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### Authors

Mei, Z.  
Chang, G.  
Morris, J.W.

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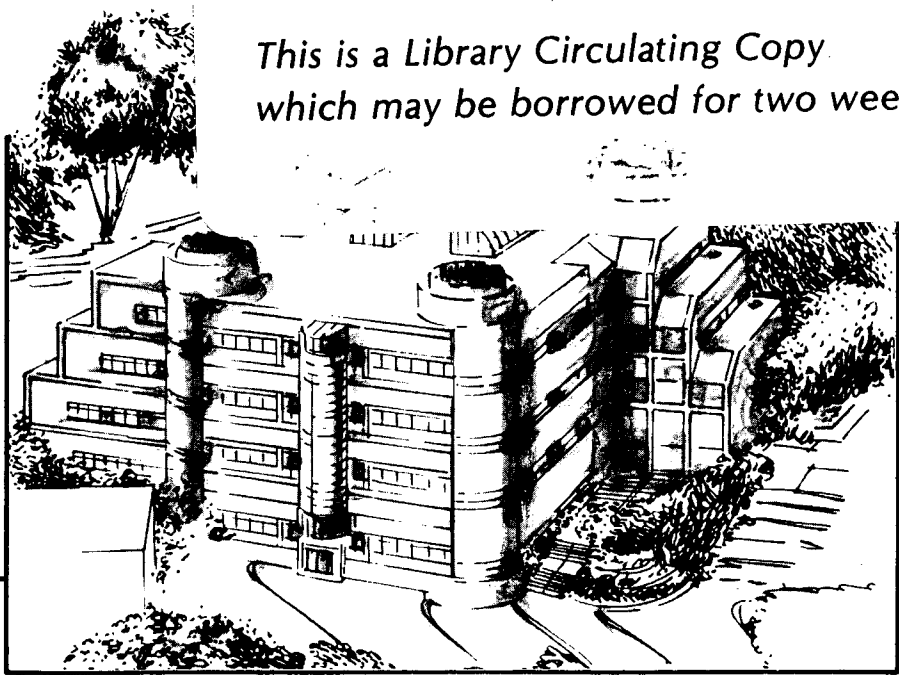
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**FATIGUE CRACK GROWTH IN METASTABLE  
AUSTENITIC STAINLESS STEELS**

Z. Mei, G. Chang and J. W. Morris, Jr.

Center for Advanced Materials  
Materials and Chemical Sciences Division  
Lawrence Berkeley Laboratory  
University of California

and

Department of Materials Science and Mineral Engineering  
University of California at Berkeley  
Berkeley, California 94720

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# FATIGUE CRACK GROWTH IN METASTABLE AUSTENITIC STAINLESS STEELS

Z. Mei , G.M. Chang, and J. W. Morris, Jr.

Center for Advanced Materials, Lawrence Berkeley Laboratory, and  
Department of Materials Science, University of California, Berkeley, California 94720

The research reported here is an investigation of the influence of the mechanically induced martensitic transformation on the fatigue crack growth rate in 304-type steels. The alloys 304L and 304LN were used to test the influence of composition, the testing temperatures 298 K and 77 K were used to study the influence of test temperature, and various load ratios (R) were used to determine the influence of the load ratio. It was found that decreasing the mechanical stability of the austenite by changing composition or lowering temperature decreases the fatigue crack growth rate. The R-ratio effect is more subtle. The fatigue crack growth rate increases with increasing R-ratio, even though this change increases the martensite transformation. Transformation-induced crack closure can explain the results in the threshold regime, but cannot explain the R-ratio effect at higher cyclic stress intensities.

## INTRODUCTION

Many common austenitic stainless steels are mechanically metastable at cryogenic temperatures and spontaneously transform into the martensite phase when subjected to sufficient stress or strain. The martensitic transformation causes a shape deformation that is evidenced by surface-relief effects [1]. There is also a volume change that is dependent on the composition, and is  $\approx 2\%$  expansion in 304-type stainless steels [2-3]. During fatigue crack growth the transformation is induced by the strain field ahead of the crack tip. The strain accompanying the transformation alters the stress state at the crack tip, and should, therefore, change the fatigue crack growth rate. This phenomenon is important in cryogenic engineering since many cryogenic structures are made of metastable austenitic steel, but is only partly understood. The work discussed here is part of a program of research to clarify the mechanisms of fatigue crack propagation in metastable austenitic steel. It involved a study of the fatigue crack growth rate in 304-type stainless steel as a function of composition, temperature and load ratio.

