

# Effect of Thermal Aging Upon the Fatigue-Crack Propagation of Austenitic Stainless Steels

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The effect of thermal aging upon the elevated temperature fatigue-crack propagation behavior of several austenitic stainless steels was investigated using linear-elastic fracture mechanics. The steels studied were AISI Types 304, 304L, 316 (both annealed and cold-worked), and weldments in Type 304 using Type 308 filler. Aging temperatures of 1000°F and 1200°F were employed, and aging times ranged between 1500 and 6000 hours. In general, thermal aging produced beneficial results (*i.e.*, lower crack growth rates) relative to unaged material in elevated temperature tests. It is suggested that the precipitation of the various carbide and intermetallic phases is responsible for the beneficial effect. One possible mechanism might be a blunting effect each time the crack tip encounters a second-phase particle, thereby requiring the crack to partially reinitiate itself before proceeding.

THE austenitic stainless steels are being utilized extensively for fuel cladding and structural components in nuclear reactors. Under service conditions, many of these components will be subjected to elevated temperatures for extended periods of time. Prolonged exposure of the austenitic stainless steels at elevated temperatures can result in the precipitation of various phases which may include (depending upon composition, prior thermo-mechanical treatments, temperature and time) the carbides  $M_{23}C_6$ ,  $M_6C$ , and  $MC$ , and the intermetallic phases  $\sigma$ ,  $\chi$ ,  $\epsilon$ , and  $\eta$  (Laves). (See Refs. 1 and 2 for two of the many studies in this area.) The precipitation of carbides is also dependent upon the amount of repeated fatigue straining. For example, Johansson<sup>3</sup> found a great increase in the precipitation of carbides at grain boundaries when going from the neutral axis toward the outer fibers of austenitic stainless steel specimens tested in reversed bending at elevated temperature. Hempel<sup>4</sup> has also observed that the carbide precipitation in austenitic stainless steels is accelerated by fatigue stressing at elevated temperatures. It has been shown that the precipitation of such phases can, under certain conditions, have an effect on the corrosion,<sup>1</sup> creep,<sup>5</sup> fatigue,<sup>6-16</sup> and mechanical properties<sup>17,18</sup> of the austenitic stainless steels.

With the exception of two recent studies,<sup>15,16</sup> all of the investigations concerning fatigue behavior have been conducted on smooth "S-N" type fatigue specimens, and hence yield no quantitative information regarding the rate at which fatigue cracks propagate under different conditions. Therefore, the objective of this study was to characterize the effect of various aging conditions (time and temperature) upon the fatigue-crack propagation behavior of several austenitic stainless steels.

The present study is one phase in an extensive program to characterize, in terms of linear-elastic fracture mechanics (LEFM), the crack growth behavior of austenitic stainless steels under various operating conditions: temperature,<sup>19-21</sup> cyclic frequency,<sup>21,22</sup> loading waveform,<sup>23</sup> stress ratio,<sup>24</sup> neutron irradiation,<sup>25</sup> environment,<sup>26</sup> crack growth in weldments,<sup>27,28</sup> and

crack growth under static loads.<sup>29</sup> LEFM techniques may be applied to elevated temperature tests so long as 1) plasticity is confined to a relatively small region in the vicinity of the crack, and 2) a linear relationship between stress and strain is maintained in the bulk of the material. If these conditions are violated to a significant degree, the stress intensity factor may not adequately describe the stress and strain distributions in the vicinity of the crack, and hence LEFM techniques would not be valid.

## EXPERIMENTAL PROCEDURE

Several austenitic stainless steels were studied in the present investigation: AISI Types 304, 304L and 316 in the solution annealed condition, 20 pct cold worked Type 316, and shielded-metal-arc weldments in Type 304 using Type 308 filler metal. With the exception of the Type 304L, all of the steels studied were from the same material heats previously used in other investigations in the author's laboratory.<sup>19-29</sup> The chemical compositions and mechanical properties are given in Tables I and II, respectively. The emphasis in the present study is on the behavior of solution annealed Type 304 (Heat No. 55697), and the thermal and mechanical processing history of this heat is detailed in Ref. 19.

The specimens were aged prior to testing in electric muffle furnaces in an environment of stagnant air at temperatures of 1000°F (538°C) or 1200°F (649°C). Aging times were 1500, 3000, or 6000 h. Several of the previous investigators studying the effects of thermal aging on the fatigue behavior of austenitic stainless steels have employed relatively high aging temperatures and relatively short aging times. Since, for a given volume fraction of particles, this may produce differences in size, shape and distributions of the particles, the present study employed somewhat lower aging temperatures (and hence longer times) that correspond more closely to expected service temperatures. The behavior of specimens that had not been aged prior to testing was also determined for comparison purposes.

ASTM "compact specimens"<sup>30</sup> were employed for this study. The specimens had the planar dimensions of a one-inch thick compact tension specimen, but were

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Table I. Chemical Composition (Pct by Wt)<sup>†</sup>

Alloy	Producer/Heat No.	C	M	P	S	Cr	Ni	Mo	Si	Cu	Al
Type 304‡	Allegheny Ludlum/55697	0.053	0.87	0.019	0.010	18.25	9.51	0.18	0.49	0.21	*
Type 304§	G.O. Carlson/600414-1A	0.064	1.42	0.019	0.015	18.79	9.48	*	0.68	*	*
Type 304L	East. S.S. Co./V50035	0.024	1.70	0.020	0.014	18.00	9.32	0.44	0.61	0.17	*
Type 308	Comb. Engr./JAFA	0.060	1.90	0.044	0.004	19.81	9.77	0.12	0.37	0.15	*
Type 316	Allegheny Ludlum/65808	0.060	1.72	0.012	0.007	17.30	13.30	2.33	0.40	0.065	0.012

Type	Ta	Cb	Ti	Pb	Sn	Co	Ca	Fe	B	Ce	N <sub>2</sub>	Total Rare Earths	Ferrite
Type 304‡	*	*	<0.01	<0.001	0.006	*	0.11	Bal.	*	*	0.030	<0.001	—
Type 304§	0.004	0.008	0.015	*	*	0.077	*	Bal.	*	*	*	*	—
Type 304L	*	*	*	*	*	0.15	*	Bal.	*	*	*	*	—
Type 308	*	*	0.06	*	*	0.17	*	Bal.	0.007	*	0.046	*	7.1
Type 316	*	*	0.003	0.0014	0.013	0.030	*	Bal.	0.005	0.0006	0.048	0.0010	—

\*Not determined.

