Effect of Gamma-Gamma Prime Mismatch, Volume Fraction Gamma Prime, and Gamma Prime Morphology on Elevated Temperature Properties of Ni, 20 Cr, 5.5 Mo, Ti, Al Alloys

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This study was designed to provide a critical test for the postulate that the mismatch between the lattices of austenite (γ) and the age-hardening gamma-prime (γ') precipitates and the resultant coherency strains have a significant influence on the elevated temperature, particularly stress rupture, properties of a nickel-base superalloy. Two experimental alloys with a base analysis of Ni, 20 Cr, 5.5 Mo were designed with variable titanium and aluminum additions. To discern the effect of mismatch, an alloy without molybdenum was also experimented with. By manipulating the mismatch and volume fraction γ' by heat treatment and chemistry, it was shown that a lower γ-γ' mismatch indeed is beneficial to stress rupture life. Importance of volume fraction γ' on this elevated temperature was also established.

NORDHEIM and Grant[1] correlated the mismatch between the lattices of austenite (γ) and age-hardening gamma-prime (γ') precipitates with creep strength in 80Ni-20Cr containing titanium and aluminum as hardeners. Bigelow *et al.*[2] and Mihalisin and Decker[3] suggested that the γ-γ' mismatch and resultant coherency strains contributed to the age-hardening in commercial nickel-base superalloys and experimental Ni-Ti-Al alloys, respectively. Very recently, however, Ansell and his colleagues[4-6] questioned the importance of coherency strains in γ' strengthened alloys. In a systematic study designed to provide a critical test for the beneficial role of coherency strains, Decker and Mihalisin[7] showed that by manipulating γ-γ' mismatch, and thus the coherency strains, aged hardness of Ni-Al alloys could indeed be controlled. In a similar investigation, Maniar *et al.*[8] correlated γ-γ' mismatch with stress rupture life in a series of Ni-Cr/Fe-Ni-Cr experimental alloys containing titanium, aluminum and, in a few cases, molybdenum. Although trends were developed to establish the critical influence of mismatch, effect of other equally important variables, such as volume fraction γ' and γ' morphology were not controlled. This study was designed to discern the effect of mismatch from some of these variables.

EXPERIMENTAL PROCEDURE

Material

Three (3) 17-lb heats, with Ni, 20Cr base, one without molybdenum and two with 5.5 Mo, were melted by vacuum induction practice. The titanium and aluminum were varied to yield 3.0 (in two heats) and 4.5 wt pct total hardener content, at a constant Ti/Al ratio of two. The C, Mn, Si, P, S and Fe were kept as minimal as possible.

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The chemical analyses of the alloys studied are given in Table I. A trace quantity of boron was added to all three heats for ease in hot working. The heats were cast into 2 by $2\frac{3}{8}$ in. ingots and homogenized at 2100°F for 6 hr. The homogenized ingots were forged to $\frac{3}{4}$ in. sq bars at 2050°F. The processing of the three heats was maintained as uniform as possible.

X-ray Diffraction Analysis

The γ-γ' mismatch determinations were made in accordance with the procedure described earlier.[8] A few samples from heat V00247 were overaged to precipitate γ' of fairly coarse size. The a_0 measurements made on the overaged solid samples were compared with the similar data obtained from the extracted γ'.

The lattice parameter values, as well as the mismatch data, are given in Table II and are plotted in Figs. 1 and 2.

Volume Fraction γ'

For volume fraction γ' determinations, the samples were subjected to electrolysis in a solution of 20 pct H_3PO_4 -H_2O and wt fraction γ' calculated for each sample. The wt fraction γ' was converted to volume fraction γ' for each sample.[8] These data are listed in Table III.

Stress Rupture and Elevated Temperature Tensile Testing

As in the previous study,[8] the alloys were tested for stress rupture properties. These data are listed in

Table I. Chemical Analyses of the Heats Studied in wt pct

Heat No.	Cr	Ni	Mo	Ti	Al	B	Ti/Al	Ti + Al	Wt Fraction Ti
V02802	20.07	76.16	–	2.02	0.92	0.0046	2.20	2.94	0.687
V00246	19.60	70.90	5.38	1.98	0.87	0.0055	2.28	2.85	0.695
V00247	19.85	70.12	5.46	2.90	1.35	0.0054	2.15	4.25	0.682

