THE DEVELOPMENT OF ODS SUPERALLOYS FOR INDUSTRIAL GAS TURBINES

R. C. Benn* and G. M. McColvin**

* Inco Alloys International, Inc.
Huntington, WV 25720 USA

** Inco Alloys, Limited
Hereford, HR4 95L, England UK

Abstract

Oxide dispersion strengthened (ODS) alloys have the significant attribute of being able to retain useful strengths up to a relatively high fraction of their melting points. Moreover at the higher operating temperatures characteristic of advanced gas turbine engines, these alloys display long-term strength beyond the capabilities of conventional superalloys. The increasing use of ODS alloys, particularly in industrial applications, emphasizes the need not only to measure the creep-rupture performance and stability of these alloys but also to predict their behavior in long-term service. Characterization of the long-term properties and structural stability of commercial alloys such as INCONEL* alloy MA 6000 has identified some unique stress rupture behavior characteristics. Two distinct regions of the rupture stress vs. time curves have been identified which together show that higher design stresses for long-term service can be realized than predicted from only short term data. This behavior will be reviewed in relation to the development of a new class of ODS superalloys, typified by INCONEL* alloy MA 760, which combine the long-term strength attributes with the requirements for severe hot corrosion resistance typical of many industrial applications such as gas turbines and similar processes.

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Introduction

The need for long-term data on new experimental alloys with only short-term results presents a familiar dichotomy. This problem of determining component performance from laboratory tests is fundamental in materials engineering. Frequently, high temperature strength data are needed for conditions where there is no experimental information. This is particularly true for long-time creep and stress rupture data where it is quite possible for the design engineer to require the creep strength to give 1% deformation in 100,000 h (~11 years) when the commercial alloy has been in existence only for about 2 years. The increasing use of ODS alloys, particularly in industrial applications, has created a need to measure the creep rupture performance of these alloys and to predict their behavior in long-term service. When other property requirements such as corrosion resistance, oxidation resistance, structural stability, etc. are progressively factored into the compositional development there are often problems of mutual exclusivity. For example, the extent to which strength is traded off against hot corrosion resistance must be balanced and preferably minimized.

From previous reported studies (1,2) on the long-term properties and related microstructures of ODS alloys it is known that INCONEL alloy MA 6000 (Table I) displays unusual behavior in that the rupture strength decreases slowly at high temperatures resulting in a leveling off (i.e. an upward departure from linearity) of the Larson-Miller curve. When the data are plotted as stress vs. log time, the upward inflection is clearly evident; as shown in Figure 1 for selected temperatures in the range 750-950°C. This unique behavior has important material design implications because the long-term stress capability of INCONEL alloy MA 6000 in the temperature range of, say, 750-1000°C is superior to initial estimates derived by extrapolation of relatively short-term data. Estimates of the long-term rupture stress capability in the 10^4-10^5 life range at a given temperature should be made strictly by using data beyond the inflection point of the stress rupture curve. As illustrated in Figure 1, σ_2 is the

