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Anisotropic deformation characteristics of an ultrafine- and nanolamellar pearlitic steel

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ABSTRACT

Micromechanical experiments with $3 \times 3 \times 6\ \mu m^3$ sized micro pillars were used to examine orientation dependencies of the mechanical properties in a severely plastically deformed high strength steel and compared with the undeformed state. For the synthesis, an initially ultrafine-lamellar (UFL) fully pearlitic steel was subjected to high pressure torsion (HPT) transforming the steel into a nanolamellar (NL) composite. Both microstructural states were then tested *in-situ* inside a scanning electron microscope. Within the individual micro pillars, fabricated by focused ion beam milling, the ferrite and cementite lamellae were aligned parallel, normal or inclined to the loading direction. The main findings are: First, the strength and strain hardening capacity is more than doubled comparing the UFL with the NL composite. Second, an anisotropic mechanical response exists in terms of i) strain hardening capacity and ii) stress level at the onset of plateau formation. Third, deformation and localization mechanisms at large compressive strains vary with the lamellae orientation, however they are independent of the lamellae thickness.

1. Introduction

Nanocrystalline (NC) and ultrafine-grained (UFG) materials have gained a lot of attraction due to their remarkable strength. Different procedures of severe plastic deformation (SPD) have been developed to refine microstructures, such as, equal channel angular pressing (ECAP), accumulative roll bonding (ARB) or high pressure torsion (HPT) [1–4]. Thereby, large amounts of strain can be imposed to the material, which causes a tremendous reduction of the structural sizes. For single phase materials, the grain refinement saturates at several hundred nanometers, whereas, for multiphase materials, even the nanometer regime can be attained, such as for pearlitic steels [5].

Pearlitic steels possess a widespread field of applications, as for instance for rails, cables for suspension bridges or steel cord wires. The great potential to control their strength by tailoring the interlamellar spacing has been extensively discussed [6–8]. Similar to single phase metallic materials, an inverse proportionality between strength and interlamellar spacing was found, also known as modified Hall–Petch effect. Hence, by strongly confining the dislocation movement in severely deformed pearlitic steels, ultrahigh strength materials can be produced [9]. To date, a cold drawn cord wire holds the highest tensile strength of all structural metallic materials with ~7 GPa and thereby approaches approximately one third of the theoretical strength limit [10].

A prominent method capable of producing nanostructured materials and so also SPD bulk pearlitic steels is HPT, where a disk-shaped sample is deformed up to large amounts of shear strain [5,11–16]. The microstructural evolution during HPT deformation is characterized by (i) the rearrangement of the colony structure into a well aligned nanolamellar (NL) composite throughout the specimen, (ii) a reduction of the ferrite and cementite lamellae spacing to approximately 25 nm and 2 nm ($\varepsilon_{eq} = 8$) [12] and (iii) a disruption of initially unfavorably aligned cementite lamellae. On the atomic scale, a modification of the chemical composition of the cementite phase takes place. Such observations have been initially reported for pearlitic steels after wire drawing [17] and later on also for HPT deformed materials [15,16], suggesting a partial or even full dissolution of the cementite. EELS measurements on the same material as used in this study, support this view by revealing a change in the electron energy-loss near-edge-fine structure of the carbon-rich areas after an equivalent strain of 8 [12].

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As widely known from composite materials, the aligned pearlitic structure is prone to possess orientation dependent mechanical properties. Up to now, the lamellae alignment has been described as an essential deformation characteristic during SPD [8,18,19], however, only few publications deal with the impact of the lamellae alignment and orientation on the mechanical properties, as for instance scrutinized for the fracture toughness [13]. The major challenge in probing the anisotropic mechanical properties of NL pearlitic steels obtained by HPT is to perform technically reliable experiments considering the small sample dimensions delivered by this technique. To our best knowledge, other SPD-processes such as ECAP or ARB cannot provide comparable high strength materials due to technical processing limitations. Another problem arises regarding the comparison with the ultrafine-lamellar (UFL) pearlitic steel of initial interlamellar spacing having a random distribution of the pearlite colonies. A careful selection and mechanical measurement of a distinctive lamellae orientation is experimentally challenging. A micromechanical testing setup, initially developed to investigate sample size effects [20], was found to satisfy both demands and further allows an *in-situ* investigation of the deformation behavior within a scanning electron microscope (SEM).

In this study, *in-situ* micro compression experiments were conducted on a NL pearlitic steel produced by HPT and compared with the undeformed state, which consists of UFL pearlitic colonies. Micro pillars were focused ion beam (FIB) milled with different lamellae orientations with respect to the subsequent loading direction including a normal, parallel and inclined one. The mechanical response, such as strength and strain hardening capacity, as well as the qualitative deformation and failure characteristics were found to be strongly dependent on the lamellae orientation in the NL as well as in the UFL state.

