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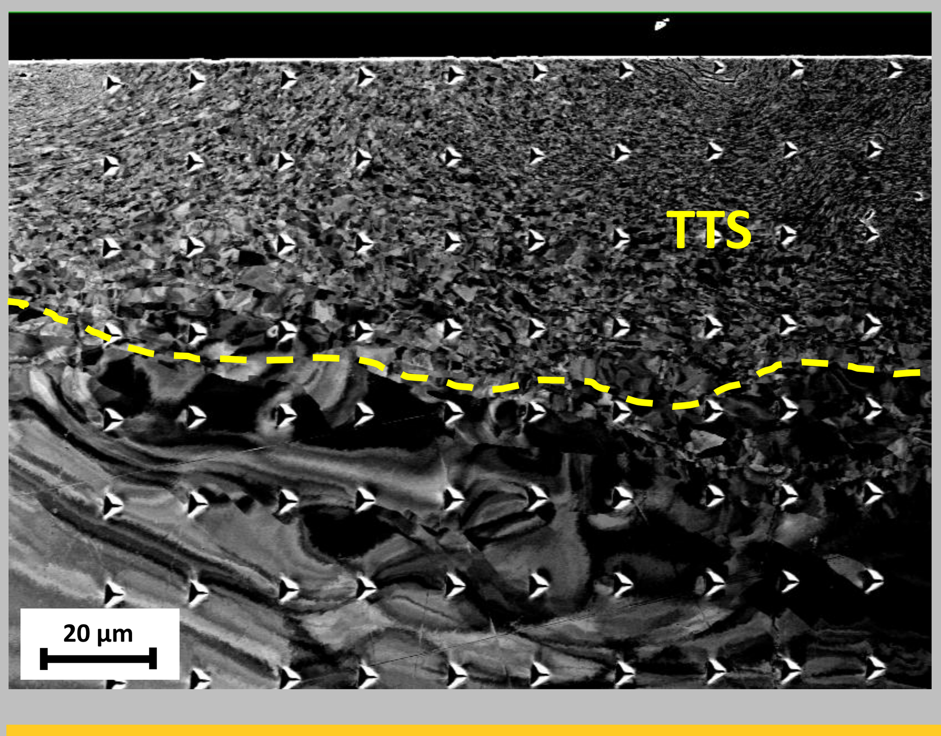


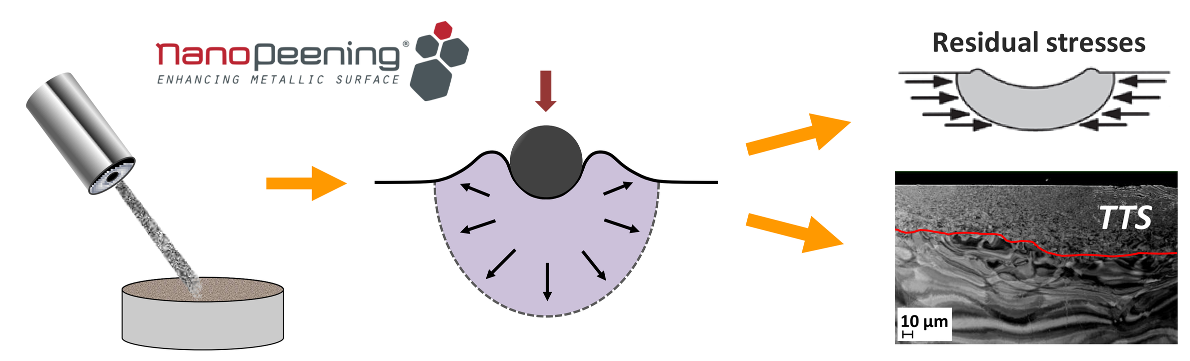
Introduction

In the industry, there exist several techniques which improve the service lifetime of materials by increasing the local mechanical properties at the near-surface. In the case of **mechanical surface treatments** (such as impact-based), the material is exposed to repeated mechanical loadings, producing a severe plastic deformation at the surface, leading to a local refinement of the microstructure in the affected zone (**Tribologically Transformed Surfaces - TTS**) [1]. Consequently, very interesting physical properties such as high hardness and better tribological properties are exhibited in these mechanically-induced transformed surfaces [2].

