Surface-hardened Layer Properties of Newly Developed Case-hardening Steel

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The effects of fine-particle peening conditions on the surface-hardened layer properties of newly developed case-hardening steel, i.e., transformation-induced plasticity-aided steel with a chemical composition of 0.2% C, 1.5% Si, 1.5% Mn, 1.0% Cr, 0.2% Mo, and 0.05% Nb (mass%) were investigated for the fabrication of automotive drivetrain components. The surface roughness decreased with decreasing arc-height of fine-particle peening after vacuum carburization. A white layer developed on the surface of the steel peened at arc-heights greater than 0.41 mm (N). The maximum Vickers hardness and maximum compressive residual stress increased with increasing arc-height in the steel. These values were higher than those of commercial case-hardening steels. The increased volume fraction and expansion strain of the strain-induced martensite increased the hardness and compressive residual stress in the surface-hardened layer of the steel, although severe plastic deformation made a substantial contribution to enhancing the surface-hardened layer properties.

KEY WORDS: vacuum carburization; fine-particle peening; hardness; residual stress; strain-induced martensite transformation; case-hardening steel; TRIP-aided steel.

1. Introduction

Recently, there has been a demand for downsized transmission precision gears with high torque capacity and excellent wear resistance to reduce the energy consumption of automobiles. Transformation-induced plasticity (TRIP)-aided steels with a martensite/bainitic ferrite structure matrix, i.e., TRIP-aided martensitic (TM) steel1–3) and quenching and partitioning steel, 4–6) which have been developed as next-generation structural steels, are expected to be suitable for use in fabricating precision gears because of their high toughness,2) good cyclic hardening,3) and high notch-fatigue strength.2,7) Sugimoto et al.8,9) have reported that fine-particle peening (FPP) further increases the rotational bending and torsional fatigue strengths of heat-treated TM steel by imparting a substantial high hardness and compressive residual stress as a result of the strain-induced martensite transformation of a large amount of metastable retained austenite. Numerous researchers have reported that high-energy shot peening also increases the fatigue strength of heat-treated steels.10–13) Even higher fatigue strengths of TM steel can be achieved by gas- or vacuum-carburizing with shot peening and FPP.14–20) However, no investigation has been conducted into the effects of FPP conditions on the surface-hardened layer properties and the fatigue strength of vacuum-carburized TM steel.

In this study, the properties of the surface-hardened layer of vacuum-carburized TM steel subjected to FPP under various conditions were investigated; the investigated properties include surface roughness, volume fractions of retained austenite and strain-induced martensite, Vickers hardness, and X-ray residual stress. These surface-hardened layer properties were correlated with the strain-induced transformation behavior of the retained austenite, as well as with its severe plastic deformation behavior.

2. Experimental Procedure

TM steel slabs with the chemical composition presented in Table 1 were vacuum-melted, hot-forged, and hot-rolled into 13-mm-diameter bars. Round bar specimens of 5 mm in diameter and 90 mm in length were machined from the bars. The specimens were subjected to vacuum carburization and then quenched in oil at 80°C followed by tempering at 180°C for 90 min. The vacuum carburization process, which was conducted in a vacuum-carburizing batch furnace (VCB, IHI Machinery and Furnace Co. Ltd., Tokyo, Japan), is shown in Fig. 1. For comparison, commercial JIS-SNCM420 steel bars (Table 1) were prepared and subjected to the same vacuum carburization treatment as the TM steel. Specimen surfaces were subjected to FPP carried out under four different sets of conditions using shot materials with different diameters and hardness (Table 2).

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Arc-height values of the FPP varied from 0.104 to 1.12 mm (N). The values of TM steel were nearly the same as those of JIS-SNCM420 steel. In the following, the effects of shot material properties on surface-hardened layer of the steels are mentioned as the effect of arc-height.

