THE HOT WORKING BEHAVIOUR OF MAR M200 SUPERALLOY COMPACTS

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ABSTRACT

The hot working behaviour of some hot isostatically pressed low carbon Mar M200 superalloy compacts has been examined by means of axisymmetric compression testing in the range 1050°C to 1200°C at constant true strain rates between $10^{-4}~\rm s^{-1}$ and 1 $\rm s^{-1}$. The compacts were pressed either below or above the γ^{t} solvus to obtain grain sizes in the range 2-8 µm or 20-200 µm respectively. The fine grained compacts deformed superplastically at all temperatures and at strain rates in the range from 10^{-4} s⁻¹ to 10^{-2} s⁻¹. Under these conditions the strain rate sensitivity exponent m was about 0.6. The coarse grained compacts produced peak flow stresses 3 to 6 times larger than the fine grained material and showed a strain rate sensitivity exponent of about 0.12 under the same working conditions. Both the fine and coarse grained materials recrystallized dynamically during deformation leading to flow softening. Uniform recrystallization and deformation occurred in the initially fine grained compacts resulting in high ductility. Localized recrystallization occurred along prior grain boundaries in the initially coarse grained material resulting in localized plastic flow and severe internal cracking at high strains. The manufacturing of powder metallurgy superalloy parts is discussed in the light of these results.

INTRODUCTION

It is widely recognized that powder fabricated superalloy billets have much superior workability than their ingot-produced, as-cast counterparts (1). The benefits derive from a reduction of dendritic segregation, with the result that wrought products can be produced from hitherto difficult to work casting-type alloys. Moreover, it has been demonstrated that stable ultra-fine grain sizes can be developed, either in cast and wrought or powder fabricated and wrought superalloys, allowing these materials to deform superplastically (2,3). For example, in the Gatorizing Process (4), the superalloy is extruded at a temperature just below the γ' solvus so that adiabatic heating causes momentary solutioning of γ ', recrystallization, and rapid re-precipitation of γ ' to stabilize the fine, recrystallized and superplastic grain structure. In the superplastic condition, such materials can be forged at low pressures into complex, close tolerance shapes, provided that the working conditions of temperature and strain rate are controlled. The purpose of this paper is to show that the necessary fine grain size for superplasticity can be retained in as-pressed HIP compacts by appropriate control of powder type and pressing conditions.

MATERIALS

The alloy examined was Mar M200, a high strength casting-type nickel-base superalloy which relies on γ' (Ni₃Al) precipitation hardening and solution hardening with tungsten. The master alloy was argon atomized and the resultant powder consolidated by hot isostatic pressing (HIP) in evacuated stainless steel tubes. The powder chemistry and mesh size distribution are given in Table 1.

Four sets of HIP pressing conditions were used as given in Table 2. Since the γ' solvus is 1200°C ± 15°C , HIP 1 represents a two phase $\gamma+\gamma'$ condition, HIP 2 a partial γ' solution condition, and HIP 3 and HIP 4 full solution conditions. The grain sizes and gamma-prime particle sizes present after pressing are given in Table 2, and selected microstructures shown in Fig. 1. The presence of coarse γ' on grain boundaries during HIP treatments 1 and 2 stabilizes the fine as-atomized grain size of the powders (Fig. 1a), while recrystallization to a coarse grain size occurs during the full solution treatment pressings of HIP 3 and 4, Figs. 1c and 1d. The compacts can therefore be classified either as fine grained (HIP 1 & 2) or coarse grained (HIP 3 & 4) materials, and their flow and fracture behaviour during hot working are examined accordingly.

TABLE 1: Chemistry in Weight % and Mesh Size of the Mar M200 Powder

Element %			W 12.82				
Mesh %			 0 +23 3 15.		~ - /	 	

TABLE 2: HIP Pressing Conditions and Microstructural Characteristics of the Compacts at Room Temperature After Pressing.

