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Influence of grain shape and orientation on the mechanical properties of high pressure torsion deformed nickel

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ABSTRACT

Severely plastically deformed (SPD) materials, for example those produced by high pressure torsion (HPT), are reported to possess outstanding mechanical properties. A typical HPT microstructure consists of elongated grains, usually of grain size well below 1 μm, which are aligned parallel to the shear plane and showing typical shear texture components. To answer the question of how these single features of a SPD microstructure affect the mechanical properties individually, such as the yield strength, the ultimate tensile strength, the uniform elongation and the reduction in area, uniaxial tensile tests have been conducted. The samples were tested in two different orientations. Within the same testing orientation the average grain aspect ratio was also varied. The variation in grain aspect ratio within a sample was achieved through a slight back rotation of the already deformed material and selective radius-dependent specimen extraction. The main results are as follows: the ductility (in terms of the reduction in area) is influenced by the grain aspect ratio. In contrast, the ultimate tensile strength is independent of the grain aspect ratio but shows an explicit dependency on the specimen orientation.

1. Introduction

Severely plastically deformed (SPD) materials are promising candidates to exhibit high strength combined with high ductility as shown for instance in [1–4]. A lot of research has been undertaken to identify and understand the physical processes behind this extraordinary phenomenon. Studies focused on both strength and ductility are often performed using equal channel angular pressing (ECAP) processed materials, because standard tension experiments can be performed. The effect of the number of passes and the types of ECAP routes have frequently been studied [5–8]. The same also applies to sheet materials processed by Accumulative roll bonding (ARB), where in two directions conventional tensile tests can be performed [9–11].

Focusig on ECAP, the numbers of applied passes have typically been between 2 and 8, which corresponds to an accumulated shear strain of about 4–16 regarding an intersection angle of 90°. In this strain regime, the grain size and the ratio of high angle to low angle grain boundaries is significantly affected by the number of passes and applied processing route. As a consequence, the strength and ductility related measures are also influenced by these main processing parameters. The shear strain applied by HPT is usually larger compared to ECAP. In most single phase materials or alloys a saturation in grain refinement and a constant ratio between high and low angle boundaries is observed for shear strains larger than 40. This usually results in a higher saturation hardness. Due to the small size of HPT samples, very often only the hardness evolution is examined. Tensile strength and ductility of the saturation microstructure is not frequently investigated and the tensile behaviour can in general only be investigated in the tangential direction due to the thinness of HPT disks [12–14]. The possible anisotropy of strength and ductility regarding different testing directions has not been studied so far—except in some micro experiments [15]. The present study analyses the orientation dependency of strength and ductility of the saturation microstructure. The questions addressed in this paper are

– Does an orientation dependency in the tensile properties exist?
– Is the strength and ductility affected by the grain shape?
– Is the crystal texture developed during HPT important for the tensile properties?

Initially as the microstructure plays a key role in answering the above questions a short summary of the most important parameters controlling the HPT microstructure is presented. The experimental results will then be presented and discussed.

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1.1. Remarks regarding the structural evolution during HPT

The structural evolution during HPT has been extensively investigated in the last 10 years [2,16–24]. The most important characteristics are summarized in order to introduce the investigated saturation microstructure in a more general sense.

At strains larger than 1, a well defined cell structures develop in a metallic material which deforms by dislocation glide. With increasing strain the cell and cell block size decreases and the misorientations between these structural elements increase. This fragmentation reaches a saturation between a shear strain of 20–60 and the boundaries become more similar to ordinary grain boundaries. Further straining does not change the grain size, grain aspect ratio, the texture or the misorientation distribution [17,20,23]. The saturation microstructure is mainly dominated by the following factors.

1.2. Deformation temperature

A dynamic recovery process based on grain boundary migration is, in conjunction with the fragmentation process, responsible for the saturation microstructure. This dynamic recovery process strongly depends on the deformation temperature. An increase in the deformation temperature leads to an increase in the average grain size. In contrast, a decrease in temperature leads to finer microstructure. Further details regarding the temperature dependency are given in [17,25,26]

1.3. Alloying and chemical composition

One major parameter which influences the minimum size of the saturation microstructure is alloying. Alloying of aluminium with magnesium causes a significant reduction of the saturation grain size [27]. The HPT deformation of an alloy consisting of two or more phases should result in smaller grains compared to a single phase material after application of the same strain. One explanation is that the dynamic recovery process is slowed as the presence of phase boundaries reduces the grain boundary mobility. For single phase alloys (solid solution) a size difference between the atoms may have an effect on the saturation microstructure but this is less effective than the presence of two phases.

1.4. Impurities

The total quantity of impurities (in terms of unintentional alloying elements) is less important than the type and amount of the different impurity elements. For example, the presence of 0.06 wt% carbon leads to a hardness increase of 70% as a consequence of the strong grain refinement. Furthermore, this carbon strengthening effect is more pronounced than a change in deformation temperature of 600 °C. A carbon free nickel deformed at liquid nitrogen temperature (−196 °C) achieves a similar saturation hardness than a nickel containing 0.06 wt% carbon (600 ppm) deformed at 400 °C [26].

