Nanostructured Aluminum and IF Steel Produced by Rolling—a Comparative Study

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In a comparative study aluminum and interstitial free (IF) steel have been deformed to very large strain by cold rolling and accumulative roll bonding. The deformation microstructure has been analyzed by transmission electron microscope techniques and microstructural parameters have been quantified together with mechanical properties. An analysis of the strain induced change in structure, strength and ductility has shown a very similar behavior for the two materials and also that their mechanical properties can be optimized by introducing a new processing step, post-process deformation.

KEY WORDS: nanostructured metals; aluminum; interstitial free (IF) steel; transmission electron microscopy; structural parameters; mechanical properties.

1. Introduction

The finer the structure the stronger the metal is a guiding principle for material scientists and technologists. This principle is the basis for the current interest in new and advanced materials with fine scale structures down to the nanometer dimension. This has led to the development of new processes and to characterization and modeling of relationships between processing, structure, and properties of materials with extremely fine microstructures of the order of tens to hundreds of nanometers. This research and development has covered a wide range of metallic materials where the structural refinement has been obtained by thermomechanical processing.

This paper analyzes the structural refinement by plastic deformation, and presents an overview of several aspects associated with deformed nanostructured metals. The focus is on wavy glide fcc and bcc metals, taking Al and interstitial free (IF) steel as examples. In Sec. 2 the universal pattern of structural evolution with strain is briefly introduced and the structural evolution during cold rolling of a commercial purity Al and an IF steel is characterized. In Sec. 3, a new deformation process, accumulative roll bonding (ARB), is described. This process allows production of bulk sheets of nanostructured metals. In Secs. 4 and 5, characteristic structural features and mechanical properties are summarized and discussed based on the results obtained for nanostructured Al and IF steel samples produced by ARB. Finally, in Sec. 6, the optimization of the structure and mechanical properties is explored by correlating processing conditions, structural parameters and mechanical properties.

2. A Universal Pattern of Structural Evolution

Plastic deformation of a metal requires mechanical energy of which a large part is transformed into heat during processing. Only a small amount of energy, of the order of few percent or less, is stored in the metal mainly as dislocations. These dislocations arrange in different configurations and patterns, depending on materials and processing parameters. It has however been found that the patterning follows general principles, and the evolution of many different structures and their characteristics can therefore be described within a common framework.1,2) Such principles are, for example, that the structures minimize their energy per unit length of dislocation line3) and that the grain orientation through the operating slip systems significantly affects the evolution of the deformation microstructure.4,5)

The microstructural evolution during cold rolling has been studied extensively in fcc metals such as Al,6) Ni7) and Cu8) and to a lesser extent in bcc metals such as iron and steel.9,10) At small strains a cell block structure forms, as shown in Fig. 1(a) for high purity Al (99.999%)11) and in Fig. 2(a) for IF steel10,12) where cell blocks are delineated by extended planar dislocation boundaries. The cell blocks are further subdivided into cells by cell boundaries.1,2) In Fig. 1(a), the extended cell block boundaries in the cold rolled Al were identified by trace analysis and sample tilting in TEM to be parallel to the (111) slip plane. Similarly, in Fig. 2(a), the extended boundaries were found to be parallel to the (101) slip plane. However, it should be pointed out that extended boundaries not parallel to the slip planes can also form depending on the grain orientation.5,9) At large rolling reduction the cell block structure is a typical lamellar structure, with cell block boundaries (lamellar
boundaries) nearly parallel to the rolling plane and short interconnecting boundaries forming a bamboo structure, as shown in Fig. 1(b) for 99.0% Al (AA1200) and Fig. 2(b) for IF steel. The lamellar boundaries are high-angle boundaries ($\theta \approx 15^\circ$) or dislocation boundaries ($\theta \approx 15^\circ$) whereas the interconnecting boundaries are typically low-angle boundaries. In the volumes between the boundaries, individual dislocations and loose dislocation tangles can be observed.

During cold rolling the microstructure subdivides on a finer and finer scale as the rolling strain is increased. This is clearly seen in Fig. 3 and Fig. 4 where the spacing between the cell block boundaries is shown for Al and IF steel, respectively, as a function of strain. It is seen that the spacing of lamellar boundaries decreases to about 300 nm at a von Mises strain of 5.8 for Al and to about 220 nm at a von Mises strain of 2.7 for IF steel. These results indicate that at high strains the structure has been refined to the nanometer scale. However, it should be noted that due to a continuous reduction in the sheet thickness during cold rolling, the sample thickness is in general very thin after high strain rolling. For example, the Al sample deformed to the highest strain ($\varepsilon_{VM} = 5.8$, see Fig. 3) is in the form of a thin foil with a thickness of only about 250 $\mu$m. The small sample thickness makes the structural and mechanical characterization difficult, and it restricts the practical application.

