Effects of Different Atomization Method on Powder Property, Microstructure and Low Cycle Fatigue Property of FGH97 Alloy

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Abstract. Two kinds of Ni-based superalloy powders (P-powder and A-powder) were prepared from masteralloy melted via the process of vacuum induction melting (VIM) by plasma rotating electrode process (PREP) and argon atomization (AA) method, which were used to fabricate two different alloys (P-alloy and A-alloy) using the same hot isostatic pressing process and heat treatment process. The study of microstructure and mechanical properties of FGH97 alloy were conducted to demonstrate the effect of different routes of powders preparation on the microstructure and low-cycle fatigue properties. Results show that the particle size distribution of P-powder is narrower than A-powder, and overall is larger, and the size of the inclusions in the A-powder is much smaller than P-powder. And it has been found that the grain size of A-alloy is finer and γ' phase is more uniform, compared with P-alloy, there is no residual dendrite, grain boundary carbides are intermittent punctate distribution; the strength of A-alloy is slightly higher, and the plasticity is better than P-alloy; at the same time, the crack initiation is formed at the site of slag inclusions, but the size of inclusions of A-alloy significantly smaller; accordingly, the resistance of low-cycle fatigue crack initiation of A-alloy is superior than that of P-alloy due to the fine powder prepared by the process of argon atomization, showing a life time of much more than 150 000 cycles, far better than P-alloy (<70000 cycles); and the fatigue property of A-alloy is very stable.

Keywords. Ni-based powder superalloy, FGH97, atomization method, powder property, microstructure, low-cycle fatigue property.

1. Introduction
FGH97 powder superalloy, which has excellent comprehensive performance in the temperature range of 650-750 °C \cite{1, 2}, is a highly alloyed and γ' precipitation-strengthened nickel-base powder superalloy. The content of γ' phase is generally about 62% \cite{2}. It is the preferred material for the manufacture of gas engine powder disks in China. At present, FGH97 pre-alloyed powders are mainly prepared by the plasma rotating electrode atomization (PREP) method \cite{2-4}. The advantages are that the powder has good sphericity and smoothness, and the oxygen content is low. However, it has fatal defects, which are mainly manifested in the overall coarseness of the powder particle size and inclusions is generally large \cite{5, 6}. Although researchers at home and abroad have made many efforts to increase the purity of powders, reduce the number of powder inclusions, and control the size of inclusions, such as the use of multiple mother alloy smelting processes \cite{7} and precision screening,
electrostatic separation of inclusions (ESS) [6] and other means to remove inclusions (especially large-sized inclusions) in the PREP powder as much as possible, and the reduction of the number of inclusions has achieved certain results as well; the control effect of reducing the size of inclusions is not ideal [6]. Therefore, due to the presence of a certain number of large-size inclusions in the PREP alloy powder, the overall level of mechanical properties (especially low cycle fatigue properties) of the powder alloys, which are formed by direct hot isostatic pressing, is not high. But also due to the randomness of the location of large-size inclusions in the distribution of specimens (or parts), also leads to the low cycle fatigue properties of the alloy is very unstable [8], a large dispersion test data; which is not conducive to engineering maximum use of the alloy. The other major method of powder preparation is argon atomization (AA) [9-11], which is characterized by high yield of fine powder, fine overall particle size of the powder, and relatively small overall size of inclusions; but its high oxygen content, the sphericity and surface finish are not as good as those of PREP powders [11], defect powder such as hollow powder is more than the PREP powder, but there is almost no hollow powder in the fine powder of less than 63 μm [9]. These factors will have a profound effect on the final comprehensive properties of the powder superalloy. However, there are few reports on the preparation of FGH97 alloys by direct hot isostatic pressing (HIP), using prealloyed powders prepared by AA method.

Based on these, the FGH97 superalloy powders were prepared by the PREP method and the AA method, and two nickel-based powder superalloys were prepared using the same hot isostatic pressing process and heat treatment process. Microstructure and mechanical properties were characterized; the influence of different atomization methods on the microstructure and low-cycle fatigue performance of FGH97 alloy formed by the direct hot isostatic pressing process route was mainly discussed, which provided a scientific basis for the preparation of FGH97 alloy using the AA+HIP process route.

