JOURNAL OF MATERIALS SCIENCE **34** (1999) 2411 – 2419

Relationship between fatigue striation height and stress ratio

Y. UCHIDA, M. SHIMOJO*, Y. HIGO
Tokyo Institute of Technology, Precision and Intelligence Laboratory, 4259, Nagatsuta, Midori-ku, Yokohama, 226-8503, Japan
E-mail: mshimojo@pi.titech.ac.jp

In order to obtain stress ratio as well as stress intensity factor range which was applied on the material before failure from the fracture surface observation, the effect of stress ratio on fatigue striation height has been investigated on two different alloys (an Fe-3% Si single crystalline alloy and a 7075 polycrystalline aluminium alloy). Striation height has been measured both microscopically using a scanning tunnelling microscope and macroscopically using a scanning electron microscope. Striation height increased with decreasing stress ratio on both the materials at a given crack growth rate. This tendency was found by all the measuring methods used. This suggests that not only stress amplitude but also stress ratio which were applied on the material before the failure could be obtained by post-failure analysis.

1. Introduction

It is known that there are characteristic ripple markings on fatigue fracture surfaces which are now called fatigue striations. Because a striation is produced by each loading cycle [1, 2] at a stress intensity factor range in excess of a certain value that is in the intermediate and high growth rate regions, striations are regarded as an indication in post-crash or post-failure analysis that the failure of the structure is caused by cyclic loading. It has been shown that striation spacing coincides with crack growth distance in a corresponding fatigue cycle in the intermediate and high growth rate regions [3]. Thus, the stress amplitude which was applied on the material can be estimated by measuring the striation spacing and by comparing laboratory data.

However, the other parameters concerning applied load such as stress ratio cannot be estimated by striation observation to date. The objective of this study is to investigate the relationship between stress ratio and striation height. Few studies have been carried out on striation height measurement [4] and such measurements have been conducted at relatively large crack growth rates of about a few micro-meters per cycle or more, because of the difficulty in measuring striation height. In order to obtain the relationship which can be applied generally, two different materials which are both single crystalline and polycrystalline are used. In addition, because a selected area measurement does not necessarily indicate the general trend, three different methods which are both microscopic and relatively macroscopic are used to measure striation height.

2. Experimental

The materials used in this study were an Fe-3% Si single crystalline alloy and a 7075-T6 polycrystalline aluminium alloy. The 7075 alloy was received as a cold-rolled plate (no heat treatment was done). The grain size of the plate was 150 μm in the longitudinal direction (L), 70 μm in the transverse direction (T) and 20 μm in the short transverse direction (S). CT specimens having two orientations (L-T and S-L) were machined from the plate. The dimension of the CT specimens was $50 \times 50 \times 20$ mm^3.

A strain-anneal method was used in order to obtain large grains of the Fe-3% Si alloy, and the grain size obtained after this treatment was approximately 3–5 cm in diameter. The material was cut into plates. Then, the work hardened surface layer which might have been introduced by machining was eliminated using chemical etching, and the plates were annealed to remove any residual stresses. The crystallographic orientations of large crystals were determined by an X-ray Laue back-scattering technique, and CT specimens of approximately $25 \times 25 \times 5$ mm^3 in size were carefully prepared from the plates. Fig. 1 shows the crystallographic orientations for the employed Fe-Si specimens plotted on a (001) standard stereographic projection. The loading direction and the notch direction of each specimen are indicated by (LD) and (ND), respectively.

Fatigue tests were performed at a sinusoidal loading frequency of 10 Hz using a closed-loop servo hydraulic fatigue testing machine under constant load conditions at stress ratios (R) of 0.1 and 0.6 on the Fe-Si alloy and of 0.05, 0.15, 0.3 and 0.6 on the 7075 alloy. The tests were performed at room temperature in laboratory air. Crack length was monitored using a direct-current potential drop technique. Crack closure measurement was also carried out using a displacement clip gauge.

* Author to whom all correspondence should be addressed.

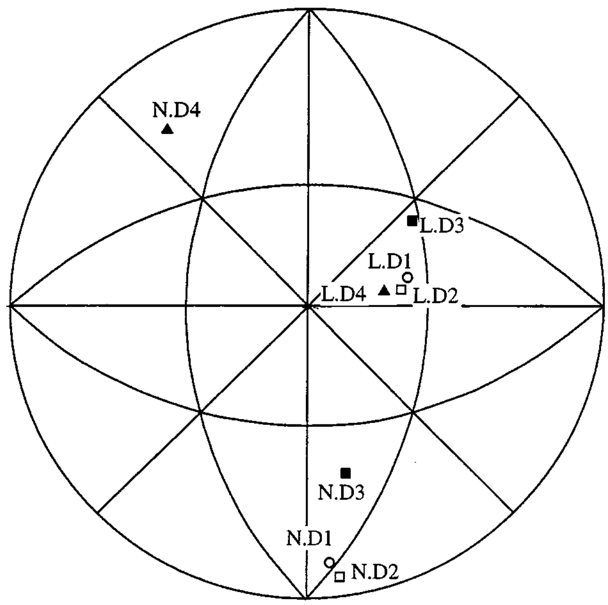


Figure 1 The orientations of Fe-3% Si single crystal specimens plotted on a (001) standard stereographic projection.

In order to measure striation heights, three different methods were used:

(i) A scanning tunnelling microscope (STM) which is installed in a scanning electron microscope (Hitachi V-3000 STM-SEM) to observe the exact position of the probe on fracture surface. The resolution of the STM in the height direction is about an order of 0.01 nm. The scanned area was 2 μm × 2 μm. The radius of the probe tip is much smaller than the striation spacings measured and the tip length was greater than the depth of striation valley. Thus no effect of convolution of the STM probe shape was considered to be observed.