HIP Sample	Pressing Conditions*	Grain Size µm		ontent cle si F	and ze µm. B	
1 2 3 4	1050°C/2 hrs/69 MN/m ² 1150°C/2 hrs/69 MN/m ² 1250°C/2 hrs/103 MN/m ² 1100°C+1230°C/2 hrs/69 MN/m ²	2 - 8 2 - 8 20 - 200 20 - 200	.1 .05	.4 .2 .2	1.5	

HF = Hyperfine (spherical), F = Fine (cuboidal) and B = Blocky (irregular). *All samples air cooled from pressing temperatures.

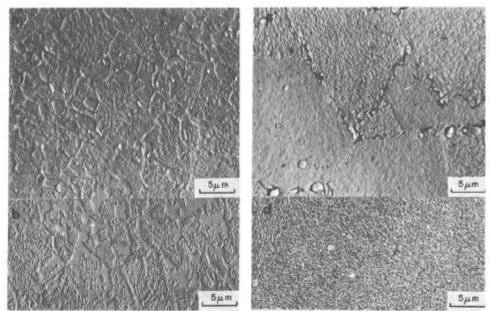


Figure 1: Microstructures in Mar M200 compacts. Pressing below the γ' solvus (HIP 1) results in a) fine grains and b) large blocky γ'; pressing above the γ' solvus (HIP 3) results in c) coarse grains and d) fine cooling γ'. (Marble's etch a) and c), Inco etch b) and d).

EXPERIMENTAL PROCEDURE

A high temperature compression testing apparatus was used to simulate hot working conditions. The isothermal axisymetric tests were carried out between silicon nitride platens in a 10,000 Kg MTS hydraulic testing machine modified for constant true strain rate deformation. The constant true strain rates were obtained by means of an analog function generator designed to operate in conjunction with the MTS controller. This device causes the compression ram velocity to remain directly proportional to the specimen height during straining. Details of the compression train and analog function generator are given elsewhere (5,6). The compression specimens were right cylinders, 9.65 mm long and 6.35 mm in diameter, machined from the HIP bars. The end faces of the cylinders were grooved in order to retain the molten glass lubricants* used to prevent barrelling of the test pieces.

The tests were carried out under flowing argon and at temperature intervals of 50°C in the range from 1050°C to 1200°C . At each temperature, 5 strain rates were used between $3.0 \times 10^{-4}~\text{s}^{-1}$ and $1\,\text{s}^{-1}$. In these tests, the specimen height was measured by means of a high temperature displacement transducer which monitored the relative displacement of the compression platens. Developed load and specimen height were continuously recorded on a x - y recorder, or at high strain rates on a high speed 2 channel galvanometric recorder.† The true stress—true strain curve for a given specimen was calculated from the load—displacement data, assuming that no volume change takes place during deformation. The compressed specimens could be quenched within 1 to 2 seconds of the end of deformation in order to retain the hot worked structures for metallographic examinations.

The deformed and quenched specimens were sectioned longitudinally, mechanically polished and chemically etched. Two etching solutions were used, one to

^{*-200} mesh sodium/calcium boro silicates with varying amounts of magnesis and alumina from Ferro-Industrial Products, Oakville, Ontario, Canada.

†Brush recorder, model 280.

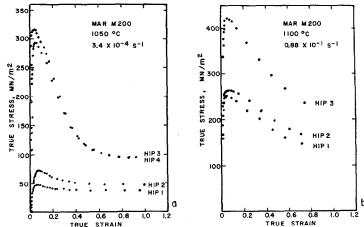


Figure 2: True stress-true strain compression curves for Mar M200 compacts, illustrating the effects of HIP processing conditions on high temperature flow. a) At 1050°C and 3.0 \times 10⁻⁴ s⁻¹, the compacts pressed above the γ' solvus (HIP 3, HIP 4) show a four to six fold increase in peak flow stress over the compacts pressed below the γ' solvus (HIP 1, HIP 2). b) At higher strain rates and higher temperatures, the differences in flow behaviour are less pronounced.

reveal the γ' morphology and distribution (Inco etch*) and the other to reveal grain boundaries (Marble's reagent†). Examinations were carried out by means of optical and electron microscopy.

