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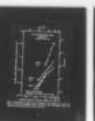
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NRL Memorandum Report 3936

Effect of Neutron Irradiation on Fatigue Crack Propagation in Type 316 Stainless Steel at 649°C

D. J. MICHEL AND H. H. SMITH

Thermostructural Materials Branch
Material Science and Technology Division

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20. Abstract (Continued)

steel during both continuous cycling and one minute hold time tests. Scanning electron microscopy (SEM) examination of the fracture surfaces of the unirradiated fatigue specimens revealed that intergranular carbide precipitation during testing at 649°C acted to delay the transition to a purely intergranular failure mode with hold time as previously observed at 593°C.

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The effect of fast neutron irradiation on the fatigue crack propagation performance of Type 304 stainless steel was investigated at 649°C and compared with previous results at 593°C. The results show that during continuous cycling no significant effect of irradiation on crack propagation rate was observed. The inclusion of a one minute hold time during the test, however, resulted in a significant increase in the crack propagation rate. The inclusion of a one minute hold time during the test, however, resulted in a significant increase in the crack propagation rate. The inclusion of a one minute hold time during the test, however, resulted in a significant increase in the crack propagation rate.

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EFFECT OF NEUTRON IRRADIATION ON FATIGUE CRACK PROPAGATION IN TYPE 316 STAINLESS STEEL AT 649°C

INTRODUCTION

A knowledge of the effects of fast neutron irradiation on the performance of reactor structural materials is becoming increasingly important as advanced reactor systems are developed. Since it is recognized that the structural components of these systems will be subjected to cyclic loads at sustained elevated temperatures, the effect of fast neutron irradiation on the fatigue crack propagation performance of the structural materials must be understood.

The available experimental results concerning the effects of fast neutron irradiation on the fatigue crack propagation performance of the austenitic stainless steels have been recently reviewed (1,2). These results at 593°C show that, for neutron fluences up to 5×10^{22} n/cm² (>0.1 MeV), irradiation produced a small increase in crack propagation rate in annealed stainless steel when compared to unirradiated tests. For cold worked stainless steels, neutron irradiation to the same fluence level was found to produce a substantial increase in crack propagation rates when compared to unirradiated material tested at 593°C. Previous results (3) also have shown that the effect of tensile hold time at 593°C in annealed, neutron irradiated Type 316 steel was to produce a small but significant increase in fatigue crack propagation rate when compared with the hold time results for unirradiated material. For 20 percent cold worked material, the results show a significant increase in crack propagation rate with increased hold time in excess of the increase observed in the unirradiated hold time tests.

The purpose of this report is to present further results concerning the effects of fast neutron irradiation and hold time on fatigue crack propagation performance of Type 316 stainless steel irradiated and tested at 649°C (1200°F). These results are compared with the 593°C results previously reported (1,2) to develop additional insight concerning the effects of irradiation and hold time on the fatigue behavior of Type 316 steel.

EXPERIMENTAL PROCEDURES

The chemical composition of the Type 316 stainless steel plate and the test specimen dimensions have been previously reported (3,4). Single-edge-

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notched (SEN) cantilever specimens and IT compact tension (CT) specimens were prepared from the plate material such that the plane of crack propagation was perpendicular to the plate rolling direction. For neutron irradiation purposes, SEN center sections, 3.81 cm by 6.35 cm were irradiated in an NRL controlled temperature subassembly at 649°C in the Experimental Breeder Reactor No. II (EBR-II) to calculated fast neutron fluences from 1.2 to 1.4 x 10²² n/cm² (>0.1 MeV). After irradiation, end tabs were welded in-cell to the center sections to produce the final specimen configuration. Similar welding procedures were employed to prepare the unirradiated SEN specimens. Previous experimental work has shown that crack propagation behavior in similarly welded specimens was the same as that in non-welded specimens (5). The unirradiated CT specimens were prepared in accordance with ASTM Standard E-399 specifications.

Complete details of the test and data analysis procedures have been reported (1,3,6). Briefly, all tests were conducted using a zero-to-tension loading cycle at 0.17 Hz (10 cpm) to a constant maximum load in air. Hold time effects were investigated for certain tests by maintaining the maximum tensile load constant for 1.0 minute during each cycle. Induction heating was employed to achieve a test temperature of 649°C with control maintained to within +3°C. Crack lengths were measured periodically using a remote high-resolution television system for the in-cell tests and using a traveling microscope for the out-of-the cell tests. The experimental data were analyzed to yield crack propagation rate, da/dN, as a function of the stress intensity factor range, ΔK, according to the relationship

$$da/dN = C (\Delta K)^m,$$

where C and m are constants dependent on test parameters, the material, and environmental factors. It is well established that this relationship describes the fatigue crack propagation behavior of metals at room and elevated temperatures under a wide variety of experimental conditions.

