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# INFLUENCE OF RENITRIDING FOR THERMAL FATIGUE PROPERTIES ON NITRIDED HOT WORK DIE STEEL (H13)

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## ABSTRACT
This paper describes the effect of renitriding for hot work die steel. We noticed a decrease of compressive residual stress on the nitrided die surface during thermal fatigue process. X-ray stress measurement was used to detect the decomposition of nitride layer on hot work die steel (H13). As a result, compressive residual stress decreased gradually during the thermal fatigue test, almost restoring to the pre-test level by renitriding. The number of cracks decreased with the frequency of test by surface removal effect of shot peening while renitriding. Comparing the renitrided specimen with non renitrided specimen (single nitride), the number of cracks had decreased.

## 1.INTRODUCTION
On the surface of hot work die, such as die-casting die, heat cycles are applied during the operation. Then, the cracks occurred due to thermal stresses caused by thermal cycles.

To extend die-casting die life, nitriding is often applied to the die surface[1)]. Even with nitriding, the nitride layer is damaged by heat during the die-casting operation. Thus, if renitriding will be available with the detecting damaged layer quantitatively, extension of die life can be expected.

On the other hand, high compressive residual stress about –900MPa is caused on the surface of hot work die steel (AISI-H13) by nitriding. And the reheating decreases the residual stress[2)]. We noticed the compressive residual stress, and tried to detect decomposition of nitride layer in thermal fatigue process by X-ray stress measurement. And repair effect of nitride layer by renitriding with shot peening was investigated.

During the thermal fatigue test, renitriding was applied several times and changes of residual stress and half-value breadth were measured. After the test, distribution of residual stress, maximum crack length and number of cracks were measured.

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## 2.EXPERIMENTAL PROCEDURE

### 2.1 SPECIMEN PREPARATION

Specimens in the form of cylinders[3)] were manufactured from hot work die steel (H13). They were heat treated to 50HRC. Gas nitriding was performed after heat treating. To reduce the grain size of specimen surface and apply of compressive stress, specimen underwent shot peening. These conditions are shown in Fig.1.

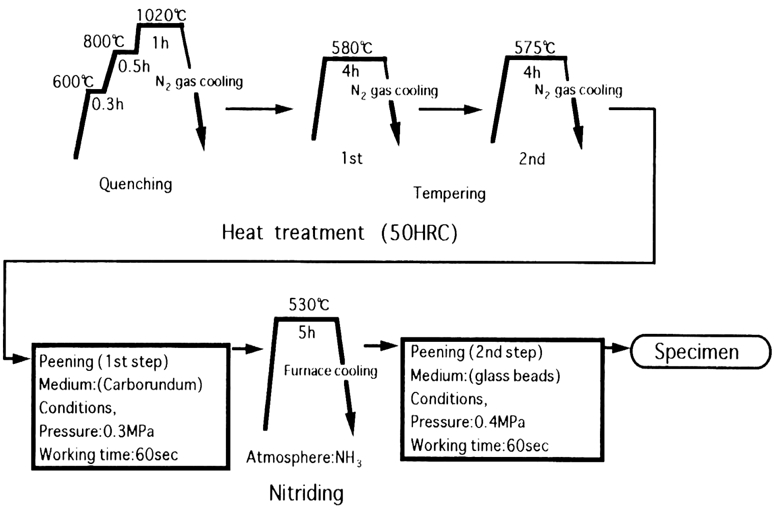


Fig 1 Conditions of heat treatment and nitriding

### 2.2 THERMAL FATIGUE TEST

Specimens were heated up to 843K (570°C) for 160 sec. by contact with heat block and cooled 373K(100°C) for 15sec. in a water bath[3)]. Heating temperatures used simulated the die surface temperature in the typical aluminum die-casting operation.

