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THERMAL FATIGUE TESTS
Materials of Construction for Denitrator Pots

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THERMAL FATIGUE TESTS
MATERIALS OF CONSTRUCTION FOR DENITRATOR POTS

by

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July 1964

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ABSTRACT

Stainless steel denitrator pots at the Savannah River Plant have deformed and cracked by thermal fatigue. Subsequent examination of the microstructure of type 347 stainless steel used in these vessels revealed the presence of sigma phase and led to speculation that sigma phase may play a role in embrittling the steel. Thermal fatigue tests carried out on 304L, 316, and 347 stainless steels showed that of the three, type 347 was superior and that sigma phase was not a factor in failure. Tests made with copper, clad on both sides with stainless steel, indicated that the composite material would prevent thermal fatigue by eliminating the large temperature gradients that lead to deformation and subsequent failure.

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MATERIALS OF CONSTRUCTION FOR DENITRATOR POTS

INTRODUCTION

The Savannah River Plant uses a one-step, batch operation to convert an aqueous solution of uranyl nitrate to solid uranium trioxide (UO_3). During the cycle, the temperature of the vessel contents is increased from 120°C to approximately 600°C. In the past few years, the bottoms of the gas-fired vessels used in the conversion process have deformed and cracked because of the high stress caused by uneven temperature distributions within the metal and the cyclic nature of the process. Failure has been attributed to thermal fatigue.

Although thermal fatigue has been recognized for many years, the subject has received increased study in the last decade. Nuclear reactors and jet engines, where cyclic temperatures are encountered and where an accurate knowledge of service life is essential, are examples of fields where considerable effort has been expended. An excellent review of thermal fatigue is given by Glenny[1].

The usual approaches to thermal fatigue problems are either (1) to find a more resistant material, (2) to alter the design to minimize thermal gradients, or (3) to alter the temperature cycle. In the present case, the basic vessel design and thermal cycle were fixed; hence, effort was directed toward finding a more resistant material. This report describes the thermal fatigue behavior of three stainless steels and a composite of copper, clad on both sides with stainless steel.

SUMMARY

Type 347 stainless steel was more resistant to thermal fatigue than either 304L or 316 stainless steel. Sigma phase, which was noted in the failed 347 vessel bottoms, had little effect on the service life of either 347 or 316; no sigma phase was observed in 304L.

On the basis of laboratory tests on relatively thin samples, stainless-steel-clad copper is a promising material for vessel bottoms. Attempts to thermally cycle thin sections of this material were not successful because the copper core dissipated heat so rapidly that thermal gradients of sufficient magnitude to cause damage could not be obtained. Furthermore, no brittle intermetallic compounds formed at the copper - stainless steel interface after heating to 850°C for two

months. The results indicate that if type 347 steel continues to fail in denitrator pot service, the composite stainless-steel-clad copper material of the proper thickness should be tested for this application.

DISCUSSION

BACKGROUND

In the Separations Area of the Savannah River Plant spent fuel from the reactors is processed to separate uranium and plutonium from fission products. One of the processes converts a concentrated solution of uranyl nitrate to UO_3 in a one-step operation. The vessels used are known as denitrator pots.

This process requires approximately 11 hours for completion and is accomplished in three phases: first, the solution is boiled for 2 hours at low heat until the intermediate product reaches 300°C; second, the rate of heating is increased and in 2 hours the vessel contents reach approximately 600°C; and third, the product is maintained at 600°C for the remaining 7 hours. In all three phases the vessel contents are continually stirred by an agitator to mix them, as well as to prevent formation of a cake on the heated bottom surface of the vessel. The powdered final product is removed by suction hoses.

The denitrator pots are made of 1/2-inch-thick type 347 stainless steel and are approximately 5 feet in diameter and 4 feet high. They are heated by gas burners from the bottom. Failures occur as bulges and cracks on the flame side of the bottoms. Corrosion by the uranyl nitrate solution is not a factor. Repair can be made by cutting out the deformed portion, usually located near the center of the bottom, and welding in a new section. A pot averages 630 cycles before the first repair is made and the average total life is 1650 cycles. Figure 1 shows the frequency of failure with respect to the number of cycles.

Failure in the vessel bottoms is caused by localized temperature differences that produce stresses by means of unequal expansion and contraction. The temperature differences probably arise from poor heat transfer caused by cake that is not scraped from local areas of the heated bottom by the agitator. The formation of bulges and cracks can be explained by the mechanism of thermal fatigue, which is defined as that type of fatigue failure in which the recurring stresses are caused by cyclic temperature differences. A brief explanation of thermal fatigue is included in the Appendix.

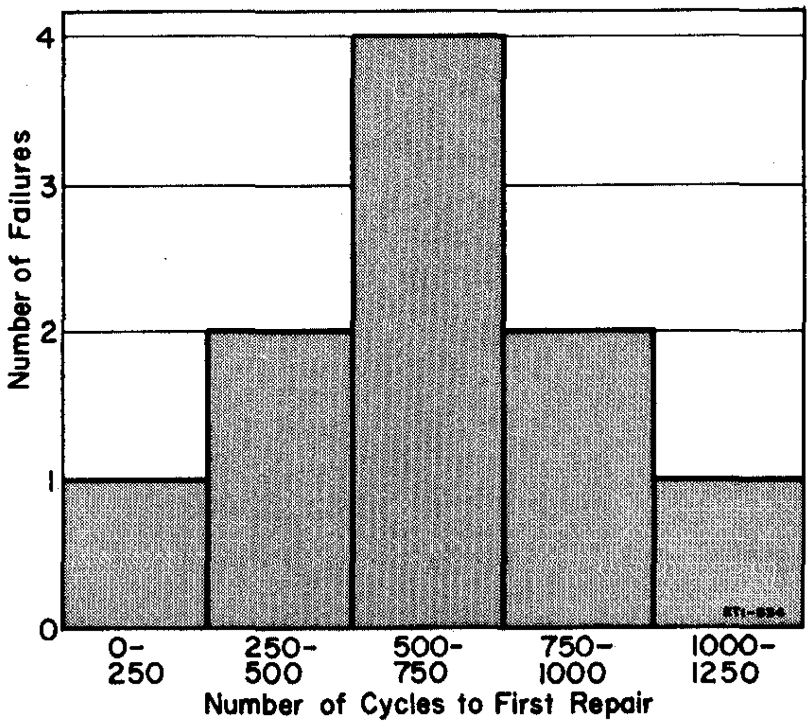


FIG. 1 PLANT EXPERIENCE ON FAILURE OF DENITRATOR POTS
The average total life of the vessels is 1650 cycles and the average life to the first repair is 630 cycles.

