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# OBSERVATIONS ON THE MICROSTRUCTURAL DEVELOPMENT DURING HIPING

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## 1 ABSTRACT

The quality of the initial packing of the powders and the microstructural development of the pore "phase", during HIPing a C1018 steel to various densities, were compared with 2D-simulations of the densification process. The results were also compared with densification and microstructural observations during HIPing of a Ni-superalloy AP1 and presintered W(Ni) solid solution. Densification during final stage HIPing was mainly controlled by the elimination of a small number of larger residual pores or by elimination of pores at grain boundaries.

## 2 INTRODUCTION

A major purpose of HIPing is the production of dense and homogeneous materials. Hence, it is of importance to be aware of effects which may lead to residual pores or an inhomogeneous microstructural development during HIPing. A special challenge in this respect is posed by the new, economically well founded high volume HIP or Sinter-HIP systems, which operate at pressures below 60 MPa. The low pressure and the particular microstructure of materials which were presintered to closed porosity require an increased awareness of defect bearing features.

In the subsequent three sections microstructural observations during HIPing of C1018-steel, AP1 (Ni base superalloy) and presintered W(Ni) are presented. Defects during HIPing are found to develop from deviations of the initial partical arrangements from the ideal random dense packing, from the different individual deformation of individual particles and from pore/grain boundary separation in the presintered microstructures.

## 3 INITIAL PARTICLE ARRANGEMENT AND PORE SIZE DISTRIBUTION DURING FINAL STAGE HIPING

Figure 1 shows a sequence of microstruc-

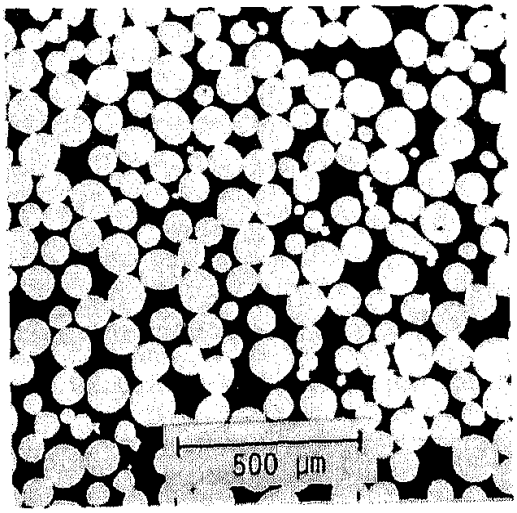



Fig.1a 1 min, 1 MPa, D=0.66, unetched.

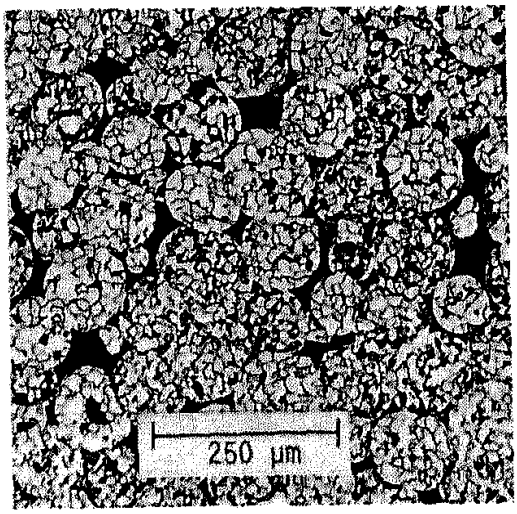



Fig.1b 20 min, 30 MPa, D=0.91.

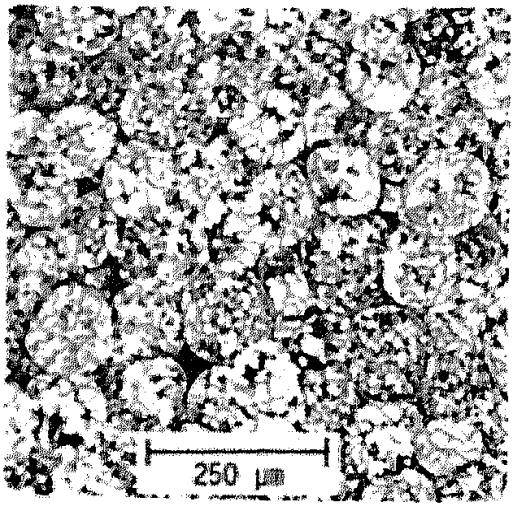



Fig.1c 10 min, 50 MPa, D=0.95.