Table II. Lattice Parameters of γ and γ', and Pct Mismatch

Heat Treatment	$\gamma\ a_0$, Å*			$\gamma'\ a_0$, Å*			Pct Mismatch		
	V02802	V00246	V00247	V02802	V00246	V00247	V02802	V00246	V00247
1950°F/1 hr/A.C. (A)	3.5636	3.5800	3.5859	No Residue	No Residue	3.5914	X	X	−0.15
A + 1350°F/15 hr/A.C.	3.5628	3.5795	3.5859	3.5920	3.5942	3.5915	−0.82	−0.41	−0.16
A + 1350°F/50 hr/A.C.	3.5619	3.5797	3.5847	3.5877	3.5907	3.5902	−0.72	−0.31	−0.16
2050°F/1 hr/W.Q. (B)	3.5656	3.5818	3.5890	No Residue	No Residue	No Residue	X	X	X
B + 1350°F/15 hr/A.C.	3.5634	3.5811	3.5859	3.5908	3.6032	3.5993	−0.76	−0.62	−0.37
B + 1350°F/50 hr/A.C.	3.5626	3.5791	3.5850	3.5884	3.5917	3.5921	−0.73	−0.35	−0.19
B + 1550°F/50 hr/A.C.	3.5624	3.5792	3.5871	3.5877	3.5921	3.5916	−0.71	−0.36	−0.13
B + 1600°F/50 hr/A.C.	3.5636	3.5800	3.5862	3.5882	3.5922	3.5917	−0.69	−0.34	−0.11
B + 1650°F/50 hr/A.C.	3.5639	3.5810	3.5879	3.5890	3.5925	3.5908	−0.70	−0.32	−0.08
B + 1550°F/50 hr/A.C.	X	X	3.5871	X	X	3.5924†	X	X	−0.15
B + 1600°F/50 hr/A.C.	X	X	3.5862	X	X	3.5924†	X	X	−0.17
B + 1650°F/50 hr/A.C.	X	X	3.5879	X	X	3.5924†	X	X	−0.13

*Precision, ±0.0005Å.
†*In situ* measurements.

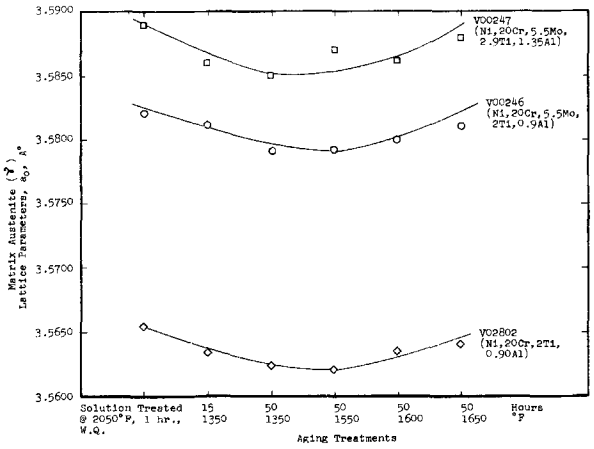


Fig. 1—Variation in matrix lattice parameter (γ-a_0) as a function of aging. (Treatments B, Table II only).

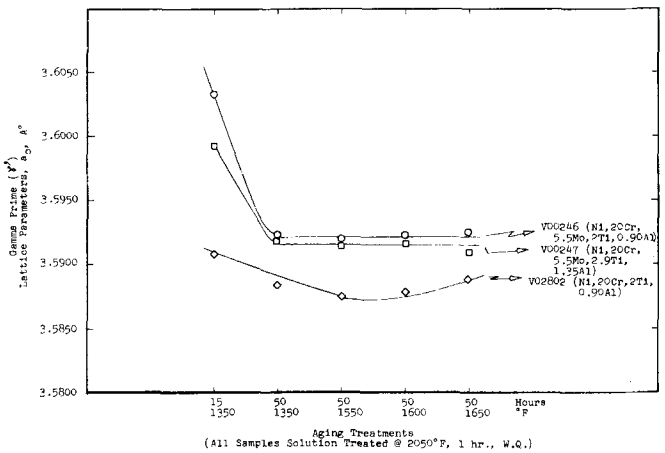


Fig. 2—Lattice parameter of gamma prime (γ'-a_0) vs aging treatment. (Treatments B, Table II only).

Tables IV to VI for 1200°F (60 ksi) and 1400°F (55 ksi) testing. The notch-smooth (K_t, the stress concentration factor, = 3.8) combination samples were utilized. For stress rupture testing, both the 1950°F solution treatment followed by air cooling and the higher temperature of 2050°F, water quenched were investigated.

The elevated temperature (1400°F) tensile strength data were obtained only from heats V00246 and V00247. These properties are given in Table VII.

Metallography

The samples were examined by optical and electron microscopy to characterize the age-hardening γ' phase. Samples were etched electrolytically in "G" etchant (12 H_3PO_4, 47 H_2SO_4, 41 HNO_3). Chromium shadowed parlodion replicas and carbon extraction replicas were examined in electron microscope.

