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Quantification of mechanical property gradient by nano-indentation and micro-compression testing on mechanically transformed surfaces

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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 shot peening, 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 [Trębicki et al, 2018]. Consequently, very interesting physical properties such as high hardness and better tribological properties are exhibited in these mechanically-induced transformed surfaces [1].

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
  - Dislocation hardening
  - Taylor’s law

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 10° and 45° 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.

Micro-Compression of pillars in TTS

Two micro-pillar 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 [4]. In this expression the H0 material constant is taken from the bulk material yield strength: \( H = 3 \times 300 \text{MPa} \). The obtained power law exponent is -0.45 and the Hall-Petch constant is \( 1.5 \times 10^2 \text{MPa} \). 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 \( 3.3 \text{ or } 3.0 \text{ GPa/MPa} \) and \( 4.2 \text{ or } 3.8 \text{ GPa/MPa} \) 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.

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Related references

[5] D. Tumbajoy, S. Descartes, B. Spinel, A. Trębicki, J.M. Bergeaheu, In situ twin boundary transformation at high rates (40 to 100 m/s). In this procedure, steel balls (0.1 – 2 mm) are repeatedly projected towards the sample surface with an impact tilt between 10° and 45° at high rates (40 to 100 m/s).

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