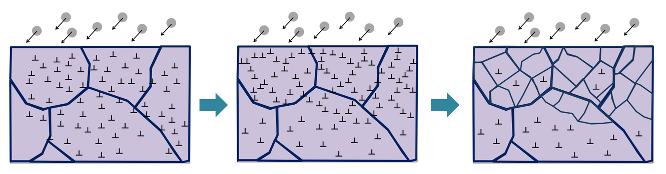
The main issue of this work is to assess and describe precisely the elastic-plastic behavior and the distribution of mechanical properties on deformed zones of a model material (pure iron). A characterization of the transformed microstructure, as well as a statistical analysis of the grain size distribution on the cross-section of the sample is presented first. Next, a methodology based on **nano-indentation** tests and in-situ **micro-pillar compression** tests is implemented to quantify the evolution of mechanical properties. A relationship between the hardness gradient and the microstructure evolution is established, as well as a comparison between the properties measured by both techniques.

Contact loading to produce TTS

Shot peening: Impact-based surface treatment

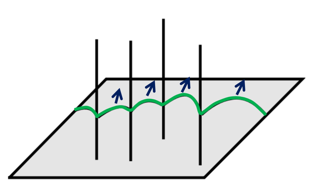


Hardening by increase of GB



Hall-Petch $\sigma_y = \sigma_0 + \frac{K}{\sqrt{d}}$

Dislocation hardening



Taylor's law $\tau = \alpha\mu b\sqrt{\rho}$

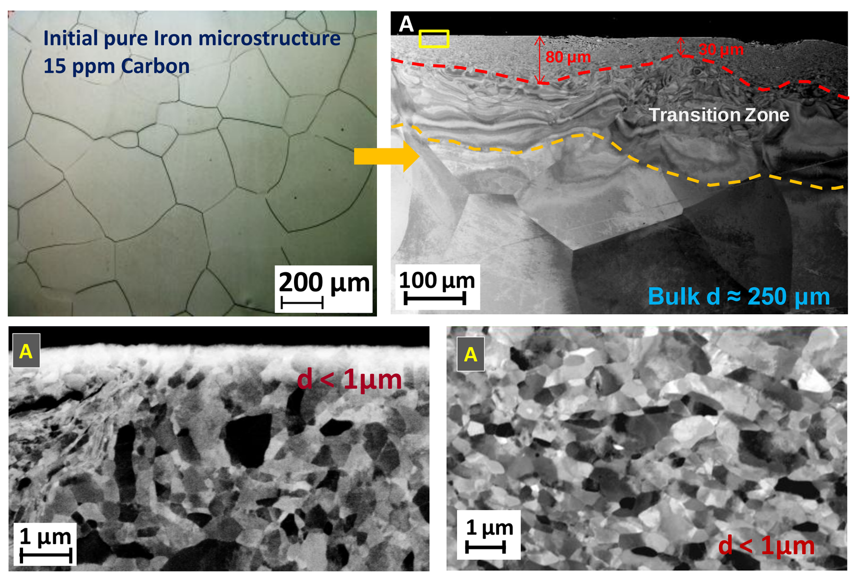
The material is submitted to an industrial shot-peening treatment: NanoPeening® [3]. In this procedure, steel balls (0.1 – 2 mm) are repeatedly projected towards the sample surface with an impact tilt between 10º and 45º at high rates (40 to 100 m/s).

This technique leads to a local microstructure transformation, characterized by a progressive grain size refinement and consequently the formation of a mechanical property gradient over a few tens of microns. The hardening on the near surface is closely associated with the increase of grain boundaries and dislocation density.

