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Fatigue Property of Stainless Steel by Ion Implantation

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The effect of residual stress by ion implantation on fatigue property of stainless steel plate have been studied. The fatigue specimen was austenitic type 304-CSP stainless steel plate with 2.0 mm thick. Fe ion implantation was carried out at energy of 3 MeV at the dose of 3.0×10^{16} at 100 K because the previous research showed stainless steel by ferrous implantation led to higher compression residual stress. The test results show that Fe implantation led to compression residual stress and reduced the fatigue crack initiation strength. There was no difference in the fatigue crack propagation rate of implanted and non-implanted specimens.
Key word: reuse, fatigue, ion implantation, residual stress, stainless steel

1. INTRODUCTION

Recently, saving resource, energy conservation and waste reduction have been very important social issues in the present industry. The recycling and reuse of products are actually recognized as the ways for achievement of them. Comparing with the recycling, the necessity of reuse is only proposed and its actual realization has not been necessarily achieved, except for cases of some parts of products, such as portable camera and copy machine. However, the reuse has the potential to become a dominant way from the view of the cost, because the recycling needs generally high cost.

The main reason why the reuse is not necessarily realized is that the concept and methodology for the evaluation of reusability for each part or product have not been established. In the scenario of the reuse of machine structural parts or products, we should evaluate their reusability, but we cannot generally evaluate whether they keep the original qualities.

Most important factor on the reusability in the case of machine structural parts is strength. Safety factor is generally introduced for strength design to secure safety on strength.

Although there are some methods to investigate the existence of damage of parts, like the nondestructive testing for using X-ray, ultrasonic or eddy current etc, the methods are not applicable to all the subjects because they are inefficient in cost and, in some cases, it is impossible to apply the methods due to the technical constraint.

Parts with the history of higher loading than designed stress could not be reused. In the previous paper by the authors, the concept for detecting sensor of overload applying to machine parts under use was proposed [1]. This concept is similar to electrical fuse in electrical appliances and is explained in Fig. 1. Pre-stressed thin plate (sensor) which is attached to the part is useful. If the overload is applied to the part under use, the sensor is damaged by priority. In the previous paper, the sensor (thin plate with a notch) inducing higher stress than one by applied load is also developed by using stress concentration based on geometrical change of sensor [1]. In the case of usual fatigue design, design stress is designed at the lower stress level than fatigue limit. The ratio of the fatigue limit stress and the designed stress is safety factor. If the stress concentration of the sensor with a notch is larger than the ratio, maximum stress in sensor is reached to fatigue limit. So, fatigue crack must be initiated in sensor earlier than structure of machine part. So, the history of overloading can be detected. Stress concentration is not necessarily related to fatigue strength property [2].

On the other hand, ion implantation technique gives a possibility that macroscopic residual stress of tension and compression can be induced in some conditions of ion implantation [3]. If we could induce both of tension and compression residual stress in a sensor (thin plate), we would get the high potential to design the sensor.

In the present paper, we focus on fatigue strength of design stress. A sensor for detecting overload to parts, and the effect of ion implantation on the fatigue properties of sensor material is investigated. Especially, the relationship between the fatigue properties and macroscopic residual stress induced by ion implantation is considered. The fatigue property is defined as the number of cyclic loading at fatigue crack initiation. In Fig. 1, fatigue limit and designed stress are shown. The fatigue limit means the number of fatigue crack initiation. So, if the pre-stress, the value of which is equal to or greater than the difference between fatigue limit stress and designed stress, is induced to sensor material, the load over designed stress causes crack initiation. As shown in Fig. 2, a thin plate sensor which is bonded to machine part by adhesive or spot welding, only have to detect the crack, and we can judge the parts can be reused.

However, ion implantation might cause metallurgical effect on fatigue properties of material during cyclic loading (fatigue process). The relationship between the behavior of fatigue crack initiation and ion implantation has been hardly investigated. So, in this research, a basic experiment is carried out to capture the phenomenon on the relationship.

As a thin material used for a sensor, the austenitic type 304 stainless steel is assumed due to its high resistance to corrosion. Because, no research has been made yet on the fatigue property of the austenitic type 304 stainless steel by ion implantation from a view point of residual stress.

