COMBINATION OF THE SURFACE MECHANICAL ATTRITION TREATMENT (SMAT) AND THE CO-ROLLING PROCESS FOR THE PRODUCTION OF A NANOSTRUCTURED COMPOSITE STRUCTURE

L. Waltz 1, D. Retraint 1, A. Roos 1, P. Olier 2, J. Lu 3

1 Institut Charles Delaunay, FRE CNRS 2848, LASMIS, University of Technology of Troyes, 10000 Troyes, France
2 DRT/LITEN/DTNM/LTMEX, Commissariat à l'Energie Atomique de Saclay, 91191 Gif-sur-Yvette, France
3 Department of Mechanical Engineering, Hong Kong Polytechnic University, Hung Hom Kowloon, Hong Kong

ABSTRACT

The purpose of the present work is to produce a 316L stainless steel nanostructured multilayer composite structure with high mechanical strength and acceptable ductility by assembling SMATed plates using the co-rolling process. First, the SMAT process is used to generate nanocrystalline layers on the elementary plates so that their mechanical properties are improved, after which they are assembled through hot co-rolling. In order to quantify the mechanical response of such a type of structure, tensile tests have been performed. In addition, sharp nanoindentation experiments have been carried out over the cross section of the samples in order to follow the local hardness evolution. Next, microscopy observations have been performed for establishing a correlation with the microstructure of the laminate and to analyse the fracture surface. Furthermore, the microstructural evolution during co-rolling as well as the deformation process has been studied.

Key words: SMAT, Co-rolling, Nanocrystallization, Nanoindentation, Tensile tests.

1. INTRODUCTION

Nowadays, ultrafine grain materials with mean grain size smaller than 100 nm are actively studied and present a widespread interest because they exhibit enhanced mechanical properties, such as high strength, hardness and superplasticity properties. In order to achieve an ultrafine grain structure, several severe plastic deformation (SPD) processes have been proposed, such as accumulative roll-bonding (ARB), equal channel angular pressing (ECAP), high pressure torsion (HPT) or mechanical milling. In addition, numerous studies have shown that a recently developed Surface Mechanical Attrition Treatment (SMAT) may induce a grain refinement up to the nanometer scale in the top surface layer of metallic materials, based on mechanisms of severe plastic deformation ((Chen, 2006), (Lu, 2005), (Roland, 2006), and (Tao, 1999)). In the present work, a method is presented combining this SMAT process and the co-rolling process for the development of a semi-massive multilayer bulk structure with improved yield and ultimate tensile strength, while conserving an acceptable elongation to failure. To characterize this new material, several tests and analyses were carried out on SMATed

316L stainless steel samples and on the resulting hot co-rolled laminate structure. A surface characterization of the treated samples, which was performed simultaneously with a local hardness analysis, will be presented. Sharp nanoindentation experiments were performed in order to follow the evolution of the local hardness through the cross-section of specimens and to establish a relation with the grain size distribution. Furthermore, complementary tensile tests highlight the effect of SMAT on the mechanical response of treated and co-rolled samples. The microstructures of SMATed but undeformed tensile specimens were analysed previously by Transmission Electron Microscopy (TEM) ((Roland, 2004), (Roland, 2006)). In this work, Scanning Electron Microscopy (SEM) micrographs of failure surfaces show how the laminate samples have been torn apart during the tensile tests.

2. EXPERIMENTAL PROCEDURES

The material used in this investigation is a commercial 316L face-centered cubic (fcc) austenitic stainless steel. For the experimental tests, 120x120x1 mm^3 plates with chemical composition (in wt%) 0.025 C, 0.38 Si, 1.33 Mn, 0.027 P, 0.002 S, 16.70 Cr, 2.09 Mo, 10.20 Ni, 0.030 N, 0.40 Cu, 0.07 Co were used. The initial microstructure of the as-received material is characterised by a grain size ranging between 40 and 120 µm. The surface nanocrystallization of these sheets was obtained by SMAT. The spherical steel shot (material 100Cr6), with a diameter of 2 mm, were placed in a reflecting chamber including an ultrasonic concentrator that was put in motion by an ultrasonic generator. Because of the high vibration frequency of the system (20 KHz), the shot reaches the resonance regime and peens the entire surface of the sample with a high number of impacts in a short period of time. It is noteworthy, that impacts during ultrasonic shot-peening are highly random and multidirectional, and the intensity induced by this technique is similar to that of conventional shot-peening processes. In this study, samples have been treated during 30 minutes in air at room temperature (see illustration in Fig. 1).