METALLOGRAPHIC EXAMINATION OF DENITRATOR POTS

During the course of the laboratory investigation, the bottoms of two vessels were available for metallographic examination. Vessel C-3-1 No. 3 failed after only 200 runs, but it was noted that the bottom was fabricated from two plates of type 347 stainless steel instead of one. Failure occurred near the weld seam and was probably caused by the combination of thermal fatigue and ductility loss due to welding.[2] A photomicrograph of this steel is shown in Figure 2. Because of the weld, this failure could not be classed as typical of thermal fatigue in hot-rolled stainless steel.

Vessel C-3-2 No. 2 survived 525 runs before it was taken out of service for repairs, and failure was attributed to thermal fatigue only, since the bottom was made from a single plate. The photograph (Figure 3) shows that the failure is primarily intergranular and that the grains are pulled apart, indicating tensile plastic flow.

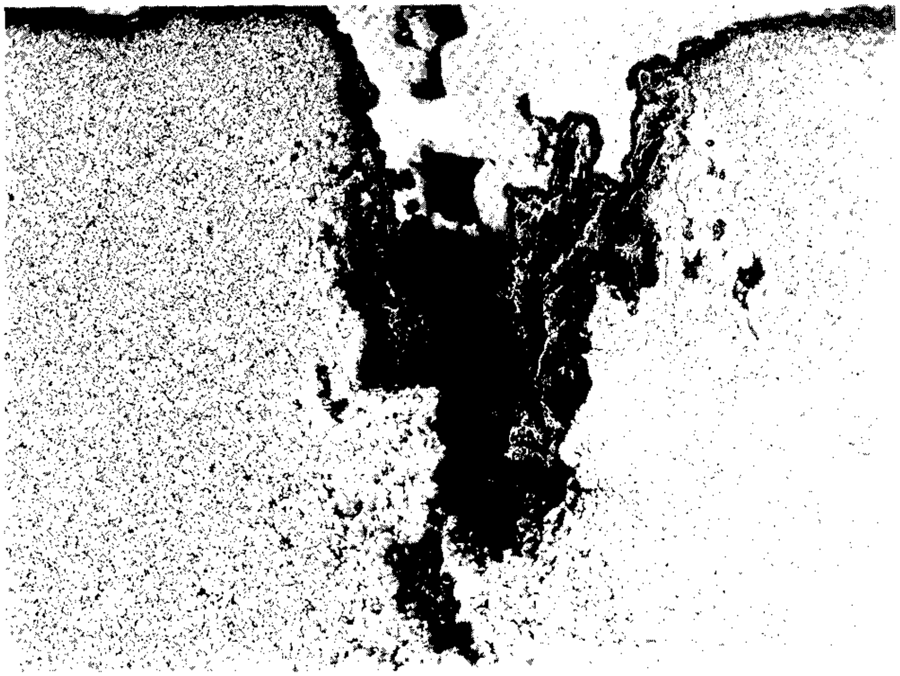


FIG. 2 THERMAL FATIGUE CRACK IN VESSEL C-3-1 NO. 3 (100X)
This crack was one of the few found on the surface of the vessel and was located near the main failure and welded zone.

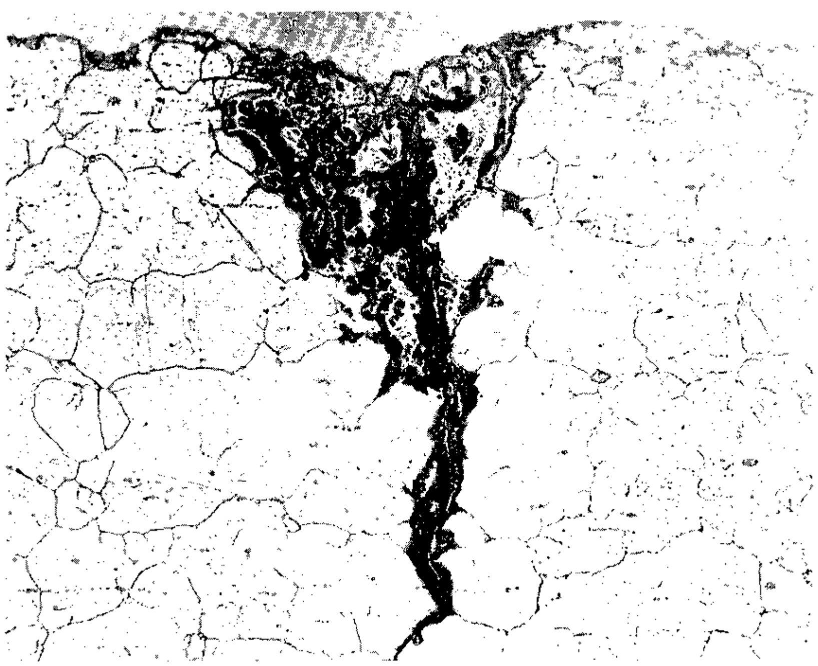


FIG. 3 THERMAL FATIGUE CRACK IN VESSEL C-3-2 NO. 2 (250X)
This crack is typical of those seen along the surface near the area of severest attack.

Of particular interest was the discovery of sigma phase in the vessel bottoms (Figures 4a and b). Sigma phase is an iron-chromium intermetallic compound that is not normally found in hot-rolled stainless steels. However, in certain types of stainless steel, such as 309, 316, 321, or 347, sigma phase will precipitate during annealing at 600 to 850°C, but will dissolve at temperatures exceeding about 1000°C; in other steels, such as types 304 and 304L, the sigma phase rarely, if ever, forms. The deciding factor for formation of sigma phase is the composition of the alloy. Jackson[8] reported that the presence of considerable sigma phase decreased the thermal fatigue life of cast 25% Cr - 12% Ni alloys, but it was not determined whether sigma phase was detrimental in smaller amounts, such as the quantities found in the vessel bottoms. Thermal fatigue of the 300 series stainless steel containing sigma phase has not been studied.

After the discovery of sigma phase in the failed pots, the hypothesis was advanced that sigma phase could reduce the thermal fatigue life of the 347 stainless steel. If this were true, then changing the bottom from 347 to 347 clad with 304L, for example, which does not form sigma phase, would improve the resistance to failure.