(ii) A scanning image analyser (SIA), which was developed by H. Sato and M. O-hori [5, 6] and the system installed in SEM has been manufactured by JEOL Ltd., Japan. Using this system, surface profile is obtained by integrating backscattered electron signal, the intensity of which is in proportion to the inclination of the surface profile. The resolution in height direction is about 1 nm. Height profiles on 7.8 μm line were measured. A gold layer of about 40 nm was deposited on the fracture surface prior to the measurements. These two methods (i) and (ii) gave direct measurements of striation height but the scanned areas were relatively small.

(iii) The apparent ratio of striated surface and the apparent ratio of plateau area to fracture surface were measured under a fixed observation condition such as magnification, tilt angle, spot size using a scanning electron microscope (SEM). The measured area was 1 mm in the thickness direction and 25 μm in the crack growth direction. The apparent ratio of striated surface represents the height of striations because higher striation can be observed more clearly at a relatively low magnification (see Appendix). This method gives an indirect value of striation height, however, the average height of a relatively wide area can be measured.

Striation heights of specimens tested at various stress ratios at given stress intensity factor ranges were compared on each material. All the striations, the heights of which were measured, were considered to be formed by each corresponding cycle, as the striation spacing almost coincided with the macroscopic crack growth rate.

3. Results and discussion

Figs 2 and 3 show fatigue crack growth rates as a function of effective stress intensity factor range ($\Delta K_{\rm eff}$) of Fe-Si and 7075 alloys, respectively. There are slight differences in fatigue crack growth rate among the stress ratios measured on both Fe-Si and 7075 alloys.

Fig. 4 shows an example of SEM fractographs with the tip of a STM probe. (The very sharp part at the end of the tip was not seen in this figure.) Striations

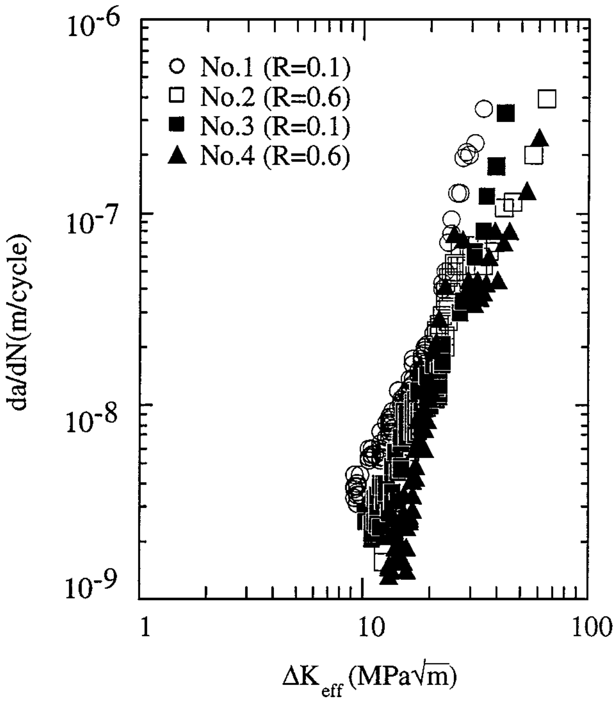


Figure 2 Crack propagation rate as a function of effective stress intensity factor range of Fe-3% Si single crystals.

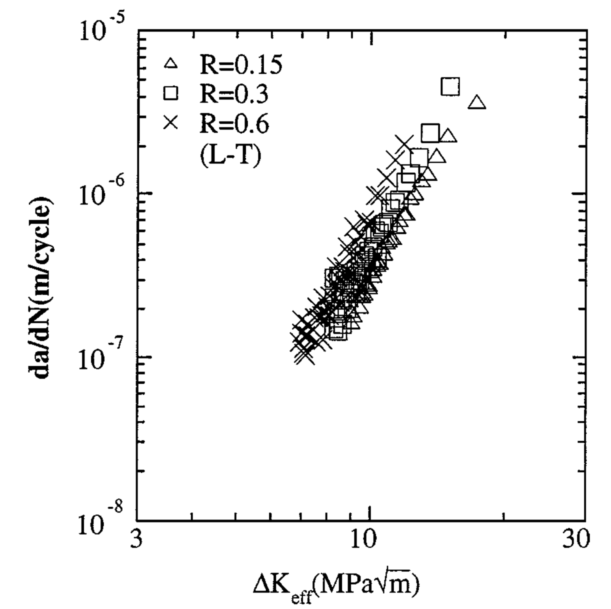


Figure 3 Crack propagation rate as a function of effective stress intensity factor range of 7075 aluminium alloy (L-T).

