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AUSTENITE STABILITY AND ITS INFLUENCE ON MECHANICAL PROPERTIES OF 18-8 STAINLESS STEEL AT CRYOGENIC TEMPERATURES

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G.-M. Chang
(M.S. Thesis)

June 1983

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CRYOGENIC TEMPERATURES 

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(M.S. Thesis) 

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June 1983 

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AUSTENITE STABILITY AND ITS INFLUENCE ON

MECHANICAL PROPERTIES OF 18-8 STAINLESS STEEL AT

CRYOGENIC TEMPERATURES

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ABSTRACT

Stress/strain induced martensitic transformation has been found to play an important role on the mechanical properties of metastable austenitic stainless steels. Typical two-staged stress-strain curves were observed for AISI 304L and 304LN stainless steels containing 18% Cr and 8%Ni. The martensite phase induced in the high-work-hardening stage was found, by transmission electron microscopy, to be accommodated by mechanical twinning. The drop of 0.2% offset yield strength caused by the dilation and induced slip associated with the martensitic transformation of the
low-carbon-content 304L was observed at liquid nitrogen temperature. Low temperature favorable fatigue properties were observed for 304L but not for 304LN. Below the $M_d$ temperature, the fatigue crack propagation in metastable austenitic steels can be assumed to be the same as crack growth in an austenite and martensite composite. Although fatigue cracks grew faster in the martensite phase than in the austenite phase, the compressive stress near the crack tip due to the martensitic transformation appeared to reduce the effective stress intensity range which resulted in a decreased crack growth rate. The controlling factor became $K_{\text{max}}$ instead of $\Delta K$ if the load ratio $R$ was not large. The addition of interstitial nitrogen, which stabilizes the austenite phase, reduced the compressive stress at crack tips and resulted in the temperature-insensitive fatigue properties. Both the austenite phase and the martensite phase were found to be ductile at liquid nitrogen temperature.

June 2, 1983
I. INTRODUCTION

The AISI 304 series stainless steels are austenitic Fe-18%Cr-8%Ni alloys offering relatively low strength but excellent cryogenic ductility and toughness. By virtue of favorable mechanical properties, service history, and availability, AISI 304 is perhaps the most widely used cryogenic alloy in the world. Carbon is known as a relative inexpensive and effective strengthener, but carbon contents must be held to low levels (less than 0.03%) to prevent sensitization (chromium carbide precipitation) during thermal excursions. These carbide precipitates in the grain boundaries may be detrimental to the toughness of the steels. Nitrogen has the same strengthening effect as carbon but does not result in sensitization. Recently, AISI 304LN has attracted attention as a possible substitute for AISI 304 in applications demanding higher strength.

All 304-type stainless steels are metastable at cryogenic temperatures, which undergo martensitic transformation when stress is applied. For martensitic transformation, as for any other reaction, the following two conditions must be fulfilled:

(i) The free energy of the system must decrease through the transformation.

(ii) Nuclei must be present.

Lecroisey and Pineau [1] and Reed and Guntner [2] found that the martensite laths formed by plastic deformation were frequently located at the intersection of two deformation bands. Since deformed metastable stainless steels are abundant in these regions and can serve as nuclei, the controlling factor for the transformation is the supply of energy. Below the $M_d$ temperature, the free energy of the martensite phase is lower than the free energy of the austenite phase; but the transformation does not start until enough of energy is supplied to the system to overcome the activation barrier. Applying stress
generally fulfills this requirement. Angel [3] showed that the martensite phase started to form when a certain value of stress had been reached. This threshold value, which was generally higher at higher temperatures and was also a function of the chemical composition, increased with the austenite stability.

The martensitic transformation plays an important role in determining the mechanical properties and is often encountered in practice as an unusual work hardening. Tensile properties of AISI 304 type stainless steels have been well studied [1,2,4,5]. As the temperature decreases (below the $M_A$ temperature), the amount of induced martensite increases, which raises ultimate tensile strength and work-hardening rate significantly, but reduces elongation. Composition variation which is important to the stability of the austenite phase can markedly change the shape of the stress-strain curves. There is also a 3% dilation associated with the transformation. Ludwigson and Berger [4] showed that the dilation plus strain hardening of the austenite before the transformation and the strengthening effect of the martensite formed during the transformation contributed appreciably to the plastic behavior of metastable stainless steels.

Tobler and Reed [6] studied the fracture toughness properties of a series of AISI 304-type stainless steels with different C+N interstitial contents which indicate the stability of the austenite phase. They observed that more martensite phase formed in the specimens of higher toughness and they also observed an inverse relationship between $K_{IC}$ and C+N contents. Parker and Zackay [7] and Antolovich and Singh [8] also claimed that a certain amount of induced martensite in the plastic deformation zone in front of a crack would increase the fracture toughness in TRIP steels.