## EXPERIMENTAL PROCEDURE

The materials used in this study are commercial grade AISI 304L and 304LN stainless steels. They differ primarily through the addition of nitrogen to 304LN, which increases the strength and the thermodynamic stability of the austenite phase. The composition of the 304LN is in weight percent, 18.7Cr-9.55Ni-1.77Mn-0.021C-0.139N. Its grain size is 70  $\mu\text{m}$ . Its mechanical properties are  $\sigma_y = 341$  MPa (RT), 724 MPa (77 K);  $\sigma_u =$

643 MPa (RT), 1476 MPa (77 K). The 304L composition is 18.7Cr-8.64Ni-1.63Mn-0.024C-0.074N. It has a 100  $\mu\text{m}$  grain size,  $\sigma_y = 294$  MPa (RT), 433 MPa (77 K), and  $\sigma_u = 658$  MPa (RT), 1524 MPa (77 K). The martensite start temperature on cooling ( $M_s$ ) and deformation ( $M_d$ ) were estimated from the empirical formula given in references [4-5], and are, for 304LN,  $M_s < 0$  K,  $M_d \approx 255$  K; for 304L,  $M_s \approx 38$  K,  $M_d \approx 299$  K. Measurement of the volume fraction of martensite as a function of tensile strain at room and liquid nitrogen temperatures [6] confirms that the austenite phase in 304L is very much less stable than that in 304LN.

The fatigue crack growth rate was determined for 12.7 mm thick compact tension (CT) specimens of the geometry and size suggested by the relevant ASTM standard [7]. The fatigue crack plane was in the L-T orientation. The specimens were tested under load control in a hydraulic testing machine, using a sine-wave load form and a frequency of 10-30 Hz. The crack length was monitored continuously using the direct current electrical potential method [8-9]. The cyclic stress intensity ( $\Delta K$ ) was calculated from the crack length and cyclic load as suggested in the ASTM standard [7]. The crack length was recorded as a function of cycle number on a strip-chart recorder. The fatigue crack growth rate,  $da/dN$ , was determined from the slope of the curve. Tests were conducted over a range of growth rates from  $10^{-11}$  to  $10^{-6}$  m/cycle to sample both the near-threshold and Paris law regions. The near-threshold region crack growth rates were measured under decreasing  $\Delta K$  conditions, using a step-wise decrement in  $\Delta K$  of less than 5% at each step. At each load level, the crack was allowed to propagate at least 3 times the computed maximum plastic zone size formed at the previous load level. After establishing the threshold, the load was increased step-wise and  $da/dN$  values were recorded until the specimen sustained general yield. The room temperature fatigue tests were conducted in air at about 25°C (298 K); the tests at liquid nitrogen temperature (77 K) were done by immersing the sample in a 25 liter dewar filled with liquid nitrogen.

The extent of crack closure during fatigue crack growth was monitored continuously using the back-face strain gauge technique [10-11]. In this technique the closure stress intensity ( $K_{cl}$ ), which represents the first contact of the fracture surfaces during unloading, is determined from the load at which the elastic compliance curve derived from a strain gauge mounted on the back face of the specimen first deviates from linearity.

The mechanically induced martensite around the fatigue crack was observed after the fatigue test by optical microscopy on samples that were sectioned perpendicular to the crack plane at center thickness. Tests showed that no martensite was induced during grinding or polishing. Two methods were used to reveal the martensite: (1) chemical etching by 15 ml  $\text{HNO}_3$  - 45 ml  $\text{HCl}$  - 20 ml methanol, which reveals the grain boundaries and interfaces between martensite and austenite, and (2) decorating magnetic phases with ferrofluid [12-13], which highlights the magnetic  $\alpha'$  martensite in the para-magnetic austenite matrix. While all the optical metallography was done at room temperature, no reversion of martensite to austenite is believed to occur [14].

## RESULTS

To explore the influence of mechanically induced martensite on the fatigue crack growth rate the extent of transformation during fatigue was varied in three different ways: (1) by changing the chemical composition from that of 304L to that of 304LN, (2) by lowering the temperature from 298 K to 77 K, and (3) by varying the load ratio. The consequences of these three changes are the following.

