Plasticity, Failure and Fatigue in Structural Materials-from Macro to Nano: Proceedings of the Hael Mughrabi Honorary Symposium
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Fracture toughness and fatigue crack propagation measurements in ultrafine grained Iron and Nickel

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Abstract

Nanocrystalline and Ultrafine-grained (UFG) materials are commonly known as materials with extraordinary mechanical properties . Due to the limited dimensions of HPT-deformed materials little is known about their fatigue properties whereas particulary this process delivers highly refined materials with little effort. In the framework of this study pure nickel and Armco-iron were subjected to High Pressure Torsion (HPT) and in combination with different heat treatments the grain-size was varied between approximately 100 nm and 10 μm. Afterwards crack-propagation measurements with reference to different grain-sizes were conducted and monitored. It could be shown that there is a distinctive difference in the crack path with respect to the grain size, which has an influence on the threshold value of crack propagation as well. With respect to fracture toughness measurements in the UFG regime a quite good combination of toughness and hardness was observed.

Introduction

Grain refinement achieved by Severe Plastic Deformation (SPD), for instance via Equal Channel Angular Pressing (ECAP) or High Pressure Torsion (HPT) has been studied intensively during the last two decades. In general such methods are capable of producing fully dense bulk-materials with grain sizes predominantly in the submicrometer range. Ultrafine grained materials ehibit extraordinary mechanical and physical properties. A good overview of the mechanical and physical benefits is given for instance in [1]. Despite the fact that HPT delivers highly refined materials, little is known about fracture and fatigue properties of materials especially produced by this technique which is comparable to ECAP-defomation via Route A. Simply because especially this process delivers mostly limited dimensions of material for material testing with respect to fracture and fatigue experiments. In the framework of this work moderate dimensions of samples were produced by HPT and fracture toughness and fatigue crack propagation measurements were performed subsequently.

Experimental Procedures

In the present study Armco-iron and Nickel, a representative for a bcc and a fcc metal was investigated. The chemical compositions are given in Tab. 1.

	Ni	Co	Cu	Fe	C	S	
Nickel	> 99.97	5.10^{-5}	0.001	0.0015	< 0.10	0.0003	
	Fe	C	Mn	Al	N	S	P
Armco-iron	> 99.98	0.0016	0.0095	0.0031	0.0034	< 0.0005	< 0.0005

Table 1: Chemical composition of the investigated materials in wt%.

The initial material with an average grain size of about 50 μm was subjected to High Pressure Torsion (HPT). The starting samples had a diameter of 14 mm and a thickness of 2 mm. The samples were deformed up to an equivalent Mises strain of 32 with reference to a radius of 3 mm. This severe deformation leads to a microstructural state where no further fragmentation takes place and is often termed saturation state [2]. Afterwards the samples were heat treated in a way so that grain sizes of 300 nm , 1 μm , and 10 μm were obtained. The datas for the individual heat treatments (time and temperature) were obtained elsewhere [3]. Additionally the severly plastically deformed state, denoted by Ni-SPD and Fe-SPD was investigated.

For fracture toughness and fatigue crack propagation experiments CT-specimens were machined out of the deformed and heat-treated HPT-discs. The CT-specimens had a width W = 8mm and a thickness of 1.6 mm. For further fracture toughness experiments samples with a width W of 5 mm and a thickness of 2.5 mm were produced. Afterwards all specimens were fatigue pre-cracked under cyclic compression-compression loading. Since the degree of deformation is dependent on the radius of the disc the final notch was situated at a radius of 2 mm where a saturation of grain fragmentation was achieved. The analysis of fracture toughness was based on the ASTM standard E399. The fatigue crack propagation experiments were performed with a frequency of 100 Hz and with load-ratios of 0.1 and 0.7 respectively. The crack length was measured by the potential drop method (DC).

Results

Fracture Toughness

Tab. 2 shows the obtained fracture toughnesses and the ultimative tensile strengths.

	$\sigma_{UTS}[MPa]$	$K_{IC}[MPa\sqrt{m}]$
Fe-SPD	1500	52.6
Fe-300nm	>1200	48.6
Ni-SPD	-	89.0
Ni-300nm	1400	74.4

Table 2: Obtained fracture toughness data and ultimative tensile strength.