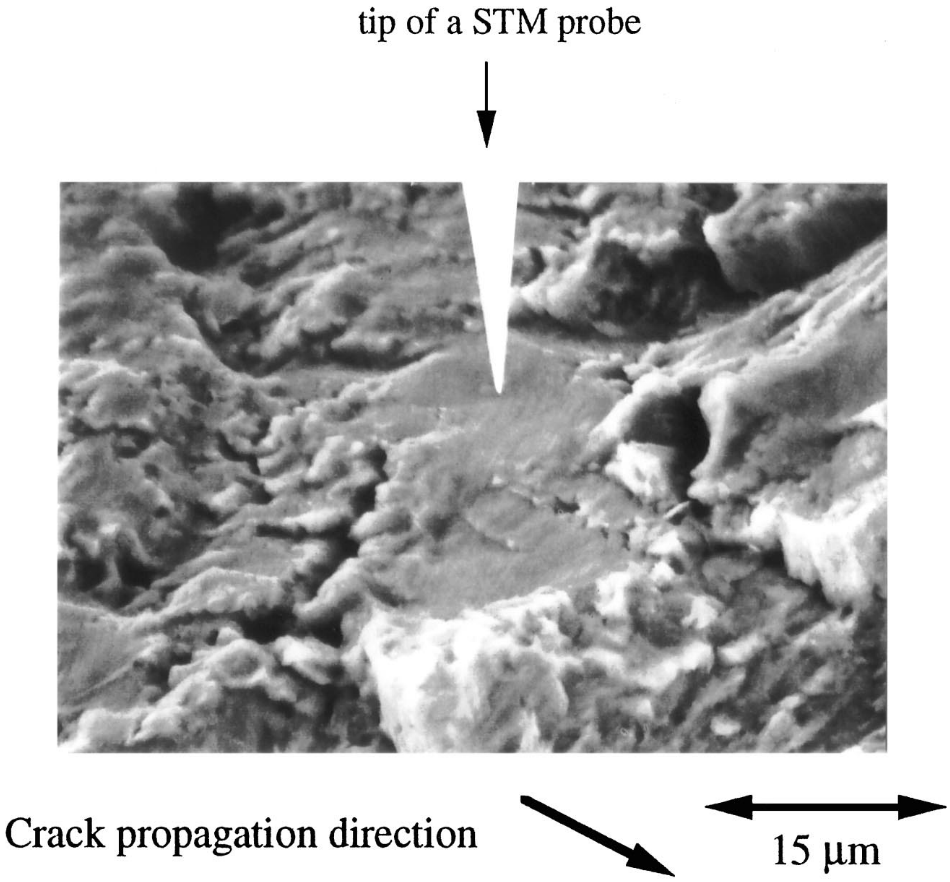


Figure 4 SEM photograph of fracture surface on a Fe-3% Si single crystal at a stress ratio of 0.6. The tip of the STM probe is also shown in the photograph.

are normally observed on plateau regions, which are relatively flat. A plateau is observed at the centre of the fractograph and ahead of the STM probe tip, and the STM probe was approached the plateau region by observing both the fracture surface and the probe tip in order to measure striation height on the plateau.

Fig. 5a and b shows STM three dimensional analyses of fatigue striations on fracture surfaces tested on Fe-Si alloy specimens at stress ratios of 0.1 and 0.6 and at a crack growth rate of 2×10^{-7} m/cycle. The striation spacings of fracture surfaces tested at stress ratios (R) of 0.1 and 0.6 were 0.25 and 0.15 μm, respectively, and were almost comparable to the crack growth rate. The average striation heights were 40 and 25 nm at stress ratios of 0.1 and 0.6, respectively. The average striation height of at a stress ratio of 0.1 seems to be larger than that of at a stress ratio of 0.6, however, the ratios of striation height to striation spacing are almost the same at both stress ratios. Thus, the effect of stress ratio on striation height cannot be revealed using STM. A reason for this may be that the area scanned by STM is so small that it is difficult to measure striation height at the same crack growth rate for both the stress ratios.

Although STMs provide detailed and accurate measurements of striation heights, which is an order of 1 nm or small, it is laborious to measure on a wide surface area using a STM and is impossible to measure a rough surface due to the limitation of probe movements. In addition, it is difficult to measure on materials which have a non-conducting oxide layer on the surface. STMs are therefore not always be able to be applied for this kind of measurements.

Figs 6 and 7 show striation height profiles, fractographs and measured heights using SIA on fatigue fracture surfaces of the 7075 alloy at stress ratios of 0.3 and 0.6 at a ΔK value of 12 MPa $m^{1/2}$. Figs 6a and 7a indicate the height profiles on the lines in the fractographs (Figs 6b and 7b). The striation heights indicated as Z at a stress ratio of 0.3 (Fig. 6c) were larger than those at a stress ratio of 0.6 (Fig. 7c), though crack growth rates which were estimated from striation spacing are almost the same at both stress ratios.

Fig. 8 shows apparent ratios of striated surface area to the observed surface area at a fixed observation condition using a SEM as a function of crack growth rate at two stress ratios of the Fe-Si alloy. Regardless of crystal orientation, the apparent ratio of striated surface at a stress ratio of 0.1 is higher than that at a stress ratio of 0.6.

Apparent ratios of plateau area to the observed surface area of the Fe-Si alloy are shown in Fig. 9. Striations are normally observed on plateau areas [7, 8]. The apparent ratio of plateau area does not change with stress ratio. Assuming that whole plateau area is covered with striations, the actual ratio of striated surface should not change with stress ratio. The main factors affecting the contrast of SEM pictures are (i) the atomic number of the material, (ii) accelerating voltage, (iii) tilt angle, and (iv) slope, curvature and edges of the surface [9]. In this work, observation conditions