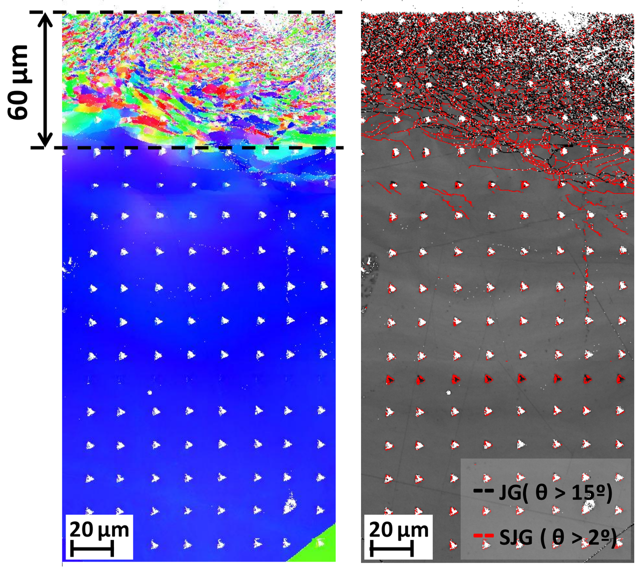
Local refinement of microstructure in pure Iron

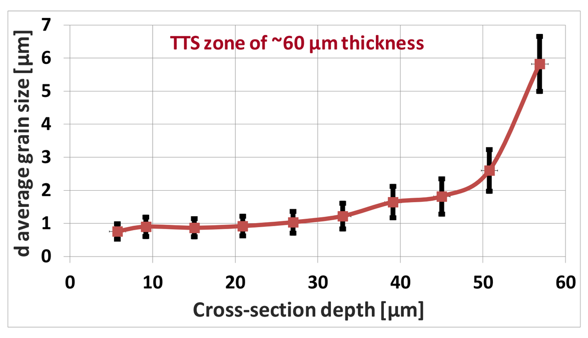
The shot peening treatment is performed on pure α-Iron (15 ppm carbon) without inclusions and with an initial homogeneous grain size of ~250 µm. After treatment, the sample cross-section presents a well-defined TTS region (d<1 µm), followed by a transition zone and the virgin bulk microstructure. An EBSD map was performed to identify the grain size distribution and to correlate it with several nano-indentation tests achieved on the same region.

