Stress Relaxation and Strain-Age Cracking in René 41 Weldments

Electron microscopy studies show that stress relaxation behavior is influenced by gamma prime precipitation during welding and postweld heat treatment

BY J. E. FRANKLIN AND W. F. SAVAGE

ABSTRACT. Strain-age cracking in nickel base superalloy weldments results from the strains which accompany the relaxation of residual stresses at temperatures within the range in which age hardening reactions occur. The mechanism by which deformation resulting in the relaxation of residual stresses occurs during post-weld heat treatment is dependent on the microstructure of the material. It has been proposed that precipitation of gamma prime during welding and post-weld heat treatment affects the stress relaxation behavior in the weldment and, as a result, affects strain-age cracking sensitivity. Precipitation causes hardening of grain interiors which may result in localized deformation at grain boundaries. In addition, the aging contraction associated with gamma prime precipitation results in the generation of stresses which may contribute to strain-age cracking. In this investigation the microstructural changes accompanying aging during welding and post-weld heat treatment were studied, and the effect of aging on stress relaxation behavior and strain-age cracking was determined.

Specimens whose structure represented that of the portion of the heat-affected zone adjacent to the weld fusion zone were prepared by exposing them to a simulated heat-affected zone thermal cycle. The structure of these specimens was studied using transmission electron microscopy and compared to the structure of the base metal. Both base metal and simulated heat-affected zone specimens were then subjected to isothermal, constant elongation, stress relaxation tests. Each test was conducted using the Gleeble by rapidly heating the tensile specimen to a predetermined aging temperature, pulling to a predetermined elongation and then maintaining constant temperature and elongation while the uniaxial stress on the specimen was monitored and recorded. The resultant structures were again examined by transmission electron microscopy. This program was conducted on specimens subjected to two different pre-weld heat treatments: (1) Solution anneal and quench to retain gamma prime forming elements in solution, (2) Overage to form large gamma prime precipitates.

Transmission electron microscopy studies showed that the same structure resulted from exposure to the simulated heat-affected zone thermal cycle regardless of initial heat treatment. Gamma prime in the overaged René 41 dissolved during exposure to temperatures above the gamma prime solvus, and in both the overaged and solution annealed René 41, additional gamma prime precipitation occurred on cooling resulting in a homogenous distribution of fine particles. Also, grain boundary carbides were dissolved in both materials and did not appear to reprecipitate on cooling.

The results of the stress relaxation tests showed that a marked difference existed between the behavior of the overaged and solution annealed material which had not experienced a simulated heat-affected zone thermal cycle. The stress in the overaged material decreased gradually and continuously for the several minutes of the duration of the test. In the solution annealed material the stress decreased rapidly at first, but after less than one second began to increase and continued this increase for about 20 seconds after which the stress decreased. The result was that during the later stages of the test the stress was significantly higher in the solution annealed material (for equal initial stress levels). This stress generation in solution annealed René 41 was attributed to aging contraction resulting from the precipitation of gamma prime during the test.

However, after exposure to the simulated heat-affected zone thermal cycle both materials behaved similarly. In this case there was a small initial decrease in stress followed by an increase which resulted in a high stress level for several minutes. These specimens failed after 100 to 200 seconds when tested at high initial stress levels. The failures were shown to occur at grain boundaries where continuous $M_{23}C_6$ carbide networks had precipitated during the test. Transmission electron microscopy studies confirmed that stress generation was

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Paper was presented at the 55th AWS Annual Meeting held at Houston during May 6-10, 1974.

accompanied by additional gamma prime precipitation and growth.

It was concluded from this investigation that precipitation of gamma prime during welding and post-weld heat treatment of René 41 contributes significantly to strain-age cracking in this alloy. Gamma prime inhibits deformation by intragranular slip while the matrix contraction accompanying precipitation causes additional stress to be generated. This results in a high stress level combined with a localized grain boundary deformation process which leads to cracking.

Overaging prior to welding allows intragranular slip to occur and eliminates generation of aging contraction stresses in the base metal. However, the overaged precipitate is dissolved in the heat-affected zone, and this region becomes susceptible to the deleterious effects of gamma prime precipitation during postweld heat treatment.

Introduction

The utilization of many precipitation hardening alloys has been limited by their susceptibility to cracking during postweld heat treatment. The gamma prime strengthened nickel base superalloys with high aluminum and titanium contents are particularly susceptible to this type of cracking. The term strain-age cracking arises from the fact that cracking occurs in highly restrained weldments, as they are heated through the temperature range in which aging occurs. Cracks usually nucleate at heat-affected zone grain boundaries and often propagate to microscopic size.

Considerable effort has been expended to determine the cause and mechanism for this cracking, and, in general, it can be said that it is a result of low ductility in the heat-affected zone accompanied by high strains in that region (Ref. 1). The mechanisms proposed as causes of low ductility include the presence of brittle grain boundary films formed by either liquation or solid state reactions during welding (Ref. 2-5); a change in deformation mode from transgranular slip to grain boundary sliding (Ref. 6-8); intragranular precipitation forcing a disproportionate amount of localized deformation near grain boundaries (Ref. 9,10); and an embrittling reaction of oxygen with grain boundaries during heat treatment (Ref. 11-14). There is evidence supporting each of these, and it seems certain that one or more mechanisms could be operating in the heat-affected zone thus contributing to cracking.

High strains in the heat-affected zone are a result of residual welding stresses, thermal expansion and contraction, and aging contraction. It is the latter which is unique to certain precipitation hardening alloys and which has been proposed by several investigators (Ref. 2,6,15,16) to be a contributing factor to strain-age cracking. Aging contraction in nickel base superalloys is a result of the precipitation of gamma prime. This depletes the surrounding gamma matrix of aluminum and titanium and results in a decrease in the lattice parameter of the matrix, hence a net contraction. Blum has reported that this contraction is of the order of 0.001 in./in. in René 41 and 0.0005 in./in. in Inconel X-750 (Ref. 15). These strains hinder the relaxation of the already high residual stresses in the heat-affected zone. The combination of high residual stress and low ductility is believed to cause cracking.

