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## Length scales and scaling laws for dislocation cells developed during monotonic deformation of (001) nickel single crystal



### Abdelali Oudriss<sup>\*</sup>, Xavier Feaugas

LaSIE, CNRS UMR 7356, Université de La Rochelle, av. Michel Crépeau, 17000 La Rochelle, France

#### ARTICLE INFO

Article history: Received 18 February 2015 Received in revised form 13 November 2015 Available online 2 December 2015

Keywords: A. Dislocations B. Crystal plasticity C. Mechanical testing C. Electron microscopy Nickel

#### ABSTRACT

An extensive collection of experimental data was gathered to improve the well-known self-organization process of dislocation resulting from a tensile strain. Statistical analyses by Transmission Electronic Microcopy were performed over a large area in order to obtain additional insights into the different relationships between the structural parameters and the flow stress. A scaling behaviour was established for the size of quasi equiaxed dislocation cells over a large plastic strain range. Additionally, the dislocation organization reveals a variety of scaling laws relative to shear stress, cell size, dislocation wall thickness and dislocation densities in dislocation walls and cells. The physical bases of these laws were demonstrated and their consequences on plastic strain behaviour are discussed.

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#### 1. Introduction

Typical strengthening behaviour of single and/or polycrystalline materials is mainly the result of spatio-temporal fluctuation of dislocation mobility and storage. It is well established that dislocation distribution in f.c.c. metals and alloys becomes significantly heterogeneous during strengthening. The general feature, that dislocation patterns are commonly heterogeneous, affects the stress-strain state during strengthening. This has been discussed extensively and seems to be a key feature for modelling mechanical behaviour in different conditions (different stress paths, temperatures and strain rates). Several groups have explored a variety of configurations to examine the possible impact of dislocation distribution on mechanical behaviour. For example, strain-temperature dependence of dislocation pattern has been rationalized in terms of a map, which depends on metals and alloys (Steeds, 1966; Anongba et al., 1993; Landau et al., 2011, 2012). Other groups have focused their attention on the impact of grain orientation and/or single crystal orientation on the dislocation structures obtained under tensile and cyclic loading (Kawasaki, 1979, Kawasaki and Takeuchi, 1980; Hansen and Kuhlmann-Wilsdorf, 1986; Feaugas, 1999; Buque et al., 2001; Holste, 2004; Huang and Winther, 2007; Feaugas and Haddou, 2007; Girardin et al., 2015; Jiang et al., 2015). For cyclic loading, a unified view of dislocation organization was proposed by Pedersen (1990) and Feaugas and Pilvin (2009) for copper, aluminium, nickel and austenitic steel. Additionally, the consequence of stacking fault energy and solute contents on dislocation pattern is well documented (see for example Mader, et al., 1963; Hong and Laird, 1990). The impact of strain path on dislocation organization under severe strain (Peeters et al., 2001; Rauch and Thuillier, 1993; Rauch et al., 2002; Gardey et al., 2005; Sakharova and Fernandes, 2006) and for multiaxial loading (Clavel and Feaugas, 1996; Bocher et al., 2001) have also extensively studied. The impact of strain rate is not so clear for f.c.c. metals (Chao and Varma, 1990; Shume et al., 1989; Kuhlmann-Wilsdorf, 1999). More recently, investigations into the impact of sample size and grain size on dislocation distribution have been motivated by technological miniaturization (Sumino and

http://dx.doi.org/10.1016/j.jplas.2015.11.003 0749-6419/© 2015 Elsevier Ltd. All rights reserved.

<sup>\*</sup> Corresponding author. E-mail address: abdelali.oudriss@univ-lr.fr (A. Oudriss).