**Chemical composition.** The measured crack growth rates of 304L and 304LN at 298 K and 77 K are plotted in Fig.1. The fatigue crack growth rates of the two alloys are very nearly the same at room temperature. However, at 77 K the crack growth rate of 304L is 10 times slower than that of 304LN at  $\Delta K = 10 \text{ MPa}\sqrt{\text{m}}$ , and is 4 times slower at  $\Delta K = 50 \text{ MPa}\sqrt{\text{m}}$ . These results correlate directly with the extent of martensitic transformation in the two alloys. Metallographic studies of the fatigue crack profiles show that at room temperature both 304L and 304LN remain essentially austenitic at the crack tip as  $\Delta K$  is varied from 3 to 40  $\text{MPa}\sqrt{\text{m}}$ . Neither is significantly affected by martensitic transformation. Moreover, the difference in their static mechanical properties does not seem to have an important effect on the fatigue crack growth rate. At LNT, on the other hand, 304L is substantially transformed while 304LN shows only a slight transformation at the higher values of  $\Delta K$ . As shown in Fig. 3, very little transformation is seen around a fatigue crack in 304L when  $\Delta K$  increases to 15  $\text{MPa}\sqrt{\text{m}}$ . However, as shown in Fig. 4, martensite is seen around the crack tip even when  $\Delta K$  approaches  $\Delta K_{\text{th}}$ , and a broad region of extensive transformation is present when  $\Delta K$  is greater than about 20  $\text{MPa}\sqrt{\text{m}}$ . The fatigue crack growth curve is apparently shifted sharply to the right leading to a significantly decreased crack growth rate when the chemical composition is changed to promote martensitic transformation.

**Temperature.** Fig.1 also illustrates the effect of decreasing the test temperature on the fatigue crack growth rate of metastable austenitic steels. The fatigue crack growth rate of 304L at room temperature, where the austenite phase is stable, is significantly greater than that at liquid nitrogen temperature, where the alloy undergoes extensive transformation. On the other hand, the fatigue crack growth rate is relatively insensitive to temperature in 304LN, which is essentially stable at both test temperatures. Again, the martensitic transformation appears to slow the fatigue crack growth. The composition and temperature effects observed here are consistent with previous work [15-22].

**Load ratio.** The influence of the load ratio on the fatigue crack growth rate at 77 K is illustrated in Fig 2. The plot shows that as the load ratio,  $R$ , increases from 0.05 to 0.5 (representing a 1.9 times increase in  $K_{\text{max}}$  for given  $\Delta K$ ), the fatigue crack growth rate curve shifts sharply to the left for the unstable alloy, 304L, but is essentially unchanged for the stable alloy, 304LN. As  $R$  increases from 0.1 to 0.75, the crack growth rate of 304L in the Paris region at 77 K increases by a factor of 18. These results suggest that the martensitic transformation induces a load-ratio effect; the fatigue crack growth rate increases with increasing  $R$ .

The influence of stability on the load-ratio effect is also suggested by the data shown in Fig. 5. In this plot the fatigue crack growth rate at given R ratio is normalized by dividing it by the growth rate at  $R = 0.1$ . The value is the same for all  $\Delta K$  in the linear, Paris region of the crack growth curve. This plot compares the high load-ratio effect in unstable 304L at 77 K to the more modest effect under more stable conditions. In all cases the fatigue crack growth rate increases with R, but by an amount that is more pronounced in the alloys and conditions where a relatively low martensite stability is expected.

To gain further insight into this behavior the size of the transformation zone and the tendency toward crack closure were measured for various R-ratios. Fig. 6 shows the transformation zone size as a function of  $\Delta K$  and  $K_{max}$ , as measured by optical microscopy on chemically etch and ferrofluid treated fatigue cracked specimen. The plots show that the transformation zone size carries roughly as  $(K_{max})^2$  for a given value of R, which may simply reflect the variation of the plastic zone size with  $(K)^2$ , but is not simply determined by  $K_{max}$ , since it varies with R (or  $\Delta K$ ) for a given  $K_{max}$ . The transformation zone size increases with R at given  $\Delta K$ , but only slightly.

Crack closure is clearly seen in backface strain gauge measurements on 304L at LNT at  $R=0.05$  and  $0.3$  when  $\Delta K$  is less than about  $20 \text{ MPa}\sqrt{\text{m}}$ , but occurs at  $R=0.5$  only at very small  $\Delta K$ . Surprisingly, the stress intensity at crack closure ( $K_{cl}$ ) at moderate  $\Delta K$  (the Paris regime) is maximal at  $R=0.3$ ; it hence cannot explain the monotonic increase of crack growth rate with R. No crack closure was observed in 304LN at room and/or liquid nitrogen temperatures or in 304L at room temperature. It follows that transformation-induced crack closure can explain the high threshold stress intensity,  $K_{th}$ , of 304L at LN temperature, but cannot explain the behavior in the Paris region.

## DISCUSSION AND CONCLUSION

Decreasing the mechanical stability of the austenite phase in 304 stainless steel by changing either composition or temperature decreases the fatigue crack growth rate at given values of R and  $\Delta K$ . These results suggest that an increase in the extent of martensitic transformation at the crack tip imparts resistance to fatigue crack growth. A plausible mechanism exists for behavior in the threshold regime: the martensitic transformation induces crack closure which reduces the effective ( $\Delta K$ ). The closure mechanism apparently does not apply in the Paris regime since closure disappears at higher  $\Delta K$ , where differences in the fatigue crack growth rate are still observed.