However, the relationship of aging contraction to residual stress relaxation and strain-age cracking is not well understood. One reason for this lack of understanding is that the aging reaction is extremely rapid in alloys susceptible to strain-age cracking, and therefore it is difficult to isolate the structural changes occurring during precipitation. In addition contraction is believed to be associated with either the early stages of precipitation or pre-precipitation (clustering) stages.

The purpose of this investigation was to determine what microstructural changes occur in the heat-affected zone and base metal during welding and postweld heat treatment, and to determine how these relate to the relaxation of residual welding stresses. This was accomplished by exposing specimens to simulated heat-affected zone thermal cycles and then studying the resulting structural changes using transmission electron microscopy. The response of restrained weldments to postweld heat treatment is determined by constant strain, isothermal aging of both base metal and simulated heat-affected zone specimens. In this manner the kinetics of stress relaxation was measured and the resulting structural changes were studied. The results provide a better understanding of the role of gamma prime precipitation in strain-age cracking in nickel base superalloy weldments.

Material

The material chosen for this study was René 41. This precipitation hardening, nickel base superalloy has found considerable application in gas turbine engines at temperatures up to about 1650 F. However, its weldability has been limited by susceptibility to strain-age cracking under conditions of high restraint.

The Rene 41 used in this investigation was prepared according to conventional mill practice, and was supplied in the form of 0.050 in. sheet. The chemical analysis of the as-received material is shown in Table 1. Prior to testing, all samples were solution annealed at 1950 F for thirty minutes and water quenched.

Experimental Procedure

Tensile specimens 0.050 × 1.0 × 4.0 in. were machined with a reduced section 0.50 × 1.5 in. as indicated in Fig. 1. Two preweld heat treatments were employed in this investigation. One set of specimens was solution annealed by individually heating to 1950 F in the R.P.I. Gleeble (Ref. 17) holding 5 min, and water quenching. A special quenching apparatus was employed which produced quench rates in excess of 50,000 F/s. This rapid quench rate produced specimens of uniform hardness and precluded precipitation of gamma prime during quenching. A second set of specimens was given the following overaging heat treatment (Ref. 5,18):

1. Solution treat in a furnace at 1975 F for ½ h, cool at 3 to 8 F per min to 1800 F.
2. Hold at 1800 F for 4 h, cool at 3 to 8 F per min to 1600 F.
3. Hold at 1600 F for 4 h, cool at 3 to 8 F per min to 1400 F.
4. Hold at 1400 F for 16 h, air cool to room temperature.

Some specimens from each set were exposed to simulated heat-affected zone thermal cycles in the Gleeble (peak temperature = 2380 F), and the balance retained in the as

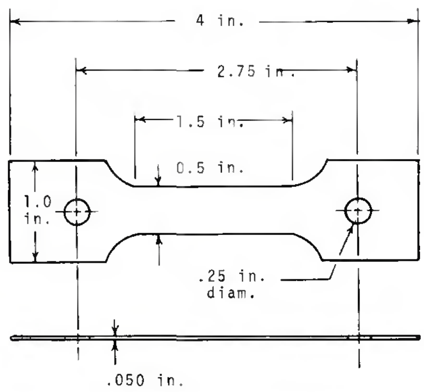

Fig. 1 — Tensile specimen configuration for isothermal stress relaxation tests

Table 1 — Rene 41 Composition

C	Mn	Si	Cr	Ni	Co	Mo	Nb	Fe	Ti	Al	B	Zr
0.09	0.02	0.26	18.94	51.21	11.10	9.68	0.03	3.12	3.11	1.48	0.005	0.01

heat treated condition. Thus four sets of specimens were available for further stress relaxation studies, two as heat treated and two thermally cycled after heat treatment. These represented both base metal and heat-affected zone for each of the two preweld heat treatments.

The stress relaxation behavior of each of these was determined by subjecting sheet tensile specimens to isothermal stress relaxation tests in the Gleeble. Specimens were heated rapidly to a predetermined temperature, then subjected to a predetermined elongation at a rate of 1.0 in./s. Once strained they were held at constant temperature and restrained from further change in length, while the stress level within the specimen was continuously monitored by recording the output of the load cell on a direct developing oscillograph. The elongation was predetermined with the aid of a differential-screw device previously described by Savage and Krantz (Ref. 19). This device limits the permissible travel of the movable jaw, thus limiting the total strain introduced in the test specimen. Stress relaxation tests on solution annealed base metal were conducted at 1400, 1600, 1700 and 1800 F. All additional stress relaxation tests on samples subjected to other prior heat treatments were conducted at 1600 F.

Samples representing each of the four conditions were examined by transmission electron microscopy both before and after isothermal stress relaxation testing. Representative sections of specimens were prepared by mechanical grinding to a thickness of 0.004 to 0.006 in. Discs 0.125 in. in diameter were then punched from these sections and electrochemically thinned by jet polishing techniques until a small hole formed at the center of the disc. Thinning was performed in a solution of 20 % perchloric acid — 80 % ethyl alcohol cooled to 32 F using 50 mA and 10 V dc.

