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EFFECTS OF SIGMA-PHASE FORMATION ON SOME MECHANICAL PROPERTIES OF A WROUGHT NICKEL-BASE SUPERALLOY (IN-100)

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16. Abstract

The effect of sigma-phase formation on an extruded and forged nickel-base superalloy with the composition of the casting alloy IN-100 was studied. By adding only aluminum and titanium to remelt stock, three compositions were produced which had varying propensities for sigma formation. These compositions were given a four-step heat treatment and were stress-rupture or tensile tested. The very sigma-prone composition had a shorter rupture life than the sigma-free or moderately sigma-prone compositions when tested at 843° and 885° C. Elongation in room-temperature tensile tests was considerably lower for the very sigma-prone composition than for the other two wrought compositions after prolonged exposure at 732° or 843° C.

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SUMMARY

An investigation was conducted to determine the effect of sigma-phase formation on an extruded and forged nickel-base superalloy with the composition of the casting alloy IN-100. One master heat of the alloy was modified by adding only aluminum (Al) and titanium (Ti) to remelt stock. Three compositions were produced. One heat was sigma free after exposures of 5000 hours at temperatures between 704° and 982° C. One heat was moderately sigma prone during elevated-temperature exposure. The third heat was very sigma prone during elevated-temperature exposure. All three compositions were given a conventional four-step heat treatment and then stress-rupture and tensile tested. Each was also tensile tested after prolonged exposure at elevated temperature.

The very sigma-prone composition had a shorter rupture life than the sigma-free or moderately sigma-prone compositions when tested at 843° and 885° C (temperatures where sigma forms). For example, the sigma-free and moderately sigma-prone compositions had average lives of approximately 1300 hours when tested at 843° C and 276 MN/m^2. But the very sigma-prone composition had an average life of only approximately 310 hours. The formation of the small amounts of sigma observed in the moderately sigma-prone wrought composition did not adversely affect the mechanical properties investigated. The formation of the sigma phase caused less reduction in the stress-rupture life of wrought alloys than in previously reported cast alloys. Room-temperature tensile tests were performed on material which had been exposed at 732° C for 1000 hours or at 843° C for 250 hours. The yield strength of all the wrought compositions decreased approximately 100 MN/m^2 after exposure to 843° C for 250 hours. The elongation measured in room-temperature tensile tests was considerably lower for the very sigma-prone composition than for the other two wrought compositions after prolonged exposure at either temperature.

INTRODUCTION

For over a decade the phase stability of the superalloys used in gas turbine engines intended for long-time service has been of concern to engine manufacturers and their metallurgists. Methods have been developed to predict whether a heat of a given alloy will be stable which relate the phase stability to effects of certain alloy additions. This investigation is a study of the effect of sigma formation on the nickel-base superalloy IN-100 in wrought form. It is a continuation of work that was initially concerned only with the cast form of IN-100 (refs. 1 and 2). This alloy was chosen for study because it had shown a tendency to form sigma in compositions within commercial specifications and because it could be processed in either cast or wrought form. Although IN-100 is commercially available in compositions which will not form significant quantities of sigma phase during long-time exposure under stress (ref. 3), sigma can form in certain ranges (refs. 1 and 3) of the Aerospace Materials Specification for IN-100, AMS 5397 (ref. 4).

Three special compositions were formulated so that one would be sigma free, one moderately sigma prone, and one very sigma prone when exposed to elevated temperatures. This variation in sigma-forming tendency was achieved by adding only aluminum (Al) and titanium (Ti) to a single master heat during remelting. Isothermal transformation curves for the onset of sigma formation for these compositions in both the cast and wrought conditions, as well as the effect of sigma-phase formation on the mechanical properties of cast alloys, have been reported in references 1 and 2.

This report concerns the three compositions in the wrought condition. It discusses the effect of sigma formation on the mechanical properties and compares the severity of the effects of sigma formation on the wrought and cast alloys. After a four-step heat treatment, specimens were stress-rupture tested at 649° to 982° C and were short-time tensile tested at room temperature. In addition, specimens heated 250 hours at 843° C and 1000 hours at 732° C after being heat treated were short-time tensile tested at room temperature. Mechanical properties were correlated with composition and the propensity to form the sigma phase.

PROCEDURE

The cast and the wrought materials discussed in this report are described in detail in references 1 and 2. The new results to be discussed have to do chiefly with the mechanical properties of wrought material. For comparison, some mechancal test results for the cast compositions (refs. 1 and 2) are also discussed. Therefore, the essential procedural details are given for the cast as well as for the wrought materials.

Materials

Wrought. - Three 45-kilogram vacuum induction heats of IN-100 were melted by a forging supplier. The starting material for each heat was a portion of a single master heat. Appropriate additions of Al plus Ti resulted in three propensities toward sigma formation. Induction-remelted IN-100 was cast into 10.2-centimeter-diameter ingots. These ingots were vacuum arc remelted to 12.7-centimeter-diameter ingots by a specialty steel producer. The ingots were then canned in steel pipe for extrusion. Extrusion was done at 1120° C, to a diameter of about 8 centimeters. The unsound ends of the extrusions were cut back to sound metal, and the extrusions were cut into thirds.

These extruded forging multiples were forged into pancakes in three flattening operations at 1120° C. The forging pressure was applied normal to the extrusion axis. The forging was done in heated dies and between steel covers. Before the last reduction the forgings were cut in half longitudinally. Each forging multiple was reduced to a thickness of 1.6 centimeters in the following steps:

(1) The 8-centimeter-diameter extrusions were forged to 4.5-centimeter-thick pancakes.

(2) The 4.5-centimeter-thick pancakes were forged to 2.5-centimeter-thick pancakes.

(3) The 2.5-centimeter-thick pancakes were forged to 1.6-centimeter-thick pancakes.

The yield was about 14 kilograms of forging per 45 kilograms of starting material. The six finished forgings from one extrusion are shown in figure 1.

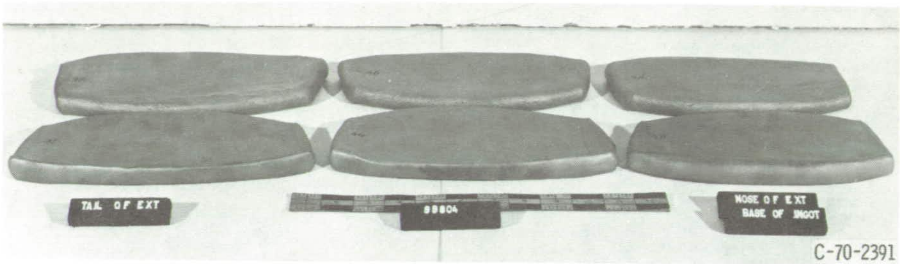


Figure 1. - Wrought 1.6-centimeter-thick pancakes of IN-100.

