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# Three-dimensional geometrical and topological characteristics of grains in conventional and grain boundary engineered 316L stainless steel

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ABSTRACT

The three-dimensional microstructures of a conventional 316L stainless steel and the same material after grain boundary (GB) engineering have been measured by serial sectioning coupled with electron backscatter diffraction mapping. While it is well known that GB engineered materials are differentiated from conventional materials because of the proportion of coincidence site lattice boundaries, the size of their twin-related domains, and their reduced random boundary connectivity, this work provides a quantitative comparison of the geometrical and topological characteristics of grains in 316L stainless steel before and after GB engineering. Specifically, the numbers of grain faces, triple lines, and quadruple unions per grain have been measured and compared. In addition, the distributions of grain sizes, surface areas, and grain boundary areas have been measured and compared. The results show that, in many ways, the three-dimensional geometrical and topological characteristics of the grains in the GB engineered and conventional materials are similar. In both materials, the distributions of the geometrical parameters are well represented by a log-normal distribution. Comparatively, the GB engineered microstructure has grains that, on average, have both fewer faces and higher (specific) surface areas that deviate more from an ideal equiaxed shape, but there are several eccentric or non-compact shaped grains that have a huge number of faces and extremely large surface area in the GB engineered material. All of these characteristics are likely to be a result of the increased number of twins in the GB engineered microstructure. These eccentric grains would have a positive influence on increasing the resistance to intergranular degradation.

## 1. Introduction

Since the early 1980s, a promising method, known as ‘grain boundary engineering’ (GBE) (Kumar et al., 2000; Liu et al., 2014b; Michiuchi et al., 2006; Randle, 2004; Tokita et al., 2017; Watanabe, 1984), has been used to improve grain boundary (GB) related properties of some not only structural but also functional materials (Watanabe, 2011), particularly face-centered-cubic (FCC) materials with low stacking fault energy. Among these properties are intergranular corrosion (Hu et al., 2011; Kobayashi et al., 2016; Xia et al., 2011), intergranular stress corrosion cracking (Gertsman and Bruemmer, 2001; Palumbo et al., 1991; Tan et al., 2013; Telang et al., 2016; West and Was, 2009), and creep (Alexandreanu et al., 2003; Alexandreanu and Was, 2006). The improved properties are thought to emerge because the GB engineering process increases the concentration of coincidence site lattice (CSL) boundaries with high coincidence. Some low-$\Sigma$ CSL boundaries (where $\Sigma \leq 29$ and measures the inverse coincidence) have shown better performance than random boundaries in FCC materials (Hu et al., 2011; Kobayashi et al., 2016; Palumbo et al., 1991; Tan et al., 2013; Telang et al., 2016; Xia et al., 2011). Numerous studies (Cao et al., 2017; Kumar et al., 2000; Liu et al., 2014a, 2013; Michiuchi et al., 2006; Randle, 2004; Xia et al., 2008) have investigated the GB character distribution (GBCD) of materials before and after GB engineering, finding that the prominent characteristics of GB engineered microstructures include a high proportion of low-$\Sigma$ CSL boundaries, large twin-related domains, and a disrupted random boundary network. However, these studies have mostly been based on comparisons of two-dimensional (2D) microstructural data from materials before and after GB engineering. One recent study has compared twin related domains in the three-dimensional (3D) microstructures of conventional and GB engineered copper (Lind et al., 2016). However, a comparison of the geometrical characteristics of

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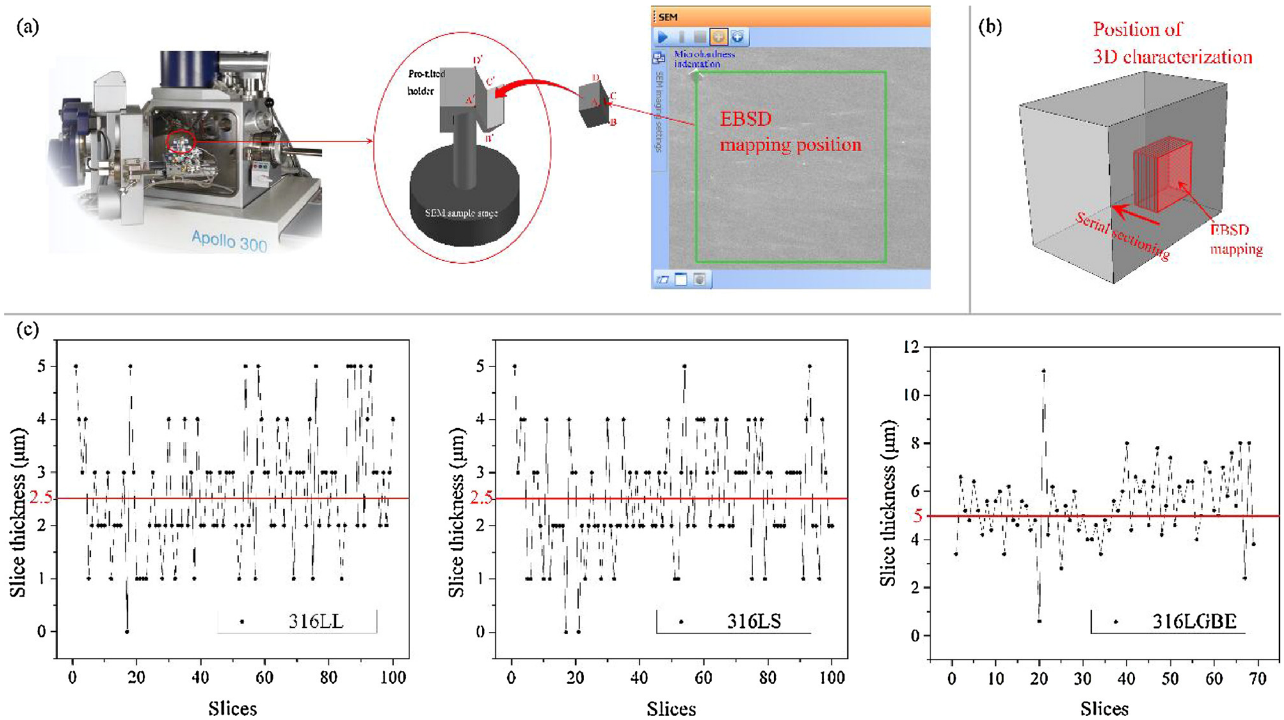


