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Exceptionally high strength and good ductility in an ultrafine-grained 316L steel processed by severe plastic deformation and subsequent annealing

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Abstract

An investigation was conducted to evaluate the effect of annealing at different temperatures on the tensile properties of ultrafine-grained 316L stainless steel processed by high-pressure torsion (HPT). A “moderate-temperature” annealing at 740 K resulted in reduced strength and elongation due to the annihilation of mobile dislocations. A “high-temperature” annealing at 1000 K yielded a remarkably good combination of yield strength (~1330 MPa) and elongation to failure (~43%) which can be attributed to the almost full reversion of $\alpha'$-martensite formed during HPT into $\gamma$-austenite while the grain size remained very fine with a value of about 200 nm.

Keywords: Metals and alloys; Nanocrystalline materials; Annealing; Microstructure; Deformation and fracture

1. Introduction

The 316L stainless is a frequently used material in both medicine and industry due to its excellent properties, such as a good corrosion resistance and the low susceptibility to neutron absorption \cite{1}. The main crystalline phases in 316L steel are the face-centered cubic (fcc) $\gamma$-
austenite and the body-centred cubic (bcc) $\alpha'$-martensite. The good ductility of $\gamma$-austenite is usually accompanied by a relatively low strength which can limit its applications under high loads. At the same time, the high strength $\alpha'$-martensite with bcc structure exhibits low ductility. Therefore, the ability to process 316L steel with high strength and good ductility is a challenging task.

The strength of ductile austenitic steels can be increased by grain refinement using severe plastic deformation (SPD) techniques such as high-pressure torsion (HPT) and hydrostatic extrusion [2,3]. However, during SPD there is a simultaneous phase transformation from $\gamma$-austenite to $\alpha'$-martensite [2]. It was suggested that annealing after SPD may yield fine austenite grains [4,5] so that a combination of high strength and good ductility is feasible. The present research was initiated to study the tensile properties of samples processed by HPT and annealed at different temperatures. Our study shows that a combination of HPT-processing and subsequent annealing to 1000 K yields an exceptional combination of high strength and good elongation to failure by comparison with the available literature data for 316L steel.

2. Material and methods

316L stainless steel samples with a coarse-grained single phase $\gamma$-austenite structure were HPT-processed for 20 turns at room temperature (RT) with an applied pressure of 6.0 GPa and rotation speed of 1 rpm. The thickness and the diameter of the HPT-processed disks were ~0.75 mm and ~10 mm, respectively. In former studies, the microstructure and the phase composition evolution during HPT processing and subsequent annealing were investigated in detail [2,5]. Differential scanning calorimetry (DSC) revealed that between ~590 and ~740 K there was only a recovery in the microstructure whereas between ~740 and ~1000 K recovery, grain growth and an almost complete reversion of $\alpha'$-martensite to $\gamma$-austenite was also detected [5]. Therefore, in this study the tensile performance was investigated for the
samples annealed to the two characteristic temperatures of the DSC thermograms, i.e., to ~740 and ~1000 K. Small specimens were cut from the regions between the half-radius and the periphery of the HPT disks and these samples were heated in the calorimeter to ~740 and ~1000 K at a heating rate of 20 K/min and then quenched to RT at ~300 K/min.

The evolution of the phase composition due to annealing was investigated by X-ray diffraction (XRD) and the dislocation density was determined by X-ray line profile analysis (XLPA). The grain size of the samples was determined by electron back-scattered diffraction (EBSD) and transmission electron microscopy (TEM). The experimental details and the results of these investigations were given in earlier reports [2,5]. For the tensile tests, two miniature specimens were cut from each HPT-processed disk by electro-discharge machining (EDM). The gauge length, width and thickness of the specimens were 1.1, 0.95 and 0.6 mm, respectively. Tensile tests were preformed on a Zwick Z030 testing machine at RT with an initial strain rate of $10^{-3}$ s$^{-1}$.