2. Materials and Test Methods

Two kinds of Ni-based superalloy powders (P-powder and A-powder) were prepared from masteralloy melted via the process of vacuum induction melting (VIM) by plasma rotating electrode process (PREP) and argon atomization (AA) method, respectively. The two prepared powders were vacuum sieved (-75μm), canned, degassed, and sealed, and then the same hot isostatic pressing process and heat treatment process were used to obtain two alloy billets (P-alloy and A-alloy, respectively); the size of alloy billet is φ100×180 mm, the chemical composition of the two alloys (mass fraction, wt %) is shown in table 1. The hot isostatic pressing process is 1200±5 °C×145±5 MPa×3 hr; the heat treatment process is: 1200±10 °C×4 hr /AC + 910±10 °C×3 hr /AC + 750±10 °C×8 hr /AC + 700±10 °C×17 hr /AC. Testing the particle size distribution of the powder with Malvern MASTERSIZER 3000 laser diffraction particle size analyzer; the oxygen content of the powders and alloys was measured with LECO ONH 836 oxygen-nitrogen-hydrogen analyzer; the density of the same sample before and after heat treatment was measured five times using the Archimedes drainage method (equipment accuracy of 0.0001 g), the average is the density of the alloy. Microstructure and grain size of hot isostatically pressed billets were analyzed by optical microscopy and scanning electron microscopy; the size and volume fraction of the gamma prime phase (at a magnification factor of 10,000) and TIP (at a magnification factor of 200) were measured using IPP (Image Pro-Plus) image processing software. The metallographic analysis uses Kalling’s reagent (100 mL HCl, 100 mL ethanol, and 5 g CuCl2), while the gamma prime etchant uses HNO3: acetic acid: H2O: HF in a volume ratio of 3: 3: 3: 1.

In order to study the effect of atomization methods on the properties of FGH97 nickel-base superalloy, the tensile properties of the two alloys at room temperature, 650 °C and 750 °C were tested, and three tensile tests were performed at each temperature condition. The fracture locations were all within the gauge length of the test specimens. The low cycle fatigue properties of the two alloys were measured using INSTRON 8862 Low Cycle Fatigue Testers. The low cycle fatigue test was performed at the condition: temperature for 650 °C, stress range for 0-1010 MPa, stress ratio for R=0, frequency for 0.33 Hz, test wave for triangular. three fatigue tests were performed on P-alloy in this test, and five
fatigue tests were performed on A-alloy. Using the scanning electron microscope to observe fractures after performance testing.

Table 1. Chemical compositions of A-alloy and P-alloy (wt%).

|       | C   | O   | N   | Cr  | Mo  | W   | Ti  | Co  | Al  | Nb  | Hf  | Zr  | B   | Mg  | Ni  |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| A-alloy | 0.0330 | 0.0117 | 0.0010 | 8.97 | 3.92 | 5.59 | 1.78 | 15.52 | 5.16 | 2.69 | 0.26 | 0.0126 | 0.0104 | 0.0028 | Bal. |
| P-alloy | 0.0351 | 0.0066 | 0.0009 | 9.25 | 3.94 | 5.77 | 1.85 | 15.60 | 5.16 | 2.68 | 0.29 | 0.0122 | 0.0120 | 0.0023 | Bal. |

3. Test Results and Analysis

3.1. Powder Characteristic Analysis

Table 2 shows the particle size measurement results of the two powders (-75 μm). The results show that the P-powder has a narrow particle size distribution, a small span, and a more uniform particle size, but the overall size is coarser; while the A-powder has a wider particle size distribution, a large span. Compared to P-powder, the particle size of A-powder is more dispersed, the mount of fine powders is much more, and is much finer than the P-powder as a whole.

Table 2. Test results of powder particle size distribution.

| Powder | Dv(10) (μm) | Dv(50) (μm) | Dv(90) (μm) |
|--------|-------------|-------------|-------------|
| P-powder | 42.596 | 59.955 | 84.020 |
| A-powder | 11.648 | 33.641 | 73.613 |

Dv(x) represents the powder particle size corresponding to the cumulative volume percentage x.

Figure 1 shows the micromorphology of two alloy powders (-75 μm). As shown in figures 1a and 1b, the P-powder particle size is more uniform than the A-powder, and the A-powder has more fine powder, which is consistent with the particle size detection results; as shown in figures 1c and 1d, the sphericity and surface cleanliness of P-powder are better than that of A-powder, and there are a considerable amount of satellite powders, coated powders, hollow powders (as shown in figure 1f) and other defective powders in A-powder, while P-powder is relatively more less.

In addition, it can be seen from figures 1e and 1f that the solidification structure of P-powder is dominated by dendrites, and the solidification structure of the A-powder has a considerable number of cell crystals or even dominated by cell crystals. This is mainly due to the fact that the A-powder has a smaller particle size and the cooling solidification rate is faster.

The physical properties of the two powders were tested in this test. The results are shown in table 3. It can be found that the P-powder has good fluidity and the A powder has no fluidity. The factors affecting the fluidity of the powder are complicated, but the main influencing factors include powder particle size, powder sphericity and surface cleanliness. In general, the larger the particle size, the better the sphericity and surface cleanliness, the better the fluidity of the powder. Therefore, the fluidity of P-powder is better than that of A-powder, which also explains the apparent density and tap density of P-powder are higher than that of A-powder. Because the surface cleanliness of A-powder is worse than that of P-powder, the powder particle size is smaller, the specific surface area is larger, and the oxygen adsorption is more. Moreover, much more surface oxidation and pollution of A-powder can be seen from figures 1c and 1d, so the A-powder oxygen content higher than P-powder.
Figure 1. Micromorphology of two alloy powders after 200 mesh sieving: (a) (c) (e) P-powder, (b) (d) (f) A-powder.