The microstructure of the steels was observed by field-emission scanning electron microscopy (FE-SEM; JSM-6500F, JEOL Ltd., Tokyo, Japan), which was performed using an instrument equipped with an electron-backscatter diffraction (EBSD) system (OIM system, TexSEM Laboratories, Inc., UT, USA). The steel specimens for the FE-SEM–EBSD analyses were first ground with alumina powder and colloidal silica and then subjected to ion thinning. The surface roughness of specimens was measured by laser scanning microscopy (LSM; VK-8510, Keyence Co., Milton Keynes, UK). The hardness was measured using a micro-Vickers hardness tester (HVM, Shimadzu Co., Kyoto, Japan) under testing load of 500 g and dwell time of 30 sec. Surface roughness was evaluated by the maximum roughness height ($R_z$, JIS B0601:2001). The retained austenite fraction of the steels was quantified from the integrated intensity of the (200)$_\alpha$, (211)$_\alpha$, (200)$_\gamma$, (220)$_\gamma$, and (311)$_\gamma$ peaks in the X-ray diffraction pattern (RINT2000, Rigaku Co., Tokyo, Japan). The cos $\alpha$ method was used in X-ray studies of residual stress in a longitudinal direction using an X-ray residual stress analyzer ($\mu$-X360, Pulstec Ind. Co. Ltd., Hamamatsu, Japan). The measurement conditions and material constants employed are given in Table 3.

<table>
<thead>
<tr>
<th>Condition</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Steel</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shot Diameter [mm]</td>
<td>0.07</td>
<td>0.05</td>
<td>0.10</td>
<td>0.30</td>
</tr>
<tr>
<td>Vickers hardness</td>
<td>800</td>
<td>900</td>
<td>850</td>
<td>950</td>
</tr>
<tr>
<td>Arc-height [mm (N)]</td>
<td>0.104</td>
<td>0.21</td>
<td>0.41</td>
<td>1.12</td>
</tr>
<tr>
<td>Coverage [%]</td>
<td>300</td>
<td></td>
<td></td>
<td></td>
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</table>

<table>
<thead>
<tr>
<th>Phase</th>
<th>$\alpha$-bcc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Characteristic X-ray</td>
<td>Cr</td>
</tr>
<tr>
<td>Voltage, current</td>
<td>30 kV, 1 mA</td>
</tr>
<tr>
<td>$\psi$</td>
<td>35°</td>
</tr>
<tr>
<td>Diffraction plane</td>
<td>(211)</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>223 GPa</td>
</tr>
<tr>
<td>Poisson ratio</td>
<td>0.28</td>
</tr>
</tbody>
</table>

Fig. 1. Vacuum carburization and tempering diagram of the TM and SNCM420 steels, where OQ represents quenching in oil at 80°C and $C_p$ is carbon potential.

Fig. 2. (a, c) Phase maps and (b, d) orientation maps of the $\alpha$-bcc phase in the cross-sectional side view of the surface-hardened layer of carburized (a, b) TM and (c, d) SNCM420 steels. Yellowish-green, gray, and red regions in (a) and (c) represent the $\alpha'$-martensite matrix, the MA-like phases, and retained austenite islands, respectively. Max volume fractions of retained austenite in TM and SNCM420 steels are 20.5 and 16.3 vol.%, respectively.
3. Results

3.1. Microstructure of the Surface-hardened Layer

The results of the EBSD analyses of vacuum-carburized TM and SNCM420 steels are shown in Fig. 2. The microstructure of the TM and SNCM420 steels was characterized by a mixture of an $\alpha'$-martensite matrix with a wide lath structure, martensite–austenite (MA)-like phases, and retained austenite phases. The microstructure near the surface is coarser than the interior microstructure in both steels. The TM steel possesses finer $\alpha'$-martensite, a greater amount of retained austenite, and a smaller amount of MA-like phase than the SNCM420 steel. Two types of retained austenite, i.e., island type in the matrix and filmy type in the MA-like phase, appear in both steels. Vacuum-carburized TM steel contains a larger amount of island type of retained austenite, compared with SNCM420 steel. Most of the MA-like phases appear to be located along prior austenitic, packet, and block boundaries.

