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# Plastic deformation to enhance plasma-assisted nitriding: On surface contamination induced by Surface Mechanical Attrition Treatment

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**Abstract.** The Surface Mechanical Attrition Treatment is a recent technique leading to the formation of nanostructured layers by the repeated action of impacting balls. While several communications have revealed possible contamination of the SMATed surfaces, the nature of this surface contamination was analyzed in the present contribution for the treatment of an AISI 316L stainless steel. It is shown, by a combination of Transmission Electron Microscopy and Glow Discharge - Optical Emission Spectrometry, that the surface was alloyed with Ti, Al and V coming from the sonotrode that is used to move the balls as well as Zr coming from the zirshot® balls themselves.

## 1. Introduction

Surface treatments and coatings are increasingly used to enhance the behavior of materials. The microstructure refinement by severe plastic deformation techniques leads to a significant increase of the materials properties (fatigue, wear, corrosion, etc ...). Among the plastic deformation techniques, the Surface Mechanical Attrition Treatment (SMAT) has been suggested as a promising way to generate surface nanostructures and sub-surface highly deformed microstructures on metallic material surfaces [1]. Such treatment involves the multidirectional repeated impact of the sample surface by high velocity balls [1, 2] at high strain rates of approximately  $10^2$ - $10^3$  s<sup>-1</sup> [3, 4]. It was also shown in recent studies that the SMAT parameters have a direct effect on the deformed microstructure evolution [5, 6]. Also, it was suggested that a contamination layer can be formed at the surface of SMATed samples [7-9]. This type of contamination phenomenon has been also observed in previous studies involving the impact of energetic balls or projectiles generating severe plastic deformation on surfaces. For example, after treating the Ni base C2000 HASTELLOY by Surface Nanocrystallisation and Hardening (SNH), Villegas et al. observed that the surface was contaminated by the colliding tungsten carbide balls [10, 11]. Another work, dealing with unidirectional repeated impacts, generated by a magnetic micro impactor, has revealed the



presence of tungsten aggregates coming from the impacting tungsten carbide tool at depth as deep as 140  $\mu\text{m}$  from the top treated surface [12].

More generally, the process of incorporating other chemical elements by using colliding balls has been used to generate surface mechanical alloying [13, 14]. While it was claimed that SMAT carried out prior to nitriding can be used as an activator for increasing the diffusion of nitrogen in iron [15, 16] and steels [17], such type of duplex treatment failed to increase the thickness of the nitrided layers for the 316 L stainless steel [8, 9]. It was suggested that oxides induced by the SMAT process could affect the surface and inhibit partially the development of the nitrided layer by blocking diffusion [9]. As it is well established that surface contamination can occur during SMAT [18], the aim of the present work is to improve our understanding of the contamination mechanisms and their exact origins.

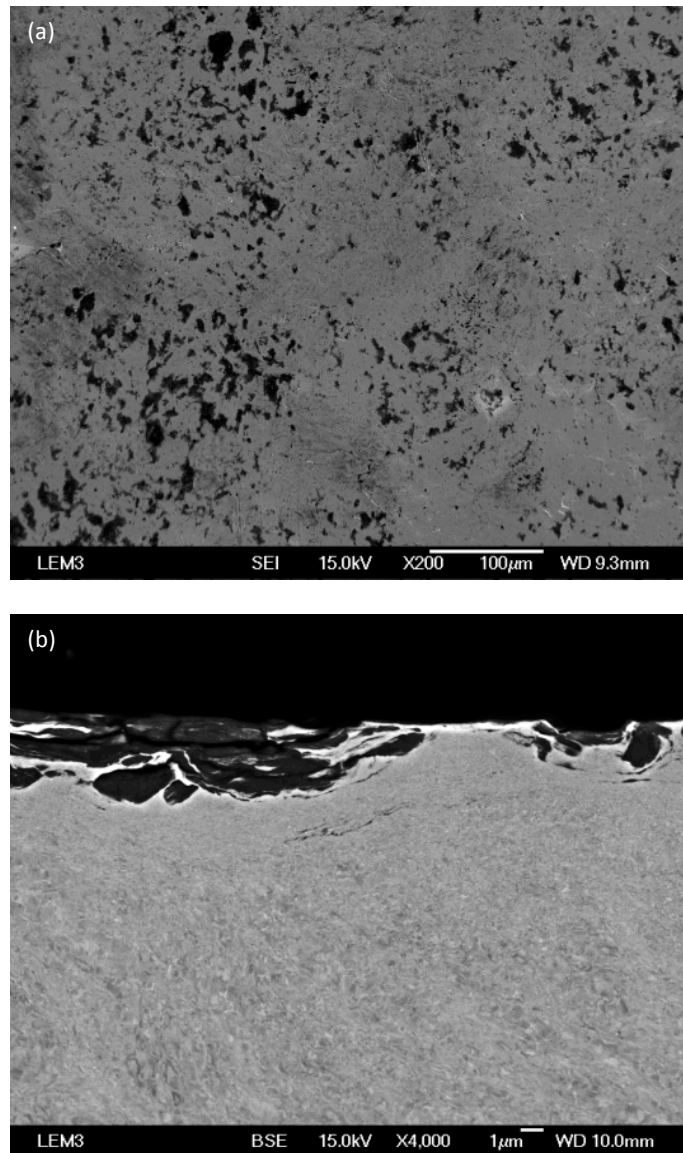
## 2. Materials and experimental methods