Tobler and Reed [9] also studied the fatigue properties. Temperature-insensitive and low-temperature-favorable behaviors were observed. There was
no general conclusion. Pineau and Pelloux [10] and Hornbogen [11] studied the fatigue properties of some metastable steels and concluded that compressive stresses at crack tips due to volume expansion associated with the transformation would reduce fatigue crack growth rate. However, all their tests were conducted at a fixed minimum/maximum load ratio, $R$, and no quantitative measurements of the compressive stress were made.

In the present study, two types of AISI 304 stainless steels with low carbon contents, but different nitrogen contents, were tested at various temperatures to study the effects of the nitrogen interstitial element. Tests of different load ratio $R$ were also performed. Besides fatigue tests, tensile tests were also performed to compare the stabilities of the steels under stresses and to study the strengthening effect of the induced martensite phase.
II. EXPERIMENTAL DETAILS

A. Materials The compositions, hardnesses, and average grain sizes of two commercial grade stainless steels, AISI 304L and 304LN, used in this study are listed in Table I. According to Pickering's summary [12] on the effect of alloying elements, the $M_s$ temperature of 304LN was estimated to be 60°C lower than 304L, which was mainly due to the high C+N and Ni contents of 304LN. No martensite phase was found in either of the steels after being quenched repeatedly in liquid nitrogen for several hours. All 304L specimens were cut from 1 inch (25.4 cm) thick plates, annealed at 1050°C for 1 hour followed by water quench. 304LN was used in the as-received condition.

B. Tensile Tests

The tensile properties were measured using sub-sized round tensile specimens with 1 inch (25.4 mm) gauge length and 1/4 inch (6.3 mm) gauge diameter (Fig. 1). The specimens were oriented along the rolling direction, so that longitudinal tensile properties were measured. Tests were conducted at room temperature, 0°C (in ice water), -78°C (in mixture of dry ice and ethyl alcohol), and -196°C (liquid nitrogen) at a constant strain rate 0.02/min. The yield strength was taken to be the stress corresponding to 0.2% offset strain. The elongation was measured using a traveling microscope with an accuracy of 0.001 inch (0.02 mm).

Pin loaded flat tensile specimens with 1 inch (25.4 mm) gauge length, 1/4 inch (6.3 mm) gauge width and 1/8 inch (3.1 mm) thickness (Fig. 2) were given different amounts of plastic tensile strain at room temperature and liquid nitrogen temperature. The gauges were then cut off from the specimens and placed in an X-ray diffractometer to determine the amounts of transformed martensites associated with the corresponding plastic strains. A set of 5 specimens were tested for each steel at every testing temperature.
C. Fatigue Tests

Fatigue tests were conducted with a 50 kip (220 kN) capacity Instron 1332 universal testing machine at room and liquid nitrogen temperatures. The Instron was modified so that the specimen could be immersed in a 25 liter liquid nitrogen container for the liquid nitrogen temperature test. Compact tension specimens with 2 inch (50.8 mm) width and 0.785 inch (20.0 mm) thickness (Fig. 3) were used. The crack plane orientation was L-T. All tests were conducted using load control. The deviation of peak-loads was controlled to be less than 1%. The load cycle was sinusoidal at a frequency of 10 Hz and the ratio R (of minimum and maximum loads) were chosen at 0.1 and 0.3.

Crack length was monitored during the tests by means of elastic compliance measurement using a C-shaped clip gauge attached directly to the notched edge of the specimens. The correlation between crack length and specimen compliance was calculated theoretically by the method of Saxena and Hudak [13]. Generally, there were two beach marks on the fracture surface of every specimen caused by load changes at the ends of the precracking and fatigue tests. The distance between the mark and loading axis represented the crack length corresponding to the mark. Since the mark was curved, crack length was determined as an average of 5 measurements at 0, 25, 50, 75, and 100% of the specimen thickness. The relation between the crack lengths measured this way and the corresponding compliances is shown in Fig. 4, there is agreement with the theoretical calculation within a deviation of 0.005 inch (0.1 mm).

The crack growth rates were determined by the secant method recommended by ASTM standard E847-81 and the corresponding stress intensity factor range ΔK was calculated from the expression for compact tension specimen.

\[
\Delta K = \frac{\Delta P}{B\sqrt{W}} \left(\frac{2+\alpha}{(1-\alpha)^{3/2}}\right) \left(0.886+4.84\alpha-13.32\alpha^2+14.72\alpha^3-5.8\alpha^4\right) \tag{1}
\]
where \( \Delta P \) is the applied load range, \( B \) the specimen thickness, \( W \) the specimen width, and \( \alpha \) the ratio of crack length and \( W \).

