P.M. SUPERALLOY FOR HIGH TEMPERATURE COMPONENTS

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ABSTRACT

The P.M. technology developed for the superalloys allows important improvements on mechanical properties at intermediate temperatures, and consistent savings on the final products. The objective of the reported studies was an extension of these technical and economical advantages to high temperature applications.

Superalloy materials have been densified from prealloyed powders of several compositions close to that of IN 100 . Powders were manufactured both by Argon Atomization of liquid metal, and by centrifugation using the CLET process. Compaction was performed both by Hot Isostatic Pressing and Hot Extrusion.

Processing characteristics are not fundamentaly affected by the powder manufacture process. For example, superplasticity can be developed by extrusion in all the materials studied. Heat treatments were chosen with metallography as a guide line, and impacts on the structure evaluated. Precipitate solution, recrystallization and grain coarsening are closely related to the type of powder.

User properties are slightly different at intermediate temperatures. They become considerably different at higher temperatures where the materials densified from CLET powders show a superiority due to their particular aptitude to grain growth, which is as marked in the HIP and forged materials as in the extruded materials. This is related to the morphologic and structural characteristics of the powder.

The influence of hardening element concentration has also been studied. An alloy has been selected and defined for the hotest turbine disks, the properties of which are, at all temperatures, superior that of the best cast-wrought superalloys.

INTRODUCTION

The superalloy materials elaboration from prealloyed powders has allowed a degree of isotropy and analytical homogeneity till then unknown in this type of material. This structural improvement has determined a progress of the endurance characteristics for the rotating components of aeronautical turbines working at temperatures lower than 650°C.

However, this limitation on the working temperature restricts their domain of application, particularly in the case of the high performing small machines, among which it is not rare to meet working temperatures higher than 750°C on the disk edges. For the alloy type IN 100, a classical material for conventionaly cast pieces, we have studied the influence of composition, powder processing hot-forming and heat-treating mode on high temperature mechanical properties. All along the study, we concentrated on solutions which should not jeopardize the important saving that the prealloyed powder metallurgy may procure. Among the diverse solutions studied, we shall present in this paper the results obtained on two composition variants derived from IN 100 by modifying the balance of different elements.

POWDERS

The powders used in this study have been processed by two different ways :

1 - by direct VIM liquid metal argon atomization (AA)

2 - by rotating electrode spraying according to the CLET* process.

The analysis of the powder batches is given in Table I.
The argon sprayed powder has been blended to - 100 Mesh and the CLET powder to - 25 Mesh. The granulometric distribution ready-for-use of both types of powders is presented on Figure 1. Blending of the argon sprayed powder is required by the occurrence of argon bubbles inside the powder grains. The proportion of hollow grains is in practice negligible if their diameter is less than 100 μ ; afterwards it increases quickly and attains 40 % for diameters greater than 250 μ. In the case of CLET powder, the particles are perfectly dense.

TABLE I - Powder batches elementary analysis

Type of powder	C	S	P	Si	Mn	Cr	Co	Mo	Ti	Al	V	Zr	B	O_2
Alloy 1 CLET	0,070	≤0,0020	0,0020	≤0,010	0,010	12,38	18,53	3,29	4,18	5,00	0,74	0,060	0,020	0,0060
Alloy 1 AA	0,070	0,0055	0,0036	0,050	0,004	12,59	18,40	3,03	4,48	4,80	0,81	0,055	0,022	0,0100
Alloy 2 - CLET	0,045	0,0030	0,0020	≤0,010	0,010	9,66	14,85	3,00	4,28	5,50	0,91	0,060	0,014	0,0060

AS-COMPACTED MATERIALS

The two types of powders, AA and CLET have been worked up with the composition 1. Densification has been carried out by hot isostatic pressure (HIP). Powders have been canned in soft steel containers. These have been sealed under vacuum after a careful hot degasing period at 500°C. Compactions have been made under 100 MPa argon pressure at 1150°C, 1190°C and 1230°C during 3 hours. The various properties (tensile and stress-rupture) have been measured.

Results and discussion

All the HIP samples are perfectly dense, except the CLET sample compacted at 1150°C which contains some residual porosity. The networks formed on the original particle boundaries represent tiny precipitates, almost all identified as TiC; it therefore reflects, on micrographic sections the granulometry of the initial powder (Figure 2). These carbides are generally present in CLET samples in shape of discrete globular particles of less than one micron in size (Figure 3A). Nevertheless, in the sample compacted at 1230°C, they are coalesced and tend to form continuous films after a partial melting at the interfaces (Figure 3B).