DISCUSSION OF RESULTS

The Ni, 20Cr, 5.5Mo, 2Ti, 1Al analysis was selected, from the earlier work,[8] as the basis for this study. To ascertain the effect of volume fraction γ', another composition with the similar analysis but with increased hardener content (3Ti, 1.5Al) was selected. Previous work had shown a considerable effect of molybdenum, in this base analysis, on the mismatch. To discriminate the effect of mismatch at a constant volume fraction γ', one heat without molybdenum was utilized.

Lattice Parameters of Solid Block Samples: Matrix Austenite

The lattice parameters (a_0's) obtained from the solid samples, solution treated and aged, are given in Table II. Generally, the matrix austenite a_0 decreases as the alloys are aged at 1350°F, and this is related to the γ' precipitation. In the case of heat V02802 and V00246, the γ' starts to solution on overaging (1500°, 1600° and 1650°F for 50 hr). This results in the matrix a_0 reverting very close to the solution treated values, Fig. 1.

The significant effect of molybdenum can be seen by comparing the a_0's of V02802 (without molybdenum) and V00246 (with molybdenum). The matrix austenite a_0 of V00246 is increased considerably by the addition of the large molybdenum atom. The effect of the increased hardener content, in V00247 as compared to V00246, is

a relatively small increase in the matrix austenite a_0.

Lattice Parameter of Extracted γ' Precipitates

The lattice parameter (a_0) values of extracted γ' precipitates are plotted in Fig. 2 as a function of aging treatments. It is seen that the γ'-a_0 is dependent on composition, and the molybdenum addition has the effect of increasing the a_0; compare V00246 and V00247 vs V02802 (no molybdenum). The effect, however, is not as large on the γ' a_0 as it is on the γ a_0. This indicates that the molybdenum, at this level (~ 5.5 pct), is partitioned favorably in the γ lattice. Loomis[9] has made similar observations. The increased hardener content, in heat V00247 as compared to V00246, has little effect on the γ' a_0, indicating the composition is nearly the same. The increased hardener content has a pronounced effect on the volume fraction γ' precipitated.

A comparison of γ'-a_0's for 15 vs 50 hr at 1350°F, Fig. 2, aging temperature indicates lowering of lattice parameter in all three heats. This decrease is not quite as pronounced in heat V02802 as the other two heats. On overaging for 50 hr at 1550° to 1600°F, the γ' a_0's do not appear to change significantly. Heat V00247 shows a gradual decrease in a_0 values as the aging temperature increased from 1350° to 1650°F. Considering the precision of such a measurement, this decrease is not considered significant. This would, then, suggest that an equilibrium γ' forms at 1350°F after a 50 hr age. Effect of overaging differs in these heats in their rates of dissolution of γ', to be discussed later.

A Comparison of γ' a_0 Measurements (*In situ* vs Extracted γ')

In the overaged samples of heat V00247, the γ' precipitates were present in a sufficient quantity and were

Table III. Wt Pct and Volume Fraction γ'

Heat Treatment	Wt Pct γ'			Volume Fraction γ'		
	V02802	V00246	V00247	V02802	V00246	V00247
1950°F/1 hr/A.C. (A)	–	–	15.0	–	–	16.2
A + 1350°F/15 hr/A.C.	7.0	9.2	18.4	8.0	10.0	19.8
A + 1350°F/50 hr/A.C.	7.6	11.5	14.8	8.4	12.6	16.1
2050°F/1 hr/W.Q. (B)	–	–	–	–	–	–
B + 1350°F/15 hr/A.C.	7.5	8.5	15.8	8.3	9.4	17.2
B + 1350°F/50 hr/A.C.	8.5 (7.8)*	9.9 (10.0)	19.8	9.4 (8.7)	10.8 (10.9)	21.4
B + 1550°F/50 hr/A.C.	4.0	6.7	18.0	4.4	7.4	19.4
B + 1600°F/50 hr/A.C.	2.6	5.4	15.9	2.9	5.9	17.2
B + 1650°F/50 hr/A.C.	1.2	3.2	14.4	1.4	3.5	15.6

*Duplicate determinations shown in parentheses.

Table IV. Stress Rupture Data—Tested at 1200°F, 60 ksi

Heat No.	Treatment	Stress Rupture Data			Pct Mismatch	Volume Fraction γ'
		Life, Hrs.	Pct El.	Pct R.A.		
V02802	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	111	5.7	10.0	−0.82	8.0
	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	148	5.0	8.0		
V00246	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	2057	12.5	15.2	−0.41	10.0
	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	3011	12.4	14.0		
V00247	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	7266* 106	9.3	12.0	−0.16	19.8
	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	7266* 94	10.3	13.2		

*Test conditions changed after 7266 hr at 1200°F, 60 ksi to 1400°F, 55 ksi. The rupture life obtained at 1400°F, 55 ksi of 106 and 94 hr. Heat V00247 only.

