Surface Characteristics of Molding Die Treated by Electron Beam Irradiation

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Abstract

In order to improve the thermal fatigue strength of EDMed molding dies, thermal fatigue tests of the treated surfaces by large-area electron beam (EB) under various conditions and gas nitriding were conducted. The thermal fatigue characteristics of the treated surface were evaluated by surface roughness measurement, microscopic observation, and X-ray residual stress measurement. These results indicate that the EB treatment has more dominant influence on the residual stress than pre-processing, such as EDM and milling, and the residual stress of EB treated surface is released in the early stage of the thermal fatigue test. Moreover, the gas nitriding treatment can inhibit the heat crack generation and its propagation on the surface in the thermal fatigue test.

Key words: EB, gas-nitriding, thermal fatigue characteristics.

1 INTRODUCTION

Electrical discharge machining is an effective method to fabricate various types of molding dies. However, the EDMed surface generally has a matte appearance, and a resolidified hard layer with micro-cracks and residual tensile stress is formed on the surface. Therefore, maintaining stability of molding performance for long term is difficult when the die surface is processed by EDM. Then, the EDMed surface is finally finished by manual polishing, and it takes long time.

On the other hand, it was reported that a large-area electron beam (EB) irradiation could improve the surface roughness and the corrosion resistance of metal surface in a short time\(^{1,2}\). The EB irradiation method will presumably be a key technique for surface finishing of dies as well as mechanical parts. The life extension of molding dies is always an important issue, and new surface treatment method has been required. In such situations, surface processing using EB, or combined EB and other surface treatments, such as gas nitriding, physical vapor deposition may have high possibility as a new surface treatment method of molding dies. However, the characteristics of surfaces irradiated with the large-area EB and those treated with other post processes to improve their resistance to thermal fatigue have not yet been sufficiently investigated.

In this study, thermal fatigue tests on a large-area EB-irradiated surface that was first EDMed and the surface after additional post processing were conducted to evaluate the fatigue characteristics of these surfaces. Furthermore, the surfaces characteristics were also discussed based on observation of test surfaces with a scanning electron microscope (SEM), measurement of residual stress, and structural analysis using X-ray diffraction.

2 EXPERIMENTAL PROCEDURE

2.1 Testpiece and Experimental Method

A testpiece material is an improved alloy tool steel for hot work (improved SKD61, HRC50). Circular plates with a diameter of 58 mm and a thickness of 20 mm were prepared for thermal fatigue test, and similar shape plates with a thickness of 10 mm were prepared for evaluation of surface characteristics. The chemical composition of the testpiece is shown in Table 1. The testpiece surface was first processed by EDM (V25F-G35, Mitsubishi Electric Corp.) under the conditions shown in Table 2, using a cylindrical copper electrode with a diameter of 60 mm. A setting area on the EDMed surface shown in Fig.1 (a) was irradiated with a large-area EB (EB300, Sodick Co., Ltd.) under the conditions listed in Table 3. The testpiece surface was also subjected to gas nitriding after the EB irradiation, since it was reported that gas nitriding was effective in preventing the deterioration of an EDMed surface with thermal fatigue\(^{3,4}\). The gas nitriding was performed at a temperature of 530°C for 6 hours in a mixed atmosphere of a reaction accelerator and NH\(_3\).

2.2 Evaluation of Surface Characteristics

The EDMed surface, the EB-irradiated surface, and the gas nitried surface were evaluated by measuring surface roughness, analyzing residual stress, and microscopic observation of each surface and its cross-section. Measuring conditions for residual stress and structural analysis using X-ray diffraction are shown in Table 4. Fig.2 shows the evaluated areas, which were 5x5 mm in size and located 15 mm from the edge of the circular plate.

2.3 Thermal Fatigue Test

Thermal fatigue tests were conducted assuming a heating and cooling cycle for aluminum die casting; heating at 570°C for 160 s...
Table 1  Chemical compositions of testpiece material (mass%)

|        | C   | Cr  | Mo  | V   | Si  | Mn  |
|--------|-----|-----|-----|-----|-----|-----|
| SKD61 (Improved) | 0.3 | 5.0 | 2.3 | 0.6 | 0.2 | 0.5 |

Table 2  EDM conditions

| Conditions | EDM1 | EDM2 | EDM3 | EDM4 |
|------------|------|------|------|------|
| $i_e$ (A)  | 8.0  | 5.5  | 3.5  | 2.5  |
| $t_e$ (μs) | 100.0| 64.0 | 20.0 | 5.4  |
| $t_o$ (μs) | 32.0 | 20.0 | 20.0 | 5.4  |

