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Necklace structure obtained by forging Astroloy supersolidus-sintered preforms

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A new and original densification process of nickel-based superalloys consists in forging supersolidus-sintered preforms. From the coarse-grained structure of the material sintered in the presence of a liquid phase, forging at a high deformation ratio can lead to a duplex structure (usually called "necklace" structure) as can be obtained by conventional HIP + forge or cast/wrought routes. Optimal forging conditions to form a "necklace" structure have been determined for Astroloy powders. The restoration and recrystallization mechanisms are described in relation to deformation conditions and heat-treatments. Structures and related mechanical behaviour have been characterized. The "necklace" material compares well with conventional direct "HIPed" or forged materials. Good creep–rupture properties are due to large warm-worked grains, and a high resistance to crack initiation and propagation results from recrystallized grains of the duplex structure.

1. Introduction

In the field of superalloys, the feasibility of densification processes based on liquid-phase sintering which can compete with current consolidation methods has already been shown [1, 2]. One such method consists in forging or "HIPing" (HIP: hot isostatic pressing) supersolidus-sintered preforms with closed porosity. The main advantage (economic) is that no can is used for densification; another one (metallurgical) is that a large variety of structures can easily be obtained, depending on the choice of densification conditions. The so-called "necklace" structure is among these structures. This duplex structure is generally achieved by HIP + forge or cast/wrought routes and corresponds to a very interesting compromise of mechanical properties [3–5], good creep properties because of large warm-worked grains and good resistance to low-cycle fatigue by virtue of the surrounding fine recrystallized grains.

The present work is based on:

(1) a study of the forging conditions (temperature and reduction ratio) leading to a duplex structure in order to show the method feasibility;

(2) a structural characterization of the various forged materials, with emphasis on the "necklace" microstructure;

(3) a mechanical characterization using tensile, creep–rupture, cyclic crack propagation, low-cycle fatigue and fatigue crack-growth rate tests.

This study has confirmed that the "necklace" structure is superior to the conventional structure especially in dynamic behaviour.

2. Densification process

Astroloy is an advanced nickel-base P/M superalloy primarily used for turbine discs, because of its high strength (> 850 MPa) at disc-working temperature (~ 650° C). Its solidification interval is rather wide (> 100° C), which is particularly suitable for liquid-phase sintering.

A single batch of 60 mesh argon-atomized powder was used (chemical composition is given in Table I). The powder is poured into a ceramic mould and then heated to sintering temperature in a high-vacuum furnace. The optimal liquid-phase sintering conditions are as follows: vacuum 10^{-5}–10^{-4} Hg mm; sintering time, 1 h; sintering temperature, 1290° C ± 10° C which corresponds to a volume liquid fraction of about 0.35. The sintered

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TABLE I Chemical composition of argon-atomized Astroloy

C	Mn	Si	S	Cr	Mo	Co	Ti	Al	O_2	N_2	Fe	B	Ni
0.026	0.01	0.01	0.002	14.80	5.0	16.99	3.60	4.09	0.008	0.002	0.21	0.023	Bal.

preform is subsequently forged. It is well known that the range of forging conditions leading to a "necklace" structure, is rather narrow and depends on the alloy type and initial microstructure. To define this range, various forging conditions were tried: 23%, 40% and 50% deformation ratios, respectively, associated with forging temperatures of 1080, 1080 and 1120° C.

3. Microstructure of the sintered plus forged materials

Optical and electron microscopy (transmission electron microscopy, TEM, and scanning electron microscopy, SEM), a microprobe and a scanning electron microscope equipped with an energy dispersive X-ray spectrometer device (EDS) were used for phase identification.

Two heat-treatments were chosen: HT1 and HT2 which are conventional for this type of nickel-base superalloy [6, 7]:

HT1: Partial solutioning of γ' 4 h/1080° C OQ; double stabilization stage 8 h/870° C AC + 4 h/980° C AC; double precipitation stage 24 h/650° C AC + 8 h/760° C AC.