3. ARB Processing

To overcome the limitation of conventional deformation processes in producing bulk nanostructured metals, new deformation processes have been developed that can impose extremely high strains into metals without changing the sample dimension. Examples are high pressure torsion (HPT) equal channel angular pressing (ECAP), and ARB. Among these processes, the ARB process was developed to extend the capability of conventional rolling in producing high strain bulk samples. However, this process differs from the conventional rolling as it consists of sheet cutting, surface degreasing, wire-brushing, sheet
stacking and finally roll bonding in one pass (one cycle). The reduction in thickness per cycle is 50% corresponding to $e_{VM} = 0.8$, i.e. if two sheets of the same thickness are stacked and roll bonded, the thickness of the rolled sheet equals to the thickness of the starting sheet. After processing by many ARB cycles, high accumulative strains can be obtained but the initial thickness is maintained.

ARB processing can be carried out under different lubrication conditions. A well lubricated ARB process is basically the same as normal rolling. However, when the ARB process is performed under dry-surface conditions without any lubricant, the process involves complicated changes in strain path and strain level, as the surface layers in each cycle are subjected to extensive shear deformation (redundant shear) in addition to rolling deformation, while the central part experiences moderate deformation by rolling. Both ARB processes have been applied to deform various metals and alloys, and bulk nanostructured sheet samples have successfully been produced. However, the results shown in the following sections are all obtained from Al and IF steel samples processed by non-lubricated ARB.

4. Characteristic Structural Features of Nanostructured Metals Produced by ARB

4.1. Structural Parameters

The key structural parameters characterizing nanostructured metals are structural morphology, spacing between boundaries, misorientation across boundaries, and density of interior dislocations present in the volume between the boundaries. Recently, the characterization of these structural parameters using various techniques has been discussed together with precautions that have to be taken in selecting sample sections and characterization techniques. It has been concluded that for sheet samples processed by rolling and ARB, characterization should be made on a longitudinal section, as being the best section to reveal the structural details, and that TEM is a suitable technique to obtain a variety of structural parameters from one sample section with high precision. It should be mentioned that electron back scatter diffraction (EBSD) has been extensively used to characterize the structural morphology, texture and misorientation characteristics for nanostructured metals and alloys. However, EBSD is incapable of revealing boundaries with misorientation angles of $\leq 2^\circ$ for highly deformed metals and it cannot resolve the dislocation structures in the volume between the boundaries. As a precise determination of these parameters is critical for understanding the characteristic mechanical behavior of nanostructured metals, all the microstructural results on nanostructured Al and IF steel to be shown in the next two subsections were obtained from TEM studies. The specific structural parameters quantified are the following (see Table 1):

- Spacing between boundaries: (i) perpendicular to the rolling plane ($d_t$), (ii) parallel to the rolling direction ($d_l$).
- Misorientation angle across the boundaries: (i) average angle, (ii) distribution of angles and (iii) frequency of high-angle boundaries ($\theta = 15^\circ$) and very low-angle boundaries ($\theta = 3^\circ$).
- Density of interior dislocations present in the volume between the different types of boundaries.

4.2. Nanostructured Al and IF Steel

Figure 5(a) illustrates a TEM observation from the longitudinal section of a commercial purity Al (JIS1100, Al–0.12Si–0.51Fe–0.11Cu–0.01Mn–0.01Mg–0.01Zn–0.04 Ti) sheet processed by 6 ARB cycles at room temperature, showing a well developed very fine lamellar structure as in the cold rolled sample (Fig. 1(b)). The boundary spacings for both the lamellar boundaries and interconnecting boundaries were measured and are given in Table 1. The lamellar boundary spacing was refined to 180nm which is finer than the lamellar boundary spacing obtained in the AA1200 Al cold rolled to comparable strain levels (see Fig. 3). Figure 5(b) shows the distribution of misorientation angles measured by Kikuchi pattern analysis in TEM.
gles measured for both lamellar boundaries and interconnecting boundaries. A bimodal distribution is seen, with one peak located below 2° and the other located between 40° and 55°. The fraction of boundaries below 3° and that of high angle boundaries (>15°) are listed in Table 1. Similar bimodal distributions have also been reported for nanostructured Al produced by cold rolling and ECAP. Within the lamellae, the presence of individual dislocations and dislocation tangles is observed. Sample tilting in the TEM confirmed that almost all lamellae contain dislocations although the dislocation density varies from lamella to lamella. The average dislocation density was measured to be about 1.3×10^{14} m^{-2}.

