Fatigue behavior of ultrafine grained medium Carbon steel processed by severe plastic deformation

C Ruffing¹, Yu Ivanisenko² and E Kerscher¹
¹ Working Group of Materials Testing, University of Kaiserslautern, Kaiserslautern, Germany
² Institute of Nanotechnology, Karlsruhe Institute of Technology, Karlsruhe, Germany
E-mail: ruffing@mv.uni-kl.de

Abstract. The endurance limit of materials has been observed to be significantly increased in materials with an ultrafine grained microstructure [1, 2]. As this effect, however, has not yet been investigated in steels, fatigue experiments of an unalloyed medium carbon steel with a carbon content of 0.45 wt.-%, which was treated by high pressure torsion (HPT) [3-5] at elevated temperature were carried out. The treatments were applied to discs which had different initial carbide morphologies and showed an increase of hardness after HPT by a factor of 1.75 – 3.2 compared to the initial states, whereby the amount of increase depends on the initial carbide morphology. The maximum hardness achieved was 810 HV. The discs were cut into fatigue specimens in the form of bars of the size of 4 mm x 1 mm x 600 µm. Until a hardness of 500 HV the endurance limits correspond linearly with the hardness. This is no longer the case at higher hardness values, where inherent and process-initiated flaws lead to lower fatigue limits. The maximum endurance limit exceeded 1050 MPa in 4-point-micro-bending and at a load ratio of R = 0.1. Fractography revealed different crack initiation sites like pre cracks and shear bands [6, 7] resulting from HPT or fisheye fractures initiated from non-metallic inclusions.

1. Introduction

Since the last century many efforts have been done to induce thermomechanical or thermal treatments which enhance the mechanical properties of materials. Improving the quasistatic and simultaneously the cyclic properties is still a main challenge in this context. Thus, it is not only the strength but the combination of strength and ductility which is said to be most beneficial to reach an optimized material for fatigue [8]. Therefore grain refining is the only method to increase strength without lowering toughness and ductility. Nanostructuring seems to be the logical way to increase fatigue properties of materials in the best manner [9, 10]. In the last 20 years a high amount of publications appeared concerning ultrafine grained and nanostructured materials but publications were mostly focused to the production processes and the quasistatic properties. Concerning fatigue only a limited selection of publications exists and in the most cases only materials like aluminum, titanium or copper with fcc and hcp crystal systems were investigated [1, 2, 11]. There are only very few publications dealing with fatigue properties of high strength ultrafine grained steels resulting from severe plastic deformation [12-14]. The following investigations represent a mechanical and microstructural characterization of medium carbon steel. Severe Plastic Deformation (SPD) during the High Pressure Torsion (HPT) treatment at elevated temperatures was used for grain refining in respect to different initial carbide distributions and morphologies. This led to significantly increased hardness and better fatigue properties in the ultrafine grained condition. Overall this paper constitutes an outline of the relationship between microstructure and the fatigue properties. Crack initiation was found to be microstructure dependent: Initiation from the surface, from nonmetallic inclusions, but also from shear bands resulted in different endurance limits.
2. High Pressure Torsion at elevated temperatures

Previous investigations have shown that samples of C45 processed by HPT at room temperature contained numerous cracks and flaws. By increasing the processing temperature to 350°C, samples with high strength and moderate ductility were obtained [15]. In the present study specimens with the diameter of 10 mm and the thickness of 1 mm were subjected to a HPT deformation under a pressure of 6 GPa for six and ten rotations (N) at the temperature of 380°C which is lower than C45 steel recrystallization temperature, using custom built HPT device (W. Klement GmbH, Lang, Austria) equipped with induction heating. The temperature was maintained during HPT deformation with the accuracy of ±1°C. After HPT deformation the thickness of samples was reduced to \( h_p = 0.8 \) mm (this reduction of thickness occurred already during the application of high pressure, therefore this final thickness was used for estimation of shear strain at HPT). The shear strain \( \gamma \) in a certain point of the sample is a function of the distance of this point from the sample center, \( R \) as given by:

\[
\gamma = \frac{2\pi NR}{h_p}
\]

3. Materials and experimental procedure

As base material for the SPD via HPT the medium carbon steel SAE 1045 (Fe balance, 0.46%C, 0.64%Mn, 0.17%Si, 0.011%P, 0.009%S) was used with four different carbide morphologies in the initial state. The steel was delivered in the normalized (n) state. Afterwards the as-received base material was heat treated in three different ways, what means patenting (p), spheroidizing (s) annealing and tempering (t) to reach overall four different initial microstructures as starting condition for HPT. For patenting the as-delivered material was annealed at 900 °C for 1 hour, quenched to 375 °C for 3 seconds and subsequently annealed at 500 °C for 30 minutes. Spheroidization annealing was made for 40 hours at 680 °C. The base material was austenitizated at 850 °C, quenched to room temperature and annealed at 450 °C for 1 hour to reach a tempered microstructure. To show the influence of the applied shear strain on the microstructure evolution after SPD two states (s+t) were investigated with six HPT rotations and all four states were investigated after ten HPT rotations. The applied shear strain at the fatigue sample extraction point at 3.1 mm distance to the midpoint was 146 respective 243, calculated using equation (1). The extraction of the micrographs was also at this position on the radius.