TABLE I. - CHEMICAL ANALYSES OF CAST AND WROUGHT IN-100

Form	Element, wt. % (balance Ni)											Average electron vacancy concentration[a], $\bar{N}_v$
	Co	Cr	Al	Ti	Mo	V	Si	Fe	Zr	C	B	
Low Al + Ti content (sigma free)												
Cast	13.58	10.26	4.95	4.10	3.45	0.96	0.08	0.07	0.02	0.160	0.014	2.27
	13.48	10.13	5.03	4.22	3.65	1.01	.08	.14	.04	.176	.015	2.31
Wrought	13.73	10.26	4.95	4.08	3.66	0.96	0.09	0.07	0.03	0.157	0.014	2.29
Medium Al + Ti content (moderately sigma prone)												
Cast	13.29	10.15	5.47	4.28	3.51	0.96	0.10	0.07	0.03	0.160	0.014	2.47
	13.35	10.13	5.51	4.30	3.60	.96	.10	.07	.03	.164	.013	2.51
Wrought	13.41	10.15	5.36	4.09	3.58	0.96	0.09	0.12	0.02	0.157	0.012	2.40
High Al + Ti content (very sigma prone)												
Cast	13.22	10.11	5.55	4.59	3.50	0.96	0.11	0.06	0.03	0.157	0.014	2.59
	13.29	10.13	5.65	4.78	3.53	.98	.09	.08	.03	.158	.012	2.71
Wrought	13.42	10.13	5.46	4.63	3.58	1.01	0.09	0.07	0.02	0.155	0.013	2.59
AMS 5397[b]												
Cast												
Minimum	13.00	8.00	[c]5.00	[c]4.50	2.00	0.70	----	----	0.03	0.15	0.01	----
Maximum	17.00	11.00	6.00	5.00	4.00	1.20	0.15	1.00	.09	.20	.02	----

[a]Data obtained by method described in reference 1 and supplied by C. T. Sims of General Electric Company. Critical $\bar{N}_v$, 2.46.

[b]Reference 4.

[c]Al + Ti > 10.0.

Table I lists chemical analyses and calculated average electron vacancy concentration numbers $\bar{N}_v$ of the extrusion billets used to produce the forgings. The higher the Al-plus-Ti content, the higher the $\bar{N}_v$ and the greater the tendency to form sigma.

Cast. - Fine-grained, vacuum-melted, investment-cast specimens were used in this work. During remelting for casting, additions of Al plus Ti were made to material from the same master heat used for the forgings. The additions were chosen to provide castings with three different propensities toward sigma formation. Compositions and average electron vacancy concentration numbers of arbitrarily selected remelted heats are shown in table I. No sigma was observed in any as-cast material. The analyses were very similar to the wrought analyses and so were the average electron vacancy numbers.

Heat Treatment

Wrought materials. - All wrought material was first heat treated in a four-step schedule similar to that used for the wrought nickel-base superalloy Udimet-700. The material was heat treated at 1215° C for 4 hours, at 1095° C for 4 hours, at 845° C for 16 hours, and finally at 760° C for 24 hours. The three compositions were all solution treated at the same temperature even though it was known that the solidification gamma prime (primary gamma prime) would not be completely dissolved (ref. 2) in the compositions containing the higher amounts of Al and Ti. The solution temperature of 1215° C was selected as the maximum temperature which could be used without causing incipient melting in any of the compositions. All specimen blanks were heated under argon and air cooled to room temperature after each heating cycle.

In order to determine the effect of preexisting sigma on short-time mechanical properties, two thermal exposures were selected. Four-step heat-treated test bar blanks were exposed to 732° C for 1000 hours and to 843° C for 250 hours. Blanks of each of the three sigma-forming tendencies were heated together.

Cast materials. - For this portion of the overall study of sigma in IN-100, the cast bars were not heat treated but were used in the as-cast condition.

Mechanical Testing

Tensile tests and constant-load stress-rupture tests were conducted on the alloys in accordance with appropriate practices recommended by the American Society for Testing Materials (ASTM). The test bar design used in references 1 and 2 was used for both tensile and stress-rupture testing. Specimens were ground to a nominal 0.6-centimeter diameter in the test section. The specimen design is shown in figure 2.

Stress-rupture specimens were heated to the test temperature in 3 to 5 hours and soaked an additional 1 to 2 hours prior to loading. Stress-rupture tests were conducted between 649^{o} and 982^{o} C at stresses of 1034, 655, 276, and 138 MN/m^2.

Wrought tensile specimens were pulled at room temperature both before and after the exposures described in the section Heat Treatment.

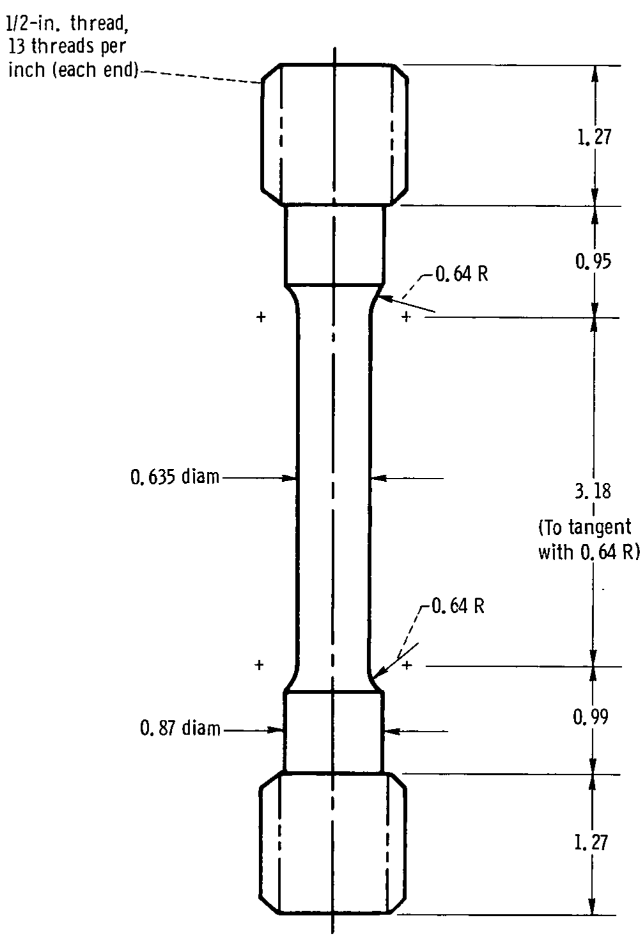


Figure 2. - Mechanical property test bar. (Dimensions are in cm, except as noted.)

Metallography

For general examination a swabbing etchant of the following composition was used: 33 parts water, 33 parts nitric acid, 33 parts acetic acid, and 1 part hydro fluoric acid. This etch reveals gamma prime and sigma. A modified Murakami's etch made from equal volumes of two solutions (10 g of potassium ferrocyanide in 90 cm^3 of water, and 10 g of potassium hydroxide in 90 cm^3 of water) was used to observe carbides and sigma, without revealing the gamma prime. An etch of 5 percent potassium hydroxide used electrolytically at about 5 volts performed this same function. The latter two etchants were used to reveal sigma when only small amounts were present.

RESULTS AND DISCUSSION

Tensile Properties

Figure 3 shows the effect of exposure at 732° and 843° C and of sigma-forming tendency on the average room-temperature tensile properties of wrought IN-100 after a four-step heat treatment. The individual results are shown in table II.