Top-view LSM images of the surface and cross-sectional side-view SEM images of the surface-hardened layer in the TM steel subjected to FPP under various conditions after vacuum carburization are shown in Fig. 3. The microstructures just below the surface were refined in all of the steel specimens. A white layer (a nanostructure or an amorphous structure) was developed on the surface of the TM steel subjected to FPP under conditions C and D. The thickness of the white layer is lower than 10 $\mu$m and increases with increasing arc-height of FPP. A similar white layer was observed at the surface-layer of vacuum-carburized JIS-SCM415 steel subjected to two-step FPP under total arc-height of 0.31 mm (N), although the white layer thickness was 10 $\mu$m. The similar tendency was confirmed for the vacuum-carburized SNCM420 steel subjected to FPP under various conditions.

3.2. Surface-hardened Layer Properties

Figure 4 shows the distributions of the volume fraction of untransformed retained austenite, the Vickers hardness, and residual stress in TM steel subjected to vacuum carburization (○) and vacuum carburization and subsequent fine-particle peening (▲: A, ●: B, △: C, □: D). $\Delta f_{\alpha m}$, $\Delta H V$ and $\Delta \sigma_{x2}$ are increments of strain-induced martensite fraction, hardness and residual stress after fine-particle peening. The x-axis shows the depth from the surface in mm.
the X-ray residual stress of the α-bcc phase in the surface-hardened layer of vacuum-carburized TM steel subjected to FPP under various conditions. The retained austenite fraction is decreased by FPP, especially on the surface. The maximum volume fraction of the retained austenite decreases with increasing arc-height of FPP and is given at depths from 20 to 100 μm. Resultantly, the strain-induced martensite fraction (Δf/m) reaches a maximum on the surface or at depths of 10 to 50 μm from the surface. The hardness and compressive residual stress reach their maximum values at the same depths. The depths corresponding to these maxima increase with increasing arc-height.

The maximum values of Vickers hardness, strain-induced martensite fraction and residual stress, and surface roughness in the TM and SNCM420 steels are shown in Fig. 5 and Table 4. The maximum strain-induced martensite fraction, the maximum Vickers hardness, and the maximum compressive residual stress in the surface-hardened layer increase with increasing arc-height. The surface roughness increases with increasing arc-height, although it increases abruptly in a range between 0.21 and 1.12 mm (N). A comparison of these properties in the surface-hardened layer of the TM steel with those of the SNCM420 steel reveals that the TM steel is characterized by a larger volume fraction of strain-induced martensite and a higher compressive residual stress compared with those of the SNCM420 steel. The maximum Vickers hardness of the two steels is approximately the same. The TM steel exhibits a slightly smaller surface roughness than the SNCM420 steel.

According to Egami et al., the Vickers hardness of 950 and residual stress of −1 850 MPa were attained in the surface-hardened layer of vacuum-carburized SCM415 steel subjected to two step FPP under total arc-height of 0.31 mm (N). Such the hardness and absolute value of the residual stress are slightly higher than those (Fig. 5) of vacuum-carburized SNCM420 steel subjected to FPP under conditions B and C.

Table 4. Surface-hardened layer properties of vacuum-carburized TM and SNCM420 steels subjected to fine-particle peening (FPP) under conditions A to D.

