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Effect of cryogenic temperatures and processing parameters on gradient-structure of a stainless steel treated by ultrasonic surface mechanical attrition treatment

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ABSTRACT

The beneficial effect of using cryogenic temperatures for the ultrasonic surface mechanical attrition treatment (SMAT) is depicted in the case of the 304 L austenitic stainless steel. The cryogenic temperatures induce an additional driving force for the formation of martensite at depth where the effect of shot peening tends to vanish, raising the subsurface hardness by 20 to 30%. The maximum amount of α′ martensite was always located within the subsurface. This is explained by different mechanisms operating at different stages during the SMAT process: (i) higher shear stresses at the subsurface due to the Hertzian contact stress, (ii) local heating due to the shot impacts and (iii) surface martensite destabilization when the microstructure enters the nanometric range. Consequently, the gradient structure formed by the SMAT superimposes different natures of gradients in terms of grain size and martensitic fraction. The steel being harder to deform at cryogenic temperatures, a significant decrease of the apparent surface roughness (down to 44%) is also obtained. An appropriate selection of processing parameters allows to tailor the surface towards a broad range of hardness and roughness values: 425 to 560 HV and 1.6 to 3.3 μm, respectively.

1. Introduction

In the past decades, several processes have been developed to produce materials with ultra fine grains through severe plastic deformation. Some of them, for example the High Pressure Torsion (HPT), Equal Channeling Angular Pressing (ECAP) or High Pressure Sliding (HPS) [1–3] are applied on the entire bulk material to generate fine sub-micrometric microstructures within the overall components [4]. Because of the load required to deform the overall parts containing a bulk refined microstructure and the associated reduction in ductility, in particular for high strength materials like steels, these techniques are rather difficult to implement in industry. On the other hand, as failure is often initiated from the surface, surface treatments focusing on the deformation of the outer part of a work piece have been developed. These include techniques for which the severe deformation is imparted to the surface either (i) directly, by mechanical shocks [5–9] or (ii) indirectly, by using pulsed beam treatments [10,11]. Some specific zones or critical parts that can be subjected to high stress field, stress concentration, friction, or corrosive environment can then be treated locally to enhance their performances. Among these mechanical surface treatment techniques, processes deriving from the traditional pre-strain shot peening but involving much longer treatment durations and often higher velocity of the shots are being developed. Different techniques are found in the literature under various names such as Surface Mechanical Attrition Treatment (SMAT) [5], Ultrasonic Shot Peening (referred to as USSP or USP) [12], Severe Shot Peening (SSP) [13] or High Energy Shot Peening (HESP) [14]. One of the major differences between the SMAT or USP techniques and the conventional shot peening lies in the fact that the shots are set in motion within a confined chamber and have a wide variety of incidence angles when colliding onto the surface. Thus, after a sufficiently large number of impacts, various slip systems are activated leading ultimately to a grain size refinement and substantial hardening of the surface and the subsurface [5,15–22]. The ultrasonic version of these techniques uses a high energy and high frequency (typically 20 kHz) ultrasonic generator as the energy source. Using these techniques, the mechanical performance of the overall material can be significantly enhanced through the formation of gradient-structures with refined surface microstructures: tensile properties of the 316 L [16] and the 304 stainless steels [17,18] and fatigue lives for a 316 L stainless steel [19] were improved. In addition,

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the formation of these refined gradient structures have been proved to activate the kinetics of chemical reactions during nitriding [23] and hydrogen storage [24] processes.

In order to control the exact nature of the gradient structure, it is primordial to understand the role of each of the processing parameter and their interactions. Indeed, the shots velocities and the impact density are the combination of various processing parameters such as the shot characteristics (diameter, nature, and quantity), the vibrating amplitude of the sonotrode that set the shots in motion and the duration of the peening treatment [22]. Several research works have investigated the effects of the processing parameters on the extent of surface hardening and the nature of the in-depth microstructure gradients [17,20,21]. For example, Chen et al. [21] have used two sets of SMAT processing parameters (vibrating frequencies, nature and diameter of the balls) to treat the 304 stainless steel under low (0.5 ms^{-1}) and high (10 ms^{-1}) speeds of the shots. In this way, for similar surface hardness, different sub-surface hardness gradients were obtained, resulting in different mechanical behavior for thin (1 mm) plates SMATed on both sides [21].

One of the important processing parameters that has received little attention is the temperature at which the severe plastic deformation of the surface is carried out. While the selection of a low temperature for SMAT processing has recently been shown to decrease further the size of the refined grains at the surface of metals [25–28], it also modifies the mechanisms involved in the accommodation of the deformation and the sub-surface hardness [28].

In this context, the present work investigates the overall surface and subsurface modifications brought by the ultrasonic SMAT treatment when applied on a 304 L steel as a function of different treatment parameters including the vibration amplitude, the treatment duration and, more importantly, the temperature at which the peening is carried out. The low Stacking Fault Energy (SFE) alloy 304 L austenitic stainless steel was selected here because the use of cryogenic processing temperatures is likely to affect significantly the interplay between plasticity (dislocation, twinning) and the formation of Stress Assisted or Strain Induced Martensites (SAM/SIM) that can be used to accommodate the deformation and, consequently, modify the nature of the gradient structure that is thus generated. After a description of the materials and experimental techniques (Section 2), the results related to the surface (Section 3.1) and the subsurface (Section 3.2) modifications will be described in terms of roughness, hardening gradients and in-depth martensite distributions. The discussion, given in Section 4, will address the effect of the cryogenic temperatures on modifying the martensitic phase transformations (Section 4.1) and the nature of the gradient microstructures (Section 4.2) as well as the potential to modify the surface hardness and roughness (Section 4.3).