Results and Discussion

Insothermal Stress Relaxation Studies

Solution annealed specimens were subjected to isothermal stress relaxation tests at 1400, 1600, 1700, and 1800 F. The change in stress level as a function of time is shown in Fig. 2. Note that the initial stress in each specimen was in the range of 55 to 60 ksi. At each temperature the stress initially decreased, and the rate of this initial decrease in stress increased with increasing temperature. At each temperature the stress reached a minimum and then began to increase. The period of increasing stress continued for several seconds after which the stress again decreased (except at 1400 F where it remained constant). At 1800 F a minimum of 22 ksi was reached after 2.0 s followed by an increase to 29 ksi after about 20 s. Stress then decreased continuously to 23 ksi when the test was interrupted after 100 s. At 1600 and 1700 F minimums of 47 ksi and 32 ksi, respectively, were reached at 0.3 s. Maximums of 54 ksi at 1600 F and 44 ksi at 1700 F occurred after 10 s. At 1400 F there was essentially no relaxation, but after about 1.0 s the stress began to increase to a maximum level of 60 ksi, at which it remained for the 200 s duration of the test. This behavior is similar to that observed by Dix in Inconel X-750 (Ref. 9) and by Duvall and Owczarski in Inconel 718 (Ref. 20), although the latter investigation involved much longer stress relaxation times.

An explanation of this behavior requires an understanding of the stress relaxation process. Stress relaxation, or the decrease in stress occurring at constant deformation, is a process related to creep. If a tensile specimen is held at a total strain ε at an elevated temperature where creep can occur, then

$$\varepsilon = \varepsilon_e + \varepsilon_p = \sigma/E + \varepsilon_p \qquad (1)$$

where

ε = total strain
ε_e = elastic strain
ε_p = plastic (creep) strain
σ = stress
E = elastic modulus at the test temperature

If the material creeps (ε_p increases), and at the same time the total strain remains constant, then it is necessary for the elastic strain to decrease. Therefore, the stress required to maintain the total strain decreases with time as creep progresses.

For a given material, the stress relaxation behavior will be determined by the temperature and the stress level, just as the creep behavior is determined by these parameters. Therefore, stress relaxation will be more rapid as the test temperature is increased as a result of increased creep rate. At relatively high test temperatures the stress initially decreases at a rapid rate. However, as the stress level drops the transient creep rate decreases with time, and therefore the stress relaxation rate levels off.

For example, one analysis shows that if creep can be described by the equation,

$$\varepsilon = B\sigma^n \qquad (2)$$

where ε = creep rate and B and n are empirical constants, then the time required to relax the stress from the initial stress σ_o to σ is given by (Ref. 21,22)

$$t = \frac{1}{BE(n-1)\,\sigma^{n-1}}\left[1-\left(\frac{\sigma}{\sigma_o}\right)^{n-1}\right] \qquad (3)$$

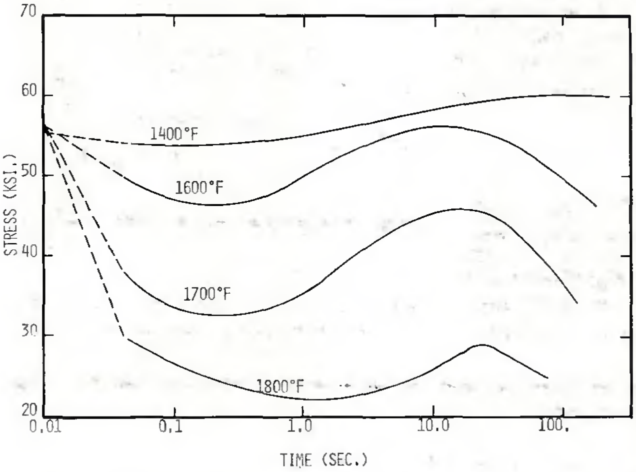

Fig. 2 — Effect of time and temperature on the isothermal stress relaxation behavior of solution annealed René 41

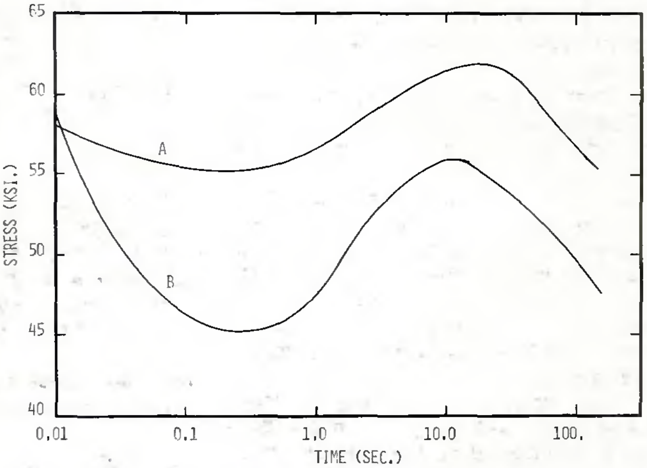

Fig. 3 — Stress relaxation behavior at 1600 F of a simulated HAZ specimen produced from solution annealed material (curve A) compared to a solution annealed base metal specimen (curve B)

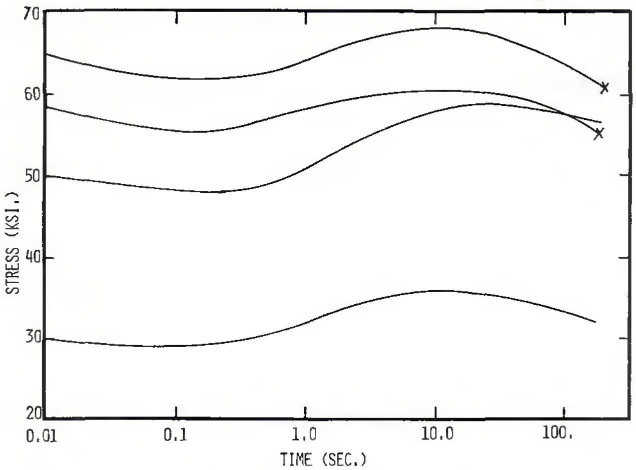

Fig. 4 — Stress relaxation behavior at 1600 F of simulated HAZ specimens produced from solution annealed base metal (x indicates specimen failed)