1.5. Strain path

After applying a large strain by HPT, a shear texture evolves in the saturation microstructure and the single grains are not equiaxed. The longest grain axis is oriented parallel to the HPT shear plane whereas the shortest grain length is achieved parallel to the HPT rotation axis. This is not completely valid as the shear deformation does not lead to a grain orientation perfectly parallel to the HPT shear plane. In reality the grains are slightly tilted with respect to the HPT shear plane by a few degrees [17,20,23]. A change in the strain path, as for example by changing the HPT rotation direction, does not affect the saturation microstructure. The only difference is that the grains are tilted in the opposite direction as shown for cyclic HPT experiments in [20].

The following described experiments have been designed to treat the previously specified questions. From cyclic HPT deformation experiments it is known that it is possible to change the grain orientation without changing the average grain size. This offers the possibility to produce a HPT sample with different grain aspect ratios while keeping a similar grain size. A nickel disc with a diameter of 30 mm is deformed to the saturation regime. Subsequently, the disc is deformed into the opposite direction for a quarter of a full rotation by a simple change of the rotation direction of the HPT equipment. This slight rotation in the opposite direction causes no change in the grain size itself but it changes the aspect ratio of the grains over the disc. As shown schematically in Fig. 1a, the aspect ratio in the disc centre and the outer region is similar but the grain orientation is opposite.

The centre still shows alignment of the grains in the primary direction whereas the outer region has grain alignment in the secondary deformation direction. The aspect ratio along the radius from the centre to the outside of the disk initially increases to a maximum at about half of the radius and then decreases towards the disk edge. In this way, equiaxed grains can be expected in a region around half of the radius. Conducting tensile tests on samples prepared in this manner gives the unique possibility to answer the aforementioned questions.

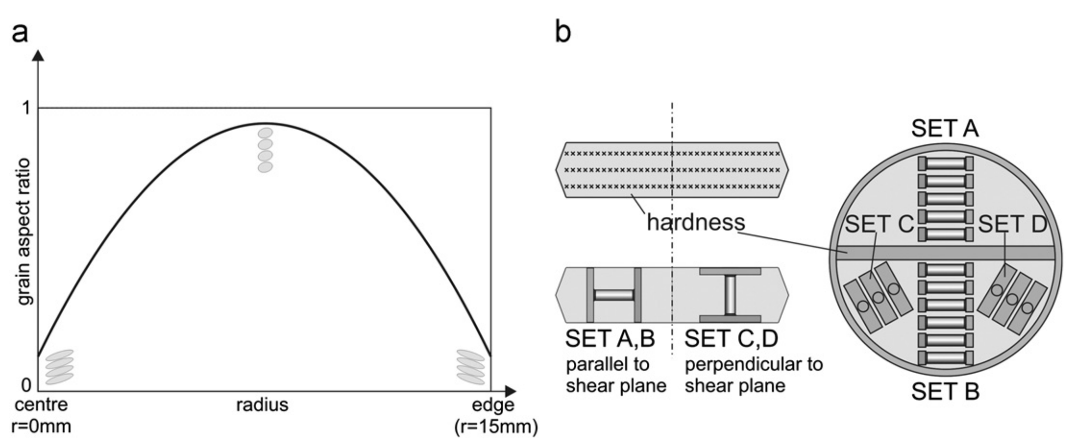

Fig. 1. (a) A schematic sketch of the expected grain aspect ratio distribution of a "back rotated" HPT disc and (b) tensile test sample orientation of the parallel and perpendicular tensile test samples and the cut disc which was used for the hardness measurement.

2. Experimental

A nickel disc with a purity of 99.69 wt% was compressed between two HPT anvils to fill the whole cavity and afterwards annealed at 700 °C for one hour to exclude any possible effects of the pressing. The type and amounts of the impurity elements in weight ppm are C(100), Co(300), Cu(1600), Fe(400), Mn(300), P(200), S(<30) and Si(200). The annealed discs were subsequently deformed for 13 revolutions in a HPT tool with an applied pressure of 3.2 GPa and a rotation speed of 0.067 revolutions/min. This large number of turns leads to a saturation microstructure at radii greater than 2 mm. In the saturation regime further strain leads to no change in the microstructure. Finally on another HPT sample, a secondary deformation of a quarter rotation in the opposite direction was performed to change the grain aspect ratio and the grain orientation over the radius as described before. To ensure the sample was really "sheared backwards", the sample was removed from the tool after the primary deformation step and marked on its top and bottom surfaces with identically positioned lines through the sample centre. The enclosed angle between the markers was measured to be 67° and both lines have not been blurred[1]. Therefore, no slip occurred between the HPT-sample and the anvils during the post deformation.