States are named by ini for initial state with a leading n, p, s, or t for the carbide morphology, or by HPT with suffix 6 or 10 depending on number of rotations during HPT.

Fatigue tests were done on a BOSE Electroforce 3230 servoelectric test system using 4-point-bending. Micro-specimens, exhibiting an inner mountings distance of 3.2 mm, were used for the load controlled fatigue tests (R=0.1) at a frequency of 40 Hz. To determine the endurance limit the staircase method [16] was used with different step sizes for the different states. Hardness measurements were done with an ASMEC UNAT 2 Nanoindenter and a proof force of 200 mN on a surface polished with colloidal SiO. A PHILLIPS SL40 Scanning electron microscope (SEM) was used for fractographic and microstructural investigations until magnification of 4000x at a voltage of 20 kV. For the micrographs with higher magnification a ZEISS Supra 40VP at 5 kV was used.
4. Microstructures

Micrographs of the different states prior and after HPT are shown in Figure 1. The micrograph of n-ini reveals a ferritic-pearlitic microstructure as typical appearance after normalizing. After patenting the pearlite lamella spacing was reduced and the volume fraction of pearlite increased. In the spheroidization annealed state s-ini pearlite was completely spheroidized. The tempered initial state t-ini reveals a needle shaped ferrite matrix with small dispersed carbides. The HPT-6 procedure caused only a small grain refinement and a slight change in the microstructure visible in the second row of micrographs. The shape of the carbides in s-HPT-6 partly changed and they seem to be elongated. The same behavior offers s-HPT-10 but the amount of carbides seems to be lower as in the initial condition. The grain size has been significantly decreased finally into the UFG regime smaller than 1 µm.

The pearlite colonies of the initial pearlitic states were nearly completely dissolved in the n-HPT-10 but also in the p-HPT-10 state. Smallest grain sizes with equiaxed grains were reached in n-HPT-10. Straining the tempered state up to ten rotations also results in an ultrafine grained microstructure similar to the patented state after the same amount of rotations with no visible needles anymore. Overall, significant microstructural changes and a high amount of grain refining was observed after HPT-10 treatment.

![Micrographs of different states](image)

**Figure 1:** SEM microstructures of the initial on the left, the HPT-6 in the middle and HPT-10 state on the right side after etching with Nital

4. Hardness distribution after High Pressure Torsion at elevated temperatures
After severe plastic deformation via HPT the ultrafine grained disks offer a gradient in hardness, like visible in Figure 2 using nanoindentation. This hardness distribution results from the above mentioned strain gradient during the torsional deformation. In Figure 2 a) an overview of HPT-10 hardness distributions compared with them of the initial states is given. The relationship between microhardness and distance to the middle of the disc, when moving on a straight line, reveals a nearly linear characteristic for p-HPT-10 from approximately 350 HV in the middle up to 800 HV at the outer diameter. Similar minima and maxima hardness values appear when regarding t-HPT-10 and n-HPT-10. Figure 2 b-d) two dimensional hardness patterns of p-HPT-10 (b), n-HPT-10 (c) and s-HPT-10 (d) are listed. They exhibit, in contrast to the patented state, a saturation tendency in hardness with higher distances than 1 mm from the middle. This is also the fact for the spheroidization annealed state after HPT but the overall hardness level is lower, analogous to the lower hardness in the initial state. In Figure 2 b-d) two dimensional hardness patterns of p-HPT-10 (b), n-HPT-10 (c) and s-HPT-10 (d) are listed. They represent an expansion from one direction to the whole disk surface what allows an appraisal concerning the homogeneity of the disk surface. Figure 2 b) shows in this context the highest homogeneity with well-defined circular color contours followed by Figure 2 d) which only shows a deviation from uniformity on the left edge. Larger inhomogeneity with clearly increased hardness is visible in Figure 2 c) for half radius distances from the middle and angles from 90°-270°.

5. Fatigue properties before and after High Pressure Torsion

A description of the execution of the fatigue test as well as their experimental circumstances are described in detail in earlier publications [17] together with the respective S-N-curves. Only n-HPT-10 was not published before but results were determined analogous. So Figure 3 reveals a relationship between the hardness and the endurance limit of the respective
material state indicated with marks. This relationship reveals a linear behavior until a hardness of about 500 HV for the initial states, HPT-6 states and s-HPT-10 state. Deeper investigations reveal crack initiation of these states to be partly located at the surface and partly at nonmetallic inclusions, mostly MnS-lines but also large Mg, Al, and Ca containing inclusions, in this regime. With higher hardness over 700 HV the values for endurance limit differ from the linear characteristic in a high extent. The fatigue limit still increases with hardness but to a much lower slope as for lower hardness. This is known from high strength materials in [18]. A maximum endurance limit of 872 MPa was determined for the normalized HPT state after 10 rotations. Not shown here is the fact, that n-HPT-10 is the only state which shows cracks that were still initiated at nonmetallic inclusions at load cycles of about 4x10^6. The results for the patented state p-HPT-10 will be published separated, but offer considerable higher fatigue limits.

