Characterization of microstructural changes in near pearlitic steels using orientation imaging microscopy - influence of pre-deformation on local sensitivity to thermal degradation

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Abstract. The focus of this study is the degradation of a near pearlitic microstructure under combined mechanical and thermal loadings leading to changes in mechanical properties. More specifically, it is examined how the orientation gradients inside the pearlite colonies, affect the spheroidisation. Samples were extracted from virgin near pearlitic railway wheels and pre-strained, thereafter heat treated at different temperatures. Microstructural characterization by scanning electron microscopy (SEM) and Electron Backscatter Diffraction Analysis (EBSD) was performed and evaluated. Results showed that spheroidised areas appear to have lost their initial orientation gradients after spheroidisation and obtain a more uniform orientation. More sub-grain boundaries are present after exposure to higher temperatures.

1. Introduction
During train operation, frictional heating occurs upon acceleration, braking, curving and occasional slippage. The temperature in the wheel tread can rise to 550 °C during freight operation with some extreme cases going up to 800 °C [1]. Apart from thermal loadings, plastic deformation in the wheel tread surface (“rim”) changes the material microstructure and leads to an increased sensitivity to thermal damage [2]. Since the initial microstructure is predominantly pearlitic, the strength and wear properties of the material depend mainly on the interlamellar spacing of the pearlite [3, 4, 5]. Because of the loadings, spheroidisation of the pearlite is observed and is found to vary between different grains. One reason could be that plastic straining is inhomogeneous, which should give differences in dislocation density and thus orientation gradients in the pearlite colonies. It is known that pearlite colony size and other microstructural characteristics can affect pearlite spheroidisation [6, 7]. However, the influence of orientation gradients within the pearlite colonies on spheroidisation has not been studied extensively.

A previous work [8] has showed that orientation gradients exist within the pearlitic ferrite and also presumably within the cementite that suggests a density of geometrically necessary dislocations is present in the pearlite colonies. It was also shown that colonies within the same nodule have a similar interlamellar spacing [9].

The aim of the present study is to examine the microstructure of pearlitic wheel steels and evaluate how the orientation gradients of the pearlite colonies affect the spheroidisation of the lamellae in pre-strained material. The microstructural changes (and following effects on strength) after exposure to elevated temperature from 250 °C up to 650 °C for three different time durations (4 minutes – 4 hours) was investigated with scanning electron microscopy (SEM) in a previous study [10]. Although lamellae start to break up around 400 °C, spheroidisation is limited below 500 °C [10, 11]. After exposure at
650°C for sufficient time, most areas are spheroidised. In the range 500–650 °C there is a mixture of the above conditions. So these two extremes were considered suitable to study the spheroidisation behaviour of this material using orientation imaging analysis of the pearlite colonies in the material microstructure. This will also help in understanding the effect of microstructural changes on the mechanical properties of this type of steels.

2. Experimental
The material studied is the railway wheel steel grade ER8 according to the standard EN13262 [12]. Its nominal composition is shown in Table 1. Here the commonly used name R8T will be used. The “T” added to the designation marks that the wheels have been rim chilled during production; the wheel tread and flange are cooled with water jets after austenitisation. This creates a fine-pearlitic microstructure with some 7–10% pro-eutectoid ferrite close to the rim with a slight increase in free ferrite and interlamellar spacing with increasing depth [10].

Table 1. Chemical composition of R8T wheel material, maximum levels, in wt%.

|   | C  | Si | Mn | Mo | Cr | Ni | S  | P  | V  | Fe |
|---|----|----|----|----|----|----|----|----|----|----|
| R8T| 0.56 | 0.40 | 0.80 | 0.08 | 0.30 | 0.30 | 0.015 | 0.020 | 0.06 | Bal. |

Samples were extracted from unused (“virgin”) wheels from around 15 to 20 mm below the wheel tread surface; this material has a representative microstructure for volumes exposed to elevated temperatures and mechanical loading during operation. The flange and the side closer to the rim face were excluded as they have shown to have a variation in hardness due to different cooling rates experienced in the rim chilling process. Tensile bars with thickness 5 mm and “dog bone” shape (see figure 1, dimensions in mm) were taken from the same depth. These bars were pre-strained using an Instron electro-mechanic tensile machine to 6.5% longitudinal strain. Two extensometers were used to prove an even strain distribution. Tests were run in strain control at a strain rate of $10^{-4} \text{s}^{-1}$. The extension and cross section in the waist were measured before and after some of the tests to verify pre-straining levels measured by the extensometers. Samples were taken from the waist and cut into pieces around 5x8x8 mm³ large, to be used for heat treatment experiments and then microstructural analysis.

![Figure 1. Pre-strain tensile specimen geometry (in mm)](image)

Two samples heat treated for 4 hours were selected for EBSD analysis. The first was heat treated at 500 °C; here some regions are spheroidised while other pearlite colonies are still intact. The second sample was heat treated at 650 °C where most of the pearlite is almost fully spheroidised. Representative microstructures of the two temperatures are shown in figure 2.
The specimens were mechanically ground and polished to 0.04 μm using a colloidal silica suspension. Etching was done using Nital (3% HNO₃ in ethanol) to gain some topographical contrast and thus be able to map regions of interest. The EBSD measurements were carried out in a LEO 1550 high resolution field emission scanning electron microscope (FEG-SEM). The system was equipped with an EBSD detector (Nordlys, Oxford instruments), a high speed camera for EBSD pattern recording and software for crystal orientation mapping (AZtecHKL). The samples were tilted at 70° and the SEM was operating at an accelerating voltage of 20kV. Crystallographic orientation maps were taken on the pearlite colonies as well as on the pro-eutectoid ferrite with a step size of 130 nm which is around the interlamellar spacing of the pearlite in this material [11, 10]. The EBSD patterns of the pearlitic cementite were of insufficient quality and were thus omitted from the analysis.