<u>As heat treated</u>. - In the heat-treated condition, the compositions had yield strengths which were essentially equal, about 970 MN/m^2. The ultimate strengths

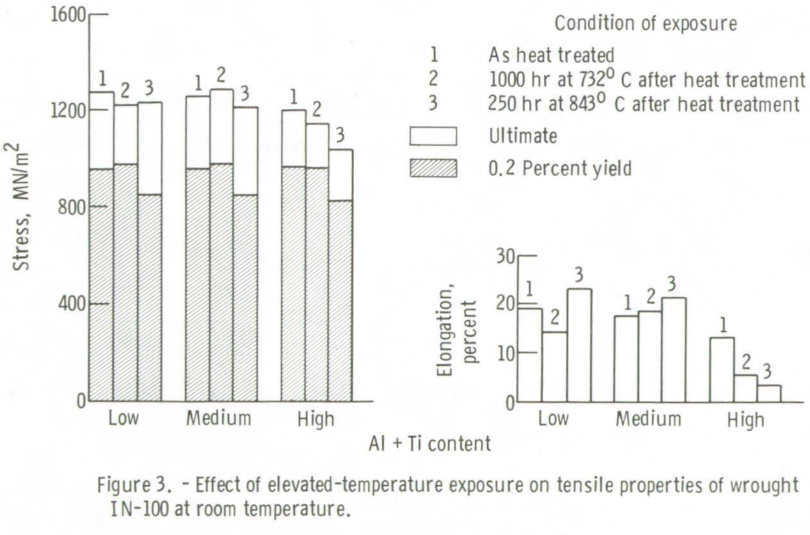


Figure 3. - Effect of elevated-temperature exposure on tensile properties of wrought IN-100 at room temperature.

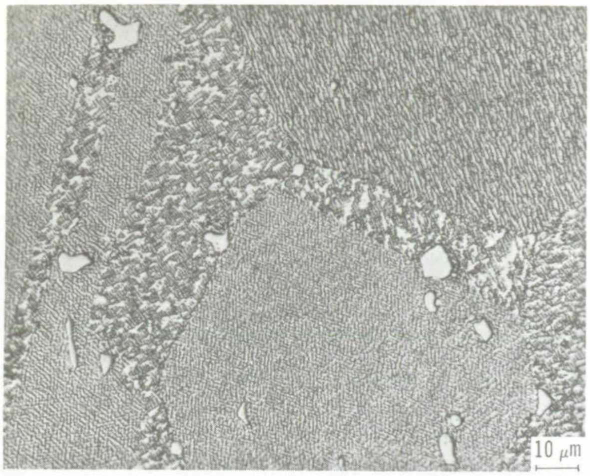
TABLE II. - ROOM-TEMPERATURE MECHANICAL PROPERTIES OF WROUGHT IN-100

Condition	Al + Ti content	Ultimate tensile strength		0.2-Percent yield strength		Elongation, percent	Reduction in area, percent
		MN/m^2	psi	MN/m^2	psi		
Heat treated[a]	Low[b]	1280	185 000	950	138 000	19	15
		1280	186 000	970	141 000	19	15
	Medium[c]	1270	184 000	960	139 000	18	15
		1260	183 000	980	142 000	17	14
	High[d]	1210	176 000	970	141 000	14	11
		1200	174 000	970	141 000	11	11
Heat treated[a] plus 1000 hr at 732° C (1350° F)	Low[b]	1250	181 000	990	144 000	13	13
		1220	177 000	990	144 000	12	12
	Medium[c]	1290	187 000	990	143 000	17	17
		1290	187 000	980	142 000	19	18
	High[d]	1130	164 000	970	141 000	5	7
		1130	164 000	970	140 000	5	9
Heat treated[a] plus 250 hr at 843° C (1550° F)	Low[b]	1230	179 000	850	123 000	24	23
		1240	180 000	860	125 000	21	19
	Medium[c]	1220	177 000	840	122 000	22	19
		1210	176 000	850	124 000	19	18
	High[d]	1130	164 000	810	117 000	5	7
		960	139 000	850	124 000	2	2

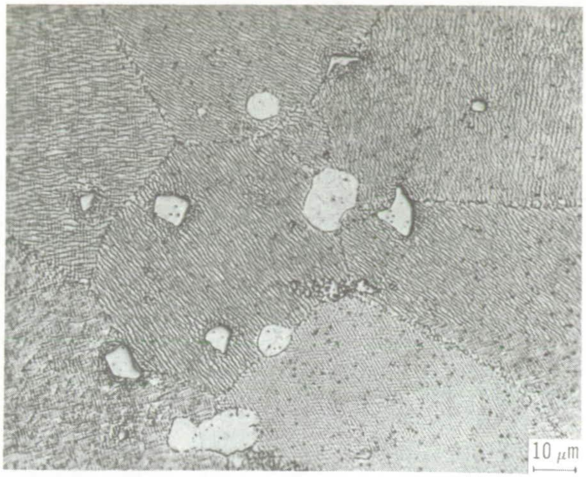
[a]Material was forged then heated to 1215° C (2215° F) for 4 hr followed by 4 hr at 1095° C (2000° F), 16 hr at 845° C (1550° F), and 24 hr at 760° C (1400° F).
[b]Sigma free.
[c]Moderately sigma prone.
[d]Very sigma prone.

of the sigma-free and moderately sigma-prone compositions were approximately equal, at 1280 and 1270 MN/m^2. The ultimate strength of the very sigma-prone composition was lower, 1210 MN/m^2. The ductility of the compositions as measured by elongation decreased as the sigma-forming tendency increased. The elongation of the sigma-free composition was 19 percent, the elongation of the moderately sigma-prone composition was 17 percent, and that of the very sigma-prone composition was 13 percent.

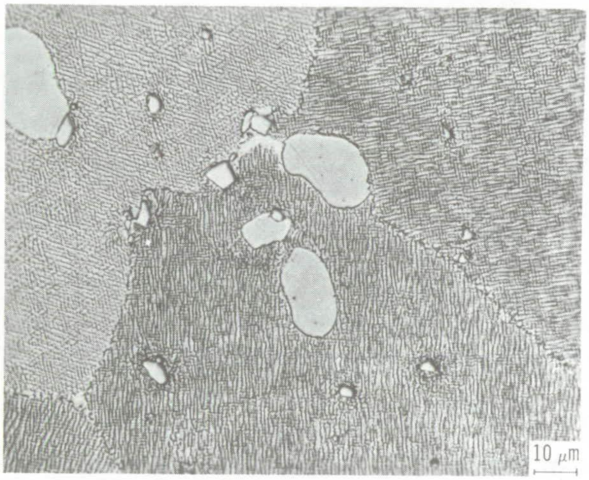
Typical microstructures of the compositions are shown in figure 4. The struc-



(a) Low Al + Ti content (sigma free).



(b) Medium Al + Ti content (moderately sigma prone).



(c) High Al + Ti content (very sigma prone).

Figure 4. - Microstructure of wrought IN-100 after four-step heat treatment. Etch, mixed acids.

ture of the compositions consists largely of grains of gamma with precipitated cooling gamma prime and with Ti-based monocarbides and solidification gamma prime dispersed throughout the structure. No sigma can be seen in the structure of any of the compositions. The sigma-free composition (fig. 4(a)) contains very little solidification gamma prime. The moderately sigma-prone composition (fig. 4(b)) contains more solidification gamma prime, and the very sigma-prone composition (fig. 4(c)) contains the most solidification gamma prime. The solidification-gamma-prime regions were largest in the very sigma-prone composition. The increase in the amount of solidification gamma prime with increasing sigma-forming tendency is consistent with the increase in the gamma-prime-forming elements, Ti and Al, in the sigma-prone compositions. The decrease in ductility of the sigma-prone heats, as compared to the sigma-free heat, may be a result of the solidification gamma prime being present in greater amounts in the lower ductility heats.