RESULTS AND DISCUSSION

The effects of neutron irradiation and hold time on the fatigue crack propagation behavior of annealed and cold worked Type 316 stainless steel at 649°C are presented in Figs. 1 through 4 together with previous results obtained at 593°C (3,4). For the continuous cycling (zero hold time) test condition, Fig. 1 shows that irradiation at 649°C produced no significant effect on the crack propagation rate in annealed material when compared with results at 593°C. However, for material in the cold worked condition, Fig. 2, the crack propagation rate was further increased by irradiation at 649°C over that observed in the irradiated, cold worked material at 593°C. On the other hand,

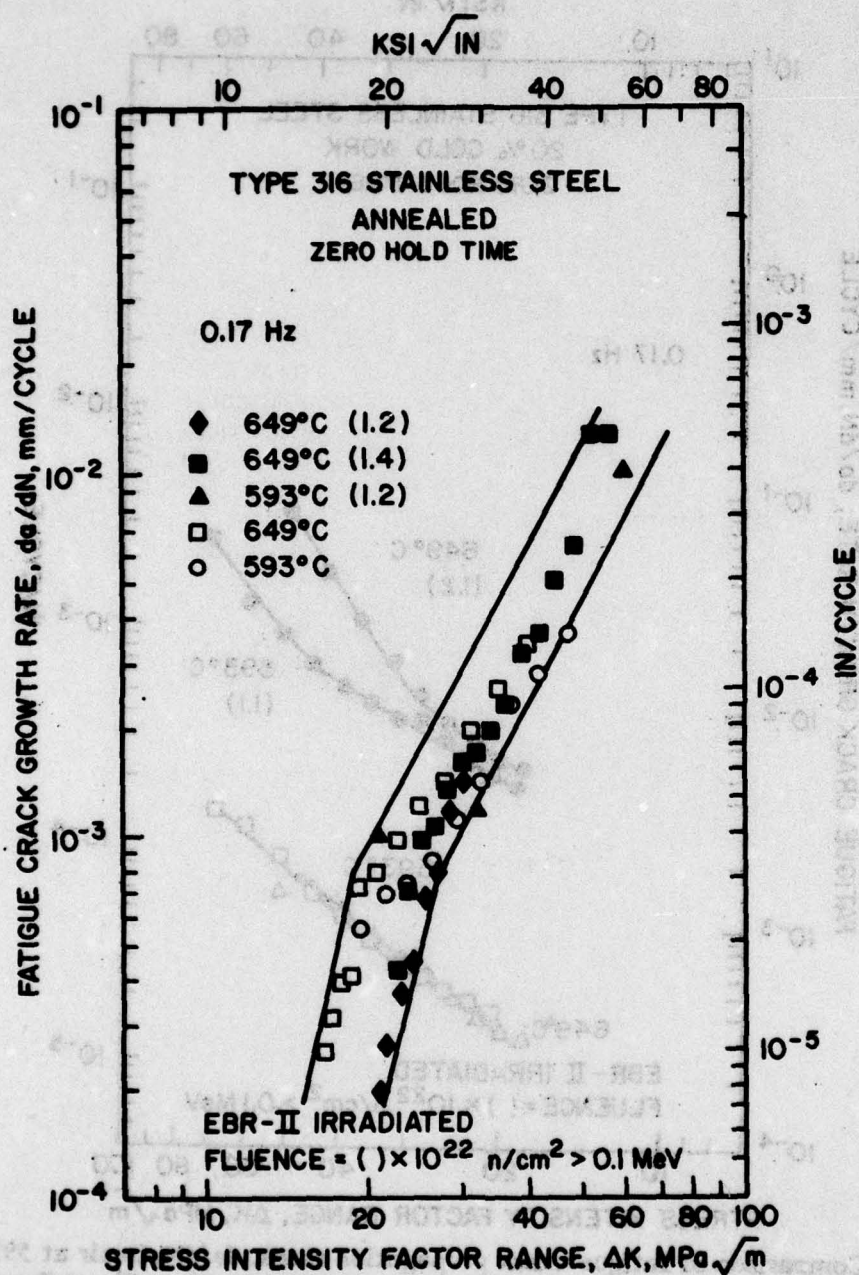


Fig. 1. Comparison of fatigue crack rates, da/dN , in air at 593 and 649°C for solution annealed, EBR-II irradiated and unirradiated Type 316 stainless steel tested for continuous cyclic loading at 0.17 Hz (10 cpm).