2. Material and experimental techniques

The composition of the 304 L austenitic stainless steel investigated here is the following [wt%]: Cr 18.11, Ni 8.01, C 0.03, Mn 1.54, Si 0.45, N 0.07, P 0.03 and Fe (balance).This composition leads to M_s and M_{d30} temperatures estimated to −136 °C and 17 °C using the Pickering [29] and Angel [30] formulas, respectively. These two characteristic temperatures represent: i) the temperature at which the transformation starts thermally without external work (M_s) and ii) the temperature at which 50% of martensite is formed under 30% true strain (M_{d30}). The initial microstructure of the 304 L stainless steel is shown in Fig. 1. It is characterized by equiaxed coarse austenitic grains having diameters in the range 50–100 μm.

Cylindrical samples having a thickness of 10 mm and a diameter of were cut and the surfaces to be treated were mechanically polished to a mirror-like finish using a colloidal silica suspension. The SMAT experiments were conducted using a Sonats Stressonic apparatus [31] with 2 mm diameter100C6 shots that were set in motion by an ultrasonic device (the sonotrode) moving at a frequency of 20 kHz for the

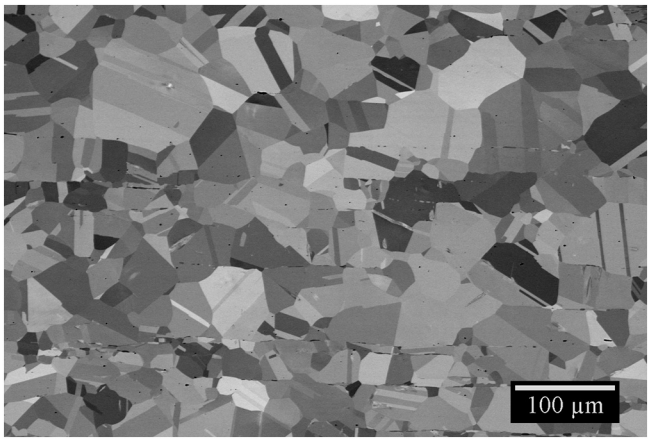

Fig. 1. SEM image of the 304 L initial microstructure.

combination of two amplitudes (40 μm and 60 μm) and two treatment durations (3 min and 20 min) as given in Table 1. The samples were peened at room temperature (RT - red), at −80 °C (black), and at −130 °C (grey) and all the data presented respect the same colour code (Table 1). A specific sample holder containing liquid nitrogen was used to treat the samples under subzero temperatures. The treatment temperatures were controlled using a thermocouple embedded within the sample at 2 mm from the treated surface. The indicated temperatures of −80 °C and −130 °C were thus the temperatures recorded 2 mm below the treated surface under a steady state reached about 3 min after the beginning of the peening treatment.

Top surface and subsurface modifications were investigated systematically after the various treatments. The roughness of the surfaces after ultrasonic SMAT was measured using a Mitutoyo SJ-400 probe roughness meter and averaged on 8 profiles of 12.5 mm. The selected roughness criterion was the quadratic mean value R_q more sensitive to roughness variations than the R_a criterion. R_q is given in Eq.1 were L is the measured length and Z(x) the profile height at the position x.

$$R_q = \sqrt{\frac{1}{L}\int_0^L Z^2(x)dx} \quad (1)$$

Vickers microhardness profiles were acquired on cross sections to determine the in-depth hardness evolutions using 5 filiations on each sample with a CLEMEX JS2000 hardness tester using a charge of 50 gf at an interdistance of 50 μm. The surface hardness was also measured directly on the treated surface after ultrasonic SMAT by a minimum of 8 indentations with the same load of 50 gf. Due to the high roughness generated by the treatment, the surface indents were carefully made at the centres of valleys of shot witness marks.

The subsurface modifications were also investigated in more details on cross sections by documenting the spatial distributions of the α′ martensite using a Zeiss Supra 40 Scanning Electron Microscope (SEM) coupled with an Electron BackScatter Diffraction (EBSD) attachment. Low magnification maps (×250, step size of 1 μm) were used to quantify the distributions of the α′ martensite as a function of the depth. Even if both the α′ and ε martensites were sometimes present in some samples, the fraction of ε martensite was so limited that it was not taken into account in the EBSD quantifications. However, as the ε martensite can be a precursor for the stress assisted formation of α′ martensite, it was investigated in some cases more precisely by X-Ray diffraction (XRD). The XRD phase quantifications were carried out using a Brucker D8 apparatus equipped with a planar detector and a cobalt radiation source ($\lambda Co_{K\alpha}$ = 1.789 Å). The measurements were integrated on the ϕ (0°–360°) and the ψ (10°, 30°, 50°) axis in order to remove potential texture effects present in the samples. The diffractograms were then post-treated with the Maud software [32] to quantify the amount of each phase in presence. To determine the in-depth phase