HT2: 4 h/1100° C OQ + 24 h/650° C AC + 8 h/760° C AC.

3.1. Microstructural aspects of the non-heat-treated materials

The 50%–1120° C forged material shows a completely recrystallized structure. A fine-grained substructure can be seen in the core of the main grains that correspond to the prior powder particle size. The 23%–1080° C forged material is not recrystallized at all whereas the 40%–1080° C material shows the start of the recrystallization process which can only be seen by transmission electron microscopy. In the latter case, the prior grain boundaries are decorated with small recrystallized grains (1–2 μm diameter). The coarsening of these grains by the appropriate heat-treatment leads to the so-called "necklace" structure although the "necklace" was already initiated before heat-treatment. This non-heat-treated structure is characterized by a cellular structure in the core of the main grain, resulting from the dynamic restoration process. The dynamic restoration process can occur if the deformation temperature is above one-third of the liquidus temperature, as is the case here. During this thermally activated process, a rearrangement of the dislocation groups leads to a cellular sub-structure (Fig. 1). The γ' precipitates and the γ/γ' interfaces are frequently associated with dislocation pile ups.

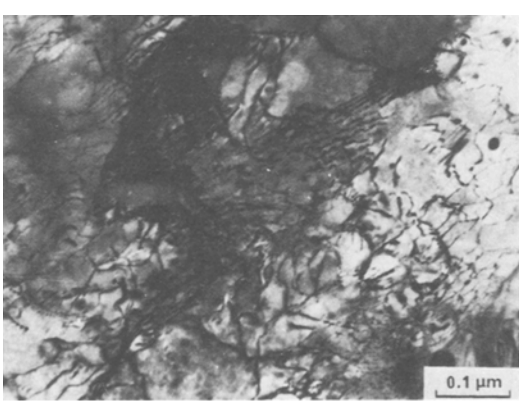


Figure 1 Dislocation cells obtained by dynamic restoration. Transmission electron micrograph (thin foil) on the non-heat-treated 1080° C–40% forged material.

This kind of structure has already been observed in other nickel-base superalloys (wrought René 95 [8] and Udimet 700 [9]). Deformation (twinning) is mostly located near the prior particle boundaries; the twins are very thin (about 0.2 μm in width) and cross the sub-structure. As a result of the deformation, recrystallized grains could nucleate and coarsen up to about 1 μm in diameter (Fig. 2): they constitute the necklace "initiation". The carbides are mainly MC (TiC) carbides that are located along the prior particle boundaries, or scattered throughout the matrix.

3.2. Microstructural study of the heat-treated 40%–1080° C forged material

Post-HT1 and post-HT2 structures are both of the "necklace" type. They are characterized by a "necklace" of recrystallized grains which outlines the prior particle boundary (Fig. 3). Such a structure cannot be obtained by direct hot isostatic pressing (HIP) of superalloy powders, since the high deformation of a coarse grain preform is

