RESPONSE OF IN-939 TO PROCESS VARIATIONS

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The development of the 4-stage heat treatment for the cast corrosion-resistant superalloy IN-939 is described. The effects on properties and structures of the various stages of heat treatment are reviewed and properties achievable for a 4h/1160°C + 6h/1000°C treatment, recommended for vane applications are provided. Cooling rate during heat treatment is shown to have a marked effect on some properties and grain boundary structure; properties are given for a simple heat treatment of 4h/1160°C + 16h/850°C, cooling at 50C°/min. Remarkable repair weldability, with the possibility of further improvement from increased ductility achieved by overaging in the eta phase range is noted and improved properties in DS castings with and without Hf additions are reported.

INTRODUCTION

IN-939 was developed to meet the need for a highly corrosion-resistant, cast, nickel-based superalloy for the blades and vanes of industrial and marine gas turbines (1) (2). The alloy has the chemical composition Ni-22.5%Cr-19%Co-2%W-1%Nb-1.4%Ta-3.7%Ti-1.9%Al-0.15%C-0.1%Zr-0.01%B. Because of the relatively high chromium content, IN-939 is several times more corrosion resistant than IN-738LC (16%Cr); creep-rupture stresses are at least as high as for IN-738LC for $>$10,000 hours life (3). In assessments for vane applications, the alloy has proved as corrosion resistant as the cobalt-based alloy FSX-414 (29.5%Cr), and has a surprising degree of repair weldability. IN-939 has been in commercial production and use since 1974, but work has continued on the alloy's response to variations in processing, in particular casting conditions, heat-treatment and welding. This paper describes the effects of heat treatment on properties and structure, the response to welding and the influence of unidirectional solidification on IN-939.

DEVELOPMENT OF 4-STAGE HEAT TREATMENT

The heat treatment used during the compositional development of IN-939 was full solution treatment for 4h/1150°C (2100°F), followed by ageing for 16h/850°C (1560°F). During the final stages of optimising the composition a parallel study of heat treatment was undertaken on a 15%Co, 3.5%Ti alloy, otherwise identical to the IN-939 composition. Development of a better heat treatment seemed necessary because the simple two-stage treatment gave very poor room-temperature tensile ductility (0.7% elongation) and marginal stress-rupture ductility.

The stratagem used to achieve good properties in IN-738, a partial solution treatment to produce coarse γ' and a single age to precipitate fine γ', did not work for the 15%Co-3.5%Ti alloy because η phase precipitated at temperatures suitable for partial solution of γ'. Nor was the four-stage heat treatment developed for Udimet 700 satisfactory: stress-rupture life was poor and there was no improvement in tensile ductility. However, when the temperature of the first age in that treatment was reduced from 1080°C to 1000°C, formation of η phase was avoided and both tensile and rupture ductility improved somewhat. A further improvement resulted from decreasing the temperatures of the third and fourth stage ageing treatments, but attempts to reduce the length and cost of treatment by omitting one stage or combining two stages at an intermediate temperature failed: all such treatments produced inferior stress-rupture life and omission of 6h/1000°C gave inadequate tensile ductility (4).

When the final composition of IN-939 was chosen, the four-stage heat treatment which gave the best properties in the 15Co-3.5Ti alloy and the original simple two-stage treatment were compared using IN-939. Again, the four-stage treatment gave better room-temperature tensile ductility, 3.0-3.8% compared with 0.5-0.7% from the two stage treatment, and better elevated temperature tensile and stress-rupture ductility. Therefore, for the standard heat treatment of IN-939 to yield the best combination of properties, it was decided to recommend a four-stage treatment, namely:

4h/1160°C + 6h/1000°C + 24h/900°C + 16h/700°C.