Although a reduction of grain size commonly leads to an increase of the tensile strength when the Hall-Petch relationship is taken into account, a reduction of grain size leads simultanously to a deterioration of toughness as well. However in this case it is obvious that a good combination between fracture toughness and strength could be achieved in ultrafine grained iron and nickel. Furthermore it seems that a small increase in grain size results in a small decrease of fracture toughness referring to the heat treated states Fe-300 nm and Ni-300 nm. Finally it should be noted that for larger average-grain sizes, 1μm and 10 μm, no valid fracture toughnesses could be measured due to the fact that these samples had a too low hardness to measure a valid plain strain fracture toughness.

An explaination for high toughness connected with high tensile strength will be derived from observing the fracture surfaces of the specimens later.

Fig. 1a shows the overload fracture surface of severe plastically deformed iron. The small arrow in Fig. 1a with the denomination SD indicates the shear direction. In advance it can be said that this fracture surface is representative for all ultrafine-grained fracture specimens.

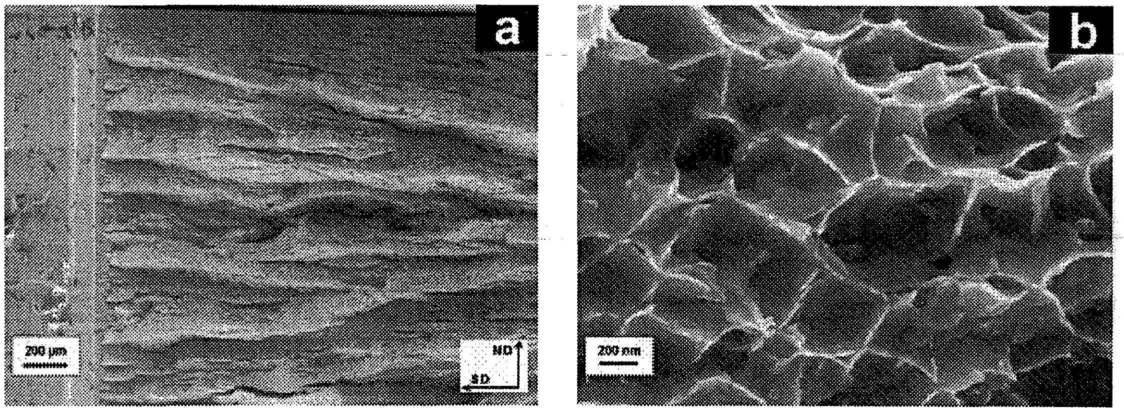

Figure 1: Overload fracture surface of Fe-SPD: (a) Macroscopic overview of the fracture surface showing typical delaminations ; (b) Typical dimple structure on the microscopic level consisting of dimples.

On the fracture surface a variety of delaminations can be seen, which are extended through the whole specimen. The reason for such delaminations could be on the one hand on account of certain texture characteristics. Wetscher et al. [4] observed in HPT-processed iron during severe plastic deformation a development of a (110)[001] texture. On the other hand small inclusions, which are aligned during the deformation process in the shear direction, could favour the formation of delaminations.

The occurence of these delaminations is crucial to achieve the high fracture toughness, which is explained by Song et al. [5]. Basically when plain strain fracture toughness is measured the region in front the crack tip is governed by a high amount of tensile triaxiality. Since delaminations during the blunting of the crack tip in the normal direction occur (indicated in Fig. 1a with ND), the tensile stress in the normal direction is decreased when a delamination occurs. Microscopically seen a plain stress state with the thickness of one delamination to the next one is generated which commomly leads to a higher toughness. To be short, on account of the immense shear deformation the specimens acts like a parallel connection of thin specimens resulting in a high global fracture toughness.

In Fig. 1b the microscopic fracture surface of Fe-SPD which is again representative for all ultrafine grained investigated specimens. It can be seen that microductile fracture occurs and that the dimple size is in the order of the grain size. Generally in conventional ductile materials voids origin from inclusions and impurities which afterwards coalescence and lead to the final fracture. Here in the case of the absence of inclusions triple junctions seem to act as initiation sites for the dimple formation, which can be seen at the bottom of the voids. So it is comprehensible that the dimple size is restricted to the distance from one triple junction to another.

Fatigue crack propagation experiments

Fig. 2 illustrates the results of the crack propagation measurements for different grain sizes in Armco iron. The experiments were performed with a high (R=0.7) and a low load ratio (R=0.1) for the severe plastically deformed state (Fe-SPD) and for specimens with average grain sizes of 300 nm, 1 μm and 10 μm, respectively. Since the plastic zone under cyclic loading is smaller it was also possible to monitor crack propagation curves for the microcrystalline states. The arrows indicate the cyclic stress intensity factor ΔK, where the first crack propagation was found for the stress-ratio R=0.7.

