Strength and ductility of heavily deformed pearlitic microstructures

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Abstract. The fracture toughness and deformation behavior of heavily deformed pearlitic steels have been investigated. A strong anisotropy of the fracture toughness and the plastic deformation behavior with respect to the lamellar orientation is observed. The consequences of this anisotropy both for processing and application, as well as for the limits in strengthening, are discussed.

1. Introduction
Eutectoid and hypereutectoid pearlitic steels are commonly used in industry. For example, nearly all rail steels have a fully pearlitic microstructure, and heavily cold drawn pearlitic wires are the standard materials for wire cables for suspension bridges, steel cord wires for tires, and the steel strings for musical instruments. In the last decades a significant increase in strength of such cold drawn pearlitic wires has been obtained [1–9]. With strengths exceeding 6 GPa in tension, such wires have further enhanced the interest in this classical structural material. In these wires the tensile strength has now reached about 1/3 of the theoretical limit, and is significantly higher than of all other currently used structural materials. Additionally, these ultra-high strength wires provide a model material for material scientists to clarify how the theoretical limit of strength of other metallic materials can be obtained.

Our interest in the last few years has been devoted to the analysis of the evolution of fracture toughness as a function of cold deformation, and to the study of the anisotropy in the deformation behavior and ductility of the nanolamellar arrangements of ferrite and cementite. The goal of the present paper is to summarize these new findings and to discuss the impact of these results with respect to the limits for processing and strengthening.

2. Fracture toughness as a function of cold working
For the processing, as well as for the in-service use of pearlitic steels, the evolution of ductility and fracture toughness as a function of strain are essential parameters. The special importance of fracture toughness and its anisotropy for ultra-strong materials has been discussed recently in [10–12]. In this section the evolution of the fracture toughness as a function of strain of pearlitic steels deformed by high pressure torsion (HPT) and of cold drawn wires are summarized. For more details, see [10,12].
The HPT processed pearlitic steel was an R 260 alloy, with a chemical composition of 0.76 wt% C, 0.35 wt% Si, 1 wt% Mn, 0.017 wt% P and 0.014 wt% S. Figure 1 contains scanning electron microscope (SEM) and transmission electron microscope (TEM) images of the undeformed and the HPT deformed microstructure for different equivalent strains viewed along the radial direction of the HPT sample. The size of the pearlite colonies in the undeformed material is in the range of 10-20 µm and the colonies are randomly oriented (figure 1a). Within the colonies the cementite lamellae spacing in the undeformed state is about 200 nm. With increasing strain the lamellae align parallel to the shear plane and the lamellar spacing decreases. As a result of the imposed strain path, unfavorably aligned lamellae become heavily bent and broken into smaller pieces, which align with further shearing (figure 1b). At equivalent strains larger than 8 the lamellae are almost fully aligned (figure 1c).

![Figure 1](image1.png)

**Figure 1.** Scanning electron microscope images of (a) the undeformed state, (b) at an equivalent strain of $\varepsilon = 2$, (c) $\varepsilon = 8$, (d) $\varepsilon = 16$ and (e) transmission electron microscope image at $\varepsilon = 16$.

At an equivalent strain of $\varepsilon = 16$ the lamellar spacing is reduced to 15-20 nm, as observed in the SEM (figure 1d) and TEM micrograph in figure 1e. In figure 2 the corresponding increase of hardness as a function of strain is plotted. The refinement of the microstructure is reflected in the change of the hardness, which increases strongly from 270 HV in the undeformed state to 770 HV at an equivalent strain of ~16. Along with the microstructural changes several studies have proven that a distinctive change in the chemistry of the cementite phase takes place, leading to a non-stoichiometric composition and a supersaturation of the ferrite phase. Therefore, the classical description of heavily deformed pearlite as “ferrite-cementite” composite is not fully straightforward [13–15].
Figure 2. Evolution of Vickers microhardness with increasing equivalent strain (data from [10]).

Compact tension (CT) specimens for fracture toughness measurements were machined from the HPT samples deformed to different numbers of rotations. Depending on the number of rotations and the sample extraction radius the fracture toughness can be measured as a function of equivalent strain. The miniaturized CT specimens had a width of 5.2 mm and a thickness of 2 mm. The fracture toughness for the three different crack plane orientations, labelled as A, B and C as described in the following, were investigated [10]. In the samples with orientation A, the crack plane is parallel to the shear plane and the propagation direction is in the tangential direction of the HPT sample or parallel to the shear direction. For orientation B (data not presented here) the crack plane is perpendicular to the shear plane and the desired crack growth direction is along the axial direction of the HPT sample and perpendicular to the shear plane. For orientation C the crack plane is perpendicular to the shear plane as in orientation B, but the crack propagation direction is in the radial direction of the HPT sample.