Table V. Stress Rupture Data—Tested at 1200°F, 60 ksi

Heat No.	Treatment	Stress Rupture Data			Pct Mismatch	Volume Fraction γ'
		Life, hrs	Pct El.	Pct R.A.		
V00246	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	3166	8.8	14.0	−0.62	9.4
	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	4040	10.7	15.2		
V00247	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	5058* 99	12.1	15.2	−0.37	17.2
	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	5058* 83	9.0	16.0		

*Test conditions changed after 5058 hr at 1200°F, 60 ksi to 1400°F, 55 ksi. The rupture life obtained at 1400°F, 55 ksi of 99 and 83 hr. Heat V00247 only.

of suitable size to enable an analysis of superlattice reflections in solid samples. These were measured and the a_0's so obtained compared to the same from extracted γ' in Table II. The agreement between the two can be considered fairly reasonable to afford some degree of confidence in the mismatch data generated using the a_0 values of extracted γ'.

Percent Mismatch

The pct mismatch between the matrix austenite and the age-hardening γ' precipitate was calculated as in earlier work.[8]

Examination of the mismatch data, Table II, shows the following trends:

a) The 5.5 pct molybdenum addition has a pronounced effect on decreasing the mismatch value (compare V00246 and V02802). Molybdenum partitions in the γ lattice to a greater degree than the γ' lattice, and therefore will reduce the mismatch.

b) Increased hardener content also decreases the mismatch; compare V00247 vs V00246.

c) Comparing the mismatch values at 1350°F, the mismatch decreases with time (50 vs 15 hr). As was shown earlier and to be further discussed later, the γ' precipitation has reached an equilibrium at 50 hr aging time at 1350°F.

d) Heat V00247 showed γ' precipitates in solution treated condition at 1950°F, 1 hr, air cooled. The pct mismatch for this γ' is in agreement with that for the sample treated for 50 hr at 1350°F (-0.15 vs -0.19 pct).

e) The mismatch value for V00246 solution treated at 2050°F, 1 hr, air cooled + aged at 1350°F for 50 hr is approximately equal to the mismatch for V00247 solution treated at 2050°F, 1 hr, air cooled + aged at 1350°F for 15 hr. This information was utilized in designing heat treatment for stress rupture testing, to be discussed later, to discriminate the effect of mismatch from that of volume fraction γ'.

Volume Fraction γ'

The effect of molybdenum and increased hardener content on the volume fraction γ' is shown in Table III. The wt pct used to derive the volume fractions are also shown. The electrolyte used in this study has been reported[10] to yield markedly less γ' recovery, in comparison with other electrolytes, due to partial dissolution of γ'. For this study, however, all the experimental conditions being identical, it is believed that the volume fraction γ' values could be used to relate one composition with the other without jeopardizing the significance of the results.

Evaluating the data in Table III, it appears that molyb-

Table VI. Stress Rupture Properties at 1400°F, 55 ksi

Test	Heat No.	Treatment	Rupture Data			Grain Size	Pct Mismatch		Volume Fraction γ'	
			Life, Hr	Pct El.	Pct R.A.	ASTM	Before*	After*	Before*	After*
(a)	V00246	1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	18.9	18.3	21.9	3.0	−0.41	−0.31	10.0	8.0
		1950°F/1 hr/A.C. + 1350°F/15 hr/A.C.	20.4	21.3	26.1					
(b)	V00246	2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	34.5	21.3	27.3	2.0	−0.35	−0.34	10.8	7.5
		2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	36.5	25.4	26.1					
(c)	V00247	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	88.6	14.0	18.4	2.0	−0.37	−0.15	17.2	23.2
		2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	126.6	14.0	17.9					
(d)	V00247	2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	151.2	14.7	19.2	2.0	−0.19	−0.20	21.4	21.1
		2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	166.4	13.9	18.0					

*"Before" and "After"–Stress Rupture Testing.

Table VII. Short-Time Elevated Temperature Tensile Data; Test Temperature 1400°F

Heat No.	Treatment	0.2 Pct Y.S. psi	Tensile Strength psi	Pct El.	Pct R.A.	Pct Mismatch	Volume Fraction γ'
V00246	2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	68,000 74,500	105,000	26.2	27.4	−0.35	10.8
	2050°F/1 hr/W.Q. + 1350°F/50 hr/A.C.	67,000 72,500	100,000	28.3	31.3		
V00247	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	86,509 92,778	111,835	11.9	21.4	−0.37	17.2
	2050°F/1 hr/W.Q. + 1350°F/15 hr/A.C.	84,002 94,032	113,340	11.0	17.4		

denum increases but very slightly the volume fraction γ' in V00246 when compared to the same in V02802. Effect of molybdenum, however, is more pronounced in increasing the γ' solvus temperature and, therefore, in inhibiting the rate of dissolution. This is apparent from the volume fraction γ' data for these two heats in overaged conditions, 1550° to 1650°F, 50 hr. The γ' quantity decreases in V00246 as the overage temperature increases but not at the same rate as that in heat V02802.