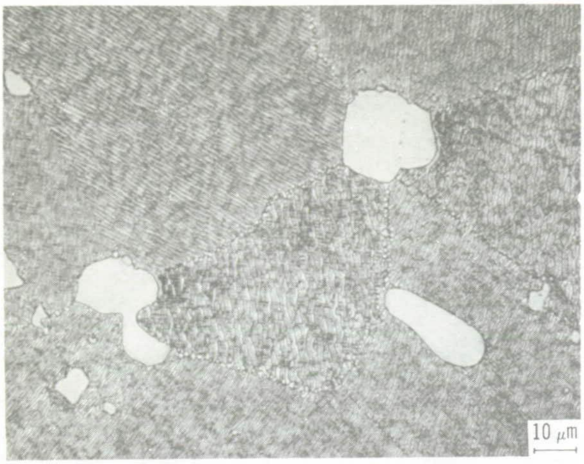
Exposed at 732° C for 1000 hours. - After exposure at 732° C for 1000 hours the yield strengths of the three compositions were approximately 980 MN/m^2. The yield strength of the moderately sigma-prone and sigma-free compositions had increased slightly. The yield strength of the very sigma-prone composition was essentially unchanged.

Exposure to 732° C caused the ultimate strength of the moderately sigma-prone composition to increase slightly but those of the sigma-free and very sigma-prone compositions to decrease. The greatest change in ultimate strength was for the very sigma-prone composition: a strength decrease of 80 MN/m^2, to 1130 MN/m^2.

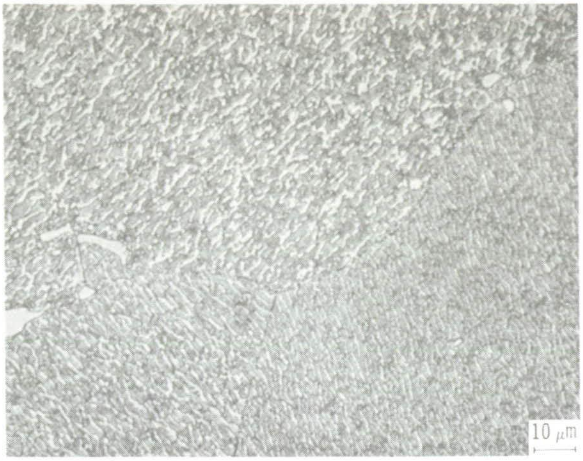
The postexposure elongation of the moderately sigma-prone composition was not significantly different from the preexposure value. The sigma-free and very sigma-prone compositions had lower elongations after being exposed to 732° C. The greatest reduction in elongation was for the very sigma-prone composition, which had an elongation of 5 percent after the exposure and 13 percent prior to it.

Typical photomicrographs of the compositions after they were exposed to 732° C for 1000 hours are shown in figure 5. Comparing figure 5(a) with figure 4(a) shows that the most noticeable effect of the exposure on the sigma-free composition is a slight increase in the amount of carbides in the grain boundaries. Although an increased carbide precipitation is not evident in figure 5(b), a visual examination of the moderately sigma-prone composition after exposure at 732° C for 1000 hours revealed that this composition also had more carbides at the grain boundaries after exposure than as heat treated. No sigma was observed in the moderately sigma-prone composition after this exposure. Discontinuous platelets of sigma can be seen in the photomicrographs of the very sigma-prone composition (figs. 5(c) and 5(d)).

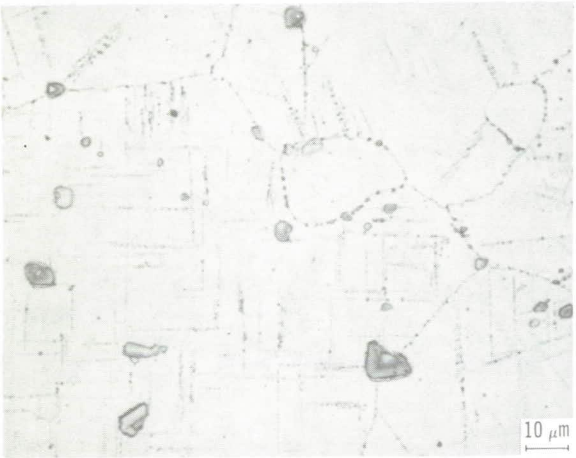
Exposed at 843° C for 250 hours. - Exposure at 843° C for 250 hours decreased the yield strength of all the compositions so that after the exposure the yield strength of the compositions was nearly equal, approximately 850 MN/m^2. The ultimate tensile strength of all the compositions was reduced. The greatest reduction in ultimate tensile strength was for the very sigma-prone composition and was 160 MN/m^2, the average strength after exposure being 1040 MN/m^2. The postexposure



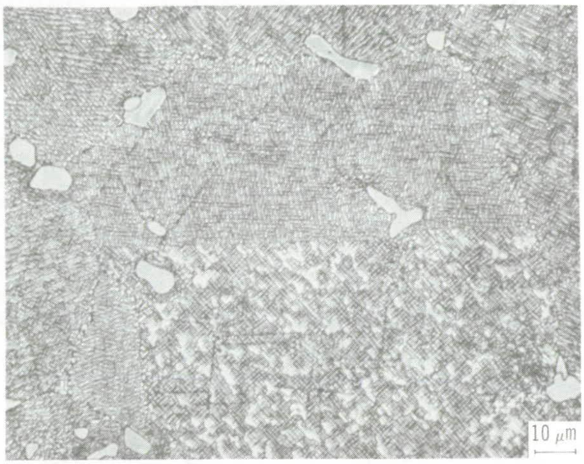
(a) Low Al + Ti content (sigma free); etch, mixed acids.



(b) Medium Al + Ti content (moderately sigma prone); etch, mixed acids.



(c) High Al + Ti content (very sigma prone); etch, KOH.



