Three-dimensional local residual stress and orientation gradients near graphite nodules in ductile cast iron

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Abstract: A synchrotron technique, differential aperture X-ray microscopy (DAXM), has been applied to characterize the microstructure and analyze the local mesoscale residual elastic strain fields around graphite nodules embedded in ferrite matrix grains in ductile cast iron. Compressive residual elastic strains are measured with a maximum strain of $\sim 6.5\text{-}8 \times 10^{-4}$ near the graphite nodules extending into the matrix about 20 μm , where the elastic strain is near zero. The experimental data are compared with a strain gradient calculated by a finite element model, and good accord has been found but with a significant over prediction of the maximum strain. This is discussed in terms of stress relaxation during cooling or during storage by plastic deformation of the nodule, the matrix or both. Relaxation by plastic deformation of the ferrite is demonstrated by the formation of low energy dislocation cell structure also quantified by the DAXM technique.

Key words: Cast iron, Residual strain/stress, Plastic deformation, Differential aperture X-ray microscopy (DAXM), Finite element modeling

1. Introduction:

Ductile cast iron (DCI) is an attractive engineering material, as it has strength and toughness very similar to steel, and the machinability advantages make it very cost effective [1]. An example of industrial applications is the heavy components for wind turbine, e.g. the main shaft. One design requirement for such components is the good fatigue resistance, as the fatigue failure is a main failure mode during their service life [2].

From a microscopic point of view, DCI is a composite material, consisting of graphite nodules embedded in a metal matrix which, in most engineering applications, can be either ferrite, or pearlite or a mixture of the two [1]. The differences in the thermal expansion coefficients between the metal matrix and the graphite nodules can lead to local thermal residual stresses in the composites during cooling from the processing temperature to room temperature [3, 4]. Due to the presence of the local residual elastic stresses, fatigue cracks may be initiated at the nodules because of **overstrain**, as the local residual stresses may be larger than the flow stress of the metal matrix. But they may also be lower as they may relax by plastic deformation of the nodules or the surrounding volume [5]. In the past many studies have been conducted to quantify and model the residual stresses in metal matrix composites containing particles that are harder than the metal matrix, e.g. SiC or Al₂O₃ reinforced aluminum matrix composite [6, 7] and Al/W metal matrix composite [8]. For a system like DCI, where the particles (graphite nodules) are considerably softer than the metal matrix, there has however not been much knowledge about the local residual stress. Many researchers believed the local residual stresses to be minor, considering the fact that graphite is soft; and the local residual stresses were neglected in most micromechanical models [9]. However, recently the formation of residual stress comparable to the material yield stress has been predicted by finite element models in DCI [10]. To optimize design and processing of DCI components, the magnitude of the local mesoscale residual stresses must therefore be known.

It is however a challenging task to quantify local residual stresses experimentally. Recently, the development of new experimental characterization techniques has given promising possibilities. For example, a novel synchrotron X-ray technique, the so-called differential aperture X-ray microscopy (DAXM), has been developed during the last 15-20 years for non-destructive 3D characterization of microstructure and local elastic strains [11,12]. With DAXM, local elastic strain distribution inside individual grains has been measured in e.g. deformed bicrystal Ni [13] and NiAl-Cr(Mo) composite [14]. The use of focused microbeam offers spatial resolutions of sub-micrometers [15].

In this study, we use DAXM to characterize the microstructures and local residual strains/stresses in matrix grains surrounding graphite nodules in a DCI sample. The objective of the study is to answer the following questions: i) are the matrix grains plastically deformed? ii) what is the magnitude of the residual elastic stress and is it comparable with the flow stresses of the cast iron? iii) how the residual elastic stress distributes? and iv) how residual stress relaxes? A DCI sample is chosen based on a previous study, where the fatigue properties of the sample are already available in a large data base. The results on the local residual stresses will provide a new aspect to evaluate the obtained fatigue properties.