Figure 6 shows a TEM image and the misorientation angle distribution for a nanostructured IF steel ARB-processed at 500°C. A high temperature was used to achieve good bonding and the processing details was described previously. The morphology of the lamellar structure, Fig. 6(a), is similar to that observed in the nanostructured Al sample (Fig. 5(a)) and the presence of loose dislocations and dislocation tangles between the lamellar boundaries is also clearly seen in the image. Figure 6(b) illustrates a bimodal distribution of misorientation angles, resembling that obtained in deformed nanostructured Al (Fig. 5(b)). The average spacing measured for the lamellar boundaries and interconnecting boundaries and the density of interior dislocations are given in Table 1.

The above results show four characteristic structural features for both the Al and IF steel samples. (1) After ARB processing to 6 cycles, a lamellar structure with a lamellar boundary spacing of about 200 nm has formed. (2) The boundaries in the structure are dominated by high angle (≥15°) boundaries (60–70%). (3) A rather high fraction (more than 15%) of dislocation boundaries with misorientation angles below 3° is present in the structure. (4) The density of interior dislocations is of the order of 10^{14} m^{-2}.

These characteristic structural features show distinct differences when compared with recrystallized materials which in general are characterized by a rather small fraction of low angle boundaries and a very low density of dislocations within the grains. It is expected that these characteristic structural features of nanostructured metals will affect their mechanical behavior, which is discussed in the next section.

5. Characteristic Mechanical Properties of Nanostructured Metals Produced by ARB

For both the nanostructured Al and IF steel samples produced by ARB, the mechanical properties were evaluated by uniaxial tensile testing at room temperature. Tensile specimens with a gauge length of 10 mm and a width of 5 mm were machined from the ARB sheets such that the tensile axis was parallel to the rolling direction. More detailed testing conditions are described in previous publications.

5.1. As-ARB-processed State

Figures 7 and 8 show the tensile curves for the 6 cycle ARB-processed Al (JIS1100) and IF steel, respectively.

![Fig. 7. Engineering stress–strain curves for 99.2% pure Al (JIS1100) in undeformed state and processed by 6-cycle ARB to a von Mises strain of 4.8.](image)

![Fig. 8. Engineering stress–strain curves for IF steel (Fe–0.002C–0.003N–0.01Si–0.17Mn–0.012P–0.01Cu–0.02Ni–0.072Ti) in undeformed state and processed by 6-cycle ARB to a von Mises strain of 4.8.](image)
For comparison, the stress–strain curves of coarse grained samples used as starting materials for the ARB process are also included in these figures. For the ARB-processed Al (Fig. 7), the yield stress is 259 MPa and the UTS is 334 MPa, both of which are several times higher than those for the coarse grained starting material. The values for the elongation of the ARB samples are, however, relatively small. The uniform elongation is low (1.8%), but a relatively large post-uniform elongation (5.2%) is observed, resulting in a larger ratio of post-uniform elongation to total elongation compared to the coarse grained starting material. For the IF steel, the stress–strain curves (Fig. 8) show a similar behavior to that of Al (JIS 1100), although the stress levels are higher, as would be expected.

The tensile properties have been examined for numerous nanostructured metals and alloys, several common and characteristic features as demonstrated above for the ARB-processed Al and the IF steel have been observed: (i) a very high strength with a value several times higher than that observed in coarse grained samples; (ii) a small uniform elongation of typically less than 3%; and (iii) a relatively large post-uniform elongation.