The first stage of this treatment takes all the γ' and practically all the η phase into solution and achieves significant homogenisation. The first age, at 1000°C, grows the γ' particles formed on cooling after solution treatment and precipitates a small amount of carbide on the grain boundaries. The second age, at 900°C, probably causes some further growth of the γ' particles and is responsible for major strengthening at the grain boundaries by precipitation of discrete particles of $M_{23}C_6$. The final age, at 700°C, precipitates fine secondary γ' particles, only about 100Å in diameter, markedly increasing the proof stress at ambient and intermediate temperatures (5).

FURTHER STUDY OF HEAT TREATMENT

Although the four-stage heat treatment adopted for IN-939 provides an excellent combination of properties, 50 hours at temperature are needed, plus heating and cooling time. Consequently, a shorter and cheaper heat treatment has been sought, by a combination of empirical experiments with a study of the effect of the various stages of treatment on mechanical properties and structure.

(i) Solution Treatment

Incomplete solution of η -phase, reported from a commercial vacuum heat treatment of large vanes, led to an investigation into the effect on mechanical properties of variations in solution treatment temperature between 1090 and 1210°C (Table 1). Poor stress-rupture properties were obtained under all test conditions, after 4-stage heat treatment involving solution treatment at 1090°C, at which temperature substantial amounts of γ' and η phase remain undissolved. After solution treatment at 1120°C, the critical longer-time rupture life was unsatisfactory, although life in the shorter-time tests was surprisingly good. The high room-temperature 0.2% proof stress suggests that solution of γ' is almost complete at 1120°C. In this set of results, solution treatment at 1150°C gave the best results although even after 1180°C treatment a life of 1832h was recorded. Tensile 0.2% proof stresses were a little lower and elongations marginally higher with 1160°C to 1180°C solution treatment than with 1150°C. With 1210°C treatment, the rupture lives plunged to low values with the onset of incipient melting at grain corners and in grain boundaries. These results, reinforced by metallographic evidence on the solution of η phase in block samples cut from the root block of a large vane, suggested that solution treatment temperature should be no lower than 1145°C; to allow a margin of error of at least $\pm$15°C in large commercial vacuum furnaces, it was decided that the recommended temperature should be 1160°C.

(ii) Primary Ageing

Short time stress-rupture properties were used to determine the effect of varying the temperature of the primary γ' precipitation treatment between 980°C and 1030°C, while keeping the other three heat-treatment stages unchanged. Stress-rupture life at 247N/mm²/900°C varied only between 49 and 60 hours, although γ' size increased from 1620 to 2510Å as ageing temperature was increased over the 50°C range. It was concluded that a practical specification of 1000°C ±15°C was satisfactory.

Omitting the 1000°C age raised 0.2% proof stress and short-time stress-rupture life (at 379N/mm²/816°C) but, as expected from the earlier work on the 15%Co-3.5%Ti alloy, decreased long-time stress rupture life (at 276N/mm²/816°C) and markedly reduced tensile ductility (Table 2). Restricting

TABLE 1

Effect of Solution Treatment Temperature upon the Stress-Rupture and Tensile Properties of IN-939

Test Conditions		Solution Treatment - 4 Hours†						
		1090	1120	1150	1160	1170	1180	1210
Stress-Rupture								
Stress N/mm²	Temp. °C	Life - h						
276 "	816 "	570	1111 1052	2057 1899	1726	-	1832 1404	791
330 "	" "	148 130	524 416	690 586	635 570	629 427	444 320	276 250
379 "	" "	21 13	207 109	212 198	173	164 143	140 97	46 46
120	927	328	623	902	753	646	574	221
20°C - Tensile Properties								
N/mm²	0.2% PS "	687 677	782 738	807 783	758 745	787 748	750 736	768 754
	U.T.S. "	803 899	978 901	967 919	981 937	968 926	928 884	951 951
%	Elong. "	1.6 4.4	4.3 2.1	3.9 2.4	4.0 3.9	3.6 3.9	4.7 3.1	3.6 2.4
	R. of A. "	4.7 3.9	8.5 7.8	14.5 -	7.8 9.3	7.8 14.5	8.5 13.8	11.6 12.3

