LONG-TIME CREEP-RUPTURE PROPERTIES AND ELEVATED TEMPERATURE STABILITY OF D-979

J. Randolph Wood

Allegheny Ludlum Research Center,
Brackenridge, Pa. 15014

ABSTRACT

Long-time creep rupture tests were conducted on D-979 wrought bar in the fully heat treated condition. Forty smooth bar tests were run in the 1000°F to 1500°F temperature range for times ranging from 100 to 70,000 hours or more. A few notch rupture tests were also conducted in this temperature range for times up to 60,000 hours. Time to rupture and time to 1% total strain were tabulated and shown as log-log plots of stress versus time and as Larson-Miller parametric curves. The long-time parametric data showed good correlation with extrapolated data based upon shorter time tests, indicating a moderate degree of stability over the temperature range studied. The alloy displayed very good stability up to 60,000 hours or more at 1000°F, 1100°F and 1200°F and reasonably good stability at 1300°F or higher.

Discontinued and fractured creep-rupture specimens were sectioned and examined metallographically utilizing both light and electron microscopic techniques. Structural changes were noted for both long and short times at each temperature studied. Tentative identification of minor phases has been made by means of X-ray diffraction analysis of extracted residues and by electron probe microanalysis.

INTRODUCTION

Designers and users of gas turbine engines, pressure vessels and nuclear reactors are interested in the long-time stability of materials especially for applications requiring service lives of ten to twenty years or more. Long-time creep-rupture data on the order of 100,000 hours or more would be desirable for design purposes; however, because of the obvious difficulty in obtaining actual data for such long times, this information does not exist for most alloys. Most of the existing long-time data is based on extrapolations from shorter-time higher temperature data through the use of time-temperature parameters. There is a certain degree of uncertainty in using extrapolated data because of the instabilities and structural changes which can occur during long-time exposure at elevated temperatures.

To remove some of the doubt surrounding extrapolated data, the present study was designed to test D-979, a wrought, age-hardenable, nickel-base superalloy, for times up to 100,000 hours. The purpose was to determine the long-time creep and rupture strength and structural stability of D-979 wrought bar when placed under stress at operating temperatures up to 1500°F. Previous structural data on this alloy have been limited to a maximum of 1000 hours static exposure in the temperature range 1200 to 2000°F(1,2). Published creep-rupture data is also limited to less than 10,000 hours duration in the temperature range 1000 to 1500°F(3,4) with no apparent correlation with microstructures. This study will present creep-rupture data up to 70,000 hours exposure in the temperature range 1000 to 1500°F.

EXPERIMENTAL PROCEDURE

Wrought, five-eighths inch diameter bar was obtained from a commercial heat of D-979 with the following chemistry:

C	Mn	Si	Cr	Ni	Mo	W	Ti	Al	B	Fe
.039	.01	.02	14.6	45.0	4.3	3.8	3.2	1.0	.011	28.0

The heat was produced by air melting followed by consumable electrode remelting. Samples were solution heat treated at 1900°F-4 hours-water quench followed by intermediate aging at 1550°F-12 hours-air cool and final aging at 1300°F 16 hours-air cool. Smooth bar rupture specimens were machined to a .252" diameter by 1" long reduced section. Notch bar specimens were ground to a .252" diameter and notched with a stress concentration factor of K_t=3.7.

Creep-rupture tests were conducted in three-zone furnaces with temperature controlled within +3°F. Creep readings were taken daily by means of linear displacement transducers connected to extensometers which were attached to the shoulders of the specimens. Total creep strain versus time was plotted and time to 1 percent total strain was taken from this curve.

Following creep testing, specimens were sectioned longitudinally, mounted, polished, etched in a mixed acids solution (10 parts HNO_3, 10 parts acetic, 15 parts HCl) and examined microscopically in both reduced (stressed) and threaded ("unstressed") portions. Selected metallographic samples were examined in the microprobe and X-ray scanning images were obtained for a qualitative analysis of minor phase constitutents. Additional samples were prepared for electron microscopic studies by electrolytically etching in a solution consisting of 60 parts methanol, 36 parts ethylene glycol and 5 parts perchloric acid, followed by standard replication techniques utilizing collodian replicas shadowed with chromium.