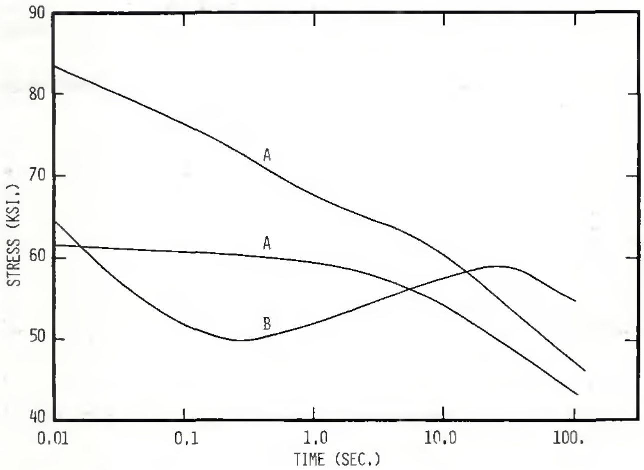

Fig. 5 — Stress relaxation behavior at 1600 F of overaged base metal specimens

However, in a precipitation hardening alloy, when stress relaxation occurs simultaneously with aging reactions, the situation is much more complex. First, aging produces a microstructure which impedes dislocation motion and thereby slows the creep rate. In addition, if aging causes a matrix contraction while the total elongation remains constant, the gage length of a specimen is effectively shortened. The test in this case is not a true constant strain test but rather a constant elongation test, and Eq. (1) can be rewitten as

$$\varepsilon = L_t/L_o = \sigma/E + \varepsilon_p \qquad (4)$$

where L_o = gage length at any time
L_t = total elongation

then,

$$\sigma = E(L_t/L_o) - \varepsilon \qquad (5)$$

During the stress relaxation test, ε_p increases as a result of creep thus causing stress to decrease. However, L_0 is decreasing as a result of aging contraction and thus tends to cause the stress to increase. The relative rate of change of ε_p (the creep rate) and L_0 (the aging contraction rate) then determines how the stress level changes with time. If aging contraction occurs rapidly relative to creep then the stress may actually increase.

This understanding of stress relaxation during aging helps to explain the behavior observed in Fig. 2. The initial rate of stress relaxation increases with increasing temperature as a result of increasing creep rate. As gamma prime begins to precipitate rapidly, aging contraction becomes the dominant factor in determining the change in stress level, thus the stress increases. Finally, as the rate of precipitation decreases, creep again becomes the dominant mechanism, and the stress again decreases.

These results indicate that aging has a significant effect on the level of stress in the solution annealed René 41 base metal. Aging contraction increases the stress level, thereby delaying relaxation. In this manner aging contraction in the base metal contributes to maintaining the high degree of restraint necessary for strain-age cracking.

All further stress relaxation tests were conducted at 1600 F. This temperature was chosen because it resulted in large contraction stresses, and because relatively high stresses were sustained for over 100 s at temperature. Specimens solution annealed and then exposed to a simulated heat-affected zone thermal cycle were tested at 1600 F. Figure 3 compares the isothermal stress relaxation behavior (for approximately equal initial stress levels) of a heat-affected zone specimen and a solution annealed base metal specimen. The initial stress relaxation rate was lower for the heat-affected zone specimen and resulted in a higher minimum stress after 0.3 s. Both experienced an increase in stress between 0.3 and 20 s, but the maximum stress was 62 ksi in the heat-affected zone specimen compared to 55 ksi in the base metal. Although the stress finally begins to decrease in both specimens it should be noted that after 200 s, the stress in the heat-affected zone specimen was still 55 ksi.

The results of additional tests on heat-affected zone specimens are shown in Fig. 4. Although these were tested at different initial stress levels the general shape of the curves is the same. However, those specimens tested at high initial stress levels (64 and 58 ksi) failed after about 180 s. Those tested at lower initial stress levels did not fail in similar time periods. These test conditions simulate the conditions experienced by the heat-affected zone of actual welds during heating to the stress relieving temperature, i.e., high residual stresses present as the weldment is heated through the temperature at which rapid aging occurs. Figure 4 clearly shows how this set of conditions can lead to cracking.

Gamma prime precipitation has been shown to be at least partially responsible for maintaining a high stress level during isothermal stress relaxation of both solution annealed base metal and simulated HAZ specimens. To reduce the effects of precipitation and thereby facilitate more rapid stress relaxation it has been suggested that René 41 be overaged prior to welding.

The results of stress relaxation tests performed on overaged specmens are shown as curves labeled A in Fig. 5. Curve B in Fig. 5 represents a solution annealed spcimen and is presented for comparison. For an initial stress of 84 ksi the initial stress relaxation rate in overaged René 41 was greater than that of the solution treated material subjected to an initial stress of 64 ksi. However, with comparable initial stress levels the initial rate of stress relaxation was less in the overaged than in the solution annealed material.

Furthermore, the averaged René 41 did not exhibit a period of increasing stress as did the solution annealed material. In fact, the stress in the overaged material relaxed continuously and was about 10 ksi less than the stress in the solution annealed material after 100 s at 1600 F. Even the specimen tested at 1600 F with an initial stress level of 84 ksi relaxed to a lower stress level than the solution annealed specimen after 14 s. In the latter stages of the tests (i.e. after approximately 20 s at 1600 F) the rate of relaxation appeared to be greater in the overaged René 41 than in the solution annealed material.