2. Experimental details

A fully pearlitic rail steel R260 with 0.76 wt% C, 0.35 wt% Si, 1 wt % Mn, 0.017 wt% P and 0.014 wt% S was used, consisting of ferrite and cementite lamellae with an average lamellae spacing of 200 nm and a cementite thickness of 25 nm. HPT was used to refine the microstructure and further details concerning the applied HPT deformation procedure can be found in Ref. [21]. The disk-shaped sample had a thickness, *t*, of 5.9 mm and a diameter of 26 mm, the number of rotations, *n*, was two and the nominal applied hydrostatic pressure was 5 GPa. The material used for the subsequent micro compression experiments was extracted at a radius *r* of 12 mm, which results in an equivalent v. Mises strain ε_{eq} of ~15 according to Eq. (1)

$$\varepsilon_{eq} = \frac{2\pi nr}{t\sqrt{3}} \tag{1}$$

The ferrite lamellae thickness at $\varepsilon_{eq} = 14.8$ is between 15 nm and 20 nm and the cementite thickness is approximately 2 nm according to earlier TEM work [12,13]. As mentioned earlier, for the described experimental conditions, the chemical composition of the cementite becomes off-stochiometric during HPT. Nevertheless, for simplicity the term cementite will be maintained for the severely deformed, off-stochiometric carbon rich phase in the NL pearlitic steel.

Fig. 1 shows the fabrication sequence for the NL micro pillars. For that, a one millimeter thick slice was cut from the HPT disk (Fig. 1a). 3×1.5 mm^2 sized rectangular platelets were cut from this slice in such a way, that the ferrite and cementite lamellae were aligned parallel, normal and inclined (50°) to the later loading direction (Fig. 1b). Subsequently, these platelets were locally thinned with a Hitachi E-3500 cross section polisher to a wedge of 20 μm thickness [22,23], which facilitates quicker focused ion beam milling (Fig. 1c). Final fabrication of the micro pillars was carried out with a Zeiss LEO 1540 XB dual beam focused ion beam workstation enabling a precise control of the microstructural orientation of the cementite and ferrite lamellae with respect to the pillar axis (Fig. 1d). Therefore, coarse milling currents of 2 nA were used, followed by a final polishing of the pillar surface with 200 pA, in order to reduce the impact of ion damage [24] to the very surface area and to ensure a smooth specimen surface. The taper-free micro pillars are $3 \times 3 \times 6$ μm^3 in size, resulting in an aspect ratio of two, which should prevent plastic buckling [25]. Using the same fabrication process, micro pillars were also cut into single pearlitic colonies of the undeformed UFL material, which were between 10 and 20 μm large, for comparison. Since in this case no macroscopically aligned structure was present (no HPT process was applied), it was required to use a platelet including multiple pearlite colonies with arbitrary orientations and selecting appropriate lamella orientations with the FIB being aligned parallel, normal and inclined (22° and 45°) to the later loading direction.

Micro compression experiments were performed *in-situ* inside a SEM (Zeiss LEO982) in compression mode. An ASMEC UNAT micro indenter with a diamond flat punch indenter tip of 20 μm in diameter was used to load the micro pillars. Misalignment between the micro pillar and the flat punch top surface was minimized by overtilting the pillar top surface by 1.3° during FIB milling.

The micro pillars were loaded under displacement control to total displacements of 1500–3000 nm in either one or two loading steps with a constant initial strain rate of 10^{-3} s^{-1}. Videos were recorded in terms of SEM-image sequences, which were taken every few seconds and allow to link the mechanical data with the material's deformation behavior. Furthermore, post-mortem SEM investigations of each micro pillar side surface were carried out to examine changes in the surface morphology. Cross-sections of the tested micro pillars were obtained by FIB milling and subsequently studied with a SEM in order to gain further insights into the underlying deformation mechanisms of the bulk material.

Supplementary videos related to this article can be found at http://dx.doi.org/10.1016/j.actamat.2015.12.037.

The non-specimen related elastic contributions of the measured displacement stemming from the wedge, SEM-stage, specimen holder, as well as from the pillar sink-in Ref. [26] were accounted for by correcting the compliance with a method implemented by Wurster et al. [27]. Finally, engineering stress–strain curves were calculated from the corrected displacement data and the measured force values.

To elucidate the mechanical behavior of the UFL polycrystalline pearlitic steel, compression experiments were performed on comparably large samples with $3 \times 3 \times 6$ mm^3. The samples were tested on a conventional testing rig from Zwick with an initial strain rate of $2*10^{-4}$ s^{-1}.

3. Results

3.1. Microhardness measurements

The microhardness distribution within the HPT-processed steel sample increases from the disk center with 270 HV1 by a factor of three to about 740 HV1 at a radius of 12 mm, similarly to [13]. This hardness gradient along the disk radius reflects the gradual decrease of the interlamellar spacing with increasing strain [5]. Under this experimental conditions a hardness plateau, as reported for many pure metals, is not observed, because of an insufficiently high applied deformation strain. Larger number of rotations would lead to a failure of the HPT-anvils or a slipping of the sample within