LOW CARBON STEEL WITH NANOSTRUCTURED SURFACE LAYER INDUCED BY HIGH-ENERGY SHOT PEENING

G. Liu¹, S.C. Wang¹, X.F. Lou¹, J. Lu² and K. Lu¹

¹ State Key Laboratory for RSA, Institute of Metal Research, Chinese Academy of Science, Shenyang 110015, China ² LASMIS, University of Technology of Troyes, 10000, Troyes, France

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Introduction

Mechanical properties of engineering materials can be improved by refining their microstructures. In the past decade, various kinds of advanced techniques by using intense plastic deformation were developed to synthesize ultra-fine grained materials, such as equal-channel angular pressing (1,2), cold-rolling (3,4) and torsion straining (5). However, most of these techniques are still difficult for practical application to conventional engineering materials.

It is known that most failures of engineering materials are very sensitive to the structure and properties of material surface, and in most cases material failures occur on the surface. Therefore, optimization of surface structure and properties may effectively improve the global behavior of material. With the increasing evidences of unique properties for nanostructured materials, it is reasonably expected to achieve surface modification by generation of a nanostructured surface layer, i.e. surface nanocrystallization (SNC) (6). By using the ultrasonic shot peening (USP) method (7), nanostructured surface layers were successfully obtained on a 316L stainless steel and the pure Fe samples (8,9). In this work, we report the synthesis of a nanostructured surface layer on a low carbon steel by using a high-energy shot peening (HESP). The microstructural evolution was characterized by means of different techniques, and the change of mechanical properties after the HESP treatment was analyzed.

Experimental

The materials used in this work is a low carbon steel plate of 1.5 mm thick, its chemical compositions contain (mass, %) 0.11C, 0.24Si, 0.35Mn, 0.018P and 0.014S. The initial grain size of the low carbon steel is in the range of 20 ~ 40 μm. Samples of 100 × 100 mm² were prepared for the HESP treatment.

The principle of the HESP treatment used in the present work is similar to that of the USP method (9), but with a lower frequency (3 kHz) and bigger shots (diameter of 8 mm). The entire surface of the sample to be treated is peened by the flying shots with a high energy. The processing durations are 30, 60, 90 and 180 min, respectively.

Surface structure of peened samples and structural evolution along the depth of the sample peened for 180 min were studied using X-ray diffraction (XRD) with CuKα radiation in a Rigaku D/max-2400 X-ray diffractometer. The average grain size and the mean microstrain were calculated from XRD line

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broadening of six Bragg reflection peaks of bcc-Fe in terms of the Scherrer-Wilson equation (10). The morphologies of cross-section and surface layer of the sample after the HESP treatment for 180 min were examined using optical microscopy and transmission electron microscopy (TEM) (Philip EM420) respectively. Tensile tests were carried out using a rigid tensile machine (40 KV Hydroplus Test System) at room temperature, and the 0.2% offset was selected as yield strength.

Results and Discussion

XRD patterns of top surface layer of samples before and after the HESP treatments are shown in Fig. 1. There is evident broadening of Bragg reflection profiles, which can be attributed to the crystalline imperfection induced by small grain size and microstrain. The structural parameters calculated from the XRD data are listed in Table 1. After the HESP treatment for 30 min, the average grain size in the top surface layer was found to be about 33 nm. With an increase of the treatment duration, the average grain size reduces slightly. The mean microstrain was found to be about 0.01–0.1%. The values of average grain size and mean microstrain derived from XRD data are similar to that of pure Fe (9) treated using the USP method.

