

## FABRICATION OF ULTRAFINE LOW CARBON METALLIC MULTILAYER BY ACCUMULATIVE ROLL BONDING

Carmela GURĂU, Gheorghe GURĂU, Lidia GURĂU, Petrică ALEXANDRU  
"Dunarea de Jos" University of Galati, Romania  
e-mail: carmela.gurau@ugal.ro

### ABSTRACT

*The effect of grain refinements by Accumulative Roll Bonding (ARB) was investigated on low carbon steel. Optical microscopy (OM) and microhardness tests were used to check the phase changes and hardness before and after ARB. A combined technique between cold rolling and ARB was used to obtain the changes of grain size under 1  $\mu\text{m}$ . Metallographic analysis reveals the change of low carbon microcrystalline one compared to the ultrafine one with increased hardness resulted from straining by ARB. The severe plastic deformation (SPD) processing of bulk metals is not straightforward due to several differences in the final microstructure at several material length scales. The plastic deformation behavior of the final material depends on many microstructural aspects, one of them being grain refinement up to amorphization caused by ARB.*

KEYWORDS: metallic multilayer, low carbon, roll bonding

### 1. Introduction

Grain refinement of metallic materials by severe plastic deformation (SPD) has been widely studied in various alloys. The fundamentals of nanostructure formation in metals by SPD have been discussed extensively [1-4]. Accumulative Roll Bonding (ARB) is one of the SPD methods that have been used to produce bulk amorphous and nanostructured materials [5-9]. During deformation by ARB, a sample is subjected to high-friction conditions without any lubricant between materials and rolls, which may cause a large amount of redundant shear strain near the sheet surface. The repetition of cutting, stacking and roll-bonding in the ARB leads to grain refinement of the sample, producing a nanostructured material with improved mechanical properties [10]. The purpose of the present study is to promote a combination between classic rolling and ARB. The effect of these two types of processes on microstructure evolution was studied. The observation of the effect of ARB on other alloys or composites was discussed [11]. Despite many reports

on the positive effect of bulk nanostructured metals produced by SPD on mechanical properties, detailed mechanisms of these effects have not been clarified yet [7]. It is also reported that SPD application resulted in improved corrosion resistance [4]. Grain refinement and amorphization induced by ARB may change the structural properties of low carbon steel thus further improving its performance. In this study, thin layered low carbon steel was fabricated and the effects of the structural and mechanical properties change of ARB were investigated.

### 2. Materials and methods

This section describes the starting microstructure of steel, the fabrication process and structure evolution as a function of rolling passes in the layered composites to a final mean layer thickness of 50  $\mu\text{m}$ .

The starting materials in this study were 5 mm thick X60 steel plates with the nominal composition presented in Table 1:

*Table 1. Chemical composition of X60 steel plates*

C	Mn	Si	P	S	Al	Ti	V	Nb	N <sub>2</sub>
0.05	1.60	0.28	0.015	0.005	0.035	0.28	0.013	0.085	0.0058

The low carbon microalloyed steel was deformed by cold rolling followed by three cycles of ARB (a thickness reduction of 99.01%) at room temperature, without lubrication and without any intermediate annealing. The rolling was performed on a quarto rolling stand, with 30 mm diameter work rolls. The equipment is operated using a DC power supply of 4.5 kW that provides high torque at low speeds, with thyristor. The thyristor drive allows for fine adjustment of speed. The cold rolled plates were cleaned with acetone and wire-brushed in preparation for bonding by accumulative roll bonding (ARB). The bonded material was cut in half and the process of surface cleaning, stacking, and roll bonding was iterated in order to produce samples having experienced couple amounts of cumulative strain. Using this technique, fine-layered steel was fabricated.

The deformation degree achieved in the ARB process was calculated with the following equation:  $\epsilon = 1 - 1/2^n$ , where n is the number of ARB cycles.

Optical micrographs were recorded using an OLIMPUS microscope, equipped with a video camera. QCapture software was used for micrograph capture. Hardness tests were made using a PMT-3-type instrument, equipped with a digital camera. Image capture and the processing of fingerprint measurements were performed using OPTIKA VIEW software. Prior to microstructural analysis and hardness tests, the samples were prepared according to the metallographic standard.

