

Fatigue Strength Reduction of Notched Component in Hydrogen Gas after Multiple Overloading

Masanobu Kubota^{1, a}, Junichiro Yamaguchi^{2, b} and Yoshiyuki Kondo^{1, c}

¹Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka 819-0395 Japan

²Graduate School of Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka 819-0395 Japan

^akubota@mech.kyushu-u.ac.jp, ^bte207386@s.kyushu-u.ac.jp, ^cykondo@mech.kyushu-u.ac.jp

Keywords: Hydrogen, Fatigue, Overload, Stainless Steel, Earthquake

Abstract. High-cycle fatigue test after multiple overloading was performed to develop a structural integrity assessment method for hydrogen utilization machines suffered seismic load. To simulate the material used in hydrogen gas for a long period, the hydrogen charged specimen whose hydrogen concentration was 80ppm was used. Three kinds of notched specimens with different notch root radii were used. The fatigue test in air using uncharged specimen was also performed. The fatigue limit of $\rho = 0.2\text{mm}$ specimen tested in air was decreased to almost one half by the application of 200 cycles overload with the amplitude of $1.25\sigma_{0.2}$. The decrease was caused by the microcracks generated by the overload. The number of cycles of overload corresponded to only 0.67% of the low-cycle fatigue life for the overload. Since no reduction of the fatigue limit was observed by the overload of $0.75\sigma_{0.2}$, there is a threshold value of overload that does not cause the reduction of high-cycle fatigue strength. The fatigue strength of hydrogen charged specimen tested in hydrogen gas was lower than that of uncharged specimen tested in air. Deeper microcracks were formed by the overload in the hydrogen charged specimen than in uncharged hydrogen.

Introduction

Utilization of hydrogen as an energy source has been strongly promoted expecting that it would be one of the solutions against energy problem and global warming. On the other hand, since Japan is one of the quake-prone countries, machines and structures are sometimes damaged due to a large-sized earthquake. Therefore, development of structural integrity assessment method of hydrogen utilization machines and structures after the experience of earthquake is required.

In the present study, high-cycle fatigue strength after multiple overloading was investigated. Since the test was intended to evaluate the fatigue strength of notched part in hydrogen utilization machines and structures, a notch was introduced and hydrogen was charged to the fatigue specimen. The points of interest in this study are the reduction of high-cycle fatigue strength after overloading due to the effect of hydrogen and the threshold of overload amplitude that no reduction of fatigue strength is caused.

Experimental Procedures

Material. The material used in the experiment was an austenitic stainless steel SUS304 designated by Japanese Industrial Standards. The material is equivalent to AISI 304 stainless steel. The chemical composition is shown in Table 1. The material was solution heat-treated. The mechanical properties are shown in Table 2.

Table 1 Chemical composition of SUS304 used in the experiment [mass%].

C	Si	Mn	P	S	Ni	Cr
0.05	0.26	1.32	0.036	0.028	8.03	18.32

Table 2 Mechanical properties of SUS304 used in the experiment.

Heat treatment condition	Proof strength $\sigma_{0.2}$ [MPa]	Tensile strength σ_B [MPa]	Elongation at fracture δ [%]	Reduction of area ϕ [%]
Solution	224	582	64	76

Fatigue specimen. Figure 1 shows the test specimen. The specimen was a round bar with a circumferential notch. A 2.5mm deep notch was introduced to constrain the macroscopic plastic deformation of the specimen by the overload. The specimen was to simulate a notched part of machine. The notch root radii of $\rho = 0.2, 0.5$ and 0.75mm were used. The notch was introduced by a lathe using formed tool. The notch root was polished lightly by Emery paper.

Fatigue test. Axial loading fatigue test was done using electro-hydraulic fatigue testing machines, which are specially manufactured to perform fatigue test in hydrogen gas. Figure 2 shows the loading pattern. Reversed multiple overloads were initially applied to the specimen for 200 cycle and then it was followed by a high cycle fatigue test with pulsating tension. The number of cycles of overload is also influencing factor. A fixed number of cycles was used in the experiment to simulate the earthquake. The overload was unloaded from compressive side to create tensile residual stress for the conservative evaluation. The loading frequency was 1Hz for the overload and 20Hz for the following high cycle fatigue. The amplitude of overload was chosen as 0.75, 1.0, and 1.25 times the proof strength of material $\sigma_{0.2}$.

