Effect of powders on microstructure and performance of inconel 718 alloy prepared by SPS

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Abstract

In this paper, 15–53 \(\mu\)m spherical powders obtained by plasma rotating electrode (PREP) and argon atomization (VIGA) were used to prepare the Inconel 718 alloy through spark plasma sintering (SPS), and then the sintered alloy was treated by solid solution and aging heat treatment. The effect of different powders on the microstructure and properties of Inconel 718 alloy as sintered and heat treated was studied. The results show that high density Inconel 718 alloy can be obtained by SPS technology, and the original particle boundary of powder can be reduced, and the original particle boundary of PREP is less. According to the densification process of spherical powder during SPS process, the change law of powder microstructure in rapid sintering is established.

1. Introduction

Inconel 718 alloy with FCC crystal structure is widely used in aerospace, automobiles, chemicals, marine engineering, industry and other fields owing to high strength, good thermal stability, excellent antioxidant, and strong corrosion resistance [1–4]. Fine casting and forging are commonly used processes to prepare Inconel 718 alloy at present. However, when casting method is adopted, the strengthening elements are easy to cause serious dendrite segregation, coarse grains, loose shrinkage cavities and other defects, and the post-treatment of parts is cumbersome. Moreover, Inconel 718 alloy parts produced by forging method are prone to defects such as white spots caused by element segregation, which leads to difficulties in forming parts with complex shapes. In recent years, advanced manufacturing technologies such as hot isostatic pressing (HIP) [5], additive manufacturing (AM) [6–9], spark plasma sintering (SPS) [10, 11], etc. have been used to prepare Inconel 718 alloy parts. Chang LT et al. [12] reported the preparation of Inconel 718 alloy using HIP technology. The tensile properties of 0.2% yield strength, ultimate tensile strengths, elongation at 650 °C were 799.1 MPa, 1041.8 MPa and 19.3%, respectively, which were equivalent to the alloy prepared by forging. DGu et al. [13] adopted the selection of laser molding to prepare the Inconel 718 alloy. This study found that with the increase in the grain of the molding energy, the surface gap decreased, where the density of the molding alloy sample increased. Moreover, its high temperature oxidation resistance was also improved. The results showed that optimally prepared fully dense Inconel 718 parts had a uniform microhardness distribution with a mean value of 395.8 HV\(_{0.2}\), a considerably low friction coefficient of 0.36 and a reduced wear rate of 4.64 \(\times\) \(10^{-4}\) mm\(^3\)/N m in sliding wear tests.

SPS technology is a fast, energy-saving and environment-friendly new technology for material preparation and processing. It shows great advantages in the preparation of nano materials, composites and other applications. Researchers have made a lot of exploration on its mechanism. The analytical model has been successfully applied to SPS which was related the densification mechanisms to power-law creep by Gendre [14]. Based on the experimental data of spark plasma sintering of ZrO\(_2\) powder, this model has been improved [15, 16]. The densification mechanism for hot pressing (HP) and spark plasma sintering AlCuFeB powder was successfully determined by the improved model [17]. SPS can realize low-temperature, short-time high-density
with controllable cooling rate. Besides the sintering technique, the characteristics of the initial powder such as particle size, purity and void ratio also affect properties of the fabricated samples. Shuaijiang Yan et al [18] prepared the Inconel 718 alloy in the SPS condition using the PREP powder (53–106 μm) as the raw material. The maximum compressive strain is 79% and the yield strength reaches 1349 MPa after aging heat treatment. So far, there are few studies on the preparation of Inconel 718 alloy by SPS with spherical fine powder as raw material. At the same time, the influence of powder preparation method on the microstructure and properties of the alloy is not clear. In this work, plasma rotating electrode and argon atomization method were used to prepare 15–53 μm spherical powder as raw material, the sintered alloy was processed through SPS, which was treated by solid solution and aging heat treatment. On the one hand, the fine particle size of the 15–53 μm powder can promote the sintering performance of SPS. On the other hand, it can also be too fine to avoid defects caused by high surface oxygen content. The effects of different powders on the microstructure and properties of Inconel 718 alloy as sintered and heat treated were studied. The densification process of spherical powder during SPS was analyzed, and the change law of powder microstructure during rapid sintering was established. Through this paper, the influence of fine powder (15–53 μm) and milling method on the properties of the Inconel 718 alloy can be inferred, which will provide experimental data and theoretical support for the subsequent forming of Inconel 718 alloy and its components.