2. Material and methods

2.1 Materials

Metal mold DCI was chosen for the study. A specimen was extracted from the head of a sample that has been fatigue tested to fail after ~5 million cycles under stress level 5.5 as described in [16]. It therefore was considered not affected by the fatigue test. The sample consists of almost spherical graphite nodules and a metal matrix with a relatively homogeneous structure, being mainly ferrite with a small fraction of perlite (~5%). The graphite nodules were distributed relatively **homogeneously** in the metal matrix (see Fig. 1a). The mean size (equivalent circle diameter) and

volume fraction of the graphite nodules were $\sim 30 \mu\text{m}$ and 11.5%, respectively. The mean distance between nodules and the maximum nodule size are both $\sim 70 \mu\text{m}$. The ferrite matrix has an average grain size of $\sim 30 \mu\text{m}$ (Fig. 1b), measured by electron backscattered diffraction in a Zeiss Supra 35 scanning electron microscope using a Channel 5 software from HKL Technology. The chemical composition of the sample is listed in Table 1.

2.2. Lattice parameter

It is known that the ferrite lattice parameter depends on the amount of solute elements [17]. For the present material, the main alloying elements, i.e. C, Si, and Mn, were considered for the calculation of the lattice parameter. The resulting ferrite lattice parameter was calculated using a function that was deduced based on various publications [17]:

$$a_0 = a_{Fe} + (3a_{Fe}^2)^{-1} \times [(a_{Fe} - 0.001297w_C)^2(a_{Fe} + 0.011606w_C) - a_{Fe}^3] - 0.0006w_{Si} + 0.0006w_{Mn} \quad (1)$$

where $a_{Fe} = 2.8664 \text{ \AA}$ was the lattice parameter of pure ferrite, and w_x is the weight percentage of x element ($x = \text{C, Si and Mn}$). For the present sample, the Si and Mn contents listed in Table 1 and a maximum solubility of 0.005wt% of C in ferrite at room temperature were used as w_{Si} , w_{Mn} and w_C , respectively. The lattice parameter for the current ferrite was calculated to be $a_0 = 2.8653 \text{ \AA}$ using Eq. 1.

2.3 X-ray tomography

To assist synchrotron measurements, the 3D distribution of the graphite nodules was characterized by X-ray tomography using a Zeiss Xradia 520 Versa micro-CT system. For the CT scan, a polychromatic conical beam with X-ray energies up to 140 keV and 1401 image projections over a rotation of 360° were used. For the present study, two nodules with size $\geq 50 \mu\text{m}$ were selected for residual stress measurements: one nodule beneath the specimen surface, defined as nodule A, and

one nodule exposed to the specimen surface, defined as nodule B, see Fig. 2. Nodule A represents a bulk nodule, while nodule B represents a surface nodule. Part of another nodule, defined as nodule C, which was neighboring to nodule B, has also been illuminated. Some of the results around this nodule are included in the analysis. The distance of center-of-mass between nodule B and C is about 95 μm . Nodule A is about 400 μm away from nodule B/C. The sizes and distances between the center-of-mass of the selected nodules and the specimen surface are given in Table 2.

2.4 DAXM experiment

The differential aperture X-ray microscopy (DAXM) was performed at beam line 34-ID-E at the Advanced Photon Source (APS), Argonne National Laboratory [18]. In the DAXM experiment, the X-rays were focused at the specimen using two non-dispersive Kirkpatrick-Baez (K-B) focusing mirrors. The resulting microbeam has a Lorentzian profile and a full-width half maximum of ~ 0.5 μm . The specimen was mounted on an inclined specimen holder at a 45° incidence angle to the incoming X-ray beam, and was scanned horizontally by moving the specimen stage with a step size of 1 μm . Two sections through the selected nodules were scanned. The detailed scanning position relative to the selected nodules is sketched in Fig. 2b. The Laue diffraction patterns from the whole volume within the incident microbeam were recorded on a flat panel detector (409.6×409.6 mm^2 , 2048×2048 pixels) mounted in 90° reflection geometry 510.3 mm above the specimen. The detector's geometry with respect to the incident beam was calibrated using a standard strain-free silicon single crystal. To resolve the diffraction pattern from each volume element at different depths, a Pt-wire of 50 μm diameter was used as a differential aperture and scanned at a distance of ~ 100 μm from the specimen surface. The Laue patterns at each depth were reconstructed by ray-tracing algorithm using the LaueGo software available at APS beamline 34-ID-E [19]. The

reconstructions were conducted to a depth of about 100 μm into the specimen with a step size of 1 μm .