The fracture toughness results for orientations A and C are summarized in figure 3. With increasing equivalent strain a significant anisotropy of the fracture toughness develops. For orientation A the fracture toughness decreases at smaller shear strains very quickly and reaches a nearly constant low value at higher strain levels, which is only slightly larger than the fracture toughness of a ceramic. For crack orientation C the fracture toughness increases at lower strains, but then decreases at higher strains to values similar to the undeformed material (about 40 MPa m$^{1/2}$). Fracture toughness values of orientation B, not shown in figure 3, lie between those of orientation A and C. However, the crack immediately deflects into the shear plane (perpendicular to the initial crack plane), and follows the direction of lowest toughness. Therefore, these values do not characterize the mode-I fracture toughness and correspond only to a lower limit for this loading case.
Figure 3. Evolution of fracture toughness with increasing pre-deformation, thus, equivalent strain, for two different loading directions: pre-crack parallel to (orientation A, black dots) and perpendicular to (orientation C, open triangles) the lamellar alignment. Data taken from [10].

Similar fracture toughness experiments have been performed on heavily cold drawn pearlitic wires [12]. The chemical composition of the hypereutectoid steel used was Fe, 0.98 wt.% C, 0.31 % wt. Mn, 0.2 wt.% Cr, 0.006 % wt. P and 0.007 % wt. S. Two wires cold drawn to true strains of 3.1 and 6.52, with corresponding ultimate tensile strengths of about 4 and 6.5 GPa, respectively, were investigated. The diameters of the wires were 120 µm and 24 µm, respectively. Again the anisotropy of these wires has been investigated. The microstructure in the view perpendicular to the drawing direction is very similar to the HPT microstructure perpendicular to shear plane. The thicker wire ($\epsilon = 3.1$) has a lamellar spacing similar to the HPT sample with $\epsilon = 16$ (figure 1d-e). In the thinner wire ($\epsilon = 6.52$) the microstructure is significantly finer. The microstructure viewed along the drawing direction is very different to the HPT deformed microstructure, showing a characteristically curled microstructure. For more details regarding the microstructure and tensile properties of these wires see [9].

In order to measure the fracture toughness micrometer-sized cantilevers with the crack propagation direction parallel to the drawing direction, and single-edge-notched tension samples for crack propagation perpendicular to the drawing direction were tested. The measured anisotropy of the fracture toughness values of the thicker wire ($\epsilon = 3.1$) are comparable to the HPT samples deformed to high strains (compare table 1 and figure 3). The low fracture toughness in the drawing direction (parallel to the lamellae) is comparable to, or somewhat larger than, the lowest values measured parallel to the shear plane of the HPT sample (orientation A). In both cases this is the brittle loading direction. The measured fracture toughness for orientation C of the HPT-processed material is similar to the crack plane perpendicular to the drawing direction in the wire and is in both cases distinctively higher. For both processing techniques this is the ductile loading direction. For the thinner wire ($\epsilon = 6.52$), the material with the highest tensile strength, the fracture toughness decreases somewhat for the crack propagation direction in the drawing direction, but more significantly in the crack propagation direction perpendicular to the drawing direction. Thus, a pronounced anisotropy of the fracture toughness remains. The reason for the pronounced anisotropy in the HPT-processed material and the drawn wires are clearly visible from the fractographs in figures 4 and 5, for the brittle and the ductile loading direction, respectively. In all cases the transition from the pre-crack to the overload fracture is
depicted. For the case of crack propagation parallel to the shear plane and the drawing direction, which is in both cases the brittle direction, a debonding of the aligned lamellar structure is evident (figure 4). In addition, the difference in the lamellar arrangement between HPT and wire drawing, namely the planar or curled microstructural arrangement, is reflected by the topography of the fractographs.

In contrast, for orientation C in the HPT sample, and for the crack orientation perpendicular to the drawing direction of the wire (figure 5), the occurrence of delamination results in a reduction of the triaxiality in front of the crack and permits a ductile failure. For further details see [10,12].

