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Electron Micrographic Digital Image Correlation: Method Optimization and Microstructural banding Case Study

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ABSTRACT

Segregation-induced microstructural banding is commonly encountered in commercial steels, yet its effect on the global mechanical behavior is disputed in the literature due to the difficulty of designing clean control experiments. This work locally compares the deformation of banded phases with unbanded regions in the same microstructure from in-situ electron micrographs analyzed with (optimized) micrographic digital image correlation. To this end, first, the employed electron micrographic digital image correlation (EMDIC) methodology is optimized in terms of its experimental parameters: specimen surface preparation settings, scanning electron microscopy imaging settings (contrast modes, magnification, resolution, and contrast-brightness), and image correlation settings (facet size and facet step size). Subsequently, the strength of the (optimized) EMDIC methodology is demonstrated on a case study on segregation-induced microstructural banding in steel, in which the influence of the banded phase and its morphology is probed by comparing the mechanical behavior of two carefully chosen, extreme cases of banded microstructures: a microstructure containing a continuous, hard band (the martensitic-ferritic system) and a microstructure containing non-continuous, softer bands (the pearlitic-ferritic system). The obtained micro-scale strain fields yield clear insight into the influence of band structure, morphology and band material properties.

1. INTRODUCTION

Microstructural banding is common in industrial steels and its detrimental effects on fracture toughness is well-known. However, contradictory results exist for its influence ductility [1-5], possibly due to the experimental methodologies commonly employed whereby the global material behavior of a banded microstructure is compared with an unbanded microstructure (produced by a homogenizing heat treatment from the banded material). Such homogenizing heat treatments also change the morphology of inclusions, thereby changing the mechanical behavior of the control specimen and making a clean comparison challenging.

This difficulty is overcome in the present work by *in-situ* micro-scale strain field measurements in the scanning electron microscope (SEM) that enable direct comparison of the banded regions with the unbanded regions in the *same microstructure*, avoiding undesired variations introduced by band-removing heat treatments. This approach enables an in-depth analysis of the influence of the band morphology (thickness, continuity, geometry etc.).

This work presents an optimization study of the electron micrographic digital image correlation (EMDIC) analysis addressing specimen preparation, SEM imaging, and image correlation settings. Subsequently, the strength of the (optimized) EMDIC methodology is demonstrated on a case study on the influence of banded phase and morphology on microstructural banding, investigated using two carefully-chosen limit cases of banded microstructures: a microstructure containing a continuous, hard band (the martensitic-ferritic system) and a microstructure containing non-continuous, softer bands (the pearlitic-ferritic system), with both microstructures processed from the same starting material for optimal comparison. The paper is finalized with the conclusions.

2. OPTIMIZATION OF THE ELECTRON MICROGRAPHIC DIGITAL IMAGE CORRELATION METHODOLOGY

Utilizing the scanning electron microscope (SEM) micrographs in digital image correlation analysis (with maximum facet correlation) requires the influence of a number of experimental parameters to be assessed. The most critical parameters for this purpose are: (i) the final finish of the specimen surface following metallographic preparation, (ii) SEM imaging settings and (iii) image correlation settings. In this section, the influence of these parameters are investigated using dual-phase 600 steel dogbone shaped tension test samples. For each parameter, SEM images are made at the cross section of a deforming sample and the facet correlation is investigated using Aramis software (GOM GmbH.).

Surface Finish

Following successive grinding and polishing steps, the dual phase steel surface is prepared with different finishes (i.e. electro-etching, chemical etching, chemical over-etching, and combined electro-etching and chemical etching) to reveal the best method in terms of the achieved contrast. Typical results for (a) electro-etching and (b) combined electro-etching and chemical etching are shown in Figure 1. From a comparison of all results, it was found that the best facet tracking is achieved with a combination of electro-etching and chemical etching.

Fig.1. Influence of specimen surface preparation on correlation quality.

SEM Imaging Settings

Obviously, the SEM imaging settings such as the contrast mode (i.e. secondary electron mode (SE), backscattering electron mode (BSE), or a mixed mode that combines SE and BSE contrast), magnification, contrast-brightness are as critical as the surface preparation of the specimen to achieve successful facet tracking. For this study, a FEI Quanta 600 scanning electron microscope is used to reveal optimized settings for the dual-phase steel considered. This SEM also allows imaging using the mixed SE–BSE mode, as shown in Fig. 2.