Increasing the load ratio (R) in 304L at 77 K increases the fatigue crack growth rate in the Paris regime. This effect is apparently also associated with the martensitic transformation since it is much stronger than in similar alloys that are more stable because of their compositions or temperatures. However, the effect is puzzling; since the extent of martensitic transformation increases with R at given  $\Delta K$ , the composition and temperature results suggest that the crack growth rate should decrease rather than increase with R. The puzzle cannot be simply solved by invoking crack closure, since the R-effect continues at stress intensities above those for which closure is significant, and since the fatigue crack growth



rate increases monotonically with R while the degree of closure does not. Noting that the fatigue crack growth rate in 304L at 77 K is never as high as that of 304LN, it appears that increasing R diminishes the beneficial effect of the martensite transformation, without completely eliminating it, even though the amount of martensite increases. The effect is under investigation. Its mechanism may become clear when the source of improved crack growth properties in metastable austenitic steels is known.

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## REFERENCES

- [1] J. F. Breedis and W. D. Robertson, *Acta Metall.*, 1962, vol. 10, pp. 1077-1088
- [2] H. Fiedler, B. Averbach and M. Cohen, *Trans. ASM*, 1955, vol. 47, p. 276
- [3] R. Reed, *Acta Metall.*, 1962, vol. 10, pp. 865-877
- [4] G. H. Eichelman and F. C. Hull, *Trans. Am. Soc. Met.*, 1953, vol. 45, pp. 77-104
- [5] I. Williams, R. G. Williams and R. C. Capellaro, *Proceedings of the Sixth International Cryogenic Engineering Conference*, IPC Science and Technology Press, Guildford, Surrey, England (1976), pp. 337-341
- [6] G. M. Chang, M.S. Thesis, University of California, Berkeley, 1983
- [7] *Annual Book of ASTM Standards*, E 647 - 83, p. 739, American Society for Test and Materials, Philadelphia, PA., 1983
- [8] *Metals Handbook*, 9th edition, vol. 8, p.386, America Society for Metals, Metals Park, Ohio, 1985
- [9] Beevers, C. J., *The Measurement of Crack Length and Shape During Fracture and Fatigue*, Engineering Materials Advisory Services LTD, West Middlelands, U. K., 1981
- [10] R. O. Ritchie and W. Yu, *Small Fatigue Cracks*, R. O. Ritchie and J. Lankford, eds., TMS-AIME, Warrendale, PA, 1986, pp. 167 - 189
- [11] W. F. Deans and C. E. Richards, *J. Test. Eval.*, vol. 7, 1979, p. 147.
- [12] *Metals Handbook*, 9th edition, vol. 9, pp. 63-70, America Society for Metals, Metals Park, Ohio, 1985
- [13] R. J. Gray, *Revealing Ferromagnetic Microstructures with Ferrofluids*, ORNL-TM-368, Oak Ridge National Laboratory, Oak Ridge, Tenn., March 1972.
- [14] T. H. Coleman, and D. R. F. West, *Metals Technology*, Feb. 1976, pp. 49-53.
- [15] A. G. Pineau and R. M. Pelloux, *Metall. Trans.*, 1974, vol. 5, pp. 1103-1112.
- [16] R. L. Tobler and R. P. Reed, in *Materials Studies for Magnetic Fusion Energy Applications at Low Temperatures-2*, NBSIR79-1609, National Bureau of Standard, Boulder, CO, 1979, pp.101-129.

- [17] R. L. Tobler and R. P. Reed, *J. of Testing and Evaluation*, 1984, vol. 12, No. 6, pp. 364-370.
- [18] G. Schuster and C. Altstetter, *Metall. Trans. A*, 1983, vol. 14, pp. 2085-2090.
- [19] G. R. Chanani, S. D. Antolovich, and W. W. Gerberich, *Metall. Trans. A*, 1972, vol.3, pp. 2661-2672.
- [20] E. Hornbogen, *Acta Metall.*, vol. 26, pp. 147-152.
- [21] C. Bathias and R. M. Pelloux, *Metall. Trans. A*, 1973, vol. 4, pp. 1265-1273.
- [22] G. Schuster and C. Altstetter, *Metall. Trans. A*, 1983, vol. 14, pp. 2077-2083
- [23] B. Yahiaoui and P. Petriquin, Note Technique RAM (73) 567, Division de Metallurgie et D 'Etude des Combustibles Nucleaires, Centre d'Etudes Nucleaires de Saclay, December 1973.
- [24] L. A. James, *Fatigue Crack Growth Measurement and Data Analysis*, STP-738, American Society for Testing and Materials, pp. 45-57, 1981.
- [25] J. L. Bernard and G. S. Slama, *Nuclear Technology*, vol. 59, No. 1, pp. 136-147, 1982.
- [26] L. A. James, Report HEDL-TME 75-20, Westinghouse Hanford Company, February 1975.

