

# Microstructural characteristics of nano-structured Fe-28.5Ni steel by means of severe plastic deformation

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**Abstract.** Microstructural evolution together with changes in mechanical properties of an high nickel content steel processed by various cycles of accumulative roll bonding (ARB) is explored. It is shown by Electron Backscatter Diffraction (EBSD) analysis that after successive roll bonding processes a stabilized nano-structure is developed containing sufficient amount of ductility under severe plastic deformation. A mean grain size of few hundred nano meter was obtained after 6-cycle of ARB process meaning that the successive ARB cycles made the structure quite refined. The starting material was mainly coming from the transformation of martensite to retained austenite, particularly under high temperature and in high cycle of ARB process. This is an early indication of the stabilization of retained austenite during the ARB process through grain refinement phenomenon. Uniaxial tensile test demonstrated that yield strength significantly improves by only one cycle of ARB process. Successive cycles of ARB process gradually increased the yield and the ultimate tensile strengths at the expense of ductility. The main cause of such a substantial increase in yield strength is discussed. Remarkably high amount of ductility was still observed in a very high amount of deformation that was applied in the 6-cycle ARB process.

## 1. Introduction

In the past two decades scientists have extensively studied bulk nano/ultrafine-grained (UFG) structure materials that had grain size below 1  $\mu\text{m}$ . The studies were mostly driven by excellent mechanical properties of these materials and their relative ease of manufacturing [1-3]. Among the manufacturing processes to achieve nano grain size range materials, several severe plastic deformation (SPD) methods have been employed [4, 5]. These methods include accumulative roll bonding (ARB) [6, 7], equal channel



angular pressure (ECAP) [8], hot pressure torsion (HPT) [9], repetitive-upsetting extrusion (RUE) [10, 11], and twist extrusion [12] etc. Of the several SPD methods used, ARB has singularly been the subject of a large number of researches, for both steels and aluminum alloys, due mainly to the relative ease of application [13]. The low ductility of SPD materials is mainly attributed to the early necking and the lack of strain hardenability of the UFG materials. Small amount of total elongation were partially overcome by a mixed of UFG microstructure and transformation induced plasticity (TRIP) effect. The total elongation is also highly dependent on the stabilization of austenite. Such stabilization, in addition to engineering the chemical composition, can be achieved through SPD [14, 15]. Kitahara et al found that the martensite transformation temperature decreases proportionally by increasing SPD amount [15].

The transformation behavior of high nickel, low carbon, content alloys has not been fully understood. Therefore, it was the subject of this research to elaborate the structural changes, and its resultant mechanical properties, that the steel would undergo during several ARB processes.

## 2. Materials and Experimental procedure

An Fe-28.5Ni-0.05C-0.003Si-0.015Mn-0.005P (wt.%) sheet steel with the thickness of 1 mm was used as the starting material. Apparently, having such a high Ni content would make the steel corrosion resistance and suitable for harsh industrial applications [14]. Initially, the sheets were subjected to annealing process at 900 °C for one hour. Cutting was performed by guillotine subsequent to this process to the dimensions of 5 × 14 cm followed by cleaning with acetone. Then, the wire brushed sheets were stacked and heated up to 500 °C in a box furnace and maintained at this temperature for 10 min. Roll bonding followed after the heat treating process. The specimen after each rolling process was quenched in water. A two-high mill with the rolls diameter of 310 mm were used to conduct ARB processes. The sheet was cut by half at the end of each rolling process and further rolling were conducted by the same procedure as explained above. The ARB processes were performed from one cycle to 4-cycle and for 6-cycle as well.