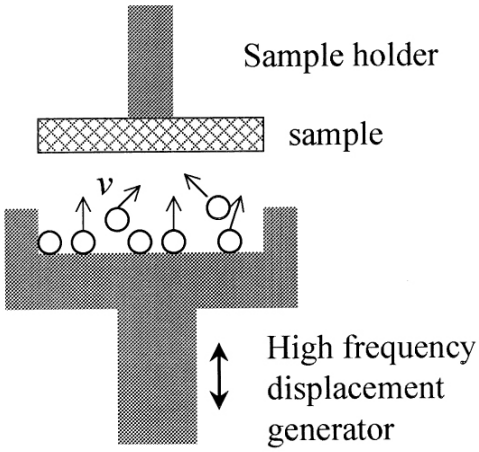

Fig. 1: Schematic illustration of the ultrasonic shot peening treatment set-up (Lu, 2005).

The co-rolling process at high temperature consists of the hot rolling of three nanocrystallised sheets as illustrated in Fig. 2. The middle plate of the piling has been SMATed on both sides, whereas the two other sheets have been treated on their inner sides only (as depicted in Fig. 2). After a preheating treatment at 550°C during 90

minutes, the stack of treated sheets was immediately co-rolled without lubrification in one pass with a global reduction ratio of 61%, and then water-cooled. The co-rolling has been carried out on a semi-industrial STANAT reversible quarto rolling equipment under a typical load of about 3500 kN.

The local hardness variation was determined on SMATed samples and on co-rolled laminate using a Nano Indenter XP^{TM} fitted with a Berkovich diamond indenter. Futhermore, in order to obtain a precise hardness profile, four indentations were performed at the same distance (depth) from the treated surface. To avoid erroneous hardness measurements due to the affected plastic zone around an indent, the distance between two indentations was fixed to at least 20 µm. The depth of the indentations themselves was fixed at 600 nm. Before testing, the samples were mechanically polished with 3 µm diamond polishing paste and polished to mirror finish with alumina.

For a better understanding of the deformation mechanism and the properties of SMATed and co-rolled stainless steel samples, tensile tests were carried out at room temperature on a screw-driven Kammrath & Weiss micro-tensile machine at a fixed strain rate of 10^{-4} s^{-1}. The dimensions of the tensile specimens were 36 mm in total length, a gauge length of 19 mm and an average cross section of 1.2 x 3.6 mm².