Selected samples were chosen for residue analysis of extracted minor phases. Samples were carefully ground to remove surface oxides and electrolytically dissolved in a 10% HCl-methanol solution for 4 hours at a current density of .07 amps per cm^2. Extracted residues were washed in a 25% H_2SO_4 solution to remove any traces of undissolved matrix phase, air dried and weighed. Residues were analyzed with a Norelco diffractometer using Cu Kα radiation with a curved pyrolytic graphite crystal monochromator. Diffraction patterns were indexed and compared with known X-ray patterns of typical phases in superalloys.

RESULTS AND DISCUSSION

Long-time creep properties are shown in Figure 1 as a plot of log stress versus log time to one percent total strain. It was observed that a linear relationship existed at 1100°F up to about 50,000 hours. At 1200°F and higher, the slope of the curves increased negatively as would be expected. It is noted that, for the most part, the linear relationship still held. There was some degree of scatter in the creep strain data, so a perfect fit was unattainable. Some long time tests were discontinued after 50,000 hours before one percent strain was reached, therefore, one percent strain data was not available for every specimen.

Much of the scatter observed in the one percent strain can probably be attributed to apparent minor stabilities in the alloy. In many cases, particularly for the lower stress-longer time tests, the creep rate was so low that the creep strain readings continued to decrease with time even after thousands of hours. In fact, it was observed that negative strain readings were recorded for long periods of time. These negative strain readings were most likely due to additional precipitation or coarsening of gamma prime particles such that the density of the material changed and a slight contraction of the sample occurred. After this transformation period, which may have lasted for several thousand hours, the strain recordings gradually increased until positive values were again obtained. Because of these low creep rates and periods of negative strain, the time to one percent total strain showed a moderate degree of scatter.

The long-time rupture properties for D-979 are shown in Figure 2. It was observed that a good linear relationship existed at 1000°F and 1100°F up to 60,000 hours or more. At 1200°F a fairly good linear relationship existed up to about 40,000 hours. Increasingly negative slope occurred at 1300°F and higher, however, the curves showed no apparent break in slope up to about 30,000 hours indicating a moderate degree of stability at these higher temperatures. It is noted that four tests are still running each with approximately 70,000 hours accumulated time.

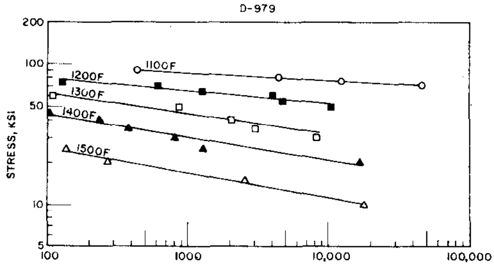

Figure 1. Long-time creep properties of D-979. Log stress(Ksi) versus log time (hours) to one percent total strain.

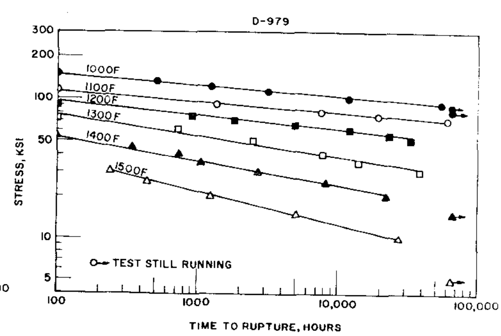

Figure 2. Long-time rupture properties of D-979. Log stress(Ksi) versus log time (hours) to rupture.

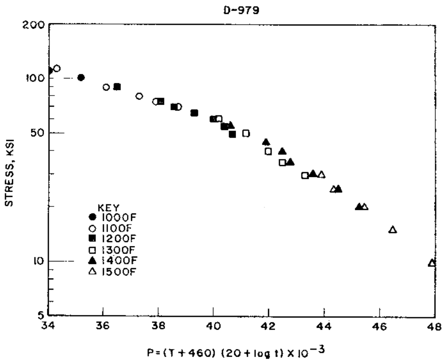

Figure 3. Larson-Miller Parameter Curve for D-979 Long-time Rupture Properties. Log stress(Ksi) versus $p=(T+460)\ (20+\log t)\ x\ 10^{-3}$.

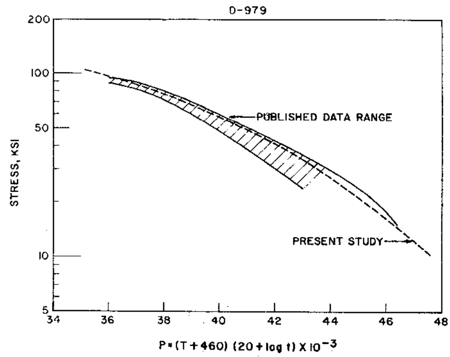

Figure 4. Larson-Miller Parameter Curve for D-979. Present study versus published results.

