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The effect of vacuum-like environment inside sub-surface fatigue crack on the formation of ODA fracture surface in high strength steel

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Abstract

Very high cycle fatigue (VHCF) of high strength steel has become an important issue for mechanical engineers in recent years. In VHCF regime over $10^7$ cycles, fatigue crack initiates not from surface but from sub-surface of materials. The sub-surface fractures even occur in a lower stress than surface-originating fractures; therefore, to clarify its mechanism is strongly needed for the safety use of high strength steel. In sub-surface fractures, a typical fracture surface with a fine concavo-convex pattern called "ODA" was discovered. To reveal the formation mechanism of ODA is regarded as a key point for sub-surface crack growth because it is never observed in surface-originating fractures. This study focuses on a special environment inside sub-surface crack. The sub-surface crack is not exposed to atmosphere and the adsorption of gaseous molecules on fresh-surface at crack tip seems to be negligible. With this in mind, fatigue crack growth tests in high vacuum were conducted to simulate sub-surface crack propagation, and fracture features were thoroughly investigated by SEM analyses. As a result, it was clarified that high vacuum is closely similar to the environment inside sub-surface crack, and is a necessary condition to form ODA in VHCF.

Keywords: Very High Cycle Fatigue, Optically Dark Area, Sub-surface Fracture, Fractography, Crack Propagation, Vacuum Environment

1. Introduction

In recent years very high cycle fatigue of high strength steel has become an important issue of growing concern for mechanical engineers. This fatigue phenomenon is mainly characterized by sub-surface fracture in the long life regime over $10^7$-$10^8$ cycles [1-9]. The sub-surface fractures even occur in a lower stress than surface-originating fractures; therefore, to clarify its mechanism is strongly needed for the safety use of high strength steel. In sub-surface fractures, a unique fracture surface with a fine concavo-convex pattern called "ODA" was discovered by Murakami [1-2]. Fig. 1(a) and (b) show a typical fracture surface of sub-surface fatigue fracture and its magnified view, respectively. The fine granular concavo-convex feature, so-called ODA, is clearly seen around the origin. To reveal the formation mechanism of ODA is regarded as a key point for sub-surface crack growth [8] because it is never observed in surface-originating fractures. Several hypotheses have been presented for the formation of ODA.

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Murakami reported that synergistic effect between cyclic stress and hydrogen trapped around the inclusion causes ODA [1-2]. Shiozawa presented that long term cyclic load leads to the dispersive decohesion of spheroidal carbides from matrix around the inclusion and forms ODA [9]. Oguma proposed the idea that crystalline structure around the inclusion is refined during cyclic loading and leads to ODA fracture surface [8,10]. At this stage, however, the close mechanism for the formation of ODA has not been unified yet.

![Fig. 1. A typical ODA region observed on the fracture surface of sub-surface crack obtained by a tension compression fatigue test of SNCM439 (R=-1, \( \sigma \max =750\text{MPa}, N_f =3.6\times10^7 \)): (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the area indicated by the arrow A in Fig. (a).](image-url)

In this study, the authors focus on the fact that sub-surface cracks are not surrounded by atmosphere. The environment around sub-surface crack tip is likely different from air because oxidation and adsorption of gaseous molecules are negligible [11]. In addition, it is known that fatigue lives of metallic materials in vacuum environment generally longer than those in air atmosphere [12,13], which corresponds to the long lives of sub-surface fractures in VHCF regime. If the environment around sub-surface crack is similar to vacuum, there is a possibility that ODA can also be generated under vacuum environment. In our previous work, granular fracture surfaces closely resembling ODA were observed in crack growth tests of Ti-6Al-4V in vacuum [14,15]. Especially, it was clarified that the granular features in Ti-6Al-4V were generated by long term repeating contacts of fracture surfaces in vacuum environment after fatigue crack was already formed. This result suggests that fine concavo-convex pattern such as ODA may be generated under repeating contacts of upper and lower fracture surfaces in vacuum environment regardless of materials. To clarify the credibility of this hypothesis, this study conducted fatigue crack growth tests of high strength steel in high vacuum environment under various conditions, and investigated the fracture surfaces by using SEM analyses focusing on the fine concavo-convex pattern.