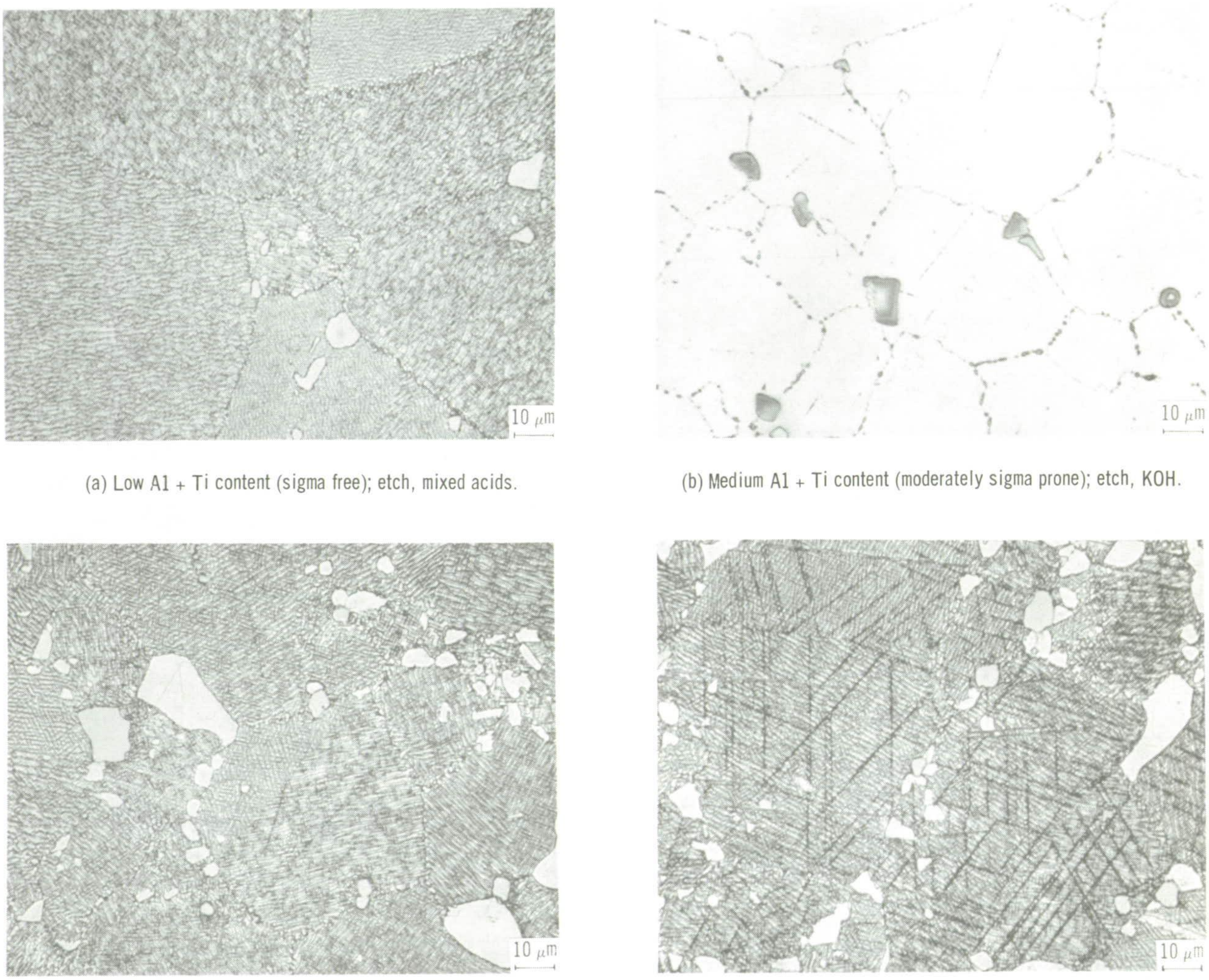
(d) High Al + Ti content (very sigma prone); etch, mixed acids.

Figure 5. - Microstructure of wrought IN-100 after four-step heat treatment and exposure to 732° C for 1000 hours.

ultimate tensile strength of the sigma-free composition was 1240 MN/m^2, which was 40 MN/m^2 less than the preexposure strength. The postexposure ultimate tensile strength of the moderately sigma-prone composition was 1220 MN/m^2, which was 50 MN/m^2 less than the preexposure value. The elongation of the sigma-free and moderately sigma-prone compositions was slightly greater after exposure at 843° C than before the exposure. The sigma-free composition's elongation increased to 23 percent, and the moderately sigma-prone composition's elongation increased to 21 percent. The very sigma-prone composition's elongation decreased from 13 percent prior to the exposure at 843° C to 3 percent after it.

Typical microstructures of the compositions which have been exposed at 843° C for 250 hours are shown in figure 6. Comparing figures 6(a) and 4(a) shows that the exposure to 843° C caused the precipitation of fine particles along the grain boundaries in the sigma-free composition. Based on previous observations of Ni-base superalloys, these grain boundary particles are assumed to be $M_{23}C_6$. No sigma phase was observed. Comparing figures 6(a) and 5(a) shows that there is little difference in appearance between the sigma-free composition exposed for 250 hours at 843° C and that exposed for 1000 hours at 732° C.

Figures 6(b) and 6(c) show that sigma needles have formed in the moderately sigma-prone composition as a result of the exposure to 843° C. This composition



(a) Low Al + Ti content (sigma free); etch, mixed acids.

(b) Medium Al + Ti content (moderately sigma prone); etch, KOH.

(c) Medium Al + Ti content (moderately sigma prone); etch, mixed acids.

(d) High Al + Ti content (very sigma prone); etch, mixed acids.

Figure 6. - Microstructure of wrought IN-100 after four-step heat treatment and exposure to 843° C for 250 hours.

contains only a few needles as compared to the very sigma-prone composition, which contains many needles in a Widmanstätten morphology. The greatest amount of sigma was observed in the very sigma-prone composition which had been exposed for 250 hours at 843° C (fig. 6(d)). Less sigma was observed in the very sigma-prone composition which had been exposed for 1000 hours at 732° C (figs. 5(c) and (d)). The least amount of sigma was in the moderately sigma-prone composition exposed for 250 hours at 843° C (figs. 6(b) and (c)). Like the sigma-free composition, the sigma-prone compositions have carbides in the grain boundaries.

Stress-Rupture Properties

Stress-rupture tests were conducted on the three compositions in the heat-treated condition. The compositions were tested at temperatures from 649° to 982° C and stresses from 1030 to 138 MN/m^2. The individual data are listed in table III and are plotted in figure 7. Also shown in figure 7 is the rupture life of the cast sigma-free alloy (refs. 1 and 2) for the same test conditions. At the lower temperatures, the lives of this cast alloy and the wrought alloys are similar. As the temperature increased, the life of the cast alloy increased relative to the wrought alloy.

Normalizing of rupture life. - To better compare the stress-rupture life of the sigma-prone compositions with that of the sigma-free composition for wrought alloys and cast alloys (refs. 1 and 2), least-squares equations were calculated for the life of the compositions at constant stress as a function of temperature. For the sigma-free and moderately sigma-prone[1] compositions the fit of the following equation was satisfactory, having a correlation coefficient R^2 greater than 0.95:

$$\text{Log (time)} = A + B\ \text{(temperature)}.$$

For the very sigma-prone wrought composition at 655 MN/m^2 and the moderately sigma-prone cast composition at 276 MN/m^2, the equation

$$\text{Log (time)} = A + B\ \text{(temperature)} + C\ \text{(temperature)}^2$$

was required to provide a correlation coefficient greater than 0.95. To show the

[1]The 364.7-hour data point at 649° C for the medium Al and Ti compositon was omitted from this analysis.