RESULTS

The effects of prior thermomechanical history and testing conditions of temperature and strain rate are examined below.

1. Flow Curves

The stress-strain curves of the four compacts were qualitatively similar as shown in Fig. 2. Each curve depicts a microstrain region and an apparent macroyield followed by rapid work hardening to a peak flow stress. The peak flow stress is followed by softening where the flow stress falls asymptotically towards a steady state value at high strains. The peak stress, the strain at peak stress and the degree and rate of work hardening and flow softening are dependent on the prior thermal history and microstructure of the compact. The finer the initial grain size, the lower the peak flow stress and the higher the strain at peak stress. Furthermore, the differences in flow behaviour are more pronounced at low strain rates and low temperatures over the ranges investigated.

Under identical testing conditions, the coarse grained compacts undergo considerably more flow softening but take longer to reach steady state as shown in Fig. 3. Figure 3b shows that at $\dot{\epsilon}=1.4\times10^{-3}~\mathrm{s^{-1}}$, the coarse grained compacts have not reached steady state flow at strains of 0.8. In the fine grained compacts under the same conditions, steady state is reached much earlier and particularly so at high temperatures. Extrapolating the flow curves of Fig. 3b to high strains indicates that the steady state stresses would be similar for fine and coarse grained materials, under similar test conditions, thus demonstrating that the effects of prior thermomechanical history are gradually eliminated during working.

The effects of working temperature are shown in Fig. 3. Flow strength diminishes rapidly with temperature in both fine and coarse grained materials,

^{*}Inco etch: 50 ml HCl, 25 ml HNO₂, 2 gm CuCl₂, 200 ml H₂O; †Marble's reagent: 10 gm CuSO₄, 50 ml HCl, 50 ml H₂O.

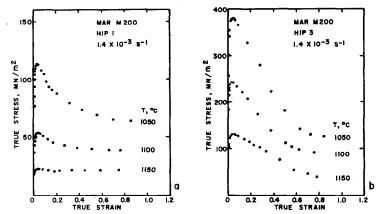


Figure 3: Effects of temperature at constant true strain rate on the compressive flow curves of Mar M200 compacts pressed a) below the γ ' solvus (HIP 1) and b) above the γ ' solvus (HIP 3).

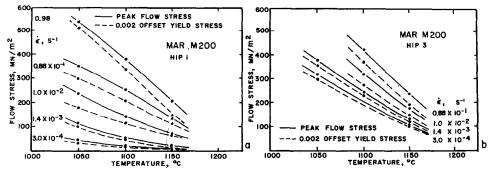


Figure 4: Temperature dependence, at various strain rates, of the high temperature compressive yield strength and peak flow stress of Mar M200 compacts pressed a) below the γ' solvus (HIP 1) and b) above the γ' solvus (HIP 3).

and flow softening is more appreciable at the lower temperatures. The temperature dependencies of the yield* and peak flow stresses in these compacts are shown in Fig. 4 for all strain rates used. The isostrain rate curves show similar trends in both materials particularly at low strain rates. For the fine grained compact (HIP 1), the reversal in curvature observed at high strain rates and low temperatures suggests that the deformation processes may be different to those occurring at low strain rates.