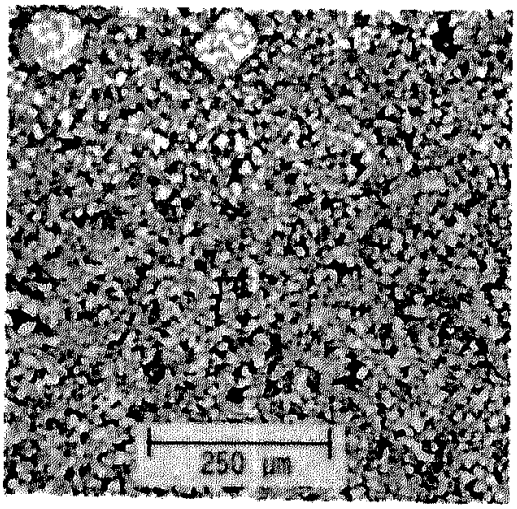



Fig.1d 20 min, 80 MPa, D=0.99.

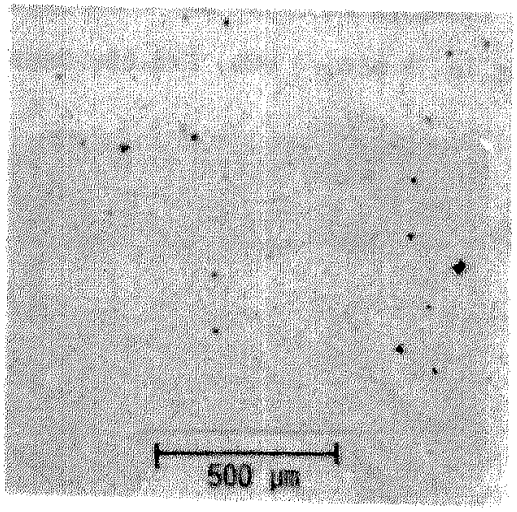



Fig.1e 20 min, 80 MPa, unetched.

Fig.1 Microstructure of C1018 after HIPing at 1000 °C (Heating rate 25 K/min, sieve fraction 100 to 125 µm).

tures of C1018 (100µm) HIPed to relative densities between 0.66 and 0.99. At densities below 0.9 the section shows areas of higher and lower particle density. Most of the virtual porosity variation is due to the 2-dimensional sectioning of the poured spheres. Part of the larger pores may indicate ,however , packing inhomogeneities which result in a few larger pores than should be present in a random dense arrangement. At HIP densities of 0.97 and 0.99 a small number of large pores is still present. Etching confuses the microstrucuture in Fig.1d. An unetched microstructure of the same sample in Fig.1e clearly shows that at a density of 0.99 some relatively large pores (approx. 40 to 50 µm) exist. In between most particles the fine regular porosity has disappeared.

The pore size distribution was determined by lineal analysis. At a relative density of 0.99 the chord length of 40µm was measured with a frequency of approximately 1%.(Fig.2). The result indicates the presence of a small number of pores with pore sizes in the order of the initial particle radius even in a material which is often considered as dense. The presence of comparatively large pores at very small porosities is expected to influence the mechanical properties of HIPed materials consider-

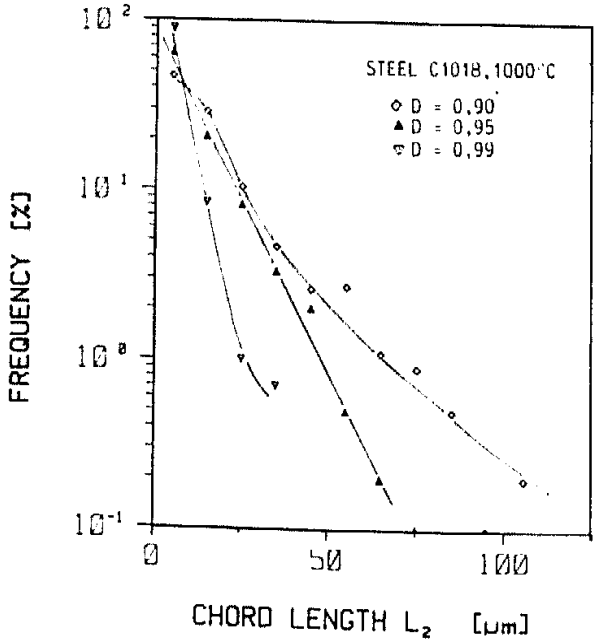



Fig.2 Pore size distribution of HIPed C1018-samples.