For any microstructure, the most challenging regions to track (using DIC) are those without significant topographic or morphologic differences. In the case of dual phase steel, the ferritic grains present the most difficult regions to correlate. It is seen in Fig. 2a that the SE contrast mode allows for visualization of fine dispersoids which appear in good contrast with the ferritic grains. This contrast is lost to a noticeable degree when BSE contrast is also incorporated in the SE image. Also, the contrast between the martensitic islands and the ferrite grains are more significant under SE contrast. Due to these reasons, pure SE contrast is clearly most suited for EMDIC.

Fig. 2. Dual phase steel microstructure viewed in different SEM contrast modes.

Next, the influence of SEM magnification is considered. For *in-situ* experiments, the choice for the level of magnification generally creates a dilemma: either high magnification, hence more details but smaller area of view (which may mean that the investigated microstructural mechanism is not captured, while also small contrast features needed for digital correlation may be lost) or low magnification, hence less details but larger area of view (especially important for capturing randomly occurring events such as damage evolution or fracture). In this work, a third option is investigated, where low magnification but with very high resolution images are captured (12 megapixels), which allows the combination of large area of view with significant level of detail (Fig. 3) at the expense of extended imaging time.

Fig. 3. Low magnification, high resolution imaging combines large area of view with high details level.

Finally, the SEM contrast-brightness have been investigated. The main conclusion from this study is that the facet tracking significantly increases with increasing imaging contrast, as shown in Fig. 4. Note that, this may result in final images that are not “visually” ideal, with respect to general SEM practice.

Fig. 4. Influence of SEM imaging contrast on facet correlation.

Image Correlation Settings

It was first indentified that the most critical parameters for facet tracking are the facet size and facet step size. To determine the ideal approach in determining these settings, computer generated images with a known strain pattern are used (Fig. 5), as this enables the comparison of the DIC-generated strain patterns for different facet size and facet step size with a well-defined reference case.

Fig. 5. Computer generated images with a known strain pattern of $\varepsilon_x = (4\pi/40) \cdot \cos(2\pi x/50)$.

The results are shown in figures 6, which shows the analytical strain pattern together with the strain patterns calculated from digital image correlation for several settings of facet size and facet step size. Optimum results are obtained using the smallest facet size and the smallest step size possible. In real applications, however, measurement noise and lack of contrast are important limiting factors to the applicable facet and step sizes.

Fig. 6. Effect of facet size and facet step size analyzed using computer generated images. In the left graph, the facet size is changed between 25 and 100 pixels for a constant facet step size of 15 pixels; in the right graph, the facet step size is changed between 5 and 20 pixels for a constant facet size of 25 pixels.

3. CASE STUDY: EMDIC ANALYSIS OF MICROSTRUCTURAL BANDING EFFECTS IN STEEL [6]

In-situ uni-axial tensile deformation tests with combined EMDIC micro-scale strain field measurements are carried out for two distinct banded microstructures: 1 mm thick martensitic-ferritic dual-phase steel sheets have a $\sim 5 \mu\text{m}$ thick martensitic band in the center, and ferritic-pearlitic banded steel (produced by austenizing the (undeformed) dual-phase steel) with a morphologically comparable microstructure but for which the continuous martensite band is replaced by a non-continuous pearlite band. Tensile test samples are cut using electro-discharge machining, while the cross sections are metallographically prepared using successive grinding and mechanical polishing steps, followed by electropolishing and etching with 2 vol. % nital solution, which is the optimum surface preparation procedure deduced from the EMDIC analysis in Section 2. Intermittent tensile tests of these materials are carried out using a Kammrath-Weiss micro-tensile stage, placed in the FEI Quanta 600 scanning electron microscope. Following the optimum SEM imaging setting obtained in Section 2, high resolution, low magnification micrographs in secondary electron contrast mode are captured at successive stages of deformation ($\sim \Delta \epsilon_{\text{global}} \approx 0.03\%$). The gray value histograms of the obtained images were adjusted for maximum overlap, and regions of interest are chosen for local strain field analysis, which is carried out by the Aramis software.