Structural parameter variation along the depth from the top surface were calculated from the XRD data for the sample peened for 180 min, layers of different thickness were removed chemically by using HF+H₂O₂+H₂O solution, and the results are listed in Table 2. From the top surface to about 20 μm depth, the average grain size increases to about 46 nm. With a further increment of the depth, the average grain size enhances more rapidly to the micrometer regime. On the other hand, the mean

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### TABLE 1

<table>
<thead>
<tr>
<th>Processing Time (min)</th>
<th>D (nm)</th>
<th>$&lt;e^2&gt;^{1/2}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>33 ± 3</td>
<td>0.0085 ± 0.0021</td>
</tr>
<tr>
<td>60</td>
<td>28 ± 3</td>
<td>0.0860 ± 0.0027</td>
</tr>
<tr>
<td>90</td>
<td>23 ± 2</td>
<td>0.0684 ± 0.0039</td>
</tr>
<tr>
<td>180</td>
<td>27 ± 3</td>
<td>0.0979 ± 0.0028</td>
</tr>
</tbody>
</table>
microstrain shows a sharp drop within the range of 10–20 μm depth. Therefore, it is reasonably estimated that the thickness of the nanostructured surface layer is not less than 20 μm.

For the sample treated for 180 min, the cross-section morphology is shown in Fig. 2. Deformation evidences can be observed at about 50 μm deep from the top surface, which can be referred as the deformation region. The plastic flows in the deformation region are found to be along one direction, which indicates that repeated mechanical loads acted on material surface are statistically concentrated in some special directions. TEM observations of the top surface layer are shown in Fig. 3. From the dark field image, one can see nanocrystallized grains of which the shape is roughly equiaxed. The average grain size in the top surface layer is approximately 8 nm, which is smaller than that obtained from the XRD calculation. The difference might be due to the fact that the XRD result averages the structure information of a surface layer of about 6 μm thick (for 95% absorption of CuKα radiation), while TEM sample of top surface layer is a very thin film (less than 1 μm thick). As shown in previous work and wear test (8,11), grain refinement is regionally inhomogeneous, there exists a grain size change which progressively increases with the distance from the peened surface.

Tensile properties of the samples before and after the HESP treatment are shown in Fig. 4. It is noticed that a pronounced increment of the yield strength without significant reduction of the elongation can be achieved for the sample peened for 30 min. With a further increase of the treatment duration, both of the yield strength and the elongation approach saturated values. The yield strength can be enhanced by about 35% (relative to the original material), and the elongation just reduced by 4% (from 38.6% to 34.6%). The strengthening after the HESP treatment might be due to work-hardening and surface nanostructure, of which the contributions can not be separated at this stage. Evidently, the increment of the strengthening is not as significant as that of bulk ultra-fine grained low carbon steel.

### TABLE 2

<table>
<thead>
<tr>
<th>Depth (μm)</th>
<th>D (nm)</th>
<th>$\langle \varepsilon^2 \rangle^{1/2}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>32 ± 3</td>
<td>0.0877 ± 0.0026</td>
</tr>
<tr>
<td>10</td>
<td>36 ± 4</td>
<td>0.0826 ± 0.0032</td>
</tr>
<tr>
<td>20</td>
<td>46 ± 6</td>
<td>0.0290 ± 0.0069</td>
</tr>
<tr>
<td>40</td>
<td>&gt;1000</td>
<td>0.0002 ± 0.0001</td>
</tr>
</tbody>
</table>

Figure 2. Optical observation on the cross-section of the sample after the HESP treatment for 180 min.
(12), nevertheless the thickness of the nanostructured surface layers is only about 3 percent of the total sample thickness.

Though the SNC and the strengthening mechanisms need to be clarified by more experimental evidences, it is noteworthy that the SNC may effectively enhance yield strength without considerable degradation of ductility and toughness. Therefore, it is reasonably expected to utilize the SNC to improve the mechanical property of engineering materials by means of the HESP technique.

**Summary**

Nanostructured surface layer can be successively obtained in a low carbon steel plate by means of the HESP technique. The average grain size in the top surface layer can be as small as a few nanometers, and gradually increases with the distance from the surface. The thickness of the nanostructured surface layer is not less than 20 µm. The yield strength was found to be significantly enhanced without considerable degradation of ductility and toughness. The strengthening of material after the HESP treatment can be attributed to work-hardening and formation of surface nanostructure. The present results show the possibility to significantly improve mechanical property of engineering materials by generation of a nanostructured surface layer induced by the HESP technique.

![Figure 3. TEM image of the top surface layer after the HESP treatment for 180 min.](image)

![Figure 4. Tensile properties vs. the HESP treatment duration for the low carbon steel samples.](image)
Acknowledgment

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References