Original Article

High mechanical performance of AISI304 stainless steel plate by surface nanocrystallization and microstructural evolution during the explosive impact treatment

Huhe Wang*, Zhiming Shi, Xinba Yaer, Zheng Tong, Zhaoxin Du

School of Materials Science and Engineering, Inner Mongolia University of Technology, Hohhot 010051, PR China

A R T I C L E   I N F O

Article history:
Received 5 December 2017
Accepted 23 May 2018
Available online xxx

Keywords:
Explosive impacting
AISI304 stainless steel
Surface nanocrystallization
Grain refinement

A B S T R A C T

The surface treatment on AISI304 stainless steel plate was successfully carried out by explosive impacting technique. We have obtained a high mechanical performance and corresponding fine microstructures compared to its base metal by the technique. Experimental results indicate that nanograins (around 30 nm of diameter) with random crystallographic orientations formed in the top impacted surface and many deformation bands in the subsurface layers. The explosive impact generated deformation induced martensite (DIM) in the plates, where DIM volume fraction increased firstly and then decreased toward inside. These surface nanocrystallization and microstructural evolution, which can be attributed to the co-actions of the grain-refinement, deformation-bands and phase transformation effects, can enhance surface hardness and mechanical properties remarkably not only near surface but entire plate.

© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

AISI304 stainless steel has been widely used in the fields of automotive, machinery, nuclear, petrochemical engineering, etc. [1–3] due to its excellent combination of strength, ductility and response to good corrosion resistance. However, its further applications have been restricted by its relatively inferior surface hardnes and the fact that hard to hardening by heat treatment. Fortunately, surface self-nanocrystallization is an effective method for improving surface hardness of AISI304 stainless steel [4]. The coarse grains can be gradually refined into nanocrystalline through introducing the severe plastic deformation in the treated surface by mechanical activation, i.e., surface mechanical attrition treatment (SMAT) [5–9], supersonic particle bombardment (SFPB) [10], ultrasonic shot peening [11], laser shock peening (LSP), etc. [12,13]. The available literatures about surface treatment of AISI304 stainless

* Corresponding author.
E-mail: huhe2009@imut.edu.cn (H. Wang).
https://doi.org/10.1016/j.jmrt.2018.05.010
2238-7854/© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

steel plates, e.g., SMAT [5,7,14] have reached 100% deformation induced martensite (DIM) on the top surface due to the repeated impact by steel ball in the low strain rate (10^{-6}–10^{-9}), and then the volume fraction decreased with the increase of depth from the top. Having said that, the martensite content treated by SMAT was closely related to processing time on the surface [5,15] and 100% DIM could be received on the treated surface nothing but processing time is long enough.

As known, although above-mentioned technologies can remarkably improve surface hardness by nanocrystallines, these cannot effectively enhance the comprehensive performance of the entire plates. The maximum thickness of hardened layer obtained by these technologies reached to 180–300 μm in the reported [5,7–9]. Nevertheless, it is insignificant for thicker plates (~3 mm) requiring outstanding surface properties with the excellent strength of entire plates in many applications such as vessel or aerospace components [16,17]. Therefore, to explore an effective method on surface nanocrystallization with strengthening of entire plates becomes an urgent and imperative issue. The explosive impact treatment is a unique processing technique, which can generate severe plastic deformation on the surface of material and receive the surface nanocrystallization [18–24]. L. Fouillaud-Pallè et al. [19] investigated the effects of explosive treatment on the fatigue life cycles of steel, indicating that the fatigue life of the treated one was seven times longer than non-treated one. This behavior is closely related to the microstructure and the state of the residual stresses. M. Vesenjak et al. [20] discussed mechanical properties and microstructure of copper before and after the explosive treatment. They also found that the explosive treatments resulted in intensive fragmentation of grains, and therefore led to increase of Young’s modulus and yield stress. F.C. Liu et al. [21] compared the microstructures and the work hardening behavior of high manganese steels which was performed both by explosion hardening treatment and static compression, deducing that the mechanism of explosion impact treatment was “in-situ” plastic deformation. F.C. Zhang et al. [22] conducted explosion hardening on high manganese steel and obtained the optimum explosive thickness and maximum strain hardening layer, suggesting that surface hardening was attributed to the dislocations and twins strengthening. Similarly, the authors have successfully fabricated nanocrystalline layers on medium carbon steels, low carbon steels (SFE 200 mJ/m²) and pure copper (SFE 80 mJ/m²) in previous works [23,24], where nano-scale austenite phases were formed on the impacted surface subjected to explosive impact, and also indicated that the nanocrystallines formed on the surface of steel plate were benefited from the dense dislocation walls and dislocation tangles.