The test environment was air and hydrogen gas. The temperature of both environments was ambient. The pressure of hydrogen gas was 0.6MPa. The purity of hydrogen gas was 99.999%. To reduce the residual air in the gas chamber, evacuation using a diffusion vacuum pump and high purity nitrogen gas purge were repeated 3 times before the injection of hydrogen gas. The achieved pressure in the evacuation process was less than 0.005Pa.

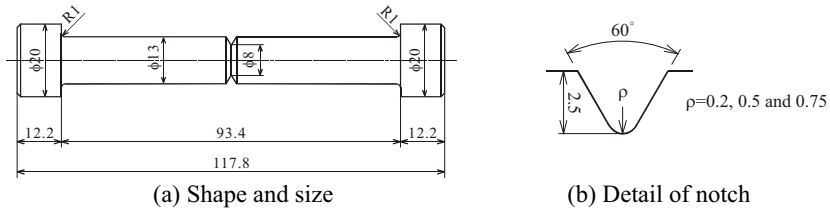


Fig. 1 Fatigue specimen (Dimensions are in mm).

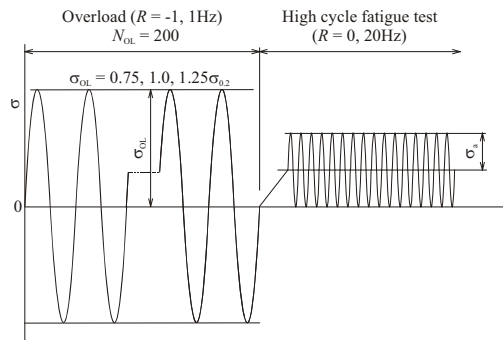
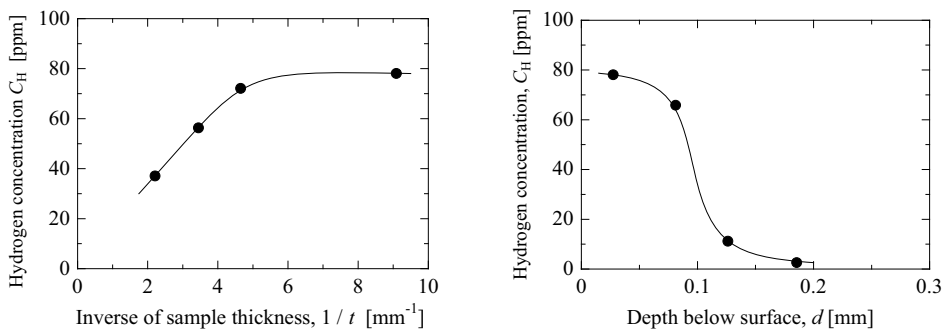


Fig. 2 Loading pattern for high cycle fatigue test after multiple overloading.

Hydrogen charge. Hydrogen charged specimen was used to simulate the material after a long-term service in hydrogen gas. The absorption of hydrogen in the material can occur in the components used in high-pressurized hydrogen gas. Considering long-term service, it is preferable to use a specimen which contains high concentration of hydrogen. Therefore, the hydrogen charge to the fatigue specimen was done by an accelerated method. The method was to expose the specimen in high-pressurized hydrogen gas at elevated temperature. The hydrogen pressure was approximately 76MPa and the temperature was 378K. The exposure time was 100h. The samples for hydrogen concentration analysis were also exposed in the same hydrogen gas. The samples with different thickness were used for the analysis. The concentration of hydrogen in the samples was measured by a thermal desorption analysis. The result is shown in Fig. 3. As shown in Fig. 3(a), the hydrogen concentration increased and leveled off with a decrease of the sample thickness. This means that the hydrogen concentration reached a saturated value under the hydrogen charge conditions used in the experiment. Fig. 3(b) shows the distribution of hydrogen concentration beneath the surface estimated from the above result. A high concentration of hydrogen was kept approximately 100 μ m beneath the surface. The concentration rapidly decreased in deeper position.



(a) Mean concentration in different thickness sample (b) Estimated distribution below surface
Fig. 3 Result of thermal desorption analysis on the absorbed hydrogen in the material.