†Average of multiple tests.

‡Excluding base metal for SMA weldments.

§Base metal for SMA weldments.

Table II. Room Temperature Mechanical Properties—(Unaged Material)<sup>†</sup>

Alloy	Producer/Heat No.	0.2 pct Yield Strength, psi	Ultimate Strength, psi	Pct Elongation	Pct Red. Area	ASTM Grain Size	Hardness
Type 304 Solution Annealed‡	Allegheny Ludlum/55697	39,600	77,050	65.0	60.7	4.5	BHN = 126
Type 304 Solution Annealed§	G. O. Carlson/600414-1A	37,000	82,000	57.0	*	*	*
Type 304L Solution Annealed	East. S.S. Co./V50035	39,300	90,750	67.4	*	*	R <sub>B</sub> = 82.5
Type 308 Shielded-Metal-Arc	Comb. Engr./JAFA	57,000	82,800	44.5	54.5	—	*
Type 316 Solution Annealed	Allegheny Ludlum/65808	44,100	82,100	68.0	63.4	4.0	BHN = 163
Type 316 20 pct Cold Worked	Allegheny Ludlum/65808	98,750	113,300	30.3	*	*	R <sub>c</sub> = 27.0

\*Not determined.

†Average of multiple tests.

‡Excluding base metal for SMA weldments.

§Base metal for SMA weldments.

nominally 0.5 in. (12.7 mm) thick (the 20 pct cold worked Type 316 specimens were nominally 0.35 in. (8.9 mm) thick). The specimens were fatigue cycled on an MTS testing machine using load as the control parameter. The majority of the tests were conducted at 1000°F (538°C) at a cyclic frequency of 40 cpm (0.667 Hz) using either a sinusoidal or saw-tooth waveform. As will be discussed later, one test was conducted at 75°F (24°C), and one test was conducted at 1000°F at a frequency of 0.083 cpm ( $1.39 \times 10^{-3}$  Hz) using a square waveform. The elevated temperature tests were conducted in an air-circulating furnace, and temperatures were controlled to within  $\pm 2^\circ\text{F}$  ( $\pm 1^\circ\text{C}$ ).

Crack lengths were obtained periodically throughout each test using a traveling microscope. The fatigue-crack growth rate,  $da/dN$ , was computed by dividing each increment of crack extension,  $\Delta a$ , by the number of cycles producing that increment,  $\Delta N$ .  $\Delta a$  generally ranged between 0.5 and 0.8 mm. The stress intensity factor range,  $\Delta K$ , associated with each increment was computed using the standard formula,<sup>30</sup> and was based on the average crack length for each increment. Crack growth rates were then plotted as a function of the stress intensity factor range.

## RESULTS AND DISCUSSION

### Type 304 Stainless Steel

Four specimens of annealed Type 304 which had not been previously aged were tested for comparison purposes. The results are given in Fig. 1 as a plot of  $\log(da/dN)$  vs  $\log(\Delta K)$ . Also shown are results for Type 304L which will be discussed later. Tests were conducted on annealed Type 304 which had been aged for 1500, 3000, and 6000 hours at temperatures of 1000°F (538°C) and 1200°F (649°C), and these results are shown in Figs. 2 and 3, respectively. It will be noted that, at the lower values of  $\Delta K$ , there is a tendency for the aged specimens to exhibit a lower crack growth rate than the unaged specimens and this tendency is somewhat more pronounced for the specimens aged at 1200°F than for those aged at 1000°F. There also appears to be a slight increase in the slope of the curve for the aged specimens, relative to the unaged specimens. In general, it appears that the results for either aging temperature are insensitive to aging time (the specimens aged 6000 hours at 1200°F appears to be an exception). In other words, the beneficial effects of thermal aging seem to occur fairly early in the aging process.

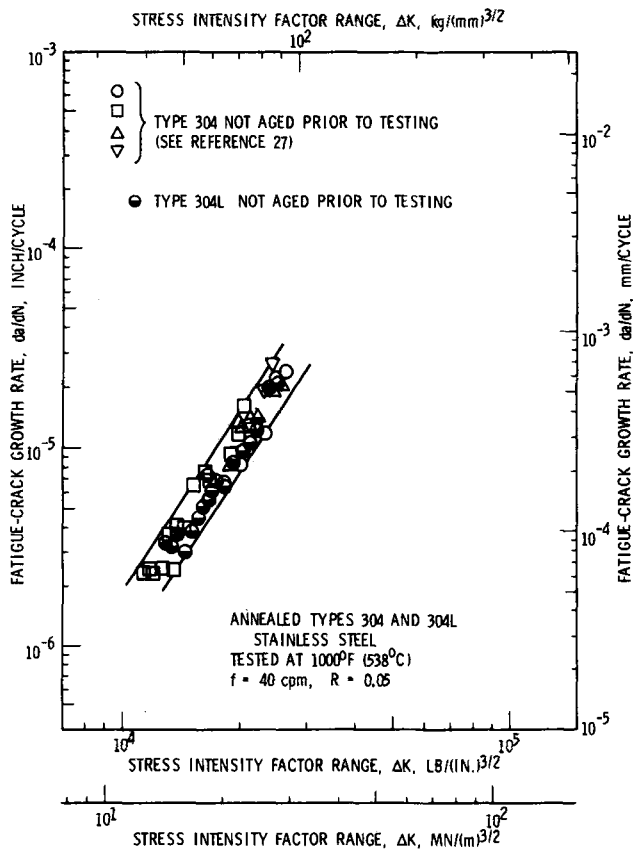


Fig. 1—Fatigue-crack propagation behavior of unaged, solution-annealed Types 304 and 304L; tested at 1000°F (538°C).