In the present study, therefore, AISI304 stainless steel was focused on to increase the surface hardness and comprehensive mechanical performance by explosive impacting treatment. And then by comparison of the microstructures and mechanical properties changes before and after treatment, the relationships between the microstructure evolution procedures including surface nanocrystallization and the properties increment of AISI 304 stainless steel were discussed systematically. Meanwhile, the mechanism of nanocrystallization on the impacted surface was also discussed.

**Table 1 – Compositions (wt.%) of AISI304 stainless steel used in the present work.**

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Mn</th>
<th>Cr</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.07</td>
<td></td>
<td>0.24</td>
<td>0.01</td>
<td>0.025</td>
<td>8.04</td>
<td>1.21</td>
<td>18.2</td>
</tr>
</tbody>
</table>

**2. Materials and methods**

The adopted material is a commercial AISI304 stainless steel plate (annealing at 1150 °C for 2 h, hereafter refers to annealed sample) with the size of 300 mm × 50 mm × 3 mm. The composition was listed in Table 1. The schematic diagram of explosive device is shown in Fig. 1. Annealed samples were installed on the anvil block (including 45# steel, smooth surface, 10 cm thick) by using four support nails in a vacuum environment (Vessel was evacuated to 5KPa). The explosive powders were placed on the AISI304 plate and a detonator was inserted into the powders. As detonator was ignited, the extremely high-speed collision was generated between the annealed sample and anvil block by detonation wave, and then resulted in the severe plastic deformation of the sample surface (hereafter the sample after explosive impact was referred as treated sample). The technique involved explosive impact treatment was described in detail in our previous papers [23,24].

Fig. 2 illustrates the sample cutting positions into the plate for microstructure observation and microhardness testing. The microstructures of annealed and treated samples were observed by optical microscope (GX51, OLYMPUS, Japan). The samples were cut cross-sectionally with the dimension of 10 mm × 10 mm × 3 mm and then mechanically polished and etched by a reagent of 2 mlHF + 3 mlHNO₃ + 95 mlH₂O. The transmission electron microscopy (TEM) (JEM-2010, NEC, Japan) and selected-areas electron diffraction (SAED) were used to analyze the microstructure of the surface layer. The constitutions of phase in different depth from the treated

**Fig. 1 – Schematic diagram of explosive impacting process.**

**Fig. 2 – Schematic diagram of TEM sample preparation.**
Surface were characterized by an X-ray diffraction instrument (D/MAX-2500, RIGAKU, Japan). Room temperature hardness was measured by using an HXD-1000YMx tester with the test loading was 0.5 N, and the distance between the adjoining testing points was 20 µm, and each value was an average of five points that were located on the same straight lines parallel to the impact surface. Tensile tests samples were conducted on a universal tensile testing machine (Instron5500R, Germany).