D. X-Ray Diffraction

The use of X-ray techniques to determine the relative volume fractions of phases in multiphase alloy specimens is well established [14]. The determination is made by comparing the relative integrated diffraction intensities of peaks associated with each of the phases which are proportional to their relative volume fractions. A Picker X-ray diffractometer equipped with diffracted beam (200) Li-F crystal monochromator was used in the present study, with Cu-K\( \alpha \) incident radiation. The fluorescence background of the X-ray was substantially eliminated by the monochromatization technique. The diffractometer is attached to a stepping motor and a controller. The integrated intensity of the diffraction peaks is automatically collected with the aid of a computer terminal.

Miller [15] has developed a simple procedure utilizing a direct comparison method to evaluate the phase fraction of steels containing the \( \gamma \) austenite phase and the \( \alpha \) martensite phase. The method involves measuring the integrated intensities under the (311)\( \gamma \), the (220)\( \gamma \), and the (211)\( \alpha \) peaks. The average area under these two austenite peaks in a completely austenitic specimen, when multiplied by a factor 1.4, is equivalent to the area of the (211)\( \alpha \) peak in a completely ferritic specimen under identical conditions of diffraction. When austenite and martensite are present in the same sample, the volume fraction of the martensite, \( V_\alpha \), is hence given by the relation:

\[
V_\alpha = \frac{I_\alpha}{I_\alpha + 1.4I_\gamma}
\]

where \( I_\gamma \) is the average of the integrated intensity from the (220)\( \gamma \) and (311)\( \gamma \) planes, and \( I_\alpha \) is that from (211)\( \alpha \) planes.
E. Optical Metallography

Samples for the optical metallograph were cut from the gauge of the strained flat tensile specimens and from the broken fatigue specimens along the crack growth direction and perpendicular to the fracture surface. These samples were ground with emery paper and polished on a rotary wheel with 1 micron diamond compound then electro-etched in saturated oxalic acid at 8 volts. Since both 304L and 304LN were relatively stable at room temperature, there was no martensite phase induced during sample preparation. Samples from the tensile specimens were examined to identify the morphology of the induced martensite phase. Samples from fatigue specimens were examined to compare the transformation zone sizes around the cracks.

F. Transmission Electron Microscopy

Transmission electron microscopic analysis was used to characterize the detailed morphology of the transformed martensite phase. Samples were cut transversely from a 304L round tensile specimen close to the necked region, then ground and electropolished in a twin jet polishing machine with a solution of 20% perchloric acid + 80% methanol. Optimum thinning was obtained using a voltage of 30 volts at -50°C. The thin foils were examined in a Siemens 101 transmission electron microscope operated at 125 kV.

G. Fractography

The fracture surfaces of the broken tensile and fatigue specimens were examined in an AMR-1000 scanning electron microscope operated at 20 kV. Because of the large depth of field of the SEM, this technique is excellent for characterizing the fracture mode.
III. RESULTS

A. Tensile Results

Tensile properties of both 304L and 304LN measured at room temperature, 0°C, -76°C, and liquid nitrogen temperature are listed in Table II. Both temperature and composition variations affected tensile properties.

The ultimate tensile strengths of both 304L and 304LN increased drastically as temperature decreased from room temperature to liquid nitrogen temperature, but the elongations decreased. The yield strength of 304LN also increased as temperature decrease while the yield strength of 304L increased first then started to decrease as temperature was lowered from -76°C to liquid nitrogen temperature. This observation was in agreement with Reed and Guntner's results [2] for low carbon content 304 type stainless steels.

Strengthening elements carbon and nitrogen increased the yield strength and kept it from decreasing as temperature decreased but had little effect on ultimate tensile strength. Strengthening elements decreased the elongation at room temperature but had good effects at liquid nitrogen temperature. The elongation of 304LN was 51.3% at liquid nitrogen temperature compared to 48.1% for 304L, while it was 71.7% for 304LN and 85.5% for 304L at room temperature.

The relations between the amounts of induced martensite and corresponding tensile strain are shown in Fig. 5. At room temperature, 304LN was stable when strained up to 27%, while up to 7% of martensite formed in 304L. At liquid nitrogen temperature, both 304L and 304LN were unstable, undergoing a stress/strain induced martensitic transformation. For 304L, the martensite phase was induced even when stressed in the elastic region.