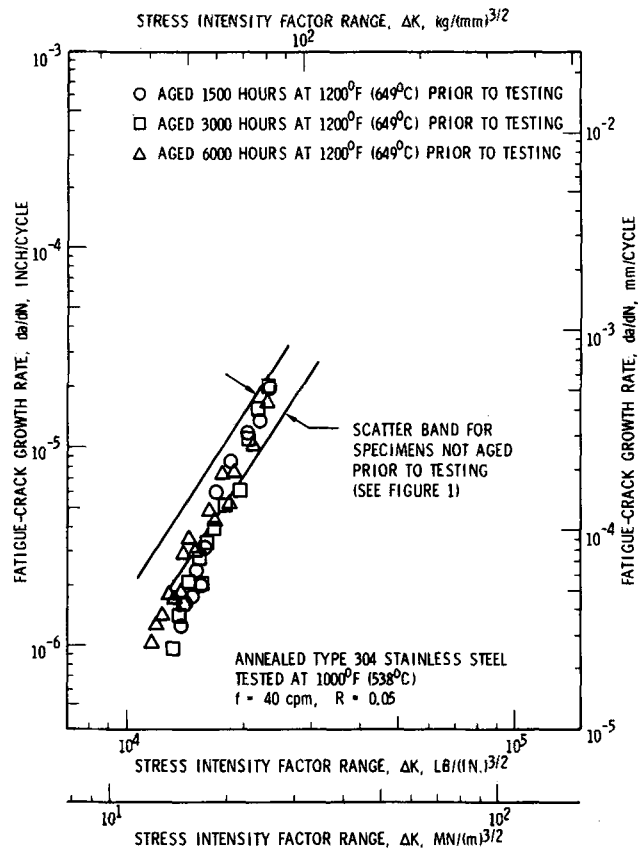


Fig. 3—Fatigue crack propagation behavior of solution-annealed Type 304, aged at 1200°F (649°C) and tested at 1000°F (538°C).

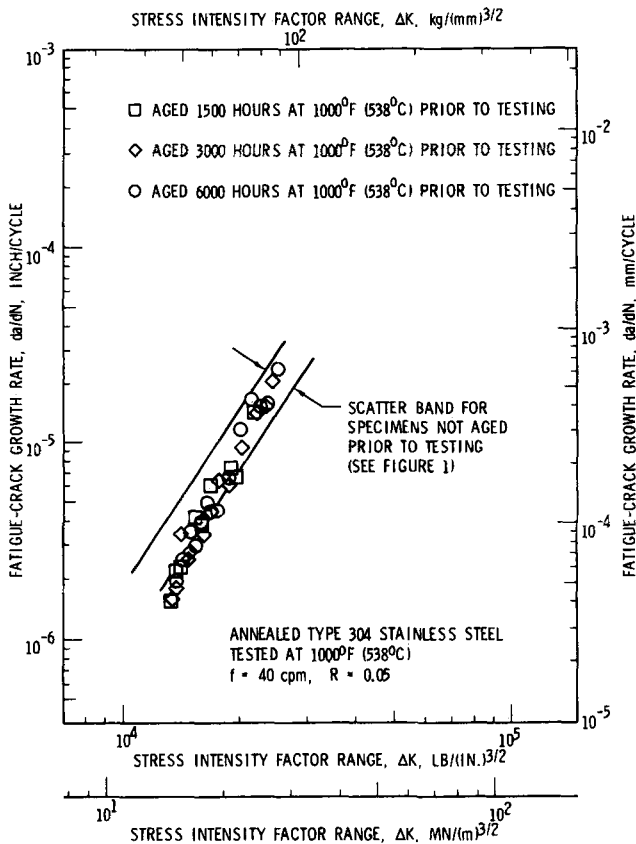


Fig. 2—Fatigue-crack propagation behavior of solution-annealed Type 304, aged at 1000°F (538°C) and tested at 1000°F (538°C).

The observation of improved fatigue properties (*i.e.*, lower crack growth rates and/or increased fatigue life) of austenitic stainless steels in the aged condition at elevated temperature is consistent with the observations of several other investigators.<sup>9-15</sup> Improvements in fatigue properties of austenitic stainless steels with thermal aging is, however, not a universal observation: James and Knecht,<sup>29</sup> tested Types 304 and 316 that had been aged 1150 h at 891°F (477°C) and observed no differences due to aging in tests conducted at 1000°F (538°C), Mahoney and Paton<sup>16</sup> observed no differences due to thermal aging in Type 316 aged 18 hours at 1300°F (705°C) for tests conducted at -321°F (-196°C), 77°F (25°C) and 1200°F (649°C), while Ueda and Yamane<sup>6</sup> and Barnby and Peace<sup>7,8</sup> have shown significantly reduced fatigue lives in aged material tested at room temperature.\*

\*The test temperature was not explicitly stated in References 6-8, but the assumption is that the tests were conducted at, or near, room temperature.

Barnby and Peace<sup>7,8</sup> have noted that fracture of the brittle carbide particles throughout the volume of the specimen preceded the initiation and propagation of the fatigue cracks, and Forsyth<sup>31</sup> obtained experimental evidence of a similar phenomenon in aged aluminum alloys. Pelloux<sup>32</sup> studied, at room temperature, two versions of an aluminum alloy with over an order of magnitude difference in the volume fraction of second-phase particles. He found a higher crack growth rate in the alloy with the higher volume fraction of particles. Pelloux attributed the higher rates to the brittle

second-phase particles within the plastic zone not being able to accommodate the large plastic strain amplitudes imposed by the surrounding matrix, thereby forming microcracks at each particle. When the number of particles within the yield zone became very large, macroscopic cracking would then proceed at a greater rate. Since the plastic zone diameter increases approximately in proportion to the square of the stress intensity factor, the process proposed by Pelloux would be more pronounced at the higher values of  $\Delta K$ . Similar findings were obtained by Lindley and Richards<sup>33</sup> who observed that, in ferritic steels at room temperature, brittle second-phase particles fractured to produce microcleavage and hence higher crack growth rates. They observed the tendency toward microcleavage increased with increasing  $\Delta K$ .