EXPERIMENTAL PROGRAM

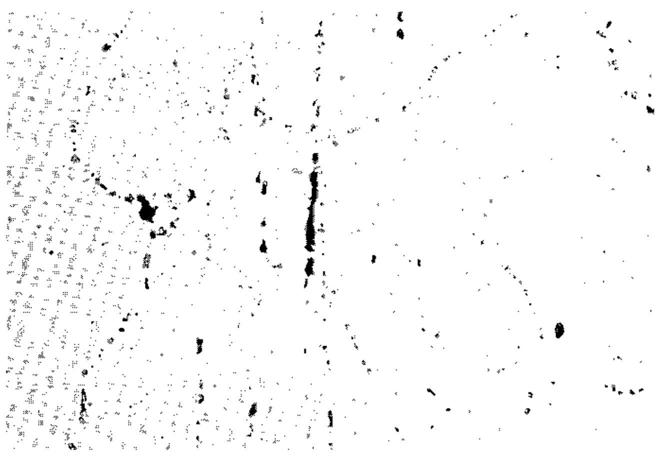
A program was initiated to answer three questions:

- Does sigma phase decrease the thermal fatigue life of various 300 series stainless steel alloys?
- Is there a stainless steel with better thermal fatigue resistance than type 347, or is there a possibility of cladding type 347 to improve performance?
- Will stainless-steel-clad copper perform better than stainless steel alone by increasing the heat dissipation and thereby reducing the temperature difference?

Various means of investigating thermal fatigue have been used. For the most fundamental studies, the tests are designed so that stress measurements can be obtained, thereby relating the number of cycles to failure with the stress level and temperature. The more common approach, which was adopted for this study, is to investigate only the relationship between temperature and cycles to failure

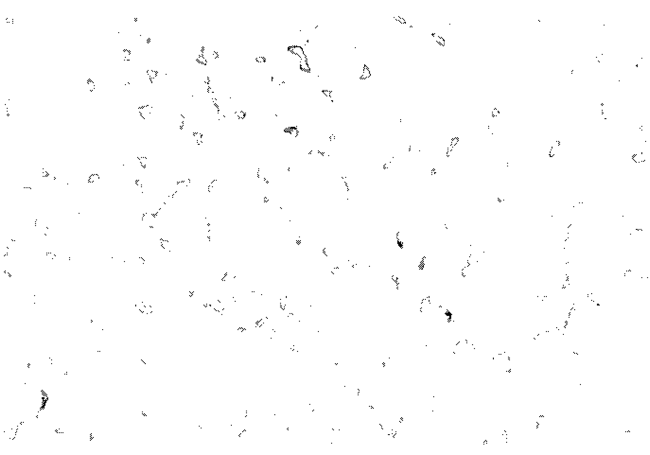
a. Vessel C-3-1 No. 3
(200 runs to failure).

Neg. 48560



b. Vessel C-3-2 No. 2
(526 runs to failure).

Neg. 48561



c. Typical sigma structure
in test specimens of
type 347 stainless steel.

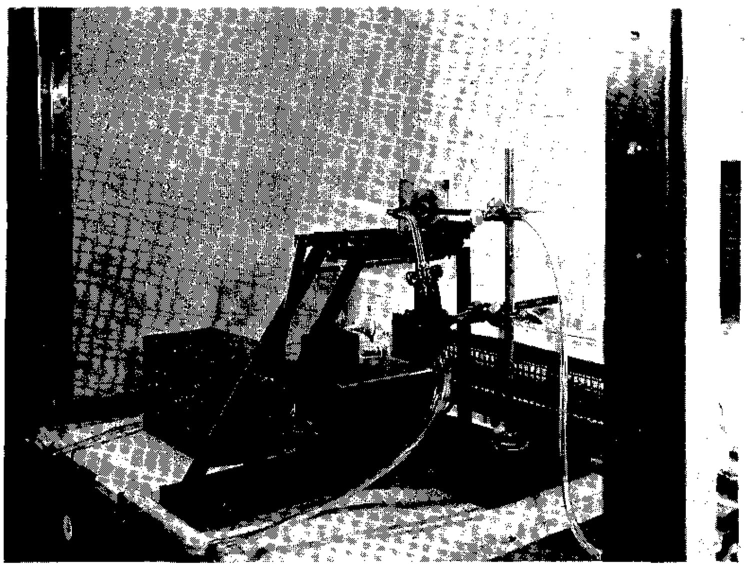
Neg. 48559

FIG. 4 SIGMA DISTRIBUTION IN TYPE 347 STAINLESS STEEL (500X)
The greater sigma content of the steel used in vessel C-3-1 No. 3 suggested that sigma was a major factor in failure. The large amount of sigma formed in test samples, although not as widely distributed, failed to decrease thermal fatigue life.

and to neglect stress completely. While this latter method is more crude, the apparatus is simpler and an indication is given of the relative performance of the various materials tested.

The apparatus is shown in Figure 5. An oxygen-propane torch heated the center of the 4 x 4 x 1/8-inch sample plates whose surfaces were ground with 120-grit abrasive; the plate was cooled by an air blast. The torch had the advantage of simulating the gaseous atmosphere of the heating chamber under the vessel bottoms.

The maximum temperature reached by a sample during the heating cycle was determined by a calibrated optical pyrometer, with correction for non-black body conditions. The low temperature (~200°C) was measured by pressing 36-gage thermocouple wires against the heated portion of the plate. The low temperatures ranged from 160 to 250°C in every case. The temperatures were controlled by the length of time the samples remained in front of the flame or air blast. A 3X telescope was used to determine the first sign of cracking, which was considered failure.



Neg. 45189

FIG. 5 THERMAL FATIGUE APPARATUS

The test specimen is shown in the cooling position where an air blast is directed at the heated portion. The rings of oxidation and interference colors on the sample are typical of a sample after a few cycles. The sample is mounted on a cart which is rolled between the torch and air line by means of the solenoid mounted on the base of the apparatus. The two timers controlling the heating and cooling cycles are shown at the rear.

Three stainless steels, 304L, 316, and 347, were chosen for study because of their structural and mechanical characteristics. Type 304L forms little or no sigma phase, although with prolonged heating chromium carbides are precipitated at the grain boundaries. Sigma phase, as well as chromium carbides, will form in type 316; this alloy has greater creep resistance than either 304L or 347. The creep resistance of 347 is intermediate between 304L and 316; it will form sigma phase, but not chromium carbides.

Samples of types 316 and 347 stainless steel containing sigma phase prior to testing were produced in two lots by annealing at 850°C, one for 1000 hours and the other for 500 hours (Figure 4c). No difference was noted in the microstructural or thermal fatigue behavior of the two lots and hereafter no distinction between them will be made. Type 304L was included in the sigma-forming anneal, although no sigma phase was detected in the samples. The presence of sigma was determined by etching electrolytically with 10N KOH.