Stress-strain curves of the tensile tests are shown in Figs. 6 and 7. At room
temperature, both 304L and 304LN showed the low strength, low work-hardening rate, and large elongation which were typical of austenitic stainless steels. As temperature decreased, the work-hardening rate began to increase as seen in the later stages of the stress-strain curves, and a plateau region of low work hardening formed immediately after yielding. This phenomenon was most pronounced at liquid nitrogen temperature. Corresponding to Fig. 5, the amount of martensite at the onset of the drastic increase in work hardening rate was about 15% for both 304L and 304LN.

Microscopy.

Optical micrographs, taken from the flat tensile specimens, are shown in Fig. 8. All specimens were given a 10% plastic strain. There is a small amount of martensite in the 304L specimen strained at room temperature but no martensite in 304LN. Large amounts of martensite can be seen in both 304L and 304LN specimens strained at liquid nitrogen temperature; however, 304L has significantly more martensite than 304LN. This is consistent with the X-ray measurements. All of the martensite was formed along the slip bands or at the intersection of slip bands, which was in agreement with the observation of previous workers [1,2].

Transmission electron micrographs taken of the 304L tensile specimen broken at liquid nitrogen temperature are shown in Fig. 9. The diffraction pattern (Fig. 9a) consists of fundamental martensite spots (BCC), twin spots and double diffraction spots arising from the interaction of diffracted electron beams of martensite and twins. The bright field image (Fig. 9b) shows the martensite lath structure. Dark field images (Figs. 9c, 9d) show that there are many micro-twins between the martensite laths which indicates that the martensite phase induced at this stage is accommodated by mechanical twinning. Scanning electron micrographs (Figs. 10, 11) show the fracture surfaces of all
the broken tensile specimens. The fracture mode is ductile and the dimple sizes decrease as temperature decreases.

B. Fatigue Results

For the stress-intensity-factor ranges studied, the fatigue crack growth rates for both 304L and 304LN can fit the Paris law equation of the form:

\[
\frac{da}{dN} = C(\Delta K)^n
\]

where the parameters C and n are material and temperature dependent. Using Figs. 12 to 15, log-log plots of \( \frac{da}{dN} \) versus \( \Delta K \) were constructed for all tests performed. The C and n values that correspond to the ordinate intercepts at \( \Delta K = 1 \) and the slopes were obtained by the least square method. Table III lists the C and n values for all material, temperature and load ratio R combinations.

As temperature decreased from room temperature to liquid nitrogen temperature, the crack growth rate decreased as well. For 304L, the crack growth rate decreased by a factor of 8 for tests of load ratio R=0.1 and by a factor of 3 for R=0.3. For 304LN, the crack growth rate decreased by a factor of 1.3 for R=0.1 and decreased only slightly for R=0.3.

As the load ratio R increased from 0.1 to 0.3, the crack growth rate increased. For 304L, the crack growth rate increased by a factor of 1.5 at room temperature and a factor of 5 at liquid nitrogen temperature. For 304LN, the crack growth rate increased by a factor of 1.3 at room temperature and by a factor of 1.8 at liquid nitrogen temperature.

The addition of the strengthening element, nitrogen, decreased the fatigue growth rate very little at room temperature, but reduced the effects of both temperature and load ratio R. Actually, the fatigue data for 304LN fell into a band with a width factor of 2, which can be related to the temperature insensitive behavior observed by Tobler and Reed [9], and the data for 304L can be
related to the low temperature favorable behavior.

Microscopy.

Optical metallographic observations of the transformation zones are shown in Figs. 16 and 17. The zone sizes are from 10 to 100 μm in 304LN specimens tested at liquid nitrogen temperature; and larger than 100 μm in 304L specimens. In both 304L and 304LN specimens tested at room temperature, the transformation zones are negligible. The morphology of the induced martensite phase in the transformation zone is shown in Fig. 18.

The results of scanning electron microscopic observation of fatigue fracture surfaces are shown in Fig. 19. The micro-failure modes, which consist of a rough surface, are the same regardless of testing conditions. Fig. 20 shows the fracture surface of the torn part of a 304L specimen broken after fatigue test at liquid nitrogen temperature. The 100% ductile dimple surface indicates that the martensite phase is still ductile at this temperature.
IV. DISCUSSION