It may be significant to note that the tests of Ueda and Yamane<sup>6</sup> and Barnby and Peace<sup>7,8</sup> who observed decreased fatigue lives in aged austenitic stainless steel, as well as those of Forsyth,<sup>31</sup> Pelloux<sup>32</sup> and Lindley and Richards<sup>33</sup> who observed fracture of brittle second-phase particles, in other alloy systems, were all conducted at room temperature. It may be possible that at elevated temperatures, the strain compatibilities of the various phases and the matrix are sufficiently similar that a larger percentage of the particles may accommodate the plastic strains imposed upon them by the matrix without fracturing. This might help explain why the elevated temperature fatigue behavior of the austenitic stainless steels was not degraded by thermal aging. The presence of the particles may, in fact, actually improve the fatigue resistance as evidenced by the lower crack growth rates at low values of  $\Delta K$  in Figs. 2 and 3. It is possible that, as proposed by Glassman and McEvily,<sup>34</sup> the particles act as partial crack arrestors, and that a crack encountering a particle becomes partially blunted and must reinitiate before proceeding. A similar proposal was made by James<sup>28</sup> and Shahinian *et al.*<sup>35</sup> to explain the improvement in elevated temperature fatigue behavior of weldments in austenitic stainless steels relative to the base metal. They suggested that the fine duplex nature of the microstructure ( $\delta$ -ferrite and austenite) with its many particles and interfaces offered greater resistance to the extension of fatigue cracks.

Further support for the concept of crack-tip blunting is found in the results of Sumita *et al.*<sup>36</sup> They investigated the fatigue behavior of steel containing various amounts of  $Al_2O_3$  particles, and found that for specimens with a ferrite matrix the endurance limit, 1) increased with increasing particle content, 2) increased with decreasing particle size for a given area fraction, 3) increased with decreasing particle spacing, and 4) increased with a change in particle shape from angular to spherical. Results for specimens with a tempered martensite matrix were generally reversed from those given above. Sumita *et al.* attribute the beneficial effects noted for specimens with a ferrite matrix to crack tip blunting as the crack encounters  $Al_2O_3$  particles.

In order to verify that thermal aging improves the high temperature fatigue properties of the austenitic stainless steels, and degrades the lower temperature fatigue properties, a specimen aged for 3000 h at 1200°F (649°C) was tested at room temperature. The

fatigue behavior of both the aged and unaged specimens tested at room temperature is given in Fig. 4. It will be noted that, at the higher values of  $\Delta K$ , the crack does indeed propagate faster in the aged material. This is consistent with the observations of Pelloux<sup>32</sup> and Lindley and Richards<sup>33</sup> that fracture of the brittle constituent particles is more pronounced at the higher values of  $\Delta K$ . It should be noted that the phase distribution for room temperature tests is further complicated by a certain amount of deformation-induced transformation to  $\alpha$ -martensite in the plastic zone.<sup>37,38</sup> Still unanswered is the question: as the temperature is increased at what test temperature does thermal aging cease to be detrimental and start to be beneficial?

Many structural components operate at low cyclic frequencies, and the loads are often maintained at the maximum level for extended periods of time. The waveform and hold-time effects in annealed Type 304 have been examined in Ref. 23, and it was shown that at 1000°F (538°C), a "square" waveform at a frequency of 0.083 cpm ( $1.39 \times 10^{-3}$  Hz) produced cracking that was predominantly intergranular, and above that frequency cracking was predominantly transgranular. Because carbide precipitation occurs chiefly along the grain boundaries in the austenitic stainless steels,<sup>1,2</sup> one test was conducted at 1000°F at 0.083 cpm using a "square" waveform on material that had been aged 3000 h at 1200°F in order to evaluate the effect of aged material undergoing intergranular cracking. (The waveform used is described in Ref. 23.) The results, shown in Fig. 5, seem to indicate little or no effect of thermal aging under these conditions. It should

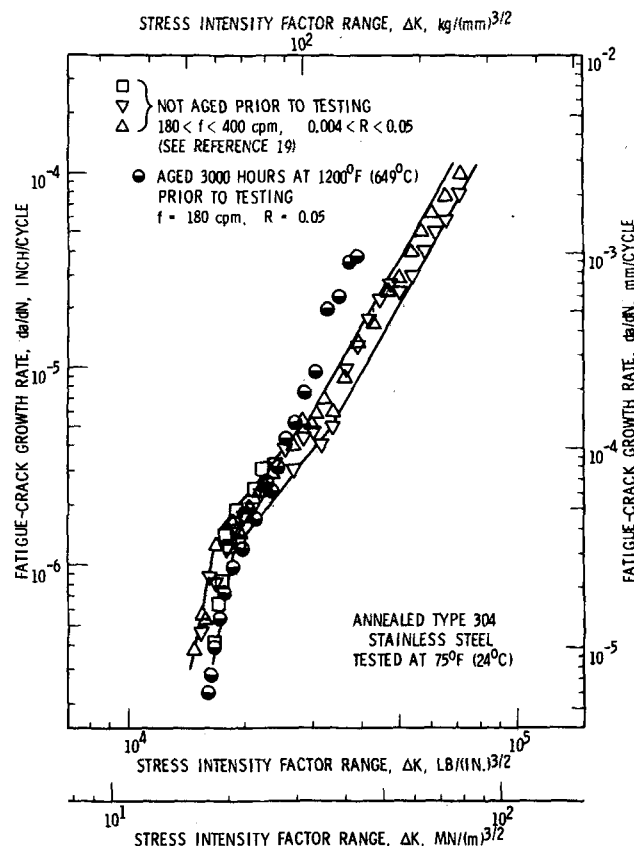


Fig. 4—Comparison of the fatigue-crack propagation behavior of aged and unaged solution-annealed Type 304 tested at 75°F (24°C).