RESULTS WITH STAINLESS STEELS

Table I and Figure 6 show the results of all runs. A linear dependence between temperature and the logarithm of the number of cycles to failure was found, which is the normal relationship for thermal fatigue. The data also show that 347 is more resistant to failure than either 304L or 316.

TABLE I

Summary of Test Results

	Hot Rolled		Sigma Annealed[(a)]	
Alloy	High Temp, °C	Cycles to Failure	High Temp, °C	Cycles to Failure
347	1150	37	1170	38
	1140	33	1095	61
	1125	38	1045	100
	1095	51	1005	229
	995	682	915	1021
	950	603	830	1917
316	1175	23	1090	58
	1125	26	920	411
	1095	48	840	1436
	1010	134		
	860	758		
304L	1205	16	1150	17
	1185	20	1010	47
	1105	36	935	165
	1095	65	870	444
	980	141		
	890	621		
	815	1630		

(a) Samples were annealed at 850°C for 500 or 1000 hr to precipitate the sigma phase in 347 and 316.

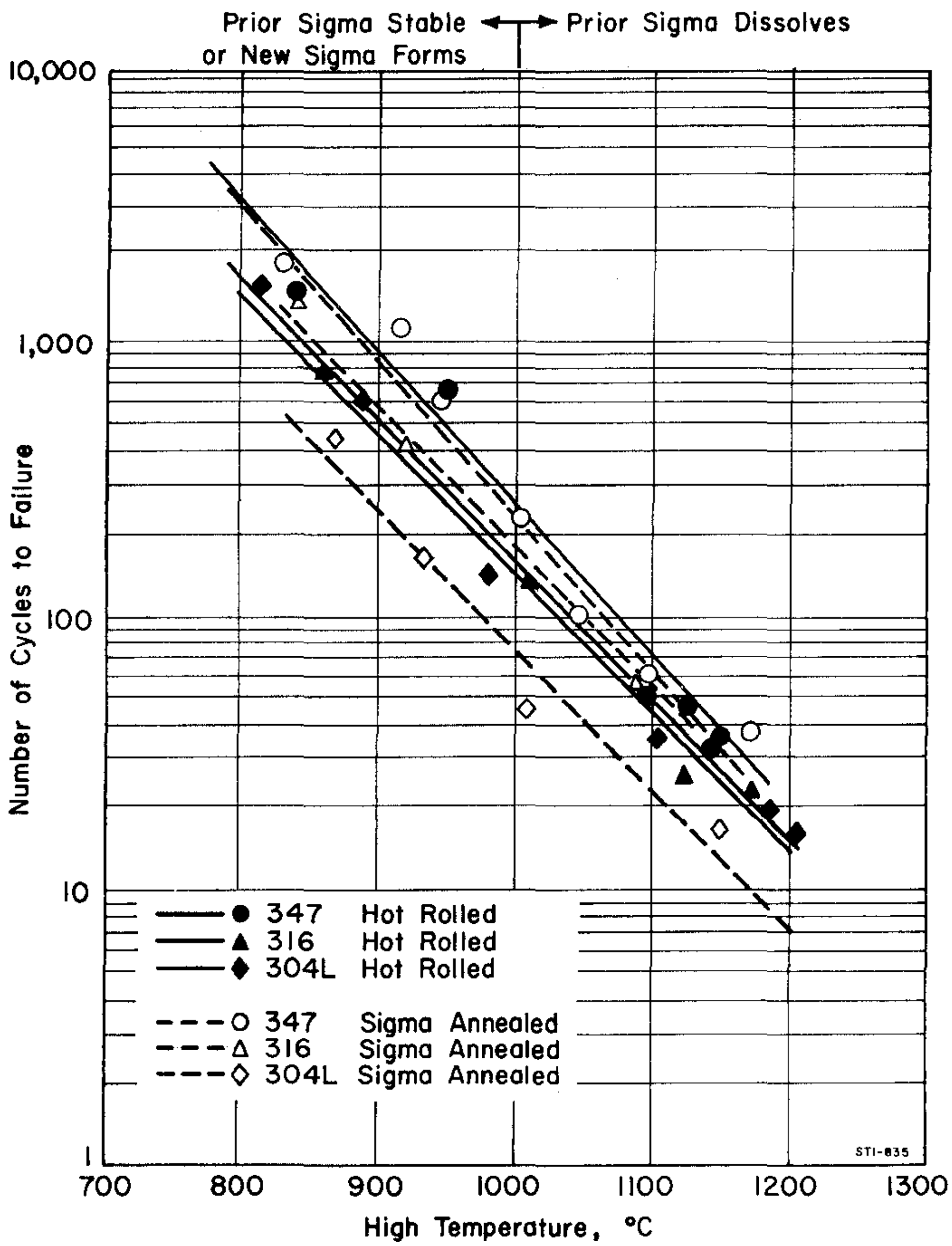


FIG. 6 THERMAL FATIGUE LIFE OF STAINLESS STEELS AS A FUNCTION OF HIGH TEMPERATURE

Type 347 stainless steel with or without sigma phase is slightly superior to type 316 also with or without sigma or to 304L. The poor resistance of type 304L subjected to the sigma-forming heat treatment is due to an increase in grain size.

The sigma-forming anneal at 850°C greatly shortened the life of the 304L alloy, even though no sigma phase formed. As shown in the table below, the shortened life is attributed to increased grain size, which resulted in a loss of ductility. Stainless steels that contain finely dispersed phases, such as types 309Cb, 347, and 321, resist grain growth and in this respect are superior to other grades.

Change in Grain Size During Sigma Anneal

Steel	ASTM Grain Size Before	ASTM Grain Size After
304L	7	2
316	6	5
347	8	8

Metallographic examinations were made of the specimens after test to reveal the possible effects of the sigma phase. Samples of 347 and 316 that initially contained sigma and were heated to temperatures in excess of 1000°C in the test showed either a decided decrease or complete disappearance of sigma in the heated zone. Samples of these steels that did not contain sigma originally, but which were subjected to similar temperature conditions, showed no evidence of sigma. In the 850-1000°C range, a fine network of sigma phase was precipitated at the grain boundaries of samples that were initially free of sigma, whereas no appreciable change in the sigma distribution was observed in those specimens that originally contained this phase.