Table 3. Testing results of the physical properties of the powders.

| Powder  | Fluidity (s/50g) | Apparent density (g/cm³) | Tap density (g/cm³) | Percentage of hollow powder (%) | Oxygen content (ppm) |
|---------|------------------|--------------------------|---------------------|-------------------|----------------------|
| P-powder | 13.2             | 4.81                     | 5.19                | 0.01               | 60                   |
| A-powder | No fluidity      | 4.16                     | 4.71                | 0.06               | 113                  |

3.2. Microstructure after Heat Treatment

3.2.1. Grain Size and Prior Particle Boundaries (PPBs). Figure 2 shows the microstructure of the P-alloy and the A-alloy after heat treatment. The average grain size of the P-alloy measured by the intercept method is 34.2 μm, which is equivalent to the grain size ASTM 6.5; while the average grain
The size of the A-alloy is 20 μm, which corresponds to the grain size ASTM 8; The grain size of A-alloy is significantly smaller than the P-alloy, see figures 2a and 2c. The P-alloy has an oxygen content of 66 ppm, a small amount of PPB, a relatively flat grain boundary (see figure 2a), and some residual dendrites apparently (see figure 2b); whereas the oxygen content of A-alloy is 117 ppm, there is a certain amount of PPB in the matrix and the grain boundary is more curved than the P-alloy (see figure 2c). This is mainly because the pinning effect of prior powder boundary particle (PPB) hinders grain boundary migration and growth, as well as straightening; at the same time, no residual dendrites were found and the microstructure was more uniform (as shown in figure 2d).

Figure 2. Grain structure and prior particle boundary (PPB) morphology of two alloys: (a) (b) P-alloy; (c) (d) A-alloy.

3.2.2. Carbides and Thermal Induced Porosities. Figure 3 is a backscattered image of the microstructure after heat treatment of P-alloy and A-alloy. According to different distribution locations, FGH97 powder superalloy carbides can be classified into grain boundary carbides (mainly $M_6C$ or $M_23C_6$) and intragranular carbides (mainly MC) [12-14]. The carbides of A-alloy is mainly composed of grain boundary carbides with less intragranular carbides, and grain boundary carbides are intermittently distributed along grain boundaries (see figure 3b), which is favorable for high temperature performance. Grain boundary carbides of P-alloy are distributed in a zigzag film, and there are a considerable number of point and massive intragranular carbides, which are about 0.5-2 μm in size, and are unevenly distributed and locally aggregated (as shown in figure 3a). The EDS-Map analysis of the intragranular carbides in the P-alloy found that these carbides are mainly rich in alloying elements such as Ti, Nb, Mo, Hf, and Zr, as shown in figures 4a-4f. In addition, as shown in figures 2h and 4g, the interdendritic carbides in the large-particle P-powder still exist after HIP and heat treatment, and EDS-Map analysis was carried out for its local area (see figure 4h) after amplification, it was found to be mainly rich in Nb (see figure 4i). This is mainly because the cooling solidification rate of the large-particle P-powder is slightly slower than that of A-powder in the atomization process, the microstructure is dominated by dendrites and Nb segregation occurs between the dendrites, while Nb is a high-melting, hard-to-diffuse element, so dendritic structure is retained after hot isostatic pressing and heat treatment; however, almost no such phenomenon is found in A-alloy.
Figure 3. Backscattered images of two alloys after heat treatment: (a) P-alloy; (b) A-alloy.

Figure 4. SEM image of (a) carbides in matrix and (g), (h) interdendritic carbides, (b) Ti, (c), (i) Nb, (d) Hf, (e) Mo, (f) C elements map scanning distributions og carbide for P-alloy.

Thermal induced porosity (TIP) can adversely affect the performance of the alloy. In this experiment, there are a certain number of hole defects in A-alloy, as shown in figure 3b; there is no hole defect in A-alloy and P-alloy before heat treatment. However, after the solution heat treatment and triple aging heat treatment, the thermal induced porosities are caused due to the heat, aggregation, and expansion of the inert gas in the A-alloy during the heat treatment. The TIP content of Alloy A is approximately 0.08%, as shown in table 4. The TIP size distribution of all these six regions is statistically analyzed, it is found that the average size of TIP is approximately 4 μm and the maximum size is ≤11 μm, as shown in figure 5.

Table 4. Thermal induced porosity (TIP) statistical results of A-alloy.

| Zone | 1   | 2   | 3   | 4   | 5   | 6   | Average | Standard deviation |
|------|-----|-----|-----|-----|-----|-----|---------|--------------------|
| TIP/ % | 0.06| 0.11| 0.07| 0.06| 0.06| 0.09| 0.08    | 0.02               |
Before heat treatment, P-alloy density $\rho_{P0}$ is 8.3169 g/cm$^3$ and A-alloy density $\rho_{A0}$ is 8.3168 g/cm$^3$; after heat treatment, P-alloy density $\rho_{P1}$ is 8.3167 g/cm$^3$ and A-alloy density $\rho_{A1}$ is 8.3103 g/cm$^3$. The results show that the density of P-alloy after heat treatment is slightly higher than that of A-alloy; the percentage reduction of A-alloy relative to P-alloy is calculated as 0.0769% according to equation (1), which is in good agreement with the TIP volume fraction of A-alloy. Therefore, it is considered that the density of A-alloy lower than that of P-alloy is due to the presence of a certain amount of TIP in the A-alloy. However, there is almost no difference in the density between A-alloy and P-alloy before the heat treatment, and there is almost no change in the density of the P-alloy before and after the heat treatment in this experiment.

\[
\Delta = \frac{\rho_{P1} - \rho_{A1}}{\rho_{P1}} \times 100\%
\]

where $\rho_{P1}$ is the density of P-alloy after heat treatment, $\rho_{A1}$ is the density of A-alloy after heat treatment, $\Delta$ is the percentage reduction of A-alloy relative to P-alloy.