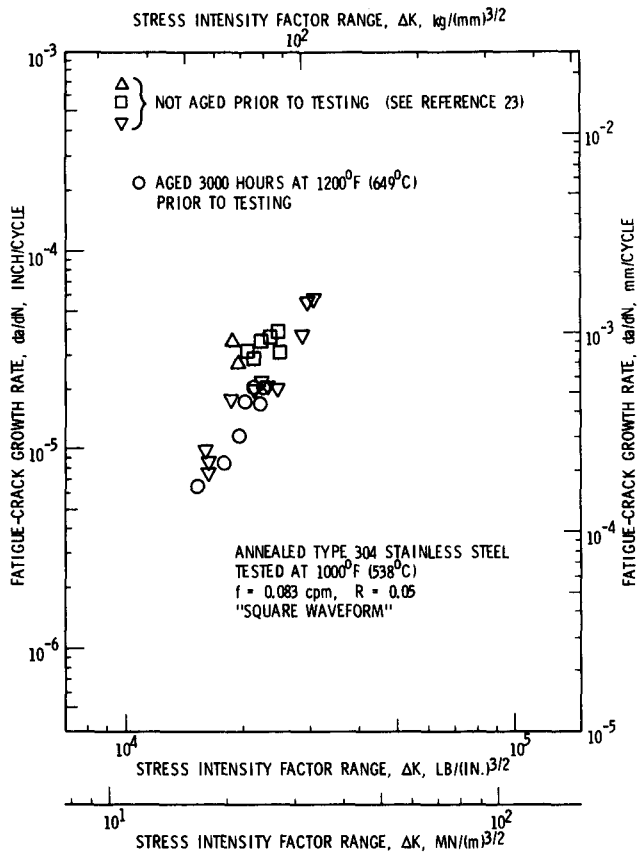


Fig. 5—Comparison of the fatigue-crack propagation behavior of aged and unaged solution-annealed Type 304 tested under conditions of intergranular cracking.

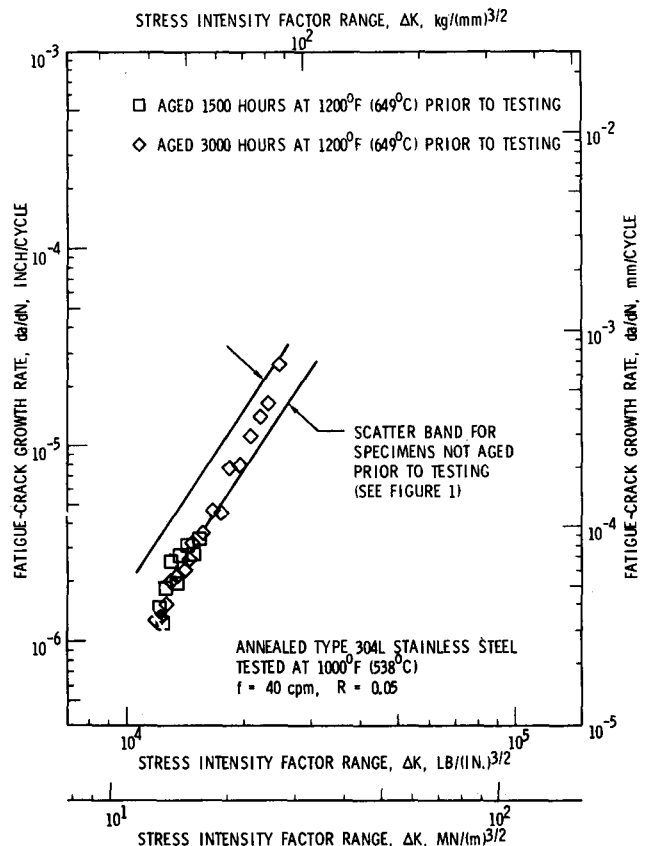


Fig. 6—Fatigue-crack propagation behavior of solution-annealed Type 304L, aged at 1200°F (649°C) and tested at 1000°F (538°C).

be noted, however, that because of the low frequencies these tests require a long time to complete, and therefore the "unaged" specimens are generally well aged by the completion of a test. For example one of the "unaged" specimens shown in Fig. 5 (designated with the symbol  $\nabla$ ) was tested over 3292 hours at 1000°F. Therefore, comparison of the aged specimen with one of the shorter durations unaged specimens (designated with the symbol  $\Delta$ , 379 total h of test time at 1000°F) suggests that aging might also be beneficial under conditions of intergranular cracking.

#### Type 304L Stainless Steel

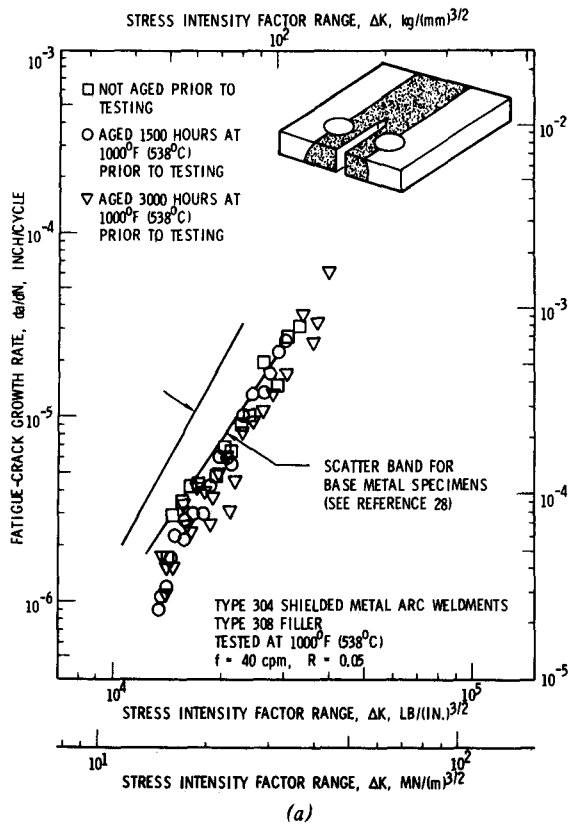
In order to evaluate the effect of carbon content on the elevated temperature crack propagation behavior of aged material, a limited number of Type 304L specimens were tested. The Type 304L (Heat V50035) used had a carbon content less than half that of the Type 304 utilized in Figs. 1 through 5 (Heat 55697). The behavior of unaged Type 304L is shown in Fig. 1, and it will be noted that there is no observable difference between the behavior of Type 304 and 304L. The results for aged Type 304L are given in Fig. 6. It will be noted that there appears to be a slight beneficial effect of thermal aging, but comparing equivalent aging times in Figs. 3 and 6, it appears that the beneficial effects (*i.e.*, lower growth rates) are more pronounced for the material with the higher carbon content. The implication is, of course, that the greater carbon content, resulting in the precipitation of more carbides, is responsible for the slight improvement in fatigue behavior.