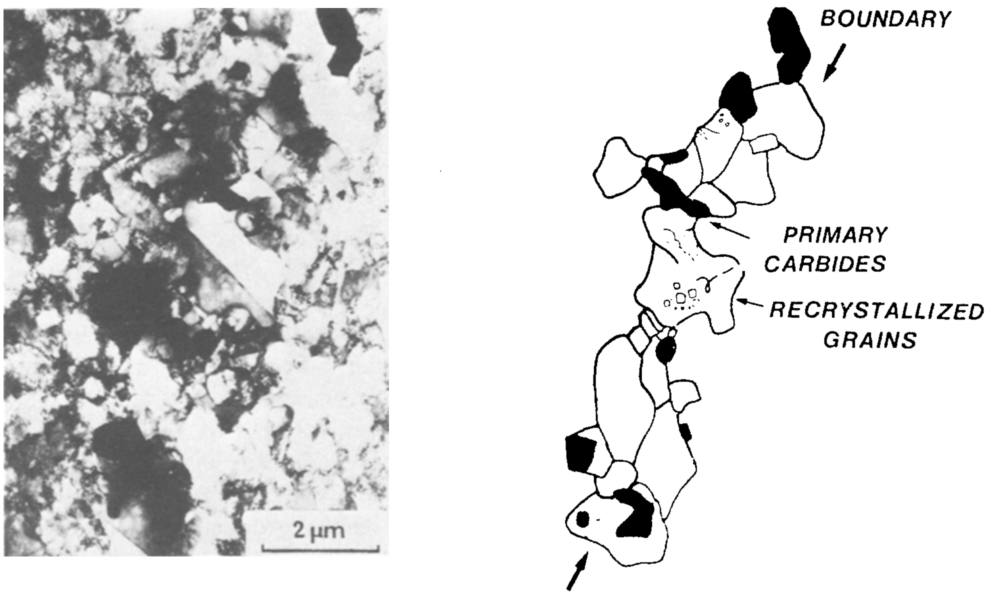


Figure 2 Starting recrystallization in the 1080° C–40% forged material. Transmission electron micrograph (thin foil).

necessary. The nucleation mechanism is shown by microscopical examinations. Recrystallization is usually linked with twinning, but here it may rather be linked to the presence of "hard" nucleating particles. For Astroloy, these "hard" particles can be carbides (TiC carbides [10]) or coarse γ' at the prior particle boundaries [11]. For the liquid-phase sintered and forged material, all the recrystallized grains are nucleated from coarse γ' (1–3 μm), as can be seen in Fig. 4.

Sometimes, the recrystallization front passes round a γ' precipitate which appears in the core of the final recrystallized grain (Fig. 5).

The structural morphology depends on the direction of motion of the recrystallization front. If the front follows the grain boundary, a typical "necklace" structure is achieved, outlining the prior powder particle as mentioned previously. On the other hand, if the front moves towards the core of the main grain, a cellular precipitation can occur. This phenomenon consists of γ' precipitates going into solution as the recrystallization front moves forward with directed reprecipitation behind it. This kind of structure can be deleterious to the mechanical properties if it extends too far into the grain as it is associated with a large-scale heterogeneity.

The "necklace" aspect depends on the heat-treatment (HT1 and HT2).

Recrystallization is a thermally activated phenomenon; since γ' partial solutioning temperature differs by 20° C (1100° C for the second heat-treatment as against 1080° C for the first one) the mean size of the post-HT2 recrystallized grains is, therefore, coarser than the post HT1.

A mean size γ' precipitation (0.25 μm) can be held in the post HT1 "necklace" grains only. It is linked with holds at intermediate temperatures (870° C and 980° C) during the double stage which only exists in HT1.

The post-HT1 recrystallized grains are outlined by molybdenum- and chromium-rich carbides which have precipitated at 870 and 980° C whereas the post HT2 ones have "clean" boundaries because there is no stage at intermediate temperature (between 800 and 1000° C). These carbides have also precipitated along incoherent twin boundaries.

In the core of the main grains (corresponding to the prior powder particles), a very fine-grained structure ($\leqslant 1\,\mu$m diameter) can be seen by transmission electron microscopy (Fig. 6). This substructure was obtained by a static restoration process from the dislocation cells of the non-heat-treated material, this structure resulting from dynamic restoration (as previously described) during forging.

The sub-grains are due to dislocation rearrangements during the first step of heat-treatment; they are free from dislocations but can contain mean size γ' (0.3 μm) which stabilize the structure.

