Effect of Combined Shot Treatment and Nitriding on Galling Property of Die Used for High Strength Steels

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The methodologies of controlling the surface texture of die by hybrid (double)-shot treatment and nitriding at a low temperature were examined for the development of long life cycle die applied to the high strength steel sheets. Hybrid-shot improved the galling property because of its lubricating effect, while nitriding at low temperature which is useful for decreasing distortion by heat-treatment had sufficient hardness of 1 500 HV for anti-abrasion. Surface texture of die material, evaluated by using Fast Fourier Transformation (FFT) analysis revealed that the ratio of the spectrum amplitude between high and low frequencies was corresponding to the galling generation load.

KEY WORDS: die; shot-treatment; nitriding; surface texture; Fast Fourier Transformation.

1. Introduction

Reducing weight of a vehicle body is an effective way to improve fuel efficiency. For lightweight, high strength steel sheet which is newly developed is used for many parts of the vehicle. The high tensile strength steel sheet contributes also to reducing fatal accidents by increasing anti-collision property. However, the application of high tensile strength steel sheet lowers the die life cycle. Moreover, the increase in tensile strength generally impairs press formability.

Many experiments on controlling microstructure in high tensile strength steel sheet have been carried out to improve press formability. TRIP steels and Dual Phase steels were developed. However, microstructure controlling has limits, so it can not solve the problem of a short life cycle of a die.

To improve the shortness of life cycle of dies according to the high strengthening of steel sheet, controlling the surface texture of die is one of the most effective methods. Coating technology using TiC etc. has been applied recently for decreasing friction of dies. However, coating has an intrinsic risk of flaking. Therefore, the development of non-coating technology is required.

In the present research, controlling the surface texture of die by hybrid (double-shot) treatment and nitriding at a low temperature was examined for developing a long life cycle die applied to the high strength steel sheet. Hybrid-shot improves lubricating property by tribological method, while nitriding at a low temperature increases abrasion property by higher hardness and decreases distortion caused by heat treatment.

2. Experimental Procedures

2.1. Material

SKD11-steel (JIS) and DC53-steel (Daido Steel Co., Ltd.), which are generally used for die material were polished by emery paper of #800, and followed by shot-treatment and low temperature nitriding.

The shot-treatments were performed via direct air pressure through a single 2 mm diameter nozzle aligned to give an 80-degree angle from horizontal. A specimen was put on a table at a vertical distance of 230 mm from the nozzle. Shot-treatment conditions are shown in Table 1. A single shot-treatment and a double shot-treatment (Hybrid–shot) were applied using various particles: Al2O3, ZrO2, Fe-0.86 mass%C and stainless steel (JIS SUS304) for controlling the surface texture, under the pressure of 0.2–0.35 MPa for 30–240 s. As a typical case, the shot coverage of 100% was achieved in 25 s as the use of Al2O3 of 0.2 mm at the pressure of 0.2 MPa. The measured Vickers hardness of materials in this work is listed in Table 2. Then the nitriding process was performed as an ammonia nitriding at 733 K for 8 h.

The nitrided specimens were etched by a nital solution and were observed by using optical microscope. The change of Vickers hardness from the surface along the thickness of test specimens after shot-treatment and nitriding was measured under the load of 50 g with testing time of 15 s. At the same time, the surface Vickers hardness was measured under the load of 25 g with the same time.

The galling property of such test specimens were examined using a draw bead test, and the surface texture was evaluated quantitatively by means of the FFT (Fast Fourier Transformation) parameter.
2.2. Draw Bead Test

The method of the draw bead test is shown in Fig. 1. Cold rolled and annealed high tensile strength steel of 440 MPa in tensile strength was used for draw bead sheet. The thickness of sheet was 1.2 mm. The dimension of test specimens was as follows: thickness 3.0 mm, width 30 mm, length 15 mm. The drawing was performed at a speed of 1 m/min and stroke of 250 mm with lubricant oil (Daphne by IDEMITSU KOSAN Co., Ltd.) after brushing sheet. The load was increased by 200 kgf. The galling property was examined by the load in which galling was observed. This load is defined as the galling generation load in this research work.