Yamamoto, 1963; Feaugas and Haddou, 2007; Norfleet et al., 2008; Keller et al., 2010, 2011; El-Awady et al., 2013). It seems that softening behaviour occurs near the surface (Fourie, 1967, 1970, 1972; Mughrabi, 1970, 1971) for small grain size (Feaugas and Haddou, 2003, 2007) or for small samples (Norfleet et al., 2008; Keller et al., 2010, 2011) in relation to less established dislocation structures than the one obtained in the bulk of large strengthening samples. The common feature of these studies is an attempt to correlate dislocation distribution with applied macroscopic stress and hardening rate. Additionally, a substantial effort to establish a relationship between hardening rate and the formation and evolution of dislocation guasi-cell structures in hardening stage II might provide a better understanding of the consequences of long-range stress on workhardening rate. Combining TEM observations and loading-unloading tensile tests or cyclic tests, Feaugas et al. (2003), Feaugas and Haddou (2007) and Feaugas (1999) showed that dislocation distributions affect the long-range internal stresses and the back stress. A broadly similar result was obtained many years ago using TEM observations of the curvature of dislocation segments pinned by neutron irradiation (Mughrabi, 1975, 1983, 1987; Mecke and Messerchmidt, 1979). More recent results obtained with XRD (Ungar et al., 1984, 1986; Straub et al., 1996; Mughrabi et al., 1986; Mughrabi and Ungar, 2002; Jakobsen et al., 2006), TEM CBED (Straub et al., 1996; Kassner et al., 2000; Legros et al., 2008) and X-ray microbeam (Levine et al., 2006, 2011) support the assumption that heterogeneous dislocation distribution impacts the long-range internal stresses at the micro-scale. Thus an attempt to define a classification of the different dislocation distributions seems to be a long-standing key motivation (see for example Steeds, 1966). Extensive Transmission Electronic Microscopy (TEM) observations have shown the variety of dislocation structures in face-centred cubic metals and alloys (Basinski and Basinski, 1979; Feaugas, 1999; Feaugas and Haddou, 2007; Huang and Winther, 2007; Mughrabi, 1975; Steeds, 1966; Sumino et al., 1963). In single crystals and/or polycrystals oriented for single-slip with a face-centred cubic (f.c., structure, dislocation patterning appears at the transition between stage I and stage II with the development of relatively planar boundaries (near the {111} plane). This microstructure corresponds to extended dislocation walls which subdivide the microstructure into blocks characterized by a strong black and white contrast difference. This TEM contrast is an illustration of an excess of dislocations of one sign in the walls. These dislocation organizations seem to result from the formation of a grid structure related to a primary and/or conjugate active slip in the early stages of plastic strain (Feaugas, 1999; Huang and Winther, 2007). Previous work on f.c.c. alloys defined these dislocation organizations as Geometrically Necessary Boundaries (GNBs) due to their accommodation of different crystal orientations that change on each side of a boundary (Feaugas and Haddou, 2007; Hansen and Jensen, 1999; Huang and Winther, 2007; Kuhlmann-Wilsdorf and Hansen, 1991). Furthermore, relatively equiaxed cells are generally observed between two GNBs. The boundaries of equiaxed cells i.e. Incidental Dislocation Boundaries (IDBs) are formed by stochastically trapping glide dislocations that arise when more than one slip system is active. A quite different situation is observed in multiple-slip oriented single crystals and polycrystals with high symmetry ((100) orientation). Indeed, the equiaxed cell structures with incidental dislocation boundaries (IDBs) readily emerge as the strain increases over the entire range of deformation up to the point of tensile fracture (Ambrosi and Schwink, 1978; Koneva et al., 2008: Lekbir et al., 2013).

Dislocation structures and distributions in f.c.c. materials have been the subject of intensive research for several decades, but these studies were generally more qualitative than quantitative. In addition, crystal plasticity models have been developed over a number of years to reflect the increasing heterogeneity of plastic strain and especially the different populations of dislocation densities and their impact on strain hardening. It is impossible to be exhaustive on this subject, but it is still important to highlight some features. Several approaches have been developed which consider the impact on mechanical behaviour of one population of dislocations (Franz et al., 2013; Gérard et al., 2013; Khan et al., 2015; Lee et al., 2010), or several populations of dislocations (Barlat et al., 2002; Bertin et al., 2013; Engels et al., 2012; Lee et al., 2013; Li et al., 2014; Ma and Roters, 2004; Ma et al., 2006; Shanthraj and Zikry, 2011; Silbermann et al., 2014; Viatkina et al., 2007b) and/or which take into account the heterogeneity of distribution of dislocation density on long-range internal stresses (Brahme et al., 2011; Feaugas and Pilvin, 2009; Viatkina et al., 2007a). Moreover, it remains difficult to have a critical approach to these models without statistically analyzing experimental data. An alternative to this problem was proposed using discrete dislocation dynamics (DDD). This approach shows a well-defined relationship between different length scales (crystal and grain size) and dislocation microstructure for the early stages of deformation (see the recent intensive calculations performed by Hussein et al., 2015). In contrast, dislocation pattern and its consequences on stress are not currently well documented in terms of a DDD calculation (Depres et al., 2004; Madec et al., 2002; Khan et al., 2004).

Moreover, an apparent conflict seems to be exist between the impact of dislocation patterns on mechanical behaviour and a direct relation between average dislocation density and macroscopic stress. It was suggested by some authors that the latter can be partially resolved using similitude relationships (or scaling laws) derived from an extensive compilation of TEM data. However, from an experimental point of view, similitude relationships are generally deduced from data compilations obtained in the literature without distinguishing between IDBs and GNBs (see for example Blum, 1993; Raj and Pharr, 1986; Sauzay and Kubin, 2011). Consequently, there is a strong possibility that the scaling laws derived from different measurements are partially wrong or are modified by errors that are the result of taking data from different sources. Essentially, two research groups performed an extensive analysis of dislocation density and distribution in polycrystalline nickel for a large range of grain size and plastic strain (Feaugas and Haddou, 2007; Huang and Winther, 2007; Hughes and Hansen, 2000; Luo et al., 2012; Zhang et al., 2008). Scaling behaviour is well established for some microstructural features (wall thickness, cell size, spacing between GNBs, misorientation between walls etc.) but the relationships between a few key structural parameters and flow stress are debatable for polycrystalline material. The equivalent stress—strain parameters that are generally used are only an average view and do not integrate the effect of grain orientation and long-range internal stresses, which

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