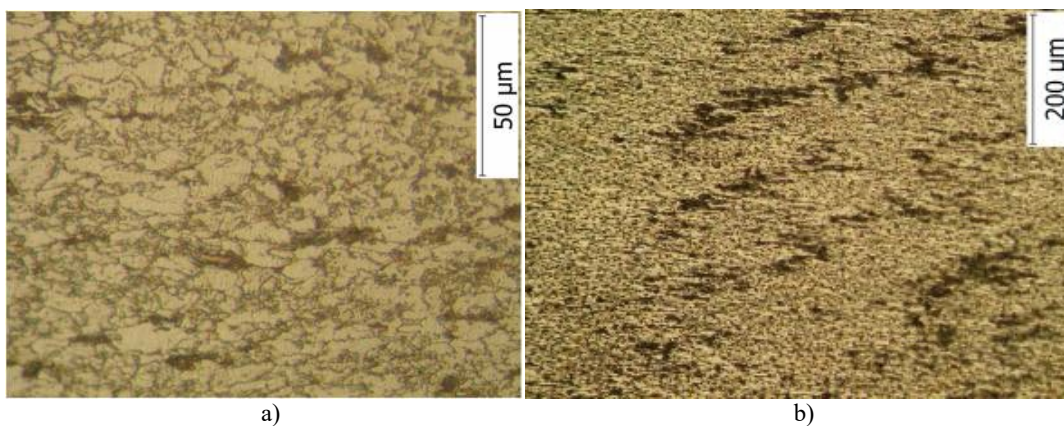
### 3. Results and discussion

The X60 steel samples were initially cold rolled at room temperature. This produced a strongly textured plate, whose normal direction is nearly

aligned with the deformation axis. As a final preparation the cold rolled plates were preceded by three ARB steps. The material underwent further straight rolling; the plates were then cut, stacked in twos and roll-bonded together at room temperature using a single high-strain rolling pass of approximately 50% reduction. Roll-bonding was performed on the same rolling mill as in the classic process.

Figure 1 shows the optical micrograph of the specimens in initial hot rolled state (Figure 1a) and subjected to 43 cold rolled steps (Figure 1b). The microstructure of the samples in initial state and after cold rolling is made up of ferrite and pearlite, specific to low carbon steels. The hot deformed grains are ferrite with few strings pearlite grains arranged in linear arrays. During the rolling process, the grains are strongly elongated in the rolling direction until lamellar boundary appears. While deformation increases, a large number of grains are distorted and refined and fibrous structure produced for clear preferred orientation [10]. In the case of cold rolling, recrystallization processes do not occur, because deformation occurs below the recrystallization temperature and is accompanied by hardening. The deformed specimen became crystallographically textured (Figure 1b) and fibrous structure is produced.

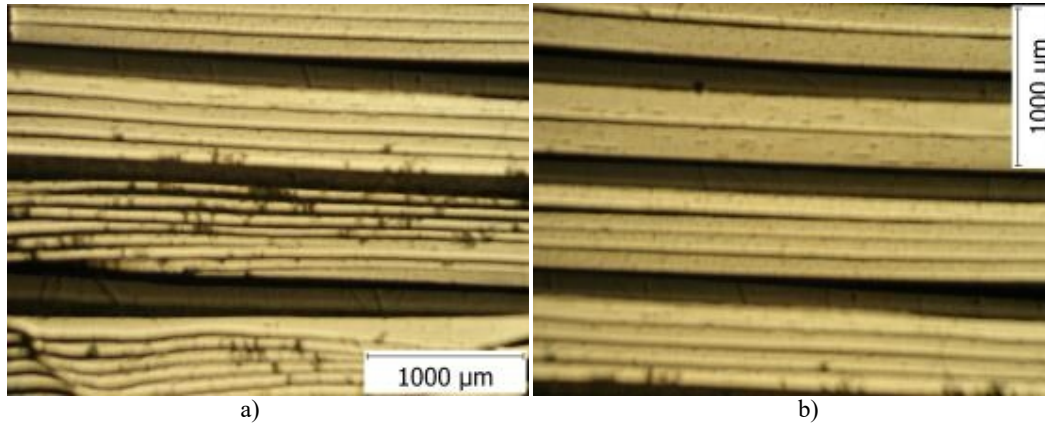
The main factors influencing the excellent deformability of steel are the size and the distribution of its structural constituents and its purity and homogeneity, incurring very large plastic strain (95.6%) without any cracks or breaks. The finer the grain size, the more likely it is that more grains appear with favorable orientation to enter the plastic deformation and to improve plasticity.