3.2.3. $\gamma’$ Phase. Figure 6 shows the morphology and size distribution of the gamma prime of the two alloys after heat treatment. The FGH97 alloy is a gamma prime strengthening alloy and the complete dissolution temperature of the gamma prime phase is about 1185 degree centigrade [15]; therefore, the solution heat treatment in this experiment is performed in the gamma single phase region. It was found that the primary $\gamma’$ phases precipitated during the hot isostatic pressing of the two alloys were completely dissolved, and the $\gamma’$ phases of both alloys were dominated by the secondary $\gamma’$ phase, while a small amount of tertiary $\gamma’$ phases distributed between the secondary gamma prime particles, as shown in figures 6a and 6b. From the morphological analysis, the secondary gamma prime phase of P-alloy and A-alloy mainly existed in the shape of square or matts, as shown in figures 6c and 6d, and they were consistent with the research results of Zhang Ying et al. [12], and there was no significant difference in the two alloys. However, some intragranular secondary $\gamma’$ phase of P-alloy precipitates along the dendritic direction (as shown in figure 6a), this is mainly due to the slower solidification and cooling rate of large-size particles of P-powder in the atomization process than that of A-powder, the growth of casting dendrites is more complete. In the HIP process, the recrystallization is incomplete and the element diffusion is insufficient, so that some secondary $\gamma’$ phases in the alloy exhibit as-cast characteristics of the powder [16]. A-alloys, in contrast, has no obvious directionality in the precipitation of the $\gamma’$ phase in the alloy microstructure. This is mainly because the A-powder is finer overall, and the as-cast microstructure of the pre-alloyed powder is mainly composed of cell crystals with more uniform composition. During the hot isostatic pressing and heat treatment, the alloy elements diffused sufficiently and recrystallization proceeded more completely, so that the $\gamma’$ phase precipitated uniformly within the crystal (as shown in figure 6b).
Figure 6. Morphology of γ’ phase for two alloys: (a) (c) P-alloy; (b) (d) A-alloy.

Figure 7 shows the statistical distribution of the intragranular gamma prime phase of the two alloys. It can be found that the size of the intragranular gamma prime phase of the P-alloy is mainly distributed in the range of 100 nm to 450 nm, while that of the A-alloy is mainly in the range of 100 nm to 500 nm, the statistical analysis results are shown in table 5. After analysis of variance, it was found that the size of the intragranular γ’ phase of the P-alloy was basically the same as that of the A-alloy; the volume fraction of the intragranular γ’ phase of the P-alloy and the A-alloy was 62.27% and 60.79%, respectively; while the γ’ phase content of FGH97 alloy is generally about 60% [2,17], so the volume fractions of gamma prime phase of the two alloys are also nearly the same. The γ’ phase on the grain boundaries of the two alloys is mainly distributed in strips/blocks, and the size is about 0.3 μm to 1.5 μm.

Figure 7. Statistical size distribution diagram of γ’ phase for two alloys: (a) P-alloy; (b) A-alloy.

3.3. Tensile Properties

Figure 8 shows the variation trend of the tensile mechanical properties and test temperature of the two alloys. As shown in figure 8a, both the yield strength and tensile strength of the two alloys decrease with increasing temperature, and their variation trends are basically the same. The strength drops of the two alloys are relatively mild within the range of 25–650 °C, but when the test temperature exceeds 650 °C, the extent of decrease in strength is significantly larger; on the whole, the strength of
A-alloy is slightly higher than that of P-alloy. As shown in figure 8b, the elongation after fracture of A-alloy increases with temperature in the range of 25 to 650 °C, reaches a maximum of 29.7% at 650 °C, then decreases rapidly, and the elongation drops to 22.5% at 750 °C; while the elongation of P-alloy after fracture in the range of 25–750 °C shows a decreasing trend overall, but the change is not obvious, and the average elongation at the 650 °C and 750 °C conditions is 22.6% (Correspond to that of A-alloy at 750 °C). The area shrinkage (as shown in figure 8c) shows that the trend of the two alloys with temperature is similar to that of the elongation. Here, it is believed that, on the one hand, because the morphology of carbides of the two alloys is different, and the thermal expansion coefficient of the carbides and the matrix are different, the carbides in the P-alloy grain boundaries are distributed in a sheet shape, so making coordination in the tensile deformation process more difficult and crack more prone to initiation [18]; causes the plasticity of the P-alloy to decrease with increasing temperature. On the other hand, grain boundary carbides of A-alloy are discontinuously distributed in dots, grain size is smaller and the components are more uniform. Thus, they are more easily coordinated and deformed in the stretching process, and microcracks are less likely to be initiated. The strength and plasticity of A-alloy will be better.