Two beam modes were used in the present study: first a polychromatic beam was used to determine the orientations and deviatoric strains of the matrix grains; then a monochromatic beam was used for determining absolute lattice parameters of selected grains in the mapped sections. Two examples of depth-resolved Laue diffraction patterns for two probed grains from polychromatic scans are shown in Figs. 3a and 3b. The patterns were indexed, from which the hkl indices of individual spots as well as their corresponding X-ray energies were determined [19]. Based on the energies and spot positions, a Laue spot with high intensity and diffraction vector approximately parallel to the specimen surface normal direction, was chosen for the monochromatic energy scan for each selected grain. From this energy scan, the intensity distributions as a function of the diffraction vector $Q = 2\pi/d$ (Q -distribution) were determined for the selected diffraction Laue spot at all depths, see for example Fig. 3c, which was obtained from the Laue spot (-3 2 5) shown in Fig. 3a. At each depth, the Q -distribution was fitted using a Gaussian function and the center of the distribution, Q_C , was used to determine the absolute diffraction plane spacing, d (see Fig. 3d).

3. Results

3.1. Depth-dependent grain orientation distribution from polychromatic scans

The depth-dependent 3D grain orientation distributions obtained from DAXM around the selected nodules are shown in Fig. 4. Here the ferrite matrix around the selected nodules is shown in color and the graphite nodules are shown as black sphere-like blocks. The three selected nodules are marked by A, B and C. Two neighboring graphite nodules are also partly seen. In the matrix grains, a critical angle of $\sim 0.1^\circ$ is used for revealing the detailed boundary structure, and a critical angle of 3° is used for defining individual grains in the matrix. It is evident that most of the grains are

deformed containing dislocation boundaries with misorientation angles below 1° , and the dislocation boundaries are organized in a cell structure. Only a few grains with relatively small sizes ($< 25 \mu\text{m}$) are nearly deformation-free (see the numbered grains in Fig. 4). This may be related to an effect of grain orientations [20].

The grains around nodule B contain evidently more dislocation boundaries as well as more boundaries with misorientation between $1\text{-}3^\circ$ (seen as thin black lines) than those around nodules A and C. To quantify the local plastic strains in the matrix grains around the nodules, the average dislocation densities grouped within cell-wall dislocation boundaries at different distances from the nodule/matrix interface are calculated based on the microstructure using the following equation [21],

$$\rho = K/D_c \cdot \theta_c/b, \quad (2)$$

where K is a number typically equal to 3, D_c is the average spacing between dislocation boundaries, θ_c is the average misorientation angle across the dislocation boundaries, and b is Burgers vector. All dislocation boundaries with misorientation angles in the range $0.1\text{-}3^\circ$ are included for the calculation, i.e. the grain boundaries are not included. The results are shown in Fig. 5. It is found that the average cell-wall dislocation densities are large at places close to the nodules, and decrease with increasing distance from the nodule/matrix interface. The maximum cell-wall dislocation density around nodule B is $\sim 6.4 \times 10^{12} \text{ m}^{-2}$, which is about twice of those around nodules A and C. The cell-wall dislocation density in the matrix away ($> 20 \mu\text{m}$) from nodule is in the range $1.2\text{-}1.6 \times 10^{12} \text{ m}^{-2}$.

3.2. Monochromatic energy scans

Two grains marked by the white boxes in Fig. 4 were chosen for the monochromatic energy scans to determine the absolute crystallographic lattice plane spacings. Diffraction from the $(-3 \ 2 \ 5)$ and $(-$

3 -3 4) planes was used for the grains around nodules A and B/C, respectively, as their normals are nearly parallel to the specimen normal direction (see Figs. 3a and 3b), with deviation angles of $\sim 3.5^\circ$ and $\sim 3^\circ$, respectively. Based on the measured absolute crystallographic plane spacings and the lattice parameter specified in section 2, the strains along the selected crystallographic directions (roughly perpendicular to the nodule/matrix interface) were determined. For the areas around nodules A and B/C, a step size of 1 μm and 2 μm , respectively, was used.