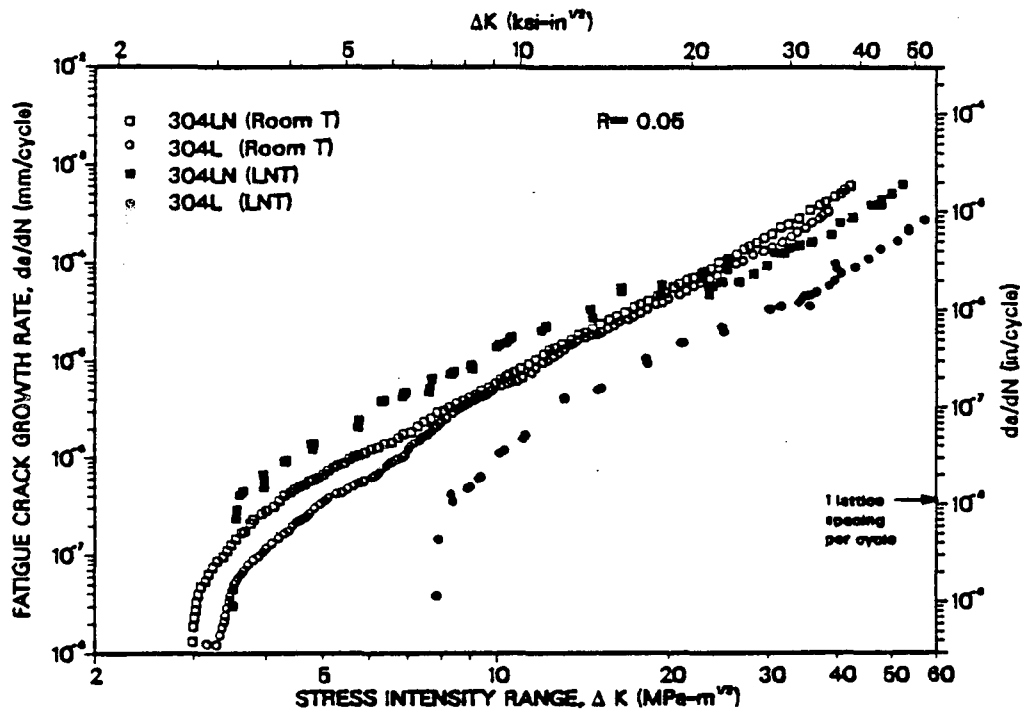


Fig. 1: Log-log plots of  $da/dN$  vs.  $\Delta K$  for 304L and 304LN tested at room and liquid nitrogen temperatures with load-ratio 0.05.