**Fig. 1.** Optical micrograph of the steel sample (a) in initial state and (b) after cold rolling in 43 passes with 95.6% degree of deformation

After cold rolling in 43 passes, the ribbon was degreased, cut into sheets and brushed. Immediately after surface treatment, in order to avoid oxidation on the surface, the sandwich samples, composed by two sheets overlapped, are subjected to concurrent rolling. The ARB process was repeated 3 times at room

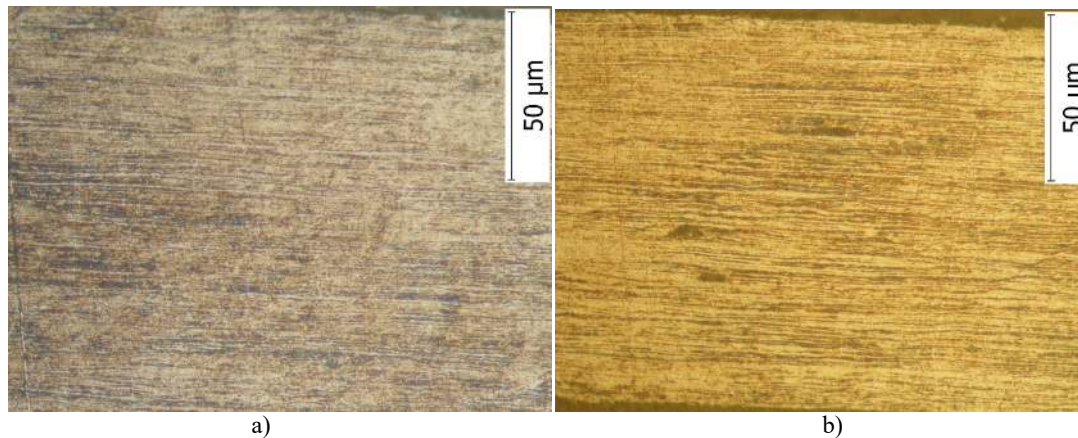
temperature. Figure 2 shows the macrostructure of the mechanically polished samples after 1, 2 and 3 ARB cycles. The resulting layered severe plastic deformation has the following total reduction [%]/number of layers/average layer thicknesses [ $\mu\text{m}$ ]: 50/2/220, 75/4/110, and 87.5/8/55.



**Fig. 2.** Macrostructure of ARB samples in longitudinal and transversal section (a) 2 and 4 layers and (b) 4 and 8 layers

Corresponding to Figure 2, as the number of ARB cycles increased, strain increased and the thickness of steel layers decreased. Unlike in the classic process, the rolling in the ARB process is not just a deformation process, being accompanied by a bonding process. The layers are joined together by rolling on the strength of the interfaces prepared preliminarily. The rolls and the surface of the samples

are not lubricated, owing to the high friction between them caused by a large amount of redundant shear strain [9-11]. In order to obtain appropriate bonding, high pressure is required concomitantly with intense friction rolling conditions. The repetition of cutting, stacking and roll-bonding in the ARB leads to grain refinement of the sample, producing ultrafine grained structure.