Hardness measurements along the radius of the HPT disc were carried out on a strip extracted from the centre of the disc, Fig. 1b. A classical metallographic water cooled cutting tool was used for all cutting procedures. For hardness measurements, the cutting face was prepared by mechanical grinding and polishing processes. Vickers hardness measurements were conducted over the radius using a load of 4.9 N and an indent spacing of 0.5 mm along three lines situated in the symmetry plane and ± 2 mm aside from the middle plane, see Fig. 1b. The mean value of the three independent measurements taken from the same radius but different axial positions has been taken as the hardness value.

In addition Fig. 1b shows a schematic of the differently oriented tensile specimens and their denominations (set A–D) extracted from the HPT-disc. All tensile samples could be fabricated from one HPT disc. For the denomination of the different sample orientations the following terms were chosen (see Fig. 1b):

"Parallel to the shear plane" (denoted as A- and B-set in the following) denotes tensile samples where the tensile axis is parallel to the shear plane.

"Perpendicular to the shear plane" (C- and D-set) is used for samples where the sample tension axis is parallel to the HPT rotation axis.

The shaping of the tensile samples from plates extracted from specific radii was performed using a water cooled circular grinding tool, which was especially developed for this purpose. The tensile specimens had a gauge length of 2.7 mm and a diameter of 0.5 mm. The tensile tests were performed using a standard tensile test stage with a load cell of 2 kN and a loading speed of 2.5 μm/s. The displacement was measured from images taken during the tensile test. Details about the grinding tool, the tensile test set-up and the data evaluation are given in [28].

In focus of the data evaluation was the uniform elongation, ε_u, and reduction in area, A, as ductility related measures. The strength of the material was measured in terms of the ultimate tensile strength, σ_{UTS}, and the proportional limit stress ($R_{p0.05}$), representing the stress at a plastic strain of 0.05%, which will in the following be defined as the yield strength, σ_y, in this work.

[1] Due to elastic deformation of the entire HPT setup and play in the transmission of the driving engine the angle is smaller than the adjusted 90°.

The images for the evaluation of the strain of the A-set samples have been taken with half of the resolution compared to the B-, C- and D-set. As a consequence of the unfortunately reduced image resolution, determining the strain resolution, the yield strength, σ_y, could not be measured satisfactorily for this set. So for the A-set, only σ_{UTS} and the reduction in area, A, were determined. To evaluate the systematic experimental errors of σ_{UTS} and σ_y, a Gaussian error calculation was used. The inaccuracy of the diameter measurement was ± 5 μm.

For comparison, tensile tests in both test directions have also been conducted on a monotonically deformed nickel sample where the total amount of strain was also equal to 13 revolutions but without back rotation. Additional compression tests parallel and perpendicular to the shear plane were performed on a monotonically deformed nickel disc. The test cylinder had a diameter of 4.5 mm and height of 5.4 mm. A lubricant was used to reduce the friction between sample and pressure plate during the test. The cross head speed was the same as for the tensile tests.

3. Results

In Fig. 2, the average hardness profile over the radius of the back rotated sample is shown. It is clearly visible that the onset of saturation in hardness is at a radius of 2 mm which would be also the case if the sample was only monotonically deformed. Therefore, all tensile test samples which are taken at a radius larger than 2 mm contain the saturation microstructure with the saturation grain size of 240 nm [26]. Surprisingly, the highest hardness values are seen around the onset of saturation which may be as a result of the one quarter back deformation. Nevertheless, the difference in hardness is not pronounced. Scanning electron microscope (SEM) images recorded with a back scattered electron (BSE) detector in tangential and radial directions at different radii are presented in Fig. 3. No significant change of the microstructure over the radius is found for the images taken in the tangential viewing direction (Fig. 3a, c and e). In the radial viewing direction (Fig. 3b, d and f), the expected variation of the grain aspect ratio is slightly visible. Most of the equiaxed grains are found at a radius of around 11 mm.

Representative tensile curves of both testing directions extracted from the back rotated HPT specimen are shown in Fig. 4 taken at a radius of ~10.5 mm. The tensile test sample in the perpendicular direction reaches a lower ultimate strength and elongation at fracture compared to the sample in the parallel direction. Beside these differences both testing orientations show a similar stress–strain behaviour with significant post-necking elongation.

σ_{UTS} as a function of the radius of the tensile test sample in the back rotated HPT disc is plotted in Fig. 5a for both testing directions. Due to the fact that all tensile samples were extracted from one back-rotated disk, the specimens with an orientation perpendicular to the shear plane could only be taken from a radius area between 9 and 13 mm approximately, compare also Fig. 1b. This is simultanously the area of interest as in this radius region the majority of equiaxed grains are observed. Regarding the samples tested parallel to the shear plane, no clear trend of σ_{UTS} along the radius was found, although along the disk radius all variations of grain aspect ratios should be present induced by the back-rotation. In case of these data points the experimental uncertainties determined by the Gausian error bars are a few times larger than the differences between the single values over the sample radius focusing exclusively on specimens tested parallel to the shear plane. However compared to these results, tensile samples tested perpendicular to the shear plane reach by