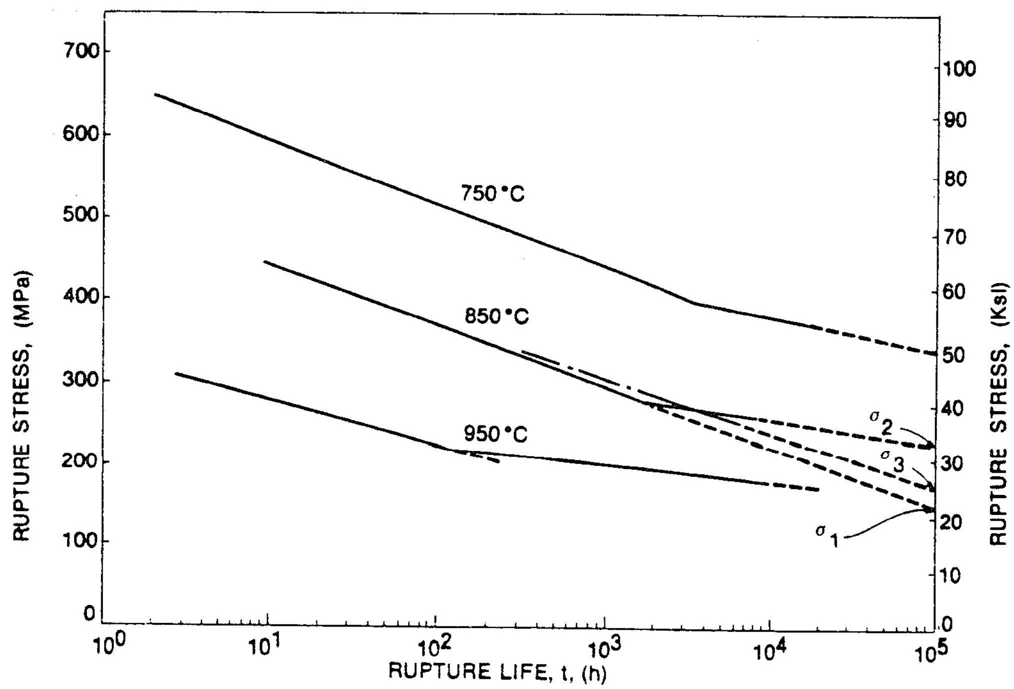

Figure 1. Implications of upward break on long term design stress estimates.

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actual long-term stress capability rather than σ_1 or σ_3 which are extrapolations or interpolations respectively of short-term data where clearly $\sigma_2 >> \sigma_3 > \sigma_1$.

While INCONEL alloy MA 6000 has excellent corrosion resistance in aerospace applications for which it was designed, new ODS superalloys are being developed by Inco Alloys International, Inc. for industrial applications that require extremely high corrosion resistance typical of IN-939. A wide range of experimental alloys, including the examples of alloys 1-3 given in Table I have been characterized with particular emphasis on long-term strength and corrosion/oxidation resistance. This work will be reviewed in relation to the development of a new class of ODS superalloys typified by INCONEL alloy MA 760.

Table I: Alloy Compositions (Wt. %)

Element	INCONEL alloy MA 6000	INCONEL alloy MA 754	IN-939	alloy 1	alloy 2	alloy 3	INCONEL alloy MA 760
Ni	Bal	Bal	Bal	Bal	Bal	Bal	Bal
Cr	15	20	22.5	19.7	19.5	20.8	20
Al	4.5	0.3	1.9	4.5	6.7	2.7	6
Ti	2.5	0.5	3.7	2.5	-	3.4	-
Ta	2.0	-	1.4	2.0	-	1.7	-
Nb	-	-	1.0	-	-	-	-
W	4.0	-	2.0	4.4	3.8	2.0	3.5
Mo	2.0	-	-	2.0	2.0	-	2.0
Co	-	-	19.0	-	-	9.7	-
C	0.05	0.05	0.15	0.05	0.044	0.057	0.05
B	0.01	-	0.009	0.012	0.011	0.014	0.01
Zr	0.15	-	0.10	0.075	0.15	0.20	0.15
Y_2O_3	1.1	0.6	-	0.6	0.6	0.6	0.95

Experimental Procedure

Experimental heats were mechanically alloyed initially in 10-S attritors (approx. 6 kg charge) used for compositional development. Selected alloys have been scaled up to commercial size 100-S (35 kg) attritors and a large-scale, commercial horizontal ball mill production route is now being developed. The mechanically alloyed powder was screened, canned and extruded followed by rolling as necessary. The bar products were directionally recrystallized, heat treated and evaluated for mechanical properties and structural characteristics. Cyclic and isothermal oxidation tests were run at 1100°C. Burner Rig hot corrosion tests were performed at 927° and 1093°C.

Results and Discussion

From consideration of the long-term rupture behavior of INCONEL alloy MA 6000 (1,2) it is reasonable to assume that the characteristics shown in Figure 1 may be advantageously used in the design of more sulfidation/oxidation resistant alloys. Three groups of ODS Ni-20 w/o Cr-based superalloys were developed through compositional variations nominally represented by experimental alloys 1-3 in Table I. The alloys contained, inter alia, Al contents in the range 3-7 w/o with various levels of other γ' phase and solid solution elements.