3. Results and discussion

3.1. Mechanical properties

The microhardness and engineering stress–strain curves of annealed and the treated sample are shown in Fig. 3. It can be seen from Fig. 3(a) that the hardness of the treated sample is effectively improved by 2–3 times in comparison with the annealed sample. The microhardness of the treated sample tends to decrease gradually from the top surface to inside. In region I, the impacted surface exhibits an excellent enhanced microhardness which reached 465 MPa. In region II, the microhardness gradually decreases along the depth to 325 MPa at the 600 µm from top surface. The microhardness tends to a constant value of 285 MPa in the region III. Fig. 3(b) revealed the explosive impact treatment directly affects the global behavior of AISI304 stainless steel plate, the yield strength increases from 250 ± 8 MPa to 700 ± 9 MPa and ultimate strength from 620 ± 8 MPa to 920 ± 5 MPa, respectively. These increments on both hardness and tensile strength were mainly dependent on the corresponding microstructures of the impacted surface and inside of the AISI304 stainless steel plate.

3.2. Characterization of microstructures

Fig. 4 shows the microscopic observations of the annealed and the treated samples. The annealed samples with grain sizes of 80–150 µm mainly composed of austenite and annealed twin (Fig. 4(a)). Fig. 4(b) showed that microstructures of the treated sample mainly can be divided into three regions. Region I refers to the area from outer surface to 40 µm depth. Region II was the area ranging from the depth of 40 µm to 600 µm from the impacted surface and its optical magnification micrographs is shown in Fig. 4(c) and (d). Region III refers to the area out of 600 µm. As can be seen in Fig. 4(c), a large number of the deformation bands and their intersections appeared in the austenite grains although macroscopic deformation is not observed. Moreover, as shown in Fig. 4(d), the deformation induced martensite (DIM) was generated in the deformation bands (as the arrow denoted in micrograph). Region III also exhibits many deformation bands, but the number of the deformation bands are much less than region II.

To further investigate the effect of explosive impacting treatment on the surface microstructure, the detailed microstructures along the cross section of impacted surface were observed by using transmission electron microscope (TEM) at every certain depth. Fig. 5(a) shows the TEM morphology corresponding to region I. Obviously, the nano-grains (with the grain size about 30 nm) were formed in the top surface. The corresponding SAED pattern dominated by circles indicates the presence of the nanocrystallites which contain austenite and martensite as shown in Fig. 5(b). Thereby, it can be suggested that, in region I, hardness and tensile strength of annealed sample were improved by grain-refinement strengthening and phase transformation strengthening resulting from the nanoscale austenite and martensite.

The microstructure at approximately 100 µm depth from the top surface as shown in the Fig. 6(a) is characterized by a number of intersections of deformation bands in the austenite grains. The deformation bands divided the original austenite grains into several rhombic blocks. Moreover, it can also be seen that the spacing between two adjacent deformation bands varies from 170 nm to 940 nm. Fig. 6(b) showed the microstructure at about 300 µm depth from the impacted surface. The spacing between the adjacent deformation bands is of ranging from 400 nm to 2.2 µm. Fig. 6(c) showed the typical feature of dislocation lines and the planar dislocation arrays in the low stacking fault metals at about 600 µm depth apart from the impacted surface. As can be seen in Fig. 6, with the increase of the depth from the surface, the number of deformation bands decrease and the spacing between the adjacent deformation bands increase due to the strain and strain rate decreasing during explosive impact treatment. Correspondingly, the intergranular defects were gradually transformed from the deformation bands to the planar dislocation arrays. Dense deformation bands consist of α-martensite, deformation twins and stacking fault [7,15,25], which can significantly impede the dislocation moving and subsequently improve
the microhardness and the mechanical properties in AISI304 stainless steel plate in our study. So, it can be deduced that the strengthening in region II and III are mainly caused by deformation bands and phase transformation.

The XRD qualitative analysis of phases with the different depth apart from impacted surface was conducted. As shown in Fig. 7(a), it was revealed that the peaks of \( \alpha \) (200) and \( \alpha \) (211) appeared at each depth, which implied that DIM was formed after the explosive impacting. The relative volume fraction of the martensite initially increased near impacted surface and then decreased toward inside. The maximum volume fraction of DIM is appeared at the subsurface (about 100 \( \mu \)m depth from surface). High strain rate and high temperature (10\(^5\)/s and 1600\( ^\circ\)K) [26] instantaneously generated in the impacted top surface would restrict the large number of nucleation and merging of the DIM during the explosive treatment [25,27]. Meanwhile, relatively low strain rate deep inside the plate also would make the volume fractions of DIM decreased a lot. So, it is likely to present a peak value at the certain depth in the subsurface from the top to inside. Furthermore, from the large numbers of deformation band as described above in Fig. 6, it is clear that there have presented a maximum volume fraction of DIM at the subsurface of approximately 100 \( \mu \)m.