2. Characteristics of ODA

Before explaining the details of the experiment, typical characteristics of ODA are summarized in this chapter. As mentioned before, ODA is not observed in surface-originating fractures but in sub-surface fractures. In addition to this point, the following four characteristics are regarded as the important morphology of ODA.

1. ODA consists of fine concavo-convex patterns smaller than microstructure [8,9]. As shown in Fig. 1(b), the size of the concavo-convex feature is similar or smaller than martensite block, which is a smallest structural unit affecting fatigue crack growth in high strength steel.

2. The concave spots on a fracture surface precisely fit to the convex spots on the opposite fracture surface, and vice versa. This feature is confirmed by mating analyses of the fracture surfaces and considered important characteristics of ODA [16].

3. The concavo-convex morph becomes much clearer under the condition of repeating compressive load. Fatigue tests under different mean stresses clarified that the feature of ODA became much clearer under negative stress.
ratio but unclear under positive stress ratio. Shiina pointed out that repeating compressive load can affect the formation of ODA [16].

(4) ODA becomes clearer with increasing fatigue lives. ODA can be observed in sub-surface fractures occurred around $10^6$ cycles. However, the fine concavo-convex feature tends to be much clearer in the fracture surfaces whose fatigue lives are over $10^7$ or $10^8$ cycles.

To compare the feature of ODA with that of fracture surfaces obtained by crack growth tests in vacuum, the above four points will be focused on in chapter 4 and 5.

3. Experimental procedures

3.1. Material and specimen

The material used was low-temperature-tempered Ni-Cr-Mo steel (SNCM439 corresponding to AISI 4340 steel), having the chemical compositions (mass%) of: 0.38 C, 0.27 Si, 0.75 Mn, 0.017 P, 0.017 S, 0.10 Cu, 1.80 Ni, 0.80 Cr and 0.16 Mo. The specimen configuration is shown in Fig. 2, which is a MT type specimen conforming to ASTM E647-00. The blank specimen slightly larger than the dimension of Fig. 2 was cut from the supplied material and the following heat treatment was given: Oil Quenching (1103K $\times$3.6ks $\rightarrow$ 343K) and Tempering (433K $\times$16.2ks $\rightarrow$ Air cooling). After finishing the blank into the shape of Fig. 2, the starter notch for fatigue pre-crack was introduced by electro discharge machining (EDM). The direction of the fatigue crack growth is set perpendicular to the rolling direction of the material. The microstructure after heat treatment was given in Fig. 3 showing uniformly distributed tempered martensite. Vickers hardness of this material was 613 HV.

![Fig. 2 Specimen configuration](image1)

![Fig. 3 Microstructure](image2)

3.2. Experimental apparatus

Fig. 4 shows an ultra high vacuum fatigue testing machine developed by the authors with the cooperation of VIC International and MOOG Japan. The vacuum system consists of a vacuum chamber, a dry scroll pump, and a turbo molecular pump. All of the connecting parts are sealed with metallic seals, and the ultimate pressure is $4.6 \times 10^{-7}$ Pa. The loading system consists of hydraulic servo actuator, gripping parts, a load cell, and a digital controller. Sinoidal wave with arbitrary mean load can be applied to the specimen precisely. In addition, the testing machine has the adjusting system of alignment to minimize bending stress in the specimen. To measure the crack length, a digital microscope (Scalar, HDM2100V, $\times$200) is used.

3.3. Experimental conditions and procedures

As mentioned in chapter 1, our previous crack growth tests using Ti-6Al-4V clarified that the granular features resembling ODA were generated in vacuum by the long term repeating contacts of fracture surfaces, which had been already formed in the former crack growth test. In addition, as summarized in 2, stress ratio and repeating number of cycles are important factors for the morphology of ODA. Based on these results, two kinds of experiments were proposed. One was a crack growth test and the other was a repeating loading test of fracture surfaces. Firstly, as for
the crack growth test, ΔK decreasing test was conducted in vacuum until the crack arrest was confirmed. Secondly, the repeating cyclic loading over $1 \times 10^7$ was applied on the fracture surfaces generated during the former crack growth test. In the repeating loading test, very low ΔK value smaller than ΔK_{th} was used so that the fatigue crack generated in the former crack growth test could not propagate.