Characterization of transformed surface

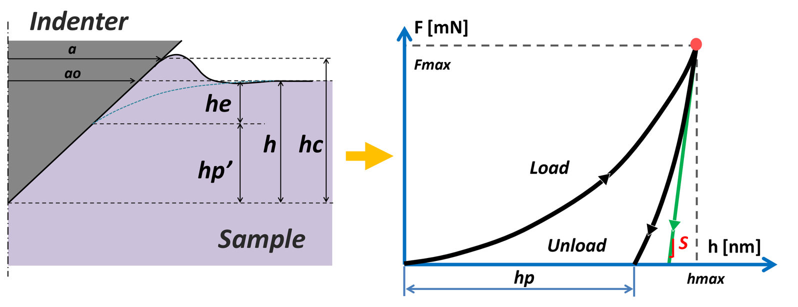


Grain size statistics by EBSD

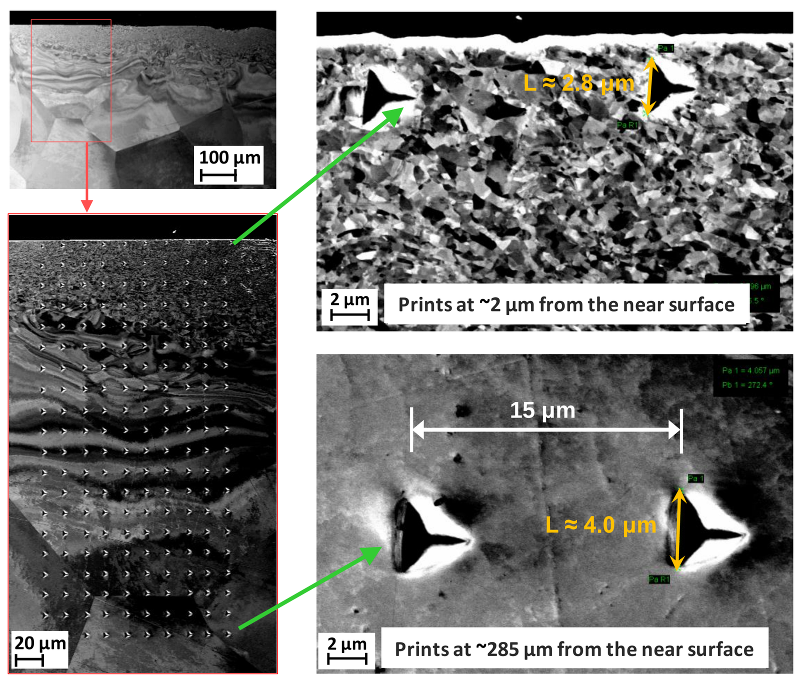


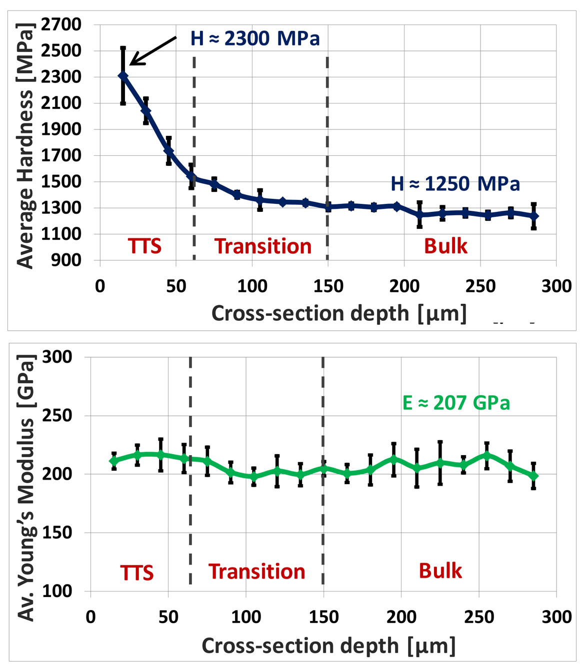


Measurement of properties by Nano-indentation



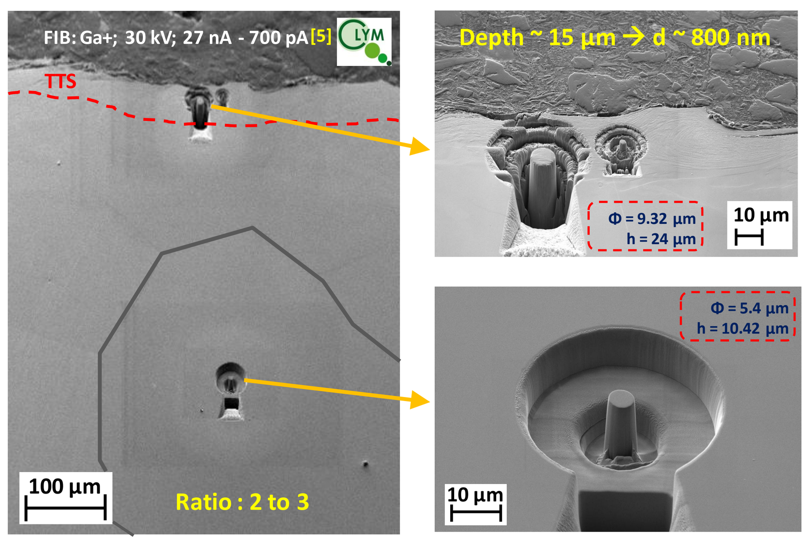
An indentation matrix (10X20) is made at the same EBSD map region. The indentation prints are done with 15 µm spacing, applying 10 mN with a Berkovich tip. Due to the bulge in the indentation profile, the force-displacement data is analyzed with the model of Loubet [4]. The measured hardness decreases 40 % from the near surface, of which 30 % drops over the 60 µm of the TTS zone.