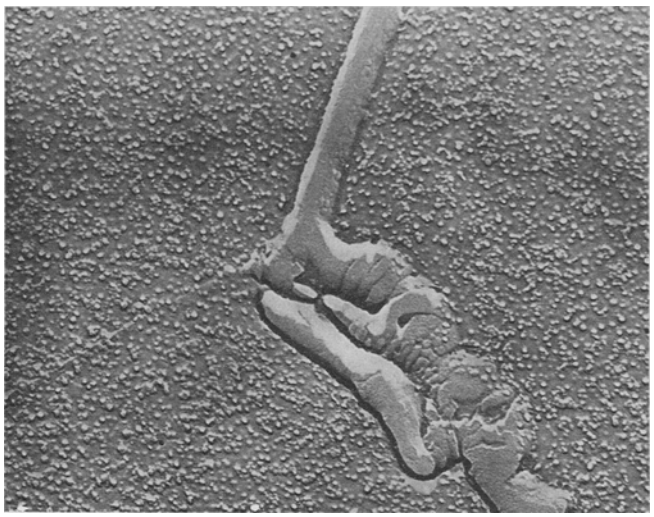
The effect of increased hardener content can be seen by comparing the volume fraction γ' for V00247 and V00246. The increased hardener content resulted in significantly higher volume fraction γ' in the former. Effect of increased (Ti + Al) on γ' solvus temperature was more pronounced than that due to molybdenum addition. Increase in overaging temperature from 1550° to 1650°F resulted in only a slight decrease in volume fraction γ'.

Stress Rupture Properties

As was stated earlier, the emphasis was placed on correlating the effect of mismatch on a high temperature mechanical property. This was done by manipulating heat treatment/chemistry to yield constant mismatch at varying volume fraction γ' or vice versa. Stress rupture data obtained at 1200°F, 60 ksi and 1400°F, 55 ksi are given in Tables IV to VI.

The molybdenum addition in heat V00246 (Table IV) decreased the mismatch to -0.41 pct as compared to -0.82 pct in V02802 (in 1950°F solution treatment) without significantly affecting the vol fraction γ', 10 and 8 pct, respectively. The improvement in rupture life at 1200°F, 60 ksi was quite significant (2500 hr vs 130 hr). In comparison, duplicate test samples from heat V00247 showed rupture life of 7266 hr at 1200°F, 60 ksi without failing. To subject these samples to a more critical test, the test temperature was increased to 1400°F with a slight lowering of stress (from 60 to 55 ksi). The test samples showed a further life of 100 hr at 1400°F, 55 ksi. Using the Larson-Miller parameter approach,[11] the rupture life data were converted from 1200°F, 60 ksi to 1400°F, 55 ksi and vice versa. Thus, heat V00247 showed a cumulative rupture life of ~ 50,000 hr at 1200°F, 60 ksi or over 120 hr at 1400°F, 55 ksi. It should be pointed out that this practice of changing test conditions midway is not to be recommended. It was done only for practical necessity since enormously long rupture life of heat V00247 at 1200°F, 60 ksi was already established.

The mismatch values for the two heats (V00247 and V00246) are –0.16 and –0.41 pct, respectively. The volume fraction γ' for the two heats for the treatments tested are 10.0 and 19.8 pct, respectively. It will be seen from the electron micrographs in Fig. 3 that the 1950°F treatment did not solution all the γ' in V00247. These residual γ' are the larger particles seen on the right of the grain boundary in Fig. 3(*b*). Thus, the V00247 sample showed a duplex γ' even though the coarse γ' was not uniformly dispersed. Also, note that the aging γ' in V00247 are of slightly larger size as compared to the ones in V00246. This is attributed to higher hardener content of the heat, V00247. On air cooling, fine γ' probably precipitated which, on aging,



(*a*)

(*b*)

Fig. 3—Electron micrographs from heats V00246 and V00247. Magnification 25,000 times. Treatment: 1950°F, 1 hr, air cooled, aged at 1350°F, 50 hr, air cooled. Chromium shadowed parlodion replicas. Etchant: "G" electrolytic. The grain boundary precipitates in both identified as $M_{23}C_6$ carbides. Fig. (*b*) shows coarse, residual γ' not solutioned at 1950°F. The matrix γ' precipitated during heat treatment slightly coarse in (*b*). (*a*)—V00246, (*b*)—V00247.