*French Patent 73-43735

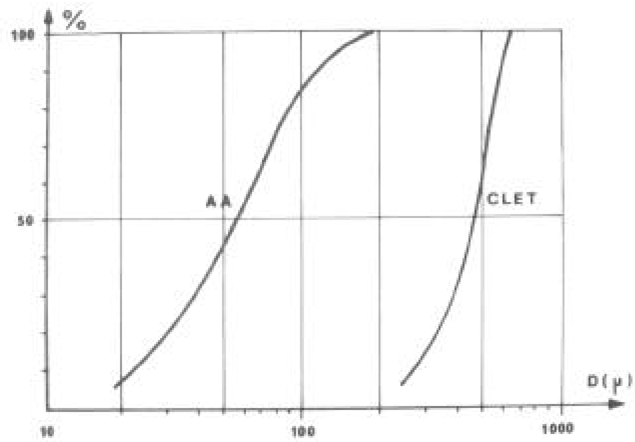
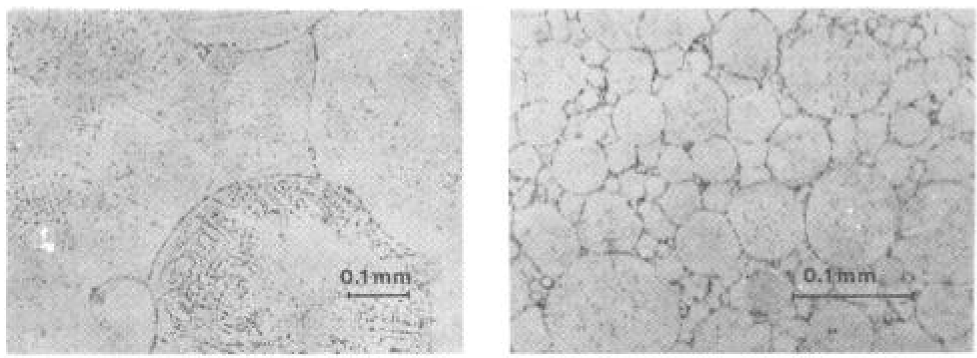
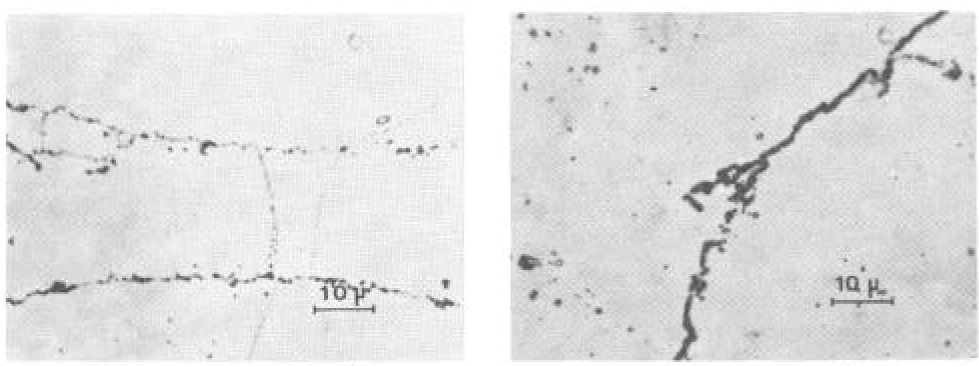


FIGURE 1 - Powder granulometry



A - CLET + HIP (1190°C, 3 h) B - AA + HIP (1190°C, 3 h)

FIGURE 2 - As-HIP Composition 1 - Carbide distribution



A - CLET + HIP (1190°C, 3 h) B - CLET + HIP (1230°C, 3 h)

FIGURE 3 - As-HIP Composition 1 - Carbide morphology

The following heat-treatment sequence has been applied to HIP materials :

(HT1) 1200°C (4 h) A.C. + 1080°C (4 h) A.C. + 850°C (24 h) A.C.
+ 760°C (16 h) A.C.

After this hot treatment, the AA samples microstructure is very little modified and is characterized by an ASTM 7-8 grain size. In the CLET samples (Figure 4), it suppresses all the structural and chemical heterogeneities, and determines an ASTM 3-4 crystallization if the sample is HIP at 1190°C or ASTM 2-3 if carried out at 1230°C.

For the tensile and stress-rupture properties (Table II), CLET samples are distinguished from AA samples by some intergranular brittleness, and stress-rupture lives shorter for the 650°C test and longer for the 980°C test. However, the hot-work torsion tests, on the HIP compacts does not reveal any influence of the powder manufacturing method.

