INFLUENCE OF POWDER PARTICLE SIZE DISTRIBUTION AND PRESSURE ON THE KINETICS

OF HOT ISOSTATIC PRESSING (HIP) CONSOLIDATION OF P/M SUPERALLOY RENE 95

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Summary

The objective of this research is to study the modes of deformation and deformation between powder particles during HIP of kinetics consolidation of superalloy powders. The kinetics of powder densification and the powder morphological changes occurring during HIP were determined as a function of applied pressure, temperature and initial powder particle size distribution for the nickel base superalloy RENE 95. It is found, for example, that at 1120°C and 103 MPa, consolidation occurs rapidly (full density is reached within five minutes) mainly by instantaneous plastic flow instead of by local creep deformation. It is also found that non-creep deformation and/or a full distribution of particle sizes can result in a greater number of undeformed particles in the consolidates. The boundaries of these undeformed particles can become prior particle boundaries (PPB). The experimental results are compared with a newly developed analytical model for HIP consolidation, which takes into account the regimes of deformation mechanisms of the superalloy powders as well as the effect of differences in the distribution of particle sizes on the kinetics of densification. The analytical model also suggests HIP conditions for more uniform deformation of all powder particles.

Introduction

Hot isostatic pressing (HIP) of alloy powders to near-net-shapes can be the most cost effective method of powder consolidation. Briefly, HIP involves consolidation of the powder particles at a high temperature and pressure. During this process, densification is believed to occur in superalloy powders principally by creep deformation of the particles. In the superalloy case, particles of unequal size are present in the initial powder mixture, and it can be visualized that the smaller particles undergo more extensive deformation in comparison with the larger particles, leaving behind undeformed boundaries of the large powder particles in the final consolidate. These undeformed boundaries, also known as prior particle boundaries (PPB), are sites for deleterious precipitation of carbide films and are partly responsible for the at times poor mechanical properties of as-HIP'ed consolidates [1-3]. The possibility exists to improve the properties of as-HIP'ed material if, during HIP, maximum mechanical deformation of all particles occurs. In what follows we will show that the heterogeneous deformation of only the smallest powder particles present in a powder distribution is the result of currently used HIP parameters of temperature and pressure. This study and the results presented provide a scientific understanding of the mechanisms of deformation during HIP and the kinetics of HIP densification. This, in turn, can provide the basis for a proper choice of HIP variables.

Experimental Procedure

Argon atomized P/M nickel-base RENE 95 superalloy powders, whose chemistry is given in Table I, were sieved and classified into three different powder particle size distributions. The three distributions were labeled as monosized, bimodal and natural and had target particle size ranges of -170,+200 mesh $(75-90\mu m)$; 80% -170,+200 mesh and 20% -400,+500 $(33-35\mu m)$; and -140 mesh $(\langle 104\mu m \rangle)$, respectively. Actual size distributions are given in Table II, along with the tap densities for the three powder distributions. Powders were canned and HIP'ed (Figure 1) at a temperature of $1120\,^{\circ}\text{C}$ for various hold times ranging from 5 minutes to 180 minutes at pressures of either 103 MPa or an order of magnitude lower at 10.3 MPa. As shown in Figure 2, all HIP cycles included a one hour programmed ramp time which preceded the attainment of steady state HIP temperature and pressure before timing of the hold periods began. After HIP, stainless steel can material was removed mechanically from the consolidates, followed by density measurements (volume was measured by an air compression pycnometer) and microstructural observations. All powder handling was carried out in an inert environment. The typical HIP temperature of 1120°C was chosen since this temperature is slightly below the γ' solvus for RENE 95, allowing the primary γ' to prevent abnormal grain growth during consolidation.

Table I. Nominal Alloy Composition of RENE 95 in Weight Percent

C B Ni Al Ti Cr Co Mo W Nb Zr

0.07 0.01 62.4 3.5 2.5 13.0 8.0 3.5 3.5 3.5 0.05

^{*} RENE is a trademark of the General Electric Company.