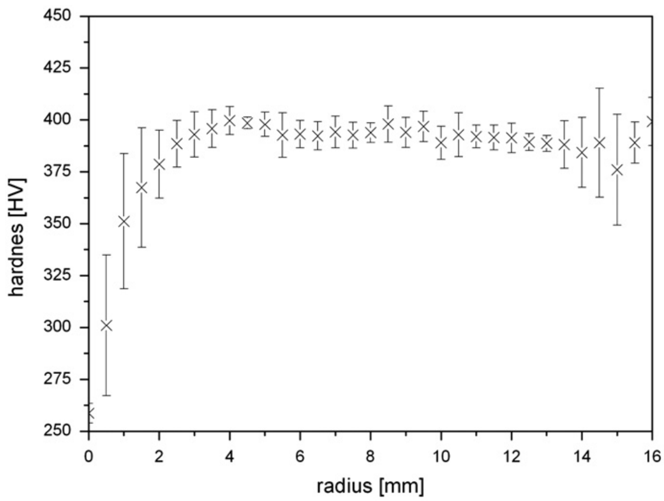

Fig. 2. Hardness as a function of the radius of the deformed and subsequently back rotated sample including error bars. The hardness profile shows an onset of saturation around a radius of 2 mm.

trend always a lower σ_{UTS} values compared to the majority of test samples oriented parallel to the shear plane.

The corresponding uniform elongation values are shown in Fig. 5b. Although the values at a radius around 11 mm are somewhat larger compared to values in the outer and centre region of the disc no statistically significant difference between the two testing directions is visible.

As mentioned, for σ_y the proportional limit stress at a plastic strain of 0.05% ($R_{p0.05}$) was measured and the yield strength values as a function of radius are presented in Fig. 5c. The samples oriented perpendicular to the shear plane reach the lower trend values of yield strength compared to samples tested parallel to the shear plane. In contrast, no clear trend along the entire radius regime can be seen for specimens tested parallel to the shear plane. The experimental uncertainties are too large to determine any clear dependence of yield strength on grain aspect ratio or in other words along the radius. Nevertheless, it should be noted that the accuracy in experimental design and execution with regard to the evaluation of σ_y is fairly remarkable. The average of the experimentally obtained values of the Young's modulus is 216 ± 31.2 GPa and are almost in perfect agreement with a literature value of 200 GPa. Therefore, the effect of bending or tilting of the sample during testing should be very small which could influence the evaluation of σ_y.

Fig. 5d presents the reduction in area values where a clear trend between the two test directions is observed. The samples tested parallel to the shear plane achieve about 10–15% higher values compared to samples tested perpendicular to the shear plane. Furthermore, the samples oriented perpendicular to the shear plane have a much larger scatter compared to the other testing direction. A detailed discussion about these phenomena is provided in the next section.

For comparison, tensile tests on monotonically deformed HPT samples have been performed as well. At least three tests have been carried out in each testing direction and a comparison of the collected values is given in Table 1. The σ_{UTS} value of the monotonically deformed nickel tested parallel to the shear plane is around 80 MPa higher compared to the perpendicular test direction. The corresponding uniform elongation values are very similar for both test directions. The reduction in area values parallel to the shear plane are about 12% higher compared to samples tested perpendicular to the shear plane.

Lastly, compression experiments were performed to check if the load path (compression or tension) has a signifcant influence on the deformation behaviour. The stress and strain curves are shown in Fig. 6 where two samples of each test orientation have been tested. The samples loaded parallel to the shear plane (dashed line) reach about 80 MPa higher values compared to the sample tested perpendicular to the shear plane (solid line). This indicates that this small difference in the yield stress is independent of tension or compression loading, compare also Table 1.

4. Discussion

4.1. The grain shape influence on strength

The presented hardness curve in Fig. 2 does not show any pronounced change as a function of the radius. Therefore, one can conclude that the hardness is mainly governed by the average grain size and is independent of the grain shape or the grain aspect ratio. In contrast, the tensile tests revealed different σ_{UTS} levels depending on the test direction. The difference is not very large but visible. For both, the monotonically and the back rotated samples, tensile tests parallel to the shear plane reach about 80 MPa higher values compared to tests performed perpendicular to the shear plane. This trend is also observed for inclusion free micro tensile samples which have been tested in a scanning electron microscope [15]. Before considering the details of the present experiments a few different reasons could be expected to be responsible for this orientation dependency of the yield stress

- the grain shape (aspect ratio),
- a strain path effect, similar to a Bauschinger effect and
- crystallographic texture effect.

As in general known from rolled materials showing elongated grains, a large influence of the grain aspect ratio on σ_{UTS} could be expected, however was not observed here. Otherwise, samples with similar grain aspect ratios should achieve similar σ_{UTS} values independent of their test direction. According to the back scattered electron micrographs of Fig. 3, this region with similar grain aspect ratios should be at a radius of 10–12 mm. However, this assumption is not confirmed by the values of Fig. 5a in this radius regime. There a clear trend comparing the two different specimen orientation is visible.