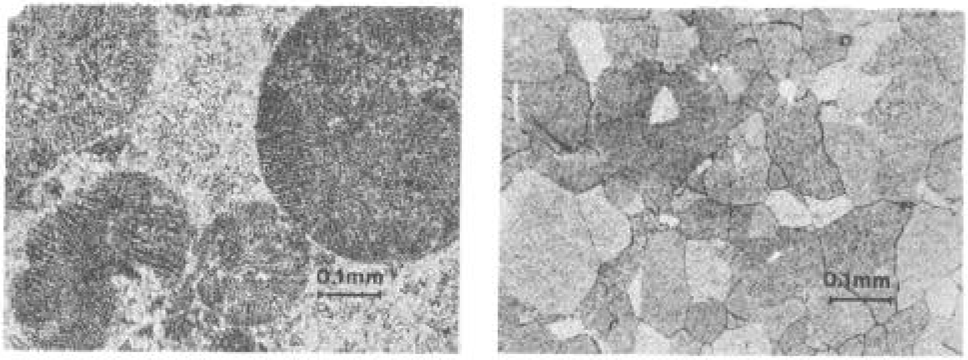
TABLE II - As-compacted composition 1 material.
Mechanical properties.

TENSILE TESTS						
Type of powder	HIP Temp.(°C)	Testing Temp.(°C)	E 0,2 (MPa)	R (MPa)	El. (%)	R.A. (%)
AA	1150	Ambient	1055	1453	22,7	24,2
	1190	Ambient	1041	1442	25,3	27,7
		650	942	1286	21,3	28,3
	1230	Ambient	1072	1380	12,9	15,2
		650	987	1307	14,3	21,5
CLET	1150	Ambient	996	1024	1,7	5,2
	1190	Ambient	1035	1062	1,3	3,5
		650	929	1050	1,9	5,2
	1230	Ambient	1028	1028	0,2	2
		650	954	1057	2,1	5,2

STRESS-RUPTURE					
Type of powder	HIP Temp.(°C)	Testing Temp.(°C)	Stress (MPa)	Rupture life (h)	El. (%)
AA	1150	650	1030	19,5	8,7
		980	122,5	6,25	11,6
	1190	650	1030	22	8,4
		980	122,5	7	7,3
	1230	980	122,5	18	3
CLET	1150	650	1030	6	4,1
		980	122,5	34,5	4,3
	1190	650	1030	1,5	3,8
		980	122,5	31,5	3,9
	1230	980	122,5	64	2,7

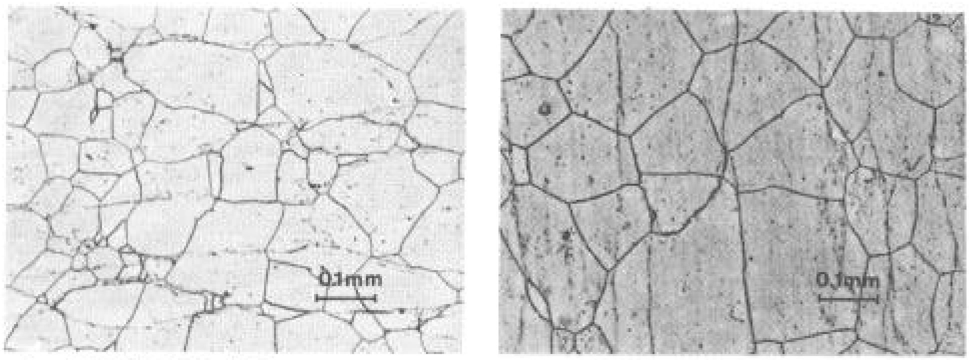
HIP AND FORGED MATERIALS

Studied material was compacted from CLET powder. The HIP compact was made at 1190°C during 3 h under a 1000 bar argon pressure. The HIP container was removed by milling and the compact put in a new steel container before forging. Pancake forging was made by press forging at 1140°C with a total forging ratio of 3. Evaluations included optical microscopy to study the influence of heat treatment, and the product was characterised by tensile, stress rupture, creep, low cycle fatigue and thermal shock tests.



A - CLET + HIP (1190°C, 3 h) B - CLET + HIP (1190°C, 3 h) + HT1

FIGURE 4 - As-HIP Composition 1 -
Effect of heat-treatment on microstructure



A - 1200°C, 32 h, A.C. B - 1200°C, 32 h + 1240°C, 4 h, A.C.