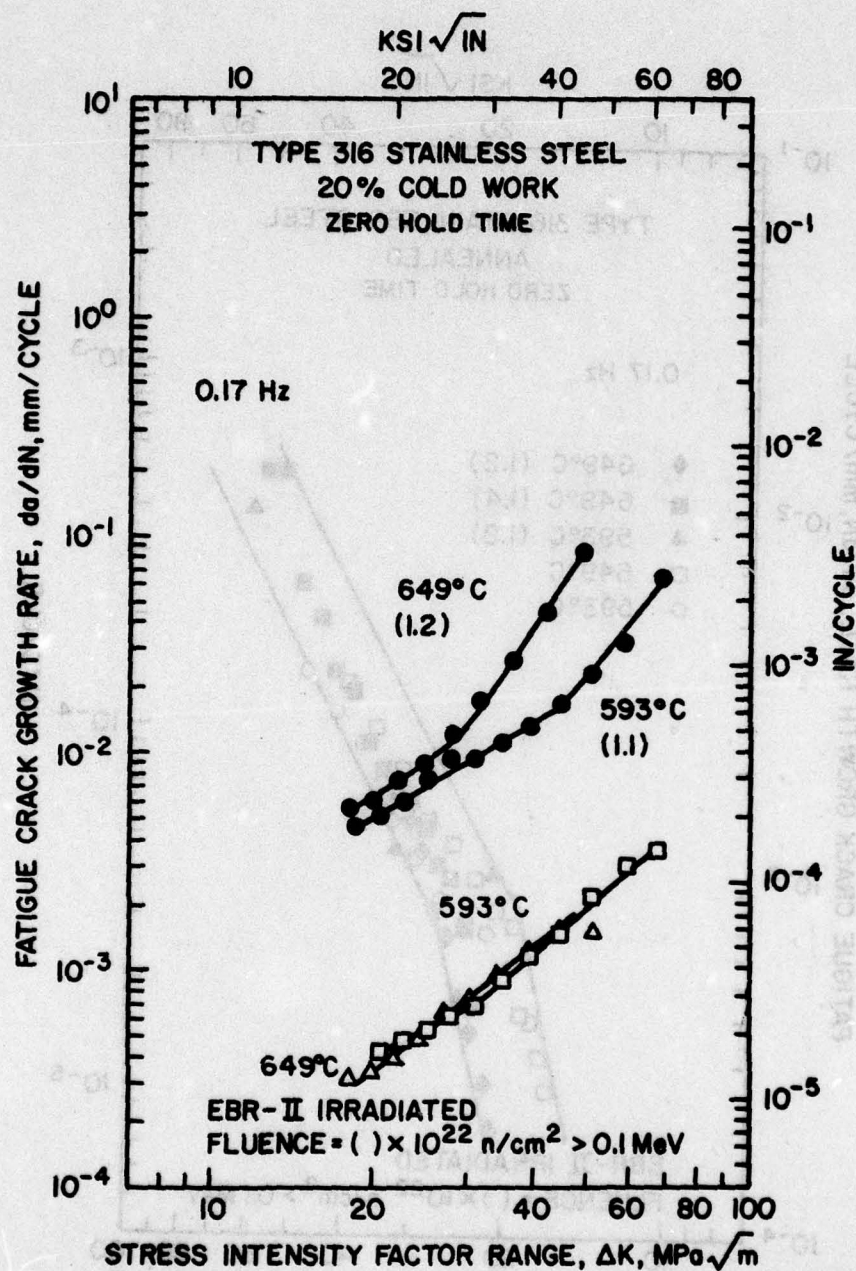


Fig. 2. Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for 20 percent cold worked, EBR-II irradiated and unirradiated Type 316 stainless steel tested with continuous cyclic loading at 0.17 Hz (10 cpm).

the unirradiated crack propagation rates for the cold worked material were nearly identical at both 649 and 593°C.