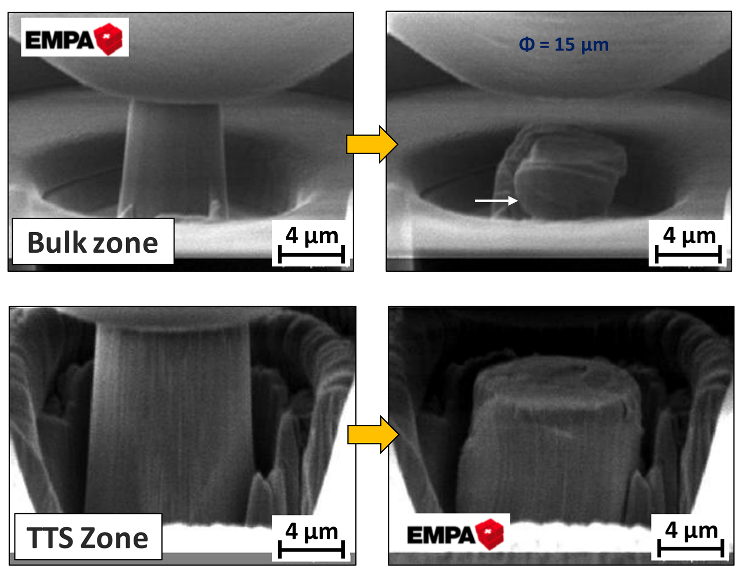


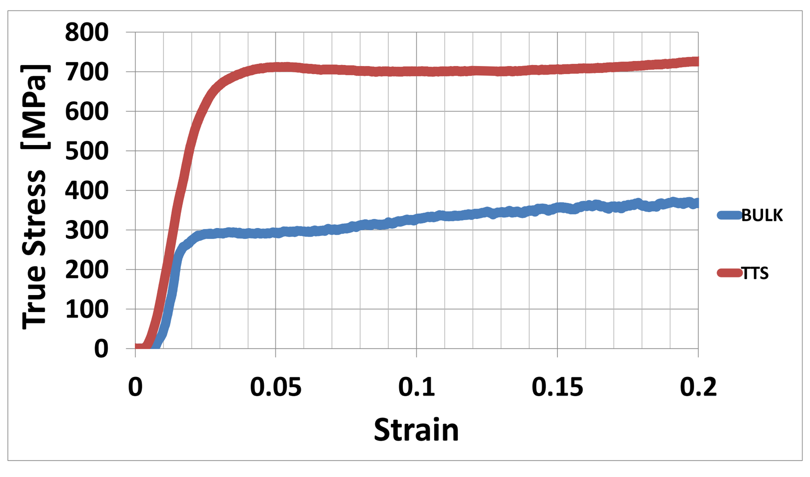


Micro-Compression of pillars in TTS

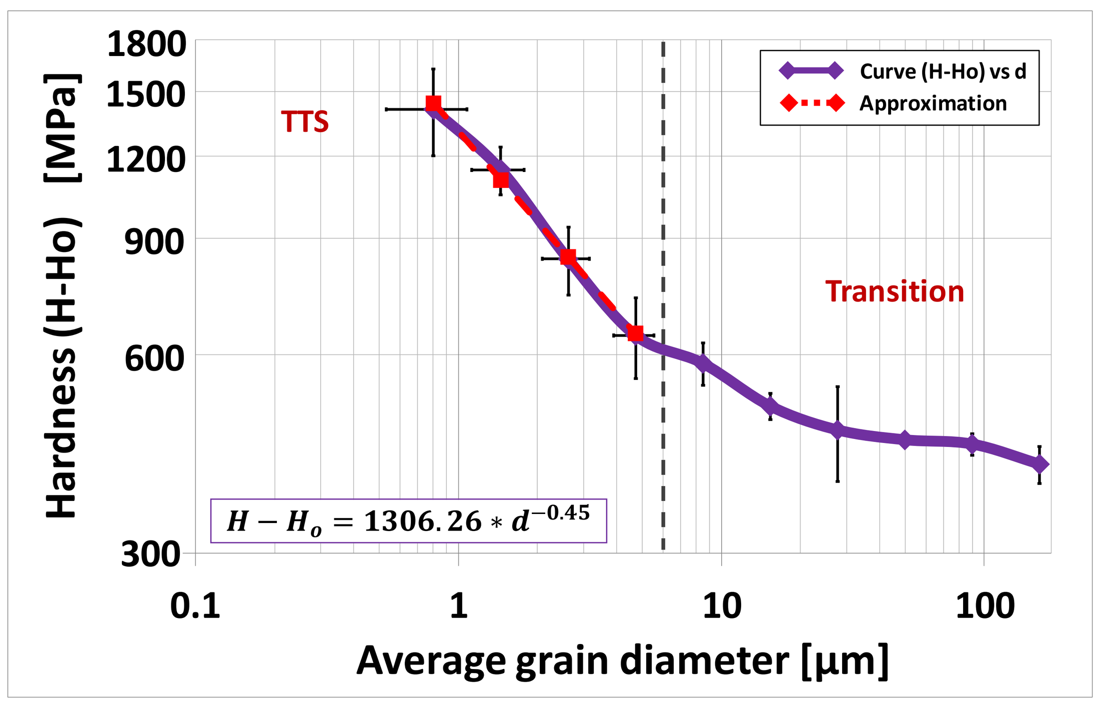
Two micro-pillars compression tests were carried out in the TTS and bulk regions using a 15 µm diameter flat punch. The geometrical ratios of the TTS and bulk pillars are 2.6 and 1.9 respectively. The bulk pillar deforms in a well defined slip plane (white arrow), while the TTS pillar deformation is entirely homogeneous. The stress-strain curves show an increase of yield strength due to the microstructure refinement.







Discussion and Conclusions



Hardness and grain size relationship: Hall-Petch

The hardness gradient quantified by the nano-indentation tests is correlated with the grain size distribution in the TTS zone. Thereupon, the Hall-Petch expression is estimated considering the hardness as three times the stress [6]. In this expression the *Ho* material constant is taken from the bulk material yield strength: *Ho ≈ 3 x 300MPa*. The obtained power law exponent is *-0.45* and the Hall-Petch constant is ~0.44 Mpa m$^{1/2}$.

$$H - H_0 = C_{h-p} * d^m \Rightarrow K_{h-p} = \frac{C_{h-p}}{3}$$

$$K_{h-p} = \frac{1306.26}{3} MPa\ \mu m^{1/2} = 435.42\ MPa\ \mu m^{1/2} \approx 0.44\ MPa\ m^{1/2}$$

Nano-indentation v.s. Micro-compression

The experimental relation proposed by Tabor expresses that the hardness is approximately three times the yield stress. Comparing both micro-mechanical tests, the obtained ratios for the TTS and bulk regions are **~3.3** (2300 MPa/700 MPa) and **~4.2** (1250 MPa/300 MPa) respectively.

Conclusions

- Both mechanical tests demonstrate an increase of mechanical properties (more than 40 %) due to the shot-peening treatment. The nano-indentation and micro-compression results are in good agreement.
- The hardness gradient estimated by nano-indentation corresponds to the grain size refinement in the near surface and they are closely related by the Hall-Petch expression.
- Pure iron is an appropriated model material to obtain a well-defined TTS region in order to compare both methods on the measurement of mechanical property gradients.

Acknowledgement

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