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# In-situ EBSD investigation of thermal stability of a 316L stainless steel nanocrystallized by surface mechanical attrition treatment

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## Abstract

In-situ Electron BackScatter Diffraction (EBSD) was used to study the thermal stability of a stainless steel nanocrystallized by Surface Mechanical Attrition Treatment (SMAT). A grain size gradient was generated after SMAT from the surface to the interior of the specimen. Observations at different temperatures were performed to study the thermal stability of the gradient microstructure. During the investigation, **oxidation** was detected which greatly affected the indexation quality especially for high temperatures. After an ionic polishing, the grains could be properly revealed. No obvious microstructure changes were highlighted up to 720°C, which indicated a good thermal stability of the nanocrystalline grains.

**Key words:** Nanocrystalline materials; SMAT; In-situ heating; EBSD; Thermal analysis

## 1. Introduction

Surface Mechanical Attrition Treatment (SMAT) is one promising surface modification technique. It can transform the surface layer of coarse grains to ultrafine grains by means of severe plastic deformation [1-3]. The nanocrystallization process is determined by high strain rate multi-directional impacts between shot and **the part**. During SMAT, a large quantity of crystallographic defects such as plastic slips can be generated. The multiplication of plastic slips can progressively lead to ultrafine grains in the near surface region if the treatment intensity is high enough. However, in the bulk region, the material state is not modified, and the microstructure remains unchanged. Between the ultrafine grained layer and the bulk, a transition region is present and characterized by a high strain hardening gradient. Some metallurgical parameters generated by SMAT including ultrafine grains can be beneficial to enhance mechanical properties such as wear [4] or fatigue [5-8].

For components exposed to high temperature in service, the thermal stability of gradient microstructure generated by SMAT is an important factor as it determines the contribution of SMAT for such conditions. During the last decade, the thermal stability has been studied for various SMATed materials [9-11]. The results show that the thermal stability is dependent on materials. For example, nanometer grains of alloys with precipitates are more stable when subjected to temperature [9], whereas nanometer grains of pure metals are less stable [10, 12]. In these studies, different techniques such as Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM) were used, and all the investigations were carried out ex-situ. The heating process must thus be interrupted in order to observe the microstructure. Compared to in-situ observations, real time microstructure evolution cannot be monitored with ex-situ techniques.

In this work, in-situ Electron BackScatter Diffraction (EBSD) was used to follow eventual microstructure changes of a SMATed steel during thermal exposure. The temperature was increased up to 720°C to study the thermal stability, especially of the ultrafine grains in the surface layer.

## **2. Material and experiments**

The investigated material is a 316L stainless steel. Its chemical composition is as follows (wt.%): 17.37Cr, 2.80Mo, 14.52Ni, 1.70Mn, 0.26Si, 0.17P, 0.08Cu, 0.07V and Fe balance. For SMAT, shot of 3 mm diameter in 100Cr6 steel was boosted by an ultrasonic generator working at 20 kHz. The specimen was **first treated during** 15 minutes with a generator power of 30%, **then treated during** 5 minutes with a power of 50%.

In-situ EBSD technique was used to characterize the microstructure under a temperature increase up to 720°C. The observations were performed using a FEG-SEM SUPRA 55-VP equipped with a platinum based heating system. The temperature was controlled in order to follow a programmed increase path. It was increased with a rate of about 40°C/minute. For different values, the temperature was kept constant so as to perform EBSD measurements. EBSD scanning was performed in an area of 200 µm×50 µm with a step size of 0.5 µm. About 4 minutes were needed to achieve a cartography. For the specimen preparation, a sample was successively SMATed, cut along cross-section, molded, mechanically ground, polished to a mirror-like finish and then polished with an OPS solution.

## **3. Results and discussion**

EBSD observations on the cross-sectional surface reveal a microstructure gradient in the SMAT affected region, similar to that obtained in a previous study [3]. A grain refinement down to the nanometer scale occurred in the top surface region. The thickness of the nanostructured layer is about 10  $\mu\text{m}$ , followed by a mixed grain size region, where both ultrafine grains and relatively large grains are present. A typical microstructure obtained at 400°C is illustrated in Fig. 1a. This microstructure can also represent the as-treated state of the material, since at low temperature the microstructure is rather stable. In addition, SMAT doesn't induce any austenitic phase transformation into martensite in this 316L alloy, as found in previous studies [11, 13].

Fig. 1 illustrates the obtained maps for temperatures from 400°C to 720°C. It can be seen that with an increase of temperature, some areas become dark (Figs. 1b to 1e), which means that these areas are no longer well indexed due to the degradation of the Kikuchi diagram quality. This phenomenon is more significant in the areas with small grains, especially in the nanostructured layer. At 720°C, the indexation is totally lost (Fig. 1f).