The results are shown in Fig. 6. For the grain around nodule A, compressive strains are observed for most part of the grain, and they are higher at regions close to the interface than in the interior volume. For the grain in-between nodules B and C, compressive strains are observed at regions close to the nodule/matrix interface, while nearly zero strains are seen in the middle of the characterized area of the grain. The compressive strains at the interface around nodule C are generally larger than those around nodule B. The maximal compressive strain is -8×10^{-4} and -6.3×10^{-4} for the grains around nodule A and B/C, respectively. It should be noted that the maximal compressive strain is found at the interface for nodule C but about 2-5 μm away from the interface for nodule A. Within the grains, strains change rather smoothly, and no abrupt change is seen at the dislocation boundaries (see the microstructure within the white boxes in Fig. 4).

4. Discussion

The present study demonstrates that DAXM is a powerful and unique technique for non-destructive 3D characterization of both microstructure and local residual strain at the mesoscale for the ferrite matrix grains within a 3D volume in the present DCI. By using focused microbeam with size of $0.5 \times 0.5 \mu\text{m}^2$, a spatial resolution of 1 μm in all directions is obtained. With the polychromatic beam mode of DAXM, the local dislocation cell structures around the nodules are revealed with an angular resolution of $\sim 0.1^\circ$. With the monochromatic energy scan of DAXM, the local elastic

strains are measured for the two selected grains with a resolution of 1×10^{-4} . To the best knowledge of authors, no other techniques can provide such good combination of depth-dependent spatial, angular and strain resolutions within local volume of $1 \mu\text{m}^3$.

With these techniques, the results have shown evidently that ferrite matrix grains at the nodules are plastically deformed, most at large nodules. The plastic deformation is reflected in the formation of a dislocation structure at or near the nodules. The dislocations are stored in cell wall boundaries with low misorientation angles, showing that the plastic strain has not been high. The plastic strains are different between matrix grains around the same nodule. This difference may be due to the fact that the grains have different crystallographic orientation, and their plastic properties and deformation microstructure will therefore depend on their orientations [20]. It may also be due to the complex stress pattern around the nodule (see for example Fig. 7a in section 4.2). To investigate the details of this nodule/matrix interaction may be possible by a local crystal plasticity analysis also encompassing the strain gradient in the matrix, which will be performed in the future.

Both plastic and elastic strain gradients are seen in the matrix grains at the nodules. In the following, the existence of the residual elastic strain gradients and their formation mechanism will be discussed. For the latter, a finite element model of residual elastic strain developed in a previous publication [10] is used to calculate the residual stress/strain distribution. The last three questions raised in the Introduction will be addresses based on the discussion. The effects of nodule sizes as well as specimen surface on the residual strains/stresses, and their roles in the material fatigue properties are discussed in the end.

4.1 Residual elastic strain gradients in the matrix grains surrounding nodules

The measured elastic strains depend on the lattice parameter, which according to Eq. (1) varies with variations in local chemical contents. The present monochromatic energy scan shows that there are

significant compressive strains along directions that nearly perpendicular to the nodule/matrix interface, with maximum of $6.5-8 \times 10^{-4}$ and a gradient of $5-6 \times 10^{-4}$ over a distance of $\sim 20 \mu\text{m}$ (see Fig. 6). Although the strain resolution is 1×10^{-4} , one may speculate that the measured strain gradients are just a reflection of gradients of chemical contents in the matrix. This speculation is not inurbane, as it is well known that during solidification process of DCI, certain alloy elements segregate, including the main alloy elements C, Si and Mn in the present DCI [22]. This possibility can however be ruled out by the following analysis.

During solidification Si is generally segregated at the first solidification region around graphite nodules, leading to a high Si content there, and a low Si content at the last solidification region, i.e. at the joints of eutectic cells. Mn segregates in a reverse way, i.e. low Mn content close to the nodules and high content within the eutectic zones [22]. Our measurements were conducted within matrix grains that neighbor the graphite nodules directly. Therefore it is very likely that the characterized region is within the first solidification region, especially for the grain in-between nodules B and C. The Si/Mn contents there should be higher/lower than the average Si/Mn contents in the material. If we assume that the Si/Mn contents were $\sim 20\%$ higher/lower within the characterized region than the average percentage of the sample, i.e. 2.76% and 0.18% for Si and Mn, respectively, the measured compressive strain will reduce only about 1×10^{-4} , which is much smaller than the observed strains. Moreover, it has to be noted that the chemical difference considered here is between the first solidification region and the last solidification regions. The chemical variation within the first solidification region should be even less than that [23]. According to Eq. (1), a strain difference of $5-6 \times 10^{-4}$ requires Si + Mn variation of 2.5-3%, which is almost more than the total average chemical contents of Si + Mn in the material. It is therefore almost impossible that such large chemical variations can exist within the two characterized matrix grains.