During the thermal fatigue test, specimens underwent renitriding every $5x10^3$ cycles. The test cycles were up to $1.5x10^4$ cycles. Hereafter, specimen tested $5x10^3$ cycles will be referred as specimen A, $1x10^4$ cycles as specimen B and $1.5x10^4$ cycles as specimen C. The interval of renitriding was decided by reduction of residual stress to 50% of its initial value[3)]. In addition, to investigate the effect of renitriding, each specimen after the test underwent renitriding. After the renitriding, distribution of residual stress was measured. This experimental flow is shown in Fig.2.

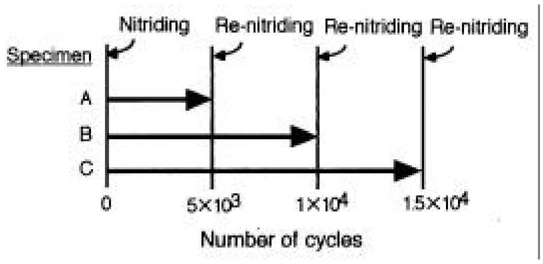


Fig2 Renitriding and testing condition

During the test, residual stress and half-value breadth were measured step by step. The values of each step were calculated from four isometric positions on the surface. Table 1 shows condition of X-ray stress measurement. The apparatus of X-ray stress measurement was a RIGAKU MSF-2M with a parallel beam system and Ω-goniometer. Because X-ray diffraction profiles showed broad αFe211 peak, its diffraction was used for X-ray stress measurement. In case of measurement for residual stress distribution, electrical polishing was used.

After the test, each specimen was cut and cracks in the sectional area were measured. The range of the crack measurement was 10mm in

Table 1 Conditions of X-ray stress measurement

| | |
|---|---|
| Diffraction | αFe211 |
| Target | Cr |
| Tube Voltage | 30kV |
| Filament Current | 8mA |
| Measuring Method | $\sin^2\psi$ |
| Stress Constant | -297 MPa/deg |

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width and the center of the range was located 15mm from the edge of the specimen[3)]. Total number of cracks and maximum crack length were measured in this range.

## 3.RESULT AND DISCUSSION

### 3.1 THERMAL FATIGUE TEST

Fig.3 shows changes of residual stress during the thermal fatigue test.

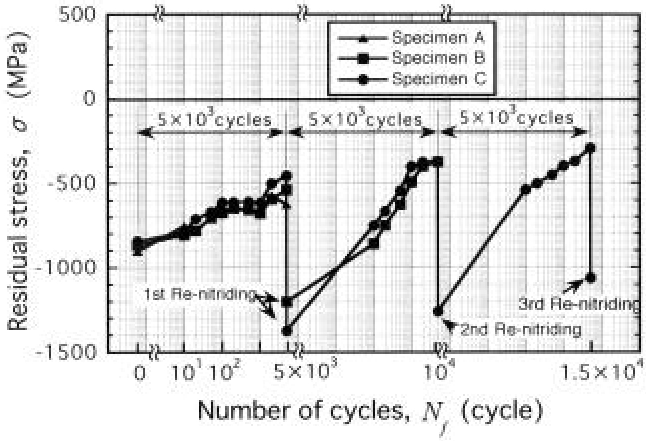

Fig3 Changes of residual stress during the test

Before the test, the values of compressive stress on the surface of every specimen were about –900MPa. This compressive stress decreased as test cycles increased and became approximately –500MPa at $5x10^3$ cycles. These values were almost half of initial stresses. It was considered that this phenomenon was the result of decomposition of nitrided layer by heating during thermal fatigue test and appearance of crack by thermal cycles. After the renitriding of specimens B and C at $5x10^3$ cycles, compressive stress increased to about –1300MPa.

At $1x10^4$ cycles, compressive stresses of specimen B and C as tested were about –400 MPa. After the renitriding of specimen C, the stress was –1300MPa. At $1.5x10^4$ cycles, it was –300MPa and –1100MPa after renitriding. These tendencies were almost the same as $5x10^3$ cycles. Also, at the end of each step of the thermal fatigue test, that is before renitriding, compressive stresses were approximately –500MPa, -400MPa and -300MPa. These values decreased as thermal cycles were increased.