Another counter argument for the grain aspect influence on σ_{UTS} is given by the compression experiments. Samples oriented parallel to the shear plane have their long grain axis parallel to the compression axis. In such samples the grains should become more equiaxed during the experiment. On the other hand, samples oriented perpendicular to the shear plane have their long grain axis perpendicular to the compression axis. In such samples the grains should become even stronger elongated during the experiment. Assuming that the grain aspect ratio or grain shape governs the strength, a change of the grain aspect ratio during deformation should lead to softening or hardening of the material depending on the specimen and grain orientation. In the case of a sample oriented parallel to the shear plane the strength should increase for tensile testing (grains become even thinner during testing) but decrease in a compression test (grains become thicker during testing). The exact opposite behaviour should be observed for samples tested perpendicular to the shear plane. The strength should decrease for tensile testing but increase in a compression test. In Fig. 6 this expected behaviour is clearly not observed and both tests performed for one testing direction show the same trend as found for tensile tests: specimens oriented perpendicular to the shear plane show a lower

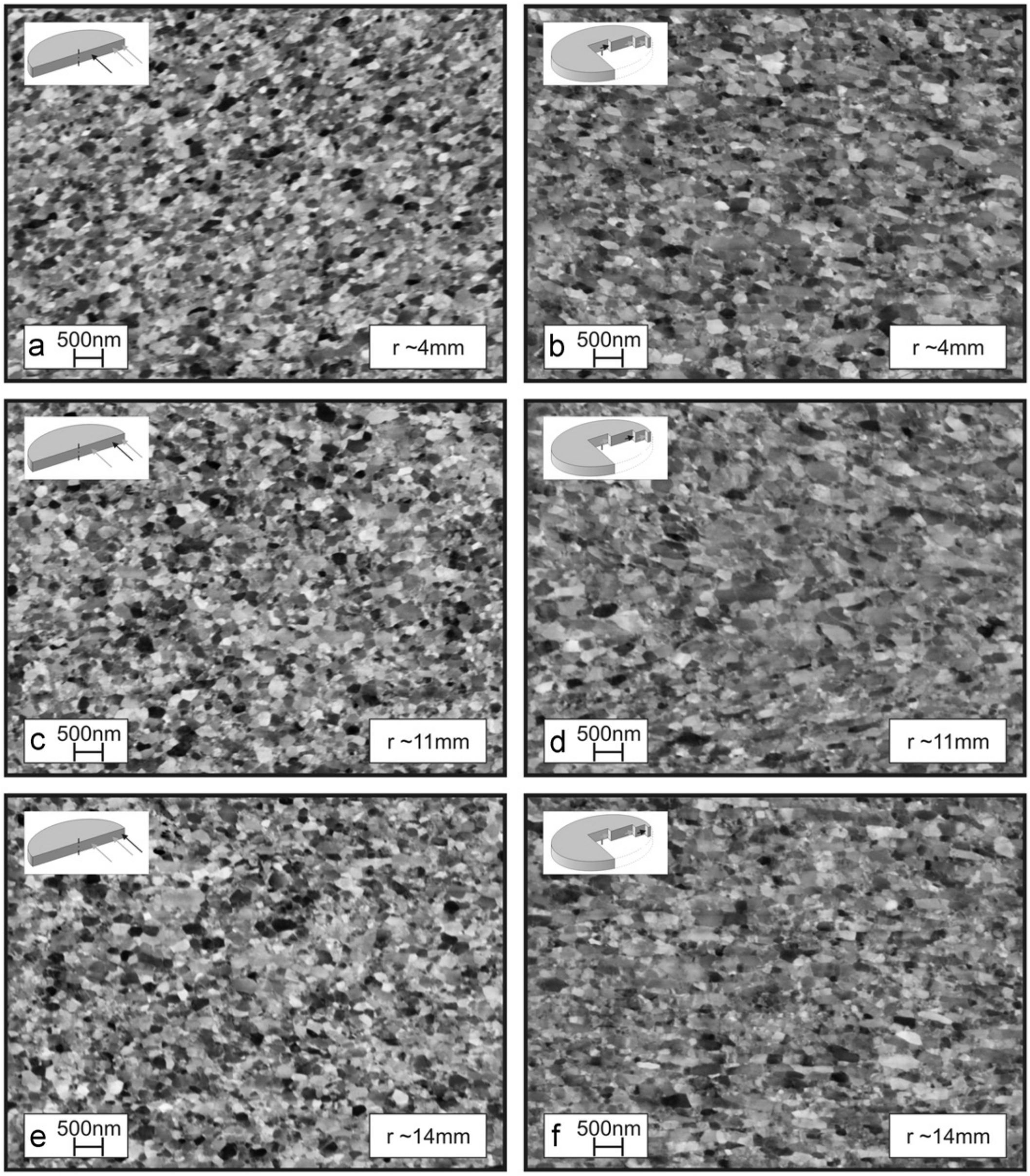

Fig. 3. Back scattered electron detector images taken at different radii, in radial and tangential directions of the back deformed HPT disc.

flow stress than the ones oriented parallel to the shear plane. Even the differences between the two test directions of about 100 MPa is very similar to that seen in the tension experiment. The fact that the compression experiments reach higher stress values as the tensile test is mainly caused by a constraint induced by the compression anvil. To conclude, a significant influence of the grain aspect ratio on σ_y and σ_{UTS} is not observed.

