Fatigue behavior of ultrafine grained medium carbon steel with different carbide morphologies processed by high pressure torsion

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\section*{Abstract}

The increased attention that ultrafine grained (UFG) materials have received over the last decade has been provoked, not least, by their high strength in combination with remarkable ductility. The main focus of our investigation was the evaluation of the effect of different carbide morphologies in the initial microstructure on the fatigue behavior after high pressure torsion (HPT) treatment of SAE 1045 steel. In our case HPT increased the hardness by a factor of 1.75 - 3.2 compared to the initial states. The achieved hardness maximum was 726 HV. The amount of increase depended on the initial carbide morphology. By stress controlled cyclic four point bending tests with a load ratio of 0.1 endurance limits were determined for the initial and HPT states. The endurance limit increased linearly with hardness until 500 HV and independently of the carbide morphology. All fracture surfaces were investigated by SEM after the fatigue tests. They revealed pretty flat fatigue fracture surfaces with crack initiation at the surface or rather at non-metallic inclusions for the UFG states. Morphology and crack initiation mechanisms were changed by severe plastic deformation compared with the coarse grained initial state. Residual fracture surfaces with a spheroidal initial microstructure showed well-defined dimple structures also after HPT at high fatigue limits and high hardness values. In contrast, the specimens with initial tempered microstructure showed rather brittle and rough residual fracture surfaces.

\section*{Introduction}

Ultrafine grained materials have been shifted more and more in focus since the last decades. They are very popular because of their combination of high strength and ductility [1]. One option to produce an ultrafine grained microstructure is severe plastic deformation (SPD). This is a top down method which begins usually with an initial coarse grained (CG) microstructure and leads to a break up of former grain boundaries and a production of very small new grains with high angle grain boundaries. Essential for SPD is the combination of a high hydrostatic pressure, to avoid crack initiation, and an enormous shear strain. Famous treatments using severe plastic deformation are Equal Channel Angular Pressing (ECAP), Accumulative Roll Bonding (ARB) and High Pressure Torsion (HPT). Mostly, materials with a face centered cubic (fcc) or hexagonal closed packed (hcp) crystal system were regarded in literature concerning SPD due to their high capability of deformation during the HPT treatment, for instance [2]. There are only a few investigations which deal with medium carbon steels and only a very small part of these considers also the fatigue properties. In this paper we present fatigue properties of an ultrafine grained medium carbon steel with respect to two different carbide morphologies. We want to draw a relationship between the hardness on the one side, which is affected by grain refining during HPT, and the endurance limits on the other side, which were determined during bending fatigue tests. Fractographic investigations were made to interpret and analyze the results.
Experimental Procedure

During HPT a cylindrical sample is placed between two anvils under a high hydrostatic pressure (see Figure 1a). After applying this pressure, in our case 6 GPa, 6 respective 10 rotations were done at a temperature of 380 °C to refine the microstructure. Four rectangular fatigue samples with 4 mm x 1 mm (w) x 0.6 mm (h) were cut equidistantly to the center of every 10 mm x 0.6 mm cylindrical disk after HPT. The fatigue samples were grinded into the final shape and polished to 1 µm grid size.

The tests were performed on a BOSE ELECTROFORCE 3230 electrodynamic testing machine at a frequency of 40 Hz under stress control. They were conducted with a load ratio of R = 0.1 and at room temperature. The dimensions are given in Figure 1 b). To determine the endurance limits of the respective state the staircase method [3] was used with different step sizes, depending of the expectable fatigue limit from hardness measurements.

Hardness measurements were done with an ASMEC UNAT 2 Nanoindenter and a proof force of 150 mN on a surface polished with colloidal Si₂O. A PHILLIPS SL40 Scanning electron microscope (SEM) was used for fractographic investigations at a voltage of 20 kV.

Materials and Microstructures

As base material for the SPD via HPT two initial states with different carbide morphologies of the medium carbon steel SAE 1045 (Fe balance, 0.46%C, 0.64%Mn, 0.17%Si, 0.011%P, 0.009%S) were used. The two carbide morphologies were reached through softening (spheroidizing) annealing and tempering.