2.3. Surface Texture Analysis

The surfaces of test specimens after shot-treatment were observed by LASER microscope, and FFT analysis shown in Eq. (1) was applied for evaluating the surface texture. From the spectrum of FFT, the ratio of spectrum amplitude in the low frequency region and that in the high frequency region \( R_{fq} \) was measured as shown in Eq. (2). The relationship between \( R_{fq} \) and the galling generation load examined by the draw bead test was investigated.

\[
R_{fq} = \frac{\text{Average spectrum amplitude in low frequency region (200 – 300 Hz)}}{\text{Average spectrum amplitude in high frequency region (400 – 500 Hz)}}
\]

3. Results and Discussion

3.1. Microstructures and Hardness after Nitriding

Optical micrographs of the specimens are shown in Fig. 2, where the particles with white contrast distributed in the matrix are carbides. In these images, blackish regions are the nitrogen diffused layers. The carbide grains in DC53 seemed to be smaller than in SKD11, but there was not so much of a difference between these steels.

The changes in hardness from the specimen surfaces along the thickness after shot-treatment and nitriding at 733 K for die steels is shown in Fig. 3. The shot-treatment condition was shot A in Table 1. In Fig. 3, the solid marks show the results, which were measured on the nitrided surface. The load was increased by 200 kgf. The galling property was examined by the load in which galling was observed. This load is defined as the galling generation load in this research work.

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Table 1. Shot-treatment conditions tested.

| Symbol | First shot | Second shot |
|--------|------------|-------------|
| Material | Diameter (mm) | Gauge pressure (MPa) | Time (s) | Material | Diameter (mm) | Gauge pressure (MPa) | Time (s) |
| A | Al₂O₃ | 0.2 | 0.25 | 240 | SUS304 | 0.1 | 0.35 | 90 |
| B | Al₂O₃ | 0.3 | 0.2 | 30 | Fe-0.86C | 0.05 | 0.2 | 30 |
| C | ZrO₂ | 0.2 | 0.2 | 30 | Al₂O₃ | 0.05 | 0.2 | 30 |
| D | Al₂O₃ | 0.3 | 0.2 | 30 | ZrO₂ | 0.05 | 0.2 | 30 |
| E | SUS304 | 0.3 | 0.2 | 30 | SUS304 | 0.2 | 0.2 | 30 |
| F | SUS304 | 0.07 | 0.2 | 30 | |
| G | SUS304 | 0.1 | 0.2 | 30 | |
| H | SUS304 | 0.3 | 0.2 | 30 | |
| I | Al₂O₃ | 0.2 | 0.35 | 150 | |

Table 2. Materials for galling test and shot treatment.

| Specimen | Material | Diameter (mm) | Average Vickers hardness |
|----------|----------|---------------|--------------------------|
| SKD11    | –        | –             | 684*                     |
| DC53     | –        | –             | 778*                     |
| Al₂O₃    | 0.05     | 930           |
| Al₂O₃    | 0.2      | 930           |
| Al₂O₃    | 0.3      | 930           |
| Fe-0.86C | 0.05     | 485           |
| SUS304   | 0.07     | 447           |
| SUS304   | 0.1      | 447           |
| SUS304   | 0.2      | 447           |
| SUS304   | 0.3      | 447           |
| ZrO₂     | 0.05     | 1100          |
| ZrO₂     | 0.2      | 1100          |

*Before shot and heat treatment

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\[
R_{fq} = \int_{0}^{\infty} f(x) e^{-2\pi n} dx
\]

\[
R_{fq} = \frac{\text{Average spectrum amplitude in low frequency region (200 – 300 Hz)}}{\text{Average spectrum amplitude in high frequency region (400 – 500 Hz)}}
\]
3.2. Surface Texture

For the shot-treatment conditions of A, G, and I in Table 1, the line profiles by LASER microscopic images of surface after shot-treatment are shown in Fig. 4. Shot A was Hybrid-shot and Shot G and I were single shot. It was revealed that the asperity of the line profile strongly depended on the shot-treatment condition, and the surface roughness became smooth by Hybrid-shot.

In Fig. 4, the roughness Rz values are illustrated. Shot G of the ordinary single shot-treatment and shot A of the Hybrid-shot treatment indicated the nearly equal Rz value of 5–7 μm, which is a typical two-dimensional index for evaluating the surface texture. From this result, it was found that the Rz is not useful in evaluating the galling property by the texture of shot-treated surface.