Characterization was performed by a Field-Emission Scanning Microscopy (SEM/Philips XL30S-FEG). Specimens were prepared by usual preparation method prior to the SEM analyses. The SEM machine was equipped with an Electron Backscatter Diffraction (EBSD) detector. EBSD raw data were subsequently analyzed by the TSL-OIM analysis software. Mechanical properties were also measured based on ASTM 8 standard for the preparation of the samples. Room temperature uniaxial tensile tests were conducted on materials. The tensile test machine was SHIMADZU AG-100 kN model and the strain rate of  $8.3 \times 10^{-4} \text{ s}^{-1}$  was used for all the tests. Visual inspection and extensometer measurements were used for these analyses.

## 3. Results and discussion

### 3.1. Microstructure analysis

*3.1.1. Initial material.* The microstructure of the starting material is shown in figure 1. Here, the structure mainly contains of retained austenite together with second phase martensite embedded in it. The former phase is shown in green color and the latter in pink. The grain boundaries map is also shown in figure 1b. The backscattered micrograph of figure 1a shows a somehow random grains structure. Moreover, the grains, to some degree, show a bimodal size distribution. As can be seen in figure 1b, some low angle grain boundaries (LAGBs) can be seen inside the martensite phase. Commonly, the LAGBs are indicative of dislocations in the microstructure. The low amount of LAGBs is associated with the low

amount of lath martensite in the structure. The martensite blocks are not differentiated by HAGBs and do not form a continuous phase in the structure.

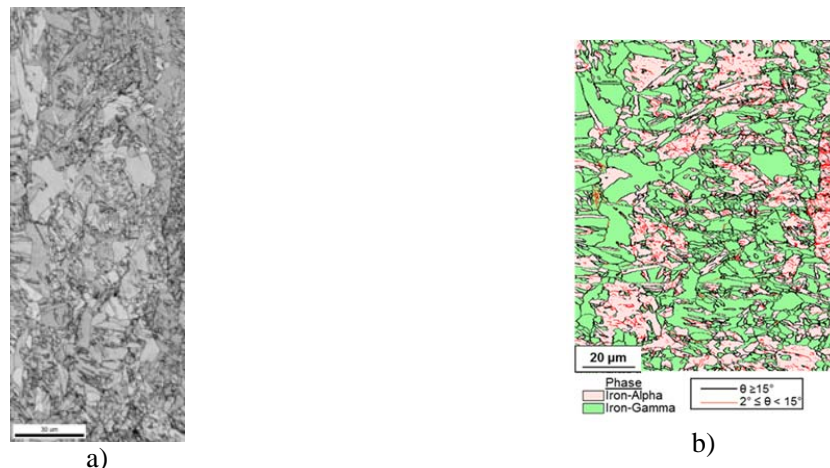


Fig. 1. In initial material, (a) SEM micrograph, (b) Grain boundary and phase map.

**3.1.2. Microstructure development during ARB process.** Two specimens of 1-cycle, and 6-cycle ARB process, as the representatives of low and high amount of plastic deformation, were analyzed by EBSD method. The grain boundary map of the 1-cycle ARB processed is shown in figure 2a. It is clear from this figure that elongated grains are developed in this situation. This is a structure very similar to typical rolling microstructure. The grain boundaries map shows the elongated cell structures. It also shows the LAGBs developed along the grains. Another aspect of this deformation path is the substantial reduction of martensite in the system. A reversion of martensite to austenite is quite plausible in this situation. Figure 2b shows the orientation color map of this condition. Unlike the starting material, here a clear grain orientation preference is observable.

Compared to the 1-cycle microstructure, in the case of 6-cycle microstructure, shown in figure 4, ultrafine grains are being developed. The grains in this case are encircled by semi-lamellar boundaries. Also, they are mostly HAGBs with large misorientation angles and with very low amount of LAGBs. The average grain size in this case is in the range of few hundred nano meter, even in some cases less than 100 nm grains, which are similar to the literature in single phase materials [6, 7]. In this case, in line with the reduction of martensite, almost no discernible amount of martensite is found. In the 6-cycle ARB processed specimen, the grains were subdivided down to nano sizes by grain subdivision mechanism. The  $M_s$  temperature is reduced by the application of severe plastic deformation in the 6-cycle ARB processed sample, as also found in a study on a similar steel [15]. The initial austenite grain size also reduces during severe plastic deformation leading to the reduction of the  $M_s$  temperature. The formation of a large amount of HAGBs is an outcome of the reduction of  $M_s$  temperature. Consequently, retained austenite would be stabilized at the expense of martensite.