coarsened slightly as compared to the aging γ' in V00246. The grain boundary precipitates in both the heats, shown in Figs. 3(*a*) and 3(*b*), were identified as $M_{23}C_6$ type carbides by electron diffraction analysis. Most of the grain boundaries in these samples were fairly clean. It is believed that the considerable improvement in rupture life of this heat may be due to a cumulative effect of lower mismatch, larger volume fraction γ', and possibly the dispersion of γ' of duplex size. The latter may be particularly true for the test condition used, 1200°F, 60 ksi.[12]

It was determined, metallographically, that a solutioning treatment of 2050°F, 1 hr dissolved all the γ' in V00247. It was also found necessary to water quench the samples after this treatment to prevent the γ' from precipitating during cooling. The microstructure of the three heats solution treated at 2050°F, 1 hr, water

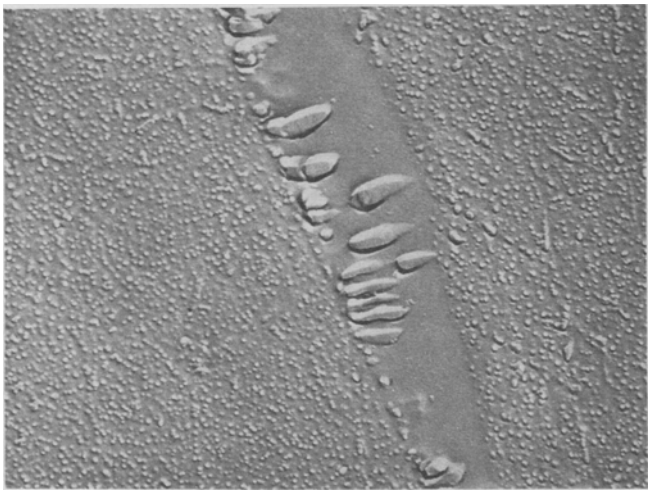
quenched, followed by aging for 15 and 50 hr at 1350°F is shown in Figs. 4 and 5. The precipitates appear to be close-packed in heats V00246 and V00247, containing molybdenum. The heat without molybdenum, V02802, shows slightly coarser γ' and larger interparticle spacing, $\langle$IPS$\rangle$, as compared to the heats with molybdenum. Also, a few grain boundaries in V02802 show cellular-like precipitates, even though it was not a predominate condition. The γ' particles were found to be slightly larger in size in V00247 as compared to V00246 and its dispersion in V00247 more uniform. The coarse grain boundary precipitates in V00246 and V00247 were identified as $M_{23}C_6$ carbides. Some fine γ' is also seen in the grain boundaries; see Figs. 4(*c*) and 5(*c*).

Samples from the two heats, V00246 and V00247, treated at 2050°F, 1 hr, water quenched + 1350°F, 15 hr, air cooled, were stress rupture tested at 1200°F, 60 ksi. The γ' size for the samples so treated was very similar. The average stress rupture life, shown in Table V, was found to be approximately 3500 hr for heat V00246. In comparison, the duplicate test samples from heat V00247 showed a rupture life of over 5000 hr without failing. Again, the test temperature was increased to 1400°F with a slight lowering of stress (from 60 to 55 ksi). The test samples showed a further life of ~90 hr at 1400°F, 55 ksi.

The mismatch and volume fraction γ' data, also shown in Table V, indicate that the increased rupture life in V00247 may be attributed to a cumulative effect of lower mismatch and increased volume fraction γ' at similar γ' size.

To discern the effect of mismatch from that of volume fraction γ', stress rupture tests shown in Table VI were carried out. Using the mismatch and volume fraction γ' data in Tables II and III, respectively, samples were heat treated to yield equivalent mismatch values but different volume fraction γ', approximately 10 and 17 pct in V00246 and V00247, respectively. Two solution treatments were used for heat V00246, 1950° and 2050°F. From the mismatch data in Table II, it was found that a 1950°F, air cooled + 1350°F, 15 hr age treatment resulted in a mismatch value (-0.41) very similar to the one found in 2050°F, water quenched samples when aged for 50 hr at 1350°F (-0.35). It was felt that since the solvus temperature in heat V00246 was below 1950°F and the fact that the γ' did not precipitate out on air cooling, this treatment will provide a direct comparison with heat V00247 solution treated at 2050°F, 1 hr, water quenched + 1350°F, 15 hr, air cooled. The volume fraction in these three test samples are also given in Table VI. The grain size, for all practical purposes, is similar. In addition, duplicate samples of heat V00247, aged at 1350°F, 50 hr, air cooled, were also tested. This treatment resulted in lower mismatch (-0.19 vs -0.37) as compared to 15 hr age with very similar volume fraction γ' (21 vs 17 pct). The γ' particle size in these samples was found to be very similar, as determined by the procedure described by Maniar *et al.*[8] Thus, the effect of stress rupture, to be discussed below, is a result of volume fraction γ' at constant mismatch and similar γ' size.