FIGURE 5 - HIP + Forged Composition 1 -
Effect of annealing conditions on microstructure

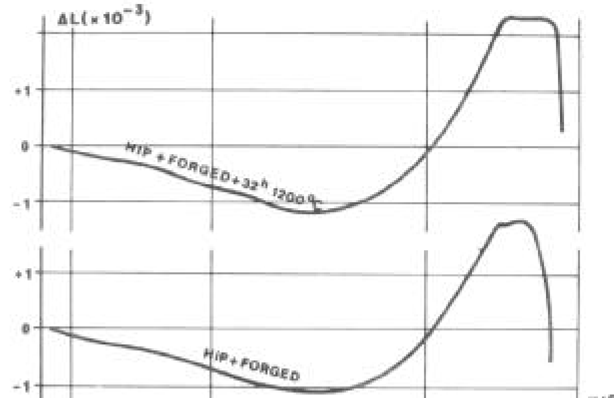


FIGURE 6 - Differential Dilatometric Curves

Metallography

As-forged material has a partially recrystallised structure. As for HIP compacts, a network of titanium carbides defines limits of the original powder particles. This structure can change by heat treatment. The carbides network solution is almost complete after a treatment at 1200°C for 16 h. After this treatment, the grain size which was limited at 4 ASTM, grows and reaches 3 ASTM after 32 h (Figure 5A). An explanation for this phenomenon has been proposed elsewhere (1). The solution and the accompanied homogenization can be followed by a dilatometric test. Figure 6 presents the differential dilatometric curves for a HIP and forged material before and after a treatment of 32 h at 1200°C. The as-forged sample presents (2) a faintly distinguishable γ' solvus at about 1145°C, typical of PM products (2). Above 1190°C, a deep falling-in is observed. Optical microscopic examinations shows that the incipient melting can be observed only above 1215°C. After annealing at 1200°C, the γ' solvus temperature is very clear and the incipient melting temperature, which is also well defined, is raised to 1247°C. During high temperature annealing, the titanium carbide solution is joined with a local excess elevation of the titanium concentration and a lowering of the incipient melting temperature in these enriched zones. This explains the observed structure of the 1230°C HIP CLET sample.

Mechanical properties

The tensile and stress-rupture results are reported on Figure 7 and Table III. After heat treatment (HT1), the ductility of the HIP + forged material is much higher, and the 650°C stress-rupture lives longer, than those of as-compacted materials. Low cycle-fatigue, carried out on notched specimens using a load cycle between $\sigma/10$ and σ with a 1 period are fairly good (Figure 8). The thermal shock test is a high stress test induced by a low frequency temperature cycle. It requires fastening a notch test sample between two bulk pieces joined by columns, the sample being alternatively heated by the Joule effect then cooled by a compressed air stream (3). The results plotted on the Figure 9 allow the comparison of the rupture cycle number as a function of the maximum, or peak temperature for several materials.

TABLE III - HIP + Wrought IN 100 - Composition 1 - H.T.1
Stress rupture properties

Material	Testing temp.(°C)	Stress (MPa)	Rupture life (hours)	El. (%)
HIP + Forged	650	1000	37,5	8,4
HIP + Extruded	650	1000	53,5	14,1
HIP + Forged	760	595	47	5,7
HIP + Extruded	760	595	60,3	14,4
HIP + Forged	850	280	293	5,1
HIP + Extruded	850	280	271	9,1
HIP + Forged	980	122,5	67,8	11,5
HIP + Extruded	980	122,5	50	14,1

The properties of the material for the tensile, stress-rupture, low-cycle fatigue and thermal shock tests are at least as good as that of the best conventionally cast-wrought alloys.

HIP AND EXTRUDED MATERIALS

For this study, billets ere densified from CLET powder by HIP (3 h at 1190°C under pressure of 1000 bar), canned in a steel container, and then extruded at 1150°C, with an extrusion ratio of 11, on an horizontal press.

Metallography

The as-extruded material presents a TiC network fixing the original powder grains, these grains being stretched along the extrusion direction. An acid attack reveals a microstructure with numerous γ' precipitates of 1 to 2 μ size, uniformly distributed. This microstructure is typical of superplasticity (4), this point being confirmed by stress-rupture tests between 950 and 1100°C. As in the case of the forged material, this structure may evolve through heat treatment with TiC network going into solution and coarsening of the metallurgical grains. After the (HT1) heat treatment, one obtains a fully-recrystallized structure with an ASTM 4 grain.