2. EXPERIMENTAL

The specimens were implanted of ferrous ions Fe^{2+} because compressive residual stress about 125 MPa induced by ferrous implantation into the stainless steel sheet was confirmed [3]. In reference [3], residual stress was measured by cantilever type testing method. The method cannot be applied to the present specimen. So, in the present study, the residual stress for the ion implanted specimen was measured by the nano-indenter technique.

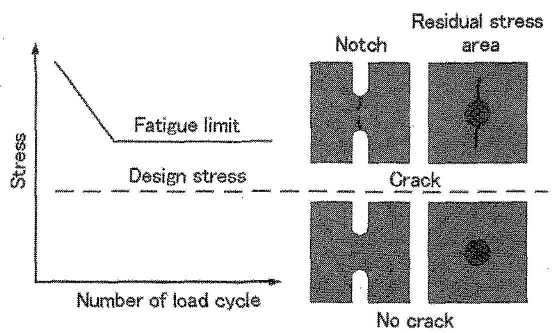

Fig. 1. The reusability evaluation by using the existence of a larger fatigue load history than design load.

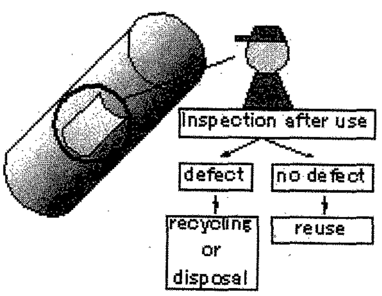

Fig. 2. The concept of the reusability evaluation.

2.1 Preparation of specimens

The compositions of the austenitic type 304-CSP stainless steel used are given in wt%: C 0.05, Si 0.62, Mn 0.80, P 0.031, S 0.002, Cr 18.12, Ni 8.14 and Fe in balance. The fatigue specimens are machined into the geometry shown in Fig. 3. After machining, a surface hole with a diameter 0.3 mm × depth 0.2 mm is made in the center of one side of specimen by electric discharge machining in order to make the fatigue crack initiation zone pinpointed. After mirror finish by buffing and ultrasonic cleaning, ferrous ion implantation was carried out at 3 MeV with a dose of 3×10^{16} ions/cm² on one side with a hole. The specimen was held on a specimen holder for the temperature control with liquid nitrogen and a heater with thermocouple feedback control. Specimen temperature was controlled with high precision at 100 K. The ion beam current density was kept below 1 μA to maintain the change of the specimen temperature within ±1 K. During all implantation, the vacuum in implantation chamber was maintained below 1×10^{-5} Pa. The non-implantation specimens were prepared for comparison.

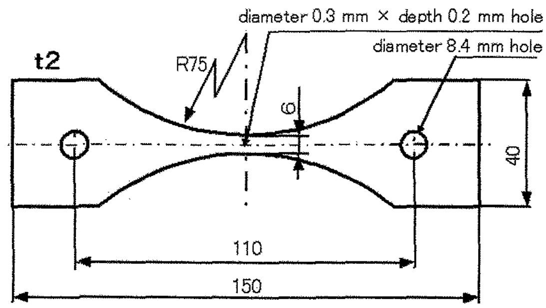

Fig. 3. Schematic of fatigue specimen geometry.

2.2 Fatigue experiment

The fatigue experiment was performed by oil pressure servo type fatigue testing machine. Experiments were carried out at a frequency of 10 Hz, stress ratio of R = 0.05 and room temperature. Loading cycle was ended to the number of cycles, $N = 1.0 \times 10^6$. After the crack initiation, the crack length was measured at several times during crack propagation to about 1 or 2 mm. The number of crack initiation (the crack length, a, = 0) was approximately calculated by linear least-squares method applying for the relation of crack length and number of loading cycle. The effect of ion-implantation on the fatigue crack propagation rate, da/dN, was considered, based on fracture mechanics. The crack propagation rate was obtained experimentally by measuring the crack length microscopically and the number of loading cycle. The fracture mechanics parameter used is the stress intensity factor range, ΔK, for the crack initiated from a surface circular hole, which is obtained from reference [4].