These samples were tested for stress rupture properties at 1400°F, 55 ksi. The test results, given in Table VI, indicate an average rupture life of 20 and 35 hr for heat V00246 in two heat treated conditions,



(*a*)

(*b*)

(*c*)

Fig. 4—Electron micrographs from samples solution treated at 2050°F, 1 hr, water quenched and aged at 1350°F, 15 hr, air cooled. Magnification 25,000 times. Chromium shadowed parlodion replicas. Etchant: "G" electrolytic. Note the γ'-denuded zone and increased $\langle$IPS$\rangle$ in V02802. The grain boundary in (*a*) shows cellular γ'. $M_{23}C_6$ carbides seen in the grain boundaries in (*b*) and (*c*). (*a*)—V02802, (*b*)—V00246, (*c*)—V00247.

respectively. Even though such a small difference may not normally be considered significant, the almost identical lives obtained in duplicate tests suggest that the higher solution treating temperature may indeed have resulted in a slight increase in rupture life. This may be due to equilibrium γ' precipitates formed at 50 hr aging time. The volume fraction γ' (10.0 vs 10.8 pct) and the grain size (ASTM 3 vs 2) difference is not considered of any significance. Comparing the test data for 15 hr age test samples of heat V00247 with that of V00246, the rupture life has increased in the former by a factor of 3 to 4. The treatment used for this heat resulted in a volume fraction γ' of 17.2 pct. The mismatch value of -0.37 pct for V00247 is very similar to that of the other two test samples from V00246. The increased life thus may be attributed to the higher volume fraction γ'. To discern the effect of a lower mismatch value, samples from heat V00247, aged for 50 hr at 1350°F, were tested. (See (d) in Table VI). The average rupture life is further increased to 160 hr and may be attributed to a lower mismatch at essentially similar volume fraction γ' (17 and 21 pct).

The broken stress rupture samples were analyzed for changes, if any, in pct mismatch and volume fraction γ' values. The thread-ends were used for this analysis. The data, also shown in Table VI, therefore reflect effect of exposure time at the test temperature, 1400°F. The following inferences may be drawn from these data:

1) The mismatch values are not significantly affected in heat V00246 from the exposure at the test temperature in both tests (a) and (b).

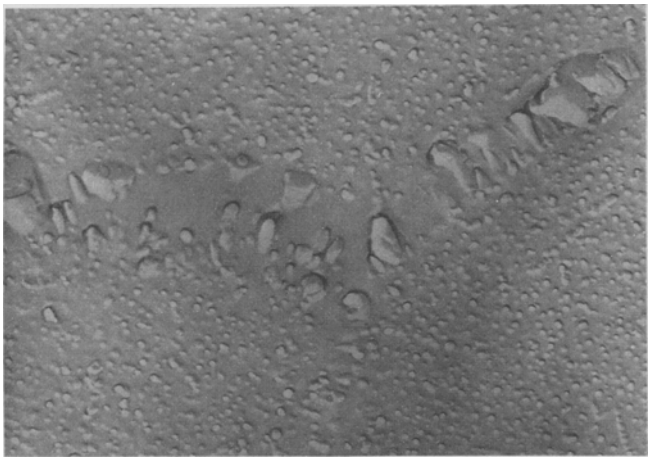
2) The volume fraction γ' appears to have slightly decreased in the two tests (a) and (b), for heat V00246, from 10 to 8 pct. This is attributed to slight coarsening of γ'.

3) The mismatch value for test (c), heat V00247, 15 hr age, however, decreased from -0.37 to -0.15 pct with a slight increase in volume fraction γ'. This, as will be discussed in (4) below, may be attributed to the aging time of 50 hr required at 1350°F to precipitate equilibrium γ'. The improved rupture life for this heat in this test was attributed earlier to increased volume fraction γ'. It is possible that lowering of mismatch from -0.37 to -0.15 pct may also have contributed beneficially.

(4) The samples from heat V00247, aged at 1350°F for 50 hr did not undergo any change in mismatch or volume fraction γ' even after an exposure of over 150 hr at 1400°F. This would indicate an "equilibrium" γ' precipitation in this treatment. Presence of "equilibrium" γ' prior to testing, accompanied by a lower mismatch value, may have contributed towards the excellent rupture life. These data also support the earlier observation of "equilibrium" γ' formation at 1350°F, 50 hr age.