ably. The fatigue behaviour of high strength materials is known to be sensitive to pores of this size. This size of residual pores may also be found in material HIPed from much finer starting powders such as ceramics. If the agglomerates do not disintegrate well during the initial HIP stages a small number of large pores may remain in the dense ceramic matrix after HIPing. Since these large pores are potential nuclei for crack initiation they are likely to increase the Weibull modulus of the ceramic materials considerably.

The residual pores are thought to result from small packing faults of the initial powder arrangement. The influence of small initial packing faults is demonstrated in a 2-dimensional model shown in Fig.3. Two arrangements of particles were shown. Three particles forming a equilateral triangle were assumed to approach their centres during HIPing by removing material from the contact areas. One half of the removed material was virtually spread uniformly on the free surface of the pore between the particles. The same procedure was carried out with four particles forming a square. At same centre approach when the pore in the triangular arrangement is completely eliminated, the arrangement of four particles still shows a relatively large pore of a size of one third of the particle radius. It must be assumed that a certain number of similar packing defects also exist in powder compacts , that means in 3D arrangements even after very carefuly processing. As a consequence, a certain number of comparatively large pores have to be eliminated at the very end of final stage HIPing. At this stage, the present models assume a much larger number of much finer pores to be present.

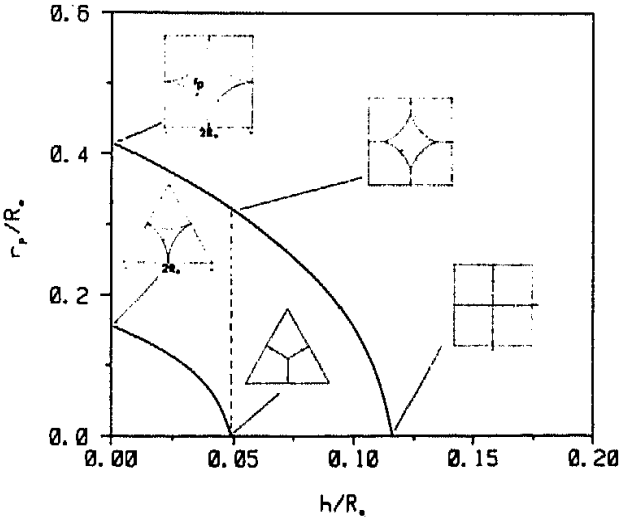



Fig.3 2-D Densification model of two typical arrangements of particles.

## 4 DEFORMATION OF INDIVIDUAL SPHERES

Figure 4 shows the microstructure of AP1 after HIPing at 1120°C for 20 min at 50 MPa. The size fraction was between 125 and 160µm. The residual porosity was below 0.5%. Figure 4a shows some particles with a much coarser grains size than the majority of the specimen. At a higher magnification the coarse grained particles reveal a high degree of residual sphericity, that means the particle appears to have been only modestly deformed during HIPing (Fig.4b). The conclusion that reduced deformation of the AP1 particles resulted in the formation of the coarse grains was supported by the microstructural development in an accidental area of the HIP capsule. Figure 4c shows the microstructure in the corner of the cylindrical steel encapsulation. The powder particles are only slightly deformed due to the insufficient deformation of the steel capsule. The reduced isostatic pressure is also indicated by the expansion of the enclosed Ar-bubbles. The slightly deformed superalloy particles exhibit coarse grains compared to the fine