Table II shows that the sulfidation resistance of these alloys was considerably improved over INCONEL alloys MA 6000 and MA 754 being comparable with IN-939. Moreover, the oxidation resistance was considerably

Table II: Oxidation and Burner Rig Hot Corrosion Resistance

Alloy	Cyclic Oxidation* Descaled Mass Change (g/m^2)	Burner Rig Corrosion** Metal Loss (μm)	Burner Rig Corrosion** Metal Attack (μm)
alloy 1	-1.55	<2.54	55.88
alloy 2	-0.093	5.08	86.36
alloy 3	-16.521	2.54	91.44
INCONEL alloy MA 6000	-1.205	25.4	88.90
INCONEL alloy MA 754	-1.703	17.78	101.60
IN-939	-25.501	2.54	78.74
IN-738	-11.678	2.54	99.06
IN-100	-1.090	Destroyed in Test	

*504 h, 1100°C, Air + 5% H_2O, 24 h Cycle to Room Temperature
**504 h, 927°C, 1 cycle/h (58 min. in flame, 2 min. out in air)
30:1 ratio of air + 5 ppm sea water to fuel (0.3% S, JP-5)

improved over IN-939 with the high Al alloy group being outstanding. The mechanical properties of these three groups were generally similar to INCONEL alloy MA 6000 with slightly lower rupture strengths as shown in Figure 2.

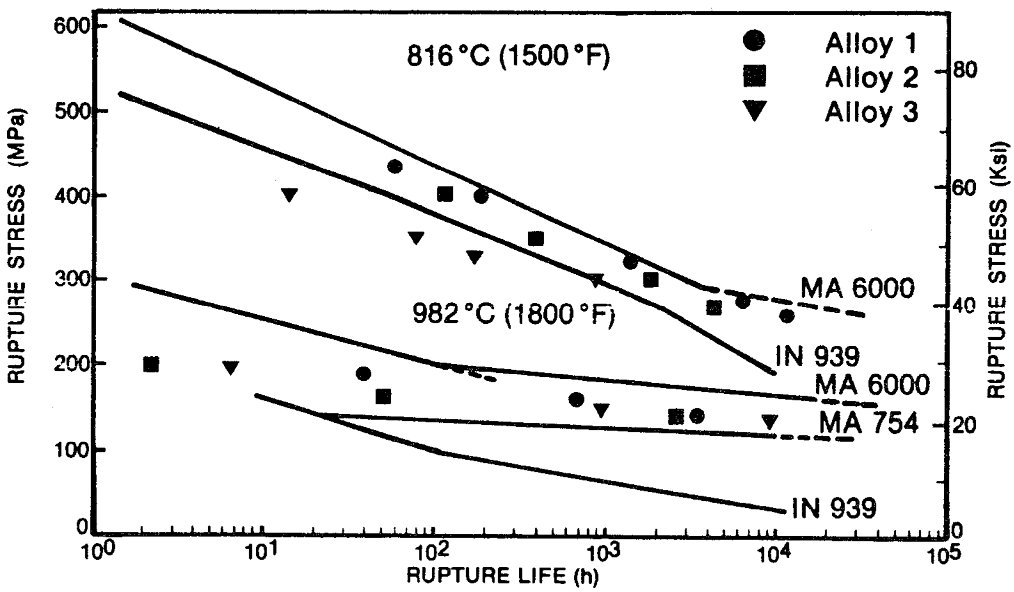

Figure 2. Comparison of stress rupture behavior of experimental alloys vs. commercial alloys.

Based on these results and some independent follow-up evaluations by an engine manufacturer, two alloys were selected from the high Al group of experimental alloys (Group 2) and one from Group 1 for scale-up production at Inco Alloys Ltd. using commercial-sized equipment. Evaluation of these alloys led to the selection of a Group 2 alloy designated INCONEL alloy MA 760 (see Table I for composition) for detailed commercial development. Similar practice to that used for other commercial ODS alloys was employed. In particular, a master alloy approach was adopted in the formulation of the final chemistry which has been found to simplify raw materials handling and enhance product reproducibility and, above all, cleanliness (3).

Based on previous studies (4) a Burner Rig hot corrosion test at 1093°C was selected as a more discerning method to compare the hot corrosion/oxidation performance of INCONEL alloy MA 760 against other materials. The results in Figure 3 and Table III clearly show the benefits of the compositional design of INCONEL alloy MA 760 in minimizing both metal loss and depth of attack.

