Investigation of graded strengthened hyper-deformed surfaces by impact treatment: micro-percussion testing

David Tumbajoy-Spinel\textsuperscript{1,2}, Sylvie Descartes\textsuperscript{1}, Jean-Michel Bergheau\textsuperscript{3}, Halim Al-Baida\textsuperscript{1}, Cécile Langlade\textsuperscript{4}, Guillaume Kermouche\textsuperscript{5}

\textsuperscript{1}Université de Lyon, INSA-Lyon, LaMCoS, CNRS UMR5259, Villeurbanne F-69621, France
\textsuperscript{2}Université de Lyon, École des Mines de Saint-Étienne, LGF, CNRS UMR5307, Saint-Étienne F-42023, France
\textsuperscript{3}Université de Lyon, ENISE, LTDS, CNRS UMR5513, Saint-Étienne F-42023, France
\textsuperscript{4}Université Technologique de Belfort-Montbéliard, IRITES-LERMPS EA 7274, 90010 Belfort, France

E-mail: david.tumbajoy-spinel@insa-lyon.fr

Abstract. In the industry, mechanical surface treatments could improve the mechanical behaviour of materials by the means of local hyper-deformation and graded strengthening. Micro-percussion test represents an interesting case scenario to emulate these kinds of conventional treatments (shot-peening, SMAT, roller-burnishing, etc) and go further on microstructural and mechanical characterization at local and global scales. For this technique, every impact is made at the same position by a rigid conical indenter, controlling the number, angle and velocity of impacts. The main issue of this work is to establish a complete description of the transformed microstructures; to understand the mechanisms involved on the formation and growth of refined structures; to make a parametric sensitivity analysis of different impact conditions.

1. Introduction
The mechanical surface treatments confer better local mechanical properties against wear or fatigue service conditions. In the case of conventional impact treatments (shot-peening \cite{1,2}, SMAT \cite{3}), the material is exposed to repeated mechanical loadings, producing a severe plastic deformation (SPD) in the near-surface. It leads to a local progressive refinement of the microstructure over a few tens of microns \cite{2,3}, commonly known as Tribologically Transformed Structure (TTS) \cite{4}. In this work, a model surface treatment (micro-percussion \cite{5,6}) is used to explore several impact conditions (angle and amount of impacts) and its effects on the microstructural transformation. In this work, three main goals are considered: (i) characterize the transformed surfaces and evaluate the possibility to emulate industrial techniques (ii) make a parametric analysis, (iii) describe the microstructural transformation process and the mechanisms involved on it. This investigation is carried out in pure $\alpha$-iron.

2. Process and Methods: micro-percussion tests in pure $\alpha$-iron samples

2.1. Material: $\alpha$-iron
The micro-percussion tests are done in a high-purity $\alpha$-iron with less than 15 ppm of carbon. This material is produced by the cold crucible melting method. The resulting metallic bar is thermomechanically treated by forging and annealing at 800 °C during 60 minutes. This treatment produces a
homogeneous microstructure with equi-axed grains (average size in the order of 1 mm). No inclusions have been observed in this model material.

2.2. Micro-percussion tests
The principle of micro-percussion tests [5]-[6] is to carry out several impacts in the same position of the sample with a rigid conical punch (carbide tungsten). For metallic materials, the SPD produces a remaining print of millimetric diameter. The indenter tip has a spherical shape with a radius of 0.5 mm and an angle of 60° (Figure 1-a). A schematic representation of the test is shown in Figure 1-b. The impact set-up permits to control the number and velocity of impacts [7]. The impact angle is introduced directly on the sample geometry (tilted plane). Three angles were explored for the impact conditions: 0°, 15° and 30°. At each angle, the prints are done for different amounts of impacts: 10, 30, 100, 300, 1000, 3000 and 10000. Prints are arranged in a rectangular matrix (2x4), spaced of 2 mm for each line and 3 mm between both lines (Figure 1-b). Only the 10000 impacts print is spaced of 4 mm because of its high diameter. Two main conditions were considered to make a parameter sensitivity analysis: (i) the angle and (ii) the number of impacts. Concerning the velocity of impacts, some preliminary tests pointed out that the set-up working range for α-iron is enclosed between 80 mm/s and 200 mm/s [7]. Then, the velocity used for all the tests was in the order of 150 mm/s.