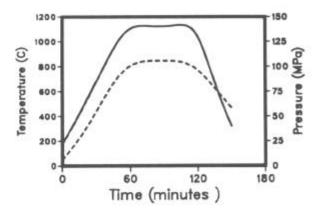


Figure 1. Canned and HIP'ed RENE 95 powders: (2) monosize, 103 MPa, (11) bimodal,103 MPa, (22) natural, 103 MPa, and (17) natural,10.3 MPa. All four cans were HIP'ed for a hold time of 60 minutes.

Figure 2. Typical temperature (solid line) and pressure (dotted line) curves plotted as a function of time showing the programmed one hour ramp time included in all HIP cycles and, for this figure, a hold time of 60 minutes at 1120°C and 103 MPa.

Table II. Tap Densities and Particle Size Distributions

Particle Distribution	Tap Density, g/cm ³ (% Theor. Density)		Actual Distribution
Monosize	4.78 (58%)	-170,+200 mesh (75-90µm)	9.5% +170 mesh (>90μm) 84.9% -170,+200 mesh 5.3% -200 mesh (<75μm)
Bimoda1	5.05 (61%)	80% -170,+200 mesh	(see monosize)
		20% -400,+500 mesh (33-35μm)	2.3% -325.+400 mesh (35-43μm) 95.2% -400,+500 mesh
			2.5% -500 mesh (<33μm)
Natura1	5.25 (63%)	-140 mesh (<104 μ m)	100% -140 mesh

Experimental Results

Using the density of the natural powder material HIP'ed at 103 MPa for 180 minutes as representing full density, it was found that within the experimental error of 0.2%, all three powder distributions reached 100% full density within the first 5 minutes of hold time at the HIP pressure of 103 MPa, as shown in Table III. Hence, densification must have occurred predominantly by athermal, time independent plastic flow of the powder particles. PPB's are clearly visible in the microstructures of all three types of consolidates, as shown in Figure 3. Increasing hold times to 180 minutes resulted in a coarsening of the γ^{\prime} (Figure 4) and an apparent decrease in porosity conceivably due to lattice and grain boundary diffusion [4,5]. However, there was no change in the number or distribution of PPB's when compared to the microstructures of the material HIP'ed for 5 minutes.

Reducing the applied HIP pressure by a factor of ten resulted in a marked slowing of the densification kinetics for the natural powder size distribution, as shown in Table IV. Powder morphological changes accompanying the increases in density with increasing hold times are represented in Figure 5. Note that within the first 15 minutes at temperature and pressure, only the smallest particles seem to deform. Prior to discussing these results and their implications with respect to HIP modeling, we will briefly present and discuss a newly derived HIP model.

Table III. Percent Full Densities at 103 MPa HIP Pressure

Particle Distribution	Hold Time 5	(min) 180
Monosize	100	100
Bimoda1	100	100
Natura1	100	100

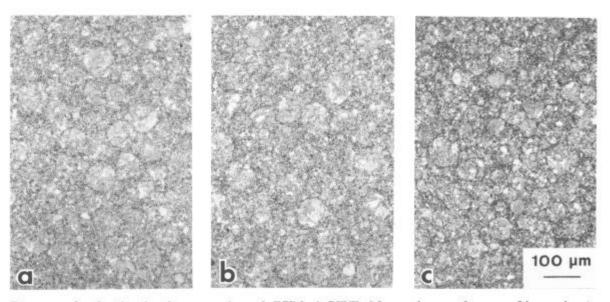


Figure 3. Optical micrographs of HIP'ed RENE 95 powders after a five minute hold time at 103 MPa and 1120°C for (a) monosize, (b) bimodal, and (c) natural powder distributions.

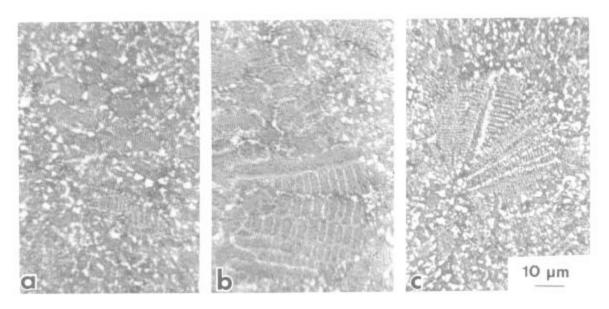


Figure 4. Optical micrographs of RENE 95 powders HIP'ed at 1120° C for 180 minutes showing γ' morphology: (a)monosized, (b)bimodal, and (c)natural.