The micrographs of the state after spheroidizing annealing for 40 hours at 680 °C and the respective HPT states after 6 and 10 rotations are shown in Figure 2 a) - c). The softening annealed state offers spheroidal carbides in a homogeneous coarse grained microstructure with well-defined grain boundaries (Figure 2 a)). After 6 rotations the grain boundaries have broken up and some new grain boundaries are visible in Figure 2 b). In Figure 2 c) the homogeneous microstructure with many new grain boundaries but still spheroidal carbides is visible. In Figure 2 d) – f) the same sequence of initial and HPT states for the tempered microstructure is given. Figure 2 d) shows the needle like
microstructure after austenitization at 850 °C, quenching to room temperature and tempering for 1 hour at 450 °C as initial state for HPT. Carbides are not visible in this SEM magnification. TEM investigations, which are not shown in this paper, revealed fine dispersed carbides between the needles. After 6 HPT rotations in Figure 2 e) stretched and elongated grains are visible; they are oriented in shear strain direction. Figure 2 f) shows the micrograph of the initial tempered state after 10 HPT rotations. A very homogeneous ultrafine grained microstructure can be observed with no visible needles anymore. The hardness values of every state are indicated in the top right corner. It is obvious that the hardness increases with the number of rotations for both carbide morphologies. The level of hardness is higher in the tempered than in the spheroidizing annealed states. Highest hardness values of 500 HV in the spheroidizing annealed and 726 HV in the tempered state were reached after 10 rotations. These correspond to increase factors of up to 3.2 and 1.75 respectively as compared to the respective CG states.

Experimental Results

4-point-bending fatigue tests were carried out with both carbide morphologies in the initial and the HPT states. With load ratio R=0.1 the virtual initial edge stress amplitude was in both cases plotted against the cycles to fracture. The arrows in the squares indicate runouts after $10^7$ cycles. Figure 3 a) reveals the values for the spheroidizing annealed state in the CG and UFG condition. Figure 3 b) illustrates the S-N-curves of the tempered state before and after HPT with 6 and 10 rotations are illustrated. In the initial state a fatigue limit of 316 MPa was determined in an analysis with the staircase method. Also fractures at cycles of more than $10^6$ were observed at 325 MPa. At 300 MPa only one fracture occurred. In contrast, the UFG state of this carbide morphology with a hardness of 289 HV exhibited only cracks until approximately 400,000 cycles. The endurance limit of 493 MPa was significantly larger than in the initial state. Crack initiation was mainly at nonmetallic inclusions as indicated in the diagram. After 10 HPT rotations resulting in a hardness of 500 HV the fatigue limit further increased to a value of 837 MPa. One specimen cracked at 800 000 cycles, all other either cracked within the first 80,000 cycles or run out. Under 800 MPa no fatigue cracks were observed anymore. Crack initiation took place at surface of nonmetallic inclusions.

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crack behavior seem to be completely different. In contrast to the initial state and the HPT state after 6 rotations, where no cracks after 200,000 cycles were observed, states with high hardness suffered cracks until 6,000,000 cycles. The very high hardness state also revealed very early cracks, which pulled to fatigue limit down to a value of 850 MPa, and thus lower as expected with a hardness of 726 HV. Again, no typical crack initiation site could be located.

**Fractographic Investigations**

After the bending fatigue tests all fracture surfaces of the broken specimen were investigated via SEM. In the bending specimens a stress gradient over the thickness leads to a tension side, a neutral axis and a compression side. The tension side, where fatigue cracks always start in this kind of material, is in every SEM investigation at the lower part of the picture.

Figure 4 a) shows the fracture surface of UFG SAE 1045 steel after 10 HPT rotations of the spheroidization annealed state. There is a large fatigue fracture surface, compared to these which are typically found in conventional high strength materials, visible on the lower site. The residual fracture surface reveals in Figure 4b) well-defined dimples which indicate a ductile fracture behavior. The fatigue fracture surface shown in Figure 4 c) is very homogeneous and appears flat. The crack initiation was at a nonmetallic inclusion. In the transition area between fatigue and residual fracture surface some small and cave-like shear bands are visible. This was reported in literature [4] regarding on nanocrystalline Palladium. These shear bands in UFG materials can partly replace the usual deformation mechanisms like dislocation motion or twinning, which take place in the CG-regime.

Figure 5 illustrates the fracture surface of one tempered sample after 10 HPT rotations and a fatigue load of 900 MPa. Low magnification here also reveals shear bands in the fracture surface.
The cave-like characteristics appear over the whole residual fracture surface and with a length of partly up to 200µm they seem to be larger than those in the spheroidizing annealed state, which are only 40 µm long. No dimples were found on the residual fracture surface, only a rather rough surface with sharp edges, see Figure 5 b). The fatigue fracture surface is smaller than in the spheroidizing annealed state although the sample was loaded at nearly the same stress level. We also observe crack initiation at a nonmetallic inclusion. The fracture appearance is homogenous with a flat fatigue fracture surface.