A summary of the long-time rupture properties in Figure 2 is shown in Figure 3 as a Larson-Miller parameter plot using a parametric constant of 20. This curve showed a very good fit and fell within a scatter band of previously published data on D-979(3,4) as indicated in Figure 4. These data indicate that higher temperature-shorter time tests have accurately described the lower temperature-longer time behavior of this alloy. The additional data from the four tests still in progress may help to confirm this observation.

Notch-rupture properties and smooth bar rupture ductility values after long times are shown in Table 1 in which both long and short-time rupture properties are shown. It was observed that at 1000°F and 1100°F the long-time rupture ductility (elongation and reduction-of-area values) was about 3 to 5 percent compared to 10 to 20 percent for the short-time (<100 hours) tests. At 1200°F, 1300°F and 1400°F the long-time ductility values ranged from about 11 to 56 percent compared to 9 to 20 percent for the short-time tests. At 1500°F both long and short time data were above the 18 percent level. Long-time rupture ductility of D-979, therefore, was somewhat low at 1000°F and 1100°F and improved considerably from 1200°F to 1500°F.

Long-time notch-rupture properties behaved similarly to the smooth bar ductility values. At 1000°F, 1100°F and 1200°F notch-rupture lives were significantly lower than their smooth bar counterparts. Differences at 1100°F and 1200°F were as much as one order of magnitude (e.g., at 1100°F, 810 hours notch life versus 7,600 hours smooth bar life). At 1300°F and higher, the notch-rupture lives were roughly two to four times the smooth bar lives at comparable stress levels. Thus, at 1200°F and below the long-time notch ductility of D-979 was adversely affected, whereas at 1300°F and above the material remained notch-ductile after very long times.

TABLE 1

D-979 LONG-TIME RUPTURE PROPERTIES

Temp. °F	Stress Ksi	Time to Rupture, hours Smooth	Notched	Smooth Bar Rupture Ductility Elongation %	R.A. %
1000	150	79	-	15.0	20.3
	100	12,450	2,500	3.0	3.9
1100	115	97	-	9.0	10.1
	80	7,600	810	3.0	3.9
	75	19,350	-	2.5	3.9
	70	60,700	-	5.0	5.6
1200	90	92	-	9.0	9.3
	65	-	2,460	-	-
	60	12,300	-	11.0	13.8
	55	23,550	2,275	18.4	23.1
	50	33,500	-	15.6	31.2
	30	-	51,300D*	-	-
1300	75	47	-	13.0	14.5
	40	7,800	29,100	31.9	37.6
	35	14,200	59,600D*	17.6	38.8
	30	39,100	-	22.8	36.2
1400	55	70	-	18.4	20.3
	25	8,180	30,200	25.4	47.5
	20	22,360	60,200	30.9	56.1
1500	30	240	-	45.0	26.9
	15	5,100	-	17.9	29.9
	10	27,700	44,800	23.9	21.6

*D - test discontinued at time indicated

Representative microstructures for the longer time tests at each temperature are shown in Figures 5 to 10. Figure A is a light micrograph and Figure B is an electron micrograph taken at 6000X. Each micrograph was taken on the reduced section away from the fractured end.

At 1000°F after 56,500 hours exposure (Figure 5), there was noted a continuous grain boundary phase with little change noted in the matrix compared to the as-heat treated condition. At 1100°F after 60,700 hours exposure (Figure 6), a slightly coarser grain boundary precipitate was observed with scattered, coarse precipitates noted in the matrix. At 1200°F after 51,300 hours exposure (Figure 7), the grain boundary phase was again visible with many fine needle-like precipitates observed in a Widmanstatten structure. At 1300°F after 59,600 hours exposure (Figure 8), a structure very similar to Figure 7 was observed. At 1400°F after 69,300 hours exposure (Figure 9), a coarse, semi-continuous, blocky grain boundary phase was observed with extremely fine, dense matrix precipitates noted, both acicular and irregular-shaped in nature. At 1500°F after 58,600 hours exposure (Figure 10), the grain boundary phase has essentially disappeared and the matrix precipitates became coarse, irregular-shaped particles. There were also observed several large needles with a precipitate-free zone around them. All microstructures contained scattered, large irregular-shaped particles of the MC type.