<table>
<thead>
<tr>
<th>Steel</th>
<th>FPP</th>
<th>AH</th>
<th>Rz [μm]</th>
<th>HVmax</th>
<th>f/mmax</th>
<th>Δf/mmax</th>
<th>ΔHV</th>
<th>ΔHV/ΔHVmax</th>
<th>Δσ/σmax</th>
<th>Δσ/σmax + abs(σ/σmax)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TM</td>
<td>A</td>
<td>0.104</td>
<td>3.9</td>
<td>760</td>
<td>16.5</td>
<td>−1 607</td>
<td>14.8</td>
<td>0.53</td>
<td>−279</td>
<td>0.17</td>
</tr>
<tr>
<td>C</td>
<td>0.41</td>
<td>39.1</td>
<td>901</td>
<td>14.3</td>
<td>−1 936</td>
<td>18.0</td>
<td>0.16</td>
<td>−340</td>
<td>0.18</td>
<td>2 798</td>
</tr>
<tr>
<td>D</td>
<td>1.12</td>
<td>82.6</td>
<td>937</td>
<td>8.6</td>
<td>−2 125</td>
<td>17.8</td>
<td>0.13</td>
<td>−336</td>
<td>0.16</td>
<td>2 910</td>
</tr>
<tr>
<td>SNCF 420</td>
<td>A</td>
<td>0.104</td>
<td>3.9</td>
<td>743</td>
<td>13.5</td>
<td>−1 555</td>
<td>7.4</td>
<td>0.39</td>
<td>−140</td>
<td>0.09</td>
</tr>
<tr>
<td>C</td>
<td>0.21</td>
<td>4.8</td>
<td>858</td>
<td>11.6</td>
<td>−1 673</td>
<td>9.4</td>
<td>0.11</td>
<td>−177</td>
<td>0.11</td>
<td>2 664</td>
</tr>
<tr>
<td>C</td>
<td>0.104</td>
<td>40.1</td>
<td>901</td>
<td>12.4</td>
<td>−1 731</td>
<td>9.6</td>
<td>0.08</td>
<td>−181</td>
<td>0.10</td>
<td>2 798</td>
</tr>
<tr>
<td>B</td>
<td>1.12</td>
<td>94.3</td>
<td>937</td>
<td>8.7</td>
<td>−1 811</td>
<td>9.7</td>
<td>0.07</td>
<td>−183</td>
<td>0.10</td>
<td>2 910</td>
</tr>
</tbody>
</table>

AH [mm (N)]: arc-height; Rz [μm]: maximum roughness height; HVmax: maximum Vickers hardness; f/mmax: maximum volume fraction of untransformed retained austenite; Δf/mmax [vol.%]: maximum residual stress; ΔHV: an increment in the hardness due to strain-induced martensite transformation; ΔHVmax: an increment in HVmax due to fine-particle peening; Δσ/σmax [MPa]: an increment in residual stress calculated from transformation strain; Δσ/σmax [MPa]: an increment in σ/σmax due to fine-particle peening; σ/σmax [MPa]: absolute value of σ/σmax.
4. Discussion

4.1. Maximum Vickers Hardness in the Surface-hardened Layer

In general, the hardness in a surface-hardened layer of the carburized steels is enhanced by the following microstructural changes developed by FPP:

(i) Formation of a white layer and increases in the dislocation density in the matrix and untransformed retained austenite because of severe plastic deformation;10–12,17–19,24

(ii) Increase in the volume fraction of hard martensite because of the strain-induced martensite transformation of the retained austenite.25–32

The relationship between the maximum Vickers hardness and the maximum volume fraction of strain-induced martensite in the TM and SNCM420 steels is shown in Fig. 6(a). The maximum Vickers hardness increases with increasing maximum strain-induced martensite fraction, although it increases substantially above the critical volume fractions of the strain-induced martensite, such as  for the SNCM420 steel and  for the TM steel. These results indicate that the microstructural change (ii) enhances the maximum hardness in vacuum-carburized TM and SNCM420 steels subjected to FPP, as well as the microstructural change (i).

The aforementioned critical volume fractions never appear in the heat-treated and fine-particle-peened steels with a low original volume fraction of retained austenite (triangles in Fig. 6(a)). This result indicates that some plastic relaxation occurs in a range below the critical volume fraction, which diminishes strain hardening due to severe plastic deformation and transformation-hardening due to the increased volume fraction of strain-induced martensite, in the vacuum-carburized steels. Because the  for the TM steel is larger than the  for the SNCM420 steel, the plastic relaxation of the TM steel may occur markedly. The relaxation mechanism is under investigation and will be reported in due course.