As shown in the results, the liquid nitrogen temperature tensile stress-strain curves of both 304L and 304LN consist of a low work hardening plateau in the first stage of deformation and a high work hardening region in later stage. Guntner and Reed [5] stated that the plastic strain in the first region was due to the stress-induced hexagonal martensite. Such an explanation is impossible in the case of Fe-Ni austenitic alloys which show the same tensile behavior, but no hexagonal martensite is formed. Suzuki et al. [16] performed an in-situ observation of the stress/strain induced transformation in a transmission electron microscope and found that α martensite, with accompanying slip dislocations, was formed at the junction of, or just outside of the intersecting slip bands. Their conclusions were: (1) It is internal stress due to the dislocation pile up at the intersection of slip bands which induces the formation of α martensite. (2) In order to accommodate itself the induced α martensite promotes slip of piled up dislocations directly related to their birth. This observation explains the presence of the low work hardening plateau as the induced slip is one of the primary slip systems in the first stage of deformation. In the later stage of deformation, α martensite is formed along all active slip plane junctions which hinders dislocation movements and results in a high work-hardening rate. The results of the present study show that the amount of α martensite at the onset of high work hardening rate were the same (15%) for both 304L and 304LN. The transmission electron microscopic observation showed that the α martensite was accommodated by mechanical twinning instead of dislocation movements. Both these results support the theory that the hardening effect was due to the α martensite formation in the later stage.

The nominal 0.2% offset yield strength of 304L was reported to be lower at liquid nitrogen temperature than at -76°C. It was also observed that there was
martensite induced even though the tensile stress was below the elastic limit. Hence, it was the 3% dilation associated with the transformation and the induced slip discussed above that made the nominal 0.2% offset yield strength drop. Subtracting the contribution of dilation on plastic strain, the true yield strength of slip, defined by the intersection of the two extrapolations from the elastic deformation and the first stage of deformation of the stress-strain curve, increased as temperature decreased.

At room temperature, the amounts of the martensite formed in the fatigue crack tip plastic deformation zone were negligible for both 304L and 304LN, indicating that the crack propagated mostly through the austenite phase. Since a large amount of martensite was formed in fatigue crack tip deformation zone at liquid nitrogen temperature, it could be assumed that the fatigue crack growth at this temperature represented crack propagation mostly through the induced martensite phase. Although the martensite had higher yield strength, the lower crack growth rates in 304L were not attributed to the fact that the fatigue crack growth rate was lower in the martensite phase than it was in the austenite phase. On the contrary, Hornbogen [11] and Pineau and Pelloux [10] observed that the fatigue crack growth rate in the fully martensitic condition was never less than in the fully austenitic condition. Tobler and Reed [12] observed a fracture mode transition from partly intergranular failure at room temperature without martensitic transformation to a fully transgranular failure at liquid nitrogen temperature with martensitic transformation for some AISI 304 type stainless steels with carbon contents up to 0.094%. The intergranular fracture could have been encouraged by the absorption of carbon interstitials at grain boundaries. As mentioned before, the carbon content must be kept below 0.03% to avoid sensitization.

There are three other factors that affect fatigue crack growth rate in
metastable austenitic steels. One is the marked increase in work-hardening rate due to the transformation, the others are the residual stress and strain near the crack tip due to dilation resulting from the transformation. Pineau and Pelloux [10] have studied the effects of the work-hardening rate by using microhardness measurements to assess the degree of hardening near the fatigue crack tip. Their results showed that an increase in the work-hardening rate in both monotonic and cyclic deformation zones should have led to a decrease of crack tip opening displacement. McClintock [17] also showed that the crack tip opening displacement decreased when the monotonic work hardening rate increased. Based on the crack extension model [20], both results showed an increase in work-hardening rate which led to a decrease in fatigue crack growth rate. However, they all admitted that the predicted change of the crack growth rate was not as large as the observed decrease of the crack growth rate of 304L when the temperature was decreased from room temperature to liquid nitrogen temperature.

Hornbogen [11] has studied the effect of residual stresses caused by martensitic transformation by treating the stress field near a crack tip as being analogous to an edge dislocation and obtained the following result:

\[
\sigma_r = \frac{\tau_t G_f \frac{\Delta V}{3V} \sin \phi}{r (1-\nu)} \text{ at } K=0
\]

where \(\sigma_r\) is the residual stress, \(\tau_t\) the transformation zone size (which is a function of \(K_{\text{max}}\)), \(f\) the fraction of induced martensite in the transformation zone, \(\Delta V/V\) the volume change, and \(r\) and \(\phi\) are polar coordinates. Estimated this way, the residual stress \(\sigma_r\) at distance \(\tau_t\) in front of a crack tip, was in the order of \(10^3\) ksi (\(10^4\) MPa, compression), which would interact with the stresses caused by the external load and modify the stress intensity factor range \(\Delta K\) by \(+K_t\). Applying this modification to the fatigue crack growth theory for work
hardening materials proposed by Weertman (20) and combining the effect of work hardening due to transformation, Hornbogen obtain the following relation:

\[
\frac{da}{dN} = B' \frac{(\Delta K + K_t)^4}{\sigma_y^2 E (U_p + U_t)}
\]

(5)

where \(B'\) is a dimensionless constant, \(\sigma_y\) the yield strength, \(U_p\) the energy for the cyclic deformation that causes fatigue crack propagation (in dimension of in-lb/in\(^2\), or J/m\(^2\), which is less for the martensite phase), \(U_t\) the modification on \(U_p\) due to the work hardening rate increase from the transformation, and \(K_t\) is the modification of stress intensity factor range whose sign depends on the volume change due to the transformation. In the present study, the sign should be negative because of volume expansion.