![Figure 1.](image)

**Figure 1.** (a) Micro-percussion punch. (b) Schematic representation of the micro-percussion tests for three angles (0°, 15°, 30°) and seven impact amounts (10, 30, 100, 1k, 3k, 10k) at 150 mm/s.

2.3. Near-surface microstructural characterization
The characterisation of the resulting prints was done using scanning electron microscopy (SEM) and electron back-scattered diffraction (EBSD) mapping. Both techniques were carried out on the print cross-section in order to observe the near-surface microstructural transformation. For both methods, the samples were cutted with a metallic wire saw and then mechanically polished up to the middle plane of each print. The sample surfaces were polished with several abrasive papers (P240 to P1200), two diamond suspensions (3 μm and 1 μm) and finished with colloidal silica. The SEM images were done in a Zeiss Supra 55 VP at 20 kV using a back-scattered detector (BSE). The EBSD maps were carried out in a Tescan Lyra3/XMU (FEG/FIB) using an indexation step of 2 μm.

3. Results: mechanical surface treatment by micro-percussion

3.1. Characteristics of a print: different deformed regions
An example of a characteristic print is shown in Figure 2-a (10000 impacts at 15°). This print has a diameter of 1750 μm and a non-axisymmetric shape due to the tilt of the impact direction. Different regions are identified in the cross-section of the print. The entire mechanically affected zone (MAZ) [8] has a thickness of hundreds of microns (430 μm). The virgin bulk microstructure (initial milimetric grains) is observed in the outside of this region (MAZ). Moreover, two main layers are observed in the
mechanically affected zone (MAZ). On the one hand, the mechanically attrited structure (MAS) [6] is essentially characterized by the presence of high local misorientations and new well-defined grain boundaries [9]. On the other hand, the low angle boundaries (LAB’s) zone corresponds to a non-microstructured region in spite of the plastic deformation (Figure 2-a) [10]. Indeed, some previous works [11]-[12] revealed that this kind of region has some slight crystal orientation nuances and it could be considered as a single crystal zone due to the initial grain sizes in the bulk material.

Furthermore, the MAS region (total thickness of 150 μm) is defined by the TTS and the transition zones (Figure 2-b). The tribologically transformed structure (TTS) corresponds to the nearest surface layer with a progressive grain size refinement in depth [4]. The transformed microstructure in this case is considerably similar to the TTS regions obtained by other surface treatments (shot peening [2],[12],[14], SMAT [3],[15], roller-burnishing [16]-[17]). Beyond the TTS layer, the transition zone is characterized by the presence of shear bands (white arrow) [13] and sub-grain boundaries (not well-formed grains) [9]. This zone is an intermediary region between the well-refined microstructure (TTS) and the “single crystal” region deformed at low strain rates (LAB’s zone).

**Figure 2.** (a) Description of the different regions observed in the cross section of the print formed at 10000 impacts and 15°. (b) Detail of the **mechanically attrited structure** (MAS).

### 3.2. Kinetics of the TTS layer transformation

The wide thickness and homogeneity of the TTS layer observed in Figure 2-a,b is definitively an interesting case scenario to emulate and investigate the transformed surfaces by the means of industrial impact treatments. However, the impact conditions will be decisive to create a significant TTS layer for this purpose. For example, as shown in Figure 3, three different prints were formed at 10000 impacts for different impact angles: (a) 0°, (b) 15° and (c) 30°. The normal impacts (0°) produce a shallow transformed region with less than 50 μm in-depth. On the contrary, the tilted impacts (15° and 30°) can produce TTS layers three times wider (more than 120 μm in-depth). Moreover, the TTS layer at 0° presents more surface heterogeneities, crack formations (white arrows) and material release [18].