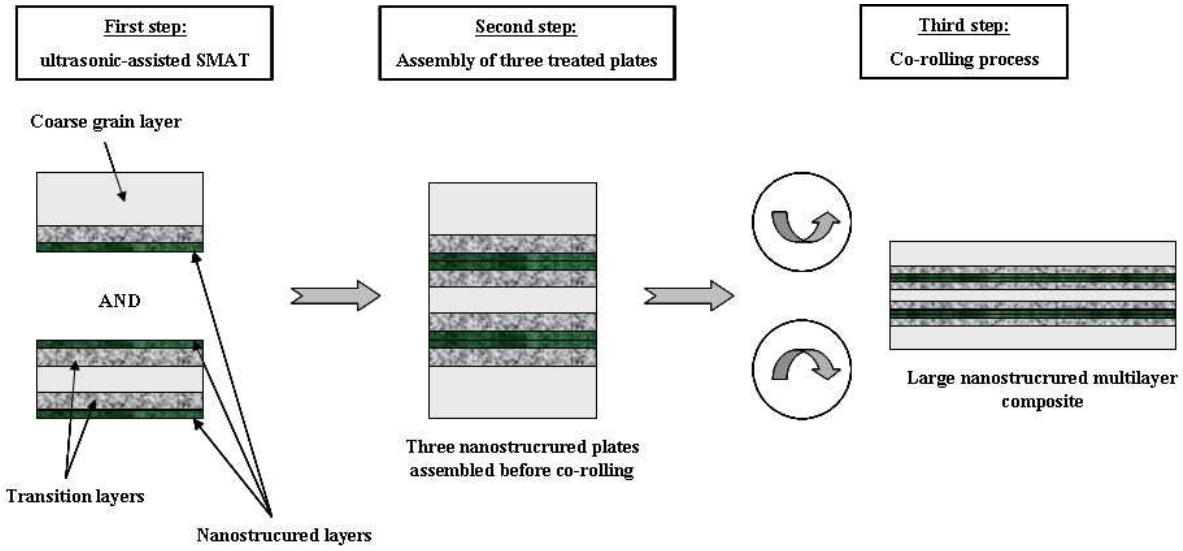

Fig. 2: Global view of the co-rolling process of SMATed samples.

3. RESULTS AND DISCUSSION

In previous studies, microstuctural characterizations of SMATed 316L austenitic stainless steel have been carried out ((Roland, 2004), (Roland, 2007)). Transmission Electron Microscopy observations (TEM) performed near the extreme top surface of the specimens have thus shown grains in the nanometer range characterized by highly random crystallographic orientations and exhibiting an average grain size of approximately 20 nm. SMAT parameters used in this work are similar to the parameters used in the previous studies.

Figure 3 shows the local hardness evolution along the depth of a specimen treated during 30 minutes with 2 mm shot. It can be observed that the ultrasonic-

assisted SMAT induces a considerable enhancement of the local hardness over the cross section of the sample. The local hardness profile exhibits very high hardness in the top surface layer of the specimen, values which decrease as the depth from the treated surface increases. For instance, at a depth of 5 µm, we record a hardness around 5.70 GPa which is more than 2.5 times higher than the coarse-grain hardness. Considering previous TEM observations, indents performed down to 15 µm below the surface are supposed to be located in the nanostructured layer which provides a partial explanation of this effect. Actually, as mentioned before, SMAT process induces a grain refinement to the nanometer scale in the top surface layer of the treated sample. This refinement process follows a Hall-Petch-like trend combined with a strain hardening phenomenon of sub-layers, and gives a simple explanation of the local hardness evolution. However, as pointed out by T. Roland (Roland, 2006), the austenite phase transformation into martensite induced by SMAT also has to be taken into account, so that a part of the high hardness could be attributed to the presence of the hard martensite phase.

Fig. 4 shows engineering stress-strain curves of a co-rolled multilayer laminate, an as-received specimen and a SMATed sample (30 minute treatment with 2 mm shot). It appears clearly that SMAT process considerably affects the yield stress of the treated materials, whereas the ductility does not seem to be changed significantly. The yield strength reaches a value as high as 460 MPa, which corresponds to an increase of the yield strength of about 56% compared to the coarse grain sample (295 MPa). This strength increase can be directly explained by the presence of SMAT-induced nanostructured layers on the top surface of both sides of the specimen, which are able to sustain a large part of the load. Furthermore, by analysing this figure, it clearly appears that SMAT combined with the co-rolling process leads to a high-strength co-rolled multilayer structure. Indeed, despite a reduction of the elongation to failure, the co-rolled laminate presents high yield and ultimate strengths reaching respectively 750 MPa and 905 MPa. The high strength of the multilayer structure can be explained by the combination of an increased volume fraction of nano and ultrafine grains combined with a hardening effect induced by co-rolling and the presence of a martensite phase.

