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THROUGH-THICKNESS VARIATIONS OF MICROSTRUCTURE AND TEXTURE IN NICKEL PROCESSED BY ACCUMULATIVE ROLL BONDING

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ABSTRACT

Through-thickness variations of the microstructure and texture have been investigated in pure nickel processed by accumulative roll bonding (ARB) to a total von Mises strain of 4.8. Significant differences between the subsurface and interior layers are found both in the fraction of rolling texture components and in the fractions of high and low misorientation regions. Through-thickness gradients of the average boundary spacing and the fraction of high angle boundaries appear less systematic. The observed variations are discussed considering the distribution of strain imposed during ARB.

1. INTRODUCTION

Accumulative roll bonding is one of the techniques for severe plastic deformation (SPD) that are capable of producing submicrometer- and nanostructures with large fractions of high angle boundaries (HABs) (Saito, Tsuji, Utsunomiya, Sakai and Hong 1998; Saito, Utsunomiya, Tsuji and Sakai 1999). Similar to heavily deformed materials produced by other deformation techniques (Liu, Huang, Lloyd and Hansen 2002; Mishin, Juul Jensen and Hansen 2003; Mishin, Juul Jensen and Hansen 2010; Zhang, Huang and Hansen 2009), in ARB-processed materials the average boundary spacing has been found to decrease and the fraction of HABs to increase with increasing strain (Saito et al. 1999). This general pattern has been obtained for different materials processed by ARB and for different ARB conditions. The situation is however less clear when microstructural homogeneity is considered. In some publications, samples after 6-8 ARB cycles have been described as being homogeneous through the sample thickness even after

deformation with no lubrication (Li, Tsuji and Kamikawa 2006), while in other studies severe through-thickness heterogeneities of both texture and microstructure have been reported after the same number of ARB cycles (Heason and Prangnell 2002; Li, Sun and Li 2010).

It seems reasonable that, as the penetration of shear into the sheet body is reduced due to lubrication, the use of lubrication during rolling should improve the homogeneity of ARB-processed materials. Indeed, Bhattacharjee, Terada and Tsuji (2009) claimed remarkable homogeneity of both the microstructure and texture achieved in nickel after large-strain ARB processing with lubrication. It is however surprising that in their study pronounced rolling-type textures were reported even for the immediate surface. Moreover, very little difference was seen in the fractions of the rolling texture components between the surface and other layers. As follows from their experimental description, Bhattacharjee et al. (2009) applied large draught rolling in their ARB process, which is expected to impose significant shear due to the roll gap geometry and thus to introduce shear texture components at the sample surface (Schoenfeld and Asaro 1996). To verify whether truly homogeneous microstructures and textures are reproducible in nickel processed with lubrication, we examine the through-thickness variations of microstructures and textures of a nickel sample after 6 ARB cycles. The results are compared with literature data for both ARB-processed Ni and other heavily deformed materials.

2. EXPERIMENT

A cold-rolled sample of pure (99.967%) nickel was annealed to produce a fully recrystallized microstructure with a nearly random texture in a $2 \times 30 \times 300 \text{ mm}^3$ strip. The average grain size (including annealing twins) in this sample was measured to be $\sim 20 \text{ }\mu\text{m}$. The initial 2 mm thick strip was cold-rolled 50% on well-lubricated rolls with a diameter of 310 mm. The 1 mm thick strip obtained was then cut in half. After degreasing and wire-brushing, the halves were stacked and rolled 50% by a single pass, applying conditions of large draught rolling and creating a bonding between sheets. This roll-bonding operation was conducted repeatedly to reach 6 ARB cycles, which corresponds to a total von Mises strain of 4.8.

The microstructure of the deformed samples was studied using the electron backscatter diffraction (EBSD) technique with a step size of 50 nm in a Zeiss Supra 35 field emission gun scanning electron microscope. All EBSD maps were collected in the longitudinal section containing the normal and rolling directions (ND and RD, respectively). In these EBSD maps, low angle boundaries (LABs) and high angle boundaries (HABs) were defined as those with misorientations of $2\text{-}15^\circ$ and $>15^\circ$, respectively. Texture components were defined within a 15° deviation from the corresponding ideal orientations. The rolling texture components are represented in this work by the Copper $\{112\}\langle 111\rangle$, S $\{123\}\langle 634\rangle$, Brass $\{110\}\langle 112\rangle$ and Goss $\{110\}\langle 001\rangle$ orientations. Shear texture components are represented by the $\{100\}\langle 011\rangle$, $\{111\}\langle 112\rangle$, $\{111\}\langle 011\rangle$ and $\{112\}\langle 110\rangle$ components. The depth was described in our work by a t/t_0 ratio, where t is the distance from the center and t_0 is the sample thickness. Thus, in the center $t/t_0 = 0$ and at the surface $t/t_0 = 0.5$.