The detailed information for XRD patterns are shown in Fig. 7(b) (marked with rectangular in Fig. 7(a)), indicating that intensity of the main peak austenite (111) was decreased and the full width at half maximum was broadened. It can be attributed to the imperfections induced by grain refinement and micro-strains. According to the full width at half maximum, the average grain size \( D \) (nm) was estimated by Eq. (1) [14,28]:

\[
D = \frac{K \lambda}{\beta \cos \theta}
\]

where \( K \) is constant value of 1 [28], \( D \) is the average grain size (nm), \( \lambda \) is the wavelength of the X-ray (1.5406 nm for Cu target), \( \beta \) is the breadth of the peak (rad.) and \( \theta \) is the Bragg angle of the

Fig. 4 – Optical microstructure of cross-section of AISI304 stainless steel samples before and after explosive treatments (a) microstructure of annealed sample; (b) microstructure of cross-section after explosive impact; (c) microstructure of deformation bands; (d) microstructure near surface.

Fig. 5 – Surface TEM micrographs of explosive impacted sample (a) TEM micrograph; (b) SAED pattern with polycrystalline rings of (a).
peak (deg). The breadth of the peak $\beta$ can be estimated using the following equation [28]: $\beta = \sqrt{B^2 - b^2}$, where $B$ (0.492 rad.) and $b$ (0.453 rad.) are the full width at half maximum of the peak for both treated and annealed samples which are calculated by using XRD analytical software JADE6.0. Average grain size of the impacted surface corresponding to main peak was calculated by Eq. (1), where the value of 35 nm was flexibly estimated. It is in well agreement with TEM results.

Consequently, the correlation between the high mechanical performance and the surface nanocrystallization and microstructural evolution of AISI304 stainless steel plate during the explosive impact treatment can be described as follows: The planar dislocation arrays and the dislocation tangles generated in the coarse grains (Fig. 6(c)). Subsequently, with the increases of strains and strain rate, the coarse grains were divided into subgrains boundaries by dislocations, and the DIM were formed in deformation band intersections during explosive impact (Fig. 6(a) and (b)). Finally, the subgrains' boundaries transfer into the large angle grain boundary by dynamic recrystallization and then formed the nanocrystallines (Fig. 5). All of these comprehensive microstructural effects contributed to the excellent mechanical properties and higher hardnesses of whole plate.

4. Conclusions

In summarizing, the surface treatment on AISI304 stainless steel plate was successfully carried out by explosive impacting technique. The correlations of mechanical properties and the corresponding microstructural evolution were analyzed. The main conclusions are as follows:

Fig. 6 – TEM micrographs apart from the impacted surface with different depths (a) 100 $\mu$m, (b) 300 $\mu$m, (c) 600 $\mu$m; the insert ellipse shows the planar dislocation arrays and rectangle shows the dislocation tangle.

Fig. 7 – XRD patterns before and after explosive treatments (a) XRD patterns for different depth from surface; (b) comparison of main diffraction peaks.
(1) A higher mechanical performance and corresponding finer microstructure than that of base metal were obtained by explosive impact technique.

(2) The nanocrystalline layer with grain sizes of 30 nm was obtained on the top explosion impacting surface, and many deformation bands were generated in the subsurface of AISI304 stainless steel plate.

(3) The hardening mechanism of entire plate is attributed to coactions of grain-refinement strengthening, deformed-bands strengthening and phase transformation strengthening.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgement

This work was supported by the National Natural Science Foundation of China (No. 51665042) and Scientific Research Foundation of Inner Mongolia University of Technology (No. X201411).

References