Results and discussions

Effect of overload on high cycle fatigue strength. The effect of overload on the fatigue strength of notched specimen is shown in Fig. 4. The stress amplitude was defined by the nominal stress at the notched section. The high-cycle fatigue strength without overloading was varied depending on the notch root radius. The constant amplitude test results without overloading (baseline) were almost the same for $\rho = 0.5$ and 0.75mm notched specimens. It is because that the stress concentrations of both specimens were not so much different. The base line of $\rho = 0.2\text{mm}$ specimen was lower than those of other specimens. The application of overloads caused a reduction of high-cycle fatigue strength.

The amount of decrease in high-cycle fatigue strength by the overload was dependent on the notch root radius as well as the overload amplitude. Figure 5 shows the effect of overload amplitude on the reduction of fatigue limit in high-cycle fatigue test following the overload. In the case of $\rho = 0.2\text{mm}$, the fatigue limit was almost one half of the baseline when the overload amplitude was $1.25\sigma_{0.2}$. No decrease was observed by the overload of $0.75\sigma_{0.2}$. For the notched specimens with $\rho = 0.5$ and 0.75mm , the decrease of fatigue limit was smaller than that of $\rho = 0.2\text{mm}$ specimen. These data show that there is a threshold value of overload that does not cause the reduction of the high-cycle fatigue strength.

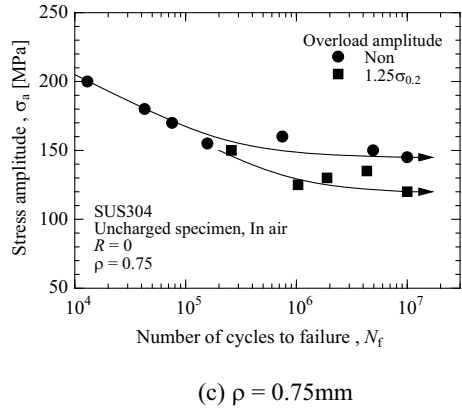
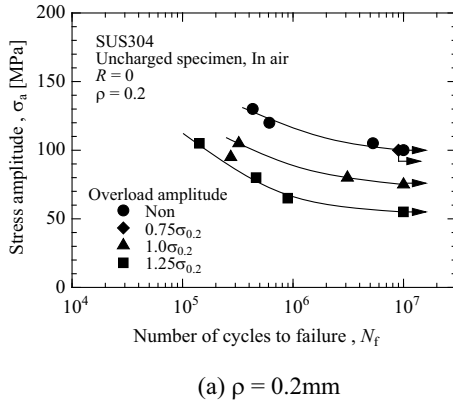


Fig. 4 High-cycle fatigue strength following the application of multiple overloads.

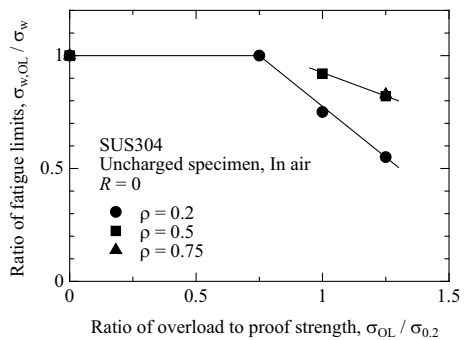
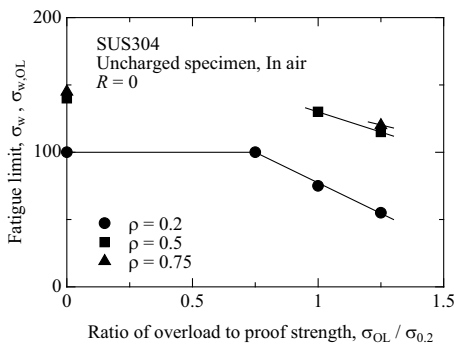
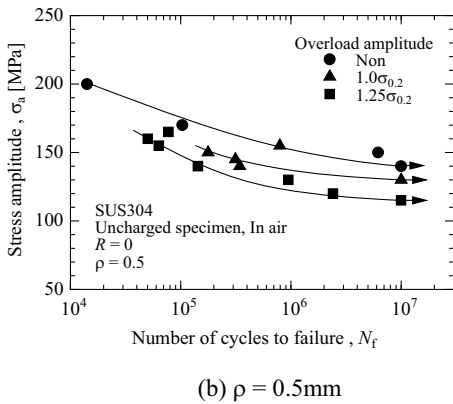


Fig. 5 Decrease of high-cycle fatigue limit after overloading.