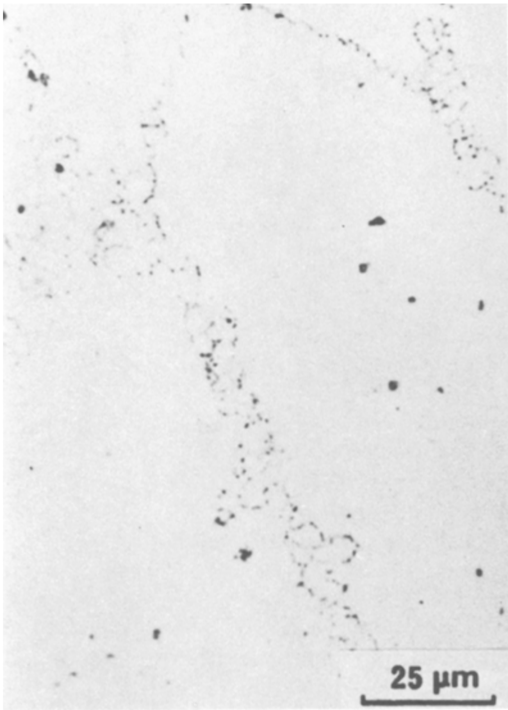


Figure 3 Optical micrograph of the HT1 1080° C–40% forged material, Murakami's reagent.

4. Mechanical behaviour of the sintered plus forged materials

Mechanical properties of the forged materials were determined using tensile, creep–rupture, low-cycle fatigue, cyclic crack propagation and fatigue crack-growth rate tests. HT1 and HT2 heat-treatments were compared.

4.1. Static tests

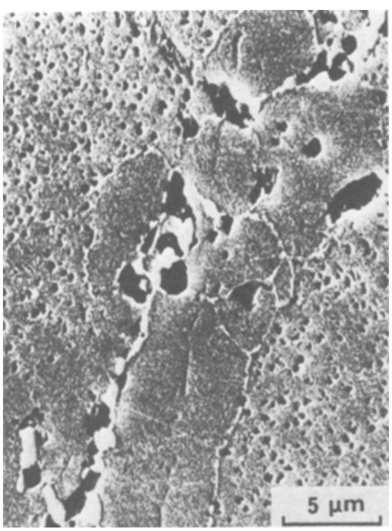
Among the three available forged materials, the 1080° C–40% and 1120° C–50% ones were chosen because the forging reductions are close; the macroscopic effect of the deformation grain size, therefore, is about the same and the microstructure influence can be isolated.

The results are given in Fig. 7a and b and Fig. 8 and compared with those of the direct HIPed Nimonic AP1* [7, 12] which is considered as one of the best high-performance materials. In the case of the creep–rupture results, a conventional HT1 HIPed (CHIP) Astroloy [13] was taken as a comparison basis.

The main properties are quite adequate although the HT1 materials exhibit the harmful effect of grain-boundary carbides.

For a given heat-treatment, the tensile properties of the 40% and the 50% forged materials are fairly close, except for the yield strength which is the highest in the case of the 40% deformed material while its elongation is the lowest.

Considering the creep–rupture (on smooth specimens) results, increasing the reduction ratio improves the elongation but reduces the rupture life as seen in Fig. 8 for HT2 materials. According



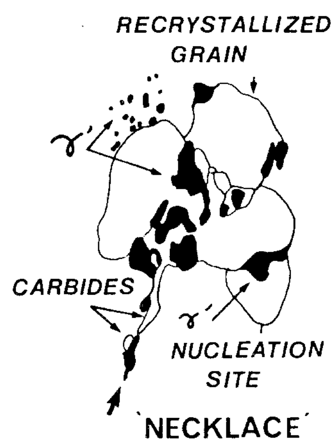


Figure 4 Recrystallized grains nucleated from coarse γ'. Scanning electron micrograph of the HT2 1080° C–40% forged material.

*Trademark of Henry Wiggin and Company Limited, similar composition to Astroloy.

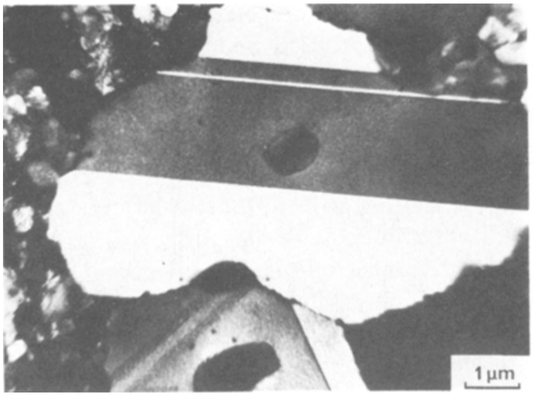


Figure 5 Coarse γ' in the core of a recrystallized grain. Transmission electron micrograph (thin foil) of the HT2 1080° C–40% forged material.