We assume that the observed difference in σ_y and σ_{UTS} seems to be caused by the shear texture which evolves during the HPT deformation [22,23] since an influence of the grain aspect ratio could be ruled out. The back rotation causes a change in the grain aspect ratio depending on the applied shear strain. However, this back rotation should not change the shear texture significantly. This is supported by the electron back scatter diffraction analyses in [20,21].

4.2. The grain shape influence on ductility

The reduction in area clearly depends on the loading direction in both samples (monotonically deformed HPT and back rotated HPT sample). The fracture surface in Fig. 7a was taken from a back-rotated sample with a parallel to the shear plane orientation. It reveals a typical ductile cup and cone fracture surface with some large pores visible over the entire surface. The white rectangle in Fig. 7a marks the enlarged area shown in Fig. 7b. In one of these large pores there is still an inclusion visible which may serve as origin of the pore formation as already reported [15,26]. Between these large pores a dimpled wavy fracture structure consisting of small pores is visible. Such fracture surfaces were found for both tensile test directions and independent from the extraction radius of the sample.

According to these fracture surface investigations it is evident that the mechanism controlling and lastly restricting the reduction in area is the growth and coalescence of small pores between the few large pores.

Focusing first on the results of the monotonically deformed samples where in the two load directions different grain shapes with respect to the loading directions are present, we assume that the grain aspect—ratio itself causes the slight difference in the reduction in area.

Mode I fracture toughness experiments on HPT processed nickel [29] and also on HPT deformed iron [30] have shown that the fracture resistance is strongly controlled by the microstructure alignment. The experiments indicate a weak crack path parallel to the shear plane along the elongated grains with low fracture resistance. Much higher fracture resistance was found for a crack propagating perpendicular to the elongated grains where even crack bifurcation into the direction of the microstructural alignment into the shear plane was found. These fracture mechanical analyses can be directly applied to explain the observed anisotropy of the reduction in area for nickel where a ductile crack propagation mechanism prevails. This mechanism can be characterized by void initiation at grain boundaries and coalescence of these small pores, as shown in Fig. 7b, with macropores initiated at non-metallic inclusions.

In case of a tensile sample with the load-line parallel to shear plane and so also parallel to the aligned grains, see Fig. 8a, a propagating crack oriented perpendicular to the load line has to

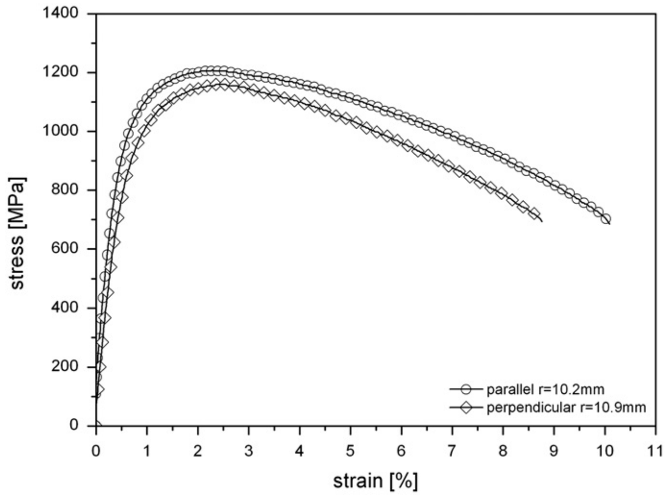

Fig. 4. Two representative stress–strain curves of a parallel and a perpendicular tested tensile sample taken at a radius of around 10.5 mm from the back rotated sample.

Table 1
Average tensile test results of a monotonically deformed HPT disc tested parallel and perpendicular to the HPT shear plane. All samples included have a saturation microstructure.

Test direction according to HPT shear plane	Ultimate tensile strength (MPa)	Uniform elongation (%)	Reduction in area(%)
Parallel to shear plane	1142 ± 29.4	1.56 ± 0.07	67 ± 2.3
Perpendicular to shear plane	1063 ± 23.6	1.43 ± 0.10	51 ± 8.9