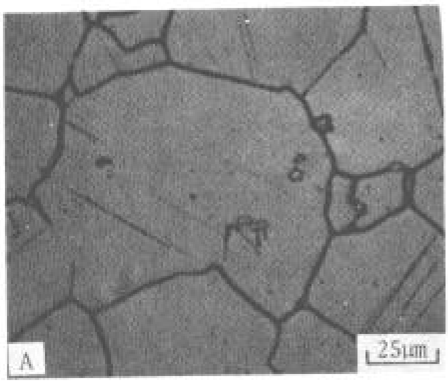

Figure 5. D-979 rupture sample. Temp-1000°F, Time - 56,500 hours, Stress-90 Ksi. Reduced section away from fracture.

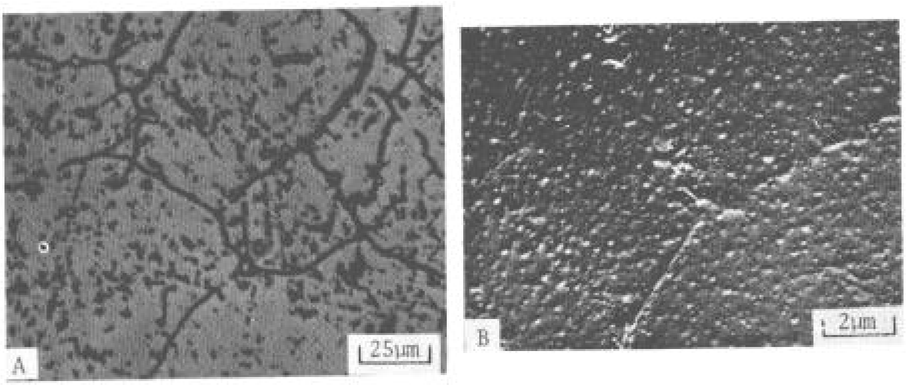

Figure 6. D-979 rupture sample. Temp-1100°F, Time - 60,700 hours, Stress-70 Ksi. Reduced section.

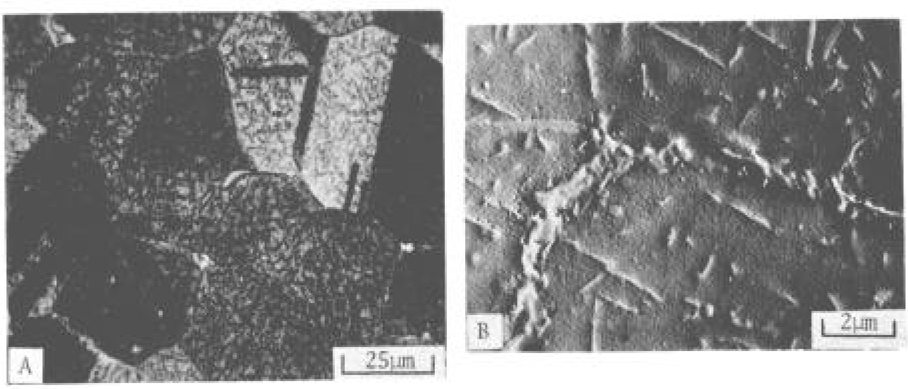

Figure 7. D-979 rupture sample. Temp-1200°F, Time - 51,300 hours, Stress-30 Ksi. Reduced section.

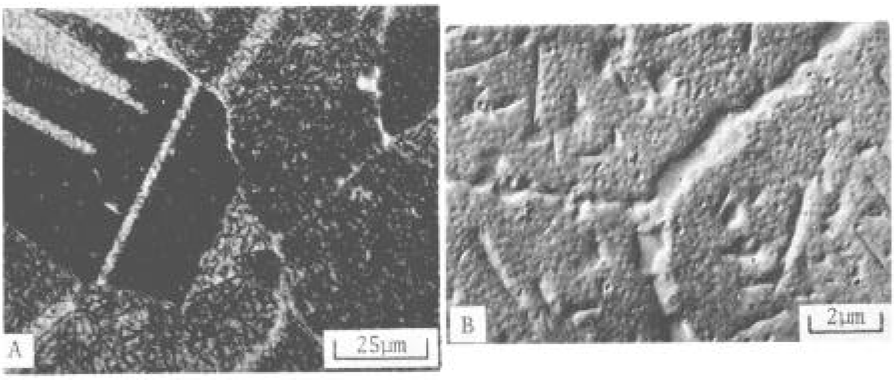

Figure 8. D-979 rupture sample. Temp-1300°F, Time - 59,600 hours, Stress-35 Ksi. Reduced section.