† +6h/1000°C + 24h/900°C + 16h/700°C

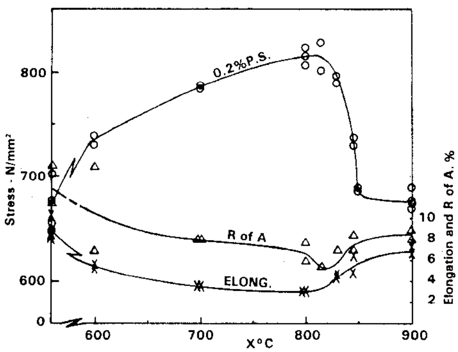


Figure 1. Room Temperature Tensile Properties of IN-939 Heat Treated 4h/1160°C + 6h/1000°C + 16h/x°C Air Cooled.

TABLE 2

Creep-Rupture and Tensile Properties of IN-939 with Various Stages of the 4 Stage Heat Treatment Omitted

Heat Tratment (Stages)				Stress-Rupture						Tensile - 20°C			
				379N/mm²/816°C		330N/mm²/816°C		276N/mm²/816°C		N/mm²		%	
1st	2nd	3rd	4th	Life h	Elong. %	Life h	Elong. %	Life h	Elong. %	0.2%PS	U.T.S.	Elong.	R. of A.
4/1150	6/1000	24/900	16/700	174 108	10.3 N.D.	586 448	8.0 8.2	1899 1808	8.6 8.1	783 739	937 929	3.2 2.7	5.0 4.0
"	----	"	"	275 218	5.4 3.7	472 462	3.3 7.5	1596 ---	4.8 ---	882 872	1034 1020	1.6 1.4	3.0 7.0
"	"	----	"	172 123	9.2 9.6	519 452	6.3 7.1	1874 1693	5.4 9.9	787 783	980 976	3.7 3.5	6.0 8.0
"	"	"	----	123 103	13.5 12.5	562 309	9.4 6.9	1579 1346	7.5 5.4	689 677	994 1020	7.3 6.8	9.0 8.0
"	"	----	----	125 71	10.6 N.D.	487 474	7.4 7.0	1722 ---	5.3 ---	648 655	970 923	10.2 8.2	15.0 12.0

heat treatment to solution treatment and primary ageing (6h/1000°C), i.e. omitting the 24h/900°C and 16h/700°C stages, reduced proof stress but only marginally affected longer-time stress-rupture life and greatly increased tensile ductility (Table 2). It seems that a simple solution treatment + primary age should be considered for applications not requiring the highest proof stress and short-time stress-rupture properties.

(iii) Secondary Ageing

Omitting the 24h/900°C age from the 4-stage treatment had little effect on stress-rupture and tensile properties (Table 2), whereas omitting the 16h/700°C age reduced stress-rupture life, particularly at higher stresses, and proof stress but increased tensile ductility. Hence the 900°C age seems to contribute least benefit to IN-939, whereas secondary γ' precipitated during the 700°C age adds to the hardening, increases proof stress and, in consequence, reduces ductility.

To explore secondary ageing further, tensile properties were determined on IN-939 heat treated for 4h/1160°C + 6h/1000°C and additionally aged for 16 hours at temperatures between 600°C and 900°C. The variation of room-temperature proof stress with temperature of the second age (Figure 1) maps out the temperature range over which secondary γ' precipitates. A 180C° difference between the temperatures of primary and secondary ageing is needed to achieve maximum additional hardening, and secondary strengthening does not occur at 850°C or above. This conclusion is consistent with the observation that the secondary γ' precipitate, formed in the final age of the 4-stage treatment, redissolves within 100 hours at 816°C or 24 hours at 870°C (6).