3. RESULTS

EBSD maps obtained from three different depths of the sample are shown in Figure 1. At each depth extended boundaries are observed to be closely aligned with the rolling plane (see Fig.1) forming structures commonly described in the literature as lamellar structures (Hughes and Hansen 2000). Evidence of localized shear was also often seen in this sample. In regions near the interfaces between bonded layers, the microstructure was typically more refined than in the

layer body (see Fig. 1b). Although mostly good bonding was produced along the interface, there were locations where incomplete bonding was seen, as those shown in Fig. 1b, where incomplete bonding is observed near a coarse oxide particle (seen in the maps as black features).

As is evident from the texture component map in Fig. 1, the shear-texture components are only pronounced in the subsurface layer, where the rolling texture is fairly weak (see Fig. 1a). The rolling texture becomes the dominant texture in the intermediate and center layers (see Fig. 1b,c). The summed fractions of either the shear or rolling texture components are presented as a function of t/t_0 in Fig. 2a. It is seen that the shear texture occupies 30% of the material volume near the surface, becoming weaker with increasing distance from the surface. The fraction of the rolling texture first increases with distance from the surface, reaching $\sim 90\%$ of all orientations at $t/t_0 = 0.15-0.25$, and then decreases slightly near the center (see Fig. 2a).

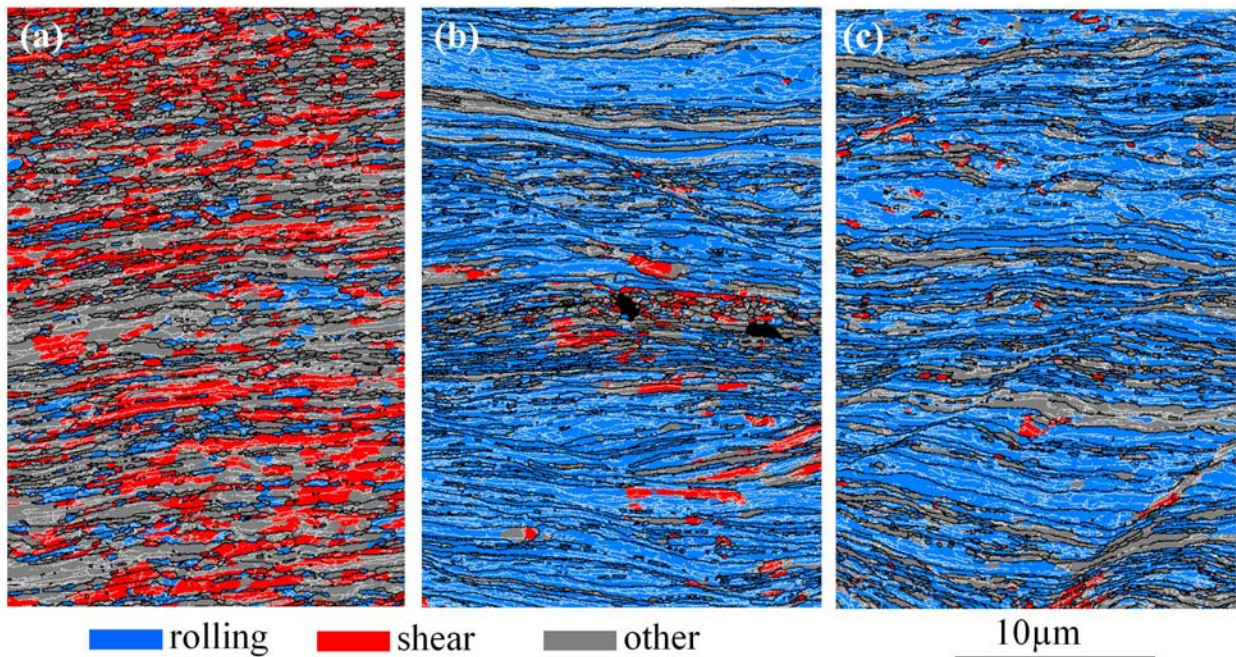


Fig. 1. EBSD maps showing microstructures in three different layers of the sample: (a) subsurface; (b) intermediate layer; (c) center. Areas having orientations of either shear or rolling texture components are shown in red and blue, respectively. White lines correspond to $2-15^\circ$ misorientations and black lines show high angle ($>15^\circ$) boundaries.