The material studied in this work is a 316L stainless steel having the following chemical composition (wt %): C: 0.017, Si: 0.38, P: 0.019, S < 0.002, Mn: 1.79, Mo: 2.74, Ni: 14.1, Cu: 0.16, Cr: 17.43 and Fe. Samples were cut to a thickness of 8 mm and mechanically polished to a mirror-like finish. The SMAT was carried out under an argon atmosphere in order to avoid excessive oxidation. The sonotrode, the tool that creates ultrasonic vibrations and transfers this vibrational energy to the balls, was made of the TA6V titanium alloy. The frequency of the sonotrode was set to 20 kHz. While the sonotrode amplitude was set at 60 or 80  $\mu\text{m}$ , the nature of the balls, either 100Cr6 stainless steel or ZrO<sub>2</sub> (zirshot®), was also modified.

The modified layer microstructure was observed by electron microscopy using a JEOL 6500F Scanning Electron Microscope (SEM) as well as a Philips CM200 Transmission Electron Microscope (TEM). EDX (Energy Dispersive X-ray spectroscopy) was done in the TEM to investigate qualitatively the chemistry of the modified layer. With regard to the in-depth chemical contamination of the samples, an analysis was carried out by Glow Discharge Optical Emission Spectroscopy using a Horiba Jobin-Yvon apparatus.

## 3. Results and discussion

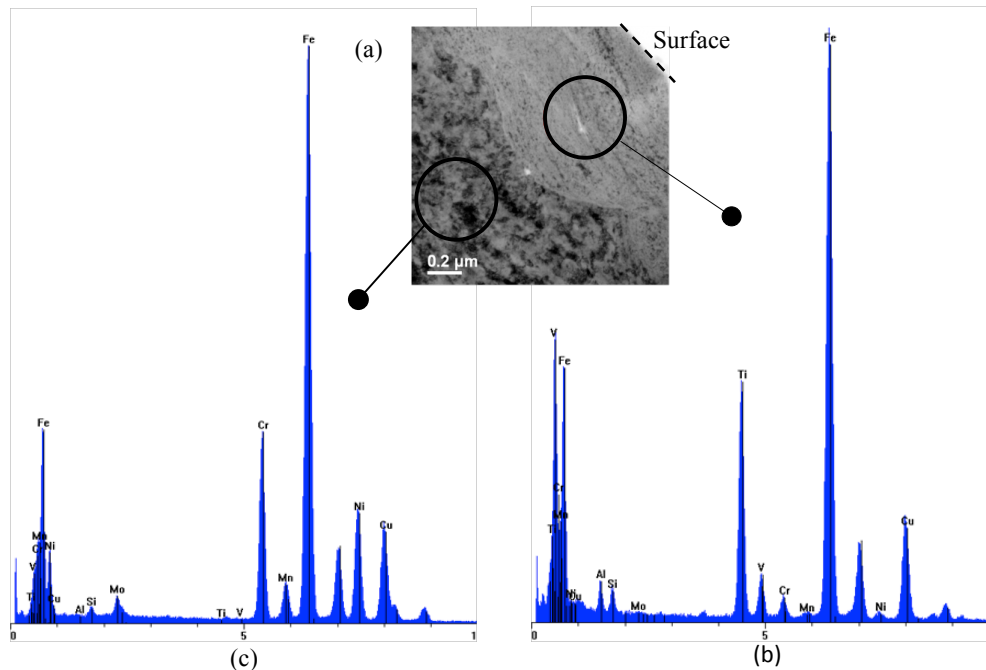
The microstructures and deformation mechanisms taking place in the 316L SS during the SMAT process have been already detailed in [6, 19, 20]. The first stage of the grain subdivision is controlled by the formation of planar dislocation arrays and twins. Then, as strain is increased, several twin systems and, depending on the exact chemistry of the alloy [6], the martensitic transformation are activated to refine further the average grain size [6, 18, 19]. Figure 1 shows SEM images, acquired under Backscattered Electron imaging conditions, from the top surface and a cross-sectional view of a sample treated by SMAT with the 100Cr6 balls for the most severe conditions (20 min, 80  $\mu\text{m}$ ). Dark spots can be seen at the top surface in Fig. 1 (a). The analysis of the cross-section in Fig 1 (b) shows that they correspond to local contaminated domains, the thickness of which can reach about 4  $\mu\text{m}$  in some zones.