Table 3  EB irradiation conditions

| Conditions | EB1 | EB2 | EB3 | EB4 |
|------------|-----|-----|-----|-----|
| Energy density (J/cm$^2$) | 3.0 | 7.0 | 10.0| 7.0 |
| maximum beam diameter (mm) | | 60 |
| Number of irradiation (shot) | 30 | 30 | 30 | 10 |
| irradiation time /1shot (micro second: μsec) | 3 |

Table 4  Conditions of X-ray stress measurement and X-ray diffraction analysis

| Conditions | X-ray stress measurement | X-ray diffraction analysis |
|------------|--------------------------|---------------------------|
| Diffraction| αFe(211)                 | ---                       |
| Target-filter | Cr-V                       | Cr-V                       |
| Tube voltage | 40kV                       | 40kV                       |
| Filament current | 40mA                       | 30mA                       |
| Stress constant | -297MPa                   | ---                       |

and cooling at 100°C for 15 s were repeated up to 15,000 cycles. A schematic of the thermal fatigue testing apparatus is shown in Fig.3.

3 RESULTS AND DISCUSSIONS

3.1 Characteristics of EB Irradiated Surface

3.1.1 Surface Roughness

The surface of testpiece was EDMed under the conditions of EDM1-EDM4 as shown in Table 2. Fig.4 shows the surface roughness of EB irradiated surface under the condition of EB2. The surface roughness of all EDMed surfaces decreased by EB irradiation. The reduction ratio of surface roughness depends on the surface roughness of EDMed surface before EB irradiation.

In order to discuss the effect of EB irradiation conditions on reduction of surface roughness, each surface was EDMed under the condition EDM2, and then the roughness values of surfaces irradiated by EB under EB conditions EB1-EB4 were compared. As shown in Fig. 5, the surface roughness decreased by EB irradiation under all EB conditions, and a larger number of irradiation (30 shots) resulted in smaller surface roughness. In addition, EB irradiation at a higher energy density resulted in smaller surface roughness.

3.1.2 Microscopic Observation

EDMed surfaces under EDM2 condition and those then irradiated with EB were observed using an SEM. The observed images are shown in Fig. 6. Pinholes and micro-cracks on the surface generated by EDM disappeared by EB irradiation under the conditions EB1-EB3, and a smooth surface could be formed. However, pinholes and micro-cracks still remained on the surface irradiated under EB4 condition.

In order to investigate the state of surfaces in detail, cross-sections were observed with an optical microscope. The results of those observations are...
shown in Fig. 7. A resolidified layer with micro-cracks generated by EDM was clearly observed on the surface before EB irradiation, and its thickness varied from 8-15 μm. However, EB irradiation under the conditions EB1-EB3 showed resolidified layers with a uniform thickness of about 10 μm, and the micro-cracks almost disappeared. Also under EB4 condition, the micro-cracks disappeared, but the layer thickness was not so uniform. Moreover, it is confirmed that the resolidified layer consists of two layers with a somewhat indistinct boundary between them, when the cross-section of a surface irradiated under condition EB2 was observed in greater detail. This boundary appeared about 5 μm from the surface, and the material above this depth was probably once generated by EB irradiation. Therefore, a white resolidified layer with uniform thickness cannot be obtained by EB irradiation when the surface roughness of resolidified layer formed by EDM are larger than the layer thickness formed by EB irradiation. In addition, on the resolidified layer with a non-uniform thickness formed under EB4 condition, craters and pinholes remained according to the SEM image in Fig. 6 (e). This indicates that the energy density under EB4 condition was not sufficient to form a resolidified layer with a uniform thickness.
Cross-sections of EB-irradiated surfaces were also observed to investigate surfaces without resolidified layer before EB irradiation. The results are shown in Fig. 8. The formation of a resolidified layer on an EB-irradiated surface was clearly confirmed under all EB conditions. The layer was approximately 5 μm thick regardless of the EB conditions. Therefore, the boundary observed in the cross-section of an EB-irradiated surface after EDM is formed by surface resolidification as a result of EB irradiation, and the layer is approximately 5 μm thick. In other words, large-area EB irradiation can modify an EDMed surface to a depth of 5 μm, smoothing out the rough surface.