The results for the effect of a one minute hold time in the annealed and cold worked material at 649°C are shown in Figs. 3 and 4 and suggest a marked dependence of crack propagation rate on temperature. In Fig. 3, the crack propagation rate of the annealed material was greatly increased at 649°C when compared with the results at 593°C. Furthermore, it is important to note that the slope of both the unirradiated and irradiated da/dN vs ΔK results at 649°C are significantly higher than for the results at 593°C. This change in slope suggests that the failure mechanism in the annealed material at 649°C during hold time tests may have been different than at 593°C. For the cold worked material, Fig. 4, the inclusion of a one minute hold time also substantially increased the crack propagation rate in the irradiated condition at 649°C when compared with the irradiated results at 593°C. The significant effect of hold time on the cold worked 316 steel can be seen by comparison of Figs. 2 and 4. In particular, the large effect of hold time observed in the irradiated material at 649°C suggests that the effects of hold time and irradiation on crack propagation rate do not saturate up to this temperature and may increase further at higher temperatures. This point is of considerable interest from the reactor design viewpoint since component lifetimes could be severely limited where hold times are expected to be encountered.

Previous fractographic examinations of failure mode in unirradiated, annealed and cold worked Type 316 stainless steel, fatigue tested at 593°C, revealed that cracks propagated transgranularly for zero hold time tests and intergranularly for 1 min hold time tests (4). The results for the unirradiated annealed steel are shown in Figs. 5a and 5b for comparison with the scanning electron microscope (SEM) examinations of fracture surfaces formed during fatigue crack propagation in the unirradiated material tested at 649°C, Figs. 5c and 5d. It can be seen that for zero hold time test conditions the crack propagation mode was primarily transgranular at 649°C, Fig. 5c. However, in contrast to the behavior at 593°C, an intergranular failure mode was not observed at ΔK 's below 30 MPa $\sqrt{m}$ for 1 min hold time tests at 649°C, Fig. 5d. At ΔK levels above this value, the crack propagated by a mixture of transgranular and intergranular fracture, but the degree of intergranular failure did not approach that seen at 593°C, Fig. 5b. In the cold worked condition, the failure mode at 649°C also was primarily transgranular or mixed for zero and 1 min hold time tests, respectively.

It is interesting to note that many features of the fracture surfaces of the unirradiated, annealed specimens at 649°C resemble those of thermally aged, annealed Type 316 stainless steel tested at 593°C (4). SEM examinations of polished and etched sections taken from unirradiated specimens tested at 649°C were conducted to determine whether the thermally enhanced precipitation kinetics at 649°C may have resulted in the formation of a partially aged microstructure during the time of test. Recent metallographic studies by the authors of unaged Type 316 stainless steel have indicated that little or no carbide precipitation occurs at 593°C during testing. However, Fig. 6a shows the microstructure of the annealed Type 316 stainless steel after a 50-hour,