The effect of strain rate is shown in Fig. 5 which indicates that all test materials are strain rate sensitive. These and earlier data indicate that thermally activated processes control the deformation over the ranges investigated. The strain rate dependencies of the yield and peak flow stresses at all testing temperatures are presented in Fig. 6. The data demonstrate that the fine grained compact is considerably more strain rate sensitive, particularly at the lower strain rates. In this material (HIP 1) the strain rate sensitivity index

$$m = \left(\frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}}\right)_{T, \text{ structure}}$$

approximates 0.6 at low strain rates and 0.2 at high strain rates. The gradual transition between the two regions is shifted to higher strain rates with an increase in temperature. In contrast, the strain rate sensitivity index for the coarse grained material (HIP 3) is 0.12 over the entire range. Similar values are obtained using either yield or peak flow stress data. Superplasticity is usually associated with strain rate sensitivity indexes higher than 0.5 (7).

^{*}Defined as the flow stress at a 0.002 strain offset.

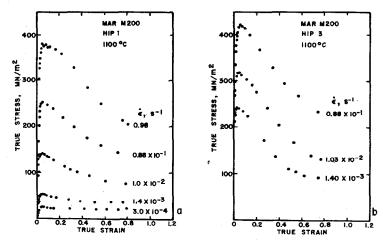


Figure 5: Effects of strain rate at constant temperature on the hot compression flow curves of Mar M200 compacts pressed a) below the γ' solvus (HIP 1) and b) above the γ' solvus (HIP 3).

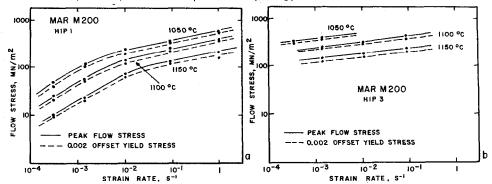


Figure 6: Strain rate dependence, at three temperatures, of the high temperature compressive yield strength and peak flow stress of Mar M200 compacts pressed a) below the γ' solvus (HIP 1) and b) above the γ' solvus (HIP 3). The strain rate sensitivity of the fine grained material (HIP 1) is higher than that of the coarse grained compact and particularly at the lower strain rates.

The important observation, therefore, is that the fine grained compacts deform superplastically under appropriate forging conditions.

2. Processing and hot worked structures

The microstructures depicted in Fig. 7 are representative of the structures developed during hot working of the powder compacts. In the fine grained material, for which the testing conditions belong to the high strain rate sensitivity region, the initial grains have been almost entirely replaced by new grains of an even finer size. Under these same conditions, the coarse grained compact shows partial recrystallization localized mainly along prior grain boundaries. The shadowed carbon replicas of Fig. 8 indicate that the recrystallized grain size is the same order of magnitude in the two compacts and therefore depends only on the deformation conditions. It increases with temperature and decreases with strain rate over the ranges investigated. Furthermore, it is apparent from these photographs that the recrystallized regions are of a microduplex nature with equiaxed γ and γ' grains uniformly distributed throughout. In view of the short quenching times involved in these tests (<2 s) and of the stability of the hot worked structures when quenching is delayed, the new grains are believed to have formed dynamically

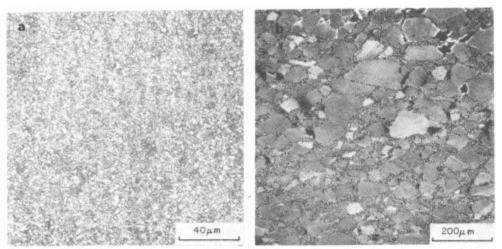


Figure 7: Effects of thermomechanical history on the microstructures developed in Mar M200 compacts during hot working to a strain of 0.6 at 1050°C and 3.0 × 10⁻⁴ s⁻¹. Samples initially HIP pressed a) below the γ' solvus (HIP 1) and b) above the γ' solvus (HIP 3). The fine grained compact (HIP 1) is uniformly recrystallized whereas in the coarse grained material, recrystallization is incomplete and localized along prior grain boundaries or twin boundaries.

