Scripta Materialia 142 (2018) 66–69

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Crack path identification in a nanostructured pearlitic steel using atom probe tomography

T. Leitner [a], S. Sackl [b], B. Völker [a], H. Riedl [c], H. Clemens [b], R. Pippan [d,*], A. Hohenwarter [a]

[a] Department Materials Physics, Montanuniversität Leoben, Jahnstr. 12, A-8700 Leoben, Austria
[b] Department Physical Metallurgy and Materials Testing, Montanuniversität Leoben, Franz-Josef Str. 18, A-8700 Leoben, Austria
[c] Institute of Materials Science and Technology, TU Wien, Getreidemarkt 9, A-1060 Wien, Austria
[d] Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Jahnstr. 12, A-8700 Leoben, Austria

ARTICLE INFO

Article history:
Received 31 July 2017
Received in revised form 10 August 2017
Accepted 15 August 2017
Available online xxxx

Keywords:
Severe plastic deformation
Pearlitic steels
Nanocrystalline
Fracture toughness
3-dimensional atom probe tomography

ABSTRACT

Severely plastically deformed pearlitic steels often possess poor crack-growth resistance along the deformation-induced elongated nanolamellar microstructure. However, it is unknown if the crack propagates in the nanocrystalline ferrite or along the ferrite-cementite interface. Here, a pearlitic steel subjected to high pressure torsion exhibiting a fracture toughness of only ~4 MPa·m$^{1/2}$ along the elongated structure was selected to address this fundamental question. For the first time 3-dimensional atom probe tomography was employed to unravel the local atomistic fractography. We present clear evidence that the low fracture toughness is controlled by crack propagation along the interface between the nanocrystalline carbon-rich and ferritic phase.

Nanostructured pearlitic steels are widely used in applications, such as the steel cording for tire reinforcement, requiring their unprecedented mechanical properties [1,2]. Their strength is a result of a nano-scaled cementite-ferrite lamellar microstructure, which can be very effectively refined for example through wire drawing. To date, the obtainable strength levels reach almost 7 GPa which is about 30% of the theoretical strength [3]. Besides the unusual high strength, the fracture properties and damage tolerance are remarkable [4]. One main factor leading to these exceptional fracture properties is the anisotropy in the fracture toughness, with relatively "weak" and "strong" crack paths evolving during deformation [4,5]. A weak direction with relatively poor fracture toughness, for example in wire products, is found parallel to the drawing direction and promotes markedly increased fracture toughness values in the strong testing direction. The strong direction is oriented perpendicular to the weak one and triggered by a delamination process dividing the thick specimen during the fracture process into thinner ligaments. The thin ligaments enable even in thick specimens critical stress intensities and, thus, fracture toughness as normally measured for thin specimens. This mechanism has been reported in metallic specimens subjected to traditional deformation processes such as rolling [6,7] and seems to be quite beneficial in high strength materials subjected to various severe plastic deformation processes as well. The crack plane of the delaminations corresponds to the weak crack path and, therefore, this fracture toughness enhancing mechanism is primarily controlled by the low toughness crack plane. Focusing on nanostructured pearlitic steels deformed by novel severe plastic deformation processes, this behavior was first studied in a pearlitic steel processed by high pressure torsion (HPT) [8]. Here, the intense shear deformation leads to a strong reduction in the lamella spacing and an alignment of the lamellae parallel to the shearing direction, combined with increased strength levels close to 4 GPa performing compression tests [9]. The fracture toughness in this material state varies between 4 and 40 MPa·m$^{1/2}$ in the weakest and strongest testing direction, respectively. This large variation is comparable to the difference between structural ceramics and modern high performance steels [10].