**Table 1.** Fracture toughness values for a pearlitic wire drawn to $\varepsilon = 3.1$ (low deformed) and $\varepsilon = 6.52$ (high deformed). Pre-cracks were introduced parallel and perpendicular to the lamellae orientation [12].

| Specimen        | $K_{IC, \text{parallel}}$ (MPa m$^{1/2}$) | $K_{IC, \text{perpendicular}}$ (MPa m$^{1/2}$) |
|-----------------|------------------------------------------|-----------------------------------------------|
| low deformed-1  | 5.1                                      | 40.1                                          |
| low deformed-2  | 4.9                                      | 42.5                                          |
| high deformed-1 | 3.7                                      | 19.7                                          |
| high deformed-2 | 3.8                                      | 21.1                                          |

**Figure 4.** SEM images of the fracture surfaces with the pre-crack parallel to the lamellae, thus parallel to (a) the HPT shear plane (orientation A) [10] and (b) the wire axis [12].

**Figure 5.** SEM images of the fracture surfaces with the pre-crack perpendicular to the lamellae, thus (a) along the radial direction of the HPT disc (orientation C) [10] and (b) perpendicular to the wire axis [12].
3. **Effect of the lamellar arrangement during compression loading**

In order to compare the mechanical behavior of an undeformed and heavily cold worked lamellar “ferrite-cementite” arrangement, micromechanical experiments on micro pillars of size about $3 \times 3 \times 6 \ \mu m^3$ have been performed. The details of this study are described in [16]. A fully pearlitic rail steel (R260), with a chemical composition as described in section 1, was used. As already mentioned, the lamellar spacing in the undeformed pearlite is about 200 nm (see figure 1) with a colony size of 10-20 \mu m. To study the orientation dependency of such lamellar composites on the plastic deformation, the sample size for the undeformed pearlite has to be significantly smaller than the colony size. The heavily deformed samples were taken from a HPT sample in a region with an applied strain of 14.8; the lamellar spacing in this case was about 15 nm. For better comparison, both the undeformed and the heavily deformed samples had approximately the same dimensions. All samples were machined by focused ion beam (FIB) milling. For each material, three loading conditions, schematically depicted in figure 6, have been investigated to study potential plastic anisotropy.

The loading directions are denoted as parallel (parallel to the aligned lamellae), normal (perpendicular to the ferrite and cementite lamellae) and inclined (a loading direction between these two extrema at an inclination angle of about 45°). Measured load-displacement curves, i.e. technical stress-strain curves, are presented in [16]. In figure 7 the characteristic features are replotted in a schematic diagram to point out more clearly the differences in the deformation behavior, as a function of the loading direction for the undeformed and heavily deformed pearlite.

![Figure 6](image-url) **Figure 6.** Schematic diagrams of the principal compression directions (normal, parallel, inclined) with respect to the ferrite and cementite lamellae alignment.

![Figure 7](image-url) **Figure 7.** Schematic stress-strain curves based on data [16] (a) for the HPT processed state after an equivalent strain of $\varepsilon = 16$ and (b) for the non-HPT processed state, in each case for the lamellae aligned parallel, normal and inclined with respect to the loading direction.

Clearly evident is the enormous effect of HPT deformation, or in other words, the reduction of lamellar spacing from about 200 nm to 15 nm, on the mechanical behavior. Despite the significant
differences in the flow stress of the two materials, there are several similarities, but also some differences with respect to the orientation dependence. The inclined direction for both microstructures is always the softest. A further pronounced similarity is the formation of only one (HPT pre-deformed to $\varepsilon = 14.8$) or two (not pre-deformed) distinct and narrow shear bands for both lamellar spacings in the normal loading direction, which are clearly visible in the SEM images of the deformed samples in figure 8. The formation of these shear bands is associated with a load drop. It seems that the realignment of the lamellar arrangement into the shear direction, compare [16], causes initially some softening and when the lamellar spacing is again sufficiently reduced, or the formed substructure is sufficiently refined (hardened), again a stable deformation takes place, presumably by a growing of the size of the shear band. Common for all three orientations is the start of plastic yielding, which is for the undeformed state at somewhat below 500 MPa and for the nanolamellar ($\varepsilon = 14.8$) at about 2 GPa. However, it should be noted that the exact determination of the onset of plastic yielding cannot be determined very precisely in such micro-compression experiments.

For the parallel and the normal lamellae arrangements with respect to the loading direction, in the undeformed material the early pronounced hardening can be explained easily by a composite model. The cementite behaves elastic and the ferrite is already nearly ideally plastic during this first few percent of plastic deformation. When the plastic deformation of the cementite starts, pronounced hardening diminishes, or is even displaced by softening, for the normal loading direction due to the formation of a shear band.

