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# Microstructure characterization of 316L deformed at high strain rates using EBSD

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#### ABSTRACT

Specimens from split Hopkinson pressure bar experiments, at strain rates between ~1000–9000 s<sup>-1</sup> at room temperature and 500 °C, have been studied using electron backscatter diffraction. No significant differences in the microstructures were observed at different strain rates, but were observed for different strains and temperatures. Size distribution for subgrains with boundary misorientations >2° can be described as a bimodal lognormal area distribution. The distributions were found to change due to deformation. Part of the distribution describing the large subgrains decreased while the distribution for the small subgrains increased. This is in accordance with deformation being heterogeneous and successively spreading into the undeformed part of individual grains. The variation of the average size for the small subgrain distribution varies with strain but not with strain rate in the tested interval. The mean free distance for dislocation slip, interpreted here as the average size of the distribution of small subgrains, displays a variation with plastic strain which is in accordance with the different strains of the small subgrain in the linear hardening range is accurately calculated using the variation of the small subgrain size with strain.

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#### 1. Introduction

Understanding the relation between the mechanical properties and the microstructure is a cornerstone for most of the process steps in the metal manufacturing industry [1–3]. One of the more challenging areas is to understand the microstructural development for high velocity and high temperature processes such as metal cutting and rolling. For machining simulations this knowledge is important to be able to model the mechanical behavior. During machining the strain rate is in the range of  $10^3$  to  $10^6$  s<sup>-1</sup> and the homologous temperature is in the range 0.16–0.9 [4]. To simulate high strain rate processes, a commonly used approach is split Hopkinson pressure bar (SHPB) tests [5]. The development in the research field of modeling plastic deformation has been intimately related to development of analytic tools such as X-ray diffraction, scanning and transmission electron microscopy (SEM and TEM, respectively), and electron backscatter diffraction (EBSD) [6–12].

When modeling the flow stress,  $\sigma$ , during plastic deformation by dislocation slip the following equation is commonly used [13–16]:

$$\sigma = \sigma_{ath} + \sigma_{th} + m\alpha Gb\sqrt{\rho} \tag{1}$$

where  $\sigma_{ath}$  is the contribution of all athermal hardening mechanisms

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(except deformation hardening) and  $\sigma_{th}$  is all contributions from thermally activated deformation mechanisms (except cross slip), e.g. bypassing solute atoms [16], to the flow stress, *m* is the Taylor factor,  $\alpha$  is a proportionality factor, *G* is the shear modulus, *b* is Burgers vector and  $\rho$  is the dislocation density.

According to Bergström [15], the variation of  $\rho$  with plastic strain,  $\varepsilon_{pl}$  can be described by:

$$\frac{d\rho}{d\varepsilon_{pl}} = \frac{m}{bL} - \Omega\rho \tag{2}$$

where *L* is the mean free distance for dislocation slip and  $\Omega$  is a parameter for remobilization and/or annihilation of dislocations.  $\Omega$  increases with increasing temperature and decreasing strain rate. The first term in Eq. (2) describes generation of dislocations and the second the recovery of dislocations. Recovery involves cross slip and dislocation climb. The inverted value of *L* is a function of  $\rho$  and grain size  $d_g$  [14]:

$$\frac{1}{L} = f\left(\sqrt{\rho}, \frac{1}{d_g}\right) \tag{3}$$

*L* is proportional to the grain diameter at low values of  $\rho$ . At higher levels of  $\rho$ , *L* is proportional to  $1/\sqrt{\rho}$ , and thus to the subgrain size,  $d_{sub}$ .

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Bergström also derived a relationship for the strain dependence of *L* in single-phase metals [15]:

$$L(\varepsilon) = L_f + (L_i - L_f) \cdot e^{-\kappa \varepsilon}$$
(4)

where  $L_i$  and  $L_f$  is the initial and final mean free distance for dislocation slip respectively, and  $\kappa$  is a rate constant determining the rate at which  $L(\varepsilon)$  goes from  $L_i$  to  $L_f$ .

Bergström, Granbom and Sterkenburg [17] later proposed a dislocation based theory for deformation hardening behavior of DP steels. This theory states that the plastic deformation process in the ferritic grains is inhomogeneous, starting near the grain boundaries and propagating towards the center of the grains with increasing strain. Furthermore, the martensitic phase is only assumed to deform elastically while the total fraction of ferrite,  $f_0$ , is divided into one active fraction,  $f_{active}$ , and one inactive fraction,  $f_{in-active}$ . The active fraction deforms both elastically and plastically whilst the inactive fraction only deforms elastically. Initially  $f_{active}$  is much smaller than  $f_o$  but increases towards  $f_o$  with increasing strain. A relationship between the active fraction of ferrite and the strain was proposed:

$$f_{active}(\varepsilon) = f_0 + (f_i - f_0) \cdot e^{-r\varepsilon}$$
(5)

where  $f_i$  is the initial active volume fraction of ferrite taking part in the deformation process and r is a material parameter which controls the formation rate of  $f_{active}$ .

