Superstrength of nanostructured alloys produced by SPD processing

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Abstract. The metals and alloys subjected to severe plastic deformation (SPD) can possess not only ultrafine-grained structure but also specific nanostructural features, such as non-equilibrium grain boundaries, nanotwins, grain boundary segregations and nano particles. The present work considers the role some of these features in exhibition of high strength of nanostructured metals and alloys. In particular, it is demonstrated that the presence of grain boundary segregations and non-equilibrium boundaries can result in yield stress values that considerably exceed those predicted from the Hall-Petch relation for the given materials.

1. Introduction

Nanostructuring is the new and promising way to enhance the properties of metals and alloys for advanced structural and functional application [1]. To date, it is well established that bulk nanostructured materials (BNM) can be produced successfully via grain refinement using severe plastic deformation (SPD), i.e. heavy straining under high imposed pressure [2]. SPD processing is an attractive procedure for many advanced applications, as it allows enhancing significantly properties of commonly used metals and alloys. Since the pioneering work on producing UFG materials by SPD processing [3], two SPD techniques have attracted close attention and have lately experienced further development [2]. These techniques are high-pressure torsion (HPT) and equal-channel angular pressing (ECAP). For the last 10-15 years there appeared a wide diversity of new SPD techniques: for example, accumulative roll bonding (ARB), multi-axial forging, twist extrusion and others (see for example [2] for a more comprehensive review). Nevertheless, processing by HPT and ECAP has remained the most popular approach and recently this has acquired a new impulse for development through the modification of conventional die-sets and demonstrations that new opportunities are now available for involving these procedures in industrial processing, e.g. using ECAP with parallel channels and continuous ECAP-Conform [4].

Grain refinement is well known to result in strength enhancement of metals and alloys, with the experimental relation between yield strength $\sigma_y$ and a mean grain size $d$ described by the Hall–Petch relationship: $\sigma_y = \sigma_{y0} + k_y d^{1/2}$, where $\sigma_{y0}$ and $k_y$ are material’s constants [5,6]. However, recently the

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results were reported that ultrafine-grained (UFG) alloys produced by SPD processing can exhibit a considerably higher strength than the Hall–Petch relationship predicts for the range of ultrafine grains [7-9]. The nature of such superstrength may be associated with another nanostructural features (dislocation substructures, nanophase particles and segregations, nanotwins et al) which could be observed in the SPD-processed metals and alloys [7,9].

In recent years in our laboratory in close collaboration with colleagues and partners there was performed a number of investigations of unusual mechanical performance of SPD-processed Al and Ti alloys as well as in several steels [7-11]. The aim of present study is to consider and investigate the manifestation and nature of high strength observed in these nanostructured materials.

As it is known for SPD processing by ECAP and HPT it is typical to form ultrafine-grained structures with mean grain sizes within the submicrometer range so that, typically, the grain sizes are ~100-500 nm [4]. However, in the process of SPD the formation of other nanostructural elements takes place as well like dislocation substructures, twins, grain boundary segregations and precipitations. They may also significantly influence the materials’ properties after processing [9, 12-15].

An example of remarkable contribution of nanostructured elements formed in the UFG materials processed by SPD to the change of yield stress and flow stress was recently studied in detail for the case of UFG Al alloys [7,8,13]. Namely, 1570 alloy (Al, 5.7 Mg, 0.32 Sc, 0.4 Mn, wt.%) and 6061 alloy of Al-Mg-Si system were nanostructured by HPT-processing, no subsequent heat-treatment was applied.

![Figure 1. A dark-field TEM image of the UFG 1570 alloy with a corresponding SAED.](image)

2. Results and Discussion

TEM observations showed that the HPT processing of the alloys leads to complete refinement of their initial coarse-grained structure. The dark field image presented in Fig. 1 illustrates the homogeneous UFG structure formed in the HPT 1570 alloy with the mean grain size of 97±2.0 nm. The SAED pattern (Fig.1 inset) testifies that the ultrafine structures are characterized predominantly by high-angle grain boundaries. The 6061 alloy demonstrated similar microstructure features [13].