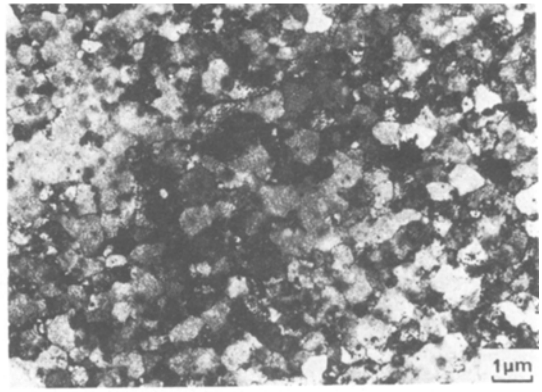


Figure 6 Fine-grained sub-structure of the HT2 1080° C–40% forged material. Transmission electron micrograph (thin foil) of the main grain core.

to the tensile results, HT2 should improve these properties further. In the case of the HT2 1080° C–40% material, it was necessary to increase the creep-stress (from 870 MPa to 1000 MPa) to accelerate the creep phenomenon.

Fractographic investigation showed that the rupture is mainly intergranular (interparticular) in the 50% forged material whereas the 40% is mixed transgranular/intergranular. The "necklace" seems to strengthen the grain boundary, provided that the grain boundary contains relatively few hard particles such as γ' or carbides (which is the case after HT2) which could initiate cracks or speed up their propagation.

4.2. Dynamic tests

The "necklace" 1080° C–40% forged material which exhibits the best static property conditions, was characterized in dynamic tests. Supersolidus-sintered plus HIP (at 1130° C and 1150° C) Astroloy [1] was taken as a reference for some of these mechanical properties.

4.2.1. Cyclic crack propagation and low-crack fatigue tests

Cyclic crack propagation (CCP) tests on notched KT 2.5 ASTM specimens, and low-cycle fatigue (LCF) tests on notched specimens (shown in Fig. 9) were performed at 550° C. In both cases, the load cycle is a trapezoidal-one-minute (symmetrical) cycle with hold periods (at σ_{max} and $\sigma_{max}/10$) of 20 sec.

Both CCP and LCF behaviours are good for the "necklace" material.

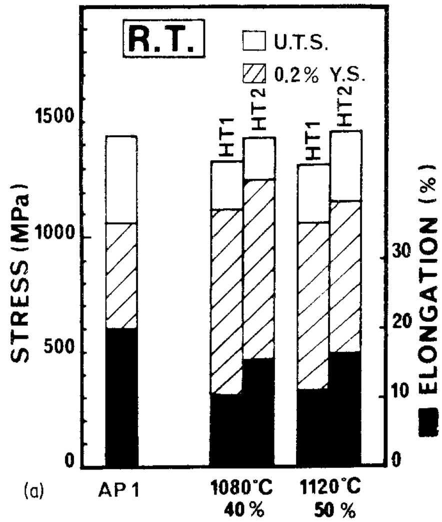
4.2.1.1. Cyclic crack propagation tests. The crack length to failure is fairly close to that of the HIPed Astroloy and the rupture life is quite adequate.

The crack path is mixed intergranular/transgranular. The intergranular crack propagation follows the "necklace" along the outline of the recrystallized grains, as along wavy grain boundaries. But if recrystallized zones spread out over a large area (more than 20 μm in extent), as in the case of cellular precipitation, the crack goes through them, and the propagation speeds up. So the "necklace" recrystallization tends to improve life when it is not too extensive.

