POWDER METALLURGY APPROACHES TO HIGH TEMPERATURE COMPONENTS FOR GAS TURBINE ENGINES

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PRICES SUBJECT TO CHANGE
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INTRODUCTION

The gas turbine engine has provided great impetus for the development of advanced high temperature alloys and their economical processing. The history of gas turbine engine development was indeed paced by significant material developments. Titanium, cobalt, and nickel-base alloys provided properties superior to iron-base alloys and were subsequently introduced as compressor and turbine components. Improved investment casting techniques coupled with the development of high strength cast alloys allowed the replacement of some forged turbine components with stronger yet lower cost castings. Directional solidification has produced unidirectional microstructures in superalloys that provide turbine blades with greatly improved rupture life and thermal fatigue resistance. And now powder metallurgy materials, offering both processing economies and improved mechanical properties, are finding their way into engines.

Recent activity has been devoted to two types of powder products for gas turbine engines. These are pre-alloyed superalloy powders and oxide dispersion strengthened materials. This paper will not provide an extensive coverage of the total activities in these fields, but will be a review of some work conducted within NASA and by NASA contractors.

POWDER METALLURGY SUPERALLOYS

Cast ingots of nickel-base superalloys often suffer the liability of severe segregation. By atomization of cast alloys, powder particles are produced which are homogeneous in chemistry and microstructure. The consolidation of such a powder results in a dense homogeneous body free of macrosegregation and consequently even alloys which were restricted to use as castings because of their high alloy content are more amenable to primary working. Such powder metallurgy (P/M) superalloys are usually characterized by a fine grain size and consequently their mechanical properties are superior to their cast counterparts at low and intermediate temperatures, however, inferior at high temperatures.

Figure 1 (ref. 1) compares the tensile strength of the NASA P/M TAZ-8A alloy to cast TAZ-8A. The P/M material is superior in strength below about 800°C while the cast form is superior above 800°C. As illustrated in figure 2 (ref. 1) ductilities are comparable below about 800°C; however, at higher temperatures, the P/M form has excessive ductility and indeed is "super-plastic".
Superplasticity allows easy fabrication of P/M superalloys to intricate shapes under low forming loads. This is illustrated in figure 3 which shows a turbine blade form of P/M TAZ-8A which was pressed in dies made of cast TAZ-8A. This ease of fabrication of P/M superalloys is currently being capitalized on to produce full scale turbine disks of intricate cross section (ref. 2).

Because of their superior strength at low and intermediate temperatures, highly alloyed P/M superalloys are becoming competitive with wrought alloys for turbine and compressor disk applications.

Figure 4 (ref. 1) shows the superiority of P/M 713C and P/M TAZ-8A to wrought Inconel 718 in stress rupture at 650°C. Under a stress at which 718 would give a 100-hour life, the P/M alloys offer lives well over 300 hours. Heat treatments of the extruded P/M alloys can improve stress rupture life even further.

In order to achieve properties in P/M superalloys comparable to their cast counterparts at high temperatures (above 800°C), it is generally believed that grain growth must be achieved. Figure 5 (ref. 1) illustrates the stress rupture behavior of 713C as P/M alloy and in the cast form at 1038°C and a stress of 68.9 MN/m². The ultra fine grain size of the as-extruded 713C results in a very short stress rupture life. A heat treatment at 1230°C resulted in considerable grain growth and improved life. However, the grain size at .4 mm is still not comparable to the large as-cast grain size and consequently stress rupture life is less than half that of the as-cast alloy. Alloy 713C did respond to grain growth heat treatments which exceeded its gamma prime solvus temperature. Once the gamma prime was in solution, the grains underwent some growth. Contrasted to this behavior, TAZ-8A was fairly unresponsive to grain growth treatments because of its high volume fraction of gamma prime and its high solvus temperature. This alloy was even heated in an autoclave to 1315°C at 68.9 MN/m². This temperature, although above the incipient melting point, was selected in order to enhance the possibility of dissolving gamma prime and promoting grain growth. The autoclave pressure served to prevent void formation where local melting occurred. Even under this severe condition, no significant grain growth occurred and properties were not at all comparable to the cast alloy.

The ultra-strong cast alloy VI-A in P/M form was also subjected to heat treatments at temperatures where incipient melting occurs (ref. 3). Grain growth occurred to increase the as-extruded grain size of 0.0014 mm to 0.032 mm; however, this did not approach the as-cast grain size of 2 to 5 mm.

With cobalt-base alloy HS-31 (ref. 4), autoclave heat treatments of P/M material gave grain sizes of the order of 0.1 mm. Although this is
still considerably smaller than an as-cast grain size, stress-rupture life at 980°C was comparable to the life of the as-cast alloy as shown in figure 6 (ref. 4).

Thus, the varying response of P/M superalloys to grain growth attempts, suggests that improved high temperature properties might be sought by P/M processing of specially designed alloys to allow grain growth. For example, an alloy of lower gamma prime solvus temperature should provide easier grain growth; however, larger grain size would have to compensate for a loss of some gamma prime strengthening.

