Materials Science & Engineering A 560 (2013) 413–432

Contents lists available at SciVerse ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

# Microstructure strengthening mechanisms in different equal channel angular pressed aluminum alloys

Marcello Cabibbo*

*Dipartimento di Ingegneria Meccanica e Scienze Matematiche (DIISM), Università Politecnica delle Marche, 60131 Ancona, Italy*

ARTICLE INFO

*Article history:*
Received 4 June 2012
Received in revised form
22 August 2012
Accepted 23 September 2012
Available online 2 October 2012

*Keywords:*
Strengthening mechanisms
Hall–Petch
Yield stress
ECAP
TEM

ABSTRACT

Microstructure characterization and identification of the different nature of boundary formation during severe plastic deformation is the basis for a quantitative analysis of material flow stress. In equal channel angular pressing, the microstructure evolves to form low-angle dislocation boundaries and high-angle boundaries whose origin is different. For this reason, boundary misorientation, size and fraction evolve differently with strain. The Hall–Petch relationship in severe plastic deformed aluminum and aluminum alloys has been extensively discussed by Niels Hansen and others in several published works. It appeared that in such cases, the dislocation boundary strengthening contribution is to be taken into account. This paper deals with further insights into the Hansen's and other authors approach to the Hall–Petch relationship. Present approach is based on a detailed microstructure characterization of the different strengthening contributions in severe plastic deformed aluminum alloys. AA1200, AA3103, AA6000 series, and AA2091 were quantitatively characterized by TEM inspections. The calculated alloys yield strengths were compared to measured tensile yield stresses obtaining a quite satisfactory matching. This, ultimately, confirmed the proposed approach and models. Finally, an experimental value of the hardness-to-yield stress, $H/\sigma_y$, was found for all the studied alloys and discussed accordingly.



## 1. Introduction

In the more than 40 years since the pioneering work on flow stress-grain size relation by Armstrong et al. [1], the interest in the Hall–Petch relation has never diminished. A main reason of such an interest has been its prediction that the strength of a material can be increased by reducing the grain size. The power-law relationship between the yield strength and the metal mean grain size is actually the basis for extensive studies of grained structures down to sub-micrometer scale [2–9]. Theoretical studies of the strength-grain size relation have been followed by numerous experimental studies, which generally have shown that the Hall–Petch relation fits quite well the experimental data [3–6]. These experimental studies have encompassed a large variety of materials (either in an undeformed and deformed state), processing techniques and testing methods. In fact, different authors have published works, using either electron microscopy (TEM) or electron back-scattered diffraction (EBSD) techniques, where the microstructure different contributions successfully met interpretative models of proof-stress determination [10–28].

Moreover, there is a strong theoretical and technological need for determining to what extent the Hall–Petch relation can be extrapolated to very small grained size materials [4–9]. The Hall–Petch relationship is expected to be based on a number of microstructure parameters. It is often derived assuming dislocation pile-up at grain boundaries, which are surrounded by dislocation-free matrix. In fact, the yield stress calculated by Hall–Petch describes the mechanical behavior of a metal through a mechanism of dislocation source activation in a dislocation-free grained matrix. Thence, studies on this issue must encompass polycrystalline materials, strengthened by grain boundaries other than also deformed materials strengthened both by grain boundaries and dislocation boundaries, which typically furthermore subdivide the sub-micrometer structured metallic materials down to a nanostructured level [5–8].

The existence of extended dislocation boundaries and cell boundaries has been known since some 50 years, but it has been only recently realized that the two types of boundaries are indeed characteristic features in polycrystalline metals, deformed under many different conditions over a wide strain range ([9] to cite but one). In contrast to the grain boundaries, cell boundaries are typically short, randomly inclined with a lower mean misorientation angle across. By a general mechanism, the cell boundaries may form by mutual trapping of glide dislocations [9,26]. This process continues with strain, leading to a progressive cell size

* Tel.: +39 0712204728; fax: +39 0712204801.
*E-mail address:* m.cabibbo@univpm.it

0921-5093/$ - see front matter © 2012 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2012.09.086

reduction and cell boundary misorientation rise. This boundary dislocation spacing shrinking on a finer and finer scale has been modeled, among others, by Prinz and Argon [29].

Since strain differences are accommodated by the formation of cell block boundaries, they were termed geometrically necessary boundaries (GNBs). To mark their different origin, cell boundaries were termed incidental dislocation boundaries (IDBs) [30]. The cell boundaries are LABs, that form by trapping of glide dislocations. This means that some LABs at large strain may develop such high misorientation angles that they become HABs (from a microstructure and mechanical point of view behaving as GNBs). In such a case the boundaries may be integrated into the lamellar structure or they may be present as high angle boundaries which interconnect the lamellar boundaries. The HABs are strong barriers to slip as they develop very high dislocation concentration with strain. On the contrary, the lower angle LABs are assumed, to a certain extent, to be penetrable by mobile dislocations. The flow may therefore take place in the channels between the lamellar boundaries with a slip length which is related to the distance between the lamellar boundaries.