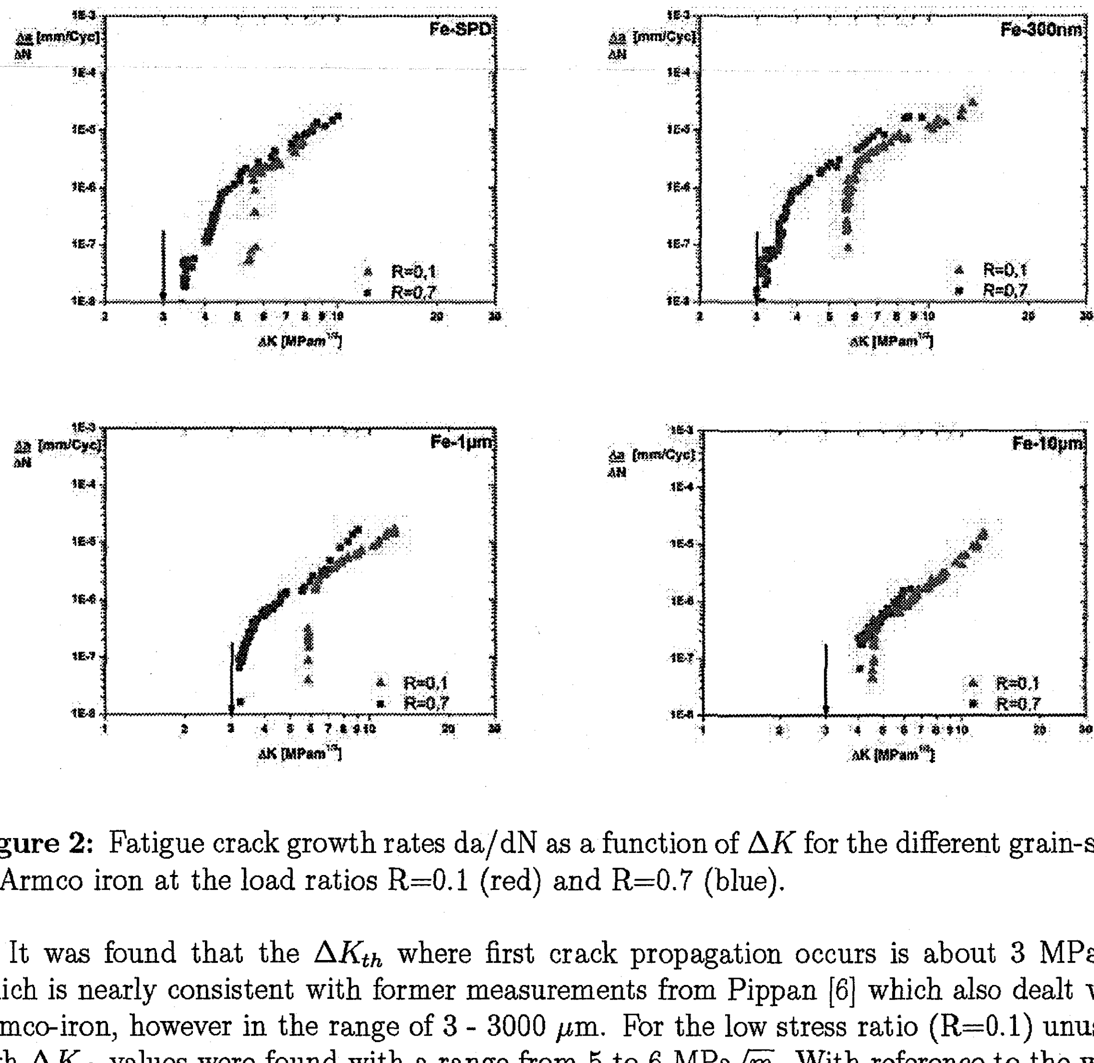

Figure 2: Fatigue crack growth rates da/dN as a function of ΔK for the different grain-sizes of Armco iron at the load ratios R=0.1 (red) and R=0.7 (blue).