The metallographic results and the lifetime-temperature curves of Figure 6 show that in the temperature range where sigma is stable, failure by thermal fatigue could not be attributed to sigma phase. The fact that the slopes of the lifetime-temperature curves are identical indicates that the same failure mechanism is operative, irrespective of the following structural sigma-phase variations: (1) one steel formed no sigma under any conditions, (2) two steels contained sigma throughout the tests, and (3) two steels containing no sigma initially formed sigma during tests.

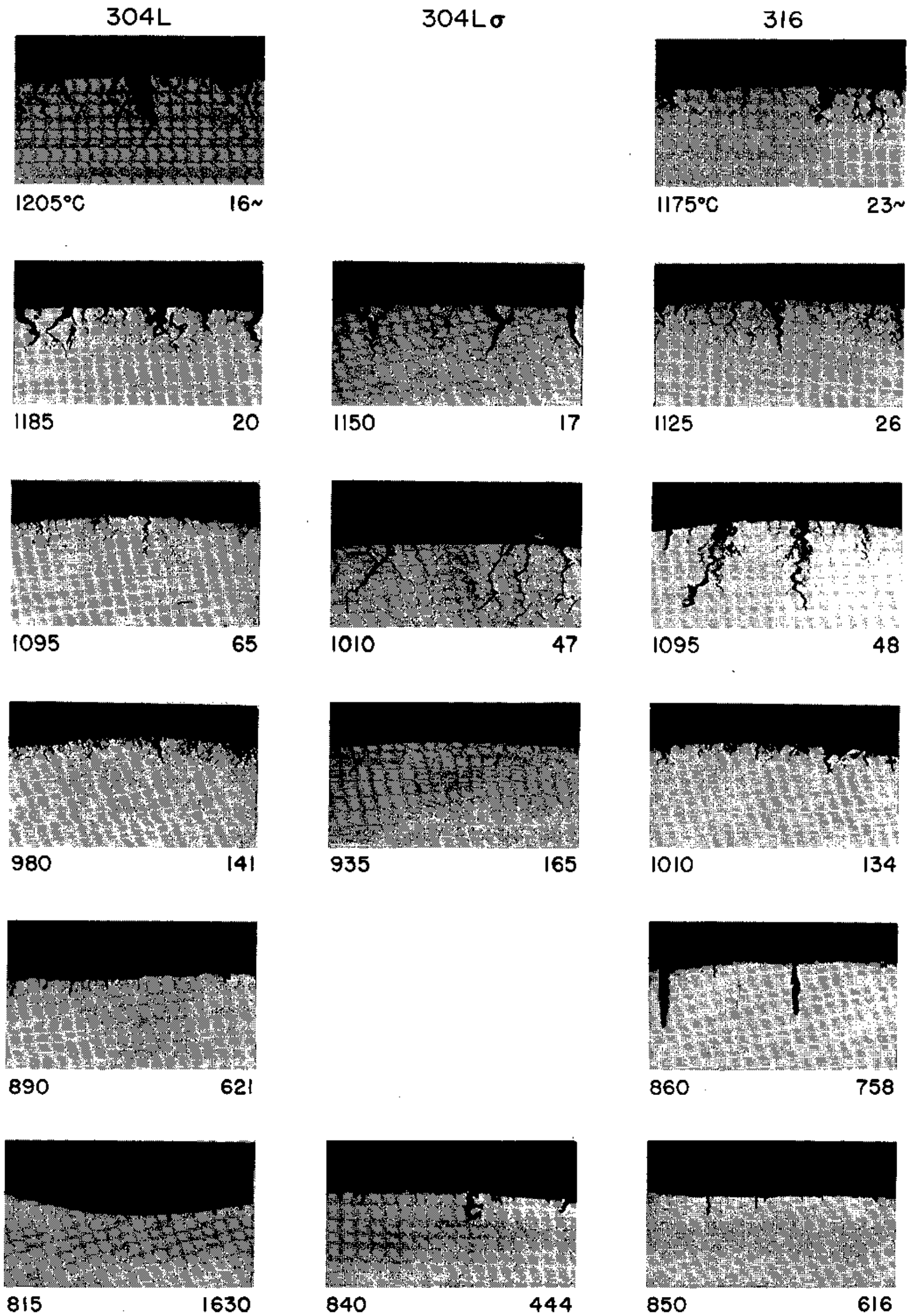
Metallographic examination of the test specimens revealed two types of cracks. The first type was characterized by intergranular fissures in which oxides were present, closely resembling failures observed in the denitrator pots. This attack occurred at test temperatures above 950-1000°C. The second type was typical of normally encountered fatigue failures; the cracks were straight and relatively smooth-faced. Both types are shown in Figure 7. It is somewhat surprising that such a change from transgranular to intergranular cracking does not produce a change in the slope of the log cycles-temperatures curve at the 950-1000°C range. However, this same behavior has been noted by Lardge[4] in tests with Nimonic-75.

The fact that intergranular cracks are present can be explained in two ways. At high temperatures it is possible that grain boundary oxidation occurs first, and then the boundaries open under the cyclic stresses. Another possibility is that the equicohesive temperature, which for stainless steels is at approximately 730°C, has been exceeded so that the grain boundaries are much weaker than the grains with the result that they open and then oxidize.

However, the fact that intergranular attack was found in the laboratory tests only above 950°C does not necessarily indicate that the pots, in which intergranular cracks were observed, also reached such temperatures. The laboratory tests were completed in a few hours, whereas the pots are exposed to the high temperatures 100 to 1000 times longer. After long exposures, grain boundary oxidation would be observed at much lower temperatures than with short exposures. It is conceivable that even the equicohesive temperature may be time dependent.

In some tests the side of the sample facing the flame did not crack first, nor did the plate always buckle toward the flame. This is surprising in view of the more severe conditions existing on the flame side. When cracking was noted on the back of the specimen, the high temperature recorded for that side was plotted in Figure 6. At temperatures of about 950°C and lower, the heated portion was found to thicken considerably at the expense of metal adjacent to the heated area, as shown in Figure 8.

The results of this work have been reported by use of only two parameters: cycles to failure and high temperature. A third parameter, the difference between high and low temperatures, would be expected to have an effect from theoretical considerations since the stress is determined by this parameter. However, Glenny and Taylor[5] investigated the temperature parameters and concluded that the maximum temperature was more important than temperature difference. The reason is obvious; strength, oxidation, and diffusion are all affected to a much greater extent at the high temperature than at the low temperature, and these factors are the ones that primarily contribute to failure. Since in these experiments the lower temperatures were held essentially constant, 160-250°C, the high temperature is in reality an approximation of both the maximum temperature and the temperature difference.