**Figure 3.** Cross-section of the TTS layer for 10000 impacts at (a) 0°, (b) 15° and (c) 30°.
Figure 4. TTS layer growth at different amounts of impacts (30°): (a) 10, (b) 100, (c) 1000 and (d) 10k. Global views of 10k impact prints are compared for three different angles: (d) 30°, (e) 15° and (f) 0°. Local views in the near-surface are presented for the prints formed at (g) 10 and (h) 10k impacts.

The evolution of the TTS layer is shown in Figure 4 as a function of the impact amounts for 30°: (a) 10, (b) 100, (c) 1000 and (d) 10000 impacts. The TTS layer and the print diameter grow progressively with the number of impacts. The microstructural refinement leads sub-micrometric grain sizes (d < 1 μm) in the TTS layer for the highest amounts of impacts (Figure 4-h). On the contrary, the TTS layer is almost invisible beneath 100 impacts and the formation of well-defined grains is essentially nonexistent (Figure 4-g). Indeed, the mechanical deformation of few impacts is not enough to refine the near-surface microstructure [10],[13]. Likewise, considering the same number of impacts (10000) at different angles (0°, 15°, 30°), the refined microstructure and the TTS layer evolves differently (Figure 4-d,e,f). As shown in Figure 3, the tilted impacts (15°, 30°) are more propitious to have wider transformed regions.

Figure 5. Parametric analysis of (a) the TTS layer thickness and (b) the print diameter as a function of the number of impacts at 0°, 15° and 30°.
Two parametric graphics are presented in Figure 5-a,b in order to quantify the impact condition sensitivity on the TTS layer formation as a function of the number of impact [6]: (a) the thickness of the TTS layer and (b) the mean diameter of the print. The first graphic (Figure 5-a) shows that the thickness of the TTS layer evolves following a power law behaviour. Indeed, the transformed region grows progressively up to 3000 impacts and then it stabilizes in a plateau. As observed in the SEM images presented below, the higher widths of the refined layer are measured for the cases of tilted impacts (15° and 30°). As shown in this graphic (Figure 5a), the thickness of these TTS layers could be more than twice the transformed region with normal impacts (0°). Likewise, it is particularly interesting that the curves for 15° and 30° are juxtaposed. The second graphic (Figure 5-b) shows the growth of the mean diameter in a log-log scale. The print average diameter evolves in the same manner for all the impact angles (0°, 15°, 30°). It would mean that the impact tilt only affects the width of the refined region and not the global size of the print, in spite of the bulge formed on the side.

4. Discussion

The parametric analysis pointed out that the impact angle has a relevant local effect on the microstructural refinement without changing the global dimensions of the print. This could mean that the impact tilt is strongly related with the microstructural transformation mechanisms in the material due to the SPD [9],[13]. To disclose this question, two sequences of the TTS layer growth at 15° are presented in Figure 6: (a, b, c) SEM images and (d, e, f) EBSD maps. In both cases, the transformed layer grows from the external side of the print and evolves progressively further below to the center, just underneath the normal impact axis. It suggests that the microstructural transformation starts from a shearing contact process (white arrows) and spreads in-depth (red and black arrows) with a combined phenomenon of shear and normal impact. Considering that shear bands and dislocation cells are quite significant for the refinement process in high stacking fault energy materials (as pure α-iron [3]), it is not surprising that shear contact and normal impact take a major role on the near-surface transformation [9]-[10]. Furthermore, the EBSD maps show that well-defined grains are not formed anymore outside the TTS region. Indeed, the remaining bulk grains beneath the refined layer perceive a low crystal misorientation in spite of the SPD induced in the mechanically affected zone (MAZ).

Figure 6. Sequence of the TTS layer formation and growth observed by the means of (a, b, c) SEM imagery (BSE detector) and (d, e, f) coarse EBSD mapping (2 μm indexation step).

5. Conclusions

A parametric investigation had been presented in this work in order to understand the effects of different impact conditions (micro-percussion) on the microstructural refinement of hyper-deformed
surfaces. The main results had shown that tilted impacts ($\theta >> 0^\circ$) could produce TTS layers much wider (at least twice) than those formed by normal impacts ($\theta = 0^\circ$). Furthermore, it had been pointed out that the refined layer (sub-micrometric grains) grows progressively with the number of impacts. However, the microstructural transformation is done by a combined effect of shearing contact and normal impact, probably related with the microstructural refinement mechanisms in high stacking fault energy materials (pure $\alpha$-iron): (i) the shear bands and (ii) the dislocation mobility [10].