The grain size continuously decreases as the number of ARB cycle increase. This is shown in the grain size profiles of figure 4 for the starting specimen as well as for the 1-cycle, and the 6-cycle ARB processed specimens. Apparently, the grain size varies from a few micron to nano meter size, even in some areas below 100 nm, as the ARB cycle increases up to 6-cycle.

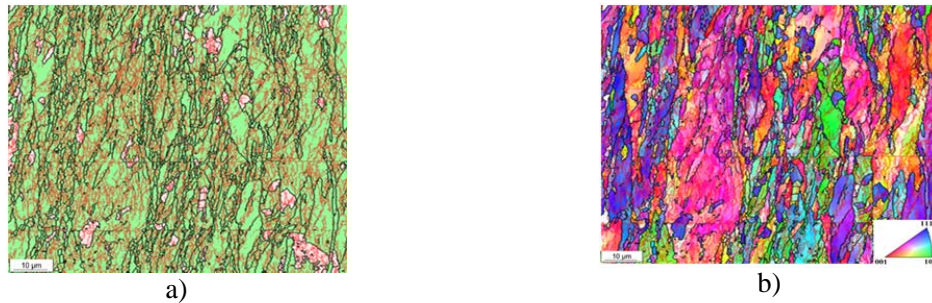


Fig. 2. In 1-cycle ARB processed sample (a) Grain boundary and phase map. (b) Orientation color map.

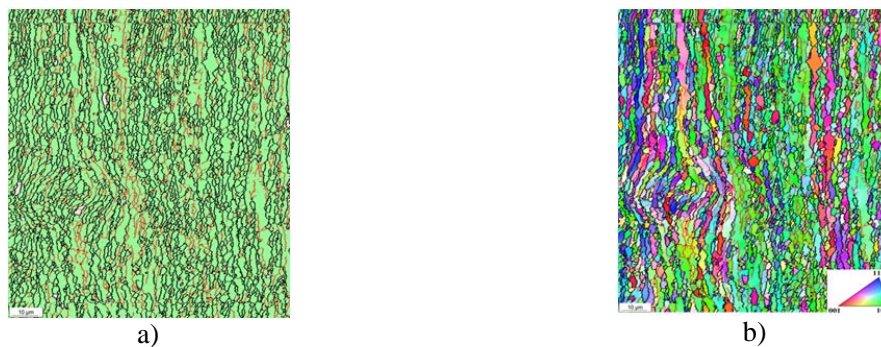


Fig. 3. In 6-cycle ARB processed sample (a) Grain boundary and phase map. (b) Orientation color map.

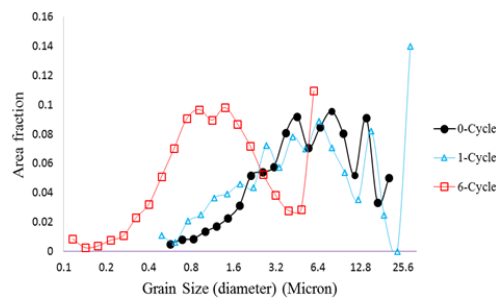


Fig. 4. Grain size distribution profile for specimens subjected to different number of ARB cycles.

### 3.2. Mechanical properties

The engineering stress-strain curves for the starting material, 1-cycle, 2-cycle, 3-cycle, 4-cycle, and 6-cycle ARB processed specimens are shown in figure 5. Here, a typical stress-strain curve is observed in the case of starting specimen. The yield strength is in the range of 300 MPa and a large strain hardening follows, together with a continuous yielding, up to an ultimate strength of about 470 MPa. As well, the ductility of the starting material is quite significant and stands at about 44%. Performing the ARB process at a relatively high temperature of 500 °C would make martensitic transformation through deformation-induced martensite mechanism possible by reducing martensite transformation. In fact, the specimen in the 6-cycle situation has substantial amount of ductility after this severe plastic deformation condition.