**Fig. 1.** (a) Schematic drawings of the positioning method used for the EBSD data collection, (b) the serial-sectioning used for 3D characterization, and (c) the slice thickness distributions of the three samples.

grains in conventional and GB engineered microstructures has not yet been extensively reported. In the current paper, we compare 3D data from a 316L stainless steel that has been GB engineered with conventional material that has not been GB engineered. The 3D data allows us to compare the microstructural parameters that cannot be derived from 2D observations including grain shapes and the numbers of grain faces, triple lines, and quadruple points. It is well-known grain boundaries have great contribution to the behavior of materials. Furthermore, the properties of materials are closely correlated with the triple junctions and quadruple junctions, such as coordination deformation between neighboring grains during deformation, and intergranular damage route at triple lines and quadruple points (Hu et al., 2011).

The most common methods of obtaining 3D orientation maps of microstructures are serial sectioning coupled with electron backscatter diffraction (EBSD) mapping (Kelly et al., 2016; Lewis et al., 2006; Rowenhorst et al., 2010; Saylor et al., 2002; Uchic et al., 2006) and X-ray diffraction techniques including high energy diffraction microscopy (HEDM) (Hefferan et al., 2012; Lin et al., 2015), differential contrast tomography (DCT) (King et al., 2008), differential aperture x-ray microscopy (DAXM) (Larson et al., 2002). Serial sectioning can be accomplished either with a focused ion beam (FIB) (Kelly et al., 2016; Saylor et al., 2002; Uchic et al., 2006) or by subtractive polishing (Alkemper and Voorhees, 2001; Lewis et al., 2006; Rowenhorst et al., 2010; Spowart, 2006). The FIB method is attractive because the process can be automated so that in a few days, a large number of grains can be characterized. However, the disadvantage is that the field of view is rather small. Even using newly available Xe-ion plasma FIBs, it is not currently possible to examine volumes with lateral dimensions much greater than 200 μm (Kelly et al., 2016). While serial sectioning by subtractive polishing is more difficult to implement, the field of view is not restricted and this makes it possible to study materials with larger grain sizes that are used in structural applications (Rowenhorst et al., 2010).

In 3D microstructures, one can identify four geometrical and topological characteristics of grains that cannot be completely quantified in 2D microstructures: the grain shape, the grain boundaries (faces), the grain edges (triple lines between three grains), and the grain vertices (quadruple points between four grains) (Bhandari et al., 2007; Groeber et al., 2008; Ullah et al., 2014). Furthermore, the connectivity of grain boundaries, including chains of twins that are important in GB engineered materials (Rohrer and Miller, 2010; Xia et al., 2009), can be identified from 3D data, but they cannot be determined from 2D data. In the current study, we will quantify and compare the geometrical and topological characteristics of the grains in GB engineered and conventional 316L stainless steels.

Past 3D studies of polycrystalline materials have found that the grain size distribution is reasonably well approximated by a log-normal distribution (Rowenhorst et al., 2010; Ullah et al., 2014; Zhang et al., 2004). Past measurements of the mean number of faces per grain have yielded 11.8 for β-brass (Hull, 1988), 12.1 for α-iron (Zhang et al., 2004), 12.8 for pure iron (Ullah et al., 2014), 12.9 for a Ni superalloy (Groeber et al., 2008), 13.7 for β-titanium (Rowenhorst et al., 2010), and 14.2 for α-titanium (Kelly et al., 2016). Therefore, to a good approximation, one can say that, on average, there are between 12 and 14 faces per grain. It has also been shown that the number of faces is positively correlated to the grain size (Hull, 1988; Kelly et al., 2016; Rowenhorst et al., 2010). In past work, features such as triple lines and quadruple points have not been given as much attention (Hardy and Field, 2016; Li et al., 2014; Ullah et al., 2014).

While the high proportion of twin-related boundaries is well known for the GB engineered materials, changes in the grain geometrical characteristics of grains before and after GB engineering are also of interest but have not been reported. The main goal of this paper is to quantify and compare the grain sizes, grain surface areas, and grain face areas within the microstructures of conventional and GB engineered 316L stainless steels. We will also examine the numbers of grain faces, triple lines and quadruple points per grain, and correlations with grain sizes. The 3D twin boundaries and GB networks of the 316L stainless steels before and after GB engineering have been reported in other papers (Liu et al., 2017).