Fatigue damage. To evaluate the fatigue damage introduced by overload, the fatigue lives for the overload were examined. Figure 6 shows the result of low cycle fatigue test. The fatigue lives were in

the range from several thousand to a few tens of thousand cycles. For example, in the case of the overload of $1.0\sigma_{0.2}$, The 200 cycles overload corresponded to only 0.67% of the low-cycle fatigue life for $\rho = 0.2\text{mm}$, and 0.18% for $\rho = 0.5\text{mm}$. The application of such small numbers of overload cycles caused substantial decrease in high-cycle fatigue strength. The Miner's liner accumulation rule was applied to evaluate the decrease of fatigue life after overload. The result is shown in Table 3. Irrespective of the overload conditions and notch root radii, the damages were much smaller than 1.

In order to investigate the cause of reduction in fatigue strength by overload, fatigue cracks in run-out specimens, which were opened at higher fatigue load after heat tint, were observed. Figure 7 shows the microcracks observed at the fracture surfaces. Microcracks were observed only in the specimens that experienced large enough overload causing the reduction of fatigue limit. No microcrack was observed in the specimen showing no decrease of fatigue limit by overload. There was no crack in the run-out specimen without overload. The depth of microcrack increased with the overload amplitude. Therefore, it can be considered that the fatigue limit following overload is governed by the threshold whether the microcracks propagate or not.

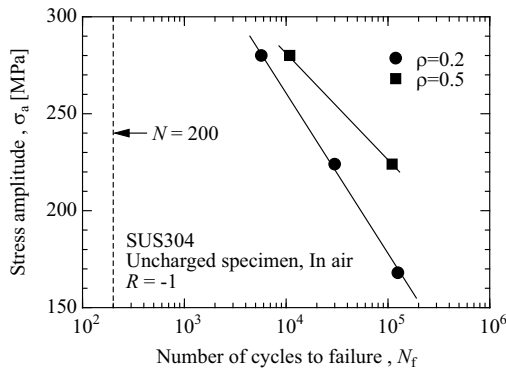


Fig. 6 Low-cycle fatigue test in the range of overload.

Table 3 Evaluation of fatigue damage based on Miner's liner accumulation rule.

Notch root radius ρ [mm]	Overload				High-cycle fatigue				Miner's damage value A+B
	Amp. $\sigma_{a,OL}$	Applied cycles N_{OL}	LCF life $N_{f,OL}$	$N_{OL} / N_{f,OL}$ (A)	Amp. σ_a	Fatigue life after overload N_f^*	Fatigue life (No overload) N_f	N_f^* / N_f (B)	
0.2	$1.0\sigma_{0.2}$	200	30,024	0.0067	105	322,298	5,294,418	0.061	0.068
0.2	$1.25\sigma_{0.2}$	200	5,710	0.035	105	142,003	5,294,418	0.027	0.062
0.5	$1.0\sigma_{0.2}$	200	110,119	0.0018	150	177,316	4,928,370	0.036	0.031

Effect of hydrogen. The fatigue test result of hydrogen charged specimens in hydrogen gas is shown in Fig. 8. The fatigue strength of hydrogen charged specimen tested in hydrogen gas was lower than that of uncharged specimen tested in air. To make clear the cause of more reduction of fatigue limit due to hydrogen, the low cycle fatigue life of hydrogen charged specimen was obtained. The result is shown in Fig. 9. The low-cycle fatigue life of hydrogen charged specimen in hydrogen gas decreased significantly. Figure 10 shows the non-propagating crack observed in the hydrogen charged specimen which run-out 10^7 cycles of high cycle fatigue loading after overload. A little deeper cracks were found in the hydrogen charged specimen comparing with the cracks found in the specimen tested in