The obtained micro-scale strain field measurements of the martensitic-ferritic steel are shown in Fig. 7 and those of the pearlitic-ferritic steel in Fig. 8, which show distinctly different behaviors. Let us first examine the martensitic-ferritic steel. Early in deformation, the formation of local slip lines within individual ferritic grains is observed. With proceeding deformation, the number of slip lines increases and the strain distribution becomes increasingly heterogeneous as a result of the formation of shear bands. The local strain fields reveal that the ferrite grains experience significant plastic deformation, whereas the martensite islands are strained only elastically at this stage ($\epsilon_{\text{global}} \leq 0.05$). The martensite band, on the other hand, shows local regions of high strain at those positions where it is the thinnest, as marked with white arrows in Fig. 1c and 1d. As can be verified from the micrographs, the martensitic band is severely deformed at these particular regions up to its fracture strain, leading to void nucleation. Careful examination of the complete series of strain maps shows that the intra-granular shear band intercepts the martensitic band exactly at these critical locations, making them the first sites where voids nucleate, while the martensite islands (and the rest of the martensite band) show hardly any deformation or void formation.

Fig. 7. EMDIC analysis of martensitic-ferritic dual phase steel with a continuous martensitic band.[6]

Let us now concentrate on the pearlitic ferritic steel shown in Fig. 8. At the first glance, examination of the micrographs and the strain maps at different stages of deformation reveals similar trends in the deformation as found for DP steel: initially formation of the slip lines in the ferritic grains and then the evolution of the strain distribution from homogeneous to increasingly heterogeneous with increasing deformation. However, more detailed analyses reveals that the first shear band initiates at a discontinuity in the pearlitic band (shown with a white arrow in Fig. 8b). As was confirmed by additional measurements, for the case that the band (morphology) is not continuous, the preferential path for the shear bands is through the gaps in the band. With further deformation, this shear band deforms steadily while the rest of the pearlitic band experiences only a limited amount of deformation (Fig. 8c). Only at much higher global strain, another shear band is developed that percolates through the narrowest section of the pearlitic band (shown with an arrow in Fig. 8d), in the absence of any void nucleation.

Fig. 8. EMDIC analysis of pearlitic-ferritic steel with a non-continuous pearlitic band.[6]

These results of the local analysis on two limit cases of banded structures show that the influence of a banded structure on the global properties (YS, UTS, ductility, etc.) of metals is critically dependent on the morphology of the band, as well as the mechanical behavior of the phase that composes the band. In microstructures where there is a continuous microstructural band, shear bands are forced to develop through the band, therefore they percolate through the narrowest section of the band. This forces the banded phase to deform beyond its plastic limit, especially if there is a significant difference in the ultimate strains of the phases composing the banded microstructure (e.g. the case of martensitic-ferritic dual phase steel). For discontinuous microstructural bands, shear bands naturally cross at the gaps within the band, thereby delaying early damage initiation. Obviously, the strength of the banded phase also plays an important role, e.g. a continuous band of a high fracture strength phase may accommodate high local stresses without damage initiation, whereas a low fracture strength phase may not.

4. CONCLUSIONS

In this work mechanical influences of segregation-induced banding in steels is investigated using in-situ tests coupled with real-time micrographic analysis. The microscopic-mechanical approach taken on board here is a strong tool that can be used for extending the understanding on many complex microstructural mechanisms, especially due the possibility of using the obtained micrographs for digital image correlation analysis to obtain the local strains at the micro-level. To optimize the micrographs for the latter analysis, first study investigated the influence of the critical experimental parameters on facet tracking, i.e. specimen surface preparation settings, scanning electron microscopy imaging settings (contrast modes, magnification, resolution, and contrast-brightness), and image correlation settings (facet size and facet step size). Results show that best correlations are achieved with a combination of electro-etching and chemical etching of the samples surfaces, by acquiring high-contrast, high resolution, low magnification SEM images in (pure) secondary electron contrast mode, and using the smallest facet size and facet step size applicable within limits of measurement noise and lack of contrast. Subsequently, these optimized settings are used for a case study on microstructural banding in steel, which revealed a clear detrimental effect for hard bands with a continuous morphology, thereby demonstrating the strength of the (optimized) EMDIC methodology.

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