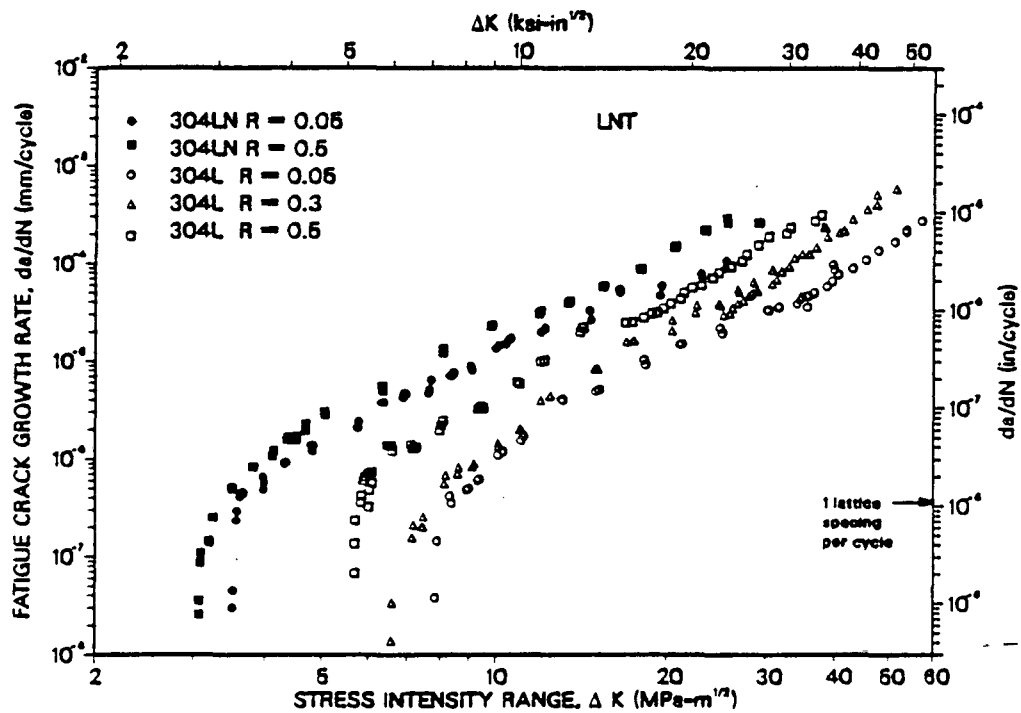


Fig. 2: Log-log plots of  $da/dN$  vs.  $\Delta K$  for 304L and 304LN tested at liquid nitrogen temperature with load-ratio varying from 0.05 to 0.5.

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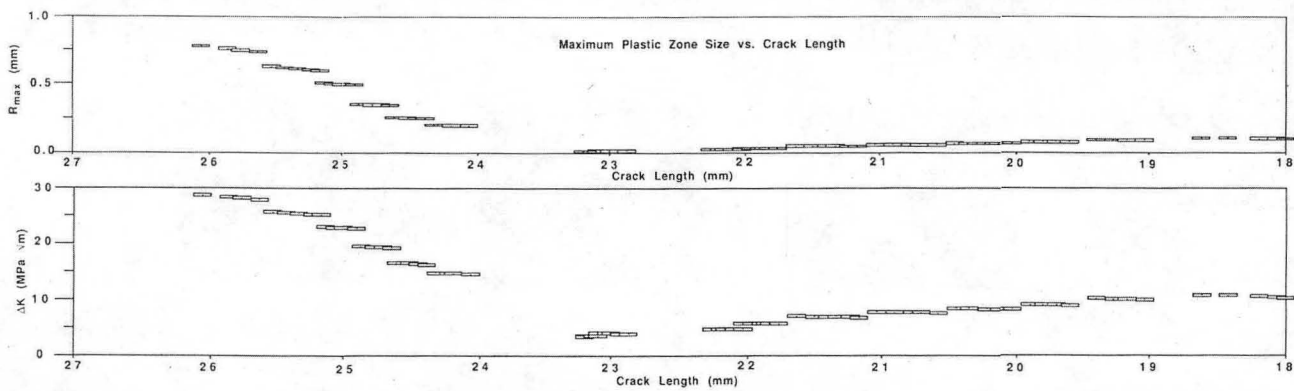
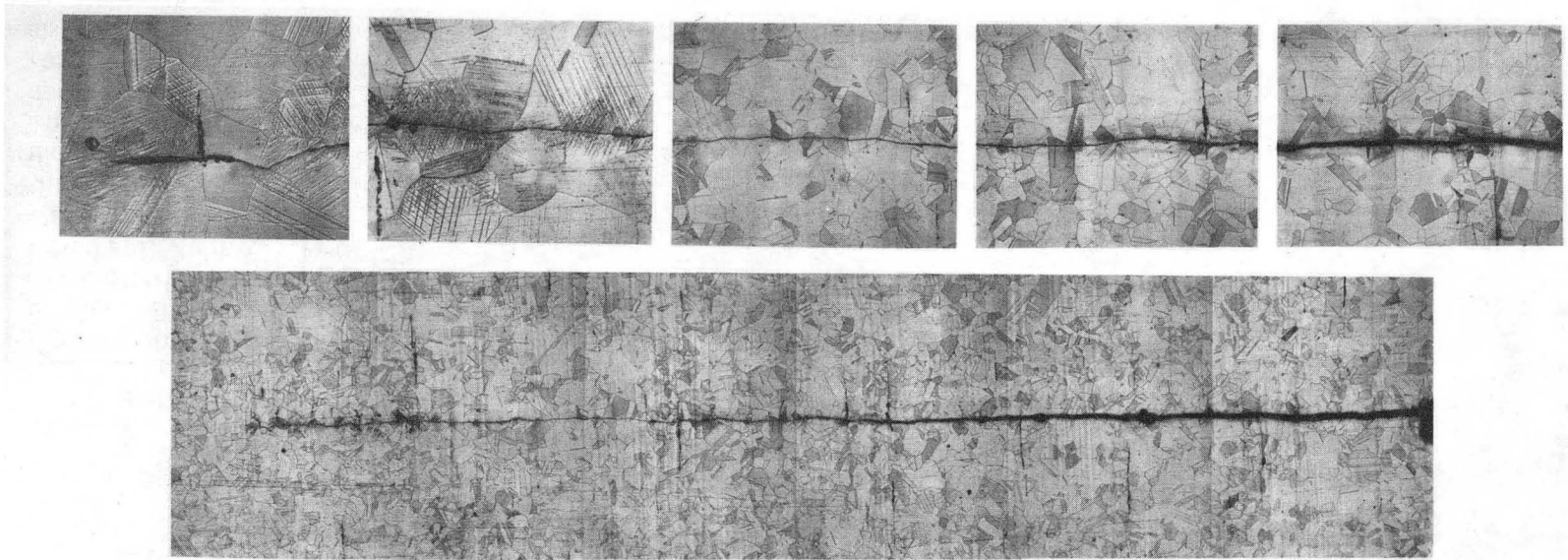