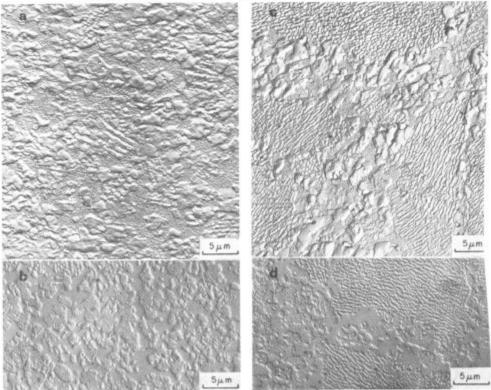


Figure 8: Microduplex nature of dynamically recrystallized areas in Mar M200 compacts: a) initially fine grained compact (HIP 1) etched to show grain boundaries and b) to reveal the Y' morphology; c) initially coarse grained compact (HIP 3) etched to show grain boundaries and d) to reveal the redistribution and coarsening of Y'.

during deformation.

A change in γ' morphology occurs during compression and this is particularly evident in the coarse grained materials. The fine (.1 to .4 μ) cuboidal precipitates coarsen and transform into oriented plates or rods (1 to $3\,\mu$ long, .4 to .6 μ wide) that are seen in the non-recrystallized areas of these compacts, Fig. 8d. The preferred alignment of the precipitates appears to depend on the crystal lattice orientation of the parent matrix and varies from grain to grain in a random fashion. In the fine grained compacts, however, these effects are not so evident since the γ' precipitate prior to compression is already of a coarse size and also these materials recrystallize dynamically to a microduplex structure during compression.

Metallographic examinations indicate that ductility in all compacts increases with temperature and decreases with strain rate. These effects are particularly evident in the fine grained materials in the high strain rate sensitivity region. Figure 7 shows that under identical conditions of strain rate, strain, and temperature, considerably more cracking occurs in the coarse grained than in the fine grained compacts. The important result, therefore, is that hot ductility is greatly improved by the presence of a fine grain size prior to deformation.

DISCUSSION

It has been shown that superplastic flow can be obtained in nickel-base superalloy compacts hot-isostatically pressed below the γ' solvus. Superplasticity is dependent on the fine grain size (7) and on the ability of the Y' particles to impede recrystallization and grain growth (8,9). This allows the inherent fine grain structure of as-atomized particles to be retained during pressing and prevents the growth of recrystallized grains during forging. It appears therefore that the intermediate extrusion process often used to generate the superplastic condition in superalloy compacts (3,4) is not entirely necessary and that superplasticity can be achieved simply by appropriate choice of powder type, mesh size and HIP pressing conditions (10). This is important in practical terms since by HIP pressing to a superplastic condition a costly extrusion process can be eliminated and greater flexibility is allowed in the design of superplastic forging preforms so that forging strains can be minimized. Furthermore, not only are fine grain size and superplasticity important in lowering working pressures, but they also provide for greater ductility during hot working than is obtained with equivalent coarse grained materials (10).

The types of powder, mesh sizes and temperature ranges required to ensure the retention of a superplastic condition during HIP processing have not been investigated. However, it is apparent that any powder having a grain size of less than about 10 µm should be suitable, provided the pressing temperature is kept well below the γ' solvus (viz. >25°C). Examinations in these laboratories (10) have indicated that most types of commercial (-60 mesh) argon atomized powder should satisfy the grain size requirements, whereas similar sized powder from the rotating electrode process appears to have grain sizes an order of magnitude larger. Forging these latter compacts in the temperature range close to, but below the γ' solvus would result in non-uniform recrystallization and non-superplastic flow characteristics as discussed below with respect to the present coarse grained compacts. Pressing above the γ' solvus leads to dynamic recrystallization and, in the absence of powder surface boundary carbide precipitation (10), the formation of a coarse grained deformation resistant microstructure.