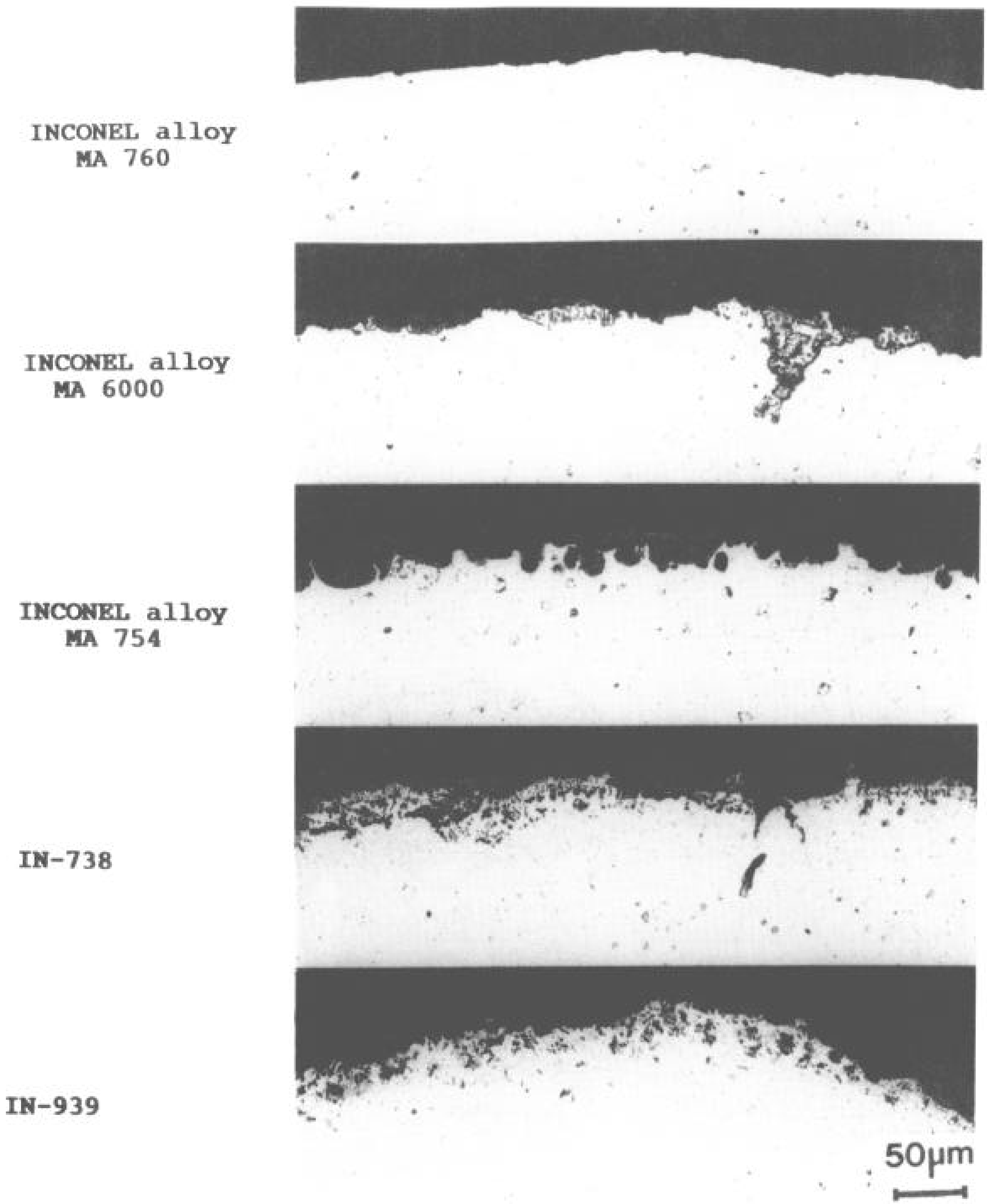

Figure 3. Micrographs showing surface integrity of tranverse sections of cylindrical specimens after 324 hours of 1093°C Burner Rig hot corrosion testing.