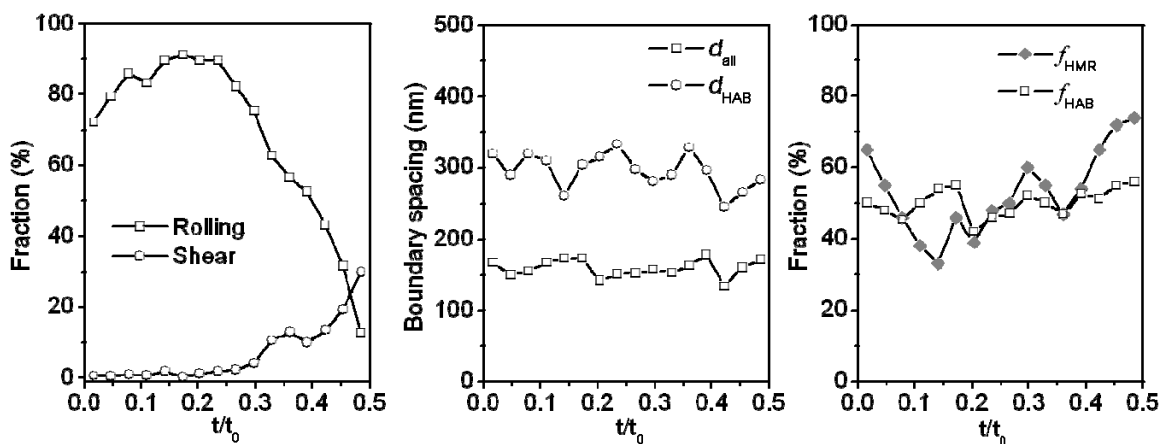


Fig. 2. Variation of microstructural parameters through the sample thickness: (a) summed fractions of texture components; (b) boundary spacing; (c) fractions of HABs and HMRs.

No systematic variation was seen in the boundary spacing data measured along the ND, as shown in Fig. 2b, where d_{all} is the spacing between all boundaries seen in the EBSD maps, and d_{HAB} is the HAB spacing. On average, d_{all} and d_{HAB} are ~ 160 nm and 300 nm. Locally the data scatter around these mean values is almost $\pm 20\%$. No strong dependence on the depth is also seen for the fraction of HABs, f_{HABs} , where on average a value of $\sim 50\%$ was found (see Fig. 2c).

It has been shown previously that the average boundary spacing and the fraction of HABs in general do not very well capture local heterogeneities of a heavily deformed microstructure (Mishin and Bowen 2009). An improved description of microstructural heterogeneity can be obtained by partitioning the microstructure into high and low misorientation regions (HMRs and LMRs, respectively). This approach has been previously applied for characterizing heterogeneities in samples deformed by either ECAE or compression (Mishin and Godfrey 2008; Mishin and Bowen 2009; Mishin, Bowen and Lathabai 2010; Luo, Mishin, Zhang, Zhang and Lu 2012). Following this previous work, LMRs are defined in the present experiment as areas greater than $2.5 \mu\text{m}^2$ surrounded by boundaries $> 5^\circ$, and the remaining areas in the deformed microstructure are HMRs. The partitioned microstructures are presented in Fig. 3. It is apparent that the fraction of HMRs, f_{HMR} , is lower in the EBSD map from the intermediate layer than in the other two maps. The through-thickness variation of f_{HMR} is further quantified in Fig. 2c, where it is seen that the f_{HMR} are large near the surface and the center. The lowest f_{HMR} is found at $t/t_0 = 0.15$ (see Fig. 2c).