The increment in the maximum Vickers hardness ( ) after FPP of vacuum-carburized steels increased with increasing strain-induced martensite fraction, where  and  are increments of the Vickers hardness because of severe plastic deformation and strain-induced martensite transformation, respectively.  can be estimated using the equation,

\[
\Delta H V_I = (H V_{\alpha_m} - H V_{m,c}) \times \Delta f_{\alpha_{m,\text{max}}} \tag{1}
\]

where  is the maximum volume fraction of strain-induced martensite (Table 4).  and  are Vickers hardness of strain-induced martensite with carbon concentration of 0.80 mass% in the retained austenite and the maximum Vickers hardness of as-vacuum-carburized TM and SNCM420 steels, respectively. If Vickers hardness  was substituted as  for Ref. 33 was 880–920 of martensite of TM steel from 713 for TM steel or 712 for SNCM420 steel, respectively. If Vickers hardness  can be estimated to be 25–30 and 12–16, respectively; the ratios of  to  are 0.13–0.53 and 0.07–0.39 for TM and SNCM420 steels, respectively (Fig. 7 and Table 4). Therefore, it is considered that severe plastic deformation mainly contributes to the  in TM and SNCM420 steels, compared to the strain-induced martensite transformation, except for arc-height of 0.104 mm (N). Higher  ratio of TM steel may be associated with a relatively large amount of isolated island type of retained austenite which effectively increases the hardness. The  ratios of both steels slightly decreased with increasing arc-height.

Fig. 6. Relationships between (a) maximum Vickers hardness ( ) and (b) maximum residual stress ( ) and strain-induced martensite fraction ( ) in the surface-hardened layer of vacuum-carburized TM (●) and SNCM420 (○) steels subjected to fine-particle peening under different conditions. In (a),  is an increment in  due to fine-particle peening and  where  and  are the increments of Vickers hardness resulting from the strain-induced transformation and the plastic strain, respectively.  is the maximum hardness of as-vacuum-carburized steels. In (b), the chain line and small marks represent calculated values of  and  and  and  are the increments of residual stress resulting from the strain-induced transformation and the plastic strain, respectively. Symbols ▲ and ■ respectively denote surface-hardened layer properties of the TM and SNCM420 steels subjected to heat-treatment and fine-particle peening (HT+FPP).

Fig. 7. Variations in  as a function of arc-height (AH) in vacuum-carburized TM (●) and SNCM420 (○) steels.
This indicates that the higher the arc-height, the smaller the contribution of the strain-induced martensite transformation.

4.2. Maximum Compressive Residual Stress in the Surface-hardened Layer

According to Sugimoto et al., FPP considerably increases the compressive residual stress in the surface-hardened layer of heat-treated TM and SNCM420 steels, although the increase is smaller than that resulting from conventional high-energy shot peening. This compressive residual stress is believed to result from the following effects:

(i) Unrelaxed strain between the matrix and second phase because of severe plastic deformation.
(ii) Expansion strain because of the strain-induced martensite transformation of the retained austenite.

According to the previous works, if transformation strain of the martensite islands is assumed to be an isotropic expansion, the transformation strain \( \varepsilon_{\text{m},t} \) of the Fe-(0.19–1.01) %C steels at martensite-start temperature is given by:

\[
\varepsilon_{\text{m},t}^* = 0.0058 + 0.0043 \times \%C; \quad \text{(2)}
\]

where %C is carbon concentration in the martensite. Natori et al. proposed that residual stress resulting from the strain-induced martensitic transformation \( \Delta \sigma_{\text{Xt},\text{p}} \) can be calculated using Eq. (3),

\[
\Delta \sigma_{\text{Xt},\text{p}} = -(d_0 - d_\text{k}) \Delta \sigma_{\text{m},\text{max}} E \varepsilon_{\text{m},t}^* / d_0; \quad \text{(3)}
\]