There is also a rapid increase of yield strength up to 498 MPa after only 1-cycle of ARB processing. Such an increase in the yield strength level after 1-cycle of ARB process have been reported

only in single phase materials, particularly for Al alloys [7]. Grain refinement and substantial increase in dislocation density were proposed as the main causes of such an increase in the yield strength level.

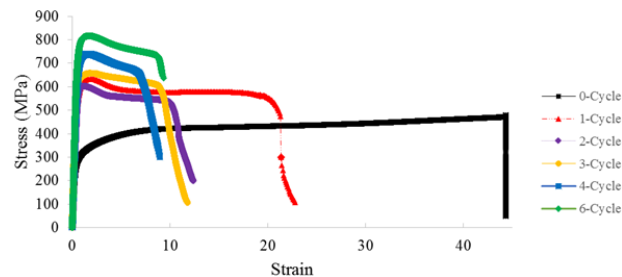


Fig. 5. Engineering stress-strain curves of different cycles of ARB processed specimens.

The extracted yield strength and ultimate tensile strength for the starting material together with the different ARB processes are given in figure 6a. Figure 6b also shows the changes of ductility under the above conditions. It can be seen in this table that the strength values increase quite rapidly in the early stages of ARB process. Ductility, on the other hand, reduces in such conditions. The pace of strength increase slows down after the first cycle and up to 4-cycle after which the rate of strength increase goes up again. The second increase in strength level is smaller than the first increment.

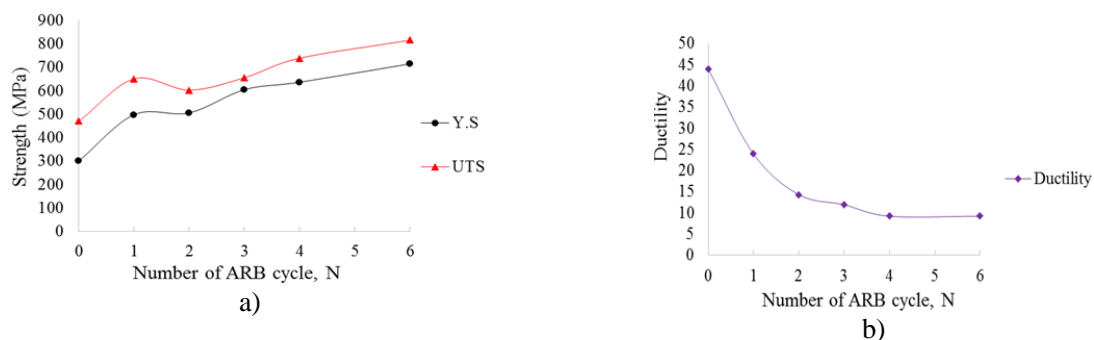


Fig. 6. Relationship between the number of ARB cycle, a) yield strength and ultimate tensile strength, (b) ductility.

#### 4. Concluding remarks

The following results were obtained by conducting this research:

- 1- The starting material contained retained austenite together with martensite phase.
- 2- The amount of martensite was substantially reduced during the ARB cycles. This was related to reversed austenite mechanism and the stabilization of austenite by lowering martensite start temperature due to severe plastic deformation.
- 3- Although dislocation density increased after only 1-cycle of ARB process, it was almost diminished after 6-cycle ARB process due to dynamic restoration phenomenon.
- 4- A substantial increase in flow strength was obtained after one cycle of ARB process. The pace of increment was greatly reduced in further cycles. The ductility on the other hand, although decreased gradually, was still significant, i.e. 6-cycle.



## 5. References

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