#### Type 304 Stainless Steel Weldments

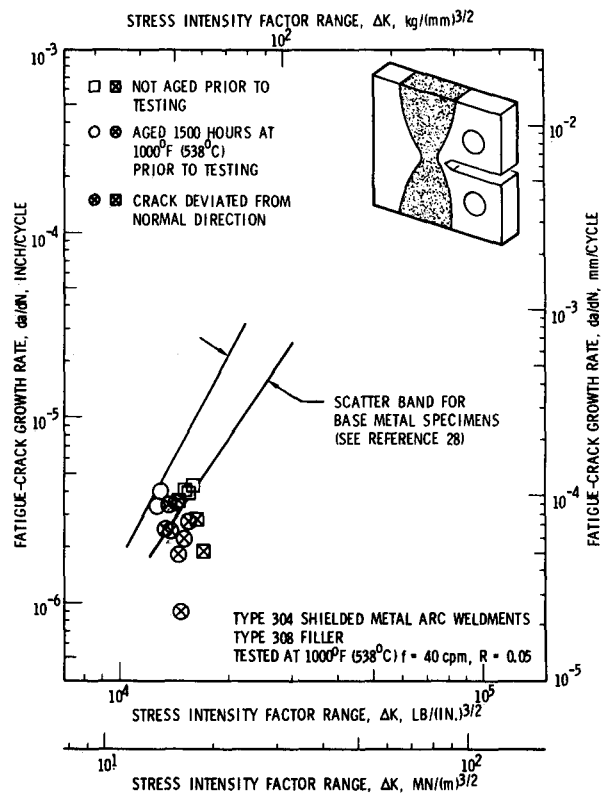
Most large engineering structures use welding as one of the principle joining methods and, despite precautions, imperfections in the weldments often act as initiation sites for fatigue cracks. For this reason, a survey was made of the effects of thermal aging upon the crack growth behavior of shielded-metal-arc weldments in Type 304 (Type 308 filler). Specimens representing three orientations of the crack, relative to the weld, were aged at 1000°F (538°C), and the results are shown in Figs. 7(a) through 7(c). The scatter for the weldment specimens appears to be somewhat greater than that for base metal specimens, and this has also been observed in previous studies on weldments.<sup>28,35</sup> Figs. 7(a) and 7(b) suggest that there is a very slight beneficial effect of thermal aging, but the effect is not great and is almost within the scatter of the data. Fig. 7(c) yields almost no definitive information. Severe crack branching and/or deviations in crack direction have been noted previously<sup>28</sup> in weldment specimens with this particular crack orientation. The data obtained under conditions of crack branching are shown, but are not considered valid.

#### Solution-Annealed Type 316 Stainless Steel

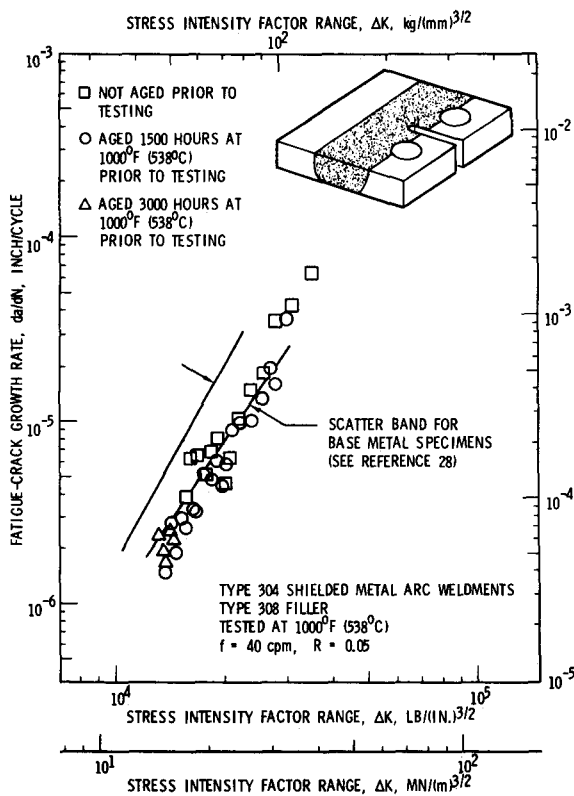
Three specimens which had not been previously aged were tested, and the results are shown in Fig. 8. The results for specimens which were aged at 1000°F (538°C) and 1200°F (649°C) are given in Figs. 9 and 10, respectively. Comparison of Figs. 8 through 10 shows that thermal aging tends to lower the rate of crack propaga-



(a)



(c)



(b)