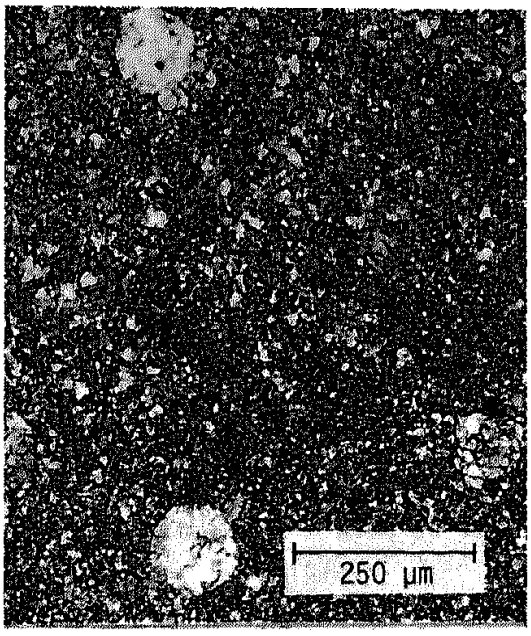



Fig.4a Fine grained microstructure with some undeformed coarse grained particles.


Fig.4b A coarse grained particle at higher magnitude.

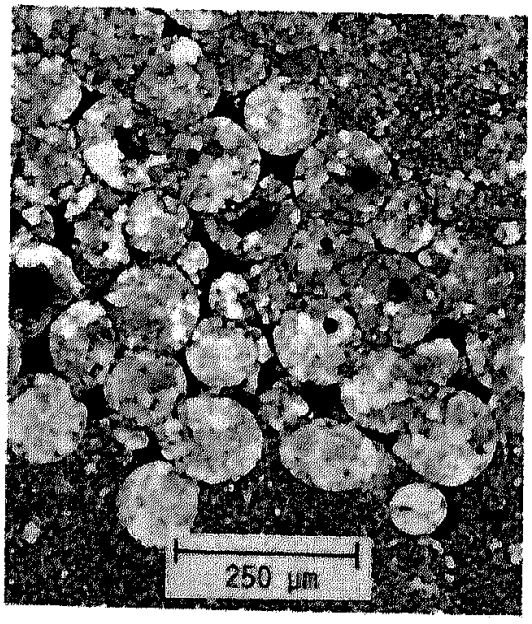


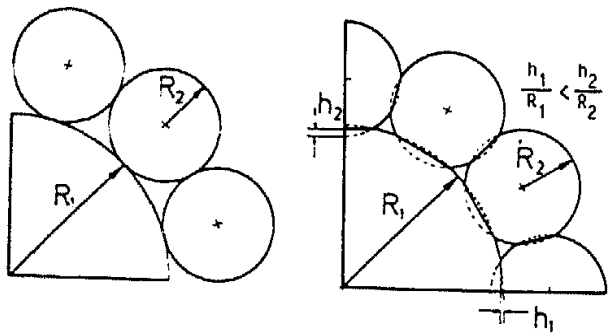

Fig.4c An area of undeformed, coarse grained particles near a corner of HIP-container.

Fig.4 Microstructure of Ni-base superalloy AP1 after HIPing at 1120 °C, 50 MPa for 90 min.

grained microstructure of the densified areas of the sample. A small number of particles which was distinguished from the rest of the material by a very coarse grain size was also found in HIPed C1018 (Fig.1d). In the latter case the particle size fraction was between 100 and 125µm.

The fact that all particles in the area of reduced deformation shown in Fig. 4b developed a coarse grain microstructure proves that the different microstructural development of a few particles in a fine grained matrix does not primarily result from a deviation of their chemical composition or initial microstructure. The different microstructural development is traced back to differences in the deformation of individual particles. Different degrees of deformation may arise from size differences of the particles. If a larger particle is surrounded by smaller particles, as schematically shown in Fig.5a, the densification during HIPing results in a minor deformation of the larger central particle only (Fig.5b). An arrangement as shown in Fig.5a may occur in a usual particle size distribution in rare cases. The particle size distributions of the AP1 and in particular the C1018 were very narrow, however, indicating other geometrical arrangements to be responsible for the development of the microstructural inhomogenities.

It is possible for example that a few particles were deformed only slightly during the intital stages of HIPing to pockets in the powder particle arrangement. If the surrounding matrix of deforming particles softens due to dynamic recrystalization the initially undeformed particle remains as a relatively hard spherical inclusion in a softer (in respect to the deformation during HIPing) matrix.




a b

Fig.5 Deformation behavior of small particles in contact with a big particle (schematic).