Table IV. Percent Full Densities at 10.3 MPa HIP Pressure

Ho1d	Time	(min)
5	15	180
92.6	96.5	99,6
	5	

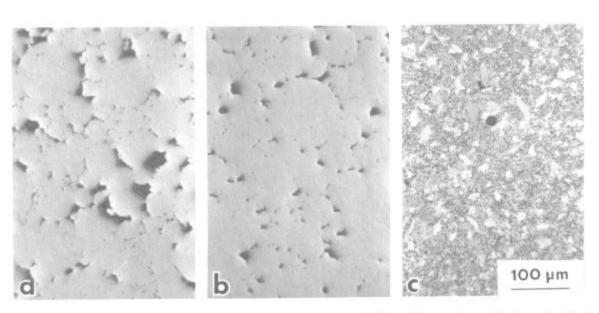


Figure 5. Optical micrographs of HIP'ed RENE 95 powders (natural size distribution) showing morphological changes of powders as a function of hold time at 10.3 MPa and 1120°C: (a)5 minutes, (b)15 minutes, and (c)60 minutes.

HIP Densification Modeling of a Bimodal Powder Distribution

There currently exists a model of HIP densification as proposed by Arzt, Ashby and Easterling [7] which establishes the deformation mechanisms (either athermal plastic flow, power law creep or diffusion) which occur during HIP densification of powder particles. This model considers particles of only one size, and predicts a single boundary between the HIP densification mechanisms of athermal plastic flow and power law creep, as shown by the dotted line in Figure 8. The regions of different deformation mechanisms of HIP densification are plotted in terms of relative density and log normalized applied pressure, P/Y, where Y is the material yield strength. For the typical RENE 95 HIP conditions of 103 MPa and 1120°C, where the applied HIP pressure approximately equals the yield strength at this temperature, Figure 8 shows that for a single particle size distribution, densification occurs predominantly by athermal plastic flow, and densification should be relatively time independent.

As mentioned, the model of Arzt et al. [7] considers only a single particle size. However, in reality P/M consolidates are made of a natural distribution of powder sizes below a given mesh size. The possible disparity between theory and practice has prompted the development of a model by Nair and Tien [6] to incorporate the influence of a particle size distribution on both the kinetics of HIP densification and the deformation mechanisms operating during HIP consolidation of superalloy powders.

How are the Arzt et al. [7] results changed when the powders are no longer of the same size, but have a somewhat more realistic bimodal distribution? In order to rederive the HIP densification mechanism map for a bimodal particle distribution, expressions are derived for (a) the distribution of unequal sized particles in a random mixture, and (b) the relationship between the interparticle contact force and the applied pressure.

The results of (a) are obtained in open form using the Percus-Yevik [9] hard sphere approximation for the molecular structure of liquid alloys as derived by J.L. Lebowitz [10], namely,

$$r g_{ij}(r) = \frac{1}{2\pi i} \int_{\delta - i\infty}^{\delta + i\infty} 12 (\eta_i \eta_j)^{1/2} G_{ij}(s) \exp(sr) ds \qquad (1)$$

where $g_{ij}(r)$ is the radial distribution function between the i^{th} and j^{th} particle; $\eta_i = \pi \rho_i/6$ where ρ_i is the density of the i-type atom in #/unit volume. G_{ij} , which is the number of atoms at any given distance, r, from a central atom and in a thin shell of thickness, dr, is given by

$$G_{ij}(\mathbf{r}) = \rho_j 4\pi \mathbf{r}^2 g_{ij}(\mathbf{r}) d\mathbf{r}$$
 (2)