### 3.1.3 Residual Stress

Fig. 9 shows the measurement results of residual stress. A tensile stress of about 650 MPa was detected on an EDMed surface before EB irradiation. The tensile stress increases by the EB irradiated surface. Each surface under conditions EB1-EB3 had a high tensile stress of more than 800 MPa. However, the surface irradiated under EB4 condition had a lower stress than other EB-irradiated surfaces. In contrast, the tensile stress on milling surface before EB irradiation is almost zero. When surfaces were irradiated with an EB...
under any conditions, they had high tensile stress. This is presumably because the shrinkage of the molten material region was constrained by solid state material immediately below the molten material during EB irradiation. These results indicated that the residual stress of the surface was dominated by EB irradiation, regardless of pre-process, such as milling or EDM.

3.1.4 Analysis using X-ray diffraction

Milling surface, surfaces processed under EDM2 conditions and under EDM2+EB1-4 conditions were analyzed with X-ray diffraction patterns, in order to investigate these metallographic and crystal structures. The results are shown in Fig. 10. The diffraction peaks due to αFe, γFe, and Fe₃C were clearly noticed on the surface processed under EDM2 condition. When this surface was irradiated with EB under EB 1-4 conditions, the intensity of the diffraction peak due to αFe and γFe tended to increase. When a milling surface was irradiated with an EB, a similar tendency was found.

3.2 Properties of Nitrided Surface Layer
3.2.1 Cross-sectional material structure and hardness distribution

Gas nitriding and shot peening have been conventionally applied to improve a die’s resistance to heat fatigue. A high tensile stress remains on the EB-irradiated surface. Then, the EB-irradiated surfaces of testpiece were subjected to gas nitriding and the surface characteristics were evaluated. The conditions of EDM4 and EB2 were used to improve the surface roughness of testpiece in thermal fatigue test.

From the SEM observation and surface roughness measurement, it was clarified that gas nitriding did not bring change in surface appearance. However, material conditions inside the testpiece changed slightly. The cross-sections of testpiece prepared under EDM4+EB2 conditions and EDM4+EB2+GN conditions were observed by an optical microscope. Fig. 11 shows the observed images.

A hardened layer with non-uniform thickness formed during EDM was not observed on the EB-irradiated surface. However, a resolidified layer with uniform thickness was formed on the EB-irradiated surfaces overall. On the other hand, a layer of about 5 μm thickness was newly formed on the surface by gas nitriding. It is considered that this layer is the remaining of the resolidified layer generated by EDM and EB irradiation⁴, because the resolidified layer by EDM remained when the EDMed surface was treated by gas nitriding. Furthermore, thick nitrogen diffusion layer of about 50 μm can be confirmed immediately below the layer.

The hardness distribution along the depth direction was measured to investigate the effects of gas nitriding on hardness. Measurement results are shown in Fig. 12. The hardness of outermost layer on the surface processed under EDM4 condition reached about 750 HV as a result of the hardening
effects by EDM. When this surface was irradiated with an EB, the hardness decreased to about 580 HV, but it was still harder than that of base metal, 520HV. This is probably because the carbide in the resolidified layer formed by EDM partially decomposed by large area EB irradiation\(^5\). In addition, the material region immediately below the surface layer was softened as a result of tempering effects. Nonetheless, the resolidified layer remains even after EB irradiation, so it showed harder hardness than the base metal.

When the surface was further treated by nitriding, the surface hardness exceeded 1,000 HV. The region with a higher hardness than the base metal extended about 50 μm below the surface. Since the nitrogen diffusion layer corresponds with the depth of the hardened layer observed in the cross-section, gas nitriding greatly increases the hardness. The hardness tended to decrease in the cases of milling surfaces (milling, milling+EB2, or milling+EB2+GN), compared to the EDMed surface treated by EB irradiation and/or GN.

### 3.2.2 Thermal Fatigue Characteristics

Testpiece processed under EDM4+EB2 conditions and EDM4+EB2+GN ones were subjected to thermal fatigue test. The residual stress values of the surfaces measured at each cycle are shown in Fig.13. A tensile strength of about 900 MPa was present on the surface prepared under EDM4+EB2 conditions before the test. However, the strength soon decreased during the thermal fatigue test, and it becomes almost 0 at 50 cycles. The testpiece surface was smooth without cracks before the test. However, cracks generated on the surface during the thermal fatigue test. The residual strain was released as the cracks extended inside, and so the stress became 0. After that, a compressive stress subsequently remained up to 15,000 cycles, since the oxide was formed on the surface as the number of cycles increased.