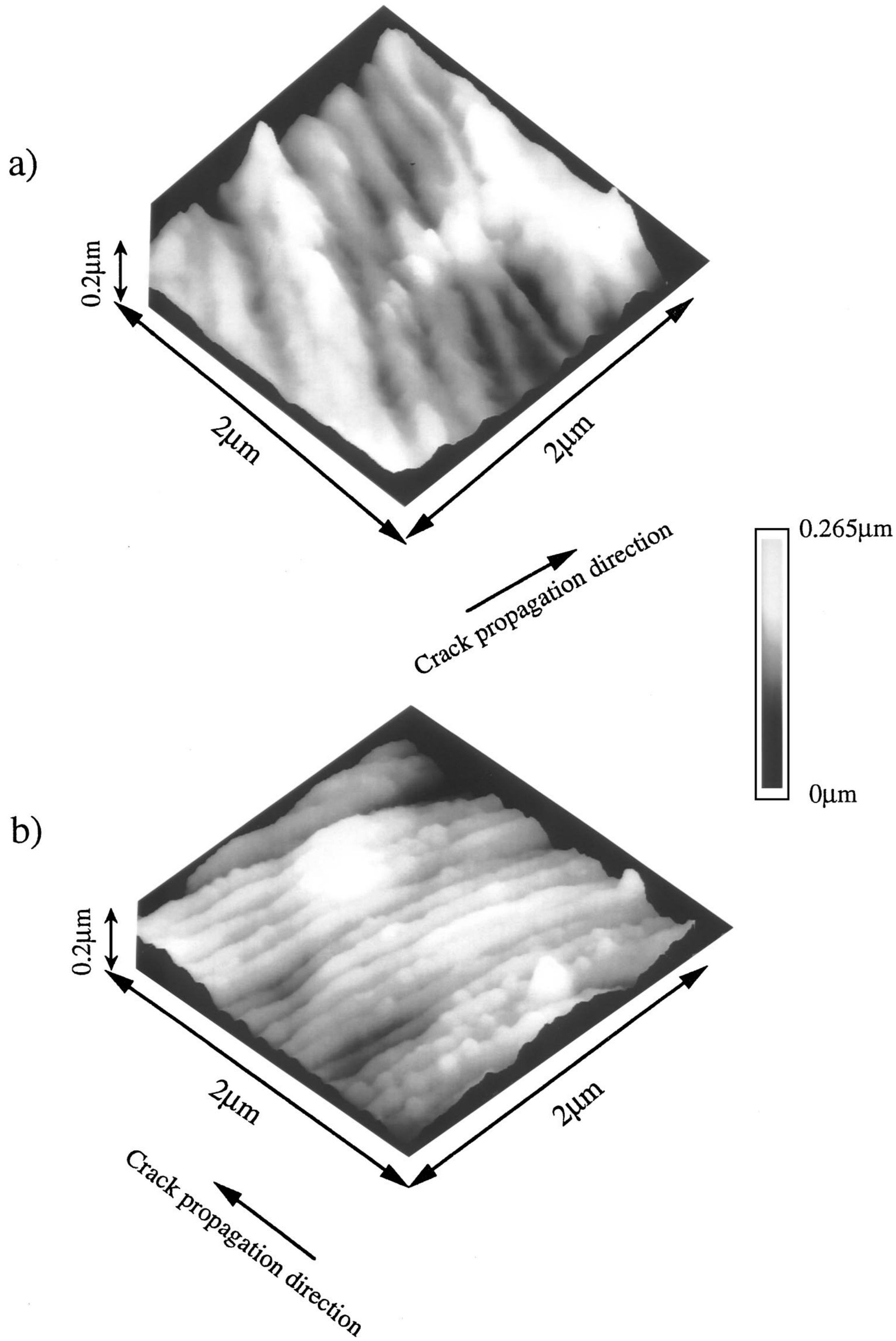
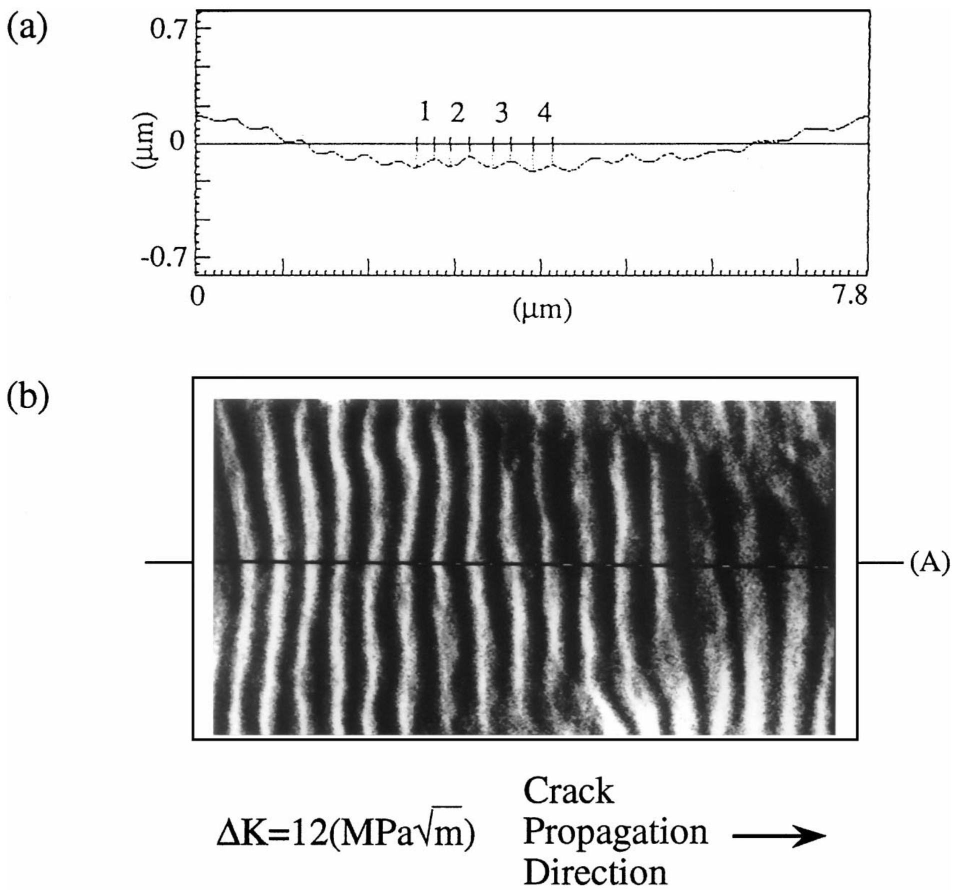


Figure 5 Striation height analysis on Fe-3% Si single crystal specimens using STM at stress ratios of 0.1 (a) and 0.6 (b).

including (ii), (iii) are fixed. It is thought that apparent ratio of striated surface depends on (iv) which means the height of striations, because striations should be observed more clearly with increasing striation height (see Appendix). It is therefore suggested that striation height at a stress ratio of 0.1 is higher than that at a stress ratio of 0.6.