The high strength observed in nanostructured metals have been related to structural parameters by suggesting that the yield stress can be estimated as the sum of contributions from dislocation strengthening and boundary strengthening expressed in the equation:

\[ \sigma = \sigma_0 + \sigma_p + \sigma_b \]  

where \( \sigma_0 \) is the friction stress, \( \sigma_p \) is the forest hardening caused by the dislocations in the low angle boundaries and in the volume between the boundaries, and \( \sigma_b \) is the grain boundary hardening caused by the high angle boundaries taken to be inversely proportional to the square root of the boundary spacing \( \text{i.e.} \) a Hall–Petch relationship. Using the assumption of a linear addition of strength contributions, good agreement between calculation and experiment has been found for the yield stress of the cold rolled IF steel, nickel and aluminum. However, when this procedure was applied to nanostructured Al and the IF steel with the structural parameters listed in Table 1, it was found that the calculated yield strength for both metals was significantly lower than the experimental value. For example, the calculated yield strength for nanostructured Al was about 190 MPa to be compared with an experimental value of about 260 MPa. This significant difference between the calculated and measured yield stress points to the operation of supplementary hardening mechanisms, which will be discussed in Subsec. 5.2.

The observed limited uniform elongation has been attributed to the reduced work hardening capacity in the fine scale structure due to the fact that the yield strength approaches the ultimate tensile strength. The relatively large post uniform elongation in nanostructured Al has been related to the elevated strain rate sensitivity of the flow stress, which can be obtained in fcc metals by increasing the dislocation density and refining the grain structure as demonstrated for ARB-processed Al. However, the same cause may not explain the behaviour of 6 cycle ARB-processed IF steel as the strain rate sensitivity for bcc materials is considered to be insensitive to the dislocation density.

5.2. Hardening by Annealing and Softening by Deformation

With an attempt to increase the low ductility while maintaining the high strength of the as-ARB-processed Al, a low temperature annealing at 150°C for 30 min was introduced. The stress–strain curve of the annealed sample is shown in Fig. 9, see curve 2. Compared with the as processed sample, curve 1, the yield stress increased to 281 MPa, and the elongation decreased markedly. This is in contrast to the expected behavior after annealing—a decrease in strength and an increase in elongation or ductility. Also unexpectedly, when the annealed sample was subsequently deformed 15% by cold rolling, a decrease in strength and an increase in elongation was observed. This unusual behavior was recently confirmed in nanostructured IF steel, as shown in Fig. 10. In this case, it was difficult to determine precisely the yield stress due to a problem associated with the stiffness of the tensile machine used. However, hardening by annealing and softening by deformation can be identified in terms of the maximum stress. Very recently, the same phenomena were also observed in ARB Al processed under well lubricated conditions after ARB processing to 10 cycles. The requirement of larger ARB cycles to show these phenomena in the well lubricated process, in contrast to the non-lubricated ARB process, is probably related to the smaller amount of redundant shear

![Fig. 9. Engineering stress–strain curves for 99.2% pure Al (JIS1100). Curve 1: processed by 6-cycle ARB to a von Mises strain of 4.8. Curve 2: same material as 1, plus annealing at 150°C for 30 min. Curve 3: same as 2, plus 15% cold rolling.](image)

![Fig. 10. Engineering stress–strain curves for IF steel (Fe-0.002C-0.003N-0.01Si-0.17Mn-0.012P-0.01Cu-0.02Ni-0.072Ti). Curve 1: processed by 6-cycle ARB to a von Mises strain of 4.8. Curve 2: same materials as 1, plus annealing at 150°C for 30 min. Curve 3: same as 2, plus 15% cold rolling.](image)
Detailed structural characterizations of the annealed and deformed samples have shown that the major change in the structural parameters is a decrease in the interior dislocation density during the annealing and re-introduction of dislocations into the structure during the cold rolling step, as shown in Fig. 11. This indicates that the presence of a certain amount of interior dislocations in the nanostructures produces softening rather than hardening as is observed in conventional coarse grained materials. When these dislocations are annealed out, a higher yield stress is required to activate new dislocation sources and to multiply dislocations during tensile straining. Consequently when dislocations are reintroduced the yield stress will decrease as new dislocation sources are operating. Such a correlation between the density of dislocation sources and the strength is typically observed in nanoscale metals both experimentally and by atomic scale modeling. The availability of dislocation sources or the lack of such sources may therefore significantly affect the yield stress. An operation of this mechanism in the as-ARB sample may explain the above observation (Sec. 5.1) that the experimental yield stress is higher than that calculated by only considering forest hardening and Hall–Petch strengthening. The operation of this source limited hardening mechanism in both the as-ARB-processed Al and IF steel samples has been explored in a preliminary experiment where the as-processed ARB sample was slightly deformed by cold rolling. A decrease in yield stress was observed in both cases indicating that the as-processed samples already lack dislocation sources, leading to additional strengthening.