Fig. 2 presents **some** characteristic areas after a thermal exposure at 720°C and cooling down to room temperature. As it can be seen, all areas give rise to Kikuchi diagrams with more or less acceptable quality, which indicates that over a certain temperature, electron diffraction is deteriorated. From 560°C to 650°C, a loss of Kikuchi pattern quality occurs at grain boundaries (Fig. 1) and especially at the SMATed surface. According to Fig. 2d, this loss of indexation quality is due to an oxidation effect, as indicated by the Energy Dispersive X-ray Spectrometry (EDS) analysis. This phenomenon occurs more easily on small grains. It seems strange that the sample is oxidized even in a secondary vacuum (around  $10^{-10}$  atm), i.e. the working condition of a conventional SEM. In fact, according to the Ellingham diagram [13], chromium oxide can be formed at 500°C even when the pressure of oxygen is as low as  $10^{-42}$  atm. However, the oxidation kinetics is quite low at 500°C and the temperature **holding** is not long enough to notice any significant changes in the indexation quality (see Fig. 1b). That is why a noticeable indexation quality degradation can be observed only beyond 560°C (Fig. 1c). Fig. 2a shows an oxidation gradient from the surface to the centre of the sample. In the transition area where the alloy was strongly plastically deformed, oxidation takes place and the indexation quality is degraded (Fig. 2c). Finally, in the area far from the surface, the material is subjected to thermal grooving while the oxidation level remains low: the indexation quality remains thus good (Fig. 2b). Note that the thermal grooving of grain boundaries is observed from 600°C.