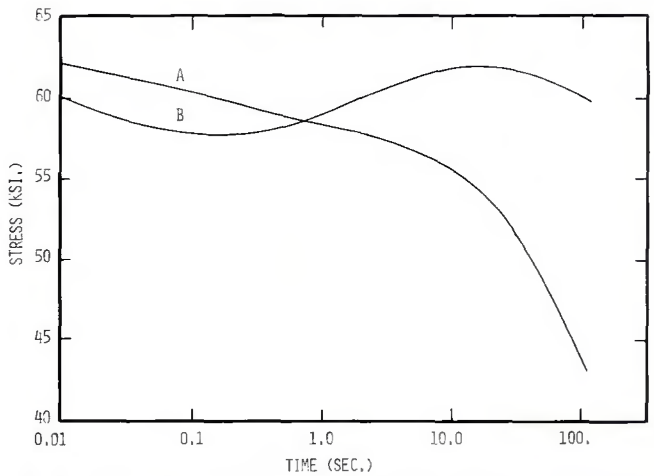

Fig. 6 — Stress relaxation behavior at 1600 F of a simulated HAZ specimen produced from overaged base metal (curve A) compared to an overaged base metal specimen (curve B)

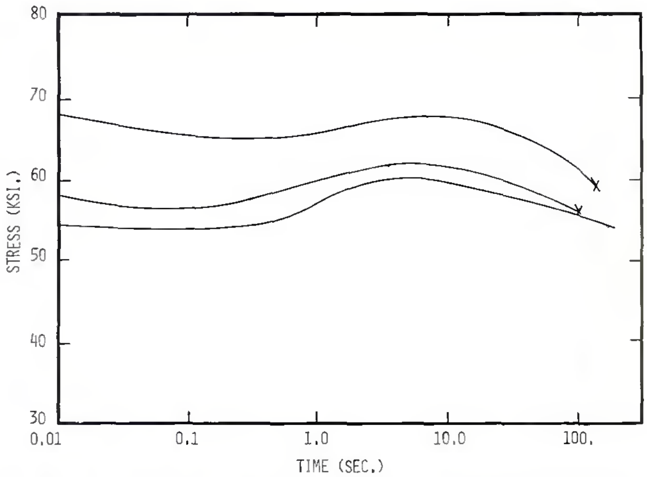

Fig. 7 — Stress relaxation behavior at 1600 F of simulated HAZ specimens produced from overaged base metal (x indicates specimen failed)

These tests indicate that overaging does have the beneficial effect of permitting more rapid stress relaxation in the base metal. Since aging contraction does not occur, no stress increase is experienced, and the more stable overaged structure allows continuous stress relaxation.

However, if the overaged René 41 is exposed to a simulated heat-affected zone thermal cycle prior to testing, the results are markedly different. Figure 6 compares the stress relaxation behavior at 1600 F for a heat-affected zone specimen with that of overaged base metal at similar initial stress. As noted above, stress in the overaged specimen relaxed without interruption. However, the heat-affected zone specimen experienced a minimum stress at about 0.3 s and a period of increasing stress between 0.3 and 10.0 s. After reaching a maximum of 62 ksi the stress again experienced relaxation. This behavior is similar to that observed in heat-affected zone specimens produced in solution annealed René 41. The result was that the stress in the heat-affected zone specimen remained at a much higher level than that in the overaged base metal. In fact, after 100 s the stress was 15 ksi higher in the heat-affected zone specimen than in the base metal specimen.

The stress relaxation behavior of three overaged heat-affected zone specimens tested at different initial stress levels is shown in Fig. 7. Those tested at initial stress levels of 59 ksi and 68 ksi both failed in 100 to 200 s. Comparison of Fig. 4 and Fig. 7 points out that the stress relaxation behavior of the heat-affected zone is remarkably similar regardless of prior heat treatment, and that both the overaged and solution annealed heat-affected zones are susceptible to cracking at high initial stress levels.

The response of René 41 weldments to postweld heat treatment has been shown to be dependent on both the initial or preweld microstructure and on the thermal cycles experienced during welding. This behavior can be explained on the basis of the microstructural changes during preweld heat treatment, welding, and postweld heat treatment.

Transmission Electron Microscopy Studies

Figure 8a and 8b respectively are electron micrographs of specimens of the solution annealed base metal and the heat affected-zone produced by exposure to a thermal cycle with a peak temperature of 2380 F. Figure 8a shows the structure associated with a grain boundary in solution annealed René 41. The large, dark globular particles along the grain boundary are M_6C carbides. The grain interiors are essentially structureless except for dislocations (short dark lines). The broader diffuse bands are "extinction contours" which result from diffraction effects created by bending of the lattice planes or changes in thickness and do not represent true structural features of the specimen (Ref. 23,24).

Figure 8b shows the appearance of a grain boundary in a specimen after exposure to the simulated heat-affected zone thermal cycle. Note that the grain boundary is void of large carbides indicating that M_6C dissolves during the thermal cycle. This is consistent with reports that M_6C dissolved at temperatures of 2150 F and above (Ref. 25). The grain interiors in this specimen have a rough, mottled structure compared to Fig. 8a. This indicates that precipitation of very fine gamma prime has occurred. Electron diffraction studies confirmed the presence of gamma prime. It is apparent that the cooling rates experienced in the heat-affected zone are slow enough to permit some gamma prime precipitation to occur, while carbide precipitation is suppressed.

Figure 9a and 9b are electron micrographs of the overaged base metal, and the heat-affected zone structure created by exposing overaged René 41 to a thermal cycle with a peak temperature of 2380 F. Figure 9a shows the gamma prime and carbide structure which results from the overaging heat treatment. Gamma prime is in the form of large cubical precipitates which have like orientations within a grain of the matrix. The grain boundary contains a dark semicontinuous phase which is different in appearance from the carbides in the solution annealed material (Fig. 8a). This phase was identified by electron diffraction as an $M_{23}C_6$ carbide. From this it appears that during the overaging heat treatment M_6C carbides transform to $M_{23}C_6$.

Figure 9b shows the effect of exposing the overaged René 41 to a heat-affected zone thermal cycle. Note the significant change in structure which occurred. First, the overaged gamma prime has completely disappeared and the grain interiors have the mottled appearance characteristic of a distribution of extremely fine gamma prime. Thus it appears that the overaged gamma prime dissolves completely during the brief interval of exposure to temperatures above its solvus (approx. 1950 F). Then, during the cooling portion of the thermal cycle, gamma prime reprecipitates in much the same manner as that noted when solution annealed material was exposed to the same weld thermal cycle.