The crack growth tests were conducted in vacuum environment according to ASTM E647-00 with a frequency of 60Hz. Fatigue pre-crack was introduced more than 1mm per one side to avoid the effect of heat affected zone (about 10μm) formed by EDM process for making starter notch. Two kinds of crack growth tests under R = -1 and R = 0.3 were carried out. The number of specimens used under R = -1 and R = 0.3 were two and one, respectively. After the crack arrest was confirmed in the crack growth test, the repeating loading test was started. The stress ratio in the repeating loading test was same as that in the crack growth test. As for R = -1, the pure crack growth test which was not followed by the repeating loading test was also carried out to obtain the basic fracture surface for reference. After these tests, cyclic overloading was applied in vacuum to break the specimen apart. In this process, the stress ratio R=0.3 was used to avoid the damage of fracture surfaces introduced in the former tests. Throughout the experiments, vacuum pressure was in the range from $7.9 \times 10^{-6}$ to $1.9 \times 10^{-5}$ Pa.

After the crack growth tests and the repeating loading tests, fracture surfaces were investigated by SEM analyses focusing on fine concavo-convex pattern.

4. Results

4.1. Crack growth test

The da/dN-ΔK curve under R = -1 is shown in Fig. 5. The crack growth rates corresponding to ΔK = 16–8MPa√m were measured. The ΔK_{th} of this material under R = -1 was about 8 MPa√m. In this research, ΔK was calculated by the following equations:

$$\Delta K = K_{\text{max}} - K_{\text{min}} \quad (K_{\text{min}} \geq 0), \quad \text{and} \quad \Delta K = K_{\text{max}} \quad (K_{\text{min}} < 0)$$

A typical fracture surface in relatively high ΔK is shown in Fig. 6(a) and (b). These figures were taken at the point where ΔK = 15MPa√m. As shown in the arrow in Fig. 6(b), the edges of martensite blocks seem to have round features. However, the fine concavo-convex pattern such as ODA cannot be observed. Fracture surfaces near ΔK_{th} are given in Fig. 7(a) and (b). The traces of martensite blocks are observed and the fine concavo-convex feature is not recognized. Consequently, it was clarified that the fracture surface similar to ODA was not observed in ΔK decreasing test in vacuum environment.
Fig. 5 Relation between fatigue crack growth rate and $\Delta K$ under $R=-1$ in vacuum.

Fig. 6. Fracture surface at $\Delta K = 15$ MPa/m in crack growth test under $R=-1$ in vacuum: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).

Fig. 7. Fracture surface near $\Delta K_n$ in crack growth test under $R=-1$ in vacuum: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).
4.2. Repeating loading test

4.2.1. The effect of stress ratio on the formation of fine concavo-convex pattern

After the crack grow tests, repeating loading tests were conducted in vacuum under $R = 0.3$ and $R = -1$. The number of cycles applied in the repeating loading test was $1 \times 10^7$. Fig. 8(a),(b) show the typical fracture surfaces under $R = -1$. These photographs were taken at the point where $\Delta K$ was relatively high value ($= 13\text{MPa}\sqrt{\text{m}}$). The granular fine concavo-convex feature was observed. Especially in Fig. 8(b), the size of concavo-convex seems to be similar or smaller than the width of martensite block. This feature corresponds to the first characteristics of ODA ((1) in Chapter 2). The granular patterns were also observed in low $\Delta K$ regions. Namely, the fine concavo-convex feature scattered in places on the whole fracture surface regardless of $\Delta K$ and crack growth rate.

Fig. 8. Fracture surface at $\Delta K = 13\text{MPa}\sqrt{\text{m}}$ in crack growth test followed by repeating loading test under $R = -1$ in vacuum: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).

Fig. 9(a),(b) show the typical fracture surfaces under $R = 0.3$. These photographs were taken at the point where $\Delta K$ was relatively high value ($\Delta K = 14\text{MPa}\sqrt{\text{m}}$). In these figures, the fine concavo-convex patterns similar to Fig. 8 are not recognized. Such kind of granular area could not be observed on the whole fracture surface. Therefore, negative stress ratio is considered necessary to form the fine concavo-convex pattern. This result corresponds to the third characteristics of ODA ((3) in Chapter 2).