When the surface processed under EDM4+EB2 conditions was nitried, the tensile stress on the outermost layer changed to compressive stress of about -700 MPa. The residual compressive stress decreased up to 50 cycles but the variation of the residual stress is as slight as about 200 MPa. The changes in stress was delayed, compared with that processed under EDM4+EB2 conditions without nitriding. The residual stress gradually changed afterwards, and the compressive stress of -400 MPa remained at 15,000 cycles. This is because the nitrogen diffusion layer with a high compressive stress given by nitriding remained immediately below the resolidified layer even after thermal fatigue test\(^6\). This is also considered that the crack generation on the surface would be inhibited by the remaining compressive stress in the nitrogen diffusion layer.

Next, the characteristics of the surface processed under EDM4+EB2+GN conditions during the thermal fatigue test were analyzed using an X-ray diffraction. Those spectra are shown in Fig.14. Decrease in \(\gamma\) Fe peaks and generation of \(\text{Fe}_3\text{N}_4\) ones were noticed in the case of the surface processed under EDM4+EB2+GN conditions. The decrease in \(\gamma\) Fe peaks depended on the tempering effect due to heat during the nitriding. After 10 cycles of the thermal fatigue test, the diffraction peaks of \(\text{Fe}_2\text{O}_3\) were newly detected in the both testpiece. The peak strength increased with the number of cycles. This is because oxide adhered to the testpiece surface, and it gradually accumulated on the surface with the number of cycles in thermal fatigue testing.

### 3.2.3 Crack Generation

Changes in surface conditions with the number of cycles in thermal fatigue testing were observed using an SEM. The SEM images are shown in Fig. 15. Crack generations were found at 10 cycles on the surface under EDM4+EB2 conditions. On the
other hand, there were no obvious cracks on the surface processed under EDM4+EB2+GN conditions even at 15,000 cycles. Cracks on the cross-section after 15,000 cycles of thermal fatigue test are shown in Fig.16. Wide-open and deep cracks were observed on the surface processed under EDM4+EB2 conditions. In contrast, narrow and shallow cracks were on the surface under EDM4+EB2+GN. Number of cracks in unit area and maximum crack length measured using the observation images are listed in Table 5. On the surface processed under EDM4+EB2+GN conditions, the number could be reduced to about one third of that under EDM4+EB2 condition. Also, the maximum length under EDM4+EB2+GN were about half of that under EDM4+EB2 condition. From these results, it is clarified that the crack generation and propagation could be decreased by gas nitriding. The propagation of cracks generated during the thermal fatigue test would be influenced by some factors, such as stress conditions on the surface by repeated compression and tensile stress, surface oxidation, and the embrittlement of material. When the EDMed surface was irradiated with an EB, chromium in the material is rearranged on the surface\([7]\). This phenomenon might provide a functional die surface with high corrosion resistance and oxidation resistance, which would inhibit the crack propagation. When the surface was further nitrided, the crack length became half as that on an EB-irradiated surface before nitriding, as shown above. This is probably caused by nitride formation on the surface and by an increase in heat fatigue strength with formation of nitrogen diffusion layer of about 50 \(\mu m\) immediately below the surface.
4 SUMMARY

The surface characteristics of molding dies treated by EDM, EB irradiation, and gas nitriding singly or in combination were experimentally investigated, and their resistance to thermal fatigue was evaluated in this study. Main conclusions are as follows;
1) When large area EB is irradiated on EDMed surface of molding die, the surface roughness can be improved with the energy density and number of irradiation of the EB.
2) When the EB is irradiated to the EDMed surface, the hardness slightly decreases, but it is still harder than that of the base metal. High hardness over 1,000 HV can be obtained by nitriding, and the depth of the hardened layer corresponds well to nitrogen diffusion layer of about 50 μm.
3) Large-area EB irradiation has dominant influence on the residual stress of the surface treated after the large-area EB irradiation, rather than pre-processes, such as milling or EDM.
4) The X-ray diffraction peak of γ Fe increases by large-area EB irradiation on the EDMed surface. Fe₃₄ generates and γ Fe decreases on the surface when the surface is nitrided.
5) Residual stress on the surface is released during an initial cycles of thermal fatigue test. Oxide adheres more to the testpiece surface as the number of cycles increases.
6) Crack generation and its propagation in thermal fatigue test can be inhibited by nitriding the EDMed and EB treated surface of molding die.

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