Fig.4 shows changes of half-value breadth for αFe211 diffraction in the thermal fatigue test. The half-value breadth of the peak was used at ψ=9° in the measurement of residual stress in Fig.3. During the test, half-value breadth of each specimen decreased. Half-value breadth increased by renitriding which is the same as the tendency for residual stress. But the amount of increased half-value breadth was 0.8 deg. for specimen B ($1.0x10^4$ cycles). Comparing the tendency of half-value breadth with residual stress, the increase level of half-value breadth by renitriding was not high. In this experiment, a factor of change for half-value breadth was considered as non-uniform strain. The causes were quenching and tempering, nitriding and shot peening as shown in Fig.1. Thus, non-uniform strain on the specimen surface was formed for each process.

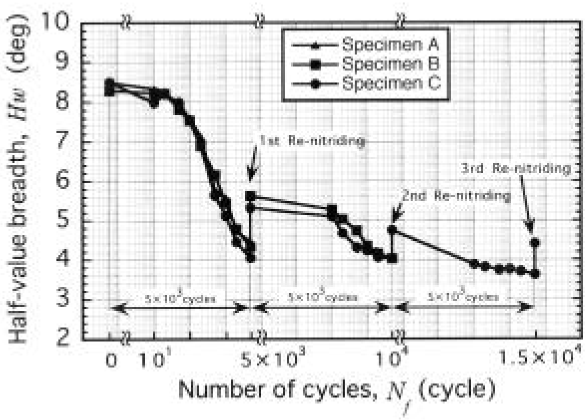

Fig.4 Changes of half-value breadth during the test

On the other hand, heating temperature 843K (570°C) at the thermal fatigue test is near the temperature of tempering. Then, decrease of half-value breadth during the thermal fatigue test was due to the release of stored non-uniform strain by tempering of matrix and decomposition of nitride layer.

At renitriding, non-uniform strain near the surface was reformed at renitriding by nitriding and shot peening. It was considered that non-uniform strain in the matrix was not recovered. Then increase of half-value breadth at renitriding was not fully recovered.

### 3.2 CHANGES OF SURFACE LAYER BY RENITRIDING

Fig.5 shows the curves of residual stress distributions for each specimen. In this figure, the result for the specimen before the test is shown with a white triangular mark. Also, non nitrided specimen after $1.5x10^4$ cycles is shown with a black triangular mark.

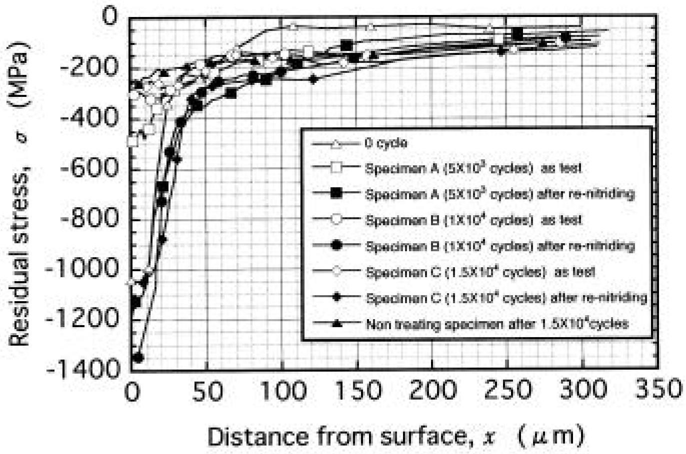


Fig.5 Distribution curves of residual stress

Before the test, the residual stress on the surface was about –1050MPa. It decreased as the distance from surface increased and became –200MPa at 100μm. At $5x10^3$ cycles of the test, the residual stress on the surface decreased to –500MPa. This phenomenon was observed in the diffusion layer. The range of residual stress increased to approximately 200μm. After renitriding, residual stress on the surface was over –1000MPa which was as the same level as before the test. Also, the distribution of every renitriding specimen showed almost the same curve.

