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## Regular article

# Simultaneous enhancement of strength and fatigue crack growth behavior of nanocrystalline steels by annealing



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### ABSTRACT

Nanocrystalline materials tend to have an inferior fatigue crack-growth behavior, compared to coarser-grained counterparts, due to the loss of crack-closure effects. Strategies to mitigate this effect are widely unknown as grain-refinement changes the strength, however, often also the fracture-mode and cyclic material behavior so that the effect of individual parameters is difficult to discriminate. To address this issue, the fatigue crack-growth behavior of a nanocrystalline austenitic-steel (316L) in two conditions with different strength but comparable grain-size is investigated. The increased strength level, realized by an annealing treatment, leads to an improvement of the fatigue crack-growth behavior.

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Severe plastic deformation (SPD) of metals can be used to study the effect of grain refinement on a large variety of mechanical properties. The influence on the mechanical properties have been the focus of a vast number of studies: The yield strength [1–4], fatigue limit [5–10], fracture toughness [11–16] and fatigue crack growth behavior [6,8,17– 23] have been studied to assess the damage tolerance of this new material class. Concentrating on the damage tolerance under cyclic loading, the majority of these studies report an overall deteriorated fatigue crack growth (FCG) behavior of nanocrystalline (NC) and ultrafinegrained (UFG) metals, compared to their coarse-grained (CG) counterparts. The origin of these changes, however, are still not fully clarified in detail in our view. The difficulties to thoroughly understand FCG can be ascribed to the complexity of the crack growth process, which is influenced by a multitude of parameters. A grain size reduction from tens or hundreds of microns to lower than 1 µm, which is typical for SPD processing of metals, strongly increases the yield strength. At the same time, however, also the fracture mode, the crack path tortuosity and the effectiveness of crack closure mechanisms during cyclic loading can be significantly altered [22,23]. Thus, comparing the FCG behavior of CG-materials with their SPD states only yields information about the overall effect and is not able to unravel the influence of the individual material parameters. This information would be essential for the targeted improvement of UFG and NC with respect to their FCG-behavior. The present study takes one step in this direction by focusing exclusively on the effect of yield strength on the FCG-behavior of

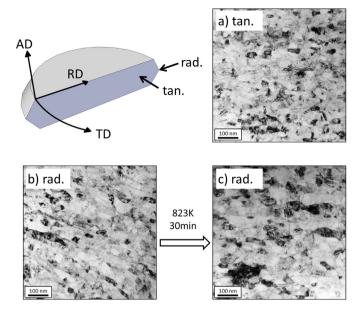
\* Corresponding author. *E-mail address:* anton.hohenwarter@unileoben.ac.at (A. Hohenwarter). high pressure torsion is investigated, which allows to increase the strength by annealing-treatments without affecting the grain size. For this purpose disks with a diameter of 30 mm and a thickness of 75 mm of an austenitic steel ture 316L (max 0.03 C 0.30 Si 1.70 Mp

nanocrystalline alloys. To realize this a NC austenitic steel produced by

7.5 mm of an austenitic steel type 316L (max. 0.03 C, 0.30 Si, 1.70 Mn, 17.50 Cr, 14.50 Ni, 2.70 Mo, all values in wt%) were subjected to high pressure torsion (HPT) at room temperature for 10 revolutions with an applied pressure of 4 GPa. A more detailed description of the deformation process is given elsewhere [24,25]. For the FCG tests, samples were taken with a distance of 3 to 15 mm from the center of the HPT disk (equivalent strain of about 22 to 109). In this area a relatively homogeneous hardness along the radius was measured and so it is guaranteed that all samples, irrespective of their radial position, possess identical microstructural features. Transmission electron microscope (TEM) investigations, Fig. 1(a,b), revealed a mean grain size of about 50 nm, with an elongation of the grains slightly inclined to the shear direction of the HPT process. With microhardness measurements a mean hardness of about 5 GPa was determined for this material state, which will be further denoted as "as-deformed NC 316L". Another set of samples was heat-treated at 823 K for 30 min, which lead to a further strengthening of the steel to about 6 GPa which is explained by defect annihilation and relaxation processes [26,27]. The grain size was only slightly affected by the heat treatment, as it can be seen in the TEM image in Fig. 1(c). The apparent increase of grain size is likely to be an effect of lowering the defect concentration in the interior of the grains rendering the former SPD-structure clearer. From both material states compact tension specimens were machined with dimensions according to ASTM E647 (W = 5.4 mm, B = 1.2-1.4 mm) as described in [22,28].

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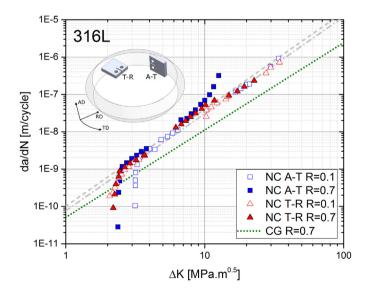
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**Fig. 1.** TEM bright field image of as-deformed 316L with the viewing direction parallel to the TD (a) and parallel to the RD (b). The annealed microstructure recorded parallel to the RD (c) exhibits a somewhat clearer microstructure, which indicates that defect annihilation processes took place.