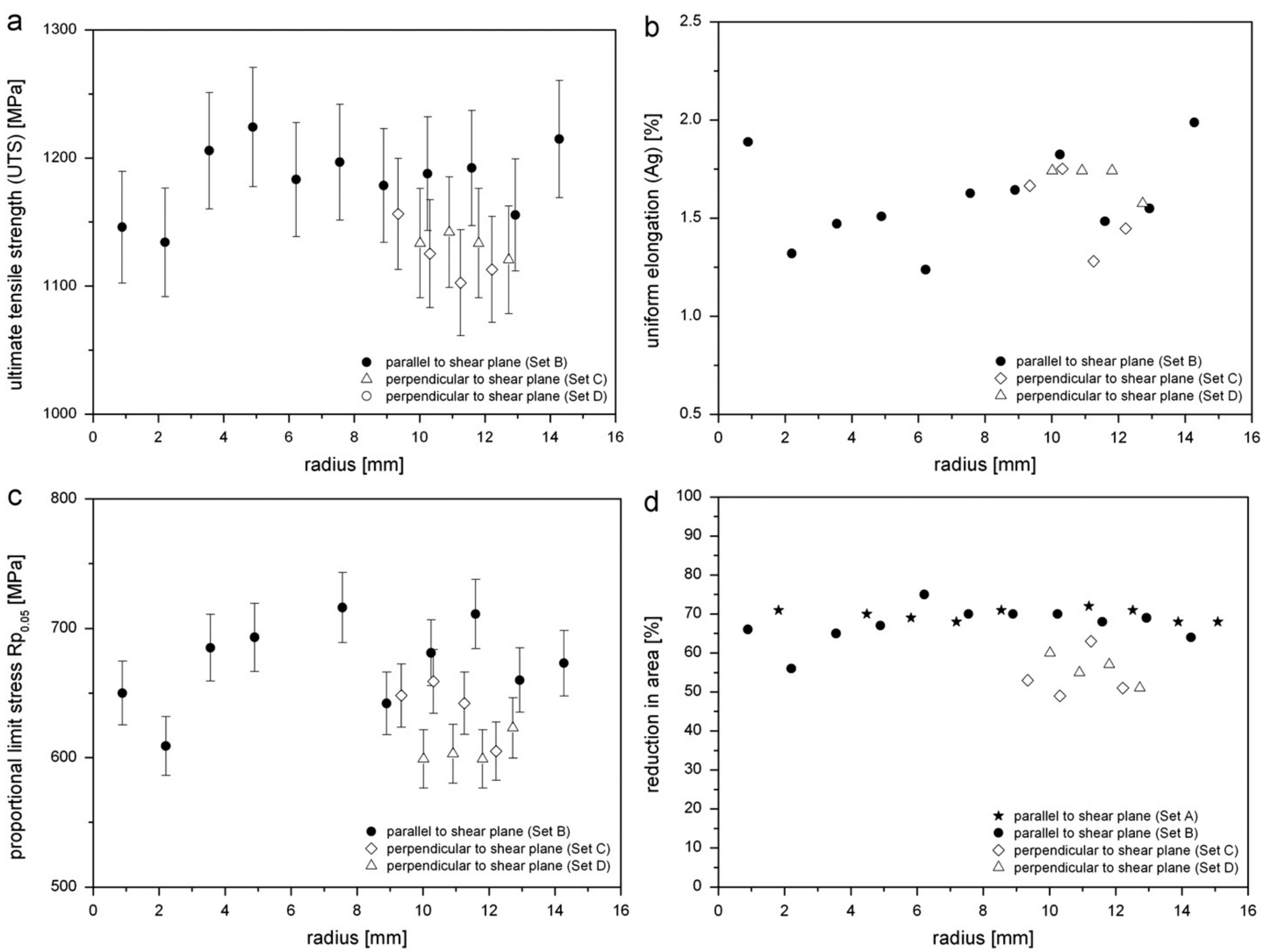

Fig. 5. Tensile test results of the different test directions versus their radii from the HPT disc: (a) ultimate tensile strength including the Gaussian error bar, (b) uniform elongation, (c) yield strength expressed with the proportional limit stress ($R_{p0.05}$) including the Gaussian error bar and (d) reduction in area.

circumvent the elongated grains or bifurcate locally. Only with this crack path can the crack propagate in the direction of maximum driving force which is perpendicular to the loading direction. The circumvention of grains or bifurcation leads to a retarded crack propagation and pore coalescence and finally to a higher reduction in area before total failing of the sample. In the other testing direction where the load-line is perpendicular to long axis of the grains, see Fig. 8b, the crack propagates parallel to the aligned microstructure serving as a weak crack path. This weaker crack path leads to a smaller reduction in area as the crack propagation is facilitated and accelerated.

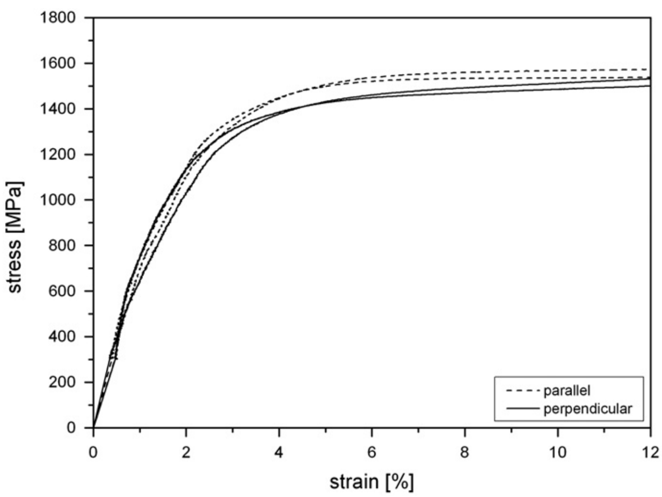

Fig. 6. Compression test curves for the samples loaded parallel to the shear plane (dashed line) and the sample tested perpendicular to the shear plane (solid line).