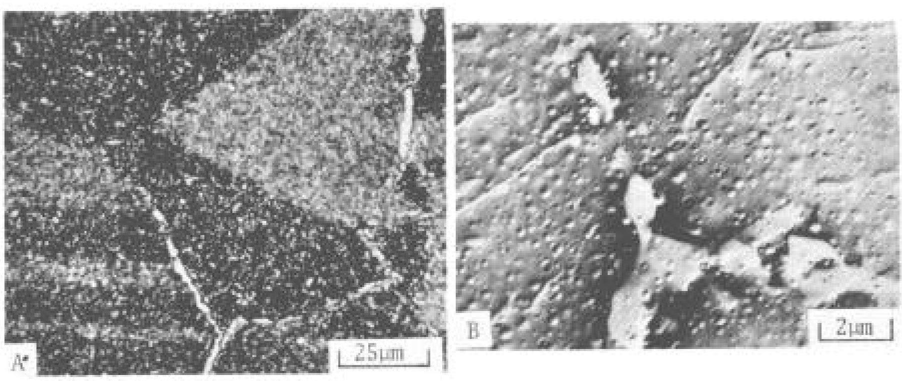

Figure 9. D-979 rupture sample. Temp-1400°F, Time - 69,300 hours, Stress-15 Ksi. Reduced section.

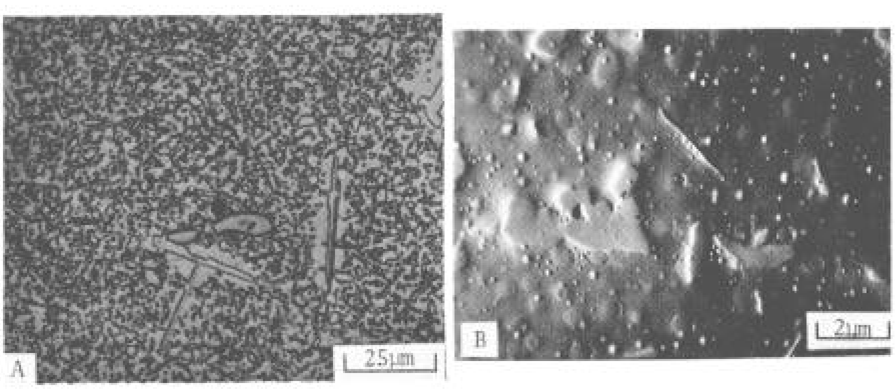

Figure 10. D-979 rupture sample. Temp-1500°F, Time - 58,600 hours, Stress-5 Ksi. Reduced section.

In general, it was observed that no noticeable differences in microstructure occurred between the stressed and "unstressed" areas of the long-time samples. For some of the shorter-time samples (i.e., less than 10,000 hours), the stressed or reduced section showed a greater amount of precipitation compared to the "unstressed" or threaded portion. This was observed at the lower temperatures where kinetics of reaction were slow and the additional strain in the reduced section accelerated precipitation. As will be discussed below, there was essentially no difference in residue analysis between the stressed and "unstressed" regions indicating similar amounts and types of precipitates.

X-ray diffraction analysis of extracted residues indicated that three phases were predominant as-heat treated and after high temperature exposure. Relative intensities of diffraction lines were determined for long-time samples at several temperatures by setting the intensity of the strongest line equal to 100 and ranking the strongest line of the other phases relative to it. The data are shown in Table 2.

TABLE 2

RELATIVE INTENSITIES OF EXTRACTED PHASES IN LONG-TIME CREEP-RUPTURE SAMPLES

Temp °F	Time Hours	Sample Location	Relative Intensities MC	Mu Phase	M_3B_2	Other*
As-heat treated		-	100	60	40	
1200	51,300	Stressed	20	100	10	
		Unstressed	15	100	25	
1300	59,600	Unstressed	5	100	15	
1400	41,200	Stressed	5	100	10	25(2.21Å)
1500	58,600	Stressed	5	100	5	25(2.21Å),80(2.15Å)

*Unknown phase

It is noted from these data that the predominant phase in the as-heat treated condition was MC followed by Mu and M_3B_2. After exposure at 1200°F for 51,300 hours, the predominant phase was Mu followed by M_3B_2 and MC. Similar results were noted for both stressed and unstressed portions of the rupture sample indicating little effect of stress on relative amounts of the three phases. After exposure at 1300°F for 59,600 hours, the Mu phase was even more pronounced compared to MC and M_3B_2. At 1400°F and 1500°F, Mu, MC, and M_3B_2 phases were roughly in the same proportion with perhaps a lesser amount of M_3B_2 observed.