**Figure 1:** SEM images showing the (a) surface morphology and (b) the cross section microstructure after 316L SS SMATed under 80  $\mu\text{m}$  of amplitude for 20 min with 100Cr6 balls.

A bright field TEM image showing a surface contaminated domain is given in Figure 2. Two zones having different aspects and different chemistry are observed. The local EDX chemical analyses of the two zones are given in Figs. 2 b and c. The bottom left of the image in Fig. 2a corresponds to the base material in which, consistently with earlier studies [6, 9, 18, 19], it is observed that the heavy deformation has generated the presence of nano-grains. Consistently with the chemistry of the base austenitic steel, peaks of Fe, Ni, Cr, Mn and Mo are clearly depicted. It is important to note that the high intensity of the Cu peak is due to electrons interacting with the sample holder in the TEM. The top right of the image corresponds to a contaminated domain revealed in Fig 1. In addition to some of the initial chemical elements

present in the 316L sample, the analysis of the top surface reveals the presence of other elements such as Ti, Al and V. This indicates that external elements were introduced during the SMAT treatment. In the present case of the specific contamination analyzed here, the chemical species introduced within the surface are the constituting elements of the Ti-6Al-4V alloy from which the sonotrode is made of.

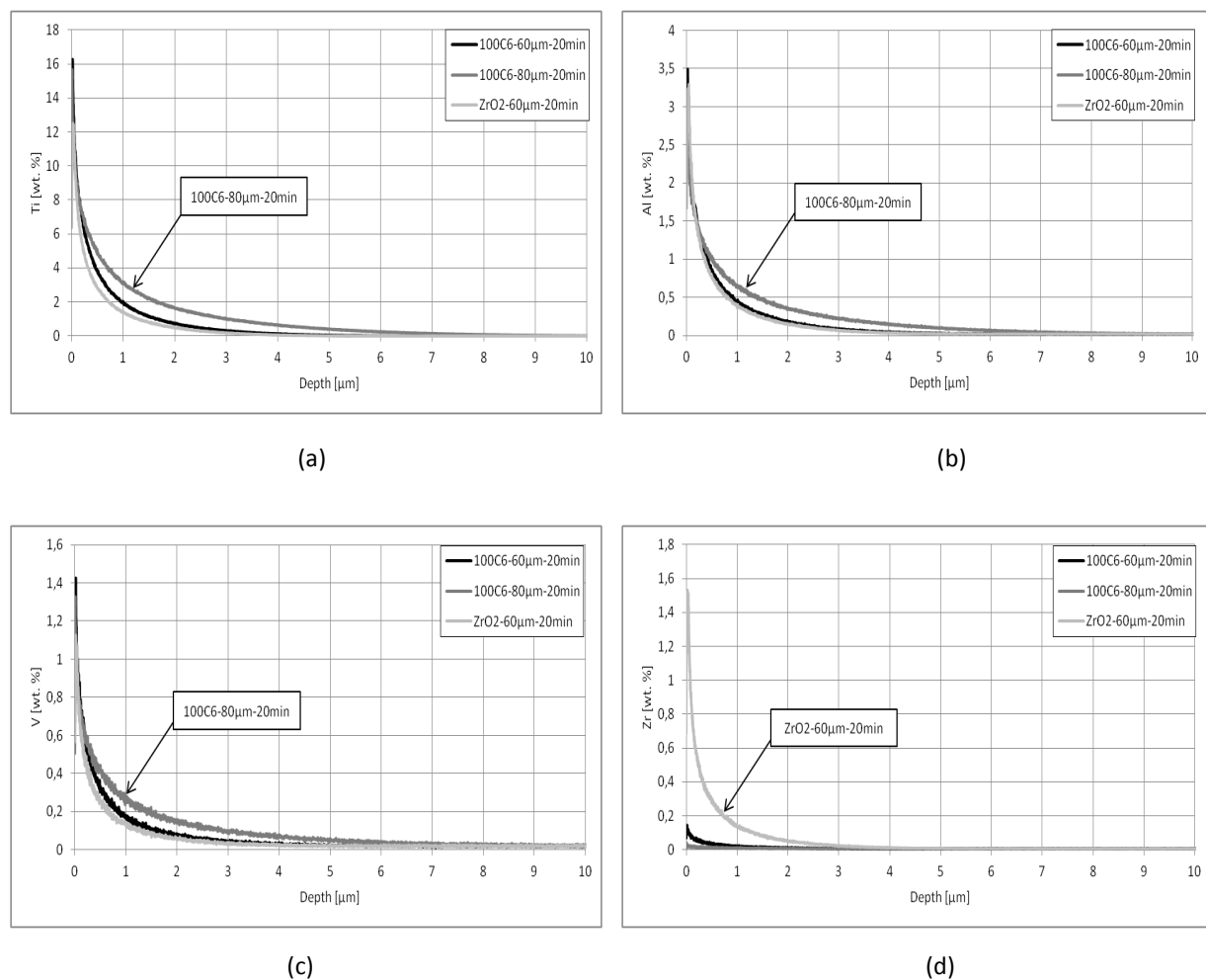


**Figure 2:** (a) TEM bright field image presenting the zones where the EDX analysis was carried out. (b) At the top surface layer, (c) at the subsurface after 316L SS SMATed under 80  $\mu\text{m}$  of amplitude for 20 min with 100Cr6 balls.

To gain a more statistical insight on the exact nature of the surface contaminations, a GD-OES analysis was carried out on different samples treated under various SMAT conditions. Figure 3 shows the GD-OES results corresponding to the content (wt%) of the different chemical elements expressed as a function of the distance from the top surface. As the data are averaged over a surface of 12 mm<sup>2</sup> and because of the surface roughness induced by the SMAT process, the curves only give a qualitative trend of the amount of contamination.

