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ULTRAFINE GRAINED MATERIALS

Microstructure and mechanical properties of UFG medium carbon steel processed by HPT at increased temperature

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Abstract Using the high pressure torsion (HPT) deformation method the medium carbon steel (AISI 1045) was the experimental material used to conduct the deformation process. The torsion deformation experiment was performed at increased temperature of 400 °C. The influence of deformation processing parameters, resolved shear strain γ (number of turns $N = 1$–6) and applied pressure p (constant pressure of 7 GPa), was evaluated by microstructure analysis and mechanical properties. The strength behaviour was assessed by microhardness measurements across the disc to detect the positional hardening, by tensile tests and in situ measured torque. In situ measurement of torque during deformation allows characterizing the changes in mechanical properties due to the large shear deformation developed across the disc. To obtain absolute values of strength the ultimate tensile strength was measured in radial direction with respect to the deformed sample. From each deformed disc two sub-sized tensile test specimens with gauge length of 2.5 mm were machined. The tensile strength in samples increased markedly with the number of turns. The hardness measured at disc edge gradually increases as straining increases until it saturates after 2–3 turns. However, the hardness values at edge were different from those measured in disc centre and for applied straining no saturation was reached across the disc. The SEM and TEM investigations were carried out to analyze the fine microstructure evolution regarding the strain introduced. To follow the difference in strain distribution across the deformed disc the microstructure analysis was performed at edge and central site of the disc in order to evaluate the effect of the strain distribution. TEM investigation confirmed the increasing misorientation even in very small grains, the fragmentation and dissolution of the cementite lamellae, (diffuse cementite/ferrite boundaries), the alignment of the fragments to the shear plane with increasing deformation. Indistinct deformation of ferrite and preserved cementite lamellae morphology were found at the centre of the disc.

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Introduction

During the last decade, bulk nanostructered materials produced by severe plastic deformation (SPD) have been investigated intensively. The production of fine grained materials by SPD, led to a large number of investigations focusing on the substructure development and related mechanical properties. It is well known that SPD of metallic materials, involving processes such as equal angular pressing (ECAP), accumulative roll bonding (ARB) and high pressure torsion (HPT) is capable of producing ultrafine grained (UFG) materials with submicrometer or nanometre grain size [1, 2]. The wide range of results available for various materials deformed by HPT confirmed a saturation in microstructural refinement and the strength and hardness as strain increases [3, 4]. The results point out that there are differences in the development of the microstructure between the pure and multiphase steels [5, 6].

In this work two phase medium carbon steel has been deformed at increased temperature of 400 °C by high pressure torsion to different strains (executing different numbers of turns) at hydrostatic pressure of 7 GPa. The underlying relationship between microstructure and mechanical properties was analyzed with respect to the local axial position of analyses on the deformed disc.

Material and experimental procedure

A commercial medium carbon steel (Fe–0.45%C, 0.42%Mn, 0.23%Si 0.18%Cr, 0.043%Al in wt%) was supplied in rod form with a diameter of 20 mm. Before HPT deformation the steel was annealed for 1.5 h at 850 °C followed by air cooling in order to obtain uniform normalized structure. The mixture of quite coarse ferrite–pearlite structure was obtained as shown in SEM micrograph in Fig. 1. Following the annealing treatment, the size of pearlite colonies was $\sim$30 μm and the ferrite grains were 15 μm. Discs of 8 mm in diameter and $\sim$0.95 mm in thickness were cut off from the rod and were deformed by torsion up to 6 turns at temperature of 400 °C and at a pressure of 7 GPa. The equivalent von Mises strain ε_{eq} as a function of the number of turns n was calculated according to the relation: $n = \varepsilon_{eq} = 2\pi nr/t\sqrt{3}$. The size of effective strain conducting $N = 1$, 2, 4 and 6 turns was $\varepsilon_{eq} \sim 15$, 30, 60 and 90. The changes in mechanical properties in relation to straining (number of turns) were determined by microhardness measurement across the deformed disc with a load of 300 g using Vickers microhardness tester, by static tensile test using sub-size tensile pieces cut off at discs' periphery (in radial direction) and by in situ measurement of the torque. The reliable and accurate possibility to measure the torque during deformation allowed quick determination of changes in mechanical strength without applying other method afterwards. Transmission electron microscopy (TEM) was used to evaluate the microstructural evolution with respect to site on disc and the number of turns. TEM micrographs were obtained by using JEOL JEM 2000FX operating at 200 kV. The purpose of the elected conditions was to evaluate the effect of difference in strain magnitude across the disc (at peripheral and axial position of disc) on ultrafine grain microstructure development across disc diameter.