(iv) Properties of IN-939 Heat Treated for 4h/1160°C + 6h/1000°C

For rotating blades requiring high strength in the blade root, where temperature is below 600°C and secondary ageing in service is unlikely, a heat treatment which includes a secondary age will be needed to develop adequate tensile strength. However, for vanes traditionally cast in cobalt-base alloys, which do not have the high tensile strength of γ' hardened nickel-base superalloys, the simple 4h/1160°C + 6h/1000°C treatment should be adequate. The good ductility provided by that treatment should also prove beneficial in adjusting castings after heat treatment and repair welding. In view of the interest shown in replacing high-cost cobalt alloys by IN-939 in some stator vanes, additional data have been determined on IN-939 given this cheap two-stage treatment.

Figure 2 compares the stress-rupture properties of IN-939 given the two- and four-stage heat treatments. As already noted, 100 hours at 816°C or 24 hours at 870°C is sufficient to dissolve any secondary γ' precipitate at 700°C, so it is not surprising that stress-rupture lives for two-stage treated material fall on the average property line for four-stage treated IN-939 at temperatures of 870°C or above. Both heat treatments give the same stress-rupture life at 816°C when lives are above about 150 hours, by when secondary γ' precipitated at 700°C in four-stage-treated material will have dissolved. The two- and four-stage heat-treated materials are indistinguishable in stress-rupture at 760°C and below, presumably because secondary γ' precipitates in two-stage treated IN-939 during the 16 hour soak at temperature before stress is applied to the stress-rupture specimen.

Tensile data (Figure 3) reveal a double peak in 0.2% proof stress and UTS after the two-stage treatment. The second peak, at 800°C, is probably the result of rapid secondary γ' precipitation and resultant strengthening during the tensile tests.

EFFECT OF COOLING RATE IN HEAT TREATMENT

All the work reported so far employed air cooling between each stage of the heat treatments, using the standard laboratory procedure of scattering the test-bar blanks on the

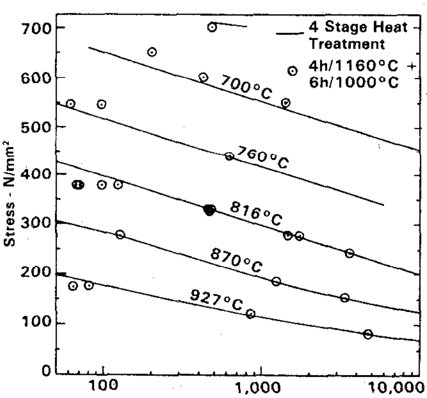


Figure 2. Creep-Rupture Properties of IN-939 Heat Treated 4h/1160°C + 6h/1000°C Air Cooled

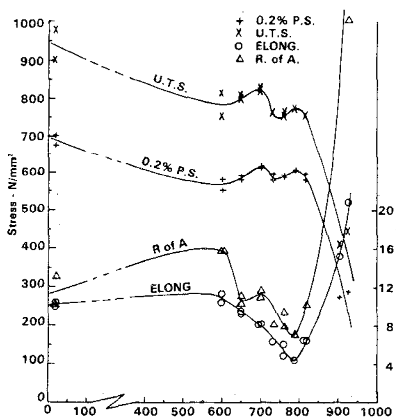


Figure 3. Tensile Properties of IN-939 Heat Treated 4h/1160°C + 6h/1000°C Air Cooled.

floor or bench. However, cooling rates achieved in commercial vacuum heat-treatment furnaces by rapid gas fan quenching are somewhat slower.