2.3 Residual stress measurement

We attempted to measure the residual stress in the ion implanted layer by X-ray diffraction, but the meaningful data were not able to be obtained. The ion implanted layer thickness was very thin. So, instead of X-ray, the residual stress was investigated by indentation experiment which is proposed by Jang et al. [5]. This experiment was carried out by the nano-indenter instrument. The indentation experiments were performed on the ion-implanted and non-implanted areas of the specimens at room temperature using a Berkovich indenter. Indentation depth was gradually increased to 500 nm for 5 s and then reversed down to zero depth for 5 s. The indentation depth (500 nm) is determined by the simulation result of implanted ion distribution (Fig. 4) obtained by simulation tool of TRIM [6]. From the result, there is a possibility that tensile residual stress is generated at the shallower depth, such as 0.5 μm, than the peak position (0.9 μm) and affects the fatigue crack initiation.

3. RESULTS AND DISCUSSION

3.1 Fatigue experiment

The fatigue test results are shown in Fig. 5 that shows the relationship between maximum stress of the specimen and number of cycles to crack initiation. The maximum stress was calculated by finite element method (MARK) and obtained at the position of the surface hole end with specimen's minimum width of 6 mm. In the results, the crack initiation life of the implanted specimens was reduced, as compared to that of the non-implanted specimens in each load levels. The results showed a possibility that the fatigue lifetime was reduced by the residual compressive stress. In past studies about the effect of ion implantation on fatigue properties [7,8], most specimens were implanted between tens keV and hundreds keV. In this study, the specimens were implanted at 3 MeV, which was comparatively higher energy. In the present experimental conditions, the peak of the depth distribution of Fe ions was around 0.9 μm from the surface (Fig. 4). If there is the peak of the residual compressive stress in the peak of the depth distribution of Fe ions, the residual tensile stress might be induced near the surface of specimen by the condition of equilibrium stress. It was thought that the crack initiation life of the implanted specimen was reduced by the tensile stress near the surface. The crack initiation generally happened in sub-micrometer scales.

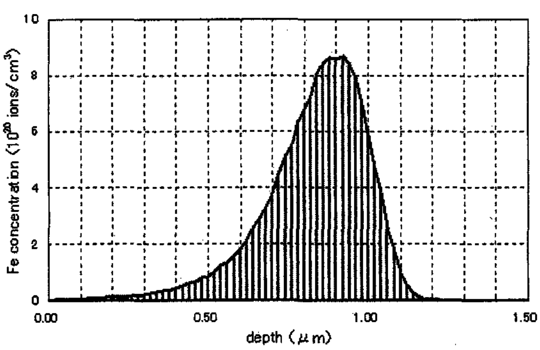

Fig. 4. Depth profile of Fe ions simulated by TRIM.

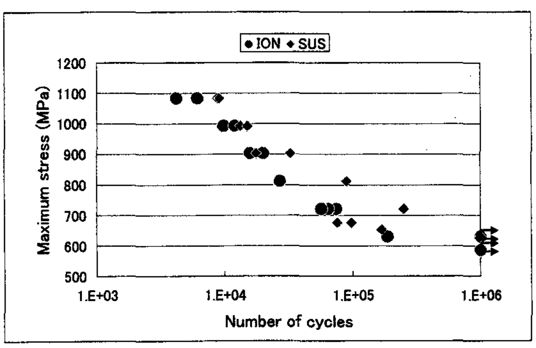

Fig. 5. Relationship between maximum stress and number of cycles to the crack initiation. (●ION: ion-implanted specimen, ◆SUS: non-implanted specimen)

The relationship between fatigue crack propagation rate and stress intensity factor range that is fracture mechanics parameter is shown in Fig. 6. This figure shows there were no major difference between crack propagation rate of ion implanted specimens and that of non-implanted specimens. The depth of the ion-implanted layer of these specimens was about 0.9 μm according to the result simulated by TRIM. While the crack was initiated in sub-micrometer scales, the crack was propagated in hundreds micrometer scale. In this study, ion implantation did influence not to the crack growth, but to the crack initiation. It might be that the metallurgical change of the ion-implanted specimen material might occur, which influenced the crack initiation.