Table 5. The statistical results of γ’ phase after heat treatment for two alloys.

| Alloy  | The average size of γ’ phase (nm) | The size standard deviation of γ’ phase (nm) | The γ’ phase volume fraction (%) |
|--------|-----------------------------------|---------------------------------------------|----------------------------------|
| P-alloy | 265                               | 104                                         | 62.27                            |
| A-alloy | 287                               | 107                                         | 60.79                            |

Figure 8. Relationship between yield strength: (a) ultimate tensile strength, (b) elongation, (c) area reduction and temperature.

3.4. Low-Cycle Fatigue Performance

Serving at high temperatures with anti-low cycle fatigue properties is one of the important characteristics of nickel-based powder superalloys. Tables 6 and 7 show the low cycle fatigue test results and fatigue source information for P-alloy and A-alloy, respectively. Comparing the low fatigue life $N_f$ values of the two alloys, the $N_f$ value of A-alloy is much higher than that of P-alloy; the highest low cycle fatigue life of P-alloy is 68517, and the lowest is 21115, the dispersion is relatively large.
However, the low cycle fatigue life of A-alloy is much higher than 150,000 cycles, and the test results are relatively stable. The fatigue sources of powder metallurgy superalloys are mainly divided into three types: platform (plastic slip damage), prior particle boundaries (PPB) and inclusions [19]. Their extent of damage to low-cycle fatigue performance of superalloys increases in turn. At the same time, the size, position and shape of inclusions have a direct effect on the low-cycle fatigue properties of superalloys. In this test, the SEM-EDS analysis of the fatigue sources of the fractures of the two alloys revealed that the fatigue cracking of the P-alloy source was caused by slag-type inclusions (see table 6); Among them, there were two inclusions in the fatigue crack initiation site of the P-1 sample, but small in size, as shown in figure 9a; P-3 specimen has an inclusion with a maximum size exceeding 200 μm, as shown in figure 9b; P-alloy fatigue crack initiation sites are all in the subsurface (fatigue source distance from surface <1 mm) of the specimens, and with the increase of the inclusion size, the lower the \( N_f \) value, the worse the anti-low cycle fatigue performance, which is consistent with the research results in reference [19]. In this experiment, the fatigue crack initiation of A-alloy was mainly caused by slag-type inclusions and \( \text{Al}_2\text{O}_3 \) ceramic inclusions (see table 7). The size of inclusions was small, and the maximum size was mainly distributed in the range of 40 μm-66 μm. The analysis, combined with \( N_f \) value, found that when the inclusion size is small (less than 45 μm in this experiment), the distribution location of inclusion has no obvious effect on the low-cycle fatigue performance of the superalloy. For example, the fatigue crack initiation sites of the samples A-2 and A-3 are all on the sample surface, the low-cycle fatigue life of the A-2 sample (see figure 9c) caused by the inclusion is comparable to that of the non-defective A-3 sample (see figure 9d), and even the former is higher than the latter. When the size of inclusions is larger (larger than 60 μm in this experiment), the location of the inclusions can significantly affect the low-cycle fatigue properties of the alloys, as shown in table 7, the size of the inclusions in Samples A-4 and A-5 is basically equivalent, but the low cycle fatigue life of A-5 sample, which the inclusions is in the subsurface, is significantly lower than that of A-4 sample which the inclusion is in the heart. In this experiment, no PPB and TIP were found in the low-cycle fatigue crack initiation sites and in the vicinity. Therefore, it is considered that the low-cycle fatigue performance of FGH97 alloy is not significantly affected by PPB and TIP in this test.

**Table 6.** Low cycle fatigue test results and fatigue source information of P-alloy.

| Sample NO. | \( N_f \) (650℃, 0.33 Hz, triangular wave) | Fatigue crack Size (μm²) | Number of the main Type of fatigue crack source elements of initiation |
|------------|--------------------------------------|--------------------------|-------------------------------------------------------------|
| P-1        | 68517                                | 572                      | 1 Al, O, Hf, Ca Slag-type inclusions                         |
| P-2        | 41980                                | 365                      | 1 Al, O, Ti, Nb Slag-type inclusions                         |
| P-3        | 21115                                | 782                      | 1 Al, O, Zr, Hf Slag-type inclusions                         |

**Table 7.** Low cycle fatigue test results and fatigue source information of A-alloy.

| Sample NO. | \( N_f \) (650℃, 0.33 Hz, triangular wave)* | Fatigue crack Size (μm) | Number of the main Type of fatigue crack source elements of initiation |
|------------|-------------------------------------------|--------------------------|-------------------------------------------------------------|
| A-1        | 173046                                    | 90                       | 1 Al, O, Ca, Ti, Hf Slag-type inclusions                     |
| A-2        | 153184                                    | 0 (Surface)              | 1 Al, O, Cr, Ni, Ti Slag-type inclusions                     |
| A-3        | 152507                                    | 0 (Surface)              | / platform                                                  |
| A-4        | 182804                                    | 1158                     | 1 Al, O ceramic inclusions                                  |
| A-5        | 166114                                    | 467                      | 1 Al, O, Ti, Zr, Hf Slag-type inclusions                     |

* Note: after the fatigue cycle number of A-alloy reached about 150,000 cycles during the low cycle fatigue test,
the stress range was modified to 0-1127 MPa (namely the maximum stress increased by 15%) while other test conditions remained unchanged.