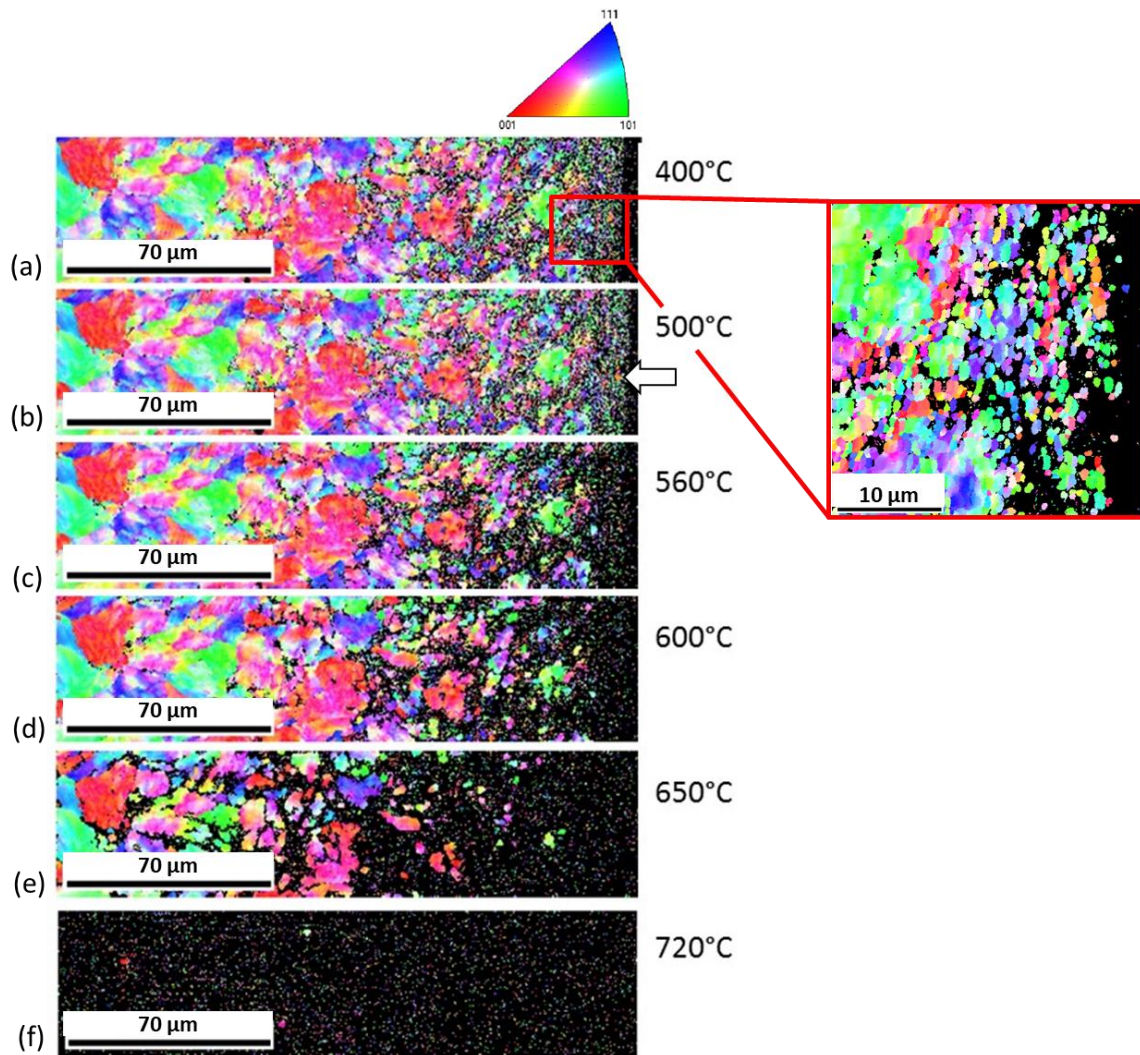


Figure 1 – EBSD maps from (a) to (f) show microstructure at different temperatures. The appearance of dark areas signifies a progressive degradation of indexing quality, especially for the treated surface (indicated by an arrow).

The observed heterogeneous oxidation can be related to the poor oxidation resistance of fine grained materials. As a matter of fact, in the region where the grain size is small, the fraction of grain boundaries is relatively high. It is well known that at grain boundaries, the atomic arrangement is less regular. Grain boundaries can thus provide short circuit diffusion path for oxygen to penetrate more easily into the material. Oxidation preferably begins at the SMATed surface where grain boundary density is high and extends to the bulk where the diffusion kinetics is relatively low. In the case of austenitic stainless steels, another mechanism can be involved in the oxidation process. When these steels are heated to 425- 815°C, chromium carbides can form at grain boundaries. As a result, the narrow areas adjacent to the grain boundaries are highly depleted in chromium and oxidation may easily occur there [14, 15]. It



is worth indicating that the formation of chromium oxide is easier than that of iron oxide for these pressure and temperature conditions, according to the Ellingham diagram [13].

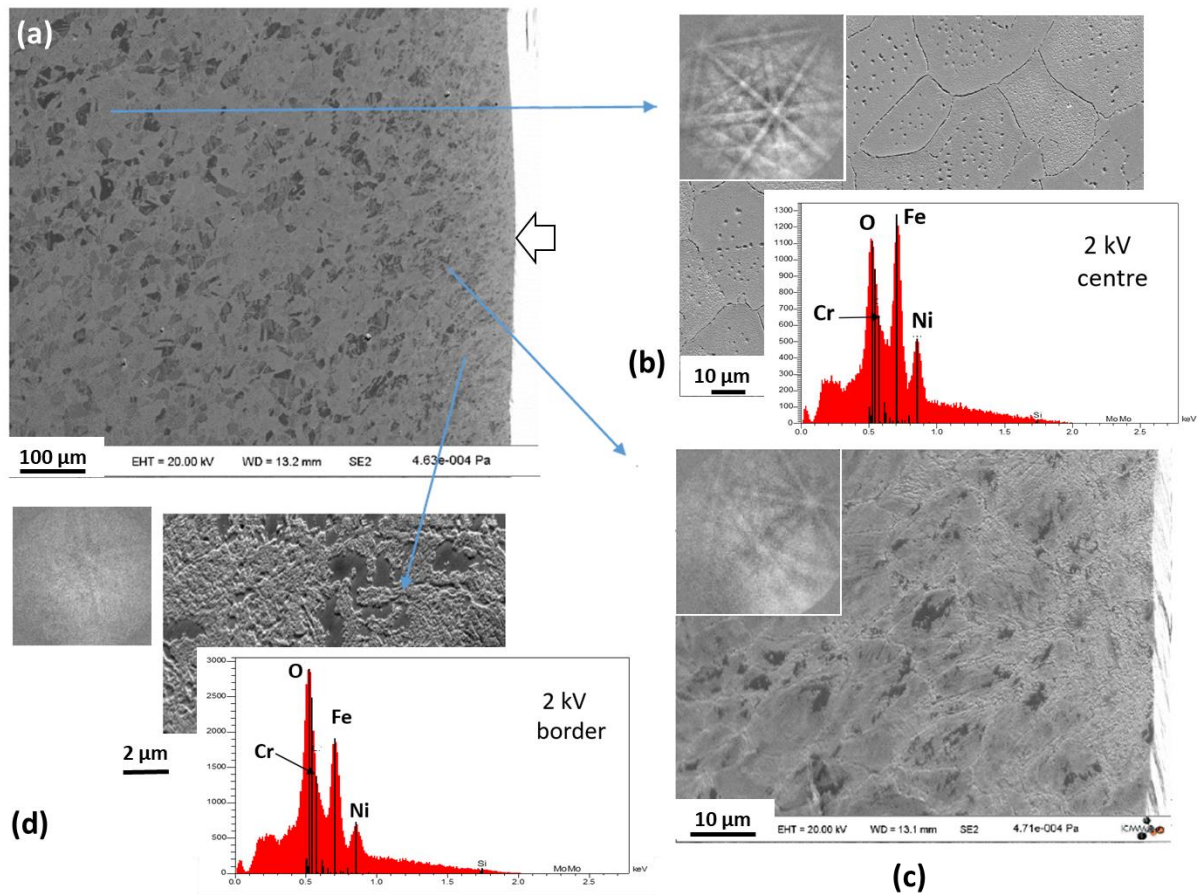


Figure 2 – Micrographs obtained after heating up to 720°C and cooling, EDS analyses and Kikuchi diffraction patterns: (a) global view of a large region (the arrow indicates the surface), (b) bulk region, (c) strongly work-hardened region, and (d) very near surface region.

