High strength state of UFG steel produced by severe plastic deformation

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Abstract. An UFG austenitic stainless steel of type 316 was produced by high pressure torsion at two different temperatures. As a result different nanostructures were observed in the investigated alloy characterized by different grain size and dislocation density. It was reported that the steel processed at both temperatures was characterized by significantly enhanced strength, which, in case of the steel processed at 430°C, exceeds the value expected for the given grain size according to Hall–Petch relation. This extra–strength is supposed to be due to the observed nanostructural features as segregations/clusters of solutes in grain boundary area formed by severe plastic deformation.

1. Introduction

The techniques resulting in grain refinement are widely used nowadays to increase strength of metals and alloys. Reduction of mean grain size is directly related to elevation of yield stress $\sigma_y$ as described by the Hall–Petch relationship [1, 2]:

$$\sigma_y = \sigma_0 + k_yd^{-1/2},$$

where $\sigma_0, k_y > 0$ are constants. This relationship operates for a wide range of materials [3] until nanometric range of grain size where Hall–Petch slope $k_y$ decays and even becomes negative [3, 4].

In the meantime the grain size is not the only factor affecting strength of ultrafine-grained (UFG) alloys, in particular produced by severe plastic deformation (SPD) [5]. Solid solution, strain hardening, precipitation can considerably contribute to the overall strengthening [6]. It was shown recently that SPD-produced Al–Mg alloy can exhibit a higher yield stress than the value predicted by Hall–Petch relationship for the given grain size which was explained by unusual grain boundary segregations induced by deformation [7].

This paper presents the latest results obtained for the UFG alloys produced by SPD with attention to this untypical redistribution of alloying elements and their impact to strengthening.

2. Experimental

The objects of this research was a stainless steel 316 (Fe–0.03C–17Cr–0.41Si–1.72Mn–0.01P–0.03S–12.9Ni–2.36Mo, wt. %). The steel was annealed at 1050°C for 1 hour and air quenched and then subjected to nanostructuring by HPT. HPT of specimens (disks 20 mm in diameter...
and 0.8 mm in thickness) was performed under a pressure of 6 GPa with 10 die–set rotations at room temperature and at 430°C. The structural characterization was performed by transmission electron microscopy (TEM) and X–ray diffraction (XRD). The grain size $d$ was calculated using TEM dark field images from an area located at half–radius of an HPT disc with the help of mean intersect lengths over 350 grains for each state. XRD measurements were performed with Rigaku Ultima IV diffractometer using CuK$_\alpha$ radiation (40 kV and 30 mA). The obtained XRD patterns were treated with Rietveld refinement method to calculate values of lattice parameter $a$, coherent domain size $d_{XRD}$ and elastic microdistortion level $<\varepsilon^2>^{1/2}$ for the initial and HPT–processed alloys using the MAUD software [8]. Microhardness was measured in terms of $H_V$ units using Micromet–5101 device. Atom probe tomography (APT) was performed with a Cameca FLEXTAP. The sample base temperature was 50K.

3. Results and discussion
The initial annealed and air quenched 316 steel exhibits typical coarse–grained structure with a mean grain size of 22 $\mu$m with log-normal grain size distribution (Fig. 1a). TEM observations of HPT steel processed at both temperatures showed highly refined microstructure with the grain size in the range of tens to hundred nm. The results of TEM investigations of the UFG steels are presented on Fig. 1b,c. The UFG steel obtained at room temperature had the mean grain size of about 60 nm. Selected area electron diffraction patterns showed spots forming uniformly distributed Debye–Scherrer rings typical for ultrafine structures dominant with high angle grain boundaries [5]. The analysis of the rings proved that the given state is characterized mainly by $\gamma$–Fe phase (FCC). Few spots corresponding to martensite were also detected, suggesting presence of a small fraction of this phase. However, XRD measurements revealed no resolvable martensite peaks for the given UFG state. It confirms that the martensite volume fraction is below XRD sensitivity (less than 5%). HPT 430°C steel had less distorted equiaxed UFG microstructure with mainly high-angle grain boundaries and a larger grain size (about 110 nm). In few grains one could observe twins (see Fig. 2) a fraction of which was estimated to be about $f = 0.07$. The analysis of selected area electron diffraction patterns testified the microstructure being formed by plain austenite which is also consistent with phase XRD analysis which shows no martensite peaks at XRD patterns for HPT 430°C steel.

| State      | $d_{XRD}$ (nm) | $a$ (Å) | $H_V$ (MPa) |
|------------|----------------|---------|-------------|
| Initial    | —              | —       | 170         |
| HPT RT     | 19±1           | 3.5952±0.004 | 585       |
| HPT 430°C  | 55±5           | 3.5975±0.0002 | 575      |

The results of XRD analysis are given in the Table 1. It is seen that UFG steel produced at room temperature has a notably less coherent domain size as the steel produced at 430°C which is in a good agreement with TEM data. It is also worth mentioning that lattice parameter had noticeably decreased for both UFG steels in comparison with initial annealed and quenched state, which could be connected with redistribution of alloying elements in the deformed samples.