Mechanical properties

The mechanical properties have been evaluated by tensile and stress-rupture tests, and the main results are grouped in Table III and Figure 7. It can be seen that, in respect of the forged material, ductility is sensibly enhanced, and that the good high-temperature creep properties are preserved.

HIGH-TEMPERATURE PROPERTIES ENHANCEMENT

With the purpose of enhancing the high-temperature properties of CLET material, two ways have been explored :

- Improvement of the heat-treatment on the base alloy.
- Modification of the base alloy composition.

Heat-treatment improvement

The tests have been carried out on a pebble HIP and forged from CLET powder in the same conditions as previously. With respect to (HT1) heat-treatment, we have sought after a more complete titanium carbide network solution and a better coarsening of the metallurgical grain.

It has been pointed out previously that heat treatments higher than 1200°C, directly applied to as-forged samples, involve the burning of the alloy. One has then proceeded to a previous heat treatment of 32 hours at 1200°C in order to raise the burning temperature. The heat treatments tested have been the following :

- 1200°C (32 h) A.C. : ASTM grain = 3
- 1200°C (32 h) A.C. + 1240°C (4 h) A.C. : ASTM grain = 1 to 3
- 1200°C (32 h) A.C. + 1225°C (24 h) A.C. : ASTM grain = 2 to 3
- 1200°C (32 h) A.C. + 1240°C (24 h) A.C. : ASTM grain = 1 to 3

Figure 5 illustrates the effect of two of these heat-treatments. After ageing at 1200°C, the carbide network has almost entirely disappeared, and only some isolated precipitates, which are impossible to put in solution, remain. One ascertains that the grain size essentially depends on annealing temperature and does not evolve beyond a 4 hours temperature maintenance.

Creep testings have been performed at 980°C on samples having undergone the following heat treatment cycle :

(HT2) 1200°C (32 h) A.C. + 1240°C (4 h) A.C. + 1080°C (4 h) A.C.
+ 850°C (24 h) A.C. + 760°C (16 h) A.C.

