Available online at www.sciencedirect.com

ScienceDirect

Scripta Materialia 100 (2015) 98–101

Scripta MATERIALIA

www.elsevier.com/locate/scriptamat

Grain boundary formation by remnant dislocations from the de-twinning of thin nano-twins

Y. Cao,[a,b] Y.B. Wang,[a] X.H. An,[a] X.Z. Liao,[a,*] M. Kawasaki,[c,d] S.P. Ringer,[a,e] T.G. Langdon[c,f] and Y.T. Zhu[g,h]

[a]*School of Aerospace, Mechanical and Mechatronic Engineering, The University of Sydney, Sydney, NSW 2006, Australia*
[b]*School of Mechanical and Manufacturing Engineering, The University of New South Wales, Sydney, NSW 2052, Australia*
[c]*Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, CA 90089-1453, USA*
[d]*Division of Materials Science and Engineering, Hanyang University, 17 Haengdang-dong, Seongdong-gu, Seoul 133-791, South Korea*
[e]*Australian Centre for Microscopy & Microanalysis, The University of Sydney, Sydney, NSW 2006, Australia*
[f]*Materials Research Group, Faculty of Engineering and the Environment, University of Southampton, Southampton SO17 1BJ, UK*
[g]*Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695, USA*
[h]*School of Materials Science and Engineering, Nanjing University of Science and Technology, Nanjing 210094, China*

Received 17 September 2014; revised 1 January 2015; accepted 2 January 2015
Available online 14 January 2015

We report a grain boundary formation mechanism in face-centred cubic metals with low stacking fault energies. Severe plastic deformation produces primary nano-twins with a twin boundary spacing of several nanometres, followed by secondary twinning through the activation of Shockley partial dislocations. The partial dislocations interact with primary twin boundaries, resulting in de-twinning of the primary twins and producing very high densities of sessile dislocations. The accumulation of these dislocations produces new grain boundaries with neighbouring grains having similar orientations.

Keywords: Grain boundary; Twinning; De-twinning; Grain refinement; Steel

In the last decades, extensive efforts have been devoted to understanding severe plastic deformation (SPD) induced grain refinement and deformation mechanisms [1–5]. It is well accepted that dislocation activities [6–9] and deformation twinning [10,11] are the two major grain refinement mechanisms. For face-centred cubic (fcc) metals with high SFE, SPD-induced grain refinement occurs via dislocation activities, including dislocation nucleation and multiplication, accumulation, interaction, tangling and spatial rearrangement [6,9,12]. Conversely, for fcc metals with relatively low SFE, stacking faults (SFs) and twin boundaries (TBs) play a key role in the grain refinement process [13–17]. Primary SFs and TBs block the motion of dislocations, leading to the accumulation of a high density of dislocations at the SFs and TBs that transforms the original atomically-flat coherent SFs and TBs into curved incoherent high-angle grain boundaries (GBs) [11,13,14]. As such, the finest grain size is largely determined by the smallest TB spacing or the smallest distance between neighbouring SFs/TBs [13,14].

Recent investigations on the HPT processing of a duplex stainless steel with fcc austenite having a very low SFE and body-centred cubic ferrite show that the average primary TB spacing in the austenite is ~7 nm [17]. Based on the grain refinement mechanism presented earlier for fcc phases with low SFE [13], where the interactions between dislocations and TBs transform coherent TBs into incoherent high-angle GBs, the smallest average grain size would be ~7 nm. However, the smallest average grain size was ~23 nm [17], thereby indicating that the mechanism is not applicable in the austenite. To understand the grain refinement mechanism operating in the austenite, detailed microstructural analyses were conducted on the austenitic phase in a duplex stainless steel processed by HPT to several strain levels. The present results reveal a hitherto unknown grain refinement mechanism that could apply to other fcc metals with low SFEs.

A commercial DP3W duplex stainless steel with a microstructure comprising ~50% volume fraction of both the fcc austenite and body-centred cubic ferrite was used in this research. The material was processed by quasi-constrained HPT [18] under 6 GPa applied pressure at room temperature for ¼, ½, and 1 revolution. The equivalent strain ε imposed by HPT was calculated using the relationship: $\varepsilon = \frac{2\pi Nr}{h\sqrt{3}}$, where N, r and h are the number of revolutions,

* Corresponding author. Tel.: +61 2 93512348; e-mail: xiaozhou.liao@sydney.edu.au

http://dx.doi.org/10.1016/j.scriptamat.2015.01.001
1359-6462/

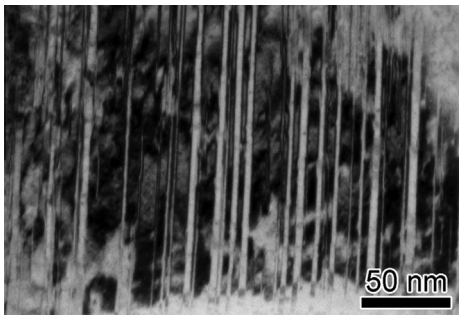

Figure 1. A typical TEM micrograph of the primary nano-twins formed in austenite grains at deformation stage 1.

the radial position, and the thickness of the disc sample, respectively [19]. Specimens for TEM investigation were carefully prepared using the standard mechanical grinding and electro-polishing methods described earlier [17]. A Philips CM12 TEM and a JEOL 3000F TEM were used for diffraction contrast imaging and high-resolution phase-contrast imaging, respectively.

