pearlitic or ferritic-pearlitic) [16] and the graphite morphology [17] can affect the crack propagation rate, so that some micro-cracks are arrested during the fatigue loading in the ferritic-pearlitic interface [18]. Verdu et al. [19] studied the early stages of fatigue crack nucleation and growth using synchrotron X-ray

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tomography, showing that the micro-cracks initiate at the graphite nodules or casting defects in the vicinity of the sample surface. However, most of the micro-cracks were arrested after their length reached the initiating defect size and did not coalesce to form short macro-cracks.

It has been proven that the strain distribution in the microstructure of ductile iron is rather inhomogeneous and micro-cracks initiate at highly strained areas [20,21]. Guillemer-Neel et al. [22] found that the Bauschinger effect in ductile iron was caused by inhomogeneous deformation between matrix and graphite particles which developed a high dislocation density originating from the local plastic deformation in the matrix close to the interface.

Digital image correlation (DIC) [23] has been used for measuring local strain distribution in the microstructure of ductile iron [24]. Fischer et al. [25] investigated the strain distribution in the microstructure of the ductile iron using 3D micro-computed tomography combined with digital image correlation. The amount of local strain was quantified showing that highly strained localized areas were favorable for crack initiation. However, the strain measurements spatial resolution in the microstructure of ductile iron needs to be improved to resolve strain in smaller regions between the graphite particles which are the critical places for crack initiation and propagation.

Two dimensional (2D) DIC can be coupled with SEM imaging for micro-scale and nanoscale deformation measurement [26,27]. In this case, one of the key challenges has been the production of the micro-scale speckle patterns which do not mask the microstructure, in order to relate the measured local strain to the microstructural features (e.g. matrix phase, grains and inclusions) [28,29]. With an ex-situ technique, Carroll et al. [30] used SEM to capture micrographs which were analyzed with DIC to measure local strain inside the grains of steel. The speckle pattern was produced by deposition of micro particles which masked the microstructure. The DIC strain patterns were related to the microstructure by overlaying on the electron backscatter diffraction (EBSD) pattern which was taken before the deformation. Using this method, since the EBSD pattern should be taken before the deformation, it is not possible to overlay the strain patterns which are measured at the maximum cyclic load (i.e. when the crack opening distance is at its maximum during in-situ testing). Therefore, there is a need to generate micro speckle patterns that do not mask the microstructure during SEM imaging.

In a previous work [31], a developed pit etching procedure was used for generating the speckle pattern to measure strain distribution in the microstructure of ductile iron using DIC. However, the method had 2 limitations: (i) the procedure could not be applied to all types of material, and (ii) the size of the speckles (pits) could not be controlled (e.g. smaller speckles are required for higher spatial resolutions). Therefore, a versatile speckle generating method needs to be developed that enables strain measurement in the microstructure depending on the required spatial resolution. In this case, an alternative can be focus ion beam milling (FIB) for generating speckles in the microstructure. A similar method has been developed before by Korsunsky et al. [32] but for different purposes; e.g. milling surface for evaluating residual stresses.

The aim of this study is to resolve the strain distribution in the microstructure of ferritic ductile iron and quantify local strain at the onset of the early micro-cracks during cyclic loading. For this purpose, in-situ cyclic tests were performed inside the SEM using CT samples. The FIB milling method was used to produce a controlled size speckle pattern on the microstructure, which is necessary for improving the resolution of the strain measurement. The strain mapping in the microstructure was performed using the DIC technique. The localized strains were measured throughout the entire test.

**Table 1**
Chemical composition of the cast material.

| Element | C | Si | Mg | Cu | Mn | P | S | $C_{eq}$ | Fe |
|---|---|---|---|---|---|---|---|---|---|
| Weight percent (wt%) | 3.20 | 3.71 | 0.042 | 0.05 | 0.18 | 0.01 | 0.006 | 4.15 | Balance |

## 2. Experimental

### 2.1. Material

The material used in this study was high silicon ductile iron grade GJS-500-14, cast in a 50 mm thick plate. The composition is presented in Table 1.

The microstructure of the material consisted of nodular and irregular shaped graphite particles in a fully ferritic matrix (see Fig. 2). Table 2 presents microstructural and mechanical properties of the cast material [33].

### 2.2. In-situ Cyclic Test

Fig. 1(a) shows the miniature tensile/compression stage (Kammrath & Weiss) with a compact-tension (CT) sample installed for cyclic test. Fig. 1(b) illustrates the dimensions of the miniature CT sample (designed and modified according to ASTM E647 [34]). The force was applied to the samples holes in the X direction. The samples were cut from the cast plate by electron discharge machining (EDM) method, using 0.3 mm wire. Samples were mechanically polished and slightly etched with 5% Nital etchant. The thickness of the samples was 1.05 ± 0.1 mm. The notch of the samples had a round tip with the radius of 0.16 mm.

### 2.3. FIB Milling for Speckle Pattern Generation

Focused ion beam (FIB) milling was used to produce the speckle pattern. This approach was used to provide a high level of freedom and flexibility control over the density and spatial resolution of the speckle pattern. 2500 squares (speckles) with the dimension of 1 × 1 × 1.5 μm (width × length × depth) were milled in a 300 × 300 $\mu m^2$ area. The speckles coordinates were produced using Python coding according to a random and homogeneous Halton sequence distribution. Such a distribution and size of speckles in the mentioned area resulted in minimum speckle size of 3 × 3 pixels. The FIB milling was done using a moderate accelerating voltage of 15 kV and beam current of < 1 nA, in order to reduce the subsurface deformation which can occur due to the high energy FIB milling. Trial measurements showed that the selected speckle depth was the minimum dimension that could provide a good contrast on the ferrite matrix. The size of field of view (FOV = 300 × 300 $\mu m^2$) was selected in order to enable strain measurement around and between the graphite particles. The speckle pattern was applied at two locations in each sample (see Fig. 2): (i) FOV1 in the vicinity of the notch tip, to investigate the strain disruption near the notch and at the onset of the micro-cracks; and (ii) FOV2 at 600 to 900 μm away from the notch, in order to investigate the strain distribution in a location further from the notch, where it was expected to comprise lower stress concentration.

### 2.4. Cyclic Test Design

Prior to the cyclic loading, monotonic tension loading up to failure was performed on several other samples which were made from the cast material. The results showed that the crack initiated (i.e. drop in force) at the notch in a critical stress intensity factor ($K_c$) equal to 48 ± 1 MPa √m. The stress intensity factor (K) was calculated for the CT samples according to ASTM E647 [34]. Thus, the cyclic tests were