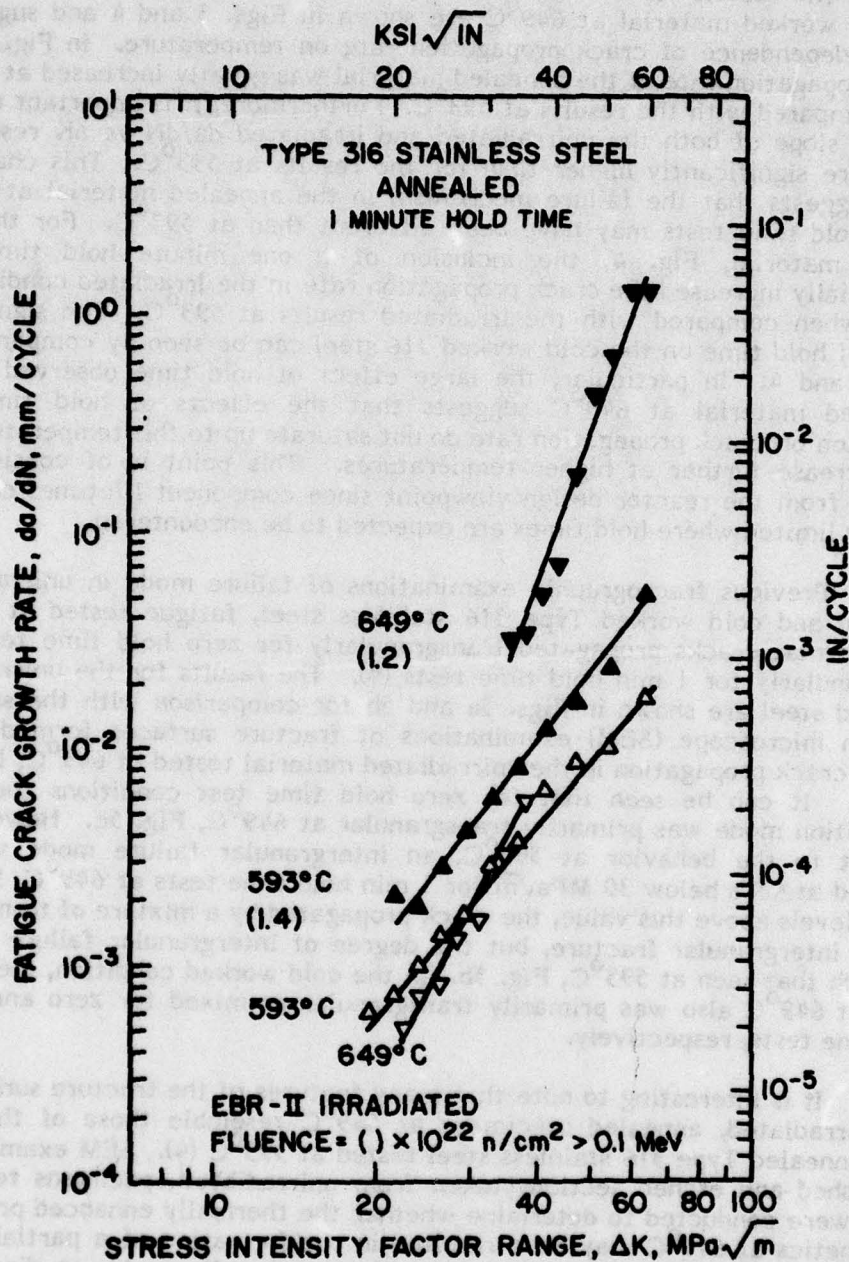


Fig. 3. Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for solution-annealed, EBR-II irradiated and unirradiated Type 316 stainless steel tested with one minute hold time.