The crack growth rates at which apparent ratios of striated surface and plateau area are measured are in a range between 2×10^{-7} and 4×10^{-7} m/cycle. It is expected that striation height varies approximately a factor of two comparing in this measured crack growth rate range. However, the apparent ratio of striated surface does not change much with crack growth rate. Thus,



(c)

No.	X(μm)	Z(μm)	D(μm)	Angle(deg.)
1	0.198	0.042	0.203	11.965
2	0.229	0.047	0.234	11.637
3	0.198	0.038	0.202	10.906
4	0.214	0.033	0.216	8.863

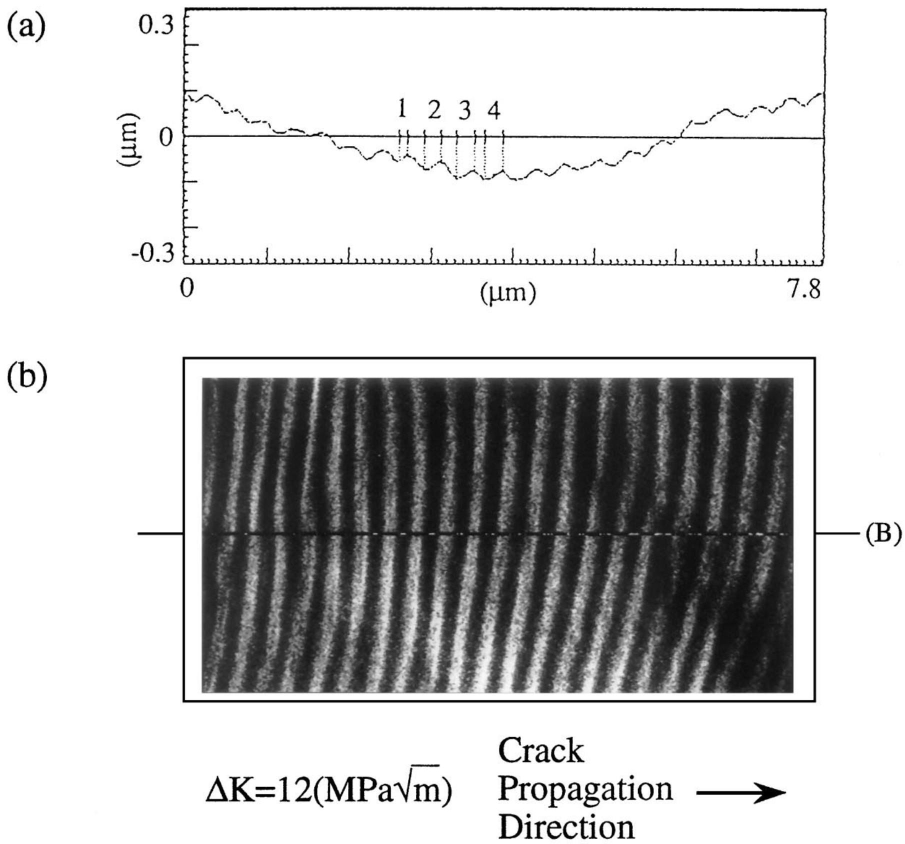
Figure 6 Striation height profiles (a), fractograph (b) and measured heights (c) using SIA on fatigue fracture surface of 7075 alloy at a stress ratio of 0.3. The profile indicates heights on the line in the fractograph. The numbers in (c) correspond to the measured points numbered in (a). X, Z, D and angle indicate projected horizontal length on the measured line, the relative heights, lengths between the corresponding points, and the averaged slope between the points, respectively.

the difference in striation height at stress ratios of 0.1 and 0.6 may be much greater than a factor of two.

Figs 10 and 11 show the apparent ratios of striated surface area to the observed surface area of the 7075 alloy having L-T and S-L orientation respectively, as a function of crack growth rate. As is observed on fracture surfaces of the Fe-Si alloy, the apparent ratio of striated surface increases with decreasing stress ratio for both orientations. The apparent ratio of striated surface should increase with an increase in crack growth rate due to an increase in striation spacing. However, the ratio does not increase in a range between 10^{-7} and 10^{-6} m/cycle, and rather decreases with an increase in crack growth rate of more than 10^{-6} m/cycle. These are because of an increase in the ratio of fracture surface area produced by static modes such as dimple fracture surface, which is caused by an increase in the maximum stress intensity factor (K_{max}).

On both materials, striation height does not depend on crystallographic orientation. This finding agrees with the fatigue crack growth data which is independent of crystal orientation in Fe-Si single crystals [10]. This also agrees with an experimental result that the formation of striation is independent of crystal orientation when multiple slip occurs [11], which suggests that striation formation is not crystallographic and that striation morphology including height, spacing and alignment depends on stress field around crack tip [12].

On both the materials of any crystal orientation tested, striation height increased with decreasing stress ratio. This is quite important for practical post-crash analysis. It could become possible to estimate stress ratio from fracture surface analysis. Crack growth rate can be measured from striation spacing on the fracture surface. From the crack growth rate, we can estimate the ΔK or ΔK_{eff} value which was applied on the material before failure. Using the results obtained in this work, we could estimate stress ratio, that is the maximum and minimum stress intensity factors in a fatigue cycle (K_{max}, K_{min}), though we need to build up a database containing ΔK and striation height data of various materials for such analysis.