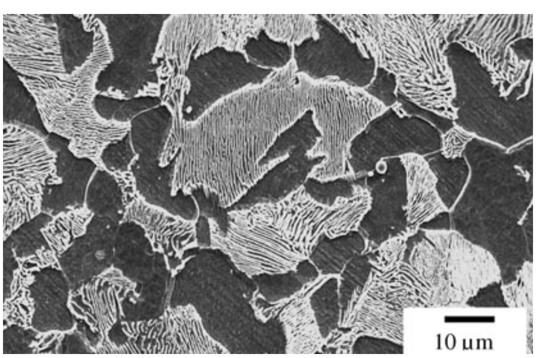

Fig. 1 SEM micrograph of initial ferrite–pearlite structure of AISI 1045 steel

Results and discussion

Investigated material comprised of the coarse ferrite–pearlitic steel AISI 1045, as shown in Fig. 1. Using HPT method for refining coarse steel structure to nanostructure size needs the application of large strain, usually with an equivalent strain ε_{eq} more than 10. Electron microscopy analysis of thin foils prepared from deformed discs exposed to different strains revealed formation of various structural characteristics in relation to strain applied and selected localization on the disc.

Microstructure

In order to compare the size of torsion straining at different position on the deformed disc, Fig. 2 shows the microstructure observed in steel after exposure to the first turn at temperature of 400 °C. Finishing the first turn ($\varepsilon_{eq} \sim 15$) the heterogeneity in structure development was still evident across the disc. At disc periphery, the alignment of smeared (partially dissolved) lamellae in pearlite next to the area of banded subgrain-like structure is documented in Fig. 2a, b.

In the centre of the disc the lamellae remained, but some dissolution of the cementite lamellae can be observed with smeared-like features, as presented in Fig. 2c. Conducting 4 and 6 turns ($\varepsilon_{eq} \sim 60$ and $\sim$90), the dual phase structure was significantly modified with respect to analysis site as presented in Fig. 3. Regardless of the strain size, successfully refined structure with small grains (grain size about of $\sim$200 nm) having random high angle orientation was found in disc edge area as presented in Fig. 3a. Selected area diffraction method (SAED) confirmed high angle disorientation of small grains. In the centre of the disc the structure has moderately deformed features. In ferrite grains tangled dislocations are present, and pearlite colonies were only slightly deformed and/or locally fragmented, Fig. 3b, c. From the TEM micrographs cementite lamellae thinning in central disc area is evident in structure. This fact is attributed to partial dissolution of cementite

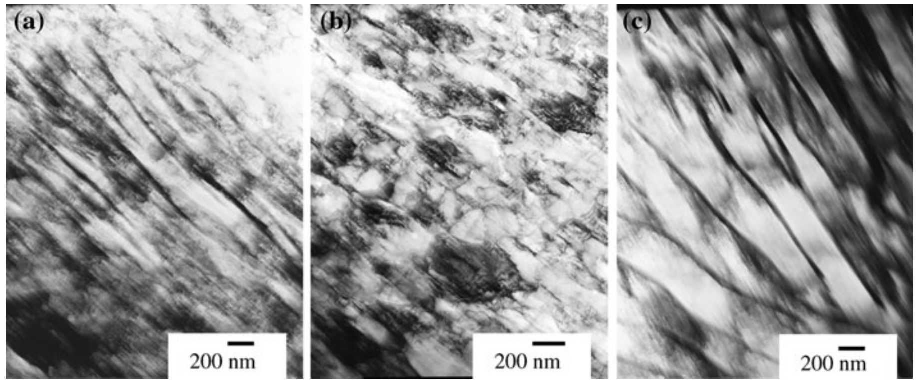

Fig. 2 TEM micrographs of microstructures observed at disc periphery after conducting the first turn (**a**, **b**), and corresponding to the centre of disc (**c**) experienced the $\varepsilon_{eq} \sim 15$