Fig. 9. Fracture surface at $\Delta K = 14\text{MPa}\sqrt{\text{m}}$ in crack growth test followed by repeating loading test under $R = 0.3$ in vacuum: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).
5. Discussion

The experimental results showed that the fine concavo-convex feature was observed after repeating loading test under \( R = -1 \) in vacuum environment. The feature matches the first and third characteristics of ODA ((1) and (3) in Chapter 2). However, it must be investigated carefully whether the fine concavo-convex feature in Fig. 8 is completely same as ODA or not. To clarify this problem, the detailed discussion will be made focusing on the second and fourth characteristics of ODA ((2) and (4) in chapter 2).

As mentioned in (4) in 2, ODA becomes clearer with increasing fatigue lives. Based on this point, the additional repeating loading test with a longer number of cycles, \( 5 \times 10^7 \), was conducted under \( R = -1 \) in vacuum environment. In this experiment, the repeating loading test was started just after fatigue pre-crack was introduced. Namely, the crack growth test was not carried out in this additional experiment. No crack growth was confirmed during the repeating loading test. Fig. 10(a),(b) show the typical fracture surfaces. These photographs were taken at the point where \( \Delta K \) was relatively high value (\( \Delta K = 15 \text{MPa}\sqrt{\text{m}} \)). It is clear that Fig. 10(a) is almost covered by the fine granular feature. This granular feature was observed on almost all area of the fracture surface regardless of \( \Delta K \) and crack growth rate. In Fig. 10(b), the feature of fine concavo-convex became much clearer compared with Fig. 8(b). By comparing this figure with ODA in sub-surface fracture (Fig. 1(b)), the both photographs have extremely similar characteristics regarding the size and feature of fine granular morphology. These results showed that long term cyclic loading was very important to form the fine concavo-convex pattern. In this experiment, the crack growth test was not conducted. Therefore, it is apparent that the fine concavo-convex feature was not formed in crack growth process but in the repeating loading process. In other words, the granular feature can be formed by applying the long term repeating load on fatigue crack in vacuum environment.

![Direction of crack propagation](image1)

![Fracture surface at \( \Delta K = 15 \text{MPa}\sqrt{\text{m}} \) in the process of introducing fatigue pre-crack followed by repeating loading test for \( 5 \times 10^7 \) cycles under \( R = -1 \) in vacuum: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).](image2)

As mentioned in (2) in Chapter 2, the concave spots of ODA on a fracture surface precisely fit to the convex spots on the opposite fracture surface. To confirm whether the fine concavo-convex feature in Fig. 10 has this characteristic or not, the mating photographs were taken. Fig. 11 shows the upper and lower fracture surfaces of the fine concavo-convex region obtained after the repeating loading test of \( 5 \times 10^7 \) in vacuum under \( R = -1 \). As shown in the white circles, the convex spots precisely fit to the concave spots, and vice versa. Therefore, the second characteristic of ODA is also satisfied in Fig. 10.

To confirm that the fine concavo-convex feature such as Fig. 10 is not observed in atmosphere, another repeating loading test was conducted for \( 5 \times 10^7 \) under \( R = -1 \) in air environment. In this experiment, the repeating loading test was started just after fatigue pre-crack was introduced in air. Fig. 12(a),(b) show the typical fracture surfaces. These photographs were taken at the point where \( \Delta K \) was relatively high value (\( \Delta K = 14 \text{MPa}\sqrt{\text{m}} \)). In these figures, the fine concavo-convex morphology cannot be observed. Although all area of the fracture surface was carefully observed,
no feature similar to Fig. 10 was detected. Namely, the fine concavo-convex feature cannot be formed in atmosphere. This result also corresponds to the fact that ODA is not observed in surface-originating fractures but in sub-surface fractures. By summarizing the various discussions focusing on the characteristics of ODA, it can be concluded that the fine concavo-convex region shown in Fig. 10 is same as ODA in sub-surface fatigue fractures.

Fig. 11. Mating photographs of the fracture surface at $\Delta K = 15$MPa$\sqrt{m}$ in the process of introducing fatigue pre-crack followed by repeating loading test for $5 \times 10^7$ cycles under $R=-1$ in vacuum.

Fig. 12. Fracture surface at $\Delta K = 14$MPa$\sqrt{m}$ in the process of introducing fatigue pre-crack followed by repeating loading test for $5 \times 10^7$ cycles under $R=-1$ in air: (a) left-hand picture shows the low magnified view; (b) right-hand picture shows the high magnified view of the rectangular area in Fig. (a).