In the heavily deformed material the parallel and normal orientations behave in a very similar manner during hardening, which indicates that the composite model cannot be applied anymore. After the initial pronounced hardening regime at a flow stress of about 3.5 GPa the parallel orientation exhibits a nearly ideal plastic behavior, whereas the normal orientation shows softening due to the pronounced shear band.

The shape of the pillars after the compression experiment is compared in figure 8 by one representative pillar for each loading direction and lamellar spacing. All samples deform at higher strains on this scale in the form of shear or kink bands, but only the normally oriented pillar deforms primarily in a single band.

Figure 8. SEM images of the undeformed (ultrafine-lamellar) and HPT pre-deformed (nanolamellar) compressed micro pillars, with the lamellae aligned parallel, normal and inclined with respect to the loading direction.
4. Concluding remarks with respect to processing and applications

The presented results have important consequences not only for the processing and application of cold worked pearlitic steels, but also for the limits of obtainable strength and ductility in such materials. There are a large number of papers dealing with the strength of cold worked fully pearlitic steels, see for example [1–9]. In most cases it is assumed that the strength is a sum of different contributions, where the most important assumed mechanisms are the Hall-Petch or Orowan mechanisms, dislocation hardening, and solution hardening by carbon dissolution [8]. In the authors' opinion, at the limit of strength of this type of material not all of these mechanisms are of similar importance. Although the micro-compression experiments provide some hints to the hardening phenomena, in the present paper only the consequences for the cold working and application will be briefly discussed.

Anisotropy of fracture toughness is a common feature of many highly stressed biomaterials like wood or bone. The exceptional properties in certain loading directions cannot be explained without this anisotropy. This is also the case for cold drawn pearlitic wires, where the anisotropy in fracture toughness is a pre-requisite for its exceptional combination of properties.

In the typical loading direction of the wires and sheets for technical applications, or along the directions where the highest tensile deformation during the processing occurs, the fracture resistance is high. It is exceptionally high if one takes into account the strength of these materials [12]. In the directions where the stresses during application are low, or where during processing compressive stresses are present, the fracture toughness is very low. Cracks on the microscale are unavoidable during processing and in service. In the case of tensile loading of sheets or wires such cracks always experience a high fracture resistance due to crack deflection or crack delamination. The decrease in fracture toughness and the decrease in the anisotropy for cold drawn hypereutectoid steel wires with strengths above 6.5 GPa might be one of the main reasons why it becomes more and more difficult to further deform such wires. An important feature of the micro-compression experiments was the pronounced formation of a single “macro” shear band for the loading of the lamellar microstructure in normal direction, i.e. compression loading perpendicular to the lamellar plane. A consequence of this anisotropy in the shear localization seems to be that during rolling the occurrence of macro shear bands at higher strains is much more pronounced than during wire drawing.

![Figure 9](image_url)

**Figure 9.** Optical micrograph of a pearlitic steel (R260) cold rolled at 298 K to a logarithmic thickness reduction of \( \varphi = 1.5 \). Macroscopic shear bands are clearly visible in the transverse section.

From simple geometric considerations, in rolling the lamellae become nearly perfectly aligned to the rolling plane at strains of about 1.5. Such an arrangement is very sensitive to the formation of localized macro shear bands, see figure 9, whereas during wire drawing a curled arrangement of the lamellae develops, and thus is less sensitive to the formation of macro shear bands. Hence, it seems to be easier to reduce the lamellar spacing by wire drawing than by sheet rolling without failure.
The shear deformation in HPT, or on the surface of rails, is realized by a relatively homogeneous shearing. This shear deformation corresponds to deformation of the inclined micro pillar, which has the lowest flow stress. Thus, in HPT or at the surface of a rail, the macro shear direction matches well with the shear direction and with the lowest flow stress, and shearing in any other direction would require higher shear stresses. Hence, this anisotropy helps to stabilize homogenous shear deformation such as that during HPT or shear deformation near the surface of a rail.

The present results demonstrate clearly the importance of the development of an orientation dependence of the fracture toughness, and the plastic deformation both during processing and in-service, of these ultra-strong materials. Whether an extension of this combination of properties to even finer and hence stronger materials is possible, remains an open question.

Acknowledgements
Financial support by the European Research Council under ERC Grant Agreement No. 340185 USMS is gratefully acknowledged.

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