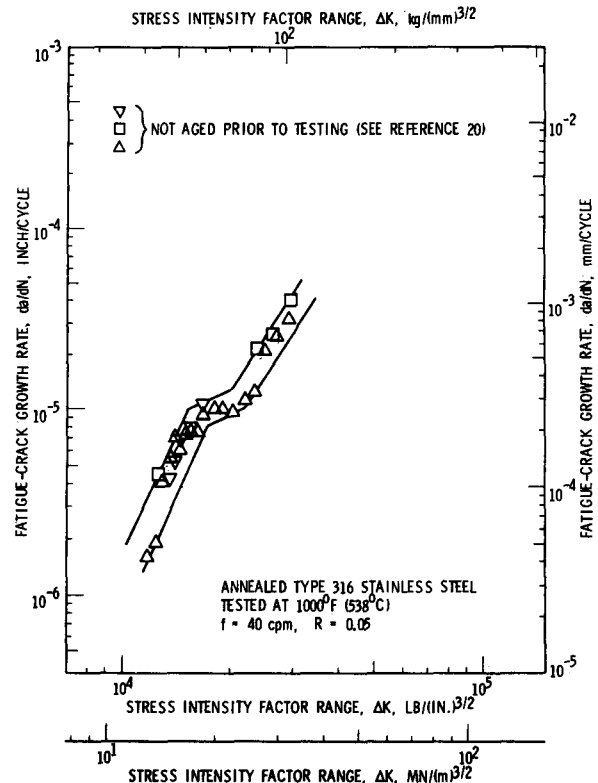


Fig. 8—Fatigue-crack propagation behavior of unaged, solution-annealed Type 316, tested at 1000°F (538°C).

Fig. 7—(a) Comparison of the fatigue-crack propagation behavior of aged and unaged Type 304 shielded-metal-arc weldments tested at 1000°F (538°C). (b) Comparison of the fatigue-crack propagation behavior of aged and unaged Type 304 shielded-metal-arc weldments tested at 1000°F (538°C). (c) Comparison of the fatigue-crack propagation behavior of aged and unaged Type 304 shielded-metal-arc weldments tested at 1000°F (538°C).

differences in behavior due to thermal aging are probably due to differences in other constituents, and this implies that the intermetallic phases may also influence the behavior.

### 20 Pct Cold Worked Type 316 Stainless Steel

The results for the specimens aged prior to testing are shown in Fig. 11, and those for specimens aged at 1000°F (538°C) are given in Fig. 12. Both Figs. 11 and 12 contain data for two crack orientations, \* TL and LT,

\*See Ref. 30 for the ASTM crack orientation system.

and since crack orientation can affect crack propagation in this material,<sup>21</sup> this accounts for the somewhat larger scatter bands. Comparison of Figs. 11 and 12 reveals little or not effect of thermal aging, although the data for the aged material does tend toward the lower bound of the scatter band. Cold working of Type 316 encourages the early precipitation of intermetallic phases upon subsequent aging.<sup>38</sup>

### SUMMARY AND CONCLUSIONS

The results of this study may be summarized as follows:

With the exception of 20 pct cold worked Type 316 where no effect was observed, thermal aging produced a beneficial effect (*i.e.*, lower crack growth rates) for all of the elevated temperature tests conducted on the austenitic stainless steels in this study. The precipitation of various carbides and intermetallic phases is

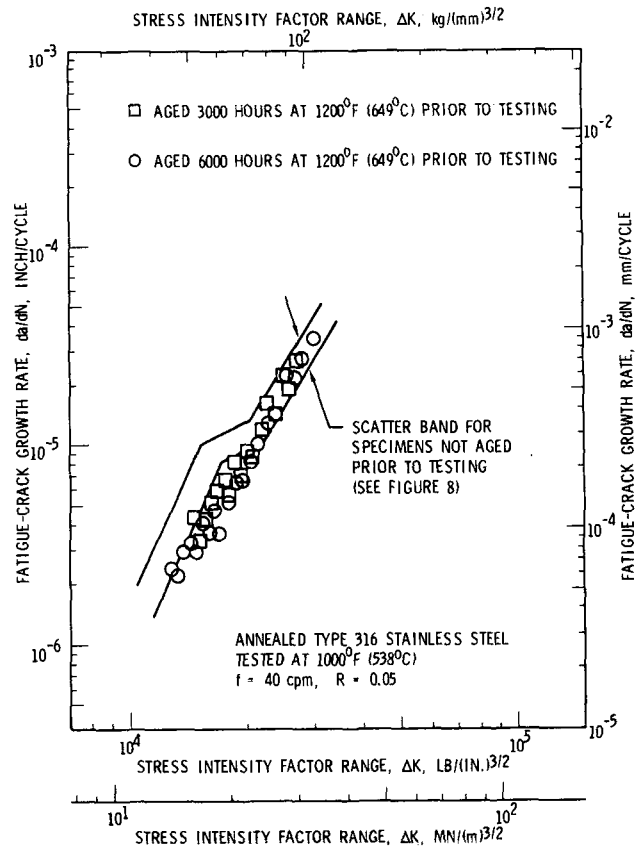


Fig. 10—Fatigue-crack propagation behavior of solution-annealed Type 316, aged at 1200°F (649°C) and tested at 1000°F (538°C).

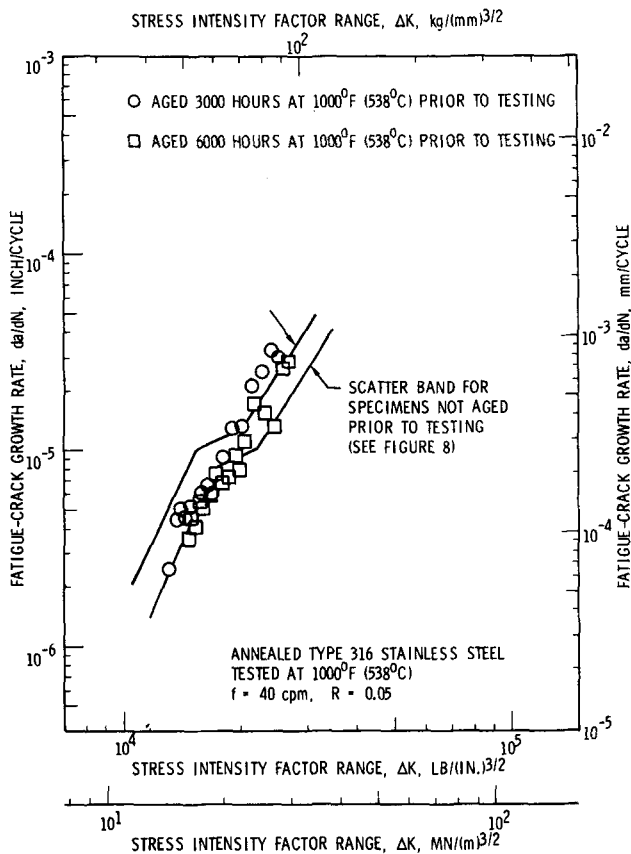


Fig. 9—Fatigue-crack propagation behavior of solution-annealed Type 316, aged at 1000°F (538°C) and tested at 1000°F (538°C).