The subdivision of crystals and grains during deformation takes place as deformation bands, on a macroscopic scale, and as cell blocks and cells, on a smaller scale. At increasing stress and strain this microstructure boundary subdivision takes place on a finer and finer scale, whose rate strongly depends on the deformation process used. The refinement of the microstructure is accompanied by an increase in the average angle across both cell block boundaries and cell boundaries [31]. In fact, severe plastic deformation (SPD) techniques are known to produce bulk metallic materials with fine-grained structure down to nanometer scale [32]. Among the various SPD techniques, equal channel angular pressing (ECAP) [12,14,18,19,33–39] is able to give grain sizes typically in the range of 400–800 nm. In particular, ECAP is an especially attractive processing method because it allows large bulk samples to be produced, which are free from any residual porosity, and are subjected to small shape changes. The evolution of the microstructure during ECAP, is closely driven by the specific pressing conditions, i.e. by the shearing deformation induced in the material at each pass through the die [31,32]. Plastic deformation of metals occurs as a result of the formation, movement and storage of dislocations. In fact, microstructure evolution during ECAP is directly linked to a complex dislocation evolution into networks and to dislocation recombination and annihilation phenomena. During shearing deformation, the evolution and accumulation of misorientation across both low-angle boundaries (LABs) and high-angle boundaries (HABs) is closely related to the crystallographic accommodation of each crystallite with its neighboring crystallites. In deformation route $B_C$, a rotation of $+90^\circ$ per passes is induced, route C implies a $180^\circ$ billet rotation per pass, and route A does not involve any sample rotation [32,39–46]. Whatever the route used, ECAP has extensively been reported to induce a severe microstructure refinement already at the earliest imposed strain levels [40–43].

In this context, Niels Hansen has extensively developed a modified Hall–Petch relationship intended to properly address and fit the yield strength dependency on both IDB and GNB contributions, which are induced by severe plastic deformation techniques. This relationship takes into account the contribution from cell boundaries (which are LAB, and essentially equivalent to IDBs), and the contribution from grain size (HAB, and equivalent to GNBs) [8]. In the Hansen's modified Hall–Petch relationship, the yield strength is the linear sum of the friction stress term plus the dislocation boundary contribution (LAB/ IDB) and the grain size contribution (HAB/ GNB). This model has been extensively proved to better fit the Hall–Petch relationship in all the cases of deformed, and severely deformed metallic microstructure with sub-micrometer scale grain size [8,25,31,47–56]. The Hansen's modified Hall–Petch relationship was here applied to different aluminum alloys subjected to severe plastic deformation by equal channel angular pressing (ECAP).

Other different approaches to model the proof stress, from the microstructure strengthening contributions, were also proposed [10–14,17,21–24,58]. Among these, Dobosz et al. [10,11], Kurzydłowski and Bucki [24], and Valiev et al. [14,58] studied the combined effect of ultrafine structure and deformation-induced segregation of solute elements at grain boundary. In these and other studies [10,11,14,15,17,19,20–22,24,25,33,35,51–58] the combination of grain boundary and Orowan-type strengthening contributions were addressed. It was exhaustively documented that dislocation sliding is the major deformation mechanism that is ultimately responsible for the most part of the material proof stress. In particular, in [11] and [24] an analytical estimation of the grain strengthening contribution was based on the concept of grain size diversity, which depends on a dimensionless coefficient of variation of grain size distribution. This approach has many similarities and analogies with the phase-mixture model developed by Estrin et al. [59,60] to explain the mechanical properties of nanocrystalline alloys. In different published works [12–14,17,21,61–69] in addition to dislocation and particle strengthening, a contribution from solid solution (solute elements) was recognized and properly addressed.

In the present study, the modified Hall–Petch formulation given by Hansen was discussed and further extended to include, other than solid solution strengthening and secondary phase particles, also the very low-angle boundaries, typically showing Moiré fringes and not detected by conventional electron backscattered diffraction (EBSD) maps. To the author's knowledge the introduction of the Moiré boundary strengthening can be considered as a new and never included term in the strengthening mechanism modeling of severely deformed metallic materials. Texture contribution and other microstructure issues which are likely to contribute to the alloys strengthening were also considered and discussed. In particular, according to several published works ([70–73], to cite but few), the texture evolution and its strengthening contribution was addressed considering the Taylor factor texture-induced changes during severe plastic deformation.

## 2. Experimental details and method

### 2.1. Materials and experimental details

Chemical composition of the studied alloys is compiled in Table 1, where the metallurgical initial status and the ECAP deformation routes are also specified. Materials were selected to include all possible strengthening contributions to yield stress in aluminum alloys. Selection of these alloys, and ECAP routes, were also made on the basis of previous works that this author carried out in the past last decade. The selection made possible to encompass all the possible microstructure strengthening mechanisms in severely plastic deformed aluminum alloys. Thus, commercially pure AA1200 was selected to study the sole role of dislocation boundary strengthening during severe plastic deformation. For this purpose, three different ECAP-routes were used, route A (no sample rotation between passes), route C ($180^\circ$ sample rotation per pass), and route $B_C$ ($+90^\circ$ sample rotation per pass). The three different routes of ECAP deformation do correspond to different dislocation boundary evolution, which in turn involves different dislocation strengthening evolution with strain [41–45,74,75]. AA3103 was selected because of the presence of non-shearable stable $Al_6Mn$ particles and for the presence of iron-rich intermetallic phase. Two different Al–Mg–Si alloys were selected: an AA6082 and an AA6106Zr, and AA6106ScZr [76,77]. These alloys were pressed after a T8 (solution treated 3 h at