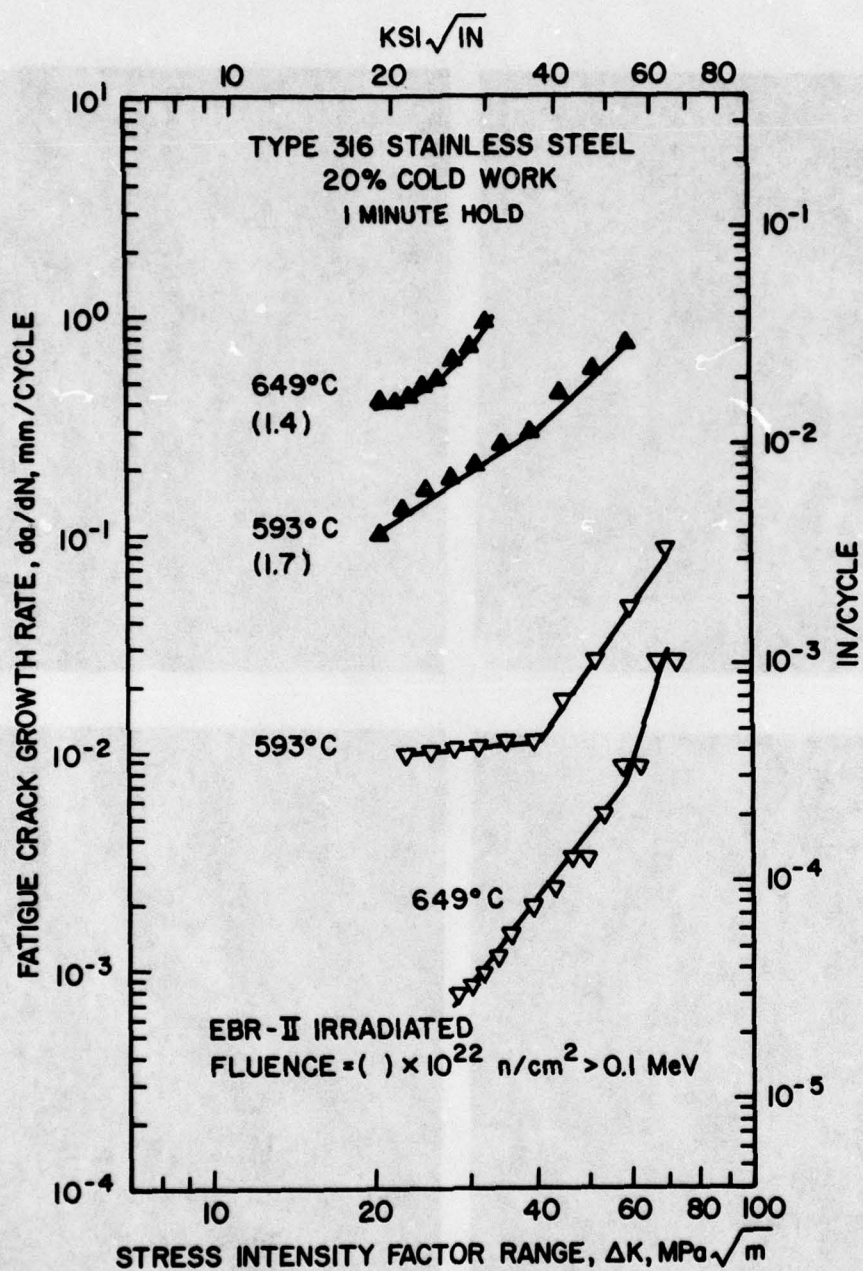


Fig. 4. Comparison of fatigue crack propagation rates, da/dN , in air at 593 and 649°C for 20 percent cold worked, EBR-II irradiated and unirradiated Type 316 stainless steel tested with one minute hold time.