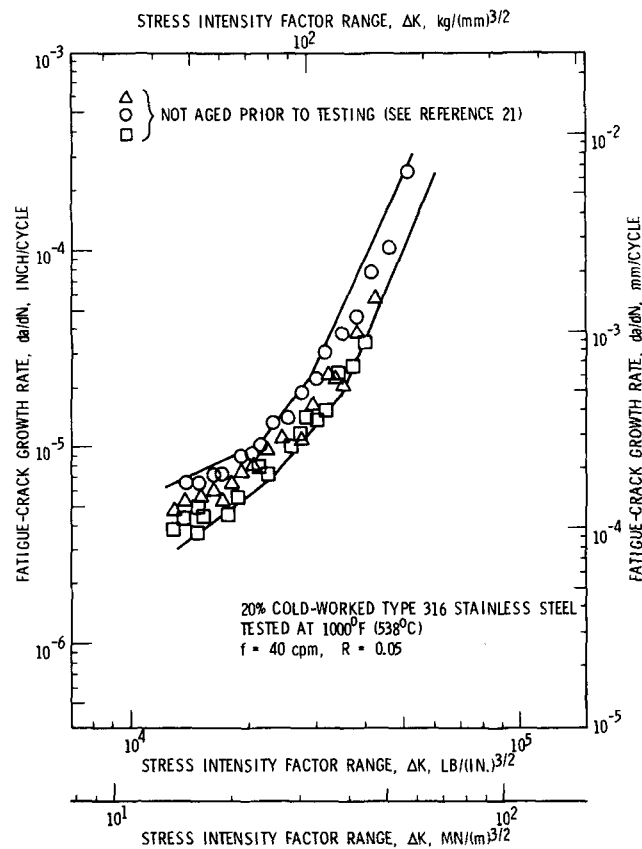


Fig. 11—Fatigue-crack propagation behavior of unaged, 20 pct cold-worked Type 316, tested at 1000°F (538°C).

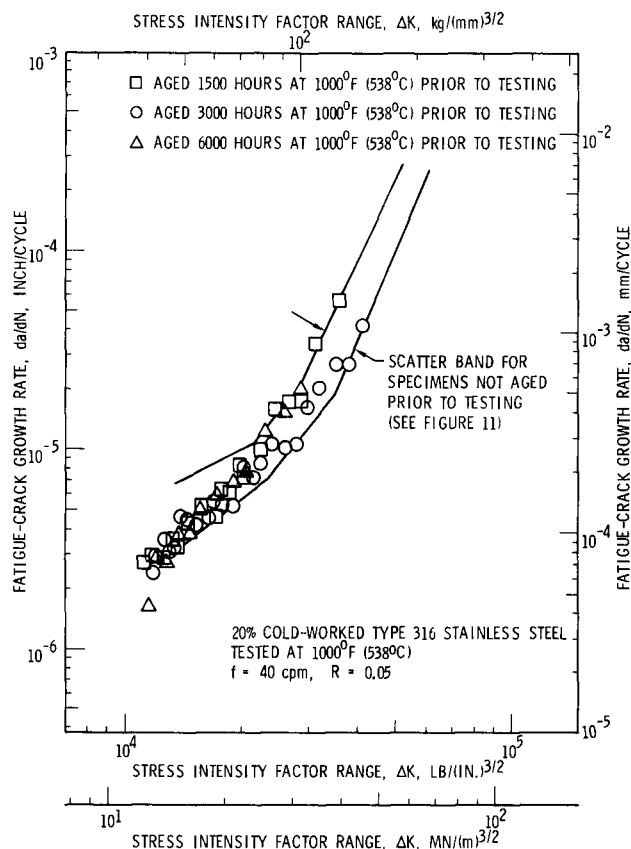


Fig. 12—Fatigue-crack propagation behavior of 20 pct cold-worked Type 316, aged at 1000°F (538°C) and tested at 1000°F (538°C).

believed responsible for this improvement. One of the possible mechanisms might be a blunting effect each time the crack tip encounters a particle, thereby requiring the crack partially reinitiate itself before proceeding.

The beneficial effect was more pronounced in Type 304 than in Type 304L, implying that carbon content is an important variable.

In both annealed Types 304 and 316, aging at 1200°F produced slightly lower crack growth rates than aging at 1000°F. The time at a given aging temperature did not seem to be as important as the temperature itself.

Carbide precipitation occurs chiefly at the grain boundaries. However, in a test conducted on annealed Type 304 under conditions producing an intergranular mode of crack extension (*i.e.*, elevated temperature, low-frequency "square" waveform), thermal aging did not appear to affect the fatigue-crack growth rate.

In a test conducted at room temperature on aged, annealed Type 304, the crack growth rate was markedly higher at the larger values of  $\Delta K$ . Other investigators have noted similar behavior in room temperature tests on other alloy systems, and have proposed that the brittle second-phase particles are unable to accommodate the large plastic strain amplitudes imposed by the more ductile matrix. The particles therefore fracture and initiate microcracks, and this accelerates the macrocracking process. The elevated temperatures results of this investigation imply that this process does not operate at the higher temperatures because the matrix and constituent particles are more compatible. Still unanswered is the question: at what test

temperature does thermal aging cease to be detrimental and start to be beneficial?

## ACKNOWLEDGMENTS

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