One useful map type for this investigation is the kernel average misorientation map (KAM). This type of map reveals short range orientation gradients and allows to distinguish local orientation changes and sub-boundaries. For this calculation the average misorientation between a point on the measurement grid and its neighbours is measured. Misorientations between all neighbouring points within the kernel are then averaged and to exclude the grain boundary effect a 2°-5° sub-grain boundary angle was used as a filter to only focus on small rotations that could also indicate a high dislocation density. For this study a square scanning grid was used as shown in figure 3 and from that the 2nd neighbour shell (5x5) was used to evaluate the material.

![Figure 2. SEM micrographs of the pre-strained R8T after heat treatment (4h) at (a) 500 °C (b) 650 °C](image)

![Figure 3. Kernel shells using a square scan grid](image)
3. Results and Discussion

Figure 4a shows a SEM micrograph from the specimen heat treated at 500 °C. It is possible to find areas that are spheroidised as well as areas with apparently intact pearlitic colonies. The inverse pole figure map (figure 4b) reveals the crystallographic orientation of the grains with respect to the sample normal direction. From the colour coding of the IPF map it can be observed that the pro-eutectoid ferrite grains have random orientations with very few substructure boundaries and minor orientation gradients. Larger orientation gradients exist within the pearlite colonies. The bottom triangle shows the relative clustering density of the poles for this field of view, where red indicates a high cluster density and blue a low cluster density. The texture is mainly remaining from the production, as pre-straining to 6.5% does not give large rotations [13]. The grain boundaries can be observed better in figure 4c where only the boundaries are shown; thick lines indicate the large angle grain boundaries and thin/red lines the low angle grain boundaries. For this study the grain boundaries are separated into large angle boundaries with a misorientation higher than 10° and low angle boundaries with a misorientation between 2° and 10°. The pro-eutectoid ferrite grains are separated mostly by large angle boundaries. The intact and the spheroidised pearlitic areas on the other hand both contain low angle boundaries. The areas that appear grey in the IPF map are the unresolved pearlitic cementite regions that were omitted from the map.

The KAM map in figure 4d reveals the local misorientation of the sample. Since it was pre-strained there is a variation in kernel average misorientation. The intact pearlite colony in the middle of the image appears to have stronger local orientation gradients than the partially spheroidised area in the left. The ferrite grains seem to have smaller orientation gradients (marked with arrows).

Figure 4. Microstructure of a sample heat treated at 500 °C (a) SEM micrograph (b) Inverse pole figure map (IPF) (c) Grain boundary map (d) Kernel average misorientation map using up to 2nd neighbour shell
In figure 5 the sample heat treated at 650 °C is shown. Strong spheroidisation is evident in most pearlitic areas of the microstructure (figure 5a). The inverse pole figure (figure 5b) reveals the crystallographic orientation of the grains with respect to the sample normal direction. Larger orientation gradients appear inside the pearlite colonies with less spheroidised microstructure (circled areas in IPF map). However, most of the spheroidised areas appear with even colours in the IPF map which indicates a more uniform orientation. It is also possible that during the annealing, carbon can diffuse from previous pearlite colonies to the pro-eutectoid ferrite grains and precipitate [14]. These grains would then appear as spheroidised pearlite colonies.

A slightly larger amount of sub-grain boundaries is also visible (red lines) but still in lesser extent inside the pro-eutectoid ferrite grains (figure 5c). The kernel average misorientation seen on the KAM map (figure 5d) is more equally distributed over the field of view. This correlates with a larger fraction of the microstructure being spheroidised. Some pro-eutectoid ferrite grains appear to have a lower local misorientation as in the 500 °C sample.

Figure 5. Microstructure of a sample heat treated at 650 °C (a) SEM micrograph (b) Inverse pole figure map (IPF) (c) Grain boundary map (d) Kernel average misorientation map using up to 2nd neighbour shell
4. Conclusions

The effect of pre-straining and high temperature annealing on the spheroidisation behaviour of a near pearlitic steel was evaluated. Samples were heat treated at various temperatures and durations. Two samples that were heat treated for 4 hours, at 500 °C and at 650 °C, were selected for EBSD examination.

1. Pearlite colonies appear to have orientation gradients presumably both from pre-deformation and initial formation of the pearlite while ferrite grains have a more uniform orientation.
2. Spheroidised pearlite colonies appear to have lost their initial orientation gradients and obtain a more uniform orientation after spheroidisation.
3. A higher annealing temperature introduces more sub-grain boundaries in the material, mostly in the pearlite colonies.

From this initial study it is concluded that further investigation is needed to fully understand the spheroidisation mechanism with respect to the orientation of the pearlite colonies. The next step would be to examine the undeformed specimens of the same material and compare between the two states. A more advanced technique to consider is in-situ EBSD with a heating element inside the SEM. The EBSD maps could then show the initial orientation state of the pearlite colonies and the orientation gradients after the spheroidisation along with intermediate stages.

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