Fig.6 shows the curves of half-value breadth for each specimen. The marks in the figure are the same as Fig.5.

Before the test, the half-value breadth on the surface was about 6.8 deg.. It decreased to 4.0 deg. at about 40μm as the distance from surface increased. At $5x10^3$ cycles of the test, the half-value breadth on the surface decreased greatly to 4.2 deg.. The converging depth of the half-value breadth increased to about 200 μm. It coincided with the residual stress distribution curve. The heat cycle range which was the maximum reduction of half-value breadth in the matrix was from 0 to $5x10^3$ cycles. After this range, the reduction range of half-value breadth became smaller.

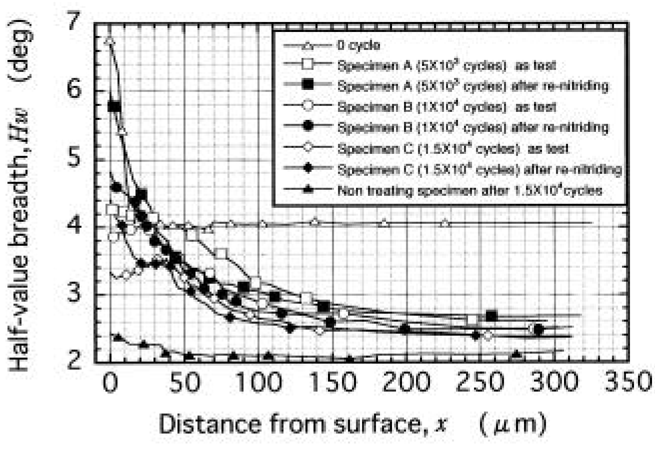


Fig.6 Distribution curves of half-value breadth

After the renitriding, though the half-value breadth increased in the range from 0 to 50μm of the depth from surface, a large change was not observed at the inside of the range. Also, it became clear that in the diffusion layer, the effect of

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renitriding for half-value breadth was smaller than that for the residual stress as shown in Fig.5.

### 3.3 OBSERVATION OF CRACKS

Fig 7 shows the result of crack observations for specimen A(5x10$^3$ cycles) as tested and after renitriding.

The oxide layer with a thickness of approximately 10μm, existed on the surface. Underneath the oxide layer, the cracks with forming oxide with a length of approximately 5 μm was observed. Also, the range of diffusion layer observed as a black color was about 125 μm. The range tended to be somewhat deeper than before the test.

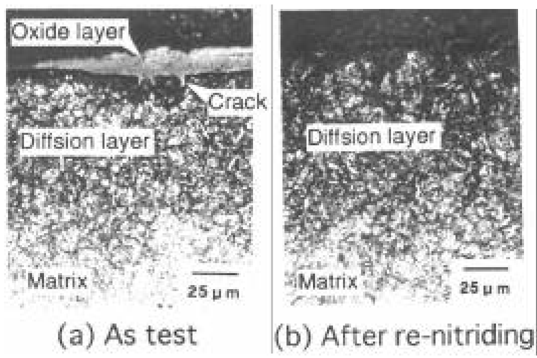


Fig.7 Microphotographs of sectional area of specimen A(5x10$^3$ cycles)

After renitriding, the oxide layer on the surface had disappeared and the cracks below the surface had almost disappeared due to the removal effect of shot peening while renitriding. This situation was almost the same as the other specimen.

Fig.8 shows the maximum crack length and the number of cracks in the sectional area for each specimen after the thermal fatigue test.

The maximum crack length increased from approximately 20 μm to 90 μm with the increase of test cycles. Conversely, the number of cracks decreased from 520 to 140. It was considered that shot peening treatment before and after renitriding removed the micro-cracks, and reduced the number of cracks. Although the micro-cracks were removed, stress concentration occurred at residual cracks, crack growth was accelerated, and maximum crack length increased.