Figure 9. The typical failure origin morphology of two alloys with low cycle fatigue fracture: (a) (b) P-alloy; (c) (d) A-alloy.

4. Discussion

The inclusions in the nickel-based superalloy powders are mainly classified into ceramic (external inclusions), slag (endogenous inclusions), organic inclusions, powder adhesion inclusions or abnormal particles formed by sublimate according to the production mechanism and source; and the mount of inclusions, which size is larger than 45 μm, is positively correlated with the amount of powders in each particle size range, that is, the inclusion size varies with the particle size [20]. As mentioned above (as shown in table 2, figures 1a and 1b), in this paper, the particle size Dv (90) of the P-powder is 84 μm, and is more uniform and larger than A-powder as a whole, so the size of inclusions in P-powder will also be larger. As shown in figures 10a and 10b, the size of the inclusions in the hot isostatically press forming P-alloy matrix is large, and the size of the inclusions observed under the metallographic conditions reaches 80 to 110 μm. In comparison with the P-powder, in this experiment, the amount of fine powder of A-powder is much more, and the overall particle size is finer, and the size of inclusions is also reduced. As shown in figures 10c and 10d, the size of the inclusions in the A-alloy matrix is controlled to be small, and the maximum size of the inclusions found under the metallographic microscopic conditions is substantially less than 50 μm, and almost no large-sized inclusions are found. These results are consistent with the inclusion size levels of the fatigue fractures (as shown in tables 6 and 7). In addition, the screening process in this paper can remove most of the large-sized inclusions and some small-sized inclusions, and to further control the inclusions. The electrostatic separation and inclusion process can effectively reduce the amount of inclusions, but the effect on decreasing the size of the inclusions is not obvious [6, 20]; as a result, the size of the inclusions in powder alloys depends on the particle size and the inclusion size distribution in the original powder which determined by atomization method. Since the powder size and the inclusion size in the A-powder are less than the P-powder as a whole, it can be considered that the gas atomization method is more advantageous for the control of the inclusion size.
Figure 10. The typical inclusion morphology of two alloys after heat treatment: (a) (b) P-alloy, (c) (d) A-alloy.

The low-cycle fatigue failure process of powder superalloys mainly includes three stages [21]: microcrack initiation (including microcrack growth), crack stable growth, and unstable fracture. The total fatigue life is

\[ N_f = N_i + N_p \] (2)

where \( N_f \) is the total fatigue life, \( N_i \) is the crack initiation life, and \( N_p \) is the crack propagation life.

The equation (2) shows that the fatigue life of powder superalloy is mainly composed of crack initiation life and crack propagation life. The fatigue crack initiation life of the powder-metallurgy superalloy FGH97 accounts for more than 80% of the entire life at 650 °C [22]. Once in the crack stable growth stage, the crack growth rate increases and the material quickly fails. Therefore, the total fatigue life of P-alloy and A-alloy is mainly determined by the crack initiation life \( N_i \). At the same time, all factors that affect the crack initiation and crack propagation of the alloy will have an important influence on the fatigue performance of the alloy.

Grain size is one of the important factors affecting the initiation and propagation rate of low-cycle fatigue cracks in superalloys. The effect of grain size on the fatigue cracking of FGH97 alloys occurs throughout the low-cycle fatigue fracture process. Because the alloy exerts stress concentration near the grain boundary due to dislocation accumulation under the tensile stress, when the stress exceeds a certain value, the interatomic bond near the dislocation plug is destroyed, thereby causing crack initiation. According to the Zener-Stroh theory based on the dislocation-sampling model, the grain size of the A-alloy is finer and has higher strength (see figure 8a), which hinders the dislocation motion caused by cyclic stress during the crack initiation stage. The critical shear stress of the crack nucleation is larger, and the crack is difficult to be nucleated, thereby increasing the crack initiation period of A-alloy [23, 24]. In addition, superalloys have a high degree of alloying and low stacking...
fault energy. The smaller the grain size, the greater the resistance to cracking in the first stage and the higher the cyclic time in the first stage. Therefore, the $N_i$ value of the fine-grained A-alloy is higher than that of the coarser-grained P-alloy. However, in the stage of crack stable growth, the large grains can promote the characteristics of the planar slip of cyclic slips and hinder the propagation of cracks. Under the action of cyclic stress, the roughness of the fracture surface is high, causing cracks to close early, resulting in a decrease in the rate of crack propagation. At the same time, the fine-grained material has more grain boundaries exposed to the air, accelerates the diffusion of oxygen along the grain boundaries under stress, aggravates grain boundary oxidation, weakens the grain boundary, and increases fatigue crack growth rate [25]. Since the P-alloy grain size is larger than that of the A-alloy, the P-alloy’s crack growth rate may be lower than that of the A-alloy during the fatigue crack propagation stage. In other words, the argon gas atomization process can be used to refine the grain structure of A-alloy and increase its fatigue crack initiation resistance. The grain size of P-alloy obtained by the PREP atomization method is coarser and reduces the fatigue crack initiation resistance; it maybe increase its resistance of fatigue crack stable growth. As the crack growth rate is accelerated when the fatigue process enters the stable growth stage, and then the material will soon fail. Therefore, the $N_p$ value of the crack propagation life is small in the total life, and the low cycle fatigue performance is mainly determined by the crack initiation life. This to some extent explains that A-alloy has better low cycle fatigue performance than P-alloy.