The grain size distribution is also important for the mechanical properties of polycrystalline materials [18]. Assuming that the grain areas, A, in a planar section are lognormal distributed [19–21], the distribution function (DF) y, and the cumulative distribution function (CDF) Y, are:

$$y(\ln (A)) = \frac{c}{s\sqrt{2\pi}} \exp\left[-\frac{\left(\ln (A) - \ln \left(\overline{A}_g\right)\right)^2}{2s_g^2}\right]$$
(6)

$$Y(\ln(A)) = \frac{c}{2} \left[ 1 + erf\left(\frac{\ln(A) - \ln\left(\overline{A}_g\right)}{s_g\sqrt{2}}\right) \right]$$
(7)

where erf(x) is the error function, c is a fitting parameter (equals one for the normalized distribution),  $s_g^2$  the variance of  $\ln(A)$  and  $\bar{A}_g$  the geometric mean grain area. The arithmetic mean,  $\bar{A}$ , and variance,  $s^2$ , for the grain area are then given by:

$$\overline{A} = e^{\ln\left(\overline{A}_g\right) + s_g^2/2} \tag{8}$$

$$s^{2} = \left(e^{s_{g}^{2}} - 1\right)e^{2\ln\left(\bar{A}_{g}\right) + s_{g}^{2}}$$
(9)

By using high angle boundaries (HABs) during grain detection  $d_g$  can be determined from the CDFs and by using low angle grain boundaries (LABs)  $d_{sub}$  and L can be determined.

In this article, we focus on the microstructure development dependence on strain and strain rate and its relation to mechanical properties. The study covers samples deformed during compression tests conducted at room temperature (RT; in this study measured to 22 °C) and 500 °C at different strain rates in the range 1000–9000 s<sup>-1</sup> performed by Wedberg and Lindgren [22]. The microstructure of stainless steel 316L samples deformed using SHPB test was characterized and compared to the undeformed microstructure using EBSD technique. The characterization includes determination of values for  $d_g$  and  $d_{sub}$  using standard methods and also by analyzing the size distributions and calculation of the Taylor factor. The aim of this study is to improve the understanding of the deformation mechanisms during high strain rate processes and to aid in the modeling of the mechanical properties by determining valuable material parameters and relating these to the stressstrain curves.

#### 2. Materials and Experimental Methods

#### 2.1. SHPB Experiment

A detailed description of the SHPB experiments is given in [22] and the equipment is described in [23]. The starting material was a solution annealed ASTM 316L alloy supplied by Sandvik AB. The chemical composition are provided in Table 1. Each sample had a cylindrical form with the dimensions adjusted to give the desired strain and strain rate.

ASTM 316L contains a small fraction of ferrite and the original steel contained about 0.25% as determined by magnetic balance measurements. Samples deformed at RT contained some extra deformation martensite and a level of around 1-1.2% was measured. At higher temperatures the ferritic levels are the same as for the annealed starting material (around 0.25%) [22].

During the experiment the sample was heated using a furnace located beside the bars. The sample was extracted from the furnace a couple of microseconds before the stroke. During deformation, part of the mechanical energy was transferred to heat causing the temperature of the sample to further increase. After the stroke the sample was air cooled.

In order to improve the readability of the paper a shorthand notation is used for the experimental parameters in the SHPB experiment, with the symbol  $S(\varepsilon, r, T)$ , where S stands for sample,  $\varepsilon$  for true strain, r for true strain rate ( $\dot{\varepsilon}$ ) and T for the temperature in degrees Celsius before the stroke. A summary of the parameters together with the shorthand notation is given in Table 2.

The  $T_{max}$  in Table 2 was calculated by assuming adiabatic heating conditions using the following relation:

$$\frac{dT}{d\varepsilon_{pl}} = \frac{\eta}{D \cdot C_p} \cdot \sigma \to T_{max} = T_{in} + \frac{\eta}{D} \int_0^\varepsilon \frac{\sigma}{C_p} d\varepsilon_{pl}$$
(10)

where D is the density,  $C_p$  the specific heat,  $\varepsilon_{pl}$  the plastic strain,  $\eta$  the heat transformation efficiency (i.e. the part of the mechanical energy which was transferred to heat and this value was set to 0.9), and  $\sigma$  the true stress.  $C_p$  was calculated using ThermoCalc with the TCFE5 database down to 400 °C.  $C_p$  data for lower temperatures were obtained by extrapolation using a second degree polynomial which was fitted to the high temperature data. The integration was carried out over the compression curves presented below. The temperature increase is highest at low temperatures.

#### 2.2. Sample Preparation and EBSD Data Acquisition

The EBSD samples were prepared in such a way that a longitudinal section through the center of the sample, in the load direction, was mechanically grinded and oxide polished. The microstructure was characterized using a Zeiss Ultra 55 FEG-SEM equipped with an Oxford Instrument HKL Nordlys F EBSD detector. The EBSD data was acquired using the Flamenco software included in the Channel 5 software or the AZtec software, both from Oxford Instruments. The SEM- and EBSD settings, see Table 3, were all optimized concerning spatial resolution and signal strength for examination of heavily deformed microstructures.

#### 2.3. Analytical Procedures for Data Cleaning, Boundary Definitions and Size Measurements

For most post-processing of the EBSD data, generation of maps and an inverse pole figure, the programs Tango and Mambo included in

 Table 1

 Chemical composition of 316L (weight-%).

С	Ν	Si	Mn	Р	S	Cr	Ni	Мо	V
0.015	0.055	0.18	1.52	0.026	0.028	16.58	9.82	2.17	0.050

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