Fig.9 shows the comparison of crack measurement for specimen C, non-treating specimen (not nitrided) and a single nitrided specimen (not renitrided).

Comparing specimen C with non-treating specimen, the maximum crack length and the number of cracks of specimen C decreased. Especially, the number of cracks decreased

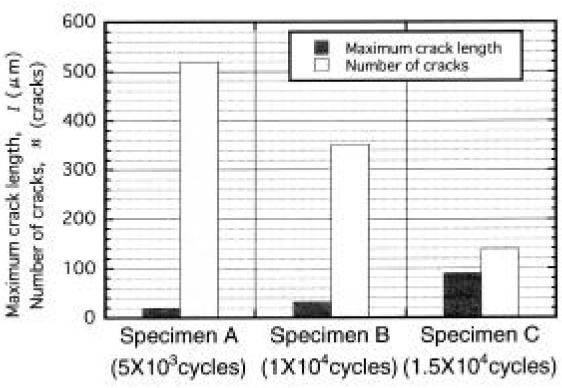


Fig.8 Result of crack measurement for the specimens

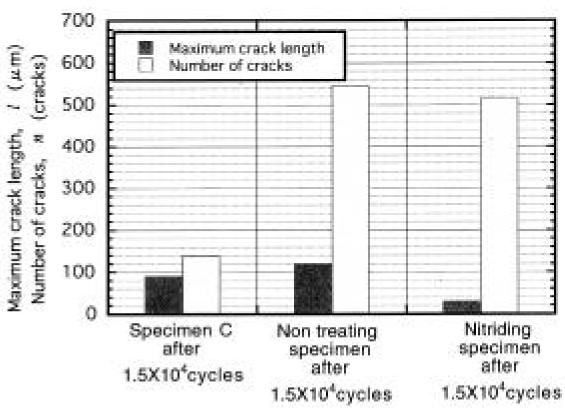


Fig.9 Comparisons of the crack measurement for various specimens

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remarkably. The cracks in specimen C were approximately 1/4 of non-treating specimen.

On the other hand, comparing nitriding specimen with specimen C, though the number of cracks of the former were 520 and the latter were 140, the maximum crack length of the former specimen was 30μm and the latter was 90 μm. This result was due to the removal of micro-cracks by shot peening while renitriding, which is the same result as shown in Fig.8.

Thus, it was clarified that shot peening while renitriding during the thermal fatigue process keeps a stable surface layer by removing micro-cracks and providing compressive residual stress. Accordingly, it is considered that suitable renitriding to nitrided die-casting die during operation maintains a good surface. And it will be an effective method to increase the die life. Also, a notable change of residual stress by degradation of nitride layer and renitriding was observed during the thermal fatigue test. Based on this result, it was clarified that X-ray stress measurement is the effective method and residual stress value being a good parameter to decide on the most suitable time to renitride.

## 4.CONCLUSION

Nitrided hot work die steel specimens were renitrided in the thermal fatigue process. The effect for surface layer was investigated by residual stress, half-value breadth and crack measurement. The results obtained were as follows;

1) During the thermal fatigue test, the residual stress and the half-value breadth of the surface layer decreased gradually. The residual stress was almost restored to the specimen before the test by rinitriding. Though half-value breadth had the same gradual decreasing tendency, the recovered value was less than the residual stress due to temper of matrix.
2) In the crack observation of the sectional area for specimen A($5x10^3$ cycles) as tested and after renitriding, the cracks were observed underneath the oxide layer. After renitriding, the oxide layer on the surface had disappeared and the cracks were almost disappeared.
3) In the crack measurement during and after the test, the number of cracks decreased with the increased number of test times by surface removal effect of shot peening while renitriding.
4) Comparing the renitrided specimen with non renitrided specimen (singe nitride), the number of cracks had decreased.

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