4.2.1.2. Low-cycle fatigue tests. The rupture life of the HT2 forged materials is about the average of the values of the two HT2 HIPed materials and meets the standard for the alloy (Fig. 10).

Fractographic investigation confirmed the results of the study on tensile fractured specimens; the "necklace" recrystallized grains do not constitute weak regions susceptible to crack initiation. Several types of initiation can be noted – carbide clusters at triple points of the grain, surface defect, cleavage – but no intrinsic crack initiation on "necklace" zones has been observed.

The influence of the initial stress was examined (Fig. 11). Low-cycle fatigue tests were performed under stresses of 650, 700 and 750 MPa. They show that the rupture life varies linearly with stress around 700 MPa: but the stress effect is not well marked in this particular range. Fractographic



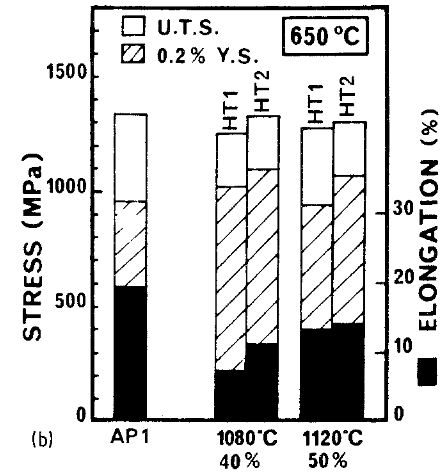


Figure 7 (a) Tensile properties (RT) of sintered plus forged materials, in comparison with HIPed Nimonic AP1 alloy [7]. (b) Tensile properties (650° C) of sintered plus forged materials in comparison with HIPed Nimonic AP1 alloy [12].

observations showed that the higher the nominal stress is and the rougher the relief of crack initiation sites.

4.2.2. Fatigue crack-growth (FCG) rate tests

10 mm thick and 40 mm wide CT type specimens were used. The R ratio ($R = K^{\min}/K^{\max}$) was kept equal to 0.02. The tests were performed using a sinusoidal wave-form signal (frequency: 40 Hz). The results (Fig. 12) are given in comparison with P/M Astroloy [14] Iconel 718 [15] Waspaloy [15] which is a high-performance superalloy as to CG behaviour; they show that the FCG properties of the "necklace" forged material are quite satisfactory. The reasons for the FCG behaviour, due to the duplex structure, are the same as those previously described (see Section 4.2.1.) for the crack-propagation tests.

5. Conclusions

This study demonstrated that "necklace" structures could be obtained with Astroloy by forging supersolidus sintered preforms. The optimal forging conditions are 1080° C and a reduction rate of 40%.

This "necklace" Astroloy is one of the best high-performance nickel-base superalloys and compares well with cast/wrought, HIPed and forged, and direct HIPed products. Its mechanical properties are always adequate due to the fact they are controlled by the fine grain or the coarse grain of the duplex structure. The microstructural aspects (grain size, carbide and γ' precipitations) can be easily modified by heat-treatment. In the present study, for an argon-atomized powder of Astroloy, a γ' solutioning at 1080° C (4 h/OQ) followed by a double ageing (650° C/24 h/AC + 760° C/8 h/AC) constitutes a very well-adapted

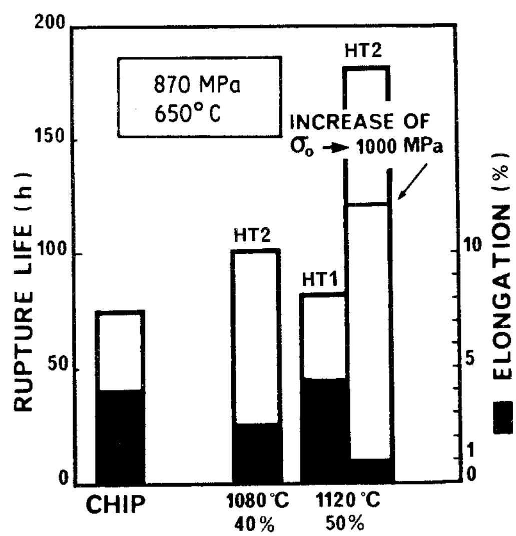


Figure 8 Creep–rupture properties of sintered plus forged materials in comparison with a conventionally HIPed (CHIP) Astroloy [13].