3. Results and discussion

The yield strength, the ultimate tensile strength, the uniform elongation and the elongation to failure for the initial coarse-grained sample, for the HPT-processed specimens and for the samples annealed to 740 and 1000 K were determined from the tensile stress-strain curves (not shown) and the evolution of these data is plotted in Fig. 1. In order to explain the changes in the tensile properties of 316L steel during annealing, the evolution of the $\gamma$-austenite fraction, the grain size and the dislocation density are plotted in Fig. 2. The dislocation density was determined only for the main phase while the grain size characterizes the whole sample. The corresponding EBSD and TEM images were shown earlier [2,5]. It is well known that the $\gamma$-austenite phase in 316L steel is ductile; therefore, the high uniform elongation and elongation to failure as well as the low strength of the initial material were attributed to the large grain size and the nearly single-phase $\gamma$-austenite structure. The very
small grains and the high dislocation density developed due to 20 revolutions of HPT were responsible for the increase of strength. The strong reduction of the ductility for this sample is attributed partly to the increase of the α’-martensite fraction and to the significant decrease of the grain size due to the reduction of the strain hardening capacity.

![Graph](image)

**Fig. 1:** Tensile properties for the initial material, the sample processed by 20 turns of HPT and the HPT-processed specimens annealed to 740 and 1000 K.

After annealing to 740 K, both the yield strength and the ductility decreased. This annealing also led to a considerable reduction in the dislocation density; however, other microstructural features, such as the grain size and the fraction of α’-martensite, remained practically unchanged. Therefore, the decrease in strength and ductility can be attributed to the reduction of the mobile dislocation density as suggested in earlier work [6].

Between 740 and 1000 K, the increase of the grain size and the softer γ-austenite fraction has a softening effect, while a further reduction in the mobile dislocation density hardened the material. These effects compensate each other, leading to a practically unchanged yield.
strength. In addition, the increase of the grain size and the fraction of the ductile fcc phase led to a significant improvement of the elongation to failure from ~2.6 to ~43%.

![Graph showing grain size, dislocation density, and γ-austenite fraction for different samples.](image)

**Fig. 2:** The average grain size, the dislocation density and the γ-austenite fraction for the initial material, the sample processed by 20 turns of HPT and the HPT-processed specimens annealed to 740 and 1000 K. For the dislocation density, the solid and open square symbols indicate that the values were determined for the γ-austenite and the α’-martensite phases, respectively (always for the main phase).

Fig. 3 shows the yield strength versus the elongation to failure for the four 316L steel samples studied in this work by comparison with data available in the literature [1,7-11]. The datum points in Fig. 3 follow a general trend which is represented by the dotted line. Thus, the higher the yield strength of the sample so the lower the elongation to failure. The sample processed by HPT and annealed to 1000 K shows a deviation from the general trend as indicated by the arrow in Fig. 3. Therefore, this study demonstrates that 20 turns of HPT followed by annealing to 1000 K is capable of producing a UFG γ-austenite microstructure in 316L steel with exceptionally high strength and elongation to failure. It should be noted that
the measured elongation to failure increases with decreasing ratio of gauge length to width and increasing specimen thickness [12]. However, the two effects nearly compensate each other by comparing the data obtained on these miniature specimens with a standard sample with dimensions of $5 \times 1 \times 1 \text{ mm}^3$.

![Graph showing yield strength versus elongation to failure](image)

**Fig. 3:** The yield strength versus the elongation to failure for the 316L steel samples studied in this work compared with data available in the literature [1,7–11].

**5. Conclusions**

1. HPT processing dramatically increased the yield strength while the elongation to failure significantly decreased. The extremely high strength and limited ductility of the HPT-processed sample were attributed to the high fraction of $\alpha'$-martensite, the small grain size and the high dislocation density.

2. DSC annealing of the HPT-processed sample to a moderate temperature of 740 K led to a pronounced embrittlement and strength reduction. The reduction of ductility was most probably caused by the decrease of the mobile dislocation density during DSC annealing.
3. After annealing of the HPT-processed sample to a higher-temperature of 1000 K, a reverse martensitic transformation yielded a nearly single-phase \( \gamma \)-austenite structure with relatively small grain size (~200 nm). This microstructure showed exceptionally high strength with a concomitant good elongation to failure compared with the literature data.

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**References**


Research Highlights

- 316L steel with a nanocrystalline microstructure was processed by HPT
- The effect of annealing on the tensile properties was studied
- Annealing to 740 K yielded a pronounced embrittlement and a strength reduction
- Annealing to 1000 K led to a good combination of strength and elongation to failure
- The excellent tensile behavior was caused by the very fine austenitic microstructure