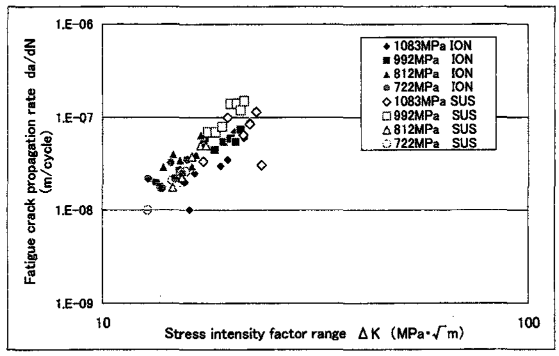

Fig. 6. Relationship between fatigue crack propagation rate and stress intensity factor range.

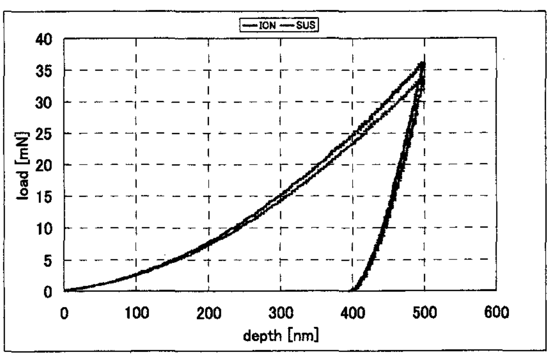

Fig. 7. Typical indentation load-depth curves for ten indentation on the ion-implanted and the non-implanted stainless steel plates.

3.2 Residual stress measurement

Typical indentation load-depth curves for ten experiments of indentation to the ion-implanted stainless steel plate and the non-implanted stainless steel plate are shown in Fig. 7. The residual stress was estimated by using these indentation results. The values used for the calculation of the residual stress are shown in Table I. Initial slope of unloading curve was the approximated value at the maximum loading depth of 500 nm using least squares approximation polynomial.

By applying the experimental data to the following evaluation equation [9], the residual compressive stress σ_{res} induced by implantation was calculated to be 367 MPa.

$$\sigma_{res} = P_i/A_{ci} - P_o/A_{co}$$

where P is the maximum load at the prescribed indentation depth, A_c is the contact area of indenter and material at the prescribed depth, and the subscripts, i and o, are ion-implanted and non-implanted cases, respectively. In this indent condition, it is thought that the influence of the substrate has been received, because the implantation layer is thin compared with the indent depth. However, when non implanted specimen and ion implanted specimen are loaded and indented to the same depth, ion implanted specimen requires more load than non implanted one does. This would be caused by the effect of implanted ion although this result is qualitative. It is reported that the material structure of the implantation area is changed by the ion implantation [10]. This structure change might influence the fatigue property. While the reliability of this residual stress value needs further discussions, it can be mentioned that the residual compressive stress induced by this implantation was hundreds MPa scale.

From Table I, it is found that the surface of ion-implanted specimen is a little harder than non-implanted SUS. It needs further consideration about the relationship between the tendency of the hardness, the residual stress of the specimen surface and the fatigue crack behavior.

Table I. Values for the calculation of the residual stress.

		ION	SUS
Maximum load	Pmax [mN]	36.17	33.92
Maximum depth	hmax [nm]	500	500
Initial slope of unloading curve	S [mN/nm]	0.456	0.406
Contact depth	hc [nm]	440.5	437.3
Contact area	Ac [nm^2]	4.75×10^6	4.69×10^6

4. SUMMARY

In this study, as a basic study to develop the sensor for the reusability evaluation of machine part using the residual stress, fatigue test and nano-indentation experiment were carried out on type 304-CSP stainless steel thin plate implanted by ion of Fe^{2+}.

As a result of fatigue test, the crack initiation life of implanted specimens was reduced, comparing to that of non-implanted specimens, and there was no major difference between fatigue crack propagation rate of ion implanted specimens and that of non-implanted specimens. From the experimental result, it was found that the residual compressive stress induced by ion implantation was the hundreds MPa scale. For practical application of sensor for the reusability evaluation using residual stress by ion implantation, it is necessary to investigate quantitative evaluation of fatigue experiments under the conditions of depth change of implantation giving the influences of residual stress.

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