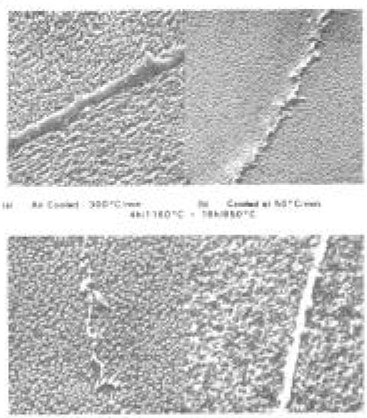
It had been noticed that the primary γ' is coarser in components vacuum heat treated commercially than in the same components heat treated in the laboratory, because slower cooling after solution treatment allows greater time for the γ' to grow. Since it is the growth of coarse primary γ' which appears to give good tensile ductility to material heat treated for 4h/1160°C + 6h/1000°C using air cooling, it seemed possible that growth of primary γ' on slow cooling from solution treatment might have the same effect and improve the ductility obtainable with the 6h/1160°C + 16h/850°C treatment originally used for composition development. To investigate that possibility, carrot-shaped test-piece blanks were given that two stage treatment, cooling from solution treatment at 300C°/min (air cool), 50C°/min (in 50mm diameter steel cans filled with foundry grog, to simulate a good vacuum-furnace gas fan quench) or 17C°/min (in refractory brick, to simulate a poor gas fan quench). The slower cooled material had much better tensile ductility, with a proof stress rather below that achievable with the four-stage treatment using air cooling (Table 3).

A marked change in the morphology of the grain-boundary carbide accompanied the reduction in cooling rate (Figure 4). The thick, continuous carbide film formed on cooling at 300C°/min was replaced by discrete particles projecting into the grains. The grain-boundary carbide in air-cooled 4-

TABLE 3

Effect of Cooling Rate on the Tensile Properties of IN-939 Given a 2 and 4 Stage Heat Treatment

Heat Treatment	Cooling Rate °C/min	20°C Tensile			
		N/mm²		%	
		0.2% PS	U.T.S.	Elong.	R. of A.
4h/1160°C + 6h/1000°C +24h/900°C +16h/700°C	300	800 771	950 965	3.0 3.8	9.0 9.0
4h/1160°C +16h/850°C	300	830 814	875 868	0.7 0.5	2.0 2.0
"	50	765 756	1006 951	6.5 6.8	9.8 7.2
"	17	701 692	1017 1007	7.1 7.1	8.0 13.4



(a) Air Cooled 300°C/min (b) Cooled at 50°C/min
4h/1160°C + 16h/850°C

(c) Cooled at 17°C/min
4h/1160°C + 16h/850°C

(d) Air Cooled 300°C/min
4h/1160°C + 6h/1000°C + 24h/900°C + 16h/700°C

Figure 4. Effect of Cooling Rate on Grain Boundary Structure of IN-939.

stage treated material (Figure 4d) has an intermediate morphology, consistent with the intermediate ductility in that condition of heat treatment, although no doubt γ' size also has an effect.

Since proof stress of the slow-cooled two-stage treated material is lower than that of air-cooled four-stage treated alloy, the shorter time stress-rupture properties at intermediate temperature would be expected to be similarly lower. That is confirmed by results to date (Figure 5), which nevertheless show a trend for the two-stage properties to match those of the four stage material in longer times - probably in 10,000 hours at 760°C and 816°C, in 1,000 hours at 870°C and in 250 hours at 900°C. Tests are planned to check the properties of slow-cooled two-stage treated material out to 10,000 hours, although some European companies have already adopted the heat treatment for vanes.

Tensile tests on material heat treated for 4h/1160°C + 16h/700, 750, 800 or 850°C with cooling at 50C°/min showed that ageing at 750°C rather than 850°C provides proof stress slightly higher than achievable with the 4-stage treatment using air cooling, together with at least as good ductility. Stress-rupture tests are now needed to determine whether this is the alternative cheaper heat treatment, for rotating blades, which the author has been seeking for some time!

WELDABILITY OF IN-939

For IN-939 to be a satisfactory substitute for cobalt alloys in vanes, the alloy must possess adequate repair weldability. It was expected that IN-939 would be as unweldable as IN-738LC, but simple tests with cast matching filler rods revealed that small and large scrap vanes can

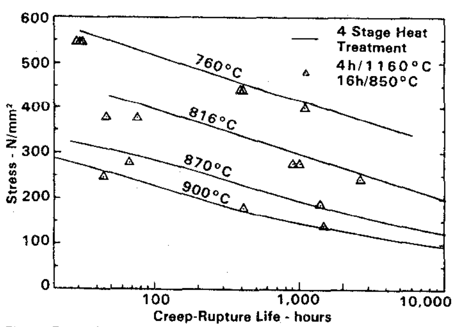


Figure 5. Creep-Rupture Properties of IN-939 Heat Treated 4h/1160° + 16h/850°C Cooled at 50°C/min.