**Fig. 3.** Optical micrographs of ARB low carbon samples: (a) 2 layers and (b) 4 layers

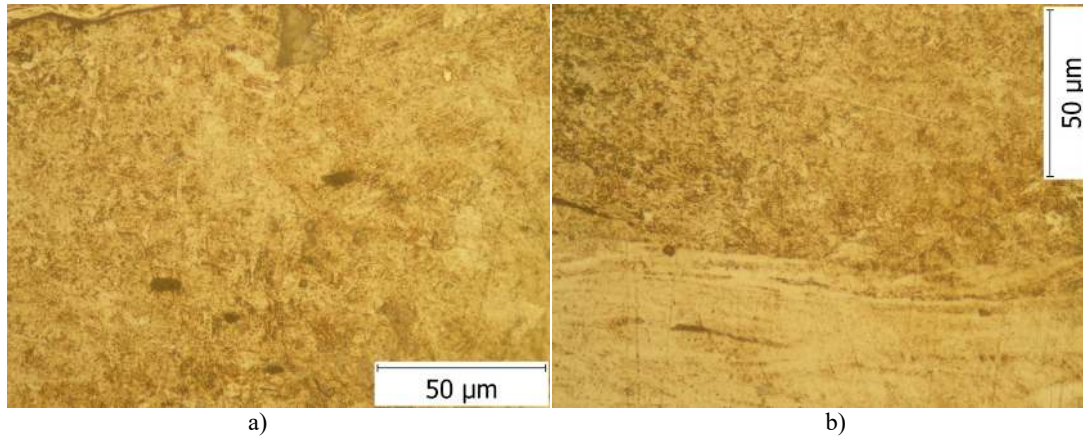
In the case of two layers, for the first roll-bonding, grain refinement and elongated grain microstructure on rolling direction as a direct effect of ARB strains can be observed (Figure 3a). Also, it can be highlighted the role of increase of trend dislocation

and agglomeration within subgrains leading to a subdivision of the microstructure. The size of subgrains falls below one micron and limits the dislocations orientation. Finally, microstructure evolves into a multilamellar structure that exists

between cells, subgrains [11]. The lamellar appearance and boundary ambiguity manifest stronger when rolling 4 layers in package. The crystalline grains loss of orientation is emphasized by the increased number of boundaries with large angles. This indicates the crystalline grain size reduction induced by severe plastic deformation [11].

The most grain refinement was experienced in the case of the samples which underwent severe

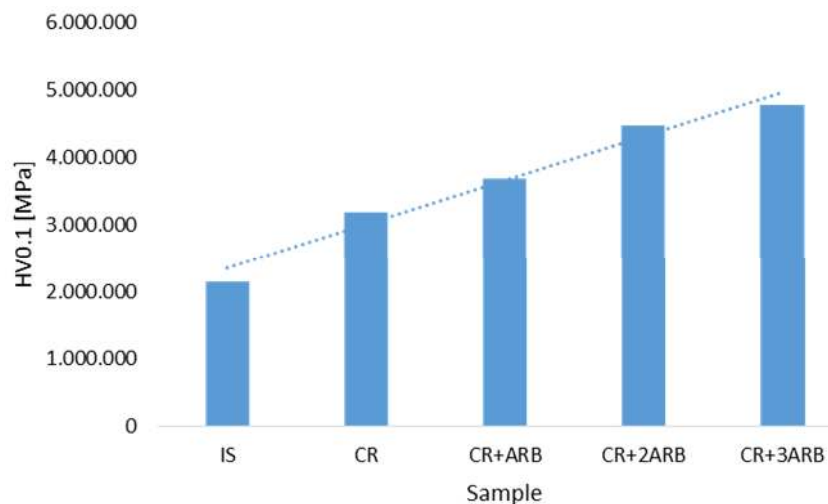
plastic deformation by cold rolling and 3 ARB cycles, having 8 layers with about 50  $\mu\text{m}$  thickness. The microstructural aspect is specific to ultrafine grained alloys. Figure 4 highlights fine distorted grains and very dense areas. Figure 4b illustrates large areas were neither lamellar grains nor boundaries are observable, after repeated metallographic etch. These areas resemble amorphous structures.