Table 1 displays the mechanical tests data for the 1570 and 6061 alloys in coarse-grained (CG) and HPT-processed states. These data demonstrate that UFG 6061 and 1570 alloys exhibit significantly increased strength as compared to CG states.

One can analyze the obtained data thanks to a Hall–Petch plot where the yield stress ($\sigma_{0.2}$) is plotted against the inverse square root of the grain size ($d^{-1/2}$) (Fig. 2). For comparison, data reported by Tsuji for a UFG 1100 Al alloy obtained by ARB [16] and data reported by Furukawa et al for the UFG Al-3%Mg alloy obtained by ECAP [17] were also added on the plot. The data for 1100 Al alloy highlight
the Hall-Petch slope resulting from the SPD-induced refinement while the Al–3%Mg alloy exhibits the additional contribution of solid solution hardening.

Table 1. Mechanical properties of the 1570 and 6061 alloys in UFG and CG states

| Alloy   | CG 1570 (solid solved) | UFG 1570 (solid solved+quenched) | CG 6061 | UFG 6061 |
|---------|-------------------------|----------------------------------|---------|----------|
| $\sigma_{0.2}$ (MPa) | 190 | 905 | 150 | 660 |
| UTS (MPa) | 376 | 950 | 275 | 690 |

From the presented data it may be seen that the $\sigma_{0.2}$ values for 6061 and, especially, 1570 alloy are notably higher than expected from an interpolation of the AA1100 or the Al–3%Mg Hall-Petch slopes.

Since the solid solution strengthening component is not a function of the grain size, one can suppose that this manifestation of superior strength exceeding Hall-Petch predictions may result from specific microstructure features of the HPT-processed alloys. The atom probe tomography technique was applied to investigate the distribution of solute elements in the alloys processed by HPT.

Fig. 3 shows some data collected on the UFG 1570 alloy [7]. The 3D reconstructed volume (Fig. 3a) clearly exhibits a planar segregation of Mg. Since Al atomic planes are resolved on the right part while they disappear on the left, it is thought that there is a significant disorientation between these two regions and that Mg atoms have segregated along a grain boundary. Such segregations give rise to a significant decrease of Mg in solid solution (from 7.0 ± 0.2 down to 6.4 ± 0.2 at.%), also confirmed by X-ray diffraction data [10]. The composition profile computed across the boundary (Fig. 3b) reveals that the local concentration is up to 30 at.% Mg within a layer of about 6 nm width. Recently, we observed a very similar behavior of segregations at grain boundaries also in a number of Ti alloys as well as low-carbon steel subjected to SPD and these data correlated with displaying by these materials of very high strength [9, 11].

It is natural to suppose that grain boundary segregations may influence the mechanical behavior of the UFG alloys. It is well established that deformation of UFG materials (with grains larger than 30–50 nm) is mainly associated with intragranular movement of dislocations [12]. Dislocations are generated at grain boundaries and move through a grain to be captured by an opposite boundary. In
this case the rate-controlling mechanism is “dislocation-grain boundary” interaction. Elevated concentration of solutes in grain boundaries can suppress emission of dislocations due to solute drag.

![Figure 3](image)

**Figure 3.** 3D image of an analyzed volume in the UFG 1570 alloy [7]: (a) a planar segregation of Mg (Al atoms are displayed as dots and Mg atoms as bubbles). (311)Al atomic planes on the right of the planar segregation are displayed; (b) concentration profile computed across the segregation.

### 3. Conclusion

The conducted investigations testify to the fact that yield stress of UFG metals and alloys produced by SPD may be considerably higher than it is predicted by Hall-Petch relation for their grain size range. The observed superstrength of these materials is attributed to nanostructural features of their grain boundaries – their non-equilibrium state and segregations. Thus, the problem of detailed investigations of grain boundaries in UFG materials produced by SPD is topical for the achievement of high strength in nanomaterials. Besides, it is important to study the role of intragranular nanostructural elements – nanotwins, nanoparticles, which can lead to additional strengthening of the materials.

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