Inclusions are one of the three major defects of powder superalloys. Due to the destruction of the continuity of alloy matrix materials causes stress concentration, which does great harm to the mechanical properties of powder superalloys, especially low-cycle fatigue properties, and often results in early failure of superalloy components. From tables 6 and 7, it can be seen that the fatigue crack sources of the two alloys are mainly caused by slag inclusions composed of elements such as Al and O. These inclusions have different linear expansion coefficients and elastic moduli with the alloy matrix. Under the effect of heating and external cyclic load, the inclusions will generate a large stress field or uneven deformation caused by stress concentration; as a result, the interface between the inclusion and the alloy matrix separates or the inclusion itself cracks. When the size of inclusions is greater than a certain value, in this experiment, a crack source will be formed; the larger the size of inclusions, the greater the tendency of crack sources to form, and the weaker the crack initiation resistance [26-28]. Since the maximum size of inclusions in P-alloy specimens is 2 to 3 times that of A-alloy specimens, it can be inferred that the fatigue crack initiation life $N_i$ of P-alloy with PREP is much lower than that of A-alloy with AA atomization method.

In the FGH97 alloy, the properly sized and uniformly arranged secondary $\gamma'$ phase is accompanied by a small tertiary $\gamma'$ phase, which can relieve the stress concentration at the crack tip, greatly increase the fatigue crack initiation resistance, and reduce the fatigue crack propagation rate [22]; as a result, greatly improve the alloy’s low-cycle fatigue performance. Due to the presence of some residual dendrites in the P-alloy (figures 2b and 4g), some of the secondary $\gamma'$-phases are directional (see figure 6a), which also affects the low cycle fatigue and other mechanical properties of P-alloy. In addition, the amount of precipitation of grain boundary carbides in P-alloy is more than A-alloy, and is distributed in a continuous network, which will accelerate the rate of fatigue crack growth [29].

5. Conclusion

(1) In this experiment, the particle size distribution of P-powder is narrower than that of A powder, and the overall particle size is larger, but the surface cleanliness and sphericity of P-powder are better, and the amount of defective powders is less than A-powder. The fluidity of P-powder is obviously better than A-powder, and its apparent density and tap density are higher than A-powder; the oxygen content of P-powder is 60 ppm, but A-powder is 113 ppm; The inclusions are mainly dominated by slag type for two powders, and the size of the inclusions in the A-powder is much smaller than P-powder.

(2) The oxygen contents of P-alloy and A-alloy are 66 ppm and 117 ppm, respectively; the grain sizes of P-alloy and A-alloy are 34.2 $\mu$m and 20 $\mu$m, respectively; the grain sizes are equivalent to ASTM 6.5 and ASTM 8, respectively; The $\gamma'$ phase volume fractions were 62.27% and 60.79%,
respectively, while their $\gamma'$ phase average sizes were 265 nm and 287 nm, respectively. The morphologies were all in the form of squares or “matts” shapes.

3) In this experiment, slag-type inclusions and ceramic-type inclusions were the main sources of fatigue cracks in the fatigue failure process of P-alloy and A-alloy, and the size of fatigue fracture inclusions in A-alloys was much smaller than that of P-alloys.

4) Fatigue crack initiation resistance of A-alloy is better in this experiment, and its fatigue life is much higher than that of P-alloy. The low cycle fatigue life $N_f$ value of A-alloy is far more than 150,000 cycles, while the maximum value of P-alloy is not more than 70,000 cycles, and the fatigue property of A-alloy is very stable.