According to this theory, an increase in \(r_t\) due to a lower stability of the austenite phase leads to an increase in \(K_t\) and results in a decrease in \(da/dN\) as temperature decreases, which is in agreement with the results of prior workers and the present study. But an increase in \(K_{max}\) also result in an increase in \(r_t\) and hence an increase in \(K_t\) because of larger stress in front of the crack tip. According to this argument, keeping the same stress intensity factor range \(\Delta K\), and increasing the load ratio \(R\) will lead to an increase in \(K_t\) and a decrease in \(da/dN\), which is contrary to the results of present study.

Actually, the residual stress \(\sigma_t\) estimated at \(K=0\), had the same effect on \(K_{max}\) and \(K_{min}\) (effective \(K_{max}=K_{max}-K_t\)), effective \(K_{min}=K_{min}-K_t\)). which meant that effective stress intensity range, \(\Delta K_{eff}\), could remain unchanged \((\Delta K_{eff}=\text{effective } K_{max} - \text{effective } K_{min})\). However, in the case that \(K_{min}\) is less than \(K_t\), effective \(K_{eff}\) is negative. Since a crack does not propagate when the corresponding stress intensity factor is less than zero, effective \(K_{eff}\) should be assume to be zero. Using this concept, the effects of load ratio \(R\) can be explained reasonably well. When the load ratio increases from \(R_1\) to \(R_2\), \(K_{max}\) increases by a factor of \(\frac{1-R_1}{1-R_2}\), while \(K_{min}\) increases by a factor of \(\frac{R_2}{R_1}\).
\( \frac{1-R_2}{1-R_1} \) for the same \( \Delta K \). In the present study, \( R_2 = 0.3 \) and \( R_1 = 0.1 \), thus \( K_{\text{min}} \)
increases faster than \( K_{\text{max}} \) by a factor of \( \frac{R_2}{R_1} = 3 \). Although the exact relation
between \( K_{\text{max}} \) and \( K_{\text{i}} \) is not known, it is reasonable to assume that \( K_{\text{min}} \)
increases more than \( K_{\text{i}} \) does. In the case that \( K_{\text{min}} < K_{\text{i}} \), the value of \( (K_{\text{i}} - K_{\text{min}}) \)
declines and the value of \( (K_{\text{max}} - K_{\text{i}}) \) increases, as load ratio \( R \) increases, sub-
sequently \( \Delta K_{eff} \) increases and crack growth rate increases. The results of
present study agree with this argument. It should be noticed that when the
load ratio \( R \) increases to certain value, \( K_{\text{min}} \) equals \( K_{\text{i}} \) and \( \Delta K_{eff} = \Delta K_{\text{nom}} \), the
effect of compressive stress due to the transformation vanishes.

Since \( K_{\text{i}} \) is dependent on \( K_{\text{max}} \), \( \Delta K_{eff} \) should be determined by \( K_{\text{max}} \)
only, if \( K_{\text{min}} < K_{\text{i}} \). Hence \( K_{\text{max}} \) is the controlling factor for the fatigue crack
growth rate; and the same \( K_{\text{max}} \) will lead to the same \( \Delta K_{eff} \) and the crack
growth rate, regardless of the differences of load ratio \( R \) for a testing condition.
Fig. 21 shows the relation between the crack growth rate of 304L tested at
liquid nitrogen temperature and \( K_{\text{max}} \). The data points for \( R = 0.1 \) and \( R = 0.3 \) fall
on the same line which conformed the above argument.

The effects of temperature, load ratio and alloying elements on fatigue
crack growth rate in metastable austenitic stainless steels can be discussed by
using the above arguments.

Temperature Effect

As the temperature decreases (below \( M_d \)), \( \sigma_y \) increases, \( U_p \) decreases
slightly since the fatigue crack grows faster in the martensite phase (\( U_p \) may
decrease drastically if temperature decreases below the DBTT of the martensite
phase), \( U_{\text{i}} \) increases due to the strengthening effect of the martensite phase,
and \( \Delta K_{eff} \) which is the most significant factor decreases for load ratio \( R \) less
than certain value. Altogether, the crack growth rate decreases as temperature decreases.