Contrary to what was initially expected, the microstructure evolution is negligible despite the presence of areas with high stored energy favorable for recrystallization and grain growth. As thermal grooving and oxidation can both hamper the grain boundary migration at the surface, it was decided to verify the microstructure under the oxidized surface. Ionic polishing was therefore carried out a posteriori on the sample heated at 720°C to remove the oxidized layer. After ionic polishing, the indexation quality became much better according to the Kikuchi diffraction pattern shown in Fig. 3. The microstructure looks very similar to that obtained after SMAT, and no notable change in grain size (Fig. 3) and misorientation is observed according to the analyses of the obtained cartographies. This means that the microstructure is not obviously changed by the temperature. The ultrafine grain size remains constant at  $0.7\mu\text{m} \pm 0.2\mu\text{m}$ . This investigation is consistent with the results presented in the literature concerning

the thermal stability of nanostructured layer generated by SMAT. For example, in the studies on 316L steels, grain size change seems to begin at about 600°C [16] or beyond 0.5T<sub>m</sub> (T<sub>m</sub> is the melting temperature) [17].

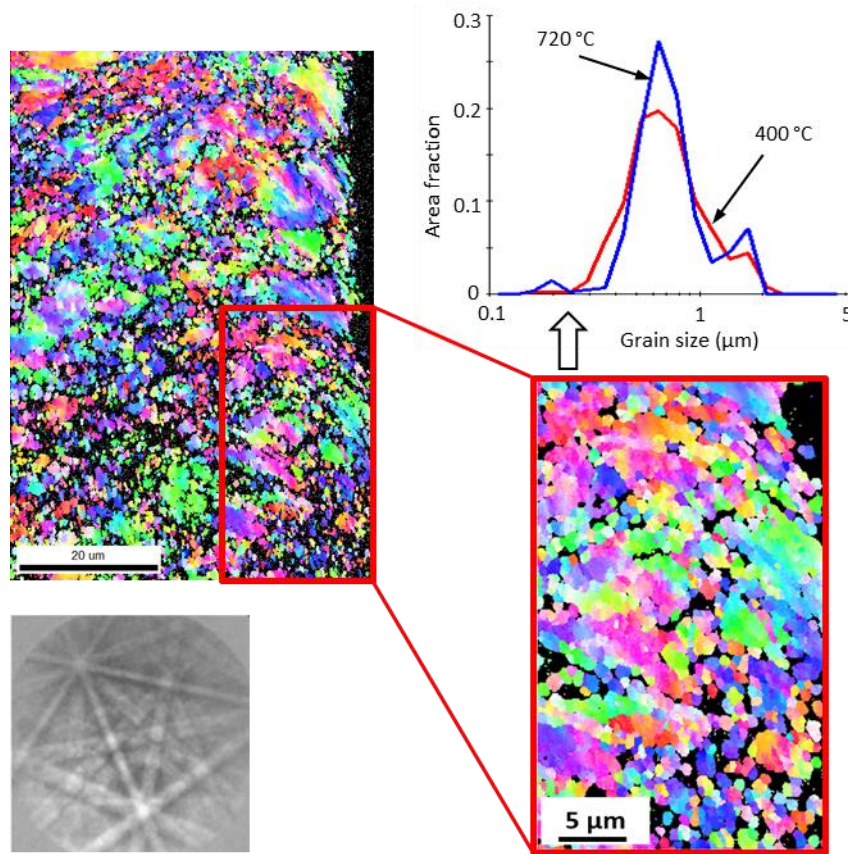


Figure 3 – EBSD observation performed after the removal of the oxidized layer (720°C) using ionic polishing. Ultrafine grain size distribution at the SMATed surface for both 400 and 720°C.

#### 4. Conclusion

In this work, the feasibility of the in-situ EBSD was illustrated by investigating the thermal stability of a 316L stainless steel gradient microstructure generated by SMAT. The initial microstructure obtained after SMAT shows a gradient microstructure from the top surface to the interior of the specimen. The thermal stability investigation shows that the microstructure does not significantly evolve up to 720°C, and especially the small grains are maintained. An oxidation phenomenon takes place and becomes more pronounced when the temperature exceeds 560°C.

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