Even though the strengthening mechanisms of nanostructured pearlitic steels have been thoroughly scrutinized [3], their fracture behavior and especially the failure mechanism in the weak testing orientation have not been investigated in detail. On the microscopic scale using scanning electron microscopy it was observed that the crack propagates parallel and along the well aligned lamellar structure, irrespective of the used production method. The exact location of the material separation, i.e. the involved interface for the crack propagation process, is unknown. In general, different scenarios are conceivable. The crack may run along the interface between the ferrite and cementite (interlamellar), in the cementite (intralamellar) or in the nanocrystalline ferrite (intralamellar). A clarification of this issue would be fruitful for future work focusing on grain boundary and interface engineering. Strategies could be developed to strengthen the involved crack path and to increase the

* Corresponding author.
E-mail address: reinhard.pippan@oeaw.ac.at (R. Pippan).

http://dx.doi.org/10.1016/j.scriptamat.2017.08.025
1359-6462/

fracture toughness or even lowering it further to tailor the degree of the fracture toughness anisotropy in a controlled fashion.

Traditional fractography and metallographic cross-sections investigated with scanning electron microscopy (SEM) can provide information about the crack path, for example in wire-drawn pearlitic steels, as long as the lamella distance is still large enough to be resolved [11]. For lamella spacings in the nanometer range a clear distinction of the crack path would become impossible with SEM. Energy dispersive spectroscopy is also often used for fractographic investigations, however, with respect to the length scales in nanolamellar steels the electron interaction volume is too large to analyze the top surface of the fracture surface satisfactorily [12]. Advanced in-situ transmission electron microscopy (TEM) studies combined with chemical analyses would grant sufficient resolution, however, the necessity of using ultrathin specimens cannot guarantee the required constraint ahead of the crack tip. Auger-electron spectroscopy possesses exceptional chemical resolution but needs to be supported by complimentary methods or modelling to yield satisfactory quantitative depth-information. In our view, a feasible method to address this issue is accomplished by advanced 3-dimensional atom probe tomography (3D APT). This technique is capable of resolving the microstructure and the involved interface at the atomistic scale directly on the fracture surface. In this study 3D APT will be employed for the first time to unravel the local atomistic fractography of a nanocrystalline material.

To evaluate this approach and to identify the exact locally occurring crack path during fracture of nanolamellar pearlitic steels, a commercially available fully pearlitic steel was subjected to high pressure torsion using disks of 30 mm diameter and a thickness of approximately 8 mm. The composition of the steel is 0.76 wt% C, 0.35 wt% Si, 1 wt% Mn, 0.014 wt% S, 0.017 wt% P, and Fe in balance. In a former study [8] compact tension C(T) fracture toughness specimens were extracted from different disk-radii of the HPT-disk and orientations, see Fig. 1a, and related to the local imposed pre-strain by calculating the v. Mises equivalent strain. By using this approach it was possible to measure the fracture toughness as a function of pre-strain since the variation of the strain within a single sample is small compared to the investigated deformation regime. For more experimental details regarding the used deformation process and fracture toughness evaluation procedure the reader is referred to the previous work [8]. After imposing an equivalent v. Mises strain of approximately 16, which is realized at a radius of about 13 mm after 3 rotations a strength of nearly 4 GPa was measured [9]. However, a very low fracture toughness of only 4 $MPa \cdot m^{1/2}$ was found for cracks propagating parallel to the HPT deformation induced elongated and aligned microstructure. This “weak” orientation, see Fig. 1a, shows the lowest fracture toughness in the entire investigated deformation range and the crack propagates parallel to the structural alignment which is at the same time parallel to the tangential direction of the HPT-disk, see Fig. 1a. Due to delamination formation the same material state exhibits a fracture toughness which is 10 times higher in another crack propagation orientation, where the crack propagates perpendicular to the tangential direction and is considered the “strong” orientation (Fig. 1a). The failure mechanism of the weak specimen orientation allowing for the exceptionally high damage tolerance in the strong orientation was the focus of the following APT-study.

Fig. 1b shows the corresponding microstructure of the investigated material inspected parallel to the radial direction. The SPD-processing leads to a strongly aligned nanolamellar microstructure parallel to the tangential direction and the lamella spacing is reduced during deformation from about 200 nm of the initial material down to a value in the range of 10 to 20 nm. The thickness of the cementite phase is diminished to only a few nanometers. The fracture surface at this microstructural state, representative for a samples with a fracture toughness of 4 $MPa \cdot m^{1/2}$ in the weak orientation, is presented in Fig. 1c. The fracture surface is fairly smooth implying that the crack follows the strongly aligned structure (Fig. 1b) and shows no further distinctive fracture characteristics except for some step like features, where the crack locally changes its crack plane along the lamellar structure.