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References
[1] Liu H, Bao R, Yue C Y, Fei B J 2016 Predominant factor related to stress intensity factor range on creep-fatigue crack growth of nickel-superalloy FGH97 Journal of Aerospace Power 31 (6) 1400
[2] Zhang Y, Zhang Y W, Zhang N, Jia J 2008 Heat treatment processes and microstructure and properties research on P/M superalloy FGH97 Journal of Aeronautical Materials 28 (6) 5
[3] Liu J T, Zhang Y W, Tao Y, Zhang Y, Zhang G X, Chi Y 2011 Microstructure of FGH97 P/M superalloy Transactions of Materials and Heat Treatment 32 (3) 47
[4] Shi C X, Zhong Z Y 2006 Fifty Years of Superalloy in China. Beijing: Metallurgical Industry Press 113
[5] Shi C X, Zhong Z Y 2010 Development and innovation of superalloy in china Acta Metallurgica Sinica 46 (11) 1281
[6] Guo W M, Wu J T, Zhang F G, Chen G S 2000 Inclusion in powder of nickel base superalloy FGH95 and the effect on LCF of the superalloy Powder Metallurgy Industry 10 (3) 23
[7] Han Z Y, Zeng G, Liang S J, Chen X L, Zhang P, Zhang P X 2014 Development in powder production technology of Ni-based superalloy Materials China 33 (12) 748
[8] Zhang Y, Zhang Y W, Zhang N, Liu M D, Liu J T 2010 Fracture character of low cycle fatigue of P/M superalloy FGH97 Acta Metallurgica Sinica 46 (4) 444
[9] Yuan H, Li Z, Xu W Y, Zhang G Q 2010 The study of argon atomized superalloy powder Powder Metallurgy Industry 20 (4) 1
[10] Hu B F, Zhang S H 1997 Study on a Nickel Base Superalloy FGH95 of Turbine Disc Transactions of Materials and Heat Treatment (3) 28
[11] Zhang Y W, Shangguan Y H 2004 Research and development in P/M superalloy Powder Metallurgy Industry 14 (6) 30
[12] Zhang Y, Zhang Y W, Zhang N, Jia J 2008 Heat treatment processes and microstructure and properties research on P/M superalloy FGH97 Journal of Aeronautical Materials 28 (6) 8
[13] Tan L M, Zhang Y W, Jia J, Liu J T 2016 Strengthening design of nickel based powder metallurgy superalloy FGH97 Transactions of Materials and Heat Treatment 37 (4) 5
[14] Gao Z J, Zhang G Q, Li Z, Yuan H, Xu W Y, Liu N 2012 Effect of Size Distribution and Oxygen Content of Powder on Microstructure of HIPed Superalloy FGH96 Chinese Journal of Rare Metal 36 (4) 665
[15] Zhang Y, Zhang Y W, Tao Y 2006 Investigation on Russian P/M superalloy EP741NP Journal of Iron and Steel Research 18 (8) 62
[16] JIA J, Tao Y, Zhang Y W, Zhang Y 2010 Effect of aging heat treatment on microstructure and mechanical properties of PM superalloy FGH95 Powder Metallurgy Industry 20 (1) 29
[17] Zhang Y, Zhang Y W, Tao Y 2006 Investigation on Russian P/M superalloy EP741NP Journal of Iron and Steel Research 18 (8) 62
[18] Ma W B, Liu G Q, Hu B F, Hu P H, Zhang Y W, Liu J T 2014 Effect of carbon on carbides in HIPed FGH96 Alloy Rare Metal Materials and Engineering 43 (1) 109
[19] Zhang Y, Zhang Y W, Zhang N, Liu M D, Liu J T 2010 Fracture character of low cycle fatigue of P/M superalloy FGH97 Acta Metallurgica Sinica 46 (4) 444
[20] Zhang Y, Zhang Y W, LÜ R H, Huang H B, Zhang G X, Han S B, Liu M D, Sun Z K 2017 Inclusions in Ni-base superalloy powder by PREP The Chinese Journal of Nonferrous Metals 27 (10) 2037
[21] Cai Z G, Liu Y K, Wang C Z, Zheng W L 1985 Metal Wear and Rupture Shanghai: Shanghai Jiaotong University Press 227
[22] Nai Q L, Dong J X, Hang M C, Zheng L, Yao Z H 2016 Fatigue behavior of powder metallurgy superalloy FGH97 Chinese Journal of Engineering 38 (2) 248
[23] Xu J, Zhang G P 2014 Effects of grain size and initial immobile dislocation density on fatigue behavior of polycrystalline metals Materials Science & Engineering A 590 (1) 194
[24] Weertman J 1986 Zener-Stroh crack, Zener-Hollomon parameter, and other topics Journal of Applied Physics 60 (6) 1877
[25] Yang J, Dong J, Zhang M, Yu T 2013 High temperature fatigue crack growth behavior of a novel powder metallurgy superalloy FGH98 Acta Metallurgica Sinica 49 (1) 71
[26] Zeng Y P, Zhang M C, Dong J X, Zhang L N, Xie X S 2005 Study on crack Initiation and propagation induced by inclusion in nickel-base P/M superalloy Journal of Materials Engineering (3) 10
[27] Guo W B 2008 Inclusion influence on fatigue property of P/M superalloy PTCA(PART: A PHYS. TEST.) 44 (5) 16
[28] Zhou X M 2006 Genetic Characteristic and Damage Mechanical Behavior of Non-metallic Inclusions in P/M Superalloy Beijing: Beijing Institute of Aeronautical Materials 58
[29] Wang P, Dong J X, Zhang Y W, Xie X S 2010 Influence of heat treatments on crack propagation rates of FGH96 P/M superalloys Rare Metal Materials and Engineering 39 (1) 157