(c)

No.	X(μm)	Z(μm)	D(μm)	Angle(deg.)
1	0.107	0.011	0.107	5.660
2	0.198	0.017	0.199	5.002
3	0.229	0.016	0.230	3.980
4	0.229	0.018	0.230	4.443

Figure 7 Striation height profiles (a), fractograph (b) and measured heights (c) using SIA on fatigue fracture surface of 7075 alloy at a stress ratio of 0.6. The profile indicates heights on the line in the fractograph. The numbers in (c) correspond to the measured points numbered in (a). X, Z, D and angle indicate projected horizontal length, the relative heights, lengths between the corresponding points, and the averaged slope between the points, respectively.

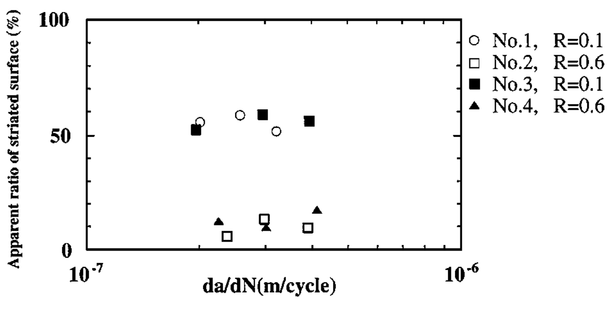


Figure 8 The apparent ratio of striated surface on fracture surface of Fe-3% Si single crystals as a function of crack growth rate.

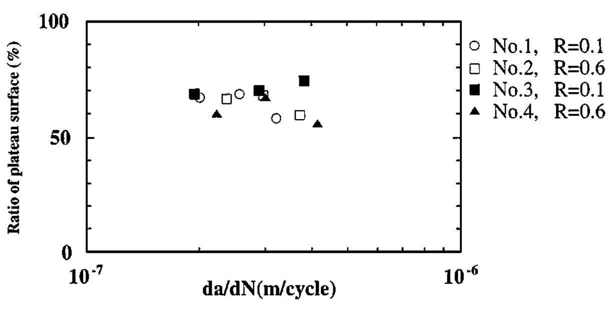


Figure 9 The ratio of plateau surface on fracture surface of Fe-3% Si single crystals as a function of crack growth rate.

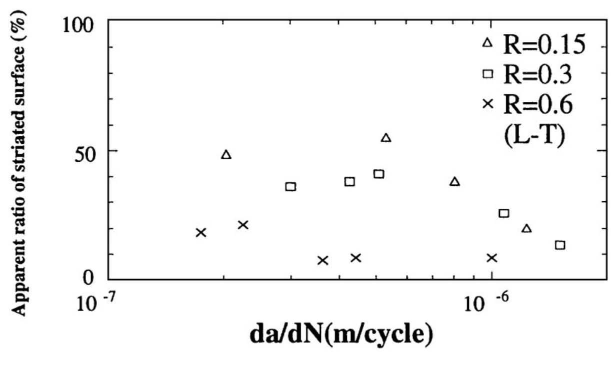


Figure 10 The apparent ratio of striated surface on fracture surface of 7075 aluminium alloy (L-T orientation) as a function of crack growth rate.

Many fatigue crack growth models have been proposed [13–18]. Neumann's model [15] is believed to be the mechanism of crack growth in single crystals. However, this model cannot account for the differences in striation height when stress ratio changes. It may also be believed that, at a low stress ratio, the fracture surfaces contact and crash together due to the closing force during unloading, and that a lower stress ratio produces

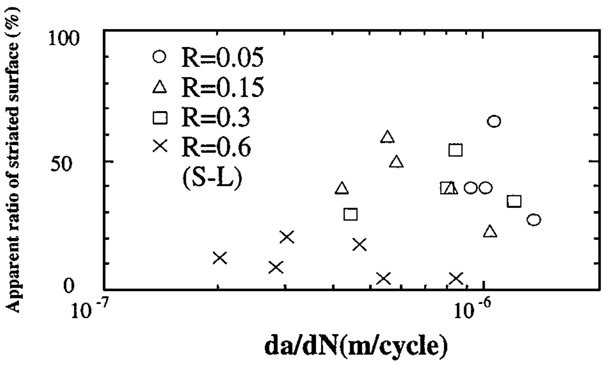


Figure 11 The apparent ratio of striated surface on fracture surface of 7075 aluminium alloy (S-L orientation) as a function of crack growth rate.

a lower striation height. This might be true, because the striation height differences are small between stress ratios of 0.3 and 0.05 (see Fig. 11). However, this surface contact mechanism cannot account for the difference in striation height between stress ratios of 0.6 and 0.3, because closure does not suppose to occur or is very small, if any, at these stress ratios, and the closing force may be very small. According to the surface contact mechanism, striation height at a stress ratio of 0.6 should be larger than that at 0.3, however, this is not. Thus, further studies are necessary to build up a striation formation mechanism which takes into account the results described above.

4. Conclusions

The effect of stress ratio on fatigue striation height was investigated on two different alloys using three different methods both microscopically and macroscopically. Striation height increased with decreasing stress ratio on both the alloys at a given crack growth rate. This suggests that not only stress amplitude but also stress ratio could be obtained by fracture surface analysis.