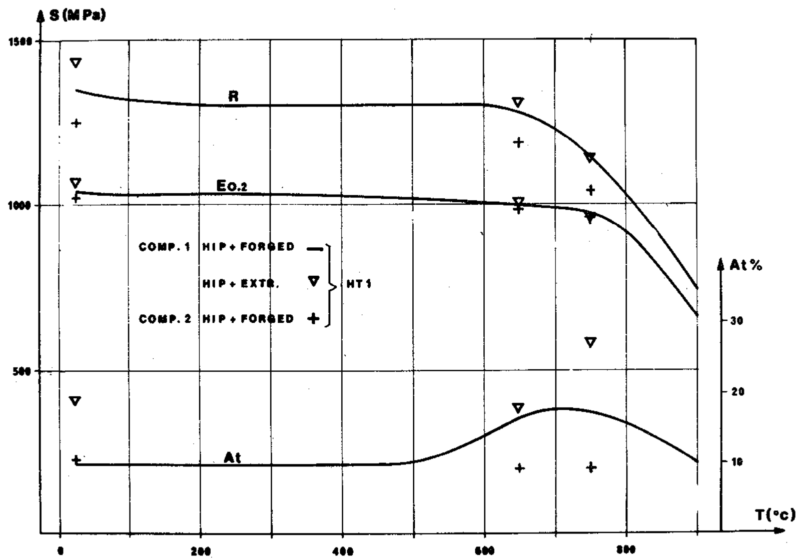


FIGURE 7 - HIP + Wrought materials - Tensile characteristics

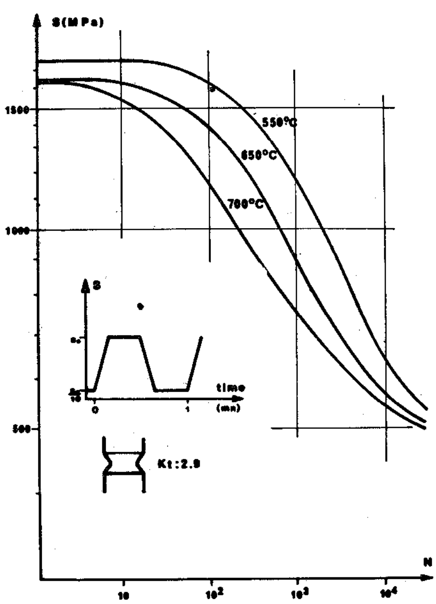


FIGURE 8 - HIP + Forged Comp. 1
Low-cycle fatigue curves

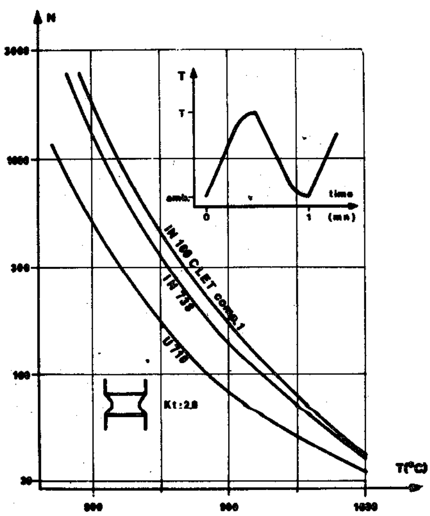


FIGURE 9 - HIP + Forged Comp. 1
Thermal shock curves

It can be seen on Figure 10 that the heat-treatment effect is more sensitive on creep strain results than on rupture life.

Modification of alloy composition balance

For the purpose of increasing the hardening phase percentage, a modified balance (composition 2) has been defined, the main modification applying to an increase of aluminium and a decrease of cobalt and chromium percentages (Cf. Table I). The material has been manufactured from HIP CLET powder and forged in the same manner as previously.

Microstructure, revealed by an optical micrographical study, is near to that obtained on alloy composition 1, with an increase of 10 to 30°C of the characteristic temperatures (solvus and incipient-melting). It is possible, with an heat treatment, to obtain a resolutioning of the titanium carbide network and a growth of the metallurgical grain.

Following forging, the material was heat-treated as follows:

(HT3) 1200°C (32 h) A.C. + 1240°C (4 h) A.C. + 1130°C (4 h) A.C. + 900°C (24 h) A.C. + 760°C (16 h) A.C.

The 980°C stress-rupture and creep tests results (Figure 10) show new property ameliorations, especially for creep test.

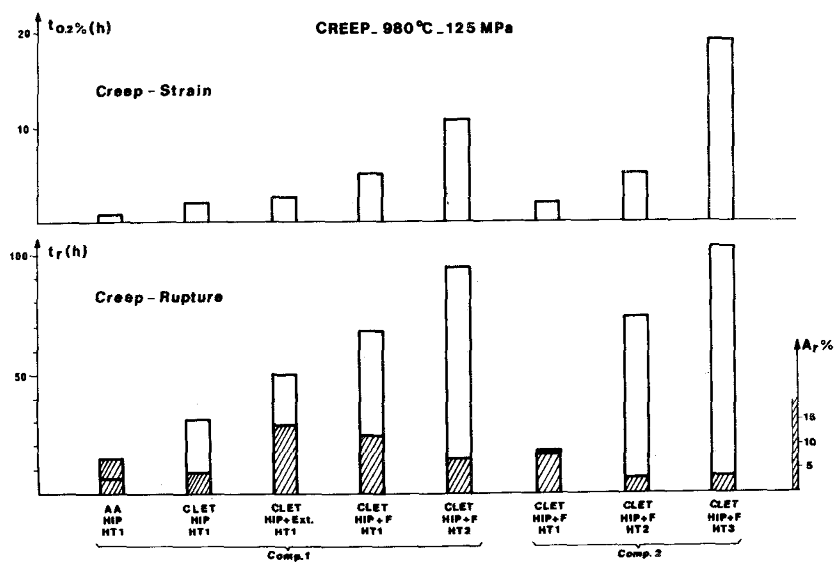


FIGURE 10 - Creep and Stress-Rupture of wrought material

CONCLUSIONS

It has been shown that the use of relatively coarse prealloyed powder allows to develop, in HIP + forged or HIP + extruded IN 100 PM materials, hot mechanical properties at least as good as that of the best conventional cast and wrought materials.

The CLET process, which consists of a centrifugal pulverization of rods, the diameter of which can exceed 250 mm, allows the production of the desired powder at competitive prices. It is possible to use electrodes which are cast molded in a large capacity vacuum induction furnace, with a fairly good yield. The slight composition heterogeneities that segregation pre-existent in the electrode may involve between powder grains are completly removed by heat-treatments.

The HIP compaction of CLET powder is a little slower than that of finer powders and this fact is the main reason for the brittleness in the as-compacted material. The good hot properties of the HIP and wrought materials are related to the powder structure and size. These properties are determined by the possibility to solutionize an important part of the primary precipitates and to coarsen the metallurgical grain.

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