TABLE III. - STRESS-RUPTURE DATA FOR WROUGHT IN-100[a]

Al + Ti content	Temperature		Stress		Life, hr	Elongation, percent	Reduction in area, percent
	°C	°F	MN/m²	psi			
Low[b]	649	1200	1034	150 000	27.0	15	13
	649	1200	655	95 000	15009.0	2	3
	649	1200			13001.0	2	2
	704	1300			401.9	2	1
	704	1300			1008.1	3	2
	773	1425			17.2	3	2
	773	1425			20.9	4	3
	843	1550	276	40 000	1340.6	5	4
	843	1550			1332.7	5	4
	885	1625			194.4	6	4
	885	1625			212.1	(c)	4
	982	1800	138	20 000	153.5	(c)	10
	982	1800	138	20 000	104.0	(c)	11
Medium[d]	649	1200	655	95 000	364.7	5	4
	649	1200			11642.4	2	3
	649	1200			12412.1	4	2
	704	1300			969.7	3	4
	704	1300			785.3	7	6
	773	1425			32.8	8	8
	773	1425			27.4	6	4
	843	1550	276	40 000	1365.8	5	5
	843	1550			1249.4	6	5
	885	1625			230.4	4	4
	885	1625			237.1	8	5
	982	1800	138	20 000	148.1	5	5
	982	1800	138	20 000	95.7	(c)	(c)
High[e]	649	1200	655	95 000	3303.0	1	0
	704	1300			740.2	5	6
	704	1300			538.6	6	8
	773	1425			20.7	3	4
	773	1425			18.1	16	18
	843	1550	276	40 000	339.2	19	21
	843	1550			281.9	3	4
	885	1625			156.9	9	6
	885	1625			132.8	7	7
	982	1800	138	20 000	20.9	14	33
	982	1800	138	20 000	140.4	9	12
	982	1800	138	20 000	103.8	(c)	7

[a]Material was forged then heated to 1215° C (2215° F) for 4 hr followed by 4 hr at 1095° C (2000° F), 16 hr at 845° C (1550° F), and 24 hr at 760° C (1400° F).

[b]Sigma free.

[c]Not available.

[d]Moderately sigma prone.

[e]Very sigma prone.

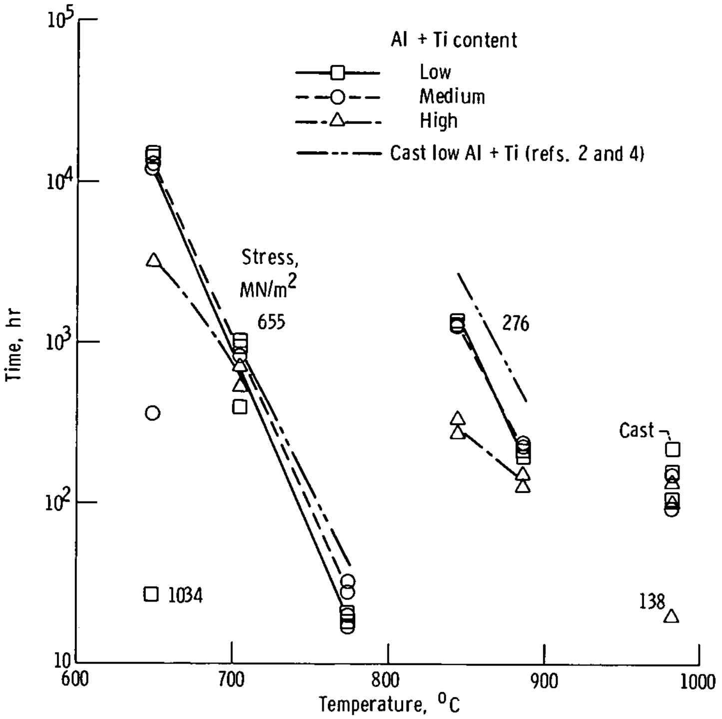


Figure 7. - Stress-rupture life of wrought IN-100.

relative effects of sigma-forming tendency on stress-rupture life, figures 8 and 9 show the lives of the sigma-prone compositions normalized to that of the sigma-free composition. The points plotted in figures 8 and 9 were calculated as follows: The expected life for each type of alloy was calculated by using the preceding equations at stresses and temperatures where data had been obtained. The expected life of the moderately sigma-prone composition was divided by the expected life of the sigma-free composition to obtain the points in figure 8. Similarly, the expected life of the very sigma-prone composition was divided by the expected life of the sigma-free composition to obtain the points plotted in figure 9.

The lives of the cast and wrought forms of the moderately sigma-prone composition and of the sigma-free composition are compared in figure 8. The moderately sigma-prone wrought composition was not observed to have a significantly shorter life than the sigma-free wrought composition at any test condition. That is, the life ratio was not significantly less than 1. In fact, the moderately sigma-prone wrought composition had a longer life than the sigma-free wrought composition at 704° and 773° C with a stress of 655 MN/m^2 and at 982° C with a stress of 138 MN/m^2. This result is in contrast to the cast compositions, for which the moderately sigma-prone

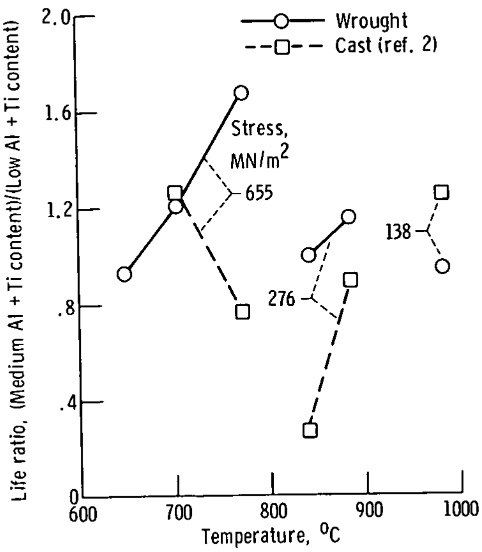


Figure 8. - Comparison of stress-rupture lives of IN-100 with medium and low Al + Ti contents.

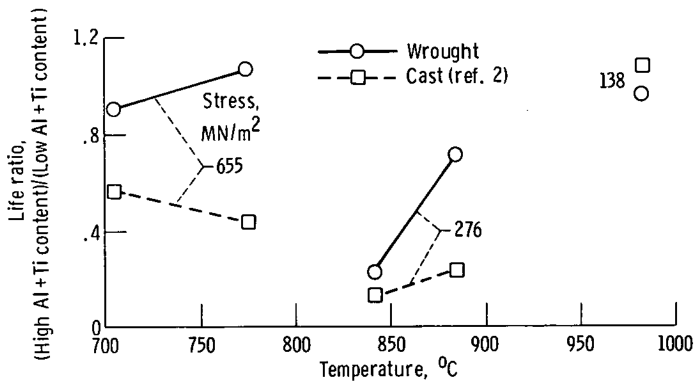
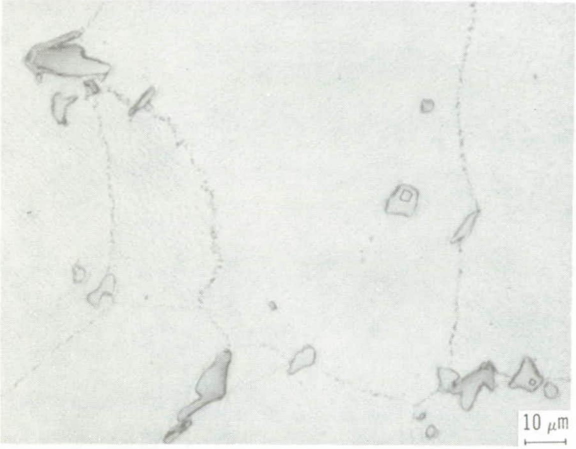


Figure 9. - Comparison of stress-rupture lives of IN-100 with high and low Al + Ti contents.