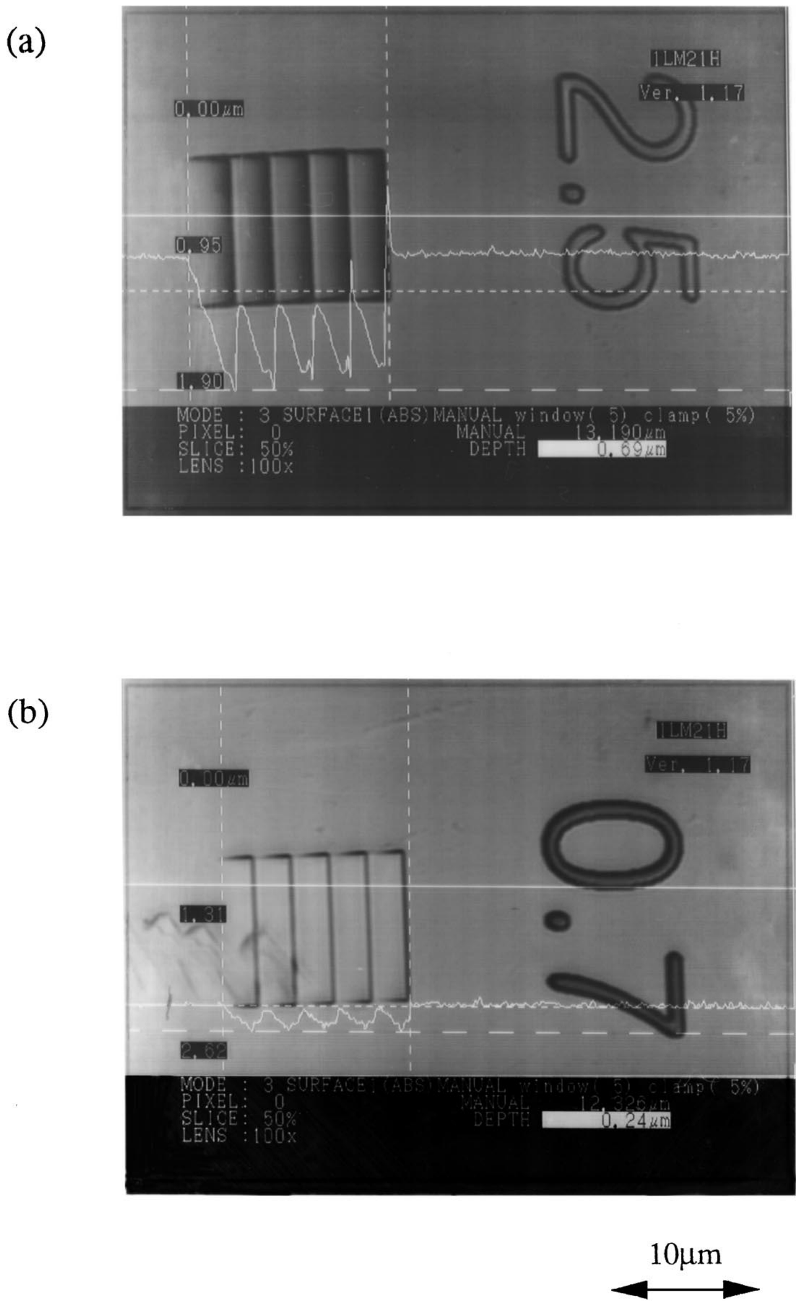


Figure 12 Scanning laser micrographs of saw-toothed steps and their depth profiles. The depths of them are 0.69 μm (a) and 0.24 μm (b).