It was found that the ΔK_{th} where first crack propagation occurs is about 3 $MPa\sqrt{m}$ which is nearly consistent with former measurements from Pippan [6] which also dealt with Armco-iron, however in the range of 3 - 3000 μm. For the low stress ratio (R=0.1) unusual high ΔK_{th}-values were found with a range from 5 to 6 $MPa\sqrt{m}$. With reference to the work of Pippan [6] lower ΔK_{th}-values were exspected. This is due to the fact, that with decreasing grain size the crack path turtosity decreases and finally the influence of roughness induced crack closure should decrease as well. With respect to ultrafine-grained and nanocrystalline materials the influence of crack path turtosity was firstly investigated in a work of Hanlon et al., however focusing on the crack propagation rate [7]. It must be admitted that residual stresses due to the high plastic deformation could play an important role for high ΔK_{th}-values, however for specimens with grain sizes of 300 nm the heat treatment should have relaxed these stresses. A possible explaination for increased ΔK_{th}-values may be given when the fatigue fracture surfaces are investigated.

Fig. 3a shows the fatigue fracture surface of iron with an average grain size of 300 nm, where individual grains can be distinguished. In Fig. 3b the fatigue fracture surface of an iron specimen with an average grain-size of 1 μm is shown where no grain structure is visible.

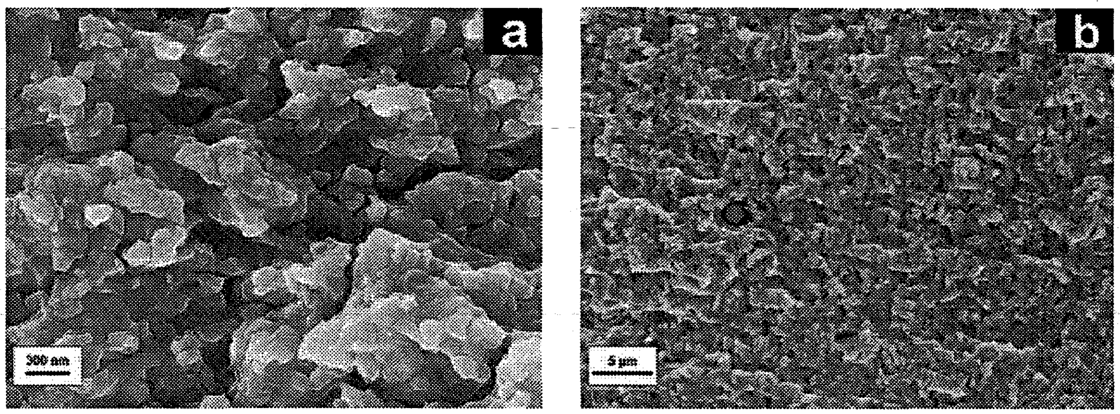

Figure 3: Comparison of the fatigue fracture surfaces: (a) Iron with an average grain size of 300nm depicting an intercrystalline crack path; (b) Iron with an average grain size of 1 μm showing a transcrystalline crack path.

Generally these SEM-pictures should point out that whereas in specimens with grain sizes smaller than 1 μm an intercrystalline crack path was found, specimens with grain sizes larger than 1 μm show a conventional transcrystalline crack path. This was found in iron and nickel as well. As shown in the section fracture toughness, under monotonic loading the submicrocrystalline materials behave ductile. As shown here, under cyclic loading a kind of "brittle" behaviour, in the form of intercrystalline crack propagation, arises. This could be attributed to the much smaller cyclic plastic zone, which seems to favour the crack propagation along grain boundaries. Further SEM-studies showed that the influence of the crack propagation rate is not significant on the crack morphology.

The intercrystalline crack path is accompanied with a certain crack turtosity, which is in the order of some grain diameters. This can contribute to roughness induced crack closure and finally lead to higher ΔK_{th}-values.

Conclusions

In the framework of this study the HPT process was used for performing a systematic study on the influence of grain size on crack propagation and fracture toughness. Following conclusions can be drawn:

- Firstly no crucial differences between Armco-iron and Nickel with respect to the general tendencies was found.
- A good combination between fracture toughness and strength in both materials was observed in the UFG microstructures.
- A transition from intercrystalline for grain sizes smaller than 1 μm to transcrystalline fatigue crack popagation for grain sizes larger than 1 μm was observed.
- In contrast to this monotonic loading leads to microductile fracture.

- For low stress-ratios, an unexspected high threshold-value of crack propagation was measured in the submicrometer regime, which seems to be caused by the change in the crack path.

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