Table III: Burner Rig Corrosion Results[a]

Alloy	Metal Loss (µm) 324(h)	Metal Loss (µm) 504(h)	Depth of Attack (µm) 324(h)	Depth of Attack (µm) 504(h)
INCONEL alloy MA 760[b]	+7.6	+10.2	+10.2	172.7
INCONEL alloy MA 6000	20.3	91.4	81.3	358.1
INCONEL alloy MA 754	40.6	322.6	40.6	469.9
INCONEL alloy 617	(c)	551.2	(c)	736.6
IN-738	726.4	(c)	(c)	(c)
IN-939	1485.9	(c)	1546.9	(c)

Notes:

(a) 1093°C (2000°F) 1 cycle/hour (58 min. in flame 2 min. out in air) 30:1 air to fuel (0.3% S in JP-5 fuel + 5 ppm sea salt) ratio. Mass flow 5.5 ft^3/min.
(b) gain in diameter due to very adherent scale
(c) Not determined

Overall, the new alloy appears to meet the surface integrity requirements of both high temperature e.g. aerospace and/or long-term exposures e.g. industrial applications.

In recrystallization studies of commercial size bar (20 x 60 mm section), the alloy showed full recrystallization after static anneals in the 1200-1280°C temperature range. Directional recrystallization (DR) studies,

DR Temp. (°C)	Speed (mm/h)
1260	115
1260	95
1240	115
1260	75

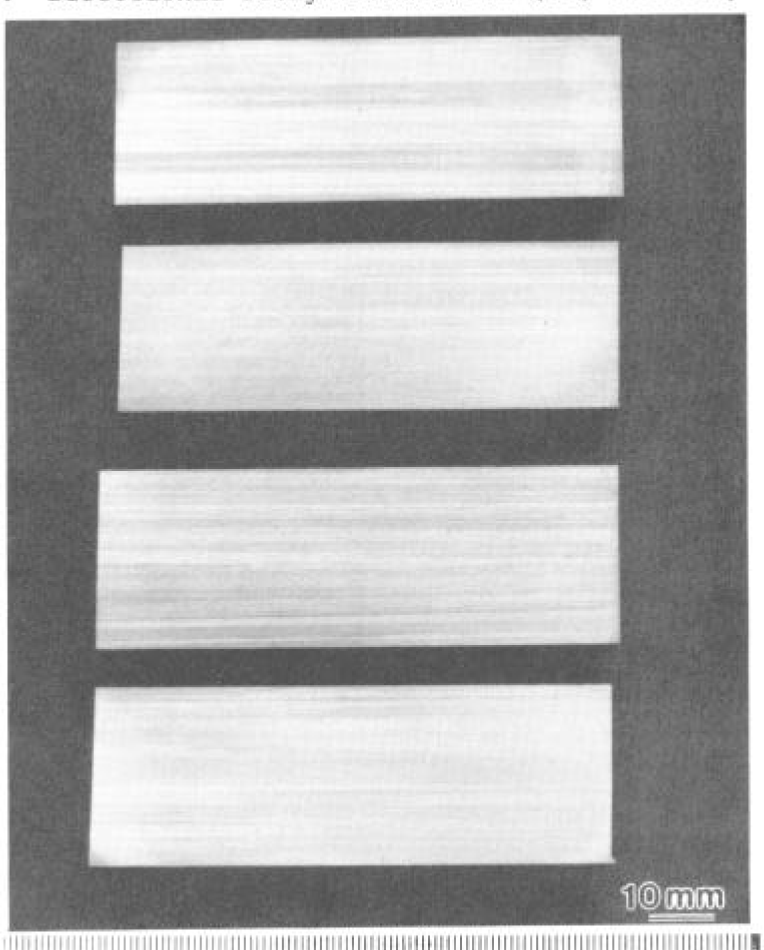

Figure 4. Macrostructure of directionally recrystallized INCONEL alloy MA 760.

normally used to enhance the grain aspect ratio, gave excellent response, as shown in Figure 4, within this range of temperatures at speeds up to approximately 150 mm/h.

The room temperature tensile properties of the scale-up bar showed little variation between the DR conditions and were similar to results obtained from laboratory-produced material given in Table IV.

Table IV: Preliminary Tensile Properties of INCONEL alloy MA 760*

Test Temperature °C	(°F)	0.2 Y.S. MPa	(ksi)	UTS MPa	(ksi)	El (%)	R.A. (%)
21	(70)	1006	(146.0)	1107	(160.0)	3.6	4.0
404	(400)	1000	(145.0)	1091	(158.2)	4.0	4.0
427	(800)	960	(139.2)	1115	(161.7)	3.0	3.3
649	(1200)	969	(140.6)	1116	(161.9)	4.0	4.1
871	(1600)	615	(89.2)	652	(94.5)	4.0	13.7
1093	(2000)	141	(20.4)	141	(20.5)	14.7	41.4

*Directionally recrystallized and heat treated 1h/1240°C/FAC + 2h/955°C/AC + 24h/845°C/AC.