To relate the applied stress, σ , to the interparticle contact force, F, the relationship by Molerus [8] for the single size case can be modified for the bimodal case [6] to give

$$\sigma_{\text{app}} = (F/4\pi) \left[(1-\epsilon-f_2) G_{12}/r_1^2 + (1-\epsilon-f_1) G_{12}/r_2^2 + (1-\epsilon-f_2) G_{11}/r_1^2 + (1-\epsilon-f_1) G_{22}/r_2^2 \right]$$
(3)

where ϵ is the void volume fraction, f_1 and f_2 the volume fractions of the two different sized particles with respective radii r_1 and r_2 , r_1 , r_2 and r_3 , r_4 , r_4 , r_5 , r_5 , r_6 , r_6 , r_7 , r_8 ,

Given the above new formulations described by equations (1), (2) and (3), the new model then proceeds to derive for the bimodal case the density as a function of time using the premise that the densification can be modeled by a fictitious growth of the particles around their centers, with the equations involved here being those of mass balance and geometry.

The final results of the theory are shown in Figures 6-9. Interestingly (see Figure 6), the interparticle deviatoric stress causing particle deformation is of the order of 0.1 to as high as 100 times the HIP pressure depending upon the particle size and the instantaneous density. Further, this deviatoric stress on the smaller particle can be of the order of 1.5 times that on the larger particle. In addition, Figure 7 indicates that the smaller particles undergo the greater degree of plastic deformation relative to their larger counterparts. These results are in contrast to the Arzt et al. model wherein all particles experience the same levels of stress and strain.

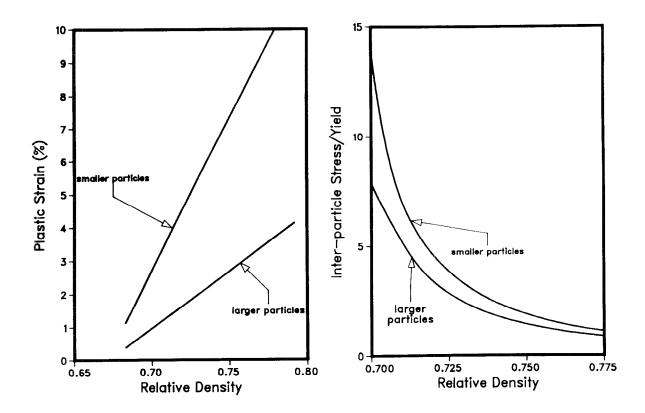


Figure 6. Plastic strain suffered by smaller and larger particles during HIP of a bimodal particle size distribution of powders as calculated by Nair and Tien [6] $(\log (P/Y) = -1.0, T = 1120^{\circ}C)$.

Figure 7. Interparticle contact stresses experienced by smaller and larger particles during HIP of a bimodal size distribution of powders as calculated by Nair and Tien [6] (log (P/Y) = -1.0, T = 1120°C).

The regimes of HIP densification mechanisms as calculated by the Nair and Tien model are shown in Figure 8, where the ratio of large to small particle diameters is 2.3 and the weight percent of small particles is approximately 20%. These values correspond to the size ratio and weight percentages obtained in the bimodal batch of powders. In comparison with the results of Arzt et al., also shown in Figure 8, we note the following: (1) The athermal plastic flow field is increased for the bimodal case relative to the single size particle case, and (2) there is a region on the map for the bimodal case, shown hatched in Figure 8, wherein the small particles deform by athermal plastic flow while the large particles undergo creep deformation. In contrast, all particles deform by the same mechanism in the Arzt model, and thus the Arzt model cannot explain powder size effects, which were observed.

A natural distribution of powders contains particles much smaller in diameter than the -400,+500 mesh (33-35µm) powders used in the bimodal batch of HIP'ed powders. Accordingly, to estimate how a natural distribution of powders might behave during HIP, Figure 9 plots the HIP densification mechanism map for a larger particle diameter ratio (4.0) and lower weight percentage of smaller particles (5%) than those used to calculate Figure 8. The results indicate that the athermal plastic flow field is futher increased (as compared to Figure 8) and the width of the hatched region increases. We tentatively conclude from these model results that in going from a bimodal to a natural powder distribution, the athermal plastic flow field may increase, as may the width of the hatched region (wherein the small particles squash plastically around the creeping large particles). The implications of these theoretical findings are next discussed with respect the experimental results of the present study.