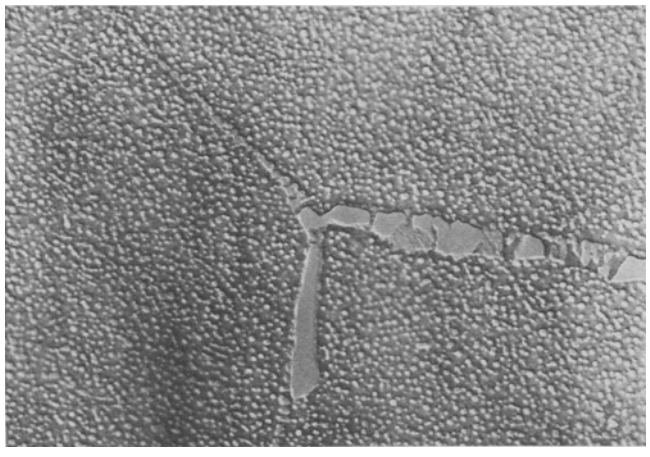
Elevated Temperature Tensile Data

In a further attempt to discern the effect of higher volume fraction γ' due to increased hardener content, heats V00246 and V00247 were tested at 1400°F for elevated temperature tensile properties. Again, the test samples from V00246 aged at 1350°F for 50 hr were used to equate the mismatch value of -0.37 pct in V00247 obtained after a 15 hr age at 1350°F. The resultant volume fractions γ' are 10.8 and 17.2 pct respectively. The tensile test results in Table VII show that at a similar mismatch value, the increased volume fraction γ' increased, but by a very small percentage,



(a)

(b)



(c)

Fig. 5—Electron micrographs from samples solution treated at 2050°F, 1 hr, water quenched and aged at 1350°F, 50 hr, air cooled. Magnification 25,000 times. Chromium shadowed parlodion replicas. Etchant: "G" electrolytic. Note coarse γ' and denuded zones in V02802. The grain boundary precipitates in (*b*) and (*c*) are $M_{23}C_6$ carbides. Some fine γ' also seen in the boundaries in (*c*). (*a*)—V02802, (*b*)—V00246, (*c*)—V00247.

the 1400°F strength in V00247. These results, when compared with the 1400°F, 55 ksi stress rupture data discussed before, would justify the earlier claim[8] that the stress rupture property may be a more critical test for such alloys.

DISCUSSION

It is shown that addition of molybdenum in heat V00246 reduced the mismatch to -0.41 pct from -0.82 pct in V02802 (of similar chemistry without molybdenum) at essentially similar volume fraction γ'. The improvement in rupture life was quite significant (2500 hr vs 130 hr). Davies and Johnston[13] have similarly shown that the rupture life can be maximized by lowering the pct mismatch (close to zero) in Ni-Cr-Al alloys.

The beneficial effect of lower mismatch is further shown by comparing the stress rupture test data for heat V00247 in 15 and 50 hr, 1350°F aged conditions (Table VI). At essentially constant volume fraction γ', the lower mismatch (0.19 vs 0.37), increased the rupture life by approximately 50 hr — a significant gain.

It is not intended to minimize the importance of volume fraction γ'. The test data in Table VI indicate that at constant mismatch value, the increased volume fraction γ' improved the rupture properties and conversely a lower mismatch value at constant volume fraction γ', likewise, results in improved rupture properties.

Prager and Shira[14] have correlated the hardener content and the maximum operating temperature. The maximum operating temperatures for Waspaloy and Udimet 500 were reported to be 1400° and 1500°F, respectively, whereas the γ-γ' mismatch values for the alloys were shown to be similar, ~0.30 pct. The volume percentages of γ', however, were 30 to 40 and 50 to 60 pct, respectively, for Waspaloy and Udimet 500. Thus, the higher operating temperature for Udimet 500 was attributed to increase in volume fraction γ'. Another important consideration in any design of superalloys should be the equilibrium γ'. This is evident from the discussion pertaining to the test data given in Table VI.

Decker and Mihalisin[7] have shown a linear relationship between the elevated temperature hardness and mismatch in Ni-Al alloys strengthened with Ta, Cb, C, and so forth. They have also pointed out the importance of service temperature in alloy design. At temperatures in the region of 0.6 Tm (Tm—melting temperature), they claim the lower coherency strains (γ-γ' mismatch) to be more useful for maximum phase stability. Assuming ~2400°F to be the melting temperature of these alloys, at 1400°F the lower γ-γ' mismatch and the resultant coherency strains play a major role in strengthening these alloys.

CONCLUSIONS

1) The mismatch between the lattices of γ and γ' has a critical effect on elevated temperature stress rupture properties. A mismatch value close to zero (*i.e.*, similar lattice parameters of γ and γ') improves the rupture life considerably.

2) Equally critical is the effect of increased hardener (within practical limits) and resultant volume fraction γ'. At equivalent mismatch value, the rupture life can be further improved by increased volume fraction γ'.

3) Molybdenum at the level studied (5.5 wt pct) does increase the γ' solvus temperatures in the analyses studied and retards the γ' dissolution on overaging.

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