Note the change from intergranular to transgranular cracking in the temperature range of 950-1000°C.

FIG. 7 MICROSTRUCTURE OF SURFACES SHOWING FISSURES PRODUCED IN THERMAL FATIGUE

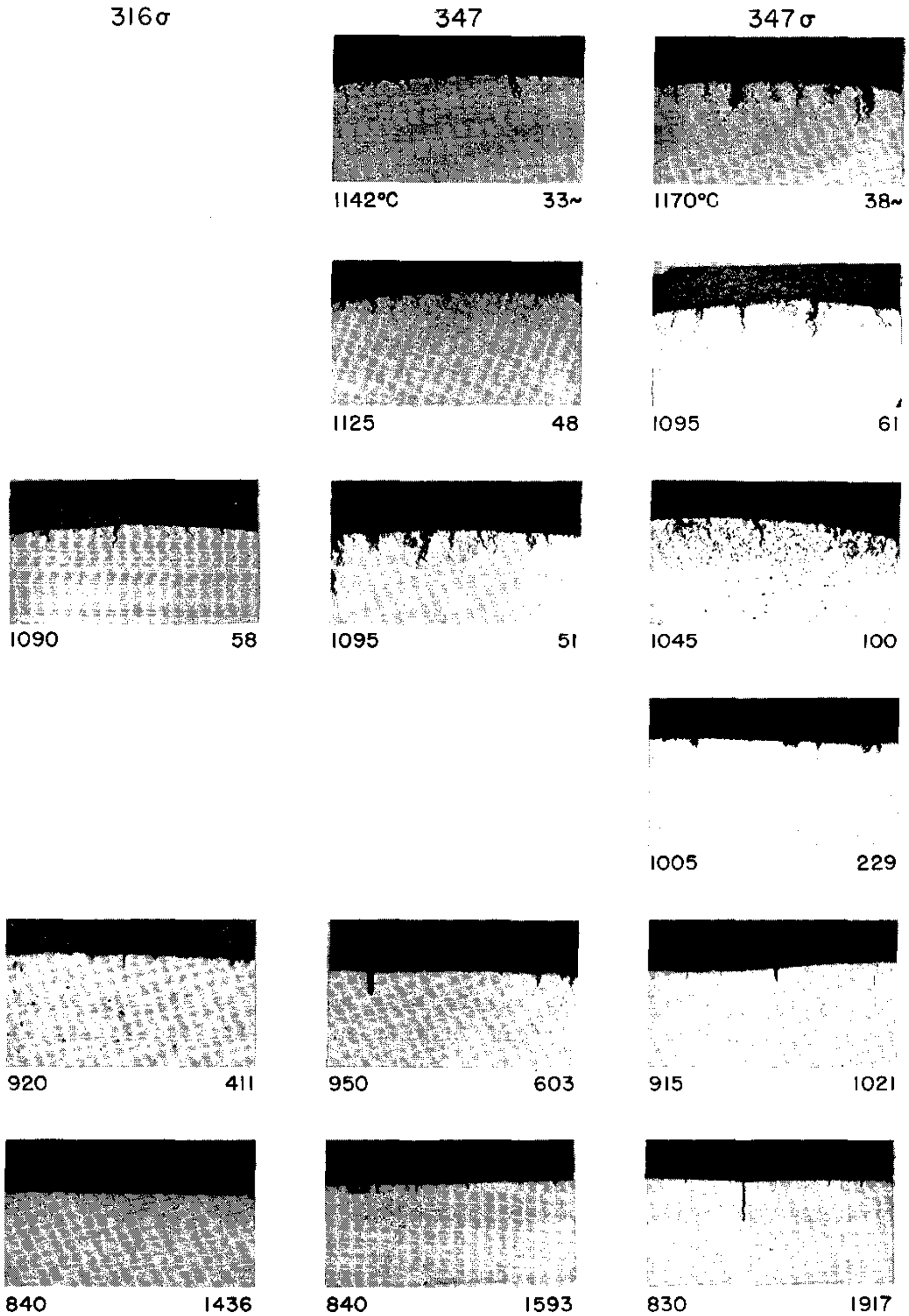


FIG. 7 CONTINUED

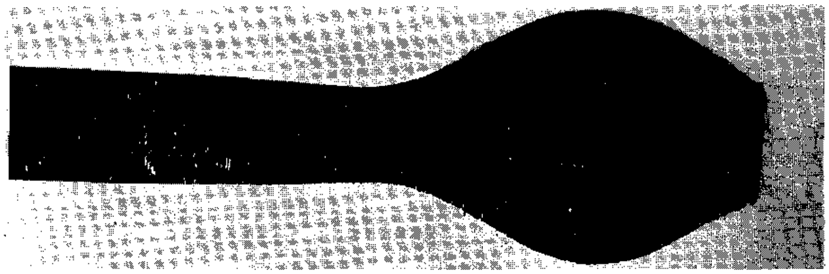


FIG. 8 EXAMPLE OF PLASTIC FLOW

The above photomicrograph shows a cross section of the center of a 304L sample that was cycled 621 times between 890 and 230°C prior to failure. The steel flowed from the surrounding area into the center bulge. This specimen was unusual in that cracks occurred on the cool side (890°C) rather than on the flame side (1030°C).

RESULTS WITH A COMPOSITE OF STAINLESS STEEL – COPPER – STAINLESS STEEL

The experiments described above indicated that type 347 stainless steel was superior to the other types of stainless steel. The next step was to investigate the possibility of eliminating the temperature differences.