The fracture surface of a tempered specimen after HPT which offered an early crack after loading at 900 MPa in the S-N curve is shown in Figure 6a). Illustrated is a very cliffy fracture surface with a high topology in the residual and also in the fatigue fracture surface. The fatigue crack seemed to grow through and jump between different levels, which is a hint that there must be special features inside the specimen, which promote this behavior. Usually fatigue cracks grow mostly in one clear direction, as visible in Figure 4 and Figure 5.

Figure 6: a) Fracture surface of a specimen after fatigue load at 900 MPa b) fatigue fracture surface c) pre crack after HPT and before fatigue loading in another specimen

The whole fracture surface offers a different characteristic concerning shear bands than for the fatigue samples with higher endurance limit. An examination of alternate samples with early cracks did not deliver a clear classification for the characteristic and existence of shear bands.

The HPT treatment itself can be a reason for the high topology of the fatigue fracture surface and the low fatigue endurance limit of this part of the tempered samples. Figure 6 c) exhibits some pre cracks prior fatigue loading in samples of the initial tempered condition, which were discovered after HPT treatment with 10 rotations. It is mentionable that only the specimens with this large strain in the tempered condition and with enormous hardness values over 700 HV seemed to contain these pre cracks.

Discussion

To get an overview concerning the actual results Figure 7 gives a relationship between hardness and endurance limit of our investigations. For 5 of our 6 tested HPT and initial states a linear correspondence appears until 500 HV and 837 MPa. The orange lines indicate the endurance limit calculated from the hardness using the equation $\sigma_0 = 1.6 \text{ HV} \pm 0.1 \text{ HV}$ by Murakami [5]. The good accordance of our results with Murakami’s studies is an unexpected coincidence because Murakami used another load ratio, larger macro specimens, and another type of testing. Important is the fact that the linear correspondence seems to be independent of the carbide and grain morphology except for the tempered state after 10 HPT rotations. In this state process flaws like reported in [6] from the HPT treatment lead to an increase of the fatigue limit that was lower than expected from the enormous hardness. The most likely reason for this behavior seems to be found in the pre cracks after HPT. They lead to an unusual high topology in the fatigue fracture surfaces, which was only observed in the tempered state after 10 rotations. Another possible reason could be different
appearance of the shear bands, but no evidence for this hypothesis was found. The red area in Figure 7 indicates the potential of UFG SAE 1045 when regarding the endurance limit, under the condition that pre cracks and other process flaws could be avoided.

![Figure 7: Relationship between hardness and endurance limit revealing a linear correspondence](image)

**Summary and conclusions**

In these investigations the fatigue behavior of medium carbon steel SAE 1045 with different initial carbide morphologies processed by HPT was analyzed. High hardness values up to 726 HV and a homogenous ultrafine grained microstructure were observed. The results of bending fatigue tests let us to the following conclusions:

- The endurance limits correspond linearly to the hardness until 500 HV and 837 MPa. The carbide morphology does not affect the linear behavior of the fatigue limit in this regime.
- Over 500 HV the fatigue limit does not correlate with the linear relationship of hardness and fatigue limit. One reason are pre cracks before fatigue loading from the HPT treatment.
- Different appearances of shear planes in the different states on the fracture surface could be determined. The influence of this difference regarding fatigue properties could not be quantified but might constitute a key for the understanding of the deformation behavior during HPT and fatigue testing.

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**References**

[1] R.Z. Valiev, R.K. Islamgaliev, and I.V. Alexandrov, Bulk nanostructured materials from severe plastic deformation, Progress in Materials Science 45 (2000), 103-189.
[2] Z. Horita and T.G. Langdon, Achieving exceptional superplasticity in a bulk aluminum alloy processed by high-pressure torsion, Scripta Materialia 58 (2008) 11, 1029–1032.
[3] M. Hück, Ein verbessertes Verfahren für die Auswertung von Treppenstufenversuchen, Z. Werkstofftech. 14 (1983), 406-417.
[4] Y. Ivanisenko, et al., Deformation mechanisms in nanocrystalline palladium at large strains, Acta Materialia 57 (2009) 27, 3391-3401.
[5] S.K. Y. Murakami, S. Konuma, Quantitative evaluation of effects of non-metallic inclusions on fatigue strength of high strength steels, Int J Fatigue 11 (1989) 5, 291-298.
[6] T.E. McGreevy and D.F. Socie, Competing roles of microstructure and flaw size, Fatigue Fract Engng Mater Struct 22 (1999), 495-508.