It is firstly observed (in Fig. 3 (a)) that Ti is present in a significant amount at the surface in comparison with the other contaminating elements. Its content reaches an average of about 10 wt% at the top surface and its presence is detected over a maximum depth of about 9  $\mu\text{m}$  for the sample treated with the most severe conditions (80  $\mu\text{m}$  / 20 min). On the same sample, contents are reaching about 4 wt% in Al and 2 wt% in V, for the same maximum affected depth of about 9  $\mu\text{m}$ . Their relative contents and this similar penetration depth confirm that these 3 elements were introduced as the consequence of some material removal from the sonotrode that was subsequently transferred to the sample surface and incorporated into the substrate under the peening process. Concerning the nature of the

surface pollution, a second interesting result is the detection of zirconium for the sample treated with the Zirshot® balls (figure 3 (d)). Its amount is globally lower with content that do not exceed 1.5 wt% for a maximum depth of 4  $\mu\text{m}$ . This element is only observed when Zirshot® balls are used, indicating thereby that the balls also transfer some of their chemical elements. Because of the nature of the treated samples, the contamination of the sample surface induced by the 100Cr6 balls is less easy to detect.



**Figure 3:** GD-OES in-depth surface chemical composition (wt%) evolution of (a) Titanium, (b) Aluminum, (c) Vanadium and (d) Zirconium for samples SMATed under different SMAT conditions.

To summarize, it is observed that the contaminated depths and the elements content are different as a function of the SMAT parameters. It is observed that increasing the sonotrode magnitude results in slightly higher Ti, Al and V concentrations while it is also clear that the nature of the balls has an influence on the surface contamination.

#### 4. Conclusion

The observations have revealed, after long attrition duration (20 min), the presence of contaminated layers on the surface of the SMATed samples. This contamination results from the material transfer from the different constitutive parts of the treatment apparatus.

This contamination by material transfer is due to the mechanical action of the ball which snatch some fragments of matter from the sonotrode and carry them to the top surface. The ball themselves can also transfer some of their chemical species.

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