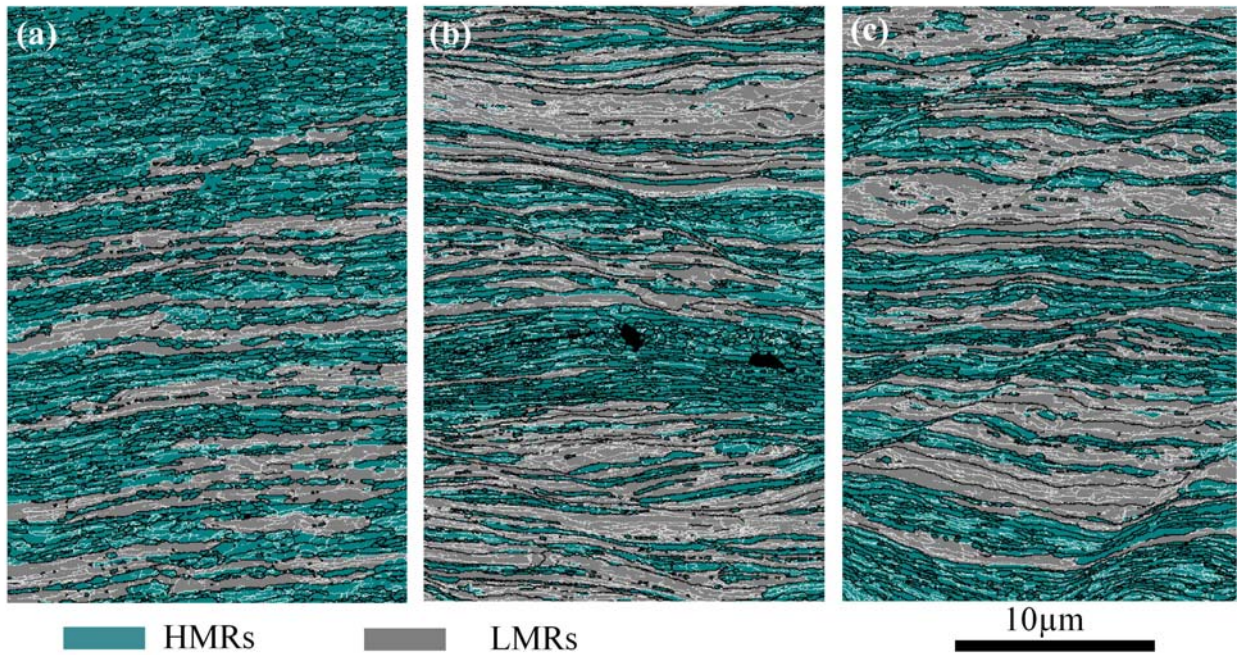


Fig. 3. HMRs and LMRs in the EBSD maps shown in Fig. 1: (a) subsurface; (b) intermediate and (c) center layers.

4. DISCUSSION

In agreement with previous reports on ARB-processed Al and Cu (Li et al. 2006), in our Ni sample we did not observe systematic through-thickness variations in the average boundary spacing and fraction of HABs. These parameters, however, do not fully describe the heterogeneity of the microstructure and thus the lack of huge variations in these parameters does not necessarily imply homogeneity of a deformed material. When a more rigorous characterization approach, based on the partition of the microstructure into HMRs and LMRs, is

employed for microstructural analysis, it becomes apparent that significant through-thickness variations are present in the sample. The higher HMR fractions near both the surface and the center of the ARB-processed sample (Fig. 2c) correlate well with expected increased shear in the subsurface layers due to large draught rolling (Schoenfeld and Asaro 1996). Note that the center layer in a given ARB sample is the surface layer in the previous ARB cycle, and thus it also experiences a significant shear strain before deformation by plane strain compression in the last rolling pass. It is therefore evident that the HMR/LMR approach is much more sensitive to the deformation prehistory than the average parameters based on the measurements of the boundary spacing and fractions of HABs. Similar conclusions have been drawn by Mishin and Bowen (2009) when analyzing through-thickness variations in ECAE-processed copper.

Severe through-thickness gradients were also seen in our work in the summed fractions of the different texture components (Fig. 2a). Again, these gradients in the fractions of the rolling and shear texture components seem to correlate with the shear strain imposed by the last rolling passes. The increased shear in the subsurface layer from the very last rolling pass reduces the strength of the rolling texture at $t/t_0 > 0.25$, although significant fractions of the shear texture are seen only much closer to the surface at $t/t_0 \geq 0.45$ (see Fig. 2a). Although plane strain compression in the last rolling pass generates a pronounced rolling texture in the layer sheared in the fifth pass, the strength of the rolling texture near the center is somewhat reduced compared to that at $t/t_0 \approx 0.2$, where the material volume has been subjected to conditions similar to plane strain compression over a large number of passes.

The significant texture gradients observed in our sample are not consistent with the conclusions drawn by Bhattacharjee et al. (2009) regarding the homogeneity of their material processed using similar rolling conditions. One possible reason of the varying data could be that in the experiment of Bhattacharjee et al. (2009) the immediate surface was mapped always in the rolling plane, where only a few grains contributed to the texture statistics, so that the limited EBSD data for the surface did not fully represent the macrotexture in this layer.

5. SUMMARY

Through-thickness variations of the microstructure and texture have been investigated by EBSD in pure nickel processed by accumulative roll bonding (ARB) to a total von Mises strain of 4.8. The ARB process produced a well-refined microstructure with an average boundary spacing of ~ 160 nm. Approximately half of the boundaries were high angle boundaries. No obvious systematic through-thickness variations were seen for either of these two parameters. In contrast, analysis of area fractions of HMRS and LMRs reveals significant differences between the layers. Increased fractions of HMRS found in the subsurface and center layers are attributed to the fact that these volumes experienced increased shear deformation in at least one of the previous two rolling passes. Summed fractions of the different texture components were significantly dependent on the deformation pre-history in different layers. A strong rolling texture was observed in the intermediate layers with somewhat reduced fractions near the center. Near the surface, the rolling texture was very weak, whereas components of the shear texture in total occupied 30% of all orientations.

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