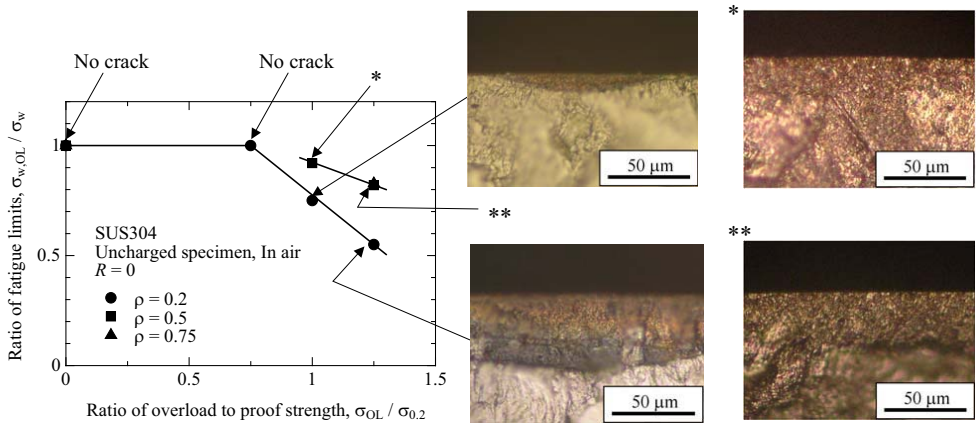
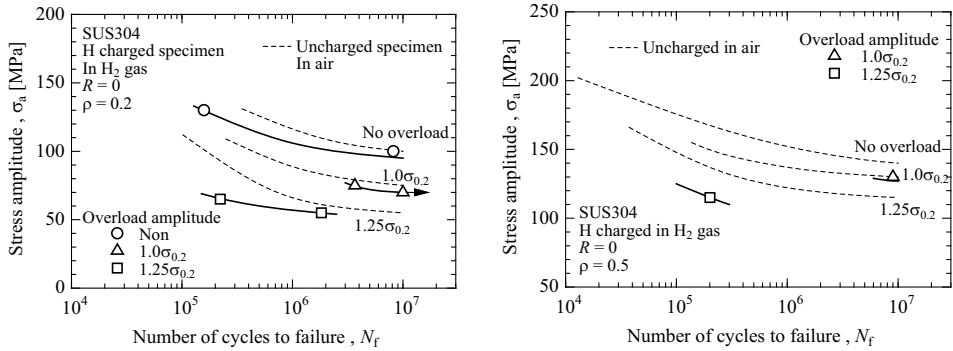


Fig. 7 Observation of non-propagating cracks generated by overload



(a) $\rho = 0.2\text{mm}$

(b) $\rho = 0.5\text{mm}$

Fig. 8 Effect of hydrogen on high-cycle fatigue strength following multiple overloading.

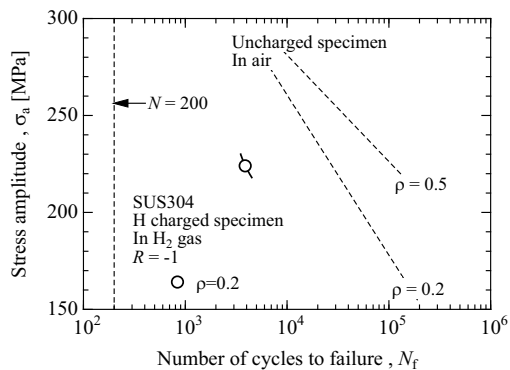


Fig. 9 Effect of hydrogen on low-cycle fatigue strength

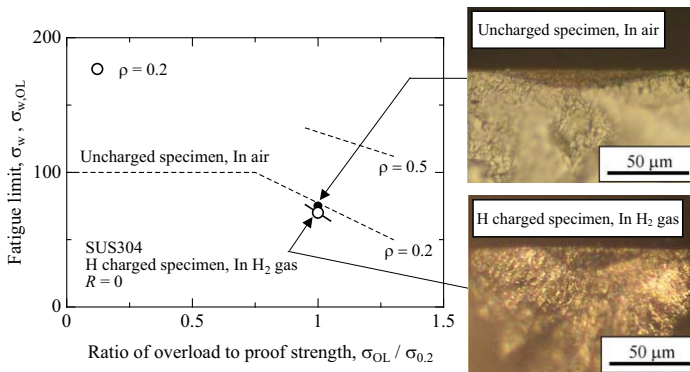


Fig. 10 Effect of hydrogen on the size of non-propagating cracks.

air. Deeper microcracks is considered to be one of the causes of decrease of fatigue strength due to hydrogen. The depth of crack was within the layer which had high hydrogen concentration.