The second major structural change shown in Fig. 9b involves the dissolution of the $M_{23}C_6$ carbides at grain boundaries, leaving the boundaries free of precipitates. Since $M_{23}C_6$ becomes unstable in the 1700 to 1800 F range in René 41 (Ref. 25), this change in structure is not unexpected. The net result of exposure to this heat-affected zone thermal cycle is the production of an extremely fine intragranular distribution of gamma prime together with carbide-free grain boundaries. It is significant to note that this is essentially the same structure as in the heat-affected zone specimen produced from solution annealed René 41 (compare Figs. 8b and 9b).

Following isothermal stress relaxation tests several specimens were examined by transmission electron microscopy. Figure 10 shows a solution annealed base metal specimen tested at 1400 F. The early stages of creep are evident in this specimen in the form of dislocation pile-ups at grain boundaries. Note that dislocations appear to be paired-up within the pile-ups. This phenomenon can be explained on the basis of the gamma prime structure. Gamma prime precipitates as ordered Ni_3Al with titanium substituting for some of the aluminum. Since it is an ordered precipitate, a dislocation moving through gamma prime creates an antiphase boundary (APB). This boundary represents a significant increase in energy. The second dislocation destroys the APB thus reducing the energy. A minimum APB area and hence a minimum APB energy results when dislocations travel in pairs. Therefore, the presence of such pairs is additional evidence of the fact that gamma prime precipitation has occurred. Note also that the grain interiors have a finely mottled texture indicating the presence of fine gamma prime. The contraction which accompanies this precipitation causes the stress to remain at a high level during the test.

Of more significance, however, are the structural changes which occur in the heat-affected zone during stress relaxation. This region is susceptible to strain-age cracking, and simulated heat-affected zone specimens failed during stress relaxation tests when a high initial stress was applied. The structure of a simulated heat-affected zone specimen produced from solution annealed René 41 and then stress relaxation tested is shown in Fig. 11. Initial stress in this specimen was 64 ksi, and the specimen failed in 190 s. Figure 11 shows that within the grains gamma prime precipitates increased in size during the test (compare Fig. 11 with Fig. 8b). In addition, precipitation has occurred at the grain boundary in the form of individual carbides arranged in a semi-continuous network along the boundary. This phase was identified by electron diffraction as $M_{23}C_6$.

In the heat-affected zone produced from overaged René 41 a similar behavior was observed during stress relaxation. Figure 12 shows that gamma prime growth has occurred within grains, while $M_{23}C_6$ carbides precipitated as a grain boundary network. Thus, it is apparent that regardless of prior heat treatment the heat-

Fig. 10 — Transmission electron micrograph of a solution annealed base metal specimen after an isothermal stress relaxation test at 1400 F X50,000, reduced 29%

Fig. 11 — Transmission electron micrograph of a simulated HAZ specimen produced from solution annealed base metal after an isothermal stress relaxation test at 1600 F X50,000, reduced 24%

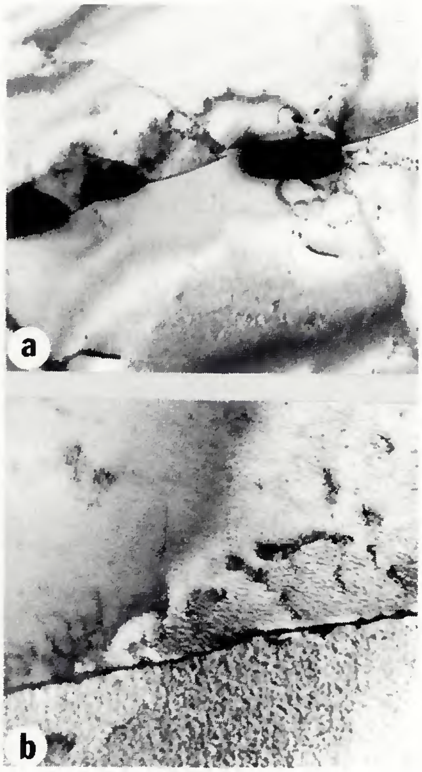

Fig. 8 — Transmission electron micrograph of (a) solution annealed base metal specimen and (b) simulated HAZ specimen produced from solution annealed base metal, X50,000, reduced 28%

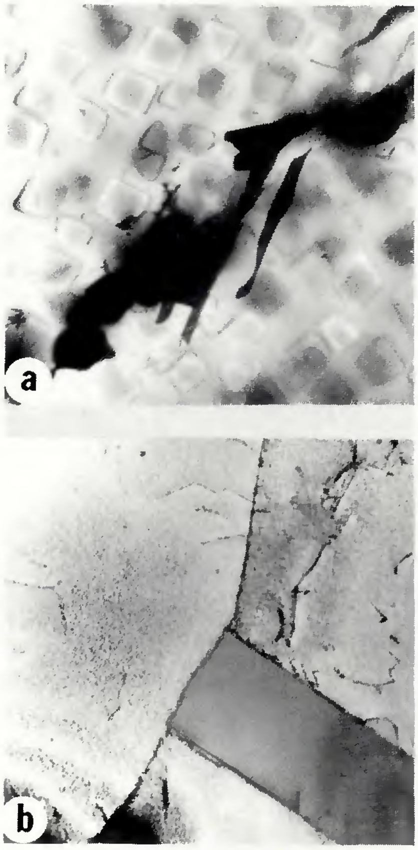

Fig. 9 — Transmission electron micrograph of (a) overaged base metal specimen and (b) simulated HAZ specimen produced from overaged base metal. X17,000, reduced 21%

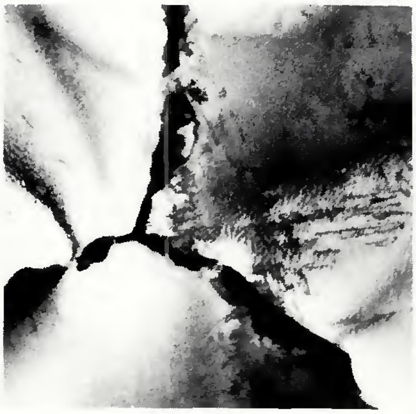

Fig. 12 — Transmission electron micrograph of a simulated HAZ specimen produced from overaged base metal after an isothermal stress relaxation test at 1600 F X50,000, reduced 28%

affected zone experiences aging during stress relaxation and the accompanying contraction causes the stress to remain at a high level. In addition, $M_{23}C_6$ grain boundary networks form. The combination of these effects can result in grain boundary fissures which lead to gross cracking.