Additionally, C is another main element in DCI that can affect the lattice parameter. In the present paper, we assumed the C content to be the maximum C solubility (0.005%) in ferrite at room temperature. From Eq. 1, it can be seen that in order to reduce the lattice parameter, and thus reduce the observed compressive strain, the C content should be even less. However, in total the 0.005% C content changes only the strain about 5×10^{-5} .

Last but not least, a similar magnitude of compressive strains and a slightly larger strain gradient are also seen in the deviatoric ϵ_{33} map for the marked grain around nodule A (see Fig. S3a in the supplementary materials). As the determination of the deviatoric strains does not depend on the input lattice parameter, the measured strain gradient therefore must be real. Based on this evidence and the analysis of chemical contents, it can be concluded that the observed compressive strains and strain gradients over the 20 μm distance from the interface are not simply due to chemical variations. To further analyze the formation of the local residual strains, finite element modeling is conducted and described in the following section.

4.2 Finite element model

Several analytical models [3] are available in the literature to predict the formation of thermal residual stresses in materials containing secondary-phase particles, where Eshelby's equivalent inclusion method [24] is central, assuming homogeneity of the material constituents at the micrometer scale. In ductile iron, however, this assumption does not hold, due to the heterogeneous and anisotropic nature of the graphite nodules [25]. To overcome this limitation, Andriollo et al [10] have recently proposed a new finite element model where the nodules' internal structure is explicitly taken into account. This model was applied to the present ductile iron assuming a graphite volume fraction of 11.5 % and 48 conical partitions in the nodule. Time-independent plastic deformation in the matrix, neglected in the original version of the model, was here simulated via a

standard J2-flow formulation, considering two sets of properties: strain-stress curve at room temperature [26], and temperature-dependent flow stress [27].

The patterns of the predicted residual elastic stresses/strains in the matrix around the graphite nodule are complex, having a cubic symmetry in the specimen coordinate system (XHF) and with each strain component varying along both the circumferential and radial directions. As an example, the predicted σ_{33} component at a section equivalent to that marked by the white line in Fig. 2 for nodule A is shown in Fig. 7a. The stress component is symmetrical with respect to the vertical Z axis but not to the horizontal X axis. This is mainly because the plane normal to the selected mapping section (see Fig. 2) is rotated about 45° from the specimen surface normal direction. For the comparison to the experimental elastic strain data, only profiles of the ϵ_{33} component (in the XHF system) within the region that is equivalent to the white box in Fig. 4a are considered.

Comparison with the results based on the monochromatic energy scans around nodule A (Fig. 6a) is reported in Fig. 7b, where the measured strains are plotted as line profiles, with each curve corresponding to one vertical line marked in Fig. 6a. It can be seen that the general tendency of the measured compressive strains to decrease with increasing distance from the interface is captured by the model. However, the maximum compressive strain close to the nodule and the corresponding decreasing rate are over-predicted by a factor ~ 2 .

There could be several reasons that the experimentally measured maximum compressive strains are smaller than those predicted by the model. Firstly, some plastic deformation in the matrix grains is seen for the selected nodules (see Figs. 4 and 5). The plastic strain predicted by the model is about 2.5×10^{-4} (Fig. 7b), which is relatively small compared to the differences between the measured and modeled strains. At the same time, when a material is plastically deformed and kept at a constant strain, stress relaxation occurs [28]. This mechanism can contribute also to the observed reduction in the measured residual strains. Secondly, it is assumed in the model that no plastic deformation

occurs in the nodule. In reality, this assumption may not hold. Moreover, high-temperature creep could also reduce the elastic strain. And in the model, an isotropic matrix is used, while in reality the anisotropy of the individual ferrite grains may also induce some strain variations (see Figs. S1 and S2 in the supplementary materials).

Another inconsistency between the model and experimental results is that the peak positions of measured maximum residual strains along each line are not as model predicted at the interface, but at a distance of 2-5 μm from the interface for nodule A. The compressive strains drop about 5×10^{-5} – 3×10^{-4} in this range for all lines. This strain drop is however not observed for nodule C (see Fig. 8c), i.e. it may not be a typical phenomenon. It could be due to some local microstructural variations in 3D that have not been captured by the present characterizations.