The flow curves and microstructural observations indicate that, while differences exist in strain rate sensitivity for the coarse grained and fine grained materials, the processes occurring during deformation are qualitatively similar under similar conditions of strain rate and temperature. The restoration mechanisms of dynamic recrystallization and second phase coarsening are operative in these compacts, as expected in nickel-base superalloys (11,12), and their initiation can be associated with the softening process that follows the peak flow stress, as observed in many other materials (13). The deformation modes are those of grain boundary sliding and shear strain accommodation by

diffusion or plastic flow within the grains (14) or, alternatively, those of intragranular flow by dislocation generation, annihilation and rearrangement (7). What process is dominant depends on the initial structure, the rate of forming, the temperature and the strain (i.e. the structure).

At slow strain rates, all materials tend to deform by grain boundary sliding, with plastic accommodation within the grains. This is the dominant deformation mode in the fine grained materials both before and after the onset of dynamic recrystallization. In contrast, in the coarse grained material, sliding is at first severely restricted by the lack of plasticity and strain accommodation within the grains which is a consequence of the small grain boundary area and an effective γ' strengthening effect. This restriction of sliding is responsible for the high yield strength and high peak flow stress in the coarse grained materials. However, once dynamic recrystallization is initiated along prior grain boundaries the local deformation mode becomes similar to that of the fine grained material.

At higher strain rates, grain boundary sliding cannot easily accommodate the required deformation rates (7,14). Intragranular deformation by dislocation glide and climb becomes the dominant deformation mode and results in the conventional low strain rate sensitivities. The decrease from m=0.6 to m=0.2 in the fine grained compacts is due to this change in deformation mode. Since the volume fraction of fine recrystallized grains increases with strain, the superplastic properties of all forged compacts can be expected to be greatly enhanced by this structure refinement (7) in accord with accepted practices (3,4).

In the fine grained material and at low strain rates recrystallization and deformation occur homogeneously due to the large initial grain boundary area, so that large crack-free strains can be developed under reduced working pressures. The growth of the recrystallized grains is restricted by γ^{\prime} (8,9) and thus a stable fine grain size is established dynamically during working. In the coarse grained material, the initial intragranular deformation leads to grain boundary distortion and local recrystallization along these boundaries. As in the fine grained materials, the recrystallized grains are prevented from growing to any extent so that the equilibrium grain structure developed during deformation consists of a duplex structure of coarse, non-recrystallized grains in a "matrix" of ultra fine recrystallized material. The new grains are of a similar size to those of the fine grained material deformed under similar conditions of strain rate and temperature.

Observations of the γ' particle sizes and distributions before and after deformation indicate that the recrystallization process involves a local resolutioning of γ' ahead of the advancing recrystallized boundaries. Both theoretical considerations (15) and experimental observations (15-17) support this contention. This is followed by reprecipitation and growth of equiaxed grains of γ' , which occurs when the austenitic matrix reaches some critical degree of supersaturation in the γ' forming elements. The net result is the formation of a microduplex structure comprising discrete equiaxed grains of (recrystallized) γ and (reprecipitated) γ' . Thus any strengthening effect associated with the original γ' distribution is destroyed during working and the soft recrystallized material deforms superplastically at very low stresses.

Deformation occurring after the onset of recrystallization in the coarse grained compacts is concentrated heavily in these soft recrystallized "grain boundary" bands. For any given macroscopic strain, the local strain in these bands is considerably higher than in the fine grained compacts where recrystallization and the soft microduplex structure are more uniformly developed. Also strain accommodation by plasticity within the γ' strengthened interior of the residual coarse grain structure is more difficult and therefore wedge type cracks develop rapidly at points of stress concentration. The net result is that ductility in the coarse grained materials is considerably less than in the fine grained materials under similar working conditions.

The above model implies that the steady state flow stress of the coarse grained materials should be controlled by the structure within the recrystal-

lized bands and that if recrystallization were complete the flow stresses would fall to the steady state values of the initially fine grained materials. While this situation was never reached, because of the early onset of fracture, the flow curves did tend to converge at high strains to those of the fine grained compacts.

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