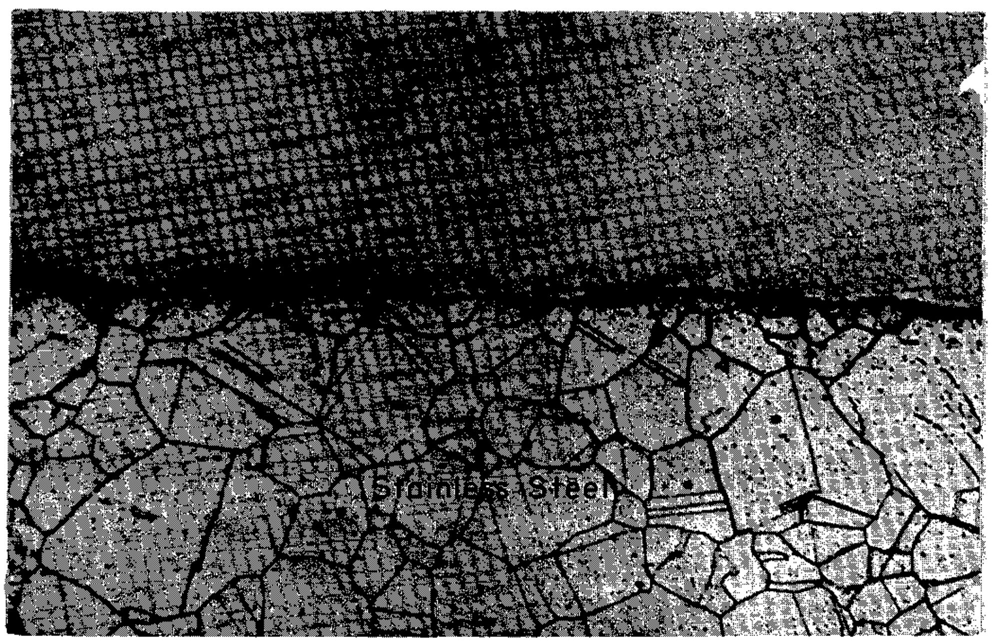
Copper, clad with 347 stainless steel, appeared promising in that the stainless steel would provide the corrosion resistance necessary for both the flame and solution sides, while the middle layer of copper would minimize thermal gradients. Previous work by Lardge[4] with Nimonic-75 – copper sandwiches showed greatly increased life over Nimonic-75 alone.

Unsuccessful attempts were made to acquire 347 stainless-steel-clad copper whose total thickness was identical to the other samples used. However, 310-clad copper and 304-clad copper with total thicknesses of 0.080 and 0.078 inch, respectively, were obtained. The stainless-copper-stainless ratio was 25-50-25 in each case, whereas a 45-10-45 ratio would have been better from the standpoint of high temperature strength for denitration service.

Tests were attempted, but a few cycles showed that the copper conducted the heat over the entire plate so well that the stresses due to temperature differences were negligible. In other words the copper was so effective in its function that thermal fatigue failure was virtually impossible. From the mechanical standpoint, the as-fabricated copper-stainless bond is strong and uniform. Furthermore, the coefficient of thermal expansion for copper is $17.5 \times 10^{-6}/°C$, almost identical with the 16.6 x $10^{-6}/^{o}C$ value of 347, thereby minimizing the small thermal stresses across the bond zone that would occur in normal operations.

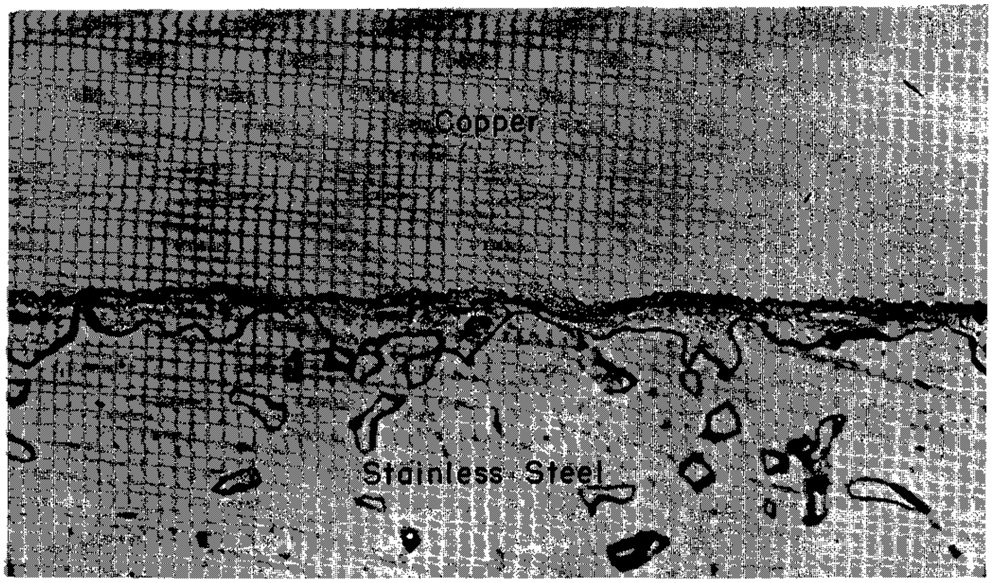
The one point that could cause difficulty is the mutual diffusion of copper and stainless steel to form a brittle interface. In order to evaluate this possibility, two 50-day diffusion tests were run, one at 850°C and the other at 600°C. In both cases the samples were bent at room temperature after test to a radius no greater than five times the thickness, and were metallographically examined for failure. No cracks were found and all material was intact. In the 850°C run, a diffusion zone of 0.005 inch was noted in the stainless steel whereas only a trace was seen for the 600°C run (Figures 9a and b). The results from these diffusion tests indicate that intermetallic compound formation is of minor importance for relatively long service.

In addition to longer life, another advantage would be gained from the copper-stainless bottom. The heat input to the vessels is determined by a temperature-sensing device located on the bottom, and if a hot spot develops at this location, the heat input is decreased, resulting in a long cycle time. By equalizing the temperature of the vessel bottom, more uniform time cycles could be expected.



NEG. 48412 400X

FIG. 9a COPPER – STAINLESS STEEL DIFFUSION AFTER 50 DAYS AT 600°C
Only a trace of a diffusion zone is noted at the interface. The type 310 stainless steel is heavily sensitized and the boundaries of large copper grains are apparent.



NEG. 48413 400X

FIG. 9b COPPER – STAINLESS STEEL DIFFUSION AFTER 50 DAYS AT 855°C
The diffusion zone is far more pronounced than at 600°C. This sample was very lightly etched with oxalic acid in order to accentuate the diffusion zone with the result that the grain boundaries of the 310 stainless steel are only vaguely visible. The rapidly attacked sigma phase in the steel is plainly visible.

APPENDIX

MECHANISM OF THERMAL FATIGUE

In the heating of an unrestrained bar (Figure 10), the bar is at a low temperature, T_1, in the initial state, (Figure 10a). When the bar is uniformly heated to a higher temperature, T_2 (Figure 10b), the length increases by ΔL as determined by the coefficient of thermal expansion, K, and the temperature difference, $T_2 - T_1$, and the original length, L, as shown in the following equation:

$$\Delta L = L(T_2 - T_1)K \qquad (1)$$

In Figure 10c, the bar has been uniformly cooled to T_1. Since no restraint was imposed on the bar, no stresses, elastic or plastic, were produced. On completion of a cycle, no change has occurred. The high temperatures must be such that no melting or weakening occurred to cause sagging of the bar under its own weight; no other restrictions on temperature are specified.