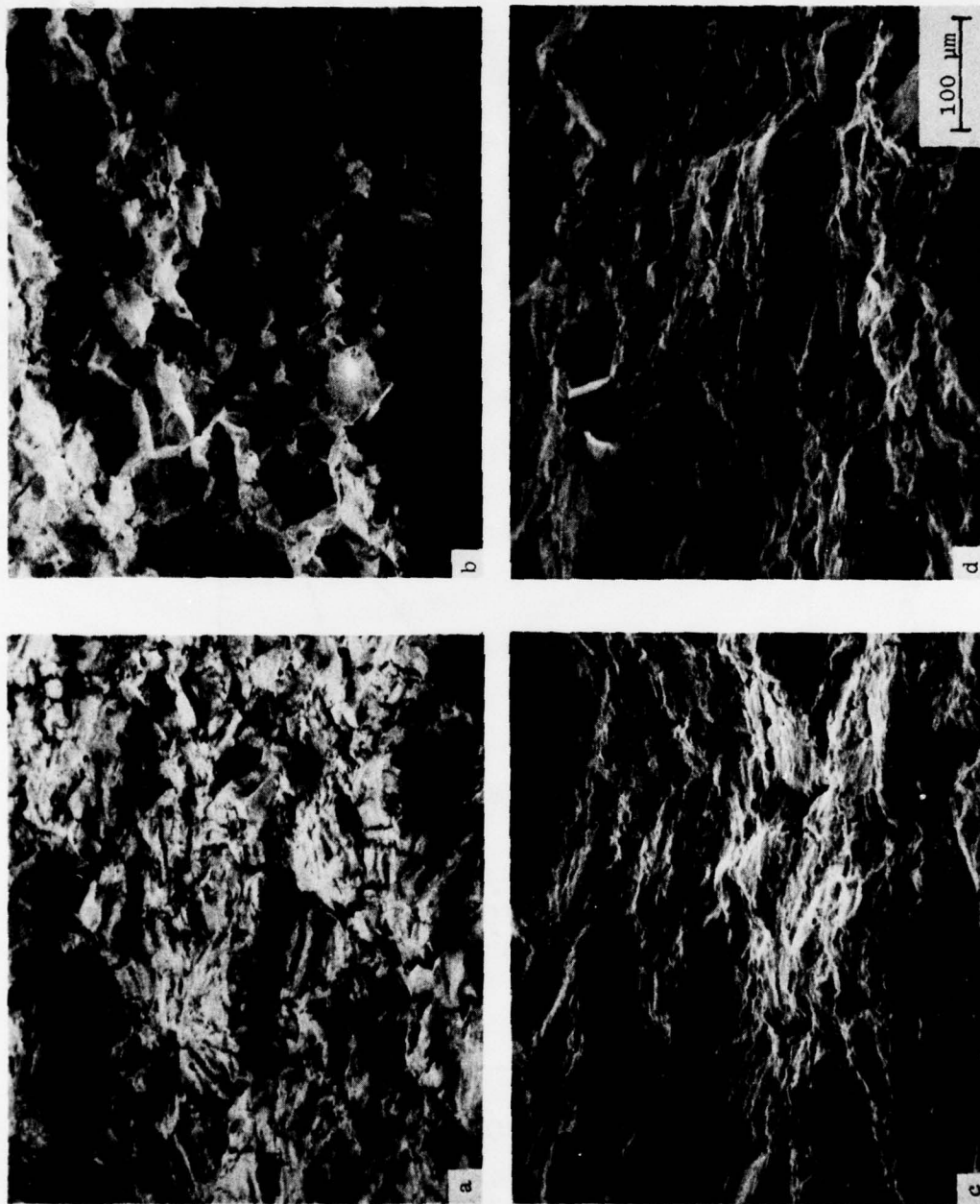


Fig. 5 — SEM fractographs of FCP specimens of annealed Type 316 stainless steel: (a) Zero hold time at 593°C, (b) One minute hold time at 593°C, (c) Zero hold time at 649°C, (d) One minute hold time at 649°C. Crack propagation direction is from left to right.

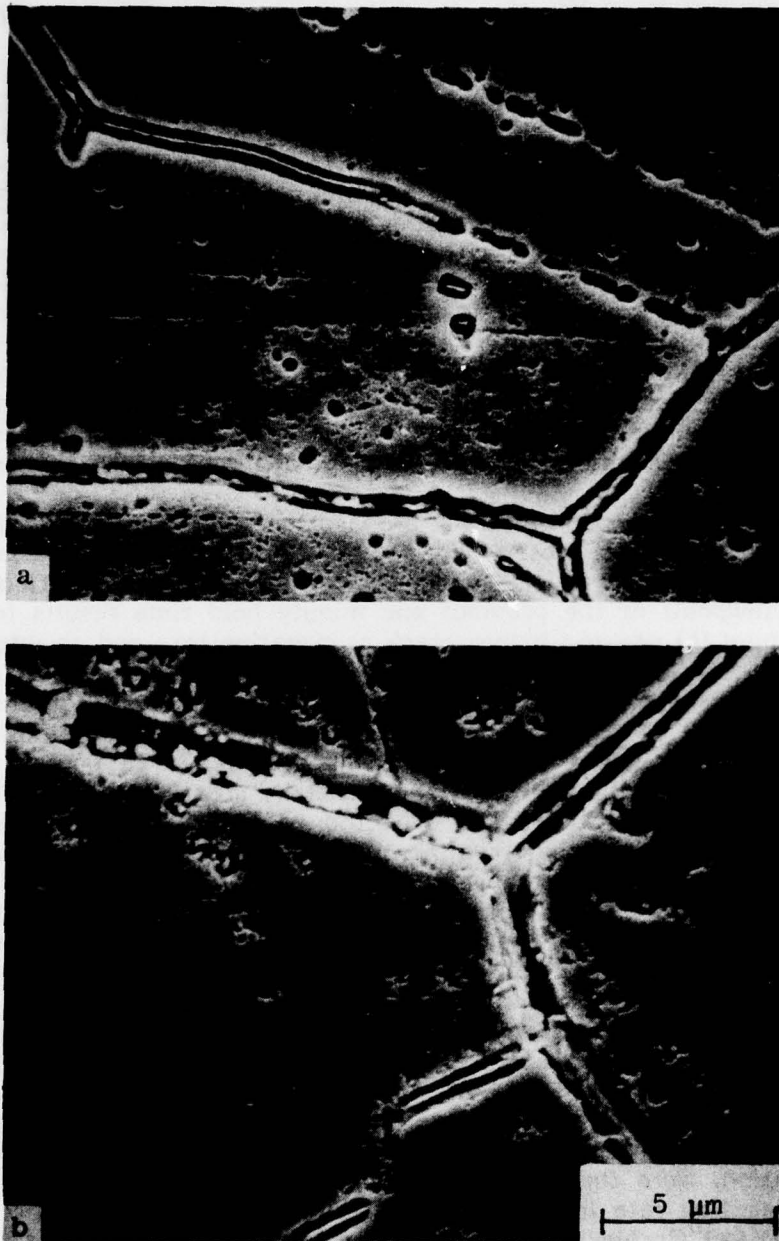


Fig. 6 — SEM micrographs of polished and etched annealed Type 316 stainless steel: (a) Unaged tested at 649°C (zero hold time), (b) Aged 5000 hr at 593°C, tested at 593°C (one minute hold time).

Areas surrounding carbides are preferentially etched because of lower corrosion resistance, thus revealing individual and conglomerated chromium carbide particles in grain boundaries and matrix.

zero hold time test at 649°C. It is evident that considerable precipitation has taken place, and when compared with material thermally aged for 5000 hours at 593°C and tested at 593°C, Fig. 6b, it is seen that precipitates were slightly smaller at 649°C but nearly as profuse.