Load Ratio Effect

As load ratio R increases, $\Delta K_{eff}$ increases, and also the crack growth rate. Since $K_{max}$ does not increase much, the effect of $(U_p + U_t)$ is not significant.

Alloying Element Effect

Alloying elements which stabilize the austenite phase reduce the effects of both temperature and load ratio R. The only beneficiary effect of the alloying elements is to increase yield strength.

McMeeking and Evans [20], however, have developed another quantitative approach which explains the change of the stress intensities of cracks in some ceramic material as being due to the effect of compressive stresses resulting from a martensite transformation. In the case of metal where the martensite phase would be accommodated by dislocation movement and twinning, the theory should be modified to be applied.

The role of residual strain near the fatigue crack tip has been demonstrated by Elber [21] who introduced the concept that a propagating fatigue crack may be partially or completely closed even at a tensile load. This concept has been widely used to explain fatigue threshold properties. Suresh and Ritchie [22] have also discovered some other closure mechanisms. In the present study, the closure effect has not been measured because crack propagation in a higher stress intensity factor range (region II) has been the main concern. However, there is a large compressive stress near a crack tip and a volume expansion due to transformation in metastable austenitic steels, crack closure is very likely to occur, especially in the threshold region.
V. CONCLUSION AND SUMMARY

1. The nominal 0.2% yield strength of 304L drops as temperature decreases to liquid nitrogen temperature because of the volume expansion and induced slip resulting from the martensitic transformation, however the true yield strength for slip increases.

2. The transformed martensite phase, which contributes to the high work-hardening rate in the later stage of deformation is accommodated by micro-mechanical twinning.

3. The transformed martensite phase is still ductile at liquid nitrogen temperature.

4. Fatigue crack grows faster in the martensite phase than in the austenite phase of metastable stainless steels.

5. The volume expansion near a fatigue crack tip due to the martensitic transformation produces a compressive stress on the crack tip which may reduce the crack growth rate if load ratio R is below a critical value.

6. In metastable stainless steels, the controlling factor of fatigue crack growth rate is $K_{\text{max}}$ instead of $\Delta K$, if the load ratio R is below a critical value over the limited range of growth rate studied.
REFERENCES


Table I. Compositions (wt.%, Hardnesses, and Grain Sizes of 304L and 304LN.

<table>
<thead>
<tr>
<th>Material</th>
<th>304L</th>
<th>304LN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>Bal.</td>
<td>Bal.</td>
</tr>
<tr>
<td>Cr</td>
<td>18.70</td>
<td>18.54</td>
</tr>
<tr>
<td>Ni</td>
<td>8.64</td>
<td>9.55</td>
</tr>
<tr>
<td>Mn</td>
<td>1.63</td>
<td>1.77</td>
</tr>
<tr>
<td>P</td>
<td>0.021</td>
<td>0.014</td>
</tr>
<tr>
<td>S</td>
<td>0.010</td>
<td>0.009</td>
</tr>
<tr>
<td>Si</td>
<td>0.51</td>
<td>0.78</td>
</tr>
<tr>
<td>C</td>
<td>0.024</td>
<td>0.021</td>
</tr>
<tr>
<td>N</td>
<td>0.074</td>
<td>0.139</td>
</tr>
<tr>
<td>C + N</td>
<td>0.098</td>
<td>0.160</td>
</tr>
<tr>
<td>Hardness (Rockwell B)</td>
<td>76</td>
<td>100</td>
</tr>
<tr>
<td>Grain size (μm)</td>
<td>78</td>
<td>100</td>
</tr>
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</table>
Table II. Tensile Properties of 304L and 304LN.

<table>
<thead>
<tr>
<th>Material</th>
<th>Testing Temperature</th>
<th>0.2% Yield Strength ksi</th>
<th>Ultimate Tensile Strength ksi</th>
<th>Elongation* %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>MPa</td>
<td></td>
<td></td>
</tr>
<tr>
<td>304L</td>
<td>RT</td>
<td>42.7 294</td>
<td>95 658</td>
<td>85.5</td>
</tr>
<tr>
<td></td>
<td>0°C</td>
<td>43.8 302</td>
<td>109 751</td>
<td>73.4</td>
</tr>
<tr>
<td></td>
<td>-76°C</td>
<td>68.5 472</td>
<td>144 993</td>
<td>66.7</td>
</tr>
<tr>
<td></td>
<td>LNT</td>
<td>62.8 433</td>
<td>221 1524</td>
<td>48.1</td>
</tr>
<tr>
<td>304LN</td>
<td>RT</td>
<td>49.4 341</td>
<td>93 643</td>
<td>71.7</td>
</tr>
<tr>
<td></td>
<td>0°C</td>
<td>55.0 379</td>
<td>104 720</td>
<td>67.1</td>
</tr>
<tr>
<td></td>
<td>-76°C</td>
<td>67.4 465</td>
<td>146 1007</td>
<td>60.2</td>
</tr>
<tr>
<td></td>
<td>LNT</td>
<td>105.0 724</td>
<td>214 1476</td>
<td>51.3</td>
</tr>
</tbody>
</table>

* 1 in. (25.4mm) in gauge length.
### Table III. Power-Law Fatigue Equation Parameters.