6. Optimization of Structure and Mechanical Properties by Post-process Deformation

The finding that the presence of a low density of dislocations can introduce softening rather than hardening of nanostructured metals has led to a suggestion of a new strategy to enhance the ductility of nanostructured metals by a small post process deformation. In a recent study, this route was further explored by applying different rolling reductions to a recovery-annealed nanostructured Al produced by 6 cycle ARB. Figure 12 shows the stress–strain curves of annealed samples followed by cold rolling to 10, 15 and 50% reductions. It is seen that the deformation by 10 or 15% cold rolling causes a significant decrease in the strength and a significant enhancement in the elongation (curves 2 and 3) as compared with the annealed sample (curve 1). These results confirm the occurrence of softening by a low strain post-process deformation. However, after 50% cold rolling (curve 4), the sample shows a stress level
higher than all the other samples over the entire plastic strain range until fracture occurs. This indicates that large strain cold rolling has hardened rather than softened the sample. It is interesting to note that curve 4 is approximately parallel to the curves 2 and 3, indicating a similar stability of tensile deformation in these cold rolled samples. One of the reasons for the lower elongation observed in sample 4 than in samples 2 and 3 might be the thinner sample thickness caused by the higher rolling reduction.

Figures 13 and 14 show the mechanical property change as a function of rolling reduction. From Fig. 13, it is clearly seen that both the yield stress and the UTS of the 10 and 15% cold rolled samples are lower than the as-ARB sample. Figure 14 shows that both the uniform and the total elongation are better in these two samples than those obtained in the as-ARB sample. However, the effect of post-process deformation on the mechanical properties of nanostructured Al is not a monotonic function of the rolling reduction. As shown in Fig. 13, the yield strength decreases quickly with strain in the low strain range, followed by a gradual increase with further deformation. In other words, a deformation induced softening in the nanostructured Al occurs only when the strain is lower than a certain value, and work hardening takes place again when the strain is above this value. The strain at which the transition from deformation induced softening to hardening takes place may depend upon structural parameters such as the boundary spacing and the high angle boundary fraction. For the present nanostructured Al with a boundary spacing of about 200 nm and a fraction of high angle boundaries of about 70%, the transition strain is in the range 25–35%, as seen in Fig. 13.

Figure 15 shows a TEM image of an annealed and 50% cold rolled sample. Compared with the 15% cold rolled sample (Fig. 11(c)), several structural changes were found based on morphological observations and structural parameter quantification. In the 15% cold rolled sample, the introduced dislocations are mostly present as single dislocations, and the extent of dislocation tangling is not pronounced. With an increase in the rolling reduction to 50%, dislocation tangles and network were often observed within the lamellae, indicating strong interaction between dislocations. Figure 16 shows the quantification of the mean dislocation density for samples subjected to different treatments. Clearly the dislocation density has increased significantly after 50% cold rolling. In such a structure, both the density of the dislocations that are free to move when a stress is applied and the free length of dislocation segments that can act as dislocation sources decreases. Therefore, most of the dislocations in the structure will contribute to forest hardening.
Figure 17 shows the variation of lamellar boundary spacing with respect to the amount of rolling reduction. Interestingly, the lamellar boundary spacing is refined to 160 nm which is the smallest spacing ever obtained by cold rolling of commercial purity Al. This refinement in the lamellar boundary may be one of the causes for the significant increase in flow stress after 50% reduction.

To summarize, it has been observed that post-process deformation has a strong effect on the mechanical behavior, which is reflected in the occurrence of softening or hardening depending on the rolling reduction applied to the annealed sample. The effect of post-process deformation on the softening and hardening is being under investigation for ARB processed IF steel and similar results have been obtained, which will be reported in a separate paper. These observations indicate that the mechanical behavior of nanostructured metals is quite sensitive to the structural conditions, which can be modified in a controllable way by the post-process annealing or deformation.

7. Conclusions

(1) A comparative study of IF steel and aluminum deformed by cold rolling or accumulative roll bonding has shown that the microstructural evolution follows a universal pattern of grain subdivision during plastic deformation.

(2) Quantification and analysis of microstructural parameters show similar strain induced changes in aluminum and IF steel. Also the stress–structure relationship is comparable in the two materials.

(3) Optimization of strength and elongation has been investigated by varying processing parameters and post-process deformation is suggested as a promising route for property improvement.

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