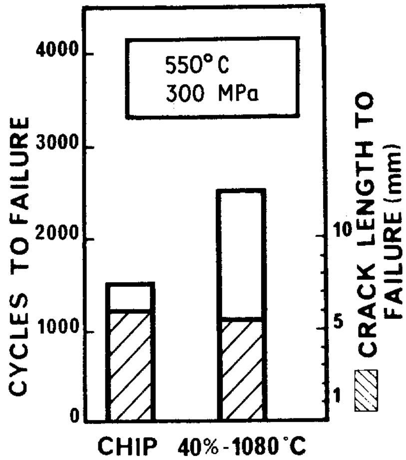


Figure 9 Cyclic crack-propagation properties of sintered plus forged material in comparison with a conventionally HIPed (CHIP) Astroloy.

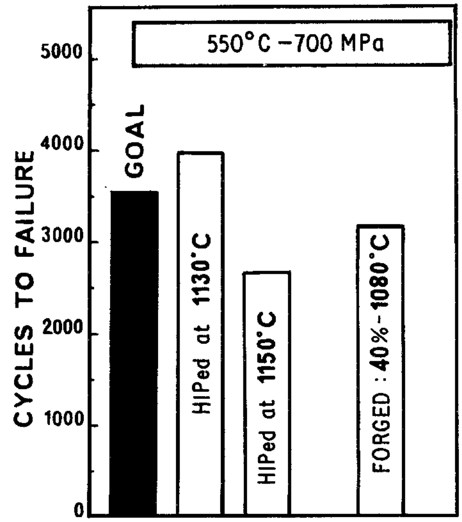


Figure 10 Low-cycle fatigue properties of HT2 sintered plus forged and sintered plus HIP materials.

heat-treatment. However, the heat-treatment can be easily modified according to the application of the alloy. For example, HT2 seems to improve the crack-propagation behaviour but lowers the crack-initiation resistance, because the recrystallized grains are too large: but, lowering a little the γ' solutioning temperature (by 20° C) rebalances these two properties.