<table>
<thead>
<tr>
<th>Material</th>
<th>Testing Temperature</th>
<th>R Ratio</th>
<th>n</th>
<th>C (Metric)*</th>
<th>AK Range ksi√in. (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>RT</td>
<td>0.1</td>
<td>4.17</td>
<td>5.15x10^-11</td>
<td>21-43 (8.45x10^-11) (23-47)</td>
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<tr>
<td></td>
<td></td>
<td>0.3</td>
<td>4.01</td>
<td>1.33x10^-11</td>
<td>23-37 (2.32x10^-11) (25-40)</td>
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<tr>
<td>LNT</td>
<td></td>
<td>0.1</td>
<td>4.72</td>
<td>9.31x10^-14</td>
<td>36-60 (1.51x10^-12) (39-65)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.3</td>
<td>4.02</td>
<td>4.16x10^-12</td>
<td>23-45 (7.23x10^-11) (25-49)</td>
</tr>
<tr>
<td>304LN</td>
<td>RT</td>
<td>0.1</td>
<td>3.00</td>
<td>2.51x10^-10</td>
<td>18-36 (4.80x10^-9) (19-39)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.3</td>
<td>3.20</td>
<td>1.65x10^-10</td>
<td>20-38 (3.10x10^-9) (22-41)</td>
</tr>
<tr>
<td>LNT</td>
<td></td>
<td>0.1</td>
<td>2.83</td>
<td>3.24x10^-10</td>
<td>19-49 (6.29x10^-9) (20-53)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.3</td>
<td>2.85</td>
<td>5.68x10^-10</td>
<td>20-45 (1.10x10^-8) (21-49)</td>
</tr>
</tbody>
</table>

* Metric da/dN is in mm/cycle instead of in/cycle.
FIGURE CAPTIONS

1. Sub-sized round tensile specimen.
2. Pin-loaded flat tensile specimen.
3. Compact tension specimen for fatigue crack growth rate tests.
4. Theoretical and experimental relations between elastic compliance and crack length.
5. Relations between the volume fraction of induced martensite and corresponding tensile strain for 304L and 304LN.
6. Stress-strain curves for 304L.
7. Stress-strain curves for 304LN.
8. Optical metallographies of strained (10%) 304L and 304LN.
9. Transmission electron microscopic images of induced martensite:
   (a). selected area diffraction pattern, (101) orientation;
   (b). bright-field image;
   (c). dark-field image from the (-101) reflection;
   (d). dark-field image from the twin reflection.
10. Scanning electron fractographs of borken 304L tensile specimens.
11. Scanning electron fractographs of broken 304LN tensile specimens.
12. Log-log plots of da/dN vs. ΔK for 304L tested at room temperature.
14. Log-log plots of da/dN vs. ΔK for 304LN tested at room temperature.
15. Log-log plots of da/dN vs. ΔK for 304LN tested at liquid nitrogen temperature.
16. Optical microscopic observations of transformation zone sizes along the fatigue cracks of 304L.
17. Optical microscopic observations of transformation zone sizes along the fatigue cracks of 304LN.

18. Induced martensite morphology along a fatigue crack.

19. Scanning electron micrographs of fatigue fracture surfaces of 304L and 304LN.

20. Scanning electron micrographs of fracture surfaces of 304L.

21. Log-log plots of $da/dN$ vs. $K_{\text{max}}$ For 304L tested at liquid nitrogen temperature.
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Fig. 2. Pin-loaded flat tensile specimen.

Thickness .125"
Fig. 3. Compact tension specimen for fatigue crack growth rate tests.
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- B: thickness
- E: elastic modulus
- V: displacement at specimen edge
- P: load

Circle markers represent data from 5 pt. average of crack front.
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Fig. 9a.

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Fig. 13. Log-log plots of $\frac{da}{dN}$ vs. $\Delta K$ for 304L, tested at liquid nitrogen temperature.
Fig. 14. Log-log plots of $\frac{da}{dN}$ vs. $\Delta K$ for 304LN XBL 829-6612 tested at room temperature.
Fig. 15. Log-log plots of da/dN vs. ΔK for 304LN tested at liquid nitrogen temperature.
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