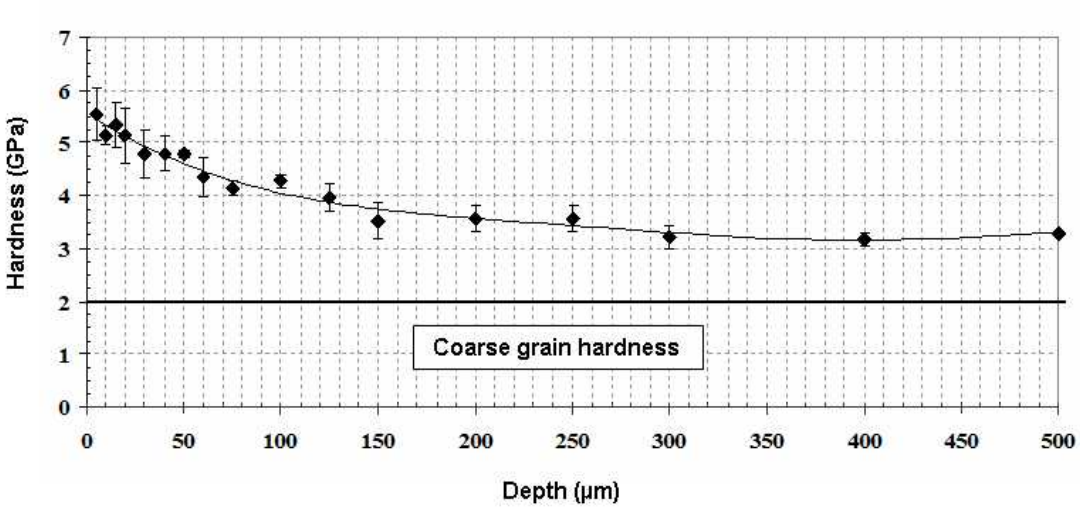

Fig. 3: Local hardness measurements obtained by nanoindentation tests on a SMATed sample treated during 30 minutes with 2 mm shot on both sides.

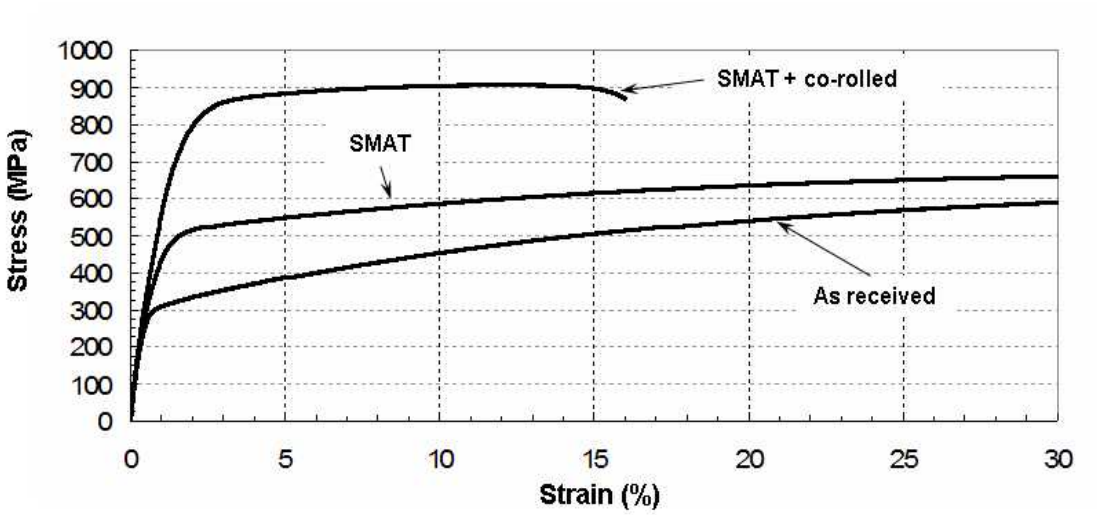

Fig. 4: Uniaxial engineering tensile stress-strain curves of (1) as-received coarse grain sample, (2) SMAT specimen, (3) SMAT + co-rolled specimen.

The SEM fractographs in Fig. 5 show an example of the fracture surface of the co-rolled multilayer tensile specimen after the tensile test. The fracture morphology of this sample presents a significant feature, since on the two lateral sheets, initially SMATed on their inner sides, two distinct failure structures can be clearly distinguished. Indeed, Fig. 5 b) presents a high magnification SEM micrograph of such an area showing a clear and straight separation between the two regions, both characterized by a specific failure mode. It is interesting to note that the flat fracture surface region near the interface (region B in Fig. 5) corresponds well with the initially nanostructured layers after SMAT ((Roland, 2004), (Roland, 2006), (Roland, 2007)). Furthermore, this flat failure surface is reminiscent of a glassy brittle material, whereas the outer side of the first sheet and the inner side of the middle sheet present a heavily dimpled morphology (region A in Fig. 5), characteristic of a ductile material. These observations indicate clearly that this sample was torn apart in a non-trivial combined ductile-brittle manner.