Figure 5 shows FFT analytical results for shot A and shot I in Table 1. The spectrum amplitude of shot I decreased with increases in frequency, while the spectrum amplitude of shot A is constant to frequency, indicating the availability of FFT analysis for examining the surface texture of die for galling property. From the FFT analysis, the spectrum amplitude ratio Rfq, which is expressed in Eq. (2) was calculated.

Rfq is an index of the flatness of power spectrum. The reason why the two frequency regions were allocated such as in Eq. (2) is described here. The enlarged micrograph in Fig. 6 shows the surface shape after shot treatment and nitriding for SKD11. The white marks in the figure indicate the scale of a dimple formed by shot particles. The average diameter of dimples was 7.9 μm in this example. On the whole, the sizes of these dimples were approximately 10 μm in each shot condition. This size is equivalent to the low frequency region, ranging from 200 to 300 Hz. Meanwhile, the spectrum amplitude exhibited to be nearly -constant in the high
frequency region (400–500 Hz), regardless of shot conditions. Therefore, its constant value was adopted as the datum level. This high frequency region corresponds to the weak impact scar, i.e. small and shallow, and which is inevitably given by the shot treatment. The relationship between $R_{fq}$ and the galling property will be discussed in section 3.

### 3.3. Galling Property

The draw bead test results are summarized in Table 3. Figure 7 shows the friction coefficient change in load to galling generation for the test specimens of shot conditions A, G and I in Table 1. It was revealed that the hybrid-shot, which is made from double stage shot-treatment has a good galling property.

The relationship between the spectrum amplitude ratio $R_{fq}$ and the galling generation load is shown in Fig. 8. The galling generation load increased linearly with the decrease in $R_{fq}$. This relationship was also obtained in the case of another die steel (DC53). This result suggests that the relationship is completely unaffected by the matrix hardness (Fig. 3). As shown in Fig. 4, the surface roughness values were approximately equal to or than 10 $\mu$m. From these data, it is considered that the extent of the impact by shot treatment is also in the 10 $\mu$m. It was reported, for instance, that the depth of shot-peened layer was mostly 2 to 5 $\mu$m.9) In contrast, the surfaces in this work had a thick hardened (nitrided) layer (~80 $\mu$m) as seen in Fig. 3. Furthermore, the surface hardness values were the same level in these both steels. Thus the relationship as the galling property is believed to reflect only the parameter of surface texture, $R_{fq}$. This finding suggests that the spectrum amplitude ratio is effective in evaluating quantitatively the die life. The experimental results

![Image 1](image1.png)

**Fig. 6.** SEM micrograph on the cross section of near surface of SKD11 after shot treatment and nitriding (No etching) Shot particle, size, pressure and time were $\text{Al}_2\text{O}_3$, 0.3 mm, 0.2 MPa and 30 s, respectively. White marks indicate the scale of a dimple formed by shot particles.

| Shot condition in Table 1 | Galling generation load (kgf) |
|---------------------------|-------------------------------|
| A                         | 3000                          |
| B                         | 2800                          |
| C                         | 2400                          |
| D                         | 2400                          |
| E                         | 2000                          |
| F                         | 1400                          |
| G                         | 1000                          |
| H                         | 2600                          |
| I                         | 1600                          |

**Table 3.** Draw bead test results.

![Image 2](image2.png)

**Fig. 7.** Relationship between pressing load and friction coefficient up to galling generation for SKD11 after various shot-treatment and nitriding at 733 K.

![Image 3](image3.png)

**Fig. 8.** Relationship between the spectrum amplitude ratio $R_{fq}$ and the galling generation load.
revealed that the lower $R_{q}$ strongly enhanced the lubricating ability by means of shot treatment. However, the reason for showing a linear relationship has not been clarified here, so further investigation will be needed.

4. Conclusions

The effect of surface texture of die on the occurrence of galling load and the quantitative evaluation of the occurrence of galling load with FFT analysis were examined to develop the long life die used for high tensile strength steel. The main findings of this research are summarized as follows:

1. The galling occurrence load increased by double shot-treatment (Hybrid-shot) and low temperature nitriding treatment.
2. The occurrence of galling load depended on the surface texture.
3. The surface texture was evaluated quantitatively by the spectrum amplitude ratio in low and high frequency regions calculated by FFT analysis.

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