## 5 FINAL STAGE SINTER-HIP OF W(Ni)

Final stage of sintering starts when the break up of interconnected pore channels leads to isolated pores which lie along three or four-grain junctions (Uematsu 1978). During the final stage sintering both densification (pore shrinkage) and coarsening (pore coarsening and grain coarsening) occur simultaneously. Shrinkage occurs by migration of vacancies from the pores along the grain boundaries to vacancy sinks (grain boundaries and free surfaces). In the final stage there are two phenomena by mass transfer between isolated pores and by pore coalescence during grain growth. During grain growth pores either remain at the boundaries or become separated and "trapped" inside the grains. Pores that are trapped usually shrink at much lower rates than pores which are at grain boundaries, since bulk diffusivity is much smaller than grain boundary diffusivity. The separation of pores and grain boundaries usually starts at densities above 90 %. Totally closed porosity, however, is expected at densities above 93%. As a consequence pore/grain boundary separation may occur during sintering in a sinter-HIP treatment as shown in Fig.6.

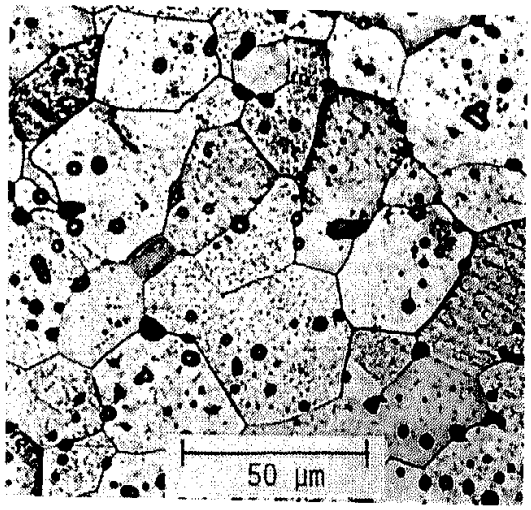



Fig.6a After sintering at 1400 °C for 60 min.

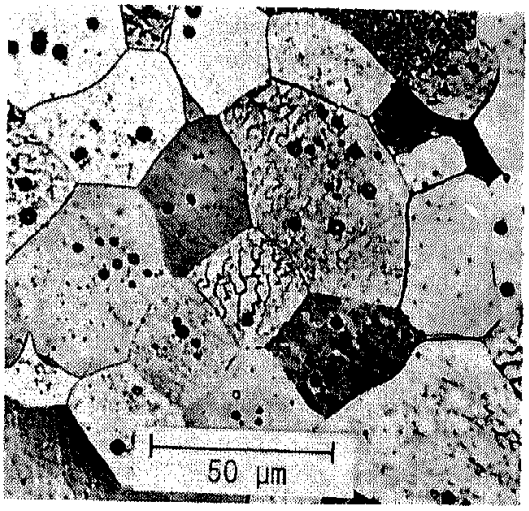



Fig.6b After post-HIP treatment at 1400 °C for 60 min.

Fig.6 Microstructure of W-0,15Ni after sintering at 1400 °C and subsequent HIPing at the same temperature.

There are two regimes where pores can remain attached to the grain boundaries (Fig.7). Pores which are large enough are immobile but retard the motion of the boundary (pore control in Fig.7) (Uematsu 1978, Brook 1969, Hsueh 1982). Pores which are small enough move along with the grain boundaries without stopping their motion. The intersection of the two regimes of pore-boundary interaction leads to a pore-boundary separation region with $G^*$ as the smallest grain size where grain boundary pore separation occurs.