The processes that have been studied for

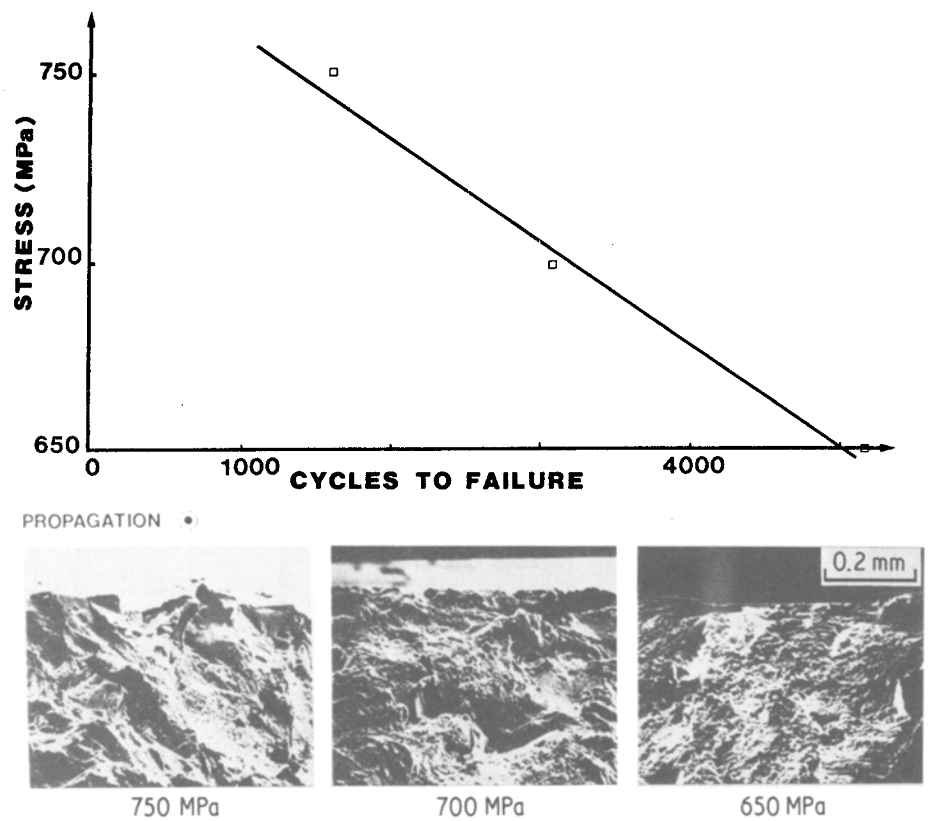


Figure 11 Low-cycle fatigue tests: effect of nominal stress on the rupture life of the HT2 1080° C–40% forged material. SEM images of the crack initiation site for nominal stresses of 750, 70p0 and 650 MPa.

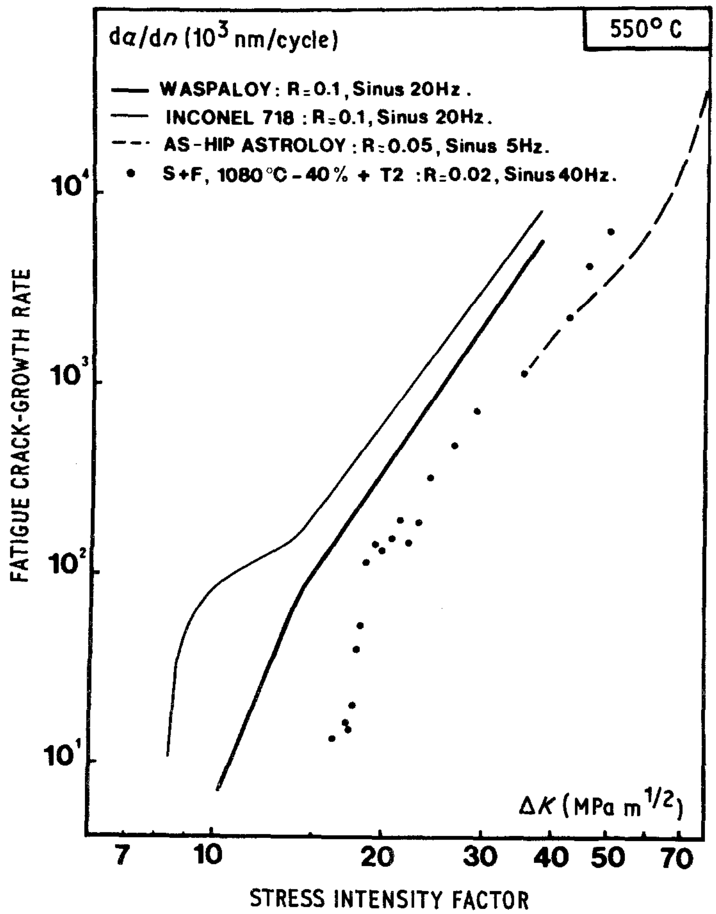


Figure 12 Variation of FCG rate (da/dN) with stress intensity factor (ΔK) at 550° C, for the HT2 1080° C–40% sintered plus forged (S + F) material in comparison with HIPed Astroloy [14], Waspaloy [15] and Inconel 718 [15] alloys.

Astroloy can be applied to other superalloys (e.g., René 95) in so far as they can be sintered in the presence of a liquid phase. Forging appears to be the most appropriate step for final densification of supersolidus-sintered preforms as large grain sizes commonly encountered in this sintering process are accommodated by the necklace structure of the forged + heat-treated material.

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