Of particular interest at 1400°F and 1500°F was the emergence of an unidentified phase(s) which was stronger in intensity than either MC or M_3B_2, but weaker than Mu phase. These unknown phase(s) may be related to the irregular-shaped precipitates observed at 1400°F (Figure 9) and 1500°F (Figure 10). It was initially suspected that the unidentified phase(s) may have been Ni_3Ti eta phase or, perhaps, a transition phase between gamma prime and eta phase. Eta phase has previously been identified in D-979 at 1600°F and above (1,2) and the needle-like phase observed in Figure 10 was characteristic of eta phase. However, the "d" values of the unidentified phase(s) were found to be 2.15Å (strongest line) and 2.21Å (next strongest line) which did not correspond to eta phase nor to any other readily identifiable phase common to superalloys. Furthermore, due to the electrolyte used for extraction, it was likely that eta phase or a transition phase dissolved thus leaving no residue for analysis.

Another possible explanation for the presence of the unknown phase(s) could be a transition phase related to Mu phase. An unknown phase involving Mu phase has been observed in Pyromet 860 (5), an alloy similar to D-979. A chromium-iron-tungsten rich grain boundary phase has also been observed in D-979 (2) which precipitated and resolutioned in conjunction with Mu phase. This phase was tentatively identified as based on tetragonal Cr_7C_3 which does not correspond to the unknown phase(s) of the present study. Additional work is needed to more completely characterize the phase(s).

Microprobe X-ray scanning images at 1000X of the samples observed in Figures 8 and 9 showed the blocky and continuous intergranular precipitates were rich in Cr, Mo, and W and depleted in C, Ni and Ti. No other major elements or phases were detected by the microprobe. The areas rich in Cr, Mo, and W suggested that Mu phase or M_3B_2 borides were present at the grain boundaries. It was not clear which phase was predominant since both can contain these elements in solution. The absence of carbon in the microprobe scan and the X-ray residue analysis suggested that grain boundary carbides of the Type $M_{23}C_6$ or M_6C were not present in D-979 after long exposures at elevated temperatures. This supports previous work which detected no $M_{23}C_6$ in D-979 (1,2).

SUMMARY AND CONCLUSIONS

Long-time creep-rupture properties of D-979 wrougnt bar showed good stability at 1000°F, 1100°F, and 1200°F and moderate stability at 1300°F, 1400°F, and 1500°F. A Larson-Miller parametric plot of long-time rupture properties showed excellent correlation with shorter time-higher temperature data previously published. At 1200°F and below, some degree of notch sensitivity was observed. At 1300°F and above, the alloy was completely notch-ductile. Minor phases identified in D-979 after long-time exposure (up to 60,000 hours) in the 1000°F to 1500°F temperature range were MC carbide, M_3B_2 boride, and Mu phase, plus an unidentified phase found only at 1400°F and 1500°F. Very little structural differences were noted between stressed and unstressed portions of the rupture samples.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the management at Allegheny Ludlum Industries for permission to publish this paper. Special thanks are due to Special Metals Corporation for helpful discussions and assistance with some of the laboratory work.

REFERENCES

1. E. A. Parker, "An Investigation of the Phases and Phase-Temperature Dependence of a Nickel-Iron-Base High-Temperature Alloy," Master's Thesis, Rensselaer Polytechnic Institute, Troy, N.Y. Sept., 1959.
2. B. A. Ewing, R. P. Dalal, and P. B. Mee, Journal of Materials, Vol. 5, No. 3, Sept. 1970, p. 651.
3. The Elevated Temperature Properties of Selected Superalloys, ASTM Publication DS-7-S1, ASTM, Philadelphia, Pa., July, 1968, p. 279.
4. Aerospace Structural Metals Handbook, Vol. 4, Belfour Stulen, Inc., Traverse City, Mich., March, 1968, p. 4109.
5. G. N. Maniar, D. R. Muzyka, C. R. Whitney, International Symposium on Structural Stability in Superalloys, Vol. 1, Sept., 1968, p. 391.