The results of mechanical tests are listed in Table 1. It is seen that the 316 steel in both UFG states demonstrates high hardness which is significantly exceeding the value of the initial state. Moreover, the UFG steel produced at 430°C is characterized by similar strength as the HPT RT steel.

The results of strength measurements were interpreted in terms of Hall–Petch relation, where the yield stress was estimated as $H_V/3$. Fig. 3 demonstrates the generalized Hall–Petch plot with...
The data obtained in the present work were plotted against this dependence. It is seen that the strength of UFG steel obtained at room temperature is in a good agreement with literature data, while the strength of UFG steel produced at 430°C considerably exceeds the value expected for the given grain size according to Hall–Petch relation for this steel. From the microstructure analysis it is seen that enhanced strength of HPT RT steel can be explained by severe grain refinement when diminishing grain size leads to significant growth of yield stress value according to Hall–Petch relation. However, for the case of HPT 430°C steel the grain size contribution itself is not sufficient to explain the observed deviation of the yield stress from Hall–Petch predictions. As it can be estimated from Fig. 3, the difference between the expected and the measured values for the given grain size exceeds the inaccuracy limits. Certain increase of strength can be provided by hardening due to presence of twins. However their rather low fraction allows suggesting that the twins hardening contribution would not be sufficient to explain the measured hardness values for the HPT 430°C steel.

Earlier direct APT observations [10, 11] showed that 316 steel subjected to HPT at room...
Figure 2. Bright field TEM image of HPT 316 steel produced at 430°C showing twins.

Figure 3. A Hall–Petch plot for the austenite stainless steels collected in [9] and the data on strength of HPT 316 steel produced at room temperature and at 430°C (yellow circles).

temperature does not exhibit a trend for segregation. It was natural to suppose that HPT at high temperatures (unlike at room temperature) might lead to re–distribution of alloying elements and formation of segregations which can be responsible for a significant hardening in the case of HPT 430°C steel, additional to the grain size hardening.

Indeed, preliminary APT studies of HPT 430°C steel (Fig. 4) gave an evidence for the formation of rich grain boundary segregations. It is seen that grain boundary area contains
elevated concentration of Mo, Si and Cr, while Ni does not exhibit any increase in concentration there. Further understanding of the nature of influencing the segregations to mechanical properties of HPT steel 316 requires additional thorough studies which are planned to be carried out in the nearest future.

![Concentration profiles for alloying elements computed across a grain boundary in UFG 316 steel processed at 430°C thanks to APT analysis](image)

**Figure 4.** Concentration profiles for alloying elements computed across a grain boundary in UFG 316 steel processed at 430°C thanks to APT analysis

### 4. Conclusion

It is shown that HPT of stainless steel 316 at room temperature and at 430°C leads to formation of UFG structures in the alloys, which are characterized by different grain size, defect density and phase composition. UFG steel produced at room temperature having grain size about 60 nm demonstrated strength which is in a good agreement with Hall–Petch relation for the austenite stainless steel. Apart from that, the UFG steel produced at 430°C exhibited the strength which significantly exceeds the value predicted for the given grain size (110 nm) by the Hall-Petch dependence. This extra-strength is suggested to be due to fine nanostructural features induced by SPD like segregations/clusters of alloying elements.

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### References

[1] Hall E O 1951 *Proc. Phys. Soc. London* **64B** 747–753
[2] Petch N J 1953 *J. Iron Steel Inst.* **174** 25–28
[3] Pande C and Cooper K 2009 *Progr. Mater. Sci.* **54** 689 – 706
[4] Louchet F, Weiss J and Richeton T 2006 *Phys. Rev. Lett.* **97** 075504(1–4)
[5] Valiev R Z, Islamgaliev R K and Alexandrov I V 2000 *Progr. Mater. Sci.* **45** 103–189
[6] Valiev R, Enikeev N and Langdon T 2011 *Kovové Mater.* **49** 1–9
[7] Valiev R, Enikeev N, Murashkin M, Kazykhanov V and Sauvage X 2010 Scripta Mater. 63 949–952
[8] Lutterotti L, Matthies S and Wenk H R 1999 Proceeding of the Twelfth International Conference on Textures of Materials (ICOTOM-12) 1 1599
[9] Shakhova Y, Yanushkevich Z and Belyakov A 2012 Rus. Metall. (Metally) 38–45
[10] Etienne A 2009 Etude des effets d’irradiations et de la nanostructuration dans des aciers austénitiques inoxydables Ph.D. thesis Université de Rouen
[11] Radiguet B, Etienne A, Pareige P, Sauvage X and Valiev R 2008 J. Mater. Sci 43 7338–7343