4.3. Size and surface effects on the residual stresses

By combining the DAXM with the X-ray μ -CT, it is possible to evaluate the effect of the nodule size on the residual stresses. To compare the results at different nodules, the residual stresses were estimated from the measured residual strains by means of the direction-dependent Young's moduli. The Young's moduli along the two crystal directions, $[-3\ 2\ 5]$ and $[-3\ -3\ 4]$, are calculated to be 220 and 270 GPa, respectively, using the elastic constants for pure iron [29].

The calculated stresses as a function of distance from interface along vertical lines are shown in Fig. 8. A similar maximum compressive stresses are seen for the grains around nodules A and C, while relatively small compressive stresses are seen for the grain around nodule B. The 3D sizes of nodules A and C are similar (see Table 2). It is thus reasonable that the compressive stresses around nodules A and C are similar. Nodule B is bigger than the other two nodules. It is therefore interesting that the compressive stresses in the matrix grains are smaller around nodule B than around nodules A and C.

Two major possibilities should be considered. Firstly, as shown in Fig. 5, the average cell-wall dislocation density in the matrix grains around nodule B is higher than those around nodule A. The residual stresses are relaxed more by plastic deformation in the matrix grain around nodule B than those around nodule A. Secondly, nodule B is exposed at the specimen surface. The residual stresses at the interface around nodule B are compressive stresses along the specimen normal direction. When nodule B was completely inside the bulk material (i.e. before polishing), the compressive stresses at one side of the interface were counterbalanced by the compressive stresses at the opposite side, and the local compressive stresses could be maintained. However, when nodule B is exposed to free surface, the compressive stresses opposite to the side we measured were released. The compressive stresses at the inner interface are then pushing the nodule out of the surface, and are consequently reduced. The free surface releases not just the stresses at the interface. The stresses in the middle of the characterized grain between nodule B and C (~15-20 μm from the two interfaces) seem also be affected, for a relatively small magnitude of compressive stresses are seen at that position compared to those away from nodule A ($> 15 \mu\text{m}$ from the interface).

Large nodules are selected for the present study, as they are the critical ones for the fatigue behavior [30]. The maximum residual stress at the selected nodules is about half of the yield stress of the matrix, which is about 297 MPa [27]. The residual stresses in the matrix grains therefore cannot be neglected for the material fatigue properties. The strength increase due to cell-wall dislocation structure in the matrix grains is maximum ~35 MPa for the largest nodule [21]. Altogether, the residual stresses are still not so critical for the fatigue properties, and that might be a reason for the high fatigue cycles for this DCI [16]. For other DCI, where the largest nodule size can be $>200 \mu\text{m}$, the resulting residual stresses may be critical for the fatigue life [16]. However, the large nodule size forms typically due to slow cooling rates, which at the other hand can reduce the residual stresses. How these two factors are balanced needs to be analyzed. At the same time, other factors

as the nodule connectivity and shape as suggested in the previous study may also be critical for the material fatigue properties [16]. The effects of these factors on the residual stresses (thus on the fatigue properties) are planned to be examined in the future.

5. Conclusions

1. The DAXM technique is suitable for characterization of both local plastic and elastic residual strains in the present ductile cast iron. With these techniques, an angular resolution of 0.1° , a spatial resolution of $1\ \mu\text{m}$, and an elastic strain resolution of 1×10^{-4} have been obtained.
2. The ferrite matrix grains at nodules have been plastically deformed as a result of the local stresses, which develop due to the interaction between the nodules and the matrix during cooling. The plastic deformation introduces dislocations which are stored as dislocation boundaries with low misorientations angle in the range $0.1\text{-}1^\circ$, and organized in a cell structure.
3. Compressive residual elastic strains along specimen normal direction are observed at interfaces that are approximately perpendicular to the specimen normal direction. The residual elastic strains have shown gradients with maximum of $6.5\text{-}8 \times 10^{-4}$ near the graphite nodules extending into the matrix about $20\ \mu\text{m}$, where the strains are near zero. These gradients are not caused by local chemical variations.
4. The finite element modeling captures the general trend that the elastic residual strain decrease as a function of the distance from the matrix/nodule interface. However, the maximum compressive strain close to the nodule and the corresponding decreasing rate are over-predicted by a factor ~ 2 . These differences are mainly originated in relaxation processes reducing the strain at the nodules by plastic deformation of the matrix.
5. The free specimen surface releases some of the compressive residual elastic stresses along the surface normal direction. The maximum residual stresses in the matrix grains are about half of

the yield stress of the matrix, which suggests that the local residual stresses cannot be neglected for the fatigue properties of ductile cast iron.