Summary

Stress relaxation in the base metal of solution annealed René 41 is interrupted by a period of increasing stress. As a result the high stress level maintained in the base metal contributes to the restraint imposed on the weldment. The period of increasing stress is caused by the precipitation of extremely fine gamma prime. The aging contraction which accompanies this precipitation causes the stress to increase. By overaging prior to welding this behavior in the base metal can be eliminated and stress relaxation continues without interruption. Therefore, the restraint imposed by the base metal eventually decreases to a lower level than in the solution annealed base metal. In this manner overaging can help to reduce the propensity for strain-age cracking.

However, during welding the heat-affected zone in both solution annealed and overaged René 41 is exposed to thermal cycles which dissolve both the coarse gamma prime and the grain boundary carbides. During cooling, extremely fine gamma prime reprecipitates, although the cooling rates involved are apparently high enough to suppress reprecipitation of carbides. This leaves the heat-affected zone grain boundaries free of carbides and a fine distribution of gamma prime within the grains. It is important to note that this structure is independent of preweld heat treatment.

Consequently, postweld heat treatment, as simulated by isothermal stress relaxation tests, does not result in a rapid decrease in stress level in the heat-affected zone. On the contrary, the stress decreases slowly at first since creep is inhibited by the strengthening effect of the fine gamma prime precipitated during the cooling portion of the weld thermal cycle. Then an increase in stress occurs as a result of precipitation and growth of additional gamma prime during the isothermal stress relaxation test. Eventually stress continues to decrease again but the net result is that stress remains near its initial value for several minutes. During this time a semi-continuous grain boundary network of $M_{23}C_6$ begins to form. If the initial stress level is high enough fissures initiate grain boundaries and may lead to gross cracking.

Conclusions

1. In René 41 the regions of the heat-affected zone subjected to peak temperatures of 2380 F or greater will experience the following microstructural changes during fusion welding:

   a. If the material is overaged prior to welding, all gamma prime will be dissolved during the time that the temperature of this region is above the gamma prime solvus.
   b. Regardless of preweld heat treatment all grain boundary carbides will be dissolved in this region. In the solution annealed material these are M_6C carbides, while in overaged material they are $M_{23}C_6$
   c. Regardless of preweld heat treatment, extremely fine gamma prime will precipitate in this region on cooling.
   d. The as-welded structure of this region is thus essentially independent of preweld heat treatment.

2. The following conclusions were reached regarding the post-weld isothermal stress relaxation behavior in René 41.

   a. In solution annealed base metal, stress relaxation is interrupted by a period of increasing stress which results from the aging contraction accompanying precipitation of gamma prime.
   b. In unwelded overaged base metal, stress relaxation occurs without interruption.
   c. In the heat-affected zone, regardless of preweld heat treatment, the initial rate of stress relaxation is slowed as a result of the fine gamma prime distribution developed during the cooling portion of weld thermal cycles with peak temperatures above 2380 F.
   d. The precipitation and growth of additional gamma prime (with its accompanying aging contraction) during postweld heat treatment then causes a period of increasing stress.

3. Intergranular failure of heat-affected zone specimens during isothermal stress relaxation was associated with the formation of $M_{23}C_6$ grain boundary networks.

4. Overaging is not a completely effective means of increasing the capacity of the heat-affected zone to accommodate the strains associated with the relaxation of residual welding stresses.

Acknowledgements

The authors wish to acknowledge the Stellite Division, Cabot Corporation for supplying the material used in this study and the Fannie and John Hertz Foundation for the graduate fellowship support which made this work possible.