On the other hand, the HS-31 experience suggests that other factors, in addition to grain size, are important. As reference 4 indicates, properties equivalent to the cast alloy were not achieved until a solidification microstructure was obtained locally by incipient melting. Such a structure may augment the benefits achieved by grain growth alone and help in achieving properties comparable to as-cast alloys.

Grain growth in P/M 713C has also been controlled to produce a columnar grain structure without transverse boundaries by annealing in a temperature gradient (ref. 5). In this case, a gradient of about 28°C/cm was established along the length of an extruded bar and the furnace power was then increased to provide a temperature rise of about 28°C/hour. In this manner, each portion of the bar was exposed to an increasing temperature in the presence of the thermal gradient. The same result would be produced by moving a bar through a furnace with a similar temperature gradient.

To illustrate the effects of gradient annealing, a bar heated in a static temperature gradient is shown in figure 7 (ref. 5). Temperatures at various locations are indicated. It is seen that large columnar grains grew in the 1250°C to 1190°C region. There is an incubation period associated with abnormal grain growth in 713C which becomes shorter at higher temperatures. This is presumably associated with the time required for gamma prime to dissolve. Grains incubate rapidly at the high temperature end and because of their high growth rate are able to grow into the cooler region before competing grains incubate there and a large grained columnar structure results. Growth down the temperature gradient continues until the gamma prime solvus temperature is reached, about 1090°C. If the specimen is now advanced slowly into the temperature gradient, growth of the columnar grains can be continued to any desired length.

Tensile test on samples cut from such a columnar region as shown in figure 7 exhibited strengths within 5 percent of as-cast 713C up to 980°C. Stress rupture lives also appear comparable to as-cast 713C as shown in figure 8 (ref. 5).
The scatter in stress rupture life may be associated with the $[110]$ fiber texture that develops in the columnar grains during the competitive growth process. Single crystal superalloy specimens with orientations near $[110]$ show similar scatter in rupture life due to a tendency for a single slip system to operate leading to rapid deformation without work hardening (ref. 6). A more favorable texture in the columnar grains, such as $[100]$, might be effected by controlling the texture of the starting material.

A number of superalloys that have gamma prime solvus temperatures below the alloy solidus temperature are amenable to gradient annealing.

OXIDE DISPERSION-STRENGTHENED ALLOYS

Since oxide dispersion strengthened (ODS) alloys derive their strength from inert refractory oxide particles, they do not suffer the severe loss of strength at elevated temperatures that is inherent to gamma prime strengthened alloys. Thus, ODS materials retain some usable strength to over $1100^\circ C$ and, therefore, are of current interest for applications requiring moderate strength at high temperatures such as burner cans and vanes.

The effect and control of P/M processing variables is of utmost importance for ODS alloys since their mechanical behavior is highly dependent upon the quality of the dispersoid distribution obtained. Process variables have been under study at NASA-Lewis for some time.

Materials are comminuted and blended in an attritor with an organic fluid in order to produce fine and homogeneous powders. After attriting, the powders are dried and precleaned in hydrogen at near $300^\circ C$. Precleaned powders are cold compacted and sintered in hydrogen at $1090^\circ C$ to about 85 percent theoretical density. Final desification is achieved by hot rolling near $1090^\circ C$. Improved properties are developed in such materials by thermal mechanical processing (TMP). In most of these studies, the TMP consists of cold rolling to about 10 percent reduction, followed by one-half hour hydrogen anneal at $1200^\circ C$. This cold roll-anneal cycle is repeated in order to increase strength. Figure 9 (refs. 7, 8) summarizes some of the results. Here, the beneficial effect of TMP on the $1093^\circ C$ tensile strength of several compositions is illustrated.

As illustrated in figure 9, the effect of doubling the amount of dispersoid from 2 to 4 volume percent $Y_2O_3$ results in dramatic strength increases. The response to TMP is also more pronounced in the 4 volume percent $Y_2O_3$ composition. The $Y_2O_3$ alloy is stronger than the corresponding composition containing ThO$_2$ processed in the same manner and can also achieve strengths comparable to TD-Ni. Tensile elongations of all compositions of figure 9 are about 2 to 5 percent.
Figure 10 (refs. 7, 8) illustrates the interesting effect of starting with NiO instead of Ni. In this case, reduction of the NiO in the hydrogen treatments results in a very homogeneous ThO$_2$ dispersion with a minimal amount of agglomerated particles. If a similar benefit could be derived from starting with NiO $+$ Y$_2$O$_3$, it appears strengths at 1093°C in excess of 180 MN/m$^2$ could be achieved.

A great deal of thought and speculation has been devoted to the relative contributions of microstructural features to the strength of ODS materials. For example, the interplay of the amount, size, and distribution of dispersoid has received attention as well as the effects of grain size and shape.