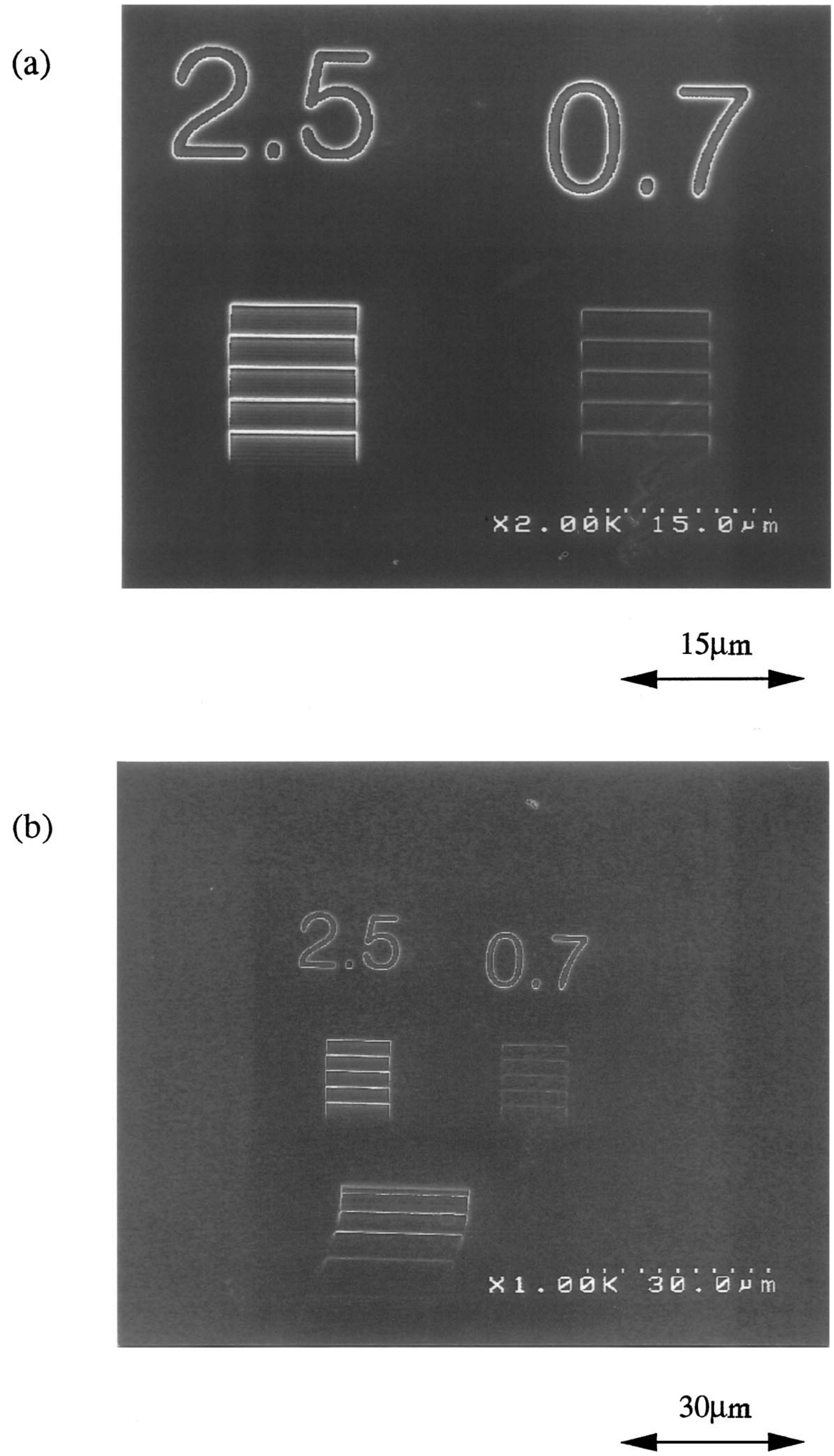


Figure 13 SEM photographs of saw-toothed steps at magnifications of 2000 (a) and 1000 (b).

Acknowledgements

The authors are grateful to Dr. S. Yamaguchi, Nippon Steel Fundamental Laboratory, for the supply of the material used in this work. This study was partly supported by Grant-in-Aid for COE Research from the Ministry of Education, Science, Sports and Culture, Japan (#07CE2003, "Ultra-parallel Optoelectronics").

Appendix

The factors affecting the contrast of SEM images [9] are:

(i) the atomic number of material
(ii) crystallographic orientation
(iii) acceleration voltage
(iv) tilt angle
(v) electric charge on the surface
(vi) magnetic field on the surface, and
(vii) surface geometry.

When we observe fracture surfaces of a metallic material having a certain crystallographic orientation, (i) and (ii) are constants, and (v) and (vi) are negligible. If we observe under a fixed condition such as (iii), (iv) and magnification, the only factor affecting the contrast of pictures is (vii). If we compare the contrast of pictures of two fatigue fracture surfaces at a given crack growth rate (thus the same striation spacing), the factor affecting contrast is striation height. Thus we can estimate striation height qualitatively.

To prove this, saw-toothed steps having two different depths and the same width were made on a flat metal surface using a focused ion beam sputtering machine (Hitachi, FIB-2000). The step depths are measured using a confocal scanning laser microscope (Lasertec, 1LM21).

Laser micrographs of the steps are shown in Fig. 12. The depth profiles are also shown in the figure. The depth was about 0.69 μm and the width of each teeth was about 2.6 μm for a deeper step shown in Fig. 12a. The other step, which is shallower, is shown in Fig. 12b. The depth of the shallower step was about 0.24 μm and the width of each teeth was about 2.5 μm, which is almost the same as that of Fig. 12a.

These steps were observed with a SEM at magnifications of 1000 and 2000, to see the difference in contrast. Micrographs are shown in Fig. 13. At a higher magnification (see Fig. 13a), both steps are clearly observed. However, at a low magnification (see Fig. 13b), the contrast of the shallower step (designated as "0.7" in the picture) is weak, though that of the deep steps (designated as "2.5" in the picture) is still strong. It is thus suggested that the contrast become stronger for deeper steps under a given observation condition. If we observe at a relatively low magnification, the striations whose height or spacing is less than the resolution will not be observed. Therefore, the height of striation can be measured qualitatively using this method.

References

1. J. C. McMILLAN and R. M. N. PELLOUX, ASTM STP 415 (American Society for Testing and Materials, 1967) p. 505.
2. J. SCHIJVE, *ibid.* p. 415.
3. P. J. E. FORSYTH and D. A. RYDER, *Aircr. Eng.* **32** (1960) 96.
4. A. J. KRASOWSKY and V. A. STEPANENKO, *Int. J. Frac.* **15** (1979) 203.
5. H. SATO and M. O-HORI, *Trans. Jpn. Soc. Mech. Eng.* **49** (1983) 227 (in Japanese).
6. *Idem., Trans. ASME, Ser. B (J. Eng. Ind.)* **109** (1987) 106.
7. P. J. E. FORSYTH, *Acta Metall.* **11** (1963) 703.
8. D. W. HOEPPNER, ASTM STP 415 (American Society for Testing and Materials, 1967) p. 486.
9. "Fundamentals and Applications of Scanning Electron Microscope," edited by Y. Tanabe (Kyoritsu Shuppan, Tokyo, Japan, 1989), p. 56 (in Japanese).
10. Y. HIGO and S. NUNOMURA, in Proc. 5th International Conference on Fracture (Cannes, France, 1981) Vol. 1, p. 291.
11. Y. UCHIDA, Y. HIGO and S. NUNOMURA, in Proc. Fatigue 90 (Hawaii, USA, 1990) Vol. 1, p. 117.
12. M. SHIMOJO, Y. UCHIDA and Y. HIGO, in Proc. 9th International Conference on Fracture (Sydney, Australia, 1997) Vol. 3, p. 1653.
13. R. M. N. PELLOUX, *Trans. ASM* **62** (1969) 281.
14. C. Q. BOWLES and D. BROEK, *Int. J. Fract. Mech.* **8** (1972) 75.
15. P. NEUMANN, *Acta Metall.* **17** (1969) 1219.
16. C. LAIRD, ASTM STP 415 (American Society for Testing and Materials, 1967) p. 131.
17. B. TOMKINS and W. D. BIGGS, *J. Mater. Sci.* **4** (1969) 544.
18. K. J. NIX and H. M. FLOWER, *Acta Metall.* **30** (1982) 1549.

Received 17 February
and accepted 12 November 1998