where \( d_0 \) and \( d_\text{k} \) are original diameters of the specimen and the depth of the surface-hardened layer, respectively. \( E \) is the Young’s modulus. In the case of the present TM steel, \( \varepsilon_{\text{m},t}^* \) was calculated to be 0.0092 because the %C was estimated to be 0.8 mass%. Substituting \( d_0 = 5 \) mm, \( d_\text{k} = 0.02 \) mm, \( \Delta \sigma_{\text{m},\text{max}} \) (Table 4), and \( E = 206 \) GPa into Eq. (3), we can obtain \( \Delta \sigma_{\text{Xt},\text{p}} \); the results are shown in Fig. 6(b) and Table 4. The \( \Delta \sigma_{\text{Xt},\text{p}} \) linearly increases with increasing maximum strain-induced martensite fraction in the TM and SNCM420 steels. The ratios of \( \Delta \sigma_{\text{Xt},\text{p}} \) to the increment in residual stress \( \Delta \sigma_{\text{Xt},\text{p}} / \Delta \sigma_{\text{Xt},\text{max}} \) are 0.16 to 0.18 and 0.09 to 0.11 for TM and SNCM420 steels, respectively, and slightly decrease with increasing arc-height of FPP. These properties were better than those of SNCM420 steel.

4.3. Estimation of Fatigue Limits

Matsui et al. reported that the fatigue limit \( (\sigma_u) \) of gas-carburized JIS-SCM822 H steel subjected to shot peening is related to the sum of the estimated yield stress \( \sigma_{\text{Y,est}} \) and the absolute value of the maximum residual stress \( \{|\sigma_{\text{Xt},\text{max}}| + \text{abs}(\sigma_{\text{Xt},\text{max}})\} / 2 \) (4)

where the value of \( \sigma_{\text{Y,est}} \) can be estimated from the maximum Vickers hardness \( (\text{HV}_\text{max}) \) by

\[
\sigma_{\text{Y,est}} = (\text{HV}_\text{max} / 3) \times 9.80665 \times (\text{YS} / \text{TS}) \times \text{(5)}
\]

where \( \text{YS} / \text{TS} \) is the yield ratio defined by a ratio of yield stress to tensile strength (assumed by Matsui et al. to be 0.95).

Figure 8 shows the relationship between \( \sigma_{\text{Y,est}} \) and arc-height of FPP for the present vacuum-carburized TM and SNCM420 steels. The values of \( \sigma_{\text{Y,est}} \) and arc-height of FPP in Table 4 increase with increasing arc-height of FPP. This relationship resembles arc-height dependences of the maximum hardness and maximum compressive residual stress. Therefore, we can expect that the fatigue limits of both steels increase with increasing the maximum hardness and increasing compressive residual stress. In case-hardening steels, however, the crack origin depth from the surface also controls the fatigue limit. Resultantly, the maximum fatigue limit can be obtained by FPP under condition B. The relationship between \( \sigma_u \) and \( \sigma_{\text{Y,est}} \) will be published elsewhere.

5. Conclusions

The effects of FPP conditions on the surface-hardened layer properties of vacuum-carburized 0.2%C-1.5%Si-1.5%Mn-1.0%Cr-0.05%Nb TM steel were investigated. The main results are summarized as follows:

(1) The surface roughness decreased with decreasing arc-height of FPP. A white layer developed on the surface of the steel subjected to FPP at arc-heights greater than 0.41 mm (N).

(2) The maximum Vickers hardness and maximum compressive residual stress of the TM steel increased with increasing arc-height of FPP. These properties were better than those of SNCM420 steel.

(3) The increased volume fraction and expansion strain of the strain-induced martensite enhanced the Vickers
hardness and compressive residual stress in the surface-hardened layer of the TM steel. However, the contribution of the strain-induced martensite to the Vickers hardness and compressive residual stress was smaller than that of severe plastic deformation.

(4) The contribution of strain-induced martensite transformation to the maximum Vickers hardness and the maximum compressive residual stress in TM steel were relatively larger than that in SNCM420 steel, except for the contribution of strain-induced martensite transformation to the maximum Vickers hardness at low arc-height. This was considered to be caused by that TM steel contained a large amount of isolated island type of retained austenite.

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REFERENCES