**Fig. 4.** Optical micrographs of ARB low carbon 8-layered samples: longitudinal section (a) and transversal section (b)

Figure 5 shows microhardness variation of microalloyed low carbon steel after hot rolling in initial state (IS), 43 passes cold rolled (9CR) and a different number of ARB cycles (one pass CR+ARB, two passes CR+2ARB and three passes CR+3ARB). The curve reveals that the increase in the number of

ARB cycles results in the number of layers increasing the microhardness of steel. The largest increase of the hardness value occurred after 43 passes, cold rolling correlated with refinement of grain and hardening. During the ARB second pass, microhardness had a sharp rise of approximately 800 MPa.



**Fig. 5.** Microhardness variation

#### 4. Conclusion

The effect of cold rolling (CR) followed by accumulative roll bonding (ARB) was investigated on low carbon steel. The evaluation of the structure and hardness properties of multilayered steel during different cycles of process leads to the following conclusions:

1. Low carbon steels can be processed by cold rolling followed by three ARB cycles at room temperature, without any intermediate annealing.
2. The deformation degree achieved in the case of cold rolling was 95.6% without any cracks or breaks. The total reduction after 3 ARB cycles was 87.5%, with a few observable cracks.
3. Starting from a fibrous structure obtained by cold rolling with high deformation, after ARB the structure became ultra-fine grained.
4. As the number of ARB cycles and the number of layers increase, the microhardness of steel increases; in the second cycle, microhardness had a sharp rise.

Further investigations should be carried on to find better bonding methods.

#### References

- [1]. Y. T. Zhu, T. G. Langdon, *The fundamentals of nanostructured materials processed by severe plastic deformation*, JOM 56, p. 58-63, 2004.
- [2]. R. Valiev, *Nanostructuring of metals by severe plastic deformation for advanced properties*, Nat. Mater., 3, p. 511-516, 2004.
- [3]. A. P. Zhilyaev, T. G. Langdon, *Using high-pressure torsion for metal processing: fundamentals and applications*, Prog. Mater. Sci., 53, p. 893-979, 2008.
- [4]. A. Azushima, *Severe plastic deformation (SPD) process for metals*, CIRP Annals Manufacturing Technology, 37, p. 216-735, 2008.
- [5]. M. Eizadjou, A. Kazemi Talachi, H. Danesh Manesh, H. Shakur Shahabi, K. Janghorban, *Investigation of structure and mechanical properties of multilayered Al/Cu composite produced by accumulative roll bonding (ARB) process*, Composites Science and Technology, 68, p. 2003-2009, 2008.
- [6]. Tsuji N., Saito Y., Lee S. H., Minamino Y., *ARB and other new techniques to produce bulk ultrafine grained materials*, 2003.
- [7]. Saito Y., Tsuji N., Utsunomiya H., Sakai T., Hong R. G., *Ultra-fine grained bulk aluminium produced by accumulative roll-bonding (ARB) process*, Scripta Materialia, 39, 9, p. 1221-1227, 1998.
- [8]. Saito Y., Utsunomiya H., Tsuji N., Sakai T., *Novel ultra-high straining process for bulk materials-development of the accumulative roll-bonding (ARB) process*, Acta Materialia, 47, 2, p. 579-583, 1999.
- [9]. Lee S. H., Saito Y., Tsuji N., Utsunomiya H., Sakai T., *Role of shear strain in ultragrain refinement by accumulative roll-bonding (ARB) process*, Scripta Materialia, 46, 4, p. 281-285, 2002.
- [10]. N. Kamikawa, T. Sakai, N. Tsuji, *Effect of redundant shear strain on microstructure and texture evolution during accumulative roll-bonding in ultralow carbon IF steel*, Acta Materialia, 55, p. 5873-5888, 2007.
- [11]. Marko Knezevic, Thomas Nizolek, Milan Ardeljan, Irene J. Beyerlein, Nathan A. Mara, Tresa M. Pollock, *Texture evolution in two-phase Zr/Nb lamellar composites during accumulative roll bonding*, International Journal of Plasticity, 57, p. 16-28, 2014.