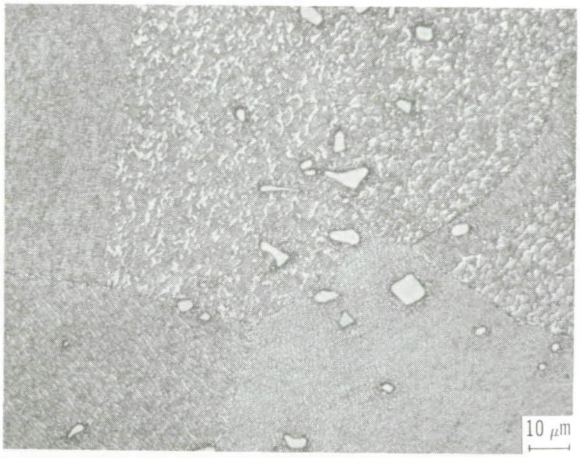
composition had significantly shorter life than the sigma-free cast composition at 773° C with a stress of 655 MN/m^2 and at 843° C with a stress of 276 MN/m^2.

Figure 9 compares the lives of the cast and wrought forms of the very sigma-prone and sigma-free compositions. For the very sigma-prone wrought composition, all test lives were significantly shorter than for the sigma-free wrought composition. At 843° and 885° C, where the rate of sigma formation has previously been shown to be fast (ref. 2), with a stress of 276 MN/m^2 the very sigma-prone wrought composition had a significantly shorter life than the sigma-free wrought composition. At 843° C and a stress of 276 MN/m^2 the very sigma-prone wrought composition had only 23 percent of the life of the sigma-free wrought composition. Except for the tests at 138 MN/m^2 at 982° C, where no sigma was observed, the life ratio of the cast composition was lower than that of the wrought composition.

For both the sigma-prone compositions, at the test conditions where the cast composition had a life ratio significantly lower than 1, the life ratio of the wrought counterpart was greater than the life ratio of the cast composition. It is evident from this and the discussion of photomicrographs which follows that during rupture testing the thermal exposures which formed sigma were less damaging to the wrought compositions than to the cast compositions.

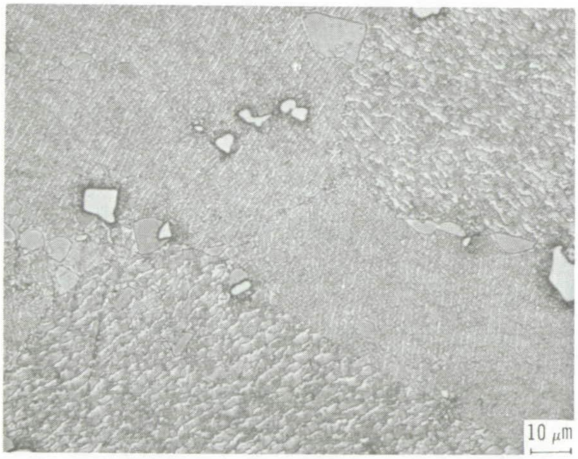


(a) IN-100 with low Al + Ti content tested at 649° C and 655 MN/m^2; etch, KOH; approximate life, 13,000 hours.



(b) IN-100 with low Al + Ti content tested at 649° C and 655 MN/m^2; etch, mixed acids; approximate life, 13,000 hours.

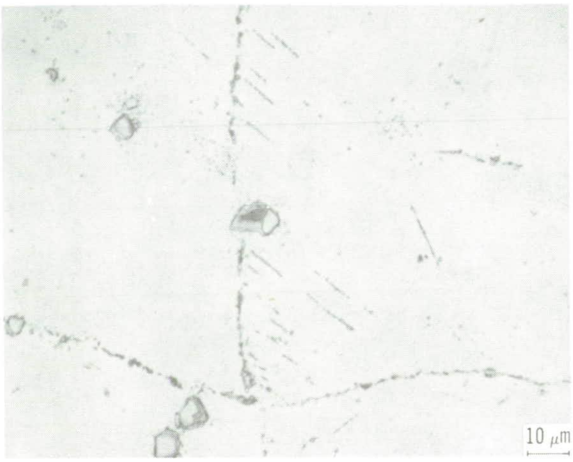
(c) IN-100 with medium Al + Ti content tested at 649° C and 655 MN/m^2; etch, KOH; approximate life, 12,000 hours.



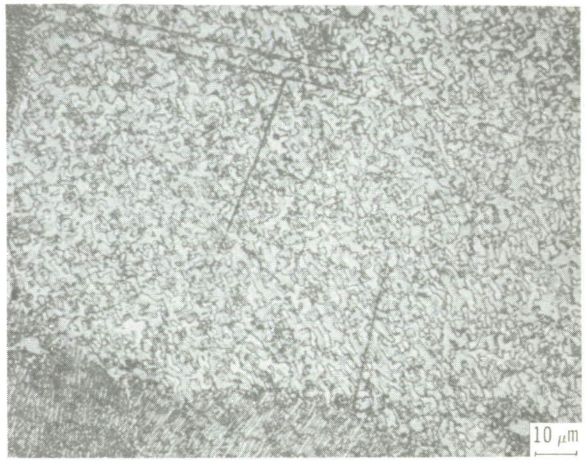
(d) IN-100 with medium Al + Ti content tested at 649° C and 655 MN/m^2; etch, mixed acids; approximate life, 12,000 hours.

Figure 10. - Microstructure of wrought IN-100 after stress-rupture testing.

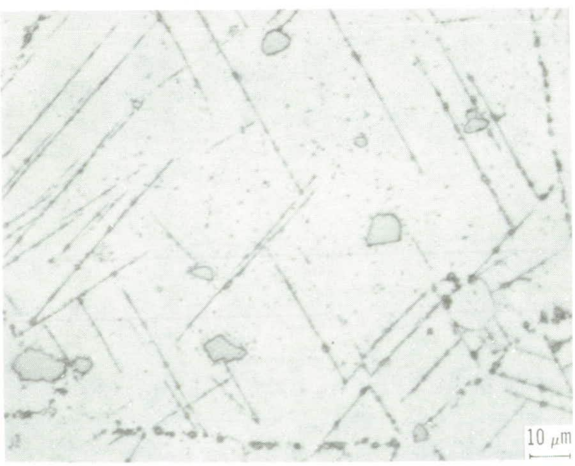
Results of stress-rupture tests. - Photomicrographs of selected samples which have been stress-rupture tested are shown in figure 10. Figures 10(a) and (b) show the appearance of the sigma-free wrought composition which was tested at 649^{o} C with a stress of 655 MN/m^2 (life, about 13 000 hr). Comparing these photomicrographs with figure 4(a) shows that the stress-rupture test has not changed the appearance of the microstucture. The microstrucure of the moderately sigma-prone composition which was tested at 649^{o} C with a stress of 655 MN/m^2 (life, about 12 000 hr) is shown in figures 10(c) and (d). A fine precipitation has occurred within the grains during the test. This type of precipitation had been previously



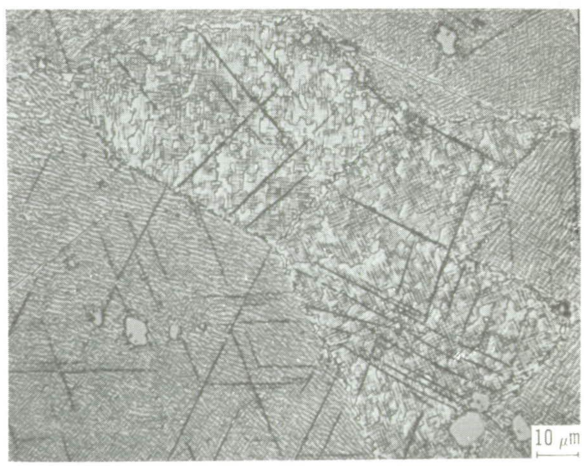
(e) IN-100 with medium Al + Ti content tested at 843° C and 276 MN/m^2; etch, KOH; approximate life, 1300 hours.



