CARBIDE PRECIPITATION IN SUPERALLOYS

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The formation of surface carbides in individual particles of superalloys has been studied. The sites of precipitation and type of carbides in as-atomized as well as heat treated powder particles was investigated to establish a basis for the development of suitable heat-treatments in connection with hot consolidation.

INTRODUCTION

Technically, the problem of carbide precipitation at previous particle boundaries (PPB) in powder metallurgy superalloys (1) has been overcome in various ways. However, the literature does not contain much information on the mechanisms involved. The present study set out to investigate carbide precipitation in loose powder particles of a nickel-based superalloy in order to provide a background for the understanding of the various solutions for the PPB problem and perhaps for the development of new approaches.

It is to be expected that precipitation in powder particles will follow a different temperature- and time dependence than in cast-and-wrought materials. Also, the chemistry of alloys for powder metallurgy processing is somewhat different, mainly in respect of lower carbon content.

EXPERIMENTAL

The alloy studied was argon-atomized Astroloy of the following chemistry:

Ni	Co	Cr	Mo	Ti	Al	C	O ₂
Bal	16.8	14.6	5.2	3.5	4.1	0.036	0.015

The carbon content is about one-half of the conventional level.

Loose powder particles were sealed in evacuated silica tubes at a residual pressure of 10^{-5} torr. The capsules were heat treated for times from 15 min. to 5 hrs at the following temperatures: 950, 1025, 1100 and 1150°C. The results shown in this paper are mainly based on one-hour heat treatments. After holding for the desired time at temperature, the capsules were quenched in water.

The powder particles were prepared for microscopic examination by placing them on a copper surface and depositing nickel on the particles and the copper substrate. The resulting nickel-plus-particle cake could be stripped from the substrate and metallographic sections were prepared for evaluation with light-optical and electron microscopy.

RESULTS AND DISCUSSION

Fig. 1 shows an etched metallographic section of an as-atomized particle, revealing the dendritic solidification pattern characteristic for gas-atomized powders. The interdendritic regions are rich in carbon and carbide formers, and they are the sites of carbide formation.

Fig. 1. Dendritic solidification structure of argon-atomized Astroloy.

Fig. 2 shows an extraction replica of such a powder particle with extracted carbides of ca. 0.02 μ m size. Electron diffraction revealed these particles to be of the MC type.

Fig. 2. MC-carbides extracted from an as-atomized powder particle.

Heat treatment of the powder particles at 960°C for 1 h produces the structure shown in fig. 3. Again the interdendritic regions are preferentially etched because they are richer in γ' and in carbides. However, the carbides present after this heat treatment are of $M_{23}C_6$ type. At 1100°C, MC

Fig. 3.
Structure formed after
1 hour heat treatment
at 950°C.

begins to reappear, and at 1150°C, MC is present alone. Fig. 4 shows the distribution of the carbides formed at that temperature. The specimen has been etched to attack only the carbides. The carbide particles are now quite coarse, but they are still situated in the interdendritic spaces.

Fig. 4. Carbide structure obtained after annealing at 1150°C for 1 hour.

At none of the temperatures was preferential precipitation of carbides at particle surfaces observed. The absence of heavy carbide nucleation at the free surfaces of heat treated particles was also confirmed by transmission electron microscopy of carbon replicas of the particle surfaces. These surface replicas revealed only a population of very fine carbide particles, much smaller in size than the carbides formed in the interior. Clearly, the free surfaces of particles do not play the role of dominating nucleation sites which is often ascribed to them, and the carbides responsible for the PPB problem in hot compacted powders must be of different origin.

However, there are many particles in gas atomized powders which have stuck together as the result of collisions during the atomization process. Fig. 5 shows a particle with another particle attached to it. The boundary between the two is clearly a preferential site for carbide precipitation at the temperature to which this specimen was annealed, 1150°C. It was also observed that normal grain boundaries inside the particle do not act as preferential nucleation sites for carbides.

Fig. 5. Carbide precipitation in contiguous surface.

In the present report, we will not go into the problem of why metal-metal boundaries formed by particle collisions are more effective nucleation sites for MC than either free particle surfaces or grain boundaries, but the mere observation of this behaviour gives some pointers to the mechanism of the various processes developed to avoid PPB precipitation in powder metallurgy superalloys.

It has been found favourable for achieving good ductility to carry out HIP densification in two steps, a precompaction step at a lower temperature which does not give full densification, and a second step at higher temperature to achieve full density (2). The precompaction step is in the regime of $M_{23}C_6$ precipitation. The heat treatment is long enough to produce fairly coarse and stable particles of this carbide without producing excessive amounts of particle-to-particle contact surface. During final compaction, MC will form, and will decorate the particle-to-particle contact surfaces, but this process will be slower than in the case of direct heating where the carbide population is finer and more easily dissolved, and where the formation of particle-to-particle contact surfaces proceeds to a considerable part concurrently with the MC formation.

A different approach in the field of pre-heat-treatments would be to heat the powder, before applying any pressure at all, to the temperature of MC formation according to the observations reported above. This would form coarse and stable particles in the interior of the particles. Since no

metal-to-metal contact surfaces had been created, there would be no coarse PPB precipitation and when pressure was applied for HIPing the coarse carbides would be stable enough to prevent carbon redistribution to the PPBs. Preliminary HIP experiments did indicate some reduction of PPB carbides compared to direct HIPing.

If hot compaction could be carried to completion below the temperature of MC formation, PPB precipitation would be considerably reduced. Low temperature compaction to full density is possible when the powder particles can be brought into a superplastic state. Our metallographic observations show that the grain size of argon atomized powders is much too coarse for superplasticity (most particles contain only a few grains). However, if the powder is deformed it will recrystallize during heating to HIP temperatures.

The recrystallization behaviour of Astroloy at various temperatures has been studied in detail at our laboratory (3). It is characterized by repeated nucleation and solute-drag restricted growth of new grains. This creates a very fine structure in primary recrystallization, with grain sizes of the order of 1-2 μm which are fairly stable against coarsening.

The process described as thermoplastic processing (4) consists in a heavy deformation of the powder particles prior to heating to HIP temperatures. This treatment would lead to an extremely fine grain size in the powder particles by primary recrystallization during heating to the HIP temperature. The densification treatment can then be carried out at temperatures (1000-1100°C) where MC forms only very slowly or not at all.

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