From this work, micro-percussion tests could be an interesting method to emulate the transformed surfaces obtained by the means of conventional techniques (as shot-peening or SMAT). Firstly, the refined microstructure in the near surface (for $\alpha$-iron) is absolutely comparable with the one obtained using industrial procedures [11],[12],[14]. Secondly, the size of the transformed layer is wide enough to be compared with conventional treatments and to analyse the microstructural and the mechanical gradients with several characterization techniques (as SEM, EBSD, nano-indentation, micro-pillar compression, etc.) [11]-[12]. Furthermore, this work highlights that $\alpha$-iron is an appropriate model material to obtain well-defined TTS and it is favourable for experimental characterization methods. For a future work, it would be interesting to go further on the local mechanical characterization (nano-indentation [19] and micro-pillar compression [15]) and match it with the microstructural characterization presented above. This coupled experimental analysis would reveal the influence of different strengthening effects in surfaces as the Hall-Petch effect or the work hardening [20]-[21].

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References
[1] Nordin E and Alfredsson B 2016 *Journal of Materials Processing Technology* 235 143–148
[2] Marteau J and Bouvier S 2016 *Surface & Coatings Technology* 296 136–148
[3] Lu K and Lu J 2004 *Materials Science and Engineering A* 375–377 38–45
[4] Descartes S, Busquet M and Berthier Y 2011 *Wear* 271 1833-1841
[5] Al-Baida H, Langlade C, Kermouche G and Ambriz R-R 2015 *J. Mater. Res.* 30 2222-2230
[6] Kermouche G, Paaquaut G, Langlade C and Bergeheau J-M 2011 *C. R. Mecanique* 339 552–562
[7] Al-Baida H, Langlade C, Kermouche G and Ambriz R-R 2014 *Materials & Techniques* 102 604
[8] Juran P, Liotier P-J, Maurice C, Valiorgue F and Kermouche G 2015 *C. R.Mecanique* 343 344–353
[9] Tan N-R, Wang Z-B, Tong W-P, Sui M-L, Lu J and Lu K 2002 *Acta Materialia* 50 4603–4616
[10] Sakai T, Belyakov A, Kaibyshev R, Miura H and Jonas J 2014 *Progress in Materials Science* 60 130–207
[11] Tumbajoy-Spinel D, Kermouche G, Descartes S, Bergeheau J.-M, Lacaille V, Guillonneau G and Michler J 2015 *Matériaux & Techniques* 103 303
[12] Tumbajoy-Spinel D, Descartes D, Bergeheau J-M, Lacaille V, Guillonneau G, Michler J and Kermouche G 2016 *Materials Science and Engineering A* 667 189–198
[13] Segal V-M 2005 *Materials Science and Engineering A* 406 205–216
[14] Lacaille V, Kermouche G, Tumbajoy-Spinel D, Feulvarch E, Morel C and Bergeheau J-M 2014 *IOP Conf. Series: Materials Science and Engineering* 63 012124
[15] Sun Z, Retraint D, Guelorget B and Waltz L 2015 *Matériaux & Techniques* 103, 304
[16] Okada M, Suenobu S, Watanabe K, Yamashita Y and Asakawa N 2015 *Mechatronics* 29 110–118
[17] Jacquet G, Kermouche G, Courbon C, Tumbajoy-Spinel D and Rech J 2014 *IOP Conf. Series: Materials Science and Engineering* 63 012039
[18] Descartes S, Desrayaud C, Niccolinic E and Berthier Y 2005 *Wear* 258 1081–1090
[19] Deng S-Q, Godfrey A, Liua W and Hansen N 2016 *Scripta Materialia* 117 41–45
[20] Frutos E, Multigner M and González-Carrasco J-L 2010 *Acta Materialia* 58 4191–4198
[21] Zou Y, Maiti S, Steurer W and Spolenak R 2014 *Acta Materialia* 65 85–97