(f) IN-100 with medium Al + Ti content tested at 843° C and 276 MN/m^2; etch, mixed acids; approximate life, 1300 hours.



(g) IN-100 with high Al + Ti content tested at 885° C and 276 MN/m^2; etch, KOH; approximate life, 140 hours.



(h) IN-100 with high Al + Ti content tested at 885° C and 276 MN/m^2; etch, mixed acids; approximate life, 140 hours.

Figure 10. - Concluded.

observed in sigma-prone cast compositions (ref. 2) but had not been previously observed by the authors in unstressed isothermal exposures of the wrought compositions investigated. C. T. Sims of the General Electric Company has suggested that this fine precipitate is $M_{23}C_6$ and is a common precursor to sigma formation.

The microstructure of the moderately sigma-prone composition after it was stress-rupture tested at 843° C with a stress of 276 MN/m^2 (life, about 1300 hr) is shown in figures 10(e) and (f). The photomicrographs show that a small amount of sigma has precipitated during the stress-rupture test. Comparing the ductility and life of this composition with its sigma-free counterpart shows that the formation of this small amount of sigma in the moderately sigma-prone composition was not detrimental to the rupture life or ductility of the composition. This behavior is consistent with the observations of Moon and Wall (ref. 5) that small amounts of sigma phase were not detrimental to the stress-rupture properties of the two wrought alloys Udimet-520 and Udimet-500. In reference 6, Sims reflects that while property degradation of some Ni-base alloys by sigma-type phases has been documented, the effect has not been clearly shown to be true for all Ni-base superalloys. In this investigation it has been demonstrated that even for an alloy where sigma phase has been shown to be detrimental to some mechanical properties when it is present in large amounts, it can be tolerated in smaller quantities.

Sigma precipitation in the very sigma-prone composition is shown in figures 10(g) and (h). These photomicrographs were of a very sigma-prone composition which was tested at 276 MN/m^2 and 885° C. The average life of the composition at this condition was only 71 percent of the life of the sigma-free composition. The rupture ductility of the very sigma-prone composition was greater than that of the sigma-free and moderately sigma-prone compositions. The data in this program and those reported in references 1 and 2 appear to show a correlation between rupture ductility and rupture life, with the longer life tests having lower rupture ductility. Because sigma formation can decrease life, it is not clear if the increase in ductility with sigma formation observed in this investigation is the direct result of the sigma formation or if it is associated with the decreased life of the sigma-containing compositions.

CONCLUDING REMARKS

Although the presence of sigma phase in Ni-base superalloys is generally avoided in commercial practice, this investigation has demonstrated that in a wrought Ni-base superalloy the formation of small amounts of sigma phase need not be detrimental to tensile or rupture properties. This fact was demonstrated for an alloy (IN-100) where larger quantities of sigma phase have clearly been shown to be deleterious to these same mechanical properties. The data in this study suggest that the use of compositions that result in the presence of small amounts of sigma may be beneficial to stress-rupture properties at temperatures to 770° C. The effects of sigma on fatigue and fracture toughness have not been investigated and would have to be known before these benefits could be exploited.

For alloys of nearly the same composition, the mechanical properties of cast forms were affected much more adversely by sigma formation than those of wrought forms. We believe that this is related to the greater segregation of alloying elements in the cast compositions as compared to the wrought compositions. This segregation would cause the cast compositions to contain local regions with large amounts of sigma. These local areas could then act as weak regions in the structure.

SUMMARY OF RESULTS

To study the effect of sigma-phase formation in a wrought Ni-base superalloy with the composition of the cast alloy IN-100, three special compositions were prepared by varying only the Al and Ti content of a single master heat. These compositions were given a four-step heat treatment before the effects of exposure to temperature on mechanical properties were determined. The resultant compositions as heat treated were sigma free, moderately sigma prone, and very sigma prone when exposed to elevated temperatures. The following results were obtained from the study:

1. The stress-rupture life of the moderately sigma-prone composition was never observed to be significantly shorter than the life of the sigma-free composition at the test conditions used. At 704° and 773° C with a stress of 655 MN/m^2 and at 982° C with a stress of 138 MN/m^2 the moderately sigma-prone composition had a greater rupture life than the sigma-free composition. The very sigma-prone composition had a

significantly shorter stress-rupture life than the sigma-free and moderately sigma-prone compositions in the time-temperature realm where sigma forms. For example, at a test temperature of 843° C and a stress of 276 MN/m^2 the very sigma-prone composition had a life of approximately 310 hours, and the moderately sigma-prone and sigma-free compositions had lives of approximately 1300 hours.

2. The formation of small amounts of sigma phase in the moderately sigma-prone composition did not have any adverse effect on the composition's stress-rupture properties as compared to the properties of the sigma-free composition.

3. Exposure to 732° C for 1000 hours had no important effect on the room-temperature tensile properties of the sigma-free and moderately sigma-prone compositions. However, this exposure did reduce the ductility of the very sigma-prone composition from approximately 13 percent elongation to 5 percent. The yield strength of the very sigma-prone composition was unaffected; and the ultimate tensile strength dropped slightly, from 1210 MN/n^2 to 1130 MN/m^2.

4. Exposure to 843° C for 250 hours caused the yield strength of all compositions to decrease such that after the exposure they all had a room-temperature yield strength of approximately 850 MN/m^2. The effect on ultimate tensile strength was slight except for the very sigma-prone composition, where it dropped from 1210 MN/m^2 to 1040 MN/m^2.

For the very sigma-prone composition this exposure caused the room-temperature elongation to decrease from 13 percent prior to exposure to 3 percent after exposure. For the sigma-free and moderately sigma-prone compositions the room-temperature elongations were greater after the exposure to 843° C than prior to the exposure.

5. Although no sigma was present, the ductility in a room-temperature tensile test of compositions in the as-heat-treated condition decreased as the sigma-forming tendency increased. For example, the sigma-free composition had an elongation of 19 percent, the moderately sigma-prone composition had an elongation of 17 percent, and the very sigma-prone composition had an elongation of 13 percent. The room-temperature ultimate tensile strength and yield strength were essentially independent of the compositions' sigma-forming tendency.

6. Sigma formation caused greater reductions in stress-rupture life relative to the life of the sigma-free composition in cast alloys than in wrought alloys tested at the same stress and temperature. For example, the very sigma-prone cast compo-

sition tested at 276 MN/m^2 and 843° C had 12 percent of the life of the sigma-free composition, and the very sigma-prone wrought composition tested at the same stress and temperature had 23 percent of the life of the wrought sigma-free composition.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 12, 1973,
501-21.

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