Focusing on the tensile tests of the back-deformed samples a slight contradiction to the introduced fracture mechanical based argumentation arises: this is because a similar difference and trend in the reduction in area of samples both parallel and perpendicular to the shear plane between 8 and 12 mm was found, see Fig. 5d, where equiaxed grains should be present.

This can however be explained by inhomogeneous back deformation: it has been observed that the change of the grain aspect ratio and the grain orientation will not vary in a continuous manner as the radius is increased. Therefore, some regions will have the new orientation, where the back deformation was more concentrated. Other areas are transforming and some regions still have the initial grain shape orientation, where only small back deformation occured. The description of a "mixed" microstructure is in good agreement to the electron back scattered images in Fig. 3 and fracture toughness experiments conducted on back rotated ultrafine-grained iron [30]. In this report it was show that already the occurrence of some equiaxed grains in the plastic zone of a crack can change the fracture behaviour in terms of the crack path and fracture toughness decisively. Nevertheless, it cannot be excluded that beside the grain aspect ratio also the HPT shear texture might also influence the slightly observed orientation dependency of the reduction in area.

The uniform elongation values show no significant difference between the two test orientations in the case of the monotonically deformed HPT sample. A similar trend is observed for the back rotated samples as shown in Fig. 5b. Both test orientations reach similar values at the same radius. In the outer region and near the disc centre these values are in good agreement to the monotonically deformed ones. The small increase of the values visible at a radius of 8–12 mm seems, to the opinion of the authors, not to have been caused by the grain aspect ratio but might be related to the "mixed" microstructure caused by the slight back rotation.

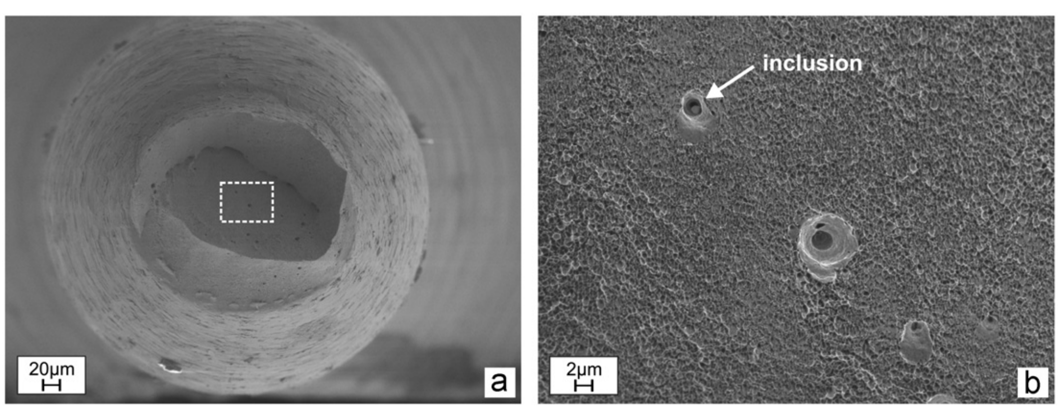

Fig. 7. (a) Scanning electron microscope image of the fracture surface in overview and (b) detailed image of the large pores and the wavy fracture surface in between them. The position of the detailed image is marked by a white rectangle in Fig. a.

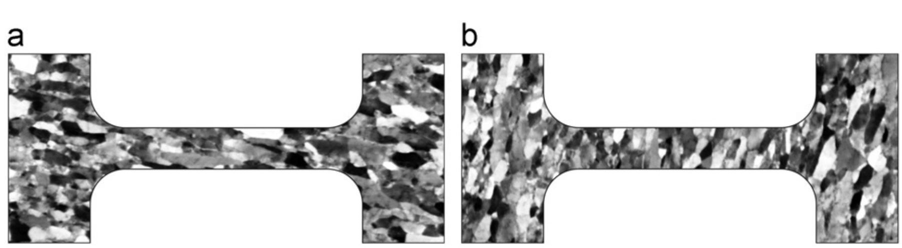

Fig. 8. Schematic of the microstructural orientation within the test samples (not to scale) (a) tensile specimen oriented parallel and (b) perpendicular to the shear plane after reaching the uniform elongation.

5. Summary

The outcome of this study can be summarized as follows:

(i) The ultimate tensile strength shows a clear orientation dependency, where samples tested parallel to the HPT shear plane reaches higher values compared to samples tested perpendicular to the shear plane. These findings are similar for compression and tension tests and an influence of the grain aspect ratio was not observed for either loading conditions. Therefore, the shear texture seems to cause the strength differences of about 100 MPa.
(ii) The reduction in area is influenced by the grain aspect ratio, where samples tested parallel to the longest grain axis reach higher values than samples tested parallel to the shortest grain axis. A further orientation dependency is not observed.
(iii) The uniform elongation is neither affected by the grain aspect ratio nor the test direction.

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