Similarly, stress rupture results to date on scale-up bar appear to be in general agreement with results on laboratory-produced bar which are shown in Figure 5. Some evidence of the upward inflection in rupture strength similar to INCONEL alloy MA 6000 is apparent from these results.

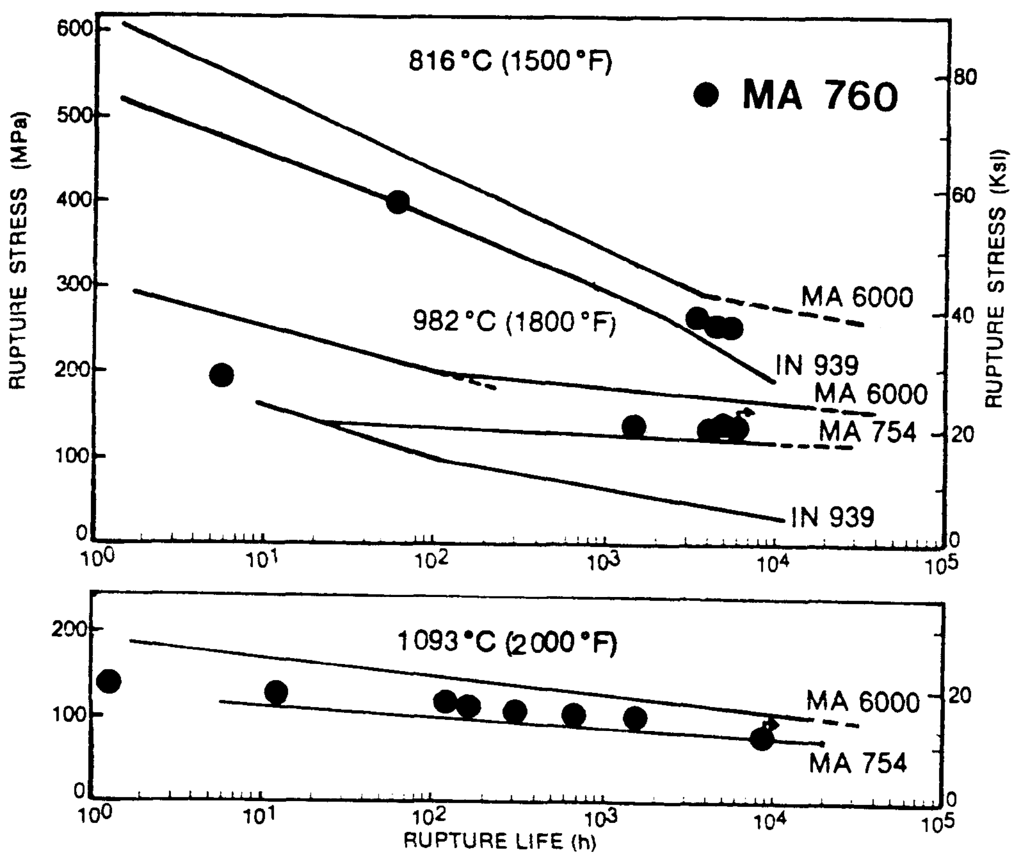

Figure 5. Preliminary stress rupture characteristics of INCONEL alloy MA 760.

A full program of mechanical property evaluations on scale-up bar is in progress. In addition, processing routes are being established to produce even larger production quantities of mechanically alloyed powder via horizontal ball mills and a range of larger section bar sizes.

Conclusions

1. Studies of the long-term stress and creep rupture characteristics of ODS alloys have identified unique stress-rupture behavior that implies significant advantages over conventional alloys particularly for long-term industrial applications.

2. A new class of ODS superalloys has been developed with strength characteristics approaching INCONEL alloy MA 6000 and hot corrosion resistance similar to IN-939 but with outstanding oxidation resistance for use, inter alia, in industrial applications.

3. An alloy composition designated INCONEL alloy MA 760 has been selected for commercial development and evaluation as, inter alia, industrial gas turbine airfoils.

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