To ensure that the pristine fracture surface (Fig. 1c) is accessible in the atom probe, a protective layer was applied to protect the surface during consecutive preparation steps. Furthermore, a site specific specimen preparation is necessary to ensure that the specimen originates from the very top of the fracture surface. In this work different approaches to apply protective layers on the fracture surface before specimen extraction were utilized. In the first approach a platinum layer was deposited on the fracture surface via the gas injection system in the vacuum chamber of a focused ion beam (FIB) Versa 3D from FEI. It was found that an unsatisfactory bonding of this layer to the nanocrystalline pearlite may lead to fracture events during the atom probe measurement. Therefore, a second approach was selected and a chromium layer was deposited by physical vapor deposition (PVD). The thin chromium layer was sputter-deposited in a pure argon atmosphere at room temperature without previous ion etching of the sample to ensure that the fracture surface is not altered in the lab scale deposition system (AJA Orion 5).

After depositing the protective layer on the fracture surface, samples for 3D APT were prepared in a dual beam microscope FIB Versa 3D by using a lift-out technique [13], whereby final tip preparation was achieved as described by Felfer et al. [14]. On the final apex of the tips a protective layer of about 100 nm was left to protect the fracture surface before the final measurement. Afterwards the specimens were measured in a Local Electrode Atom Probe (LEAP) 3000X HR from Cameca Instruments in the laser mode at 60 K. The pulse rate was set to 250 kHz and the laser energy was 0.3 nJ.

In Fig. 2 3D APT reconstructions of a specimen coated with Pt (Fig. 2a) and Cr (Fig. 2b) is presented. For the color-code of the atoms

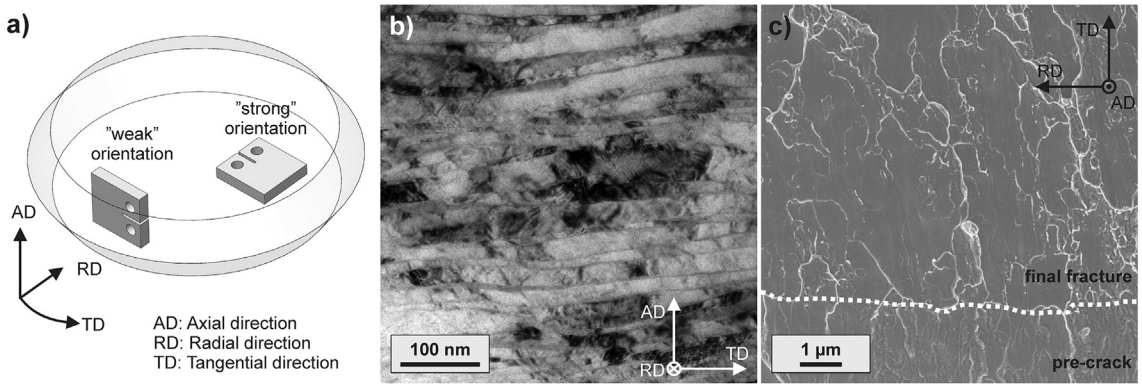

Fig. 1. Features of the investigated material. a) Schematic of the HPT-disk, the C(T) specimens with the weak and strong specimen orientation and the coordinate system with the three principal directions. b) TEM-brightfield micrograph of the nanolamellar steel inspected into the radial direction consisting of a nanocrystalline ferritic and carbon-rich (cementite) phase aligned parallel to the tangential direction. c) Typical fractograph of this microstructure tested in the weak orientation exhibiting a fracture toughness of approximately 4 $MPa \cdot m^{1/2}$ with relatively flat fracture surface. The dotted line separates the fatigue pre-crack surface from the final fracture surface.