References

1. Baker, R. G. and Newman, R. P., "Cracking in Welds," *Metal Construction and British Welding Journal*, 1-4, Feb. 1969.
2. Chang, W. H., "Basic Causes of Cracking in René 41 Alloy Sheet," General Electric Report DM58302 (58AD-16), October 1958.
3. Morris, R. J., "René 41 — New Higher Strength Nickel-Base Alloy," *Metal Progress*, 76, (6), 67-70 (1959).
4. Hughes, W. P. and Berry, T. F., "A Study of the Strain-Age Cracking Characteristics in Welded René 41-Phase I," *Welding Journal*, 46 (8), 361s-370s (1967).
5. Berry T. F. and Hughes, W. P., "A Study of the Strain-Age Cracking Characteristics in Welded René 41-Phase II," *Welding Journal*, 48 (11), 505s-513s (1969).
6. Wu, K. C. and Herfert, R. E., "Microstructural Studies of René 41 Simulated Weld Heat-Affected Zones," *Welding Journal* 46 (1), 32s-38s (1967).
7. Owczarski, W. A. and Sullivan, C. P., "A Microstructure Study in a Welded Superalloy," *Welding Journal*, 43 (9), 393s-399s (1969).
8. Owczarski, W. A., Duvall, D. S., and Sullivan, C. P., "A Model for Heat-Affected Zone Cracking in Nickel-Base Superalloys," *Welding Journal*, 45, (4), 145s-155s (1966).
9. Dix, A. W., "An Investigation of the Deformation and Aging Characteristics of Inconel X-750 at Elevated Temperatures," Ph.D. Thesis, Rensselaer Polytechnic Institute, Troy, New York, June 1971.
10. McKeown, D., "Re-Heat Cracking in High Nickel Alloy Heat Affected Zone," *Welding Journal*, 50 (5), 201s-206s (1971).
11. Thompson, E. G., Nunez, S. and Prager, M., "Practical Solutions to Strain-Age Cracking in René 41," *Welding Journal*, 47 (7), 299s-313s (1968).
12. Prager, M. and Thompson, E. G., "A Study of the Mechanical Properties and Strain-Age Cracking of René 41 for F-1 Rocket Engine Applications," Rocketdyne Report R-7111, Sept. 1967.
13. Carlton, J. B. and Prager, M., "Variables Influencing the Strain-Age Cracking and Mechanical Properties of René 41 and Related Alloys," *Welding Research Council Bulletin*, No. 150, 13-23, May 1970.
14. Prager, M. and Sines, G., "A Mechanism for Cracking During Postweld Heat Treatment of Nickel-Base Alloys," *Welding Research Council Bulletin*, No. 150, 24-32, May 1970.
15. Blum, B. S., Shaw, P., and Wickesser, A., "Improved Methods for Welding Age Hardenable High Temperature Alloys," Technical Report ASD-TRD-63-601 (Republic Aviation Corp.), June 1963.
16. Schwenk, W. and Trabold, A. F., "Weldability of René 41," *Welding Journal*, 42 (10), 460s-465s (1963).
17. Batra, R., "An Investigation of the Fracture Behavior of Low Carbon Steels at Hot Working Temperatures," Ph.D. Thesis, Rensselaer Polytechnic Institute, Troy, New York, January 1971.
18. Lyman, T. (ed.) *Metals Handbook Vol. 6.* Welding and Brazing, p. 282, American Society for Metals, Metals Park, Ohio (1971).

19. Savage, W. F. and Krantz, B. M., "An Investigation of Hot Cracking in Hastelloy X," *Welding Journal,* 46 (1) (1966).

20. D'Annessa, A. T. and Owens, J. S., "Acoustic Emission Characteristics of Heat Treat Cracking in Rene 41 Weldments," *Welding Journal,* 51 (3), 156s-168s (1972).

21. Dieter, G. E., *Mechanical Metallurgy,* p. 368, McGraw-Hill Book Company, New York (1961).

22. Robinson, E. L., "High-Temperature Bolting Materials," *Proc. ASTM,* 48, 214-235 (1948).

23. Hirsch, P. B., Howie, A., Nicholson, R. B., Pashley, D. W., and Whelan, M. J., *Electron Microscopy of Thin Crystals,* p. 336, Butterworth & Co., London, England (1965).

24. Thomas, G., *Transmission Electron Microscopy of Metals,* p. 238, John Wiley & Sons, Inc., New York (1966).

25. Collins, H. E. and Quigg, R. J., "Carbide and Intermetallic Instability in Advanced Nickel-Base Superalloys," *Trans ASM,* 61 (1), 139-148 (1968).

WRC Bulletin No. 195
June 1974

"A Review of Bounding Techniques in Shakedown and Ratcheting at Elevated Temperatures"
by F. A. Leckie

"Upper Bounds for Accumulated Strains due to Creep Ratcheting"
by W. J. O'Donnell and J. Porowski

"A Review of Creep Instability in High-Temperature Piping and Pressure Vessels"
by J. C. Gerdeen and V. K. Sazawal

"Cyclic Creep — An Interpretive Literature Survey"
by Erhard Krempl

In recent years considerable effort has been devoted to developing a methodology based on detailed analysis to design structures which will operate under conditions of high temperature and periodic large thermal transients such that there exists a high level of confidence in their structural integrity. This methodology encompasses analytical methods, material behavior and design criteria. There has been excellent progress in all of these areas; however, it has become obvious that simplified procedures are needed, since the costs associated with performing a rigorous time-history analysis of a structure which is subjected to significant transient loadings while operating in the creep regime are very high, particularly if three-dimensional representation is required.

The Pressure Vessel Research Committee believes that progress in further developing this methodology will be assisted by the creation and wide distribution of a series of topical reports. This report series will serve to inform both by making available techniques and data which are relatively unknown in this country and by summarizing the current state of the art. In this manner the PVRC believes that technical progress can be stimulated and focused. However, the technology is in the developmental state and a full description of ancilliary information is often not available (e.g., a complete description of the creep and plasticity response of a candidate material). Also, sufficient confirmatory experimental data on structures of similar geometries, materials and operating conditions does not exist for many of the proposed design methods such as those contained in the following report. Experimental programs such as those sponsored by the USAEC are expected to provide such confirmation and define the range of applicability of proposed methods. Thus the topical reports published in *WRC Bulletin 195* are not recommendations by the PVRC to industry on the appropriate technique for pressure-vessel design at this time, but rather are topical reports of the status of an aspect of elevated temperature design at a point in time to aid the current development work in this field.

For structures other than semi-infinite right circular cylinders of uniform thickness subjected to continuous internal pressure and cyclic radial thermal gradients, no closed form analytical methods of demonstrated conservatism exist. The use of finite element time-history analysis has proven to be, on occasion, extremely expensive. Thus a clear and urgent need exists for the development of simplified analytical techniques to permit the economic evaluation of potential ratcheting configurations.

The concepts discussed in these reports are expected to have significant value in reducing the analytical efforts for the design of elevated temperature structures. At the current time insufficient experimental data are available to permit the PVRC to endorse the techniques for bounding the response of potential ratcheting problems. Further experimental data on the basic response of candidate materials as well as ratcheting experiments on typical structures are required. These reports are recommended to the industry as a source of potentially valuable techniques. It is believed that these proposals deserve detailed examination and should be tested against the body of experimental data as it becomes available.

The price of *WRC Bulletin 195* is $11.00. Orders should be sent to the Welding Research Council, United Engineering Center, 345 East 47th St., New York, N.Y. 10017.