Discussion

The experimental results using the monosized batch of powders show rapid densification at the typical HIP conditions of 103 MPa and 1120°C. This is consistent with the results of both the orginal Arzt et al. [7] model and with the Nair and Tien model when a single particle size is used in that model. The models indicate, as seen in Figure 8, that most of the densification at these HIP conditions should occur by athermal, time independent plastic flow of the powder particles.

The experimental results of the bimodal batch of powders HIP'ed at 103 MPa and 1120°C also show very rapid densification. Qualitatively, it appears that many of the smaller particles have undergone extensive deformation, while the majority of larger particles appear unchanged (Figure 3b). The Arzt et al. model, of course, cannot explain the above observation since the model does not distinguish between particles based on size. Such a distinction is only brought out in the Nair and Tien [6] model and the results of the latter model can explain the experimental observations. For example, Figure 8 predicts that the bimodal batch will suffer a greater degree of densification by an extremely rapid athermal plastic flow (as compared to the single size case) and, further, a regime is predicted (the hatched region in Figure 8) wherein the smaller particles are 'squashed' between the larger ones. Even outside this region, the smaller particles are predicted by the model to deform by a greater extent when compared to the larger particles (Figure 7). This is due to the intrinsically higher stresses exerted on these smaller particles (Figure 6).

With regard to the experimental results for the natural size distribution batch of powders HIP'ed at 1120°C and the lower pressure of 10.3 MPa, we have evidence (Figure 5) that the fines (very small particles) tend to get completely smeared out, apparently more so than in the bimodal case. The theoretical result of Nair and Tien (Figure 9) is even consistent with this finding. Figure 9 predicts, for example, that as both the size and the number of smaller particles decrease, the width of the hatched region appears to increase and so does the athermal plastic flow regime in the HIP densification mechanism map.

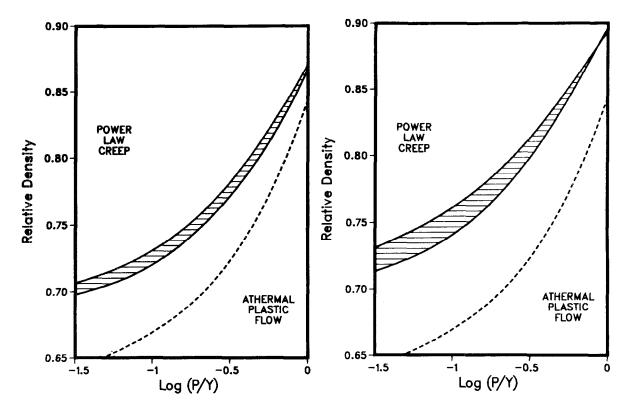


Figure 8. Boundary between the power-law creep and athermal plastic flow regions in a HIP densification mechanism map. The dashed line is as calculated by Arzt et al.[7] for deformation of single sized particles. The upper solid line is for the smaller particles and the lower solid line is for the larger particles as calculated by the model of Nair and Tien [6] (T=1120°C, particle size ratio=2.3, and weight % of smaller particles is 20%).

Figure 9. Same as Figure 8 except the particle size ratio = 4.0 and the weight % of smaller particles is 5%.

Conclusions

It would appear that the HIP parameters of pressure and temperature, where the temperature range is just below the γ' solvus, result in component entirely by time independent plastic densification achieved almost deformation of powder particles. The smaller particles in a distribution of particle sizes are observed to suffer a higher degree of plastic deformation in comparison with the larger particles. The boundaries of these larger particles are observed to be the sites for PPB's. These results are consistent with a new model which takes into account particle size differences in HIP densification mechanisms and kinetics. The model teaches that more microstructural uniformity of the final HIP'ed product can result if the pressure/temperature conditions are so chosen that the dominant densification mechanism is creep or creep-like deformation. Further work needs to be done to extend these results to different temperature and pressure regimes wherein the mechanisms of deformation and densification during HIP are not necessarily dominated by athermal plastic flow and can include localized creep and/or superplastic flow of the powder particles.

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