The effect of grain size on yield strength for TD-Ni is shown in figure 11 (ref. 9). In addition to this grain size effect, a dependency of strength on grain shape has been noted as shown in figure 12 (ref. 9). Here, $l/d$ is the ratio of grain length to grain diameter; i.e., the grain aspect ratio.

Wilcox (ref. 10) found a Hall-Petch relationship to hold for yield strength at room temperature but not at 1093°C. However, a good correlation was found between stress-rupture behavior and grain aspect ratio. Figure 13 (ref. 10) illustrates the beneficial effect of elongated grain structure on strength and also suggests that a number of dispersion strengthened alloys conform to this relationship.

This observed dependence of stress rupture behavior on aspect ratio is explained (ref. 10) in terms of grain boundary sliding being the predominant deformation mode at elevated temperatures. A high value of grain aspect ratio minimizes the resolved shear stress on grain boundaries which therefore minimizes the amount of grain boundary sliding and leads to a more creep-resistant material. Grain boundary sliding must be accommodated by dislocation motion or diffusional processes to prevent void formation at boundaries. Dislocation motion is believed to be effective at moderate temperatures while diffusional processes become more predominant at high temperatures and low strain rates. The loss of ductility of ODS materials at high temperatures is then attributed to inability to maintain accommodation at boundaries under conditions of large amounts of grain boundary sliding thus leading to void formation and early low ductility failure.
CONCLUDING REMARKS

Powder metallurgy superalloys offer distinct strength advantages over conventional wrought alloys. Their superior mechanical properties at low and intermediate temperatures and their high degree of formability due to superplasticity is, of course, an ideal combination for expanded usage. The goal remains to capitalize on superplasticity for forming and then destroy it in order to provide strength at high temperatures as well as at intermediate temperatures. Only when this is accomplished will P/M superalloys truly compete with cast superalloys. We will, no doubt, eventually achieve much improved high temperature properties by pursuing imaginative means to enhance grain growth, by developing unique alloy compositions that are designed specifically for P/M processing, and by gaining a full understanding of the factors in addition to grain size that affect high temperature strength in such alloys.

A workable unified theory seems to be emerging in the field of dispersion strengthening. The desirability of a high aspect ratio grain structure appears apparent. Effort is required to identify the most economical and effective means of obtaining such a structure; e.g., by thermomechanical processing or by directional recrystallization. Recent unpublished work (ref. 11) also indicates that aligned microstructures can be obtained in ODS alloys by directional forging. Essentially, unidirectional metal flow is achieved by proper design of preform and die. Directional forging appears to offer the possibility of providing more complex geometrics in hardware having properties equivalent to current extruded ODS bar stock. Effective alloying with chromium and aluminum of the nickel-base matrix of ODS alloys has demonstrated much improved oxidation resistance. Continued efforts in this area should reduce our dependence on protective coatings.

Finally, an obvious challenge to the powder metallurgist is to combine the attributes of the two types of materials; i.e., gamma prime strengthening offered by P/M superalloys and dispersion strengthening. The feasibility of this concept has already been demonstrated by Benjamin (ref. 12) and indeed a commercial alloy of this type is currently available. To carry this to its full fruition, dispersion strengthening should now be combined with an ultra-high strength superalloy of high gamma prime volume fraction. The complexities of each type of alloy are compounded in such an approach, however, the reward of superior strength over the entire temperature spectrum of the alloy is enticing. Here the combined effects of microstructural features and the complementary, as well as opposing features of multiple strengthening mechanisms must be understood.
REFERENCES


Figure 1. - Tensile strength of P/M TAZ-8A and cast TAZ-8A.

Figure 2. - Tensile elongation of P/M TAZ-8A and cast TAZ-8A.

Figure 3. Formability of TAZ-8A prealloyed powder product.
P/M 713 C AS EXTRUDED GRAIN SIZE 0.01 mm

Figure 4. Stress rupture lives of P/M 713 C, P/M TAZ 8A, and wrought Inconel 718 at 650°C, 725 MN/m².

Figure 5. Stress rupture lives of P/M 713 C and as-cast 713 C at 1038°C, 68.9 MN/m².

P/M HS-31 AS EXTRUDED GRAIN SIZE 0.01 mm

Figure 6. Stress rupture lives of P/M HS-31 and as-cast HS-31 at 982°C, 90 MN/m².
Figure 7. - Gradient-annealed specimen of 713C sectioned longitudinally and etched to show grain structure developed.

Figure 8. - Stress rupture lives of P/M 713 C gradient annealed and as-cast 713 C.

Figure 9. - 1093°C strength of ODS Ni alloys.
Figure 10. - Response to TMP schedule of several dispersion strengthened nickels.

Figure 11. - Effect of grain size on yield strength: TD-Ni.

Figure 12. - Effect of grain shape on yield strength: TD-Ni.

Figure 13. - Dispersion strengthened nickel alloy grain aspect ratios vs creep rupture strength for 100 hr life at 1093° C.

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