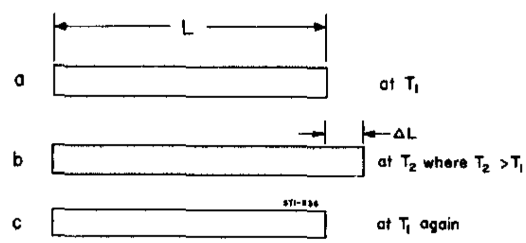


FIG. 10 CASE OF THE UNRESTRAINED AND UNIFORMLY HEATED BAR

In the situation in which the bar is restrained from expanding or contracting (Figure 11), the difference between the maximum and minimum temperature is of importance. If the bar is heated from its initial temperature of T_1, it attempts to expand along its length (and diameter) by the amount determined by equation 1. The expansion is resisted, however, and a compressive stress is produced that has a magnitude sufficient to produce a decrease in length identical to the increase due to thermal expansion. Since the net length change is zero, the two effects are equal as long as

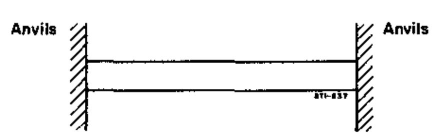


FIG. 11 CASE OF THE RESTRAINED BAR

the stress remains below the yield stress (i.e., only elastic behavior is encountered).

$$\Delta L = 0 = L(T_2-T_1)K - (S/E)L \qquad (2)$$

or

$$S = (T_2-T_1)KE$$

where

S = stress

E = Young's modulus

The stress-strain diagram for this example is shown in Figure 12.

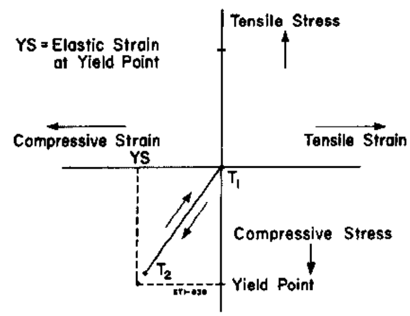


FIG. 12 STRESS-STRAIN DIAGRAM FOR THE CASE OF ELASTIC DEFORMATION DUE TO THERMAL CYCLING

This figure shows the stress-strain relationship when the compressive stresses induced by the heating from T_1 to T_2 remain in the region of compressive elastic stress. Note that strain could be replaced by temperature since strain is defined by $\Delta L/L$ and in this case ΔL is directly proportional to (T_2-T_1).

If the yield stress is substituted ($S = 30{,}000$ psi), and the appropriate values of E and K are used ($E = 29 \times 10^6$ psi, $K = 16.6 \times 10^{-6}/^{o}C$) for stainless steel, a ΔT of 62.4°C is obtained. The significance of this temperature difference, ΔT_E, is that below a difference of 62.4°C all stresses are elastic and no permanent deformation occurs on cycling.

In Figure 13 the path of the stress-strain (or temperature) diagram is plotted for the case where the temperature difference, ΔT, exceeds ΔT_E, but not $2\Delta T_E$. Initially on heating, the same path is traced as before, starting from the origin A, but this time the elastic limit is reached.

At this point the stress remains constant while the strain continues to increase to the value determined by the upper temperature, T_3. In this example the metal is assumed to be ideal in that no work hardening occurs in the plastic region, no Bauschinger[6] effect is noted, and no creep occurs at any temperature. On cooling, the path follows CD, which represents completely elastic strain, since the process proceeds from the compressive elastic region into the tensile elastic region at stresses less than the yield stress. A repetition of the temperature cycle will follow path CD only, and purely elastic behavior occurs. Plastic behavior is encountered only on the first cycle. The fatigue life of metals subjected to thermal fluctuations such as described in the first two examples would be very long.

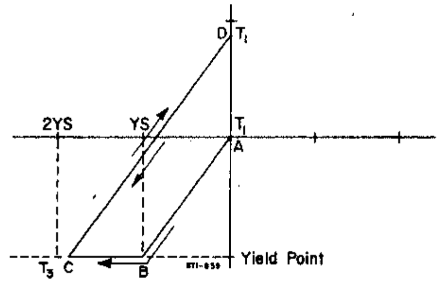
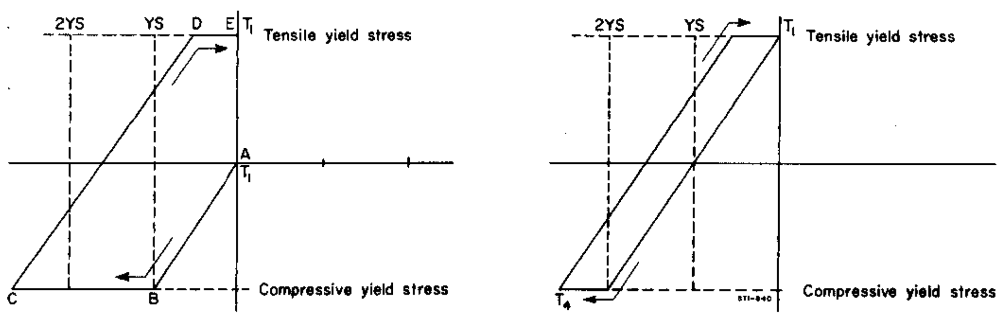


FIG. 13 STRESS-STRAIN DIAGRAM FOR THE CASE WHERE ΔT IS GREATER THAN ΔT_E AND LESS THAN $2\Delta T_E$

In the final example, Figure 14a, the temperature difference exceeds $2\Delta T_E$. The path followed on the first cycle starts at A, and the sample is elastically compressed to point B and then flows plastically to point C, corresponding to the high temperature T_4. Cooling starts and the elastic region is traversed from C to D, and plastic flow occurs again from D to E. Repetition of the cycle is shown in Figure 14b. Both tensile and compressive plastic flow occur on each cycle; fatigue life is limited in this circumstance.



a. First cycle

b. Repetitive cycles

FIG. 14 STRESS-STRAIN DIAGRAM FOR THE CASE WHERE ΔT EXCEEDS $2\Delta T_E$

In the example described in Figure 14, plastic flow occurred in both directions, but this is not to say the specimen will maintain the same shape cycle after cycle. In real bars, deformation would occur by buckling, and the bars would never attain their straight shape again. Where constant loads, compressive or tensile, are applied throughout the cycle, such as in pressure vessels, one type of plastic flow will be opposed, and the other increased. The net result is a "ratchet effect", which produces rapid permanent deformation.

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