Fig. 3: Optical micrographs of the fatigue crack profile of 304LN tested at LN temp with load-ratio 0.05, showing deformation induced martensite. The calculated maximum plastic zone size and  $\Delta K$  are also indicated

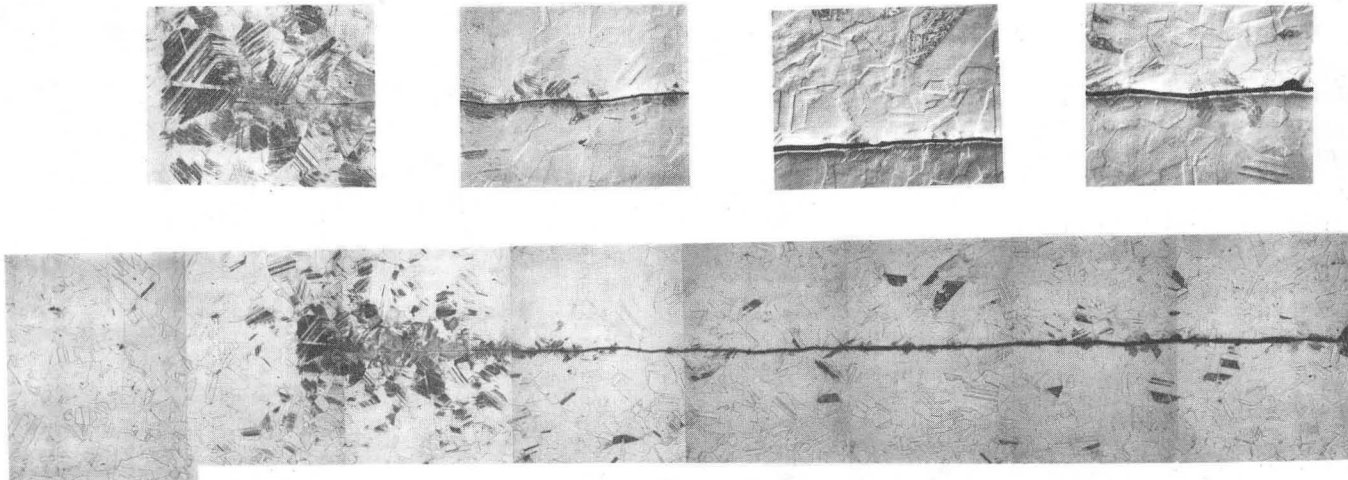
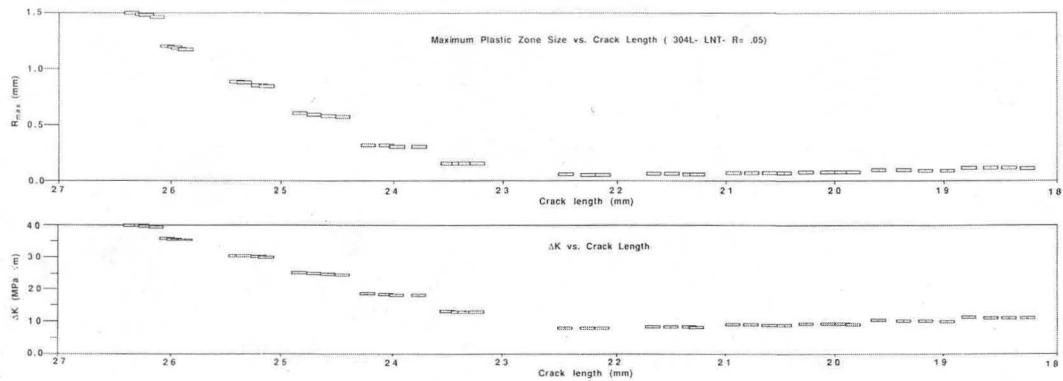


Fig. 4: Optical micrographs of the fatigue crack profile of 304L tested at LN temp. with load-ratio 0.05, showing deformation induced martensites. The calculated maximum plastic zone size and  $\Delta K$  are also indicated.