TABLE 4

Effect on the Tensile Ductility of IN-939 at 788°C (1450°F) of a Pre-Weld Heat Treatment*

Heat Treatment		Tensile- 788°C (1450°F)			
Time	Temp.	N/mm²		%	
h	°C	0.2% PS	U.T.S.	Elong.	R. of A.
4	1038	621	806	7.7	19.5
"	1052	641	856	7.8	15.9
"	1066	621	825	8.5	20.3
"	1080	593	833	8.8	11.6
"	1093	597	816	9.4	16.0
20	1038	640	843	8.0	16.7
"	1052	604	809	13.0	26.5
"	1066	624	865	7.5	12.3
"	1080	612	828	7.9	23.8
"	1093	607	833	9.3	N.D.

* As-cast carrot shaped test-bar blanks heat treated in foundry grog in 2in O.D. mild steel can to give a cooling rate of 50°C (90°F)/min.

be repair welded and are crack free when redressed to size and dye penetrant tested (7). In addition, 12mm thick cast plates have been successfully butt-welded together, using a double-V joint preparation, and 80% joint efficiency was obtained on a creep-rupture test-piece cut out across the weld. For the repair of very large vane segments and large fabrications, pre-weld heat treatments may be found helpful. For material given the suggested heat treatment for vanes of 4h/1160°C + 6h/1000°C, the minimum tensile ductility was found to occur at 788°C (Figure 3). The ductility at that temperature was much improved by over-ageing in the range of temperature in which η-phase precipitates (1038-1093°C). A 4h or 20h age at 1093°C, followed by cooling at 50C°/min, attained an elongation of over 9% (Table 4) compared with 4.5% for material treated for 4h/1160°C + 6h/1000°C (air cooled). Further work on welding of IN-939 is continuing, at the Inco Research and Development Centre, Sterling Forest.

DIRECTIONAL SOLIDIFICATION

Although IN-939 was originally developed as a conventionally cast (C-C) alloy, creep-rupture properties have been found to benefit from directional solidification (DS). When CC and DS materials were compared using the 4-stage heat treatment, this benefit was evident only at the higher temperatures (870° and 927°C) and at times in excess of ~600h at 816°C (8). At 760°C, the DS material gave inferior lives to those of the C-C material. These surprising results were attributed to the extremely high ductility of the DS material. Since the 4 stage treatment was developed to produce greater ductility, which

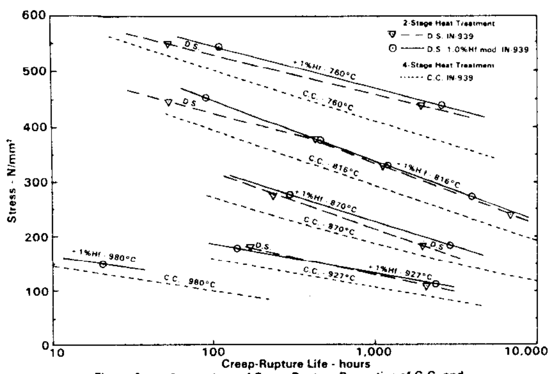


Figure 6. Comparison of Creep-Rupture Properties of C.C. and D.S. IN-939 and 1%Hf Modified IN-939

is not necessary for DS material, the tests were repeated with DS material heat treated for 4h/1160°C + 16h/850°C (air cooled). Benefit was then obtained from directional solidification at all test times and temperatures, including short lives at 816°C and 760°C (Figure 6). Hafnium additions with slight reductions in Nb, Ta and W contents produced further benefits, 1%Hf appearing to be the best in the combinations tested (Figure 6).

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