![Graph showing relationship between hardness and endurance limit for initial and UFG states](image)

**Figure 3: Relationship between hardness and endurance limit for initial and UFG states**

Crack initiation was mostly located at nonmetallic inclusions containing Mg, Al and Ca in the n-HPT-10 state or inhomogeneity in the t-HPT-10 state.
Figure 4 reveals the different crack initiation sites for the HPT-10 states as imaged from scanning electron microscopy (SEM). In Figure 4 a) crack initiation from a relatively large Al, Ca, S inclusion at the surface of an s-HPT-10 specimen is shown. Around this no special features are visible on the rather flat and unruffled fatigue fracture surface. A very uneven and unsteady fatigue fracture surface is shown in Figure 4b) when regarding a fatigue specimen of t-HPT-10. This is a typical crack appearance for this state, where irregular features from HPT act as a crack initiator. Figure 4 c-d) show the same fisheye fracture in a SEM and optical microscopy picture. The low resolution of the optical image (Fig 4d) reveals the typical bright circular plane around the nonmetallic inclusion. In the SEM picture (Fig 4c) the flat appearance of this face and the nonmetallic inclusion is shown in higher resolution. The size of the fisheye initiating inclusion is around half as large as in the s-HPT-10 case of Figure 4 a).

6. Discussion – Conclusions

The previous explanations represent a summary of micrographs, hardness measurements and fatigue properties of ten different material conditions of the medium carbon steel SAE 1045. These are four material states with different heat treatments as initial conditions. After six HPT rotations two material states were investigated and furthermore every single of the four initial states after 10 HPT rotations.

The micrographs reveal a very high extend of grain refining and homogenization for the HPT-10 states depending on the initial hardness and initial carbide structure of the specimen. So, the state with initially lowest hardness and spheroidal carbides obtains the highest grain size after HPT-10. One reason can be the more dispersed state of carbides. An initially pearlitic or tempered microstructure seems to result in finer grain sizes with a high amount of decomposition of pearlite. The HPT-6 states exhibit a clearly different behavior. Only a small grain refining can be observed. There are two general possible reasons for this behavior. One reason is a wrong applied too high, temperature which results in grain coarsening or recrystallization. Another reason is a too small amount of shear strain. Because temperature was kept under the recrystallization temperature under surveillance a too small amount of shear strain led to the low extend of grain refining in our case. This is in fact because of the
lower number of rotations, but also a slipping between the rotating anvils and the SAE 1045 specimen is possible. Because of the only small differences between initial and HPT-6 micrographs slipping is the most feasible theory. As a consequence only the HPT-10 states were investigated more intensive concerning their hardness. All states show well defined hardness gradients around the midpoint, following the shear strain distribution at HPT (Eq. 1). Inhomogeneity in this gradient can be a hint to possible cracks inside the HPT specimen. This is the case for the normalized state, where often cracks after HPT were observed in microstructural investigations of the cross sections of HPT specimen. The spheroidizing annealed state didn’t offer cracks at all and the patented state only in a few cases. So, the different appearance of the hardness gradient for the patented state on contrast to all other after HPT-10 can also be a hint to cracks but more intensive investigations have to be done in future.

Figure 3 reveals a typical behavior for metallic materials concerning their fatigue properties like reported in [19]. Because of the grain sizes of the low hardness states, which are larger than most of the nonmetallic inclusions, the matrices of these materials are decisive for determining the endurance limit. Most of the specimens of the s-HPT-10 state initiate cracks at nonmetallic inclusions at the surface but also cracks direct from the surface without any distinctive feature were observed. This is a hint to a competing behavior between intrinsic (microstructure) and inherent (e.g. nonmetallic inclusions) flaws what means that microstructure and the non-metallic inclusions offer similar dimensions. The state t-HPT-10 offers large inhomogeneity probably from HPT. This can be seen as process flaws which lower the endurance limit compared to states that offer only intrinsic and inherent flaws. In the case of the n-HPT-10 state mostly non-metallic inclusions are responsible for crack initiation. The formation of a fish-eye fracture for a high severely plastic deformed UFG-material implicates a high homogeneity at the location of highest stress around the crack initiating inclusion what makes crack initiation possible also from such a rather small inherent flaw.

Overall the investigated ultrafine grained material states of SAE 1045 show exceptional hardness and fatigue properties. The amount of increase of these two properties depends on the grain refining. But also the homogeneity of the highly loaded material volume plays an import role when regarding the endurance limit because irregularities from HPT lower the fatigue limit of the t-HPT-10 state. It is well known that nonmetallic inclusions lead also to a deviation from the linear behavior. But the relatively low fatigue limit of n-HPT-10 when comparing with the linear correspondence of hardness and endurance limit could not completely be clarified. The small size of crack initiating inclusions in the ultrafine grained materials is noticeable. Also the fact, that cracks initiate after \(4 \times 10^6\) cycles indicate a low possibility of the n-HPT-10 microstructure to stop micro-cracks initiated at the inclusions in contrast to the other states.

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