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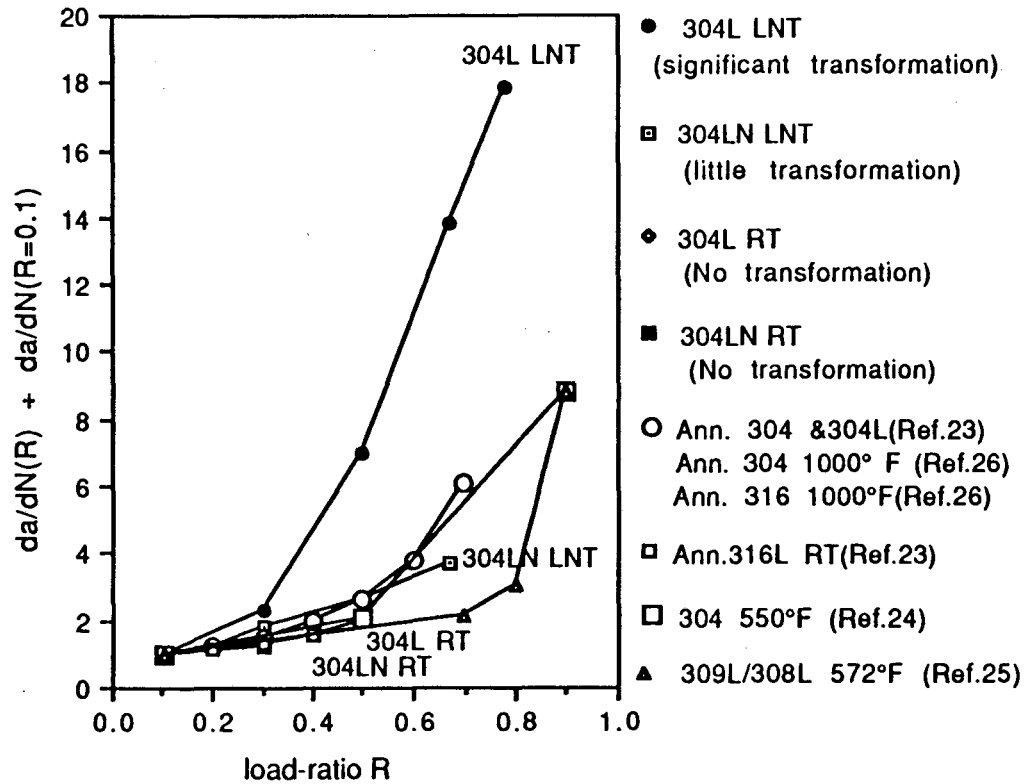


Fig. 5: Load-ratio effect on fatigue crack growth rate of several stainless steels at different conditions.

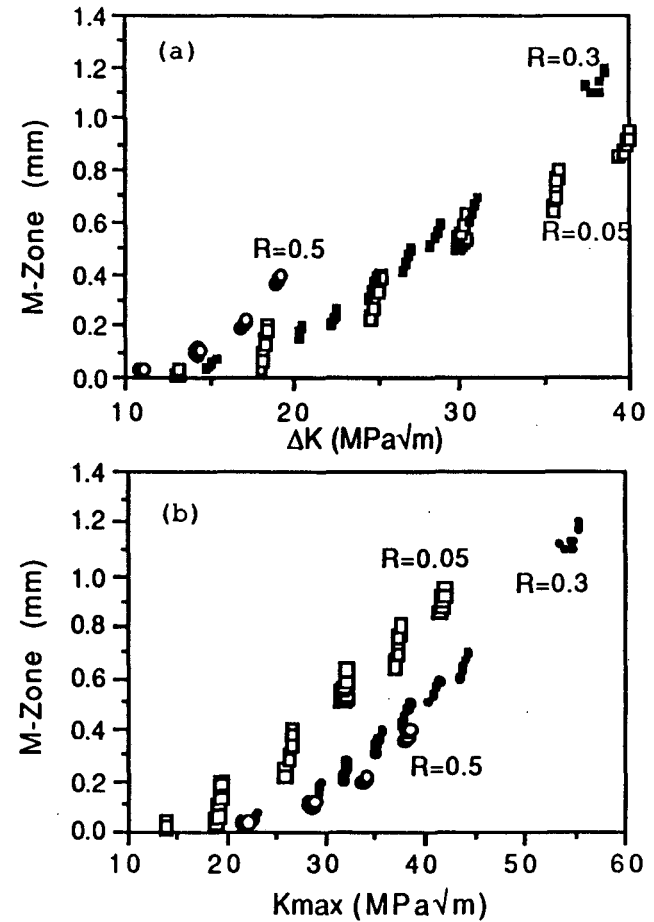


Fig. 6: The 10-20% martensite transformation Zone size around the fatigue cracks of 304L tested at LN temperature with three load-ratio  $R$  as a function of (a)  $\Delta K$  (b)  $K_{max}$ .

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