Table 4 shows the evaluation of the stress intensity factor of non-propagating cracks. The value is regarded as the crack propagation threshold. The K value was calculated by combining the solutions for a crack emanating from an elliptical hole under tension [1] and that for an embedded semi-elliptical crack in finite plate in tension [2]. Since the distribution of residual stress at the notch root produced by the overload was unknown, the effect of residual stress was not taken into account. In air, the ΔK_{th} was almost constant. The hydrogen charged specimen had a little smaller ΔK_{th} .

Figure 11 shows the hardness distribution below the notch root after overloading. The solid line and hatching zone show the mean value and standard deviation of the hardness of base material. The hardness of subsurface after the application of overload was increased. There is a report that ΔK_{th} decreased by the absorbed hydrogen in the material depending on the hardness of material and as a result fatigue strength can be decreased [3]. Although that report didn't treat the same kind of material used in this experiment, the increase of hardness can be one of the causes of decrease of fatigue strength due to hydrogen. On the contrary, there is a report that environmental hydrogen can increase ΔK_{th} [4]. The fatigue strength of hydrogen charged specimen tested in hydrogen gas might be resulted by the opposite influence between both increase and decrease effects on ΔK_{th} . Encouragement of further study to clarify the effect of hydrogen on fatigue strength is required.

Table 4 Estimation of stress intensity factor range of non-propagating cracks.

Conditions	Uncharged specimen, In air		H charged specimen, In H ₂ gas
Overload amplitude	1.0 $\sigma_{0.2}$	1.25 $\sigma_{0.2}$	1.0 $\sigma_{0.2}$
Crack depth [μm]	17.7	53.1	20.9
σ_w after overloading [MPa]	75	55	70
ΔK_{th} [MPa m ^{1/2}]	4.7	4.8	4.0

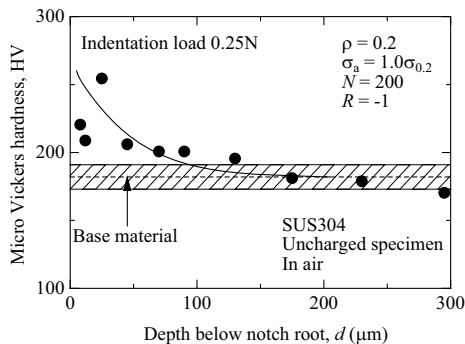


Fig. 11 Hardness distribution produced by overloading
(Uncharged specimen tested in air, $\rho = 0.2$, $\sigma_a = 1.0\sigma_{0.2}$, $N = 200$)

Summary

The effect of overload, which might be applied in an earthquake, on high-cycle fatigue strength of notched specimens was investigated for an austenitic stainless steel SUS304. The effect of hydrogen superposed on the effect of overload was also studied. The notch root radii of 0.2, 0.5 and 0.75mm were used. The hydrogen charged specimen whose hydrogen concentration was approximately 80ppm was tested in hydrogen gas environment. The results obtained are summarized as follows:

- (1) The fatigue strength of notched specimen decreased depending on the overload amplitude. In the case of $\rho = 0.2$ mm specimen, the overload of $0.75\sigma_{0.2}$ had no effect on the high cycle fatigue strength after the overload.
- (2) The cause of decrease of fatigue strength was the microcracks formed at the notch root by the overload.
- (3) The fatigue strength of hydrogen charged specimen tested in hydrogen gas was lower than that of uncharged specimen tested in air.
- (4) The depth of microcracks was deeper in the hydrogen charged specimen than in the uncharged specimen.

Acknowledgement

This work was supported by New Energy and Industrial Technology Development Organization (NEDO) in Japan.

References

- [1] Y. Murakami, P. Lukas, M. Klesnil, in: *Stress Intensity Factors Handbook Vol. I*, edited by Y. Murakami / Pergamon Press, Oxford (1987).
- [2] J. C. Newman, Jr. I. S. Raju, in: *Stress Intensity Factors Handbook Vol. II*, edited by Y. Murakami / Pergamon Press, Oxford (1987).
- [3] K. Shishime, M. Kubota, Y. Kondo: *Mater. Sci. Forum* Vol. 567-568 (2008) p. 409.
- [4] Y. Ueda, M. Kubota, Y. Kondo: *Proc. of 39th Meeting of JSME Kyushu Student Council*, Fukuoka, Japan, JSME No. 088-2 (2008), p. 177.