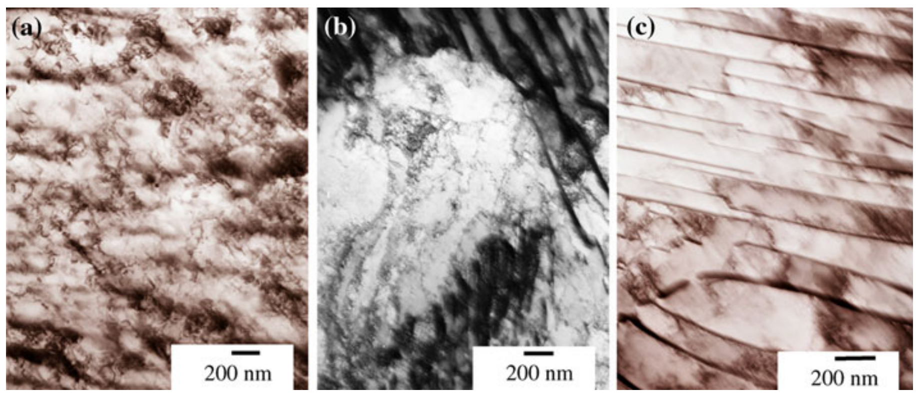

Fig. 3 TEM micrographs of deformed microstructures conducting 4 turns found at periphery and at the centre of disc experienced the $\varepsilon_{eq} \sim 60$: **a** edge; **b**, **c** centre

during shearing and results in decreasing of the lamellae thickness.

Mechanical properties

In order to characterize the changes in mechanical properties due to the large shear deformation, various methods were applied. Local Vickers microhardness and tensile properties were evaluated at room temperature. The torque, which was measured, contains the torque necessary to deform the sample as well as solely a contribution from the region of the burr [5].

Microhardness development after different deformation exposure was the quickest and the most easily available method to estimate the mechanical strength and is often applied in the literature [6, 7]. In present work, this experimental method was mainly used to determine the change of the strength across the disc (at disc periphery and in the centre) after application of different ε_{eq} and compared with hardness of the initial steel. The measured Vickers hardness (HV30) records for different numbers of turns are stated in Table 1. The results show that some effect of softening was detected in disc after the fourth turn regardless of the position, which can result from ultrafine grained structure dominancy and some dynamic recovery of dislocation structure in ferrite grains at disc centre. To obtain mechanical strength values, the specimens for

Table 1 HV hardness as function of *N* and indentation position

N turns	Edge 1	Centre	Edge 2
1	530	364	509
2	524	342	528
4	467	308	426
6	536	517	491
In[a]	185	192	182