Final stage sintering of W with Ni shows extensive grain boundary pore separation

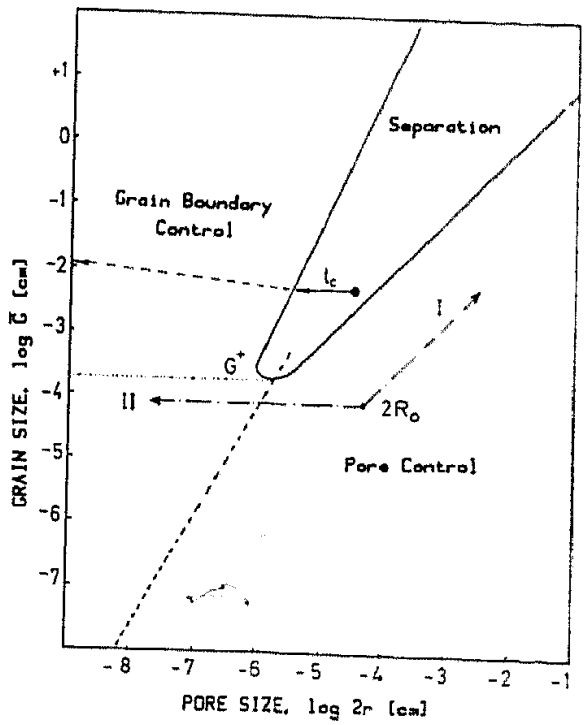



Fig.7a Pore/grain boundary separation program.

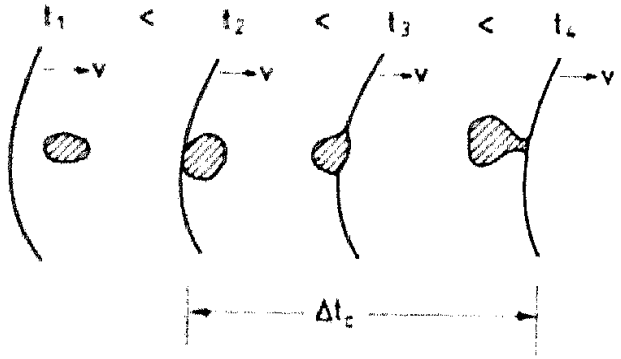



Fig.7b Contacting of grain boundary and pore; migration to the breakaway position.

Fig.7 Pore/grain boundary separation during sintering and HIPing.

since both the grain boundary and surface diffusion are increased by the presence of Ni at the grain boundaries. Figure 6a shows the microstructure of W-0.15Ni after final stage sintering.

When samples are subject to a high hydrostatic pressure as during HIP, the surface and grain boundary diffusion constants essentially remain constant, but the applied pressure gives much steeper gradients of the chemical potential of the vacancies and results in a faster vacancy flow towards the grain boundary sinks. The driving force for diffusional pore coarsening on the other hand remains unchanged. Figure 6a clearly shows the grain boundary pore separation in a W-0.15wt.%Ni compact during sintering (Kaysser 1980). The large pores are too immobile to remain attached to the grain boundaries. As shown in Fig.6b the application of 200 MPa at 1400°C changes the microstructure dramatically.

In the wake of migrating grain boundaries all pores were eliminated. It can be shown that densification by other mechanisms (e.g. power law creep which is most frequently observed in other systems and which depends on the bulk diffusivity) is small compared to the pore shrinkage by vacancy elimination at the grain boundaries (Kaysser 1984). The presence of Ni increased the grain boundary diffusivity considerably, whereas bulk diffusivity is too low to yield any influence even at HIP conditions. The pore grain boundary breakaway diagram can be used for the HIP conditions after some additional kinetic considerations. If pores come into contact with a grain boundary they were subject to separation after a short time. During HIPing, pores which come into contact with a grain boundary shrink in a short time interval $t_c$ to a size at which the attachment due to boundary control becomes dominating (Fig.7b). This time interval can be shorter than the time which elapses before the contacting grain boundary migrates into a breakaway condition.

## 6 SUMMARY

During final stage HIPing at densities above 98% a small number of relatively large pores,of 1/3 of the particle radius, are still present, which mainly trace back to the irregularities in the initial powder packing.

During HIPing some percent of the particles undergo only minor deformation. It was found in AP1 that these particles showed coarse grains whereas the more heavily deformed particles displayed a fine microstructure.

During final stage HIPing of Ni-doped W, complete pore elimination occurs when pores come into contact with grain boundaries migrating due to normal grain growth. The absence of power-law creep allows the description of the pore/grain boundary separation conditions by a critical time interval $t_c$ which elapses between the first contact of the grain boundary with the pore and the instant when the grain boundary has moved into the breakaway position.

## 7 ACKNOWLEDGEMENT

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