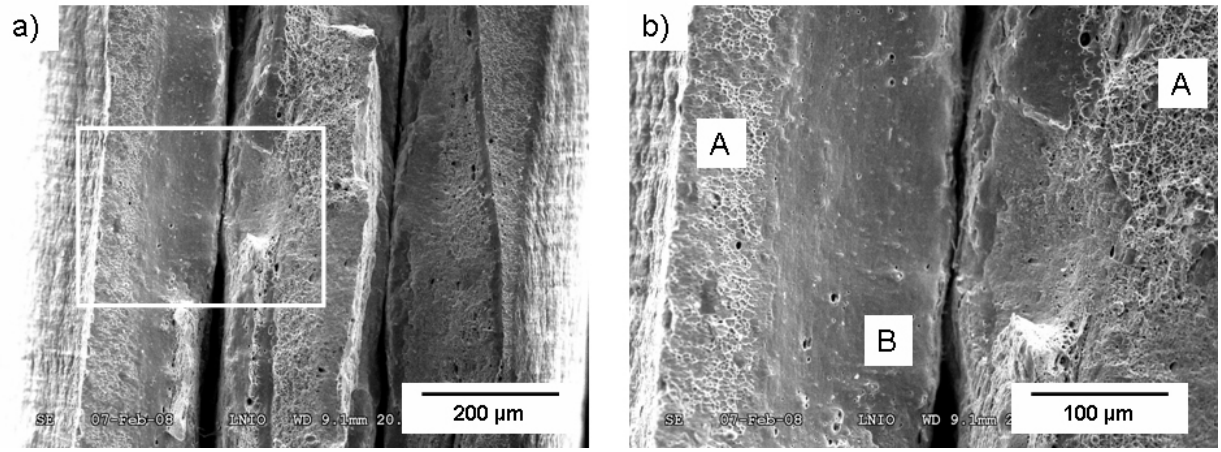

Fig. 5: a) SEM micrographs of the fracture surface of the co-rolled multilayer tensile specimen; b) high magnification SEM micrographs of selected regions (white square).

CONCLUSION

In the present study, generation of a high strength semi-massive multilayer structure by a combination of Surface Mechanical Attrition Treatment (SMAT) and the co-rolling process at high temperature has been investigated. To characterize SMATed and the co-rolled laminate, local hardness measurements, tensile tests and electron microscopy observations have been performed. It has been shown that SMAT process has a significant influence on the local hardness evolution through the cross section of the sample, and values recorded near the treated top surface are very high (≈ 5,70 GPa). The hardness increase near the surface can be attributed to the presence of a SMAT-induced nanocrystalline layer, which is confirmed by an enhanced yield strength obtained by tensile tests. Furthermore, the new co-rolled structure presents improved yield and ultimate strengths (750 MPa and 905 MPa respectively), while keeping an acceptable plastic strain to failure of approximately 16%. Scanning electron micrographs of the failure surface obtained after tensile tests show clearly a separation between flat and dimpled areas, indicating that the specimen was torn apart in a ductile-brittle manner.

ACKNOWLEDGEMENTS

The authors thank Prof. Pentti Karjalainen from the Materials Engineering Laboratory at the University of Oulu for supplying the transmission electron microscopy equipment and for helpful discussions.

REFERENCES

(Chen, 2006) J. Chen, L. Lu and K. Lu, Scripta Materialia 54 (2006) 1913-1918.

(Lu, 2005) J. Lu and K. Lu, Comprehensive Structural Integrity, Fracture of Materials from Nano to Macro 8 (2005) 1039-1044.

(Roland, 2004) T. Roland, M. Ya, D. Retraint *et al*, Journal of Materials Science and Technology 20 (2004) 55-58.

(Roland, 2006) T. Roland, D. Retraint, K. Lu *et al*, Scripta Materialia 54 (2006) 1949-1954.

(Roland, 2007) T. Roland, D. Retraint, K. Lu *et al*, Materials Science and Engineering A 445-446, pp. 281-288, 2007.

(Tao, 1999) N.R. Tao, M.L. Sui, J. Lu *et al*, Acta Metallurgica 11 (1999) 443-440.