[a] Hardness of initial state of steel after annealing

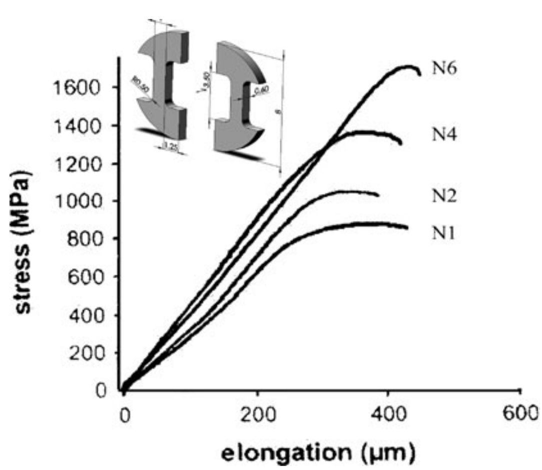

Fig. 4 Tensile test records of steel after different HPT straining ε_{eq}

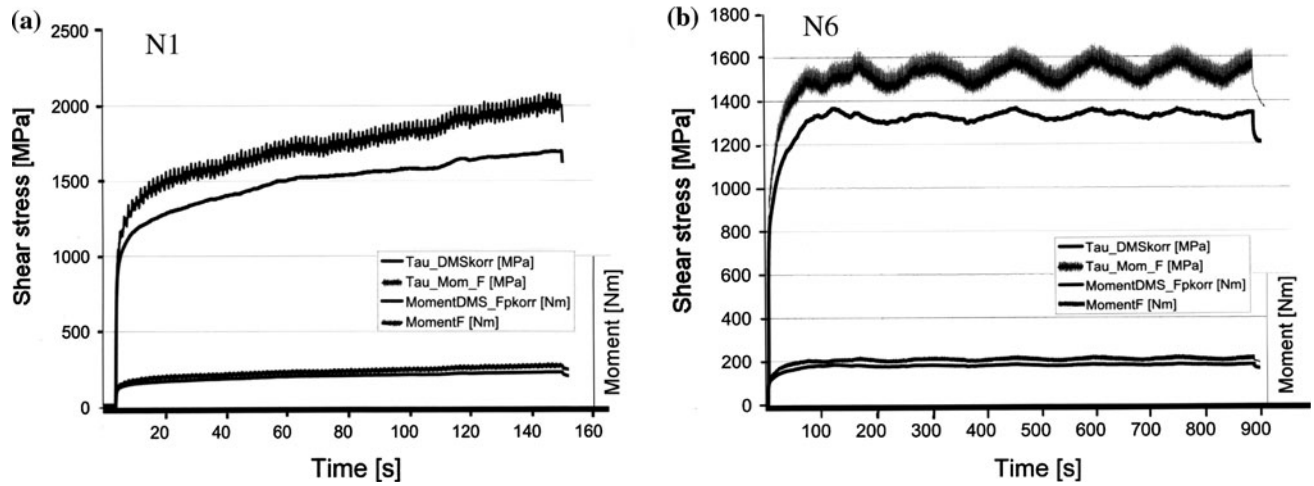

Fig. 5 Records of the shear stress (τ) and in situ torque (Nm) for **a** N1 and **b** N6 turns

tensile tests were machined out of HPT samples [8]. Two specimens were cut off from each of the deformed discs (see Fig. 4). Tensile behaviour was evaluated at room temperature and results in the form of stress and elongation are documented in Fig. 4. The ultimate strength increased with the applied shear strain and maximum value obtained was higher than 1700 MPa. The development of the ultimate tensile strength is similar to the in situ measured shear stress and torque. The records of shear stress and torque measured in situ for turn's $N = 1$ and $N = 6$ are presented in Fig. 5. The possible explanation could be that the onset of steady state coincidences with saturation in the decrease of the grain structure size and tensile tests values. The strain necessary to reach this state deformation depends on the material's structural state.

Discussion

High pressure torsion method at increased temperature of 400 °C was applied to refine microstructure in AISI 1045 steel. There are marked differences in the development of the microstructure in relation to strain applied as regards the site of analysis. Grain refinement and cementite lamellae dissolution was observed after the first turn ($\varepsilon_{eq} \sim 15$) at disc periphery. In disc centre, the deformed structure of ferrite with dislocation tangles had moderately deformed features of coarse cementite lamellae in pearlite grains. At higher strain cementite lamellae are directionally aligned, thinner and fragmented in relation to the strain introduced. As the effective strain increased, probable set equilibrium between the cementite phase fragmentation and new grains formation in UFG structure, and led to saturation of the refinement process. With regards to tensile properties, the yield strength and ultimate strength increased with increasing ε_{eq}. A small decrease in the hardness across the disc was measured after execution of 4 turns, which may be related to formation of ultrafine grain structure in the disc but more probably only due to dislocation structure recovery in disc centre and preservation of initial structure.

Summary

Severe plastic deformation of medium carbon steel AISI 1045 by high pressure torsion process produced ultrafine grained structure at periphery of the disc already after finishing the first turn. Structure heterogeneity was evident across the disc disregarding the number of turns increased to $N = 6$ and effective strain ε_{eq} reached value of ~ 90. The dissolution of cementite lamellae contributed to their thinning. The fragmentation and alignment of cementite into the shear direction was accompanied by an increase of hardness. Tensile tests records confirmed that ultimate strength increased with increasing shear strain and a maximum value of ~ 1700 MPa was reached after $N = 6$ turns. The torque measurements during the deformation confirmed strong hardening at the beginning, whereas saturation behaviour for higher strain was observed, as number of turns increased.

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