Samples were taken with two different orientations of the crack with respect to the deformation process to additionally investigate possible orientation effects, as shown in the inset of Fig. 2. The used coordinate system is presented in Fig. 1 containing the axial (A), radial (R) and tangential (T) directions of the HPT disk. The sample orientations are named after this coordinate system with a two letter code indicating to the crack plane normal and designated crack propagation direction with A-T and T-R.

Before testing the samples were pre-fatigued with a compressioncompression loading to generate a fully open sharp pre-crack. The samples were tested with an electrodynamic testing machine Instron



**Fig. 2.** Fatigue crack growth curves of A-T and T-R oriented samples of as-deformed NC 316L tested at R = 0.1 and R = 0.7. Dash-dotted and dashed lines show fitted curves of R = 0.1 and R = 0.7 data, respectively. The R-ratio, as well as the sample orientation only slightly influence the FCG rates in the Paris regime. NC 316L exhibits about four times higher FCG rates compared to results of coarse-grained 316L from literature [33] (see dotted line in the diagram).

ElectroPulse™ E3000, with a sinusoidal load and a frequency of 45 Hz at load ratios R of 0.1 and 0.7. The load rising method was in use to evaluate the FCG-curve, for more details see [29]. The experiments were performed with a constant amplitude test starting at a  $\Delta K$ -level of 1.5 MPa $\cdot$ m<sup>0.5</sup> and after 2  $\times$  10<sup>5</sup> cycles without distinct crack growth (less than 3 µm) the load was increased by an increment of 0.2 MPa  $\cdot$  m<sup>0.5</sup>. With this procedure  $\Delta K_{th,eff}$  and after exceeding the load level for  $\Delta K_{th}$ , the FCG-curve was recorded. The crack length was measured during the experiment by using the direct current potential drop method [22] and the FCG-rate was determined according to ASTM E647. One specimen was tested per orientation and load-ratio. The repeatability of the results and the possible error sources within the measurements using the same testing setup have been discussed in a previous study [23]. Variations less than 5% difference in  $\Delta K$  and da/dN were calculated by considering typical error ranges within a measurement. Fracture surface analyses were performed in a field-emission scanning electron microscope (SEM) LEO Gemini 1525.

Before presenting the FCG data it is helpful to distinguish between intrinsic and extrinsic mechanisms controlling the FCG to isolate the influence of the yield strength from other effects [30]. Intrinsic mechanisms directly influence the material separation at the crack tip, whereas extrinsic mechanisms act behind the crack tip and reduce (shielding effect) or increase (anti-shielding effect) the crack advance. Typical extrinsic contributions in ductile microcrystalline metals are, for example, contact shielding, also known as crack closure mechanisms, which reduce the cyclic load at the crack tip by premature contact of the crack faces upon unloading. Besides such extrinsic contributions it is essential to identify the microscopic crack propagation and material separation process at the crack tip which represents the intrinsic crack growth mechanisms. The propagation process can be fully ductile or a combination with micro-cracking ahead of the crack tip.

The FCG diagrams in Fig. 2 show only a small difference in the threshold and Paris regime between the two sample orientations focusing on the same load ratio. This is in strong contrast to findings for other SPD processed materials, such as pure Ni and ARMCO Fe [22,23] especially regarding the Paris regime. The orientation dependency of these UFG metals originates from the intergranular crack path and the grain elongation parallel to the shear direction of the HPT process. Therefore, more frequent and larger crack deflections are observed for cracks growing perpendicular to the long grain axis (T-R samples), than for cracks parallel to the grain elongation direction (A-T samples) [22,23]. As regular crack deflections reduce the crack driving force [31], the FCG resistance of T-R samples is improved and thus, FCG-rates are lower for T-R oriented specimens than for A-T samples [22,23]. The vanishing FCG anisotropy in NC 316L can be explained by the less pronounced grain elongation and especially the even smaller grain size in combination with intergranular fracture, which is the prevailing crack propagation mechanism in the NC-state of this material and will be presented later in Fig. 4. Crack deflections are smaller and in general deviate in a lower angle from the straight crack path, which reduces the shielding effect of this extrinsic mechanism and also reduces the differences between the two orientations. Furthermore, FCG data from as-deformed NC 316L tested at R = 0.1 and R = 0.7 almost coincide, which implies that the mean stress effect is further reduced for NC 316L compared to the afore-mentioned other UFG metals. This indicates that extrinsic effects and so crack closure contributions only play a minor role in the FCG of NC 316L, even at low mean stresses.

In Fig. 3 the heat-treated NC 316L for both orientations in comparison with the pure NC-state are presented. Again, an orientation independent FCG-behavior and a reduced mean stress effect was observed. In Table 1 the effective threshold,  $\Delta K_{th, eff}$ , and threshold of stress intensity factor range,  $\Delta K_{th}$ , are presented showing only small variations. This indicates that also for the heat-treated state crack closure and, thus, extrinsic mechanisms play only a minor role. Therefore, the differences in the FCG-behavior of the as-deformed and heat-treated NC 316L can be solely attributed to the changes of the intrinsic FCG resistance. The effect

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