The average grain size of the austenitic phase in the as-received material was ~8 μm. The austenite GBs were smooth and sharp and TBs were observed having spacings in the micrometre range. The estimated dislocation density in the as-received material was $\sim 5 \times 10^{13}$ m^{-2}. Depending on the microstructural features observed in this study, the deformation process was divided into three stages. At deformation stage 1, the HPT-imposed shear strain (ε) was smaller than ~3. In this stage, a high density of primary nano-twins with an average TB spacing of ~7 nm, based on TB spacings varying between ~1 and ~25 nm, and lengths of several micrometres was formed throughout all austenite grains, as shown in Figure 1. SFs and secondary nano-twins were rarely observed between primary TBs under detailed HRTEM analysis. The formation of a high density of primary nano-twins in coarse-grained austenite is attributed to the low SFE of the material (~26 mJ/m^2) and high external stress [20–24].

At the deformation stage 2, the shear strain value was between ~2 and ~5 and large numbers of secondary SFs and nano-twins inclined to the primary TBs were formed within the primary twin/matrix (T/M) lamellae, as shown in Figure 2. The long bands spanning across the whole image from the top-left to bottom-right in Figure 2(a) are primary T/M lamellae. Note that neither secondary SF nor secondary nano-twin were found within narrow lamellae with widths smaller than 7 nm: some of the narrow lamellae with no secondary nano-twin are marked with white arrows in Figure 2(a). A comparison between the primary TB-spacing (between ~1 and ~70 nm) at deformation stage 2 (Fig. 2(a)) and the primary TB-spacing (between ~1 and ~25 nm) at deformation stage 1 (Fig. 1) reveals an increase in the primary TB spacing. This gives evidence for the occurrence of de-twinning of the primary twins via interactions between partial dislocations and primary TBs, leading to a hierarchical nano-twined structure with a broadened primary TB-spacing. This phenomenon was also observed by Wei et al. [25] although there was no discussion of de-twinning.

Examples of the interactions are provided in Figures 2(b and c). For example, a high density of SFs (some of which are indicated by white arrows) were inclined to the primary TBs (marked with TB1) in Figure 2(b). The region enclosed by the two TB1 is a primary twin, the region enclosed by TB2 is a secondary twin, and the surrounding region is the matrix. A close examination of the primary twin indicates that the TB spacing on the left of TB2 is thinner than on the right by 7 {1 1 1} atomic layers, which is equivalent to the TB spacing of the secondary twin. At the intersection of the primary and secondary twins, the atomic arrangement is distorted by dislocations. The occurrence of dislocation/TB interactions at the intersection of the secondary and primary twins produced Shockley partial dislocations on 7 consecutive {1 1 1} atomic layers parallel to TB1 that slipped to the left (Fig. 2(b)), leading to de-twinning of the primary twin [26,27]. One dislocation at the top of TB1 in Figure 2(b) is marked by a black "T" and a magnified image of the local area surrounding the dislocation is shown in Figure 2(c). A close examination of the local distorted region magnified in Figure 2(c) reveals that a few consecutive atoms on a {1 1 1} plane are slightly distorted. The Burgers circuit in Figure 2(c) indicates the existence of the trailing partial dislocation of a SF (also marked by a black arrow in Figure 2(b)) immediately next to the dislocation. Therefore, various reactions may have occurred as the SF was driven towards the TB [28–30] and it is clear that some of these reactions can lead to the formation of new dislocations with Burgers vectors along other directions [29–31].

At deformation stage 3, the shear strain was between ~4 and ~11. Elongated grains with widths of less than ~200 nm and lengths of up to ~2 μm were formed in the austenite, as shown in Figure 3(a): one fully developed GB is marked with asterisks and two dislocation walls or low-angle GBs are indicated by two dashed lines. Detailed analysis of this microstructure reveals that the elongation direction of these grains is approximately parallel to the primary TBs, thus indicating a close relationship between the GBs and the primary T/M lamellae. Previous research suggested that coherent TBs can lose their coherency and transform into conventional GBs after trapping a large number of dislocations [13,15,16]. Based on this mechanism, two newly formed neighbouring grains should have an orientation relationship that is close to the twin relationship, i.e., a

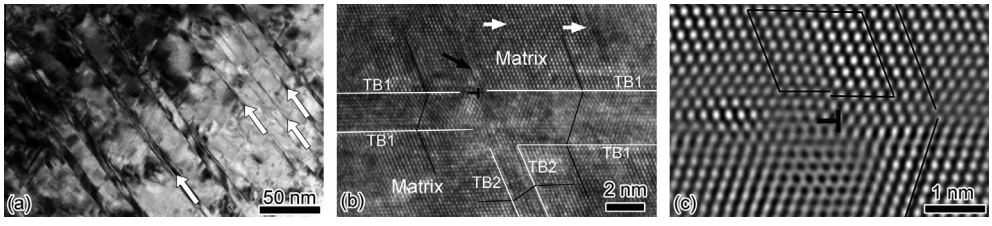

Figure 2. (a) A typical TEM micrograph showing the microstructures of secondary twinning within primary T/M lamellae; (b) a typical HRTEM image showing the de-twining to the primary twins via the interactions with secondary nano-twins/SFs; (c) a Fourier-filtered image showing the atomic arrangement around the dislocation marked by a black "T" in (b); a Burgers circle is drawn to help identify the partial dislocation.