Previous thermal aging studies (4) on unirradiated Type 316 stainless steel have shown that aging at 593°C reduced the effects of hold time and delayed the transition from a transgranular to an intergranular failure mode to hold times longer than one minute. The similar behavior seen in the present study for the unaged and unirradiated, annealed and cold worked Type 316 stainless steel tested at 649°C with a one minute hold time suggests that the carbide precipitation which occurred during testing was responsible for the reduction in crack propagation rates to levels equivalent to those in aged material at 593°C. Furthermore, these results indicate that, despite the higher test temperature, the more rapid carbide precipitation kinetics reported for Type 316 stainless steel at temperatures above 600°C (7,8) were responsible for maintaining a transgranular or mixed failure mode throughout the 649°C one minute hold time tests despite the higher slope of the da/dN vs ΔK curves.

Both the reduction in crack propagation rate in unirradiated material at 649°C to the level observed at 593°C and the maintenance of the transgranular or mixed failure mode are considered to be beneficial effects from the viewpoint of the previous one minute hold time results for unaged Type 316 steel (4) where a higher crack propagation rate and an intergranular failure mode was observed at 593°C. From the standpoint of structural applications, the unirradiated test results and metallographic observations at both 593 and 649°C suggest that thermal aging of Type 316 stainless steel, intended for unirradiated service at temperatures up to 650°C, could be employed to suppress the transition to potentially unstable intergranular crack propagation when hold times are expected to be encountered. Studies to specifically explore such heat treatments will be discussed in a future report. It is noted, however, that the reduction in crack propagation rate found at 649°C for the unirradiated material was not observed for the irradiated material, regardless of prior thermomechanical history, when tested with a one minute hold time.

Based on previous work at 593°C concerning hold time effects in irradiated 316 stainless steel (3) and recent studies concerning the deformation mechanism responsible for the hold time effects in unirradiated 316 stainless steel (4), the present results suggest that thermally activated processes, possibly related to grain boundary cavity formation and growth and to precipitate formation, were the dominant mechanisms which produce the increased crack propagation rate in the irradiated specimens tested with a one minute hold time at 649°C. Nevertheless, until the ongoing examination of the failure mode and the carbide precipitation in the irradiated specimens has been completed, it will not be possible to determine the contribution of these mechanisms to the crack propagation behavior. Separate experiments designed to investigate the synergistic relationship of these mechanisms also are in progress.

SUMMARY AND CONCLUSIONS

The effects of neutron irradiation at 649°C on the fatigue crack propagation performance of annealed and 20 percent cold worked Type 316 stainless steel were investigated for zero and one minute hold time at 649°C. The following conclusions are drawn from the results:

1. Neutron irradiation at 649°C was found to have no significant effect on fatigue crack propagation rate during continuous cycling in annealed Type 316 stainless steel when compared with unirradiated tests at this temperature.
2. The inclusion of a one minute tensile hold period significantly increased the crack propagation rates in annealed Type 316 stainless steel neutron irradiated and tested at 649°C when compared to both unirradiated results at 649 and 593°C and neutron irradiated results at 593°C.
3. For 20 percent cold worked Type 316 stainless steel, neutron irradiation at 649°C was found to increase crack propagation rate during both continuous cycling and one minute hold time tests when compared to results at 593°C.
4. In the unirradiated, annealed and cold worked one minute hold time tests at 649°C, the reduced crack propagation rate, relative to that at 593°C, was accompanied by a purely transgranular failure mode at ΔK levels less than 30 MPa $\sqrt{m}$ and a significantly higher da/dN vs ΔK slope. It is concluded that the carbide precipitation which occurred during testing acted to delay the transition to a mixed intergranular-transgranular failure mode at a ΔK of 30 MPa $\sqrt{m}$ and to reduce the crack propagation rate.
5. The increased crack propagation rates at 649°C in the neutron irradiated, 20 percent cold worked Type 316 stainless steel with hold time are suggested to result from thermally activated, processes such as those concerned with grain boundary cavity formation and growth and with precipitate formation.
6. Based on the fatigue crack propagation performance of neutron irradiated, annealed and 20 percent cold worked Type 316 stainless steel at 649°C, it is concluded that the effects of hold time and irradiation may not saturate at this, at this temperature and that further increases in crack propagation rate may occur at higher temperatures.

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