In this research, influential factors affecting the formation of ODA were clarified. The fine concavo-convex pattern: ODA is not formed during crack growth process but formed by the long term repeating loading after fatigue crack was introduced. In addition, considering both the effect of stress ratio and the effect of environment, it can be said that the long term repeating contact of fracture surfaces in vacuum environment is a necessary condition to form ODA. Especially, the similarity between Fig. 1(b) and Fig. 10 suggests that environment around sub-surface crack is quite similar to high vacuum environment. The knowledge obtained in this research clearly matches our previous work using Ti-6Al-4V [13-15]; therefore, the morphology of ODA can be formed regardless of materials when next three conditions are satisfied: vacuum environment, repeating contact of fracture surfaces, and long term loading over about $10^7$ cycles. These three conditions are automatically satisfied in sub-surface fractures under rotating bending or tension-compression fatigue tests in very high cycle regime.
6. Conclusion

To clarify the influential factors on the formation of ODA observed in sub-surface fractures in very high cycle fatigue, crack growth tests and succeeding repeating loading tests of high strength steel were conducted under various conditions in vacuum and air environments. After the detailed observation of fracture surfaces by SEM, it was clarified that ODA is not formed during crack growth process but formed by the long term repeating compressive loading after fatigue crack was generated. By comparing the similar granular fracture surface observed in Ti-6Al-4V, it was concluded that the morphology of ODA can be formed regardless of materials when next three conditions are satisfied: vacuum environment, repeating contact of fracture surfaces, and long term loading over about 10^7 cycles. These three conditions are automatically satisfied in sub-surface fractures under rotating bending or tension-compression fatigue tests in very high cycle regime. This can be the main factor for the formation of ODA in VHCF.

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References

[1] Murakami, Y., Nomoto, T., Ueda, T., Fatigue Fract. Engng. Mater. Struct., Vol. 22, pp.581-590, 1999.
[2] Murakami, Y., Nomoto, T., Ueda, T., Murakami Y., Fatigue Fract. Engng. Mater. Struct., Vol. 23, pp.893-902, 2000.
[3] Shiozawa, K., Lu, L., Ishihara, S., Fatigue Fract. Engng. Mater. Struct., Vol. 24, pp.781-790, 2001.
[4] Bathias, C., Fatigue Fract. Engng. Mater. Struct., Vol. 22, pp.559-565, 1999.
[5] Sakai, T., Sato, Y., Nagano, Y., Takeda, M., Oguma, N., Int. J. Fatigue, Vol. 28, pp.1547-1554, 2006.
[6] Furuya, Y., Matsuoka, S., Abe, T., Yamaguchi, K., Scr. Mater., Vol. 46, pp.157-162, 2002.
[7] Wang, Q.Y., Berard, Y., Rathery, S., Bathias, C., Fatigue Fract. Engng. Mater. Struct., Vol. 22, pp.673-677, 1999.
[8] Sakai, T., J. Solid Mechanics and Materials Engineering, Vol.3, No.3, pp.425-439, 2009.
[9] Shiozawa, K., Morii, Y., Nishino, S., Lu, L., Int. J. Fatigue, Vol.28, No. 11, pp.1520-1532, 2006.
[10] Oguma, N., Harada, H., Sakai, T., J. Mater. Sci. Japan, Vol.52, pp.1292-1297, 2003, in Japanese.
[11] Nishijima, S., Kanazawa K., Fatigue Fract Engng Mater Struct, Vol. 22, pp.601-607, 1999.
[12] Grinberg, N.M., Int. J. Fatigue, April, pp.83-95, 1982.
[13] Nakamura, T., Oguma, H. and Shiina, T., Ti-2003, Science and Technology, pp.1775-1782, 2003.
[14] Nakamura, T., Oguma, H., Yokoyama, S. and Noguchi, T., Proc. of the Third International Conference on Very High Cycle Fatigue, VHCF-3, pp.201-208, 2004.
[15] Nakamura T., Oguma H., Proceedings of the Materials Science & Technology 2008 Conference, MS&T08, CD-ROM, (2008),
[16] Shiina T., Nakamura, T., Noguchi, T., Trans. Japan. Soc. Mech. Eng., Vol. 70A, No.696, pp.24-31, 2004, (in Japanese).