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Figure and table captions

Fig. 1 Microstructures of the DCI showing the graphite nodules and metal matrix. (a) Scanning electron microcopy image and (b) EBSD map. In (a) dark regions are graphite nodules and the rest is metal matrix, while in (b) the black particles are graphite nodules and the colored grains are metal matrix. The colors of the matrix grains correspond to the crystallographic orientation along the specimen normal direction (ND) (see the insert).

Fig. 2. Sketches showing a side view of the detailed scanning positions relative to the selected nodules. The selected nodules are marked by A, B and C. Specimen normal direction (ND) is marked by the black arrow. The white lines represent the projections of the mapping planes illuminated by the incoming X-rays, which are along the Z direction.

Fig. 3 Illustration of orientation indexing based on white beam Laue diffraction patterns and determination of crystallographic plane spacing. (a) and (b) show two examples of indexed depth-resolved Laue diffraction patterns. (a) and (b) are from the grains marked by white boxes in Figs. 4a and 4b, respectively. The crosses in dashed lines in (a) and (b) mark the diffraction center. (c) The diffraction vector, Q , as a function of depth for one wire energy scan corresponding to the (-3 2 5) Laue spot in (a). (d) Fitted intensity distribution for the (-3 2 5) Laue spot for the depth marked by black vertical line at the frame in (c). The center of the fitted Q -distribution, Q_c , is used for determination of the crystallographic plane spacing. The dashed line in (d) marks the Q value for d_0 of the (-3 2 5) crystallographic plane determined based on the lattice parameter in section 2.2.

Fig. 4 Microstructures of the DCI near selected nodules characterized using white beam DAXM. (a) and (b) showing microstructures around the selected graphite nodules A and B/C, respectively. In the maps, dislocation boundaries with misorientation angles in the range of $0.1 - 1^\circ$, $1 - 3^\circ$, and $> 3^\circ$ are shown in thin white, thin black and thick black lines, respectively. The colors of the matrix grains correspond to the crystallographic orientation along the specimen normal direction in the specimen coordinate system (XHF system in Fig. 2). The color code is the same as that in the insert

in Fig. 1b. The two white boxes mark regions where monochromatic energy scans were conducted. The numbers mark grains, which are nearly deformation-free. The two black lines in (a) were caused by a technical fault during the Pt-wire scans, and the data were not recorded for that two positions. The black individual pixels in the matrix away from the nodules are non-indexed.

Fig. 5. Average dislocation density as a function of distance from interface. The dislocation densities are calculated from the dislocation boundaries with misorientation angles in the range 0.1-3° [21]. The error bars were estimated based on the variations in the distances between the dislocation boundaries, reflecting the differences between matrix grains.

Fig. 6 Maps colored according to the strains determined based on the monochromatic energy scans. The strains in (a) and (b) are calculated along the $[-3\ 2\ 5]$ and $[-3\ -3\ 4]$ directions for the grains marked by the white boxes in Fig. 4a and 4b, respectively.

Fig. 7 Comparison between the experimental and modelling results. (a) distribution of σ_{33} component at a X-Z section equivalent to that marked by white line in Fig. 2 for nodule A, calculated assuming linear elastic behavior of the matrix. (b) The ε_{33} profiles from experiments and modeling. The experimental profiles are converted from the data in Fig. 6a, each curve corresponds to each vertical line. The modeling profiles are averaged over 9 lines geometrically equivalent to those in Fig. 6a.

Fig. 8 Residual stresses calculated based on the measured strains from the monochromatic energy scans as a function of distance from interface for nodules. (a) - (c) are for the strains measured in the matrix grains near nodules A - C. The line profiles are calculated based on the strain data shown in Fig. 6, each curve corresponds to each vertical line.

Table 1. Chemical composition of the sample (mass%).

Table 2. Sizes and positions of the selected three nodules.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Graphical Abstract