2. Experimental method

The same batch of rods were used to prepare the Inconel 718 alloy spherical powder by plasma rotation electrode (PREP, P powder) and gas atomization method (VIGA, A powder), respectively, and then 15–53 μm powders were obtained by classified the granularity of sieving machine. The oxygen content of P powder is 70 ppm and the carbon content is 240 ppm, while the oxygen content of A powder is 324 ppm, and the carbon content is 230 ppm. Then using the SPS process to sinter the raw material of 15–53 μm powder at the temperature of 1150 °C and the heating rate of 100 °C min−1, plus thermal insulating for 10 min under the pressure of 50 MPa, followed by furnace cooling to room temperature. The sintering process is shown in figure 1. The sintered samples were treated by solid solution + aging heat treatment, in which the solid solution temperature was 980 °C and the holding time was 1 h, and then air cooled to room temperature. The aging temperature was 720 °C with holding time 8 h, and then the temperature was reduced to 620 °C at the rate of 50 °C h−1, and the furnace was cooled out after the holding time 8 h.

The density of the alloy was measured by Archimedes drainage method, and the microhardness was measured by microhardness instrument. The detection method of element C is high-frequency combustion infrared absorption method (CS844, LECO, American). The detection method of element O is inert gas fusion method (ONH836, LECO, American). XRD patterns of the powders and alloy were got on a Japan Rigaku D/Max-III C diffractometer (40 kV, 40 mA, Cu Kα radiation (λ = 1.5406 Å)), employing a scanning rate of 4°min−1 in the 2θ ranging from 20° to 80°. JSM-6700F field emission scanning electron microscope was used for SEM. Samples of Φ 6 × 10 mm for compression experiments were prepared by an electrical glorious cutting machine and the compression rate is 0.1 mm s−1.
3. Experimental results

3.1. Characterization of Inconel 718 alloy powder

Figure 2 shows the particle size distribution curves corresponding to the two powder methods. It can be seen that for particle size range of 15–53 μm, the yield distribution curves of the two methods satisfied the lognormal distribution, but the average particle size of A powder is 20–25 μm, while the average particle size of P powder is 33–37 μm. Obviously, A powder is finer.

Figure 3 presents the surface morphology and cross-section photos corresponding to the two milling methods. The powders are regular spherical. However, the sphericity of P powder is significantly higher than that of A powder. The surface of A powder is adhered by satellite powder, while surface of P powder is smooth. The surface and cross section of the powder corresponding to two methods are in dendrite structure. Moreover, because the alumina crucible is used in the production of A powder, fine alumina ceramic inclusions are found.
in the powder, while no ceramics and other inclusions are found in P powder. By testing the content of hollow powder through Micro-CT, it can be observed that there was a small amount of hollow powders in A powders, accounting for 0.01%, while no hollow powder was found in P powder. The appearance of A powder hollow core powder is mainly caused by the following facts: (i) the droplet was immersed in the atomized gas during the physicochemical process; (ii) the flight process, or the droplet dissolves the gas during the cooling process, but the gas is not discharged and gathered during the solidification process. At the same time, during the process of powder metallurgy, especially in the process of hot pressing, hot isostatic pressing or plasma sintering, the cavity volume of hollow powders would be rapidly reduced under pressing condition. However, during the process of alloy post-treatment, for lack of external pressure, the powder expands under heating condition, resulting in thermally induced holes in the material and reducing the microstructure and properties of the final components [19, 20]. The above research reveals that the hollow powder proportion of Inconel 718 alloy powder prepared by the two methods is very low, thereby not having a great impact on the microstructure and properties of subsequent alloy samples.

The EDS analysis of the cross section of Inconel 718 alloy spherical powder is shown in figure 4. The cross section is in dendritic shape, which is related to the micro zone segregation during solidification due to the different solidification distribution coefficients of different elements. Because of the fast cooling structure of the powder, the distance between such dendrite segregation is very small, basically less than 3 μm. Through energy spectrum analysis, it is found that there was component segregation between the branches. The contents of Nb, Ti and Mo in the branches and stems are high, as well as the contents of Cr, Fe and Ni in the branches. This is a typical phenomenon of micro segregation and dendrite growth during the solidification process of superalloys.

The XPS analysis on the surface of the Inconel 718 alloy spherical powder is presented in figure 5. It is worth noting that the metal elements Ni, Fe, Cr, Ti, and Nb, and non-metallic elements O, C were found in the samples. The corresponding combination can be different while there are differences in the content of the
The content of O and C in A powder reached 40.03 at.% and 44.63 at.% respectively. It is higher than that of O and C in P powder of 36.46 at.% and 42.17 at.%. Through further analysis of its combination, it is found that the content of O for A powder and P powder surfaces is mainly based on free state, and there are still a small amount of $\text{–1}$ and $\text{–2}$ oxides. Meanwhile, there are also a small amount of MC carbides, which is related to the larger specific surface area caused by the finer particle size of a powder. The existence of these oxides and carbides is the main factor for the formation of the original particle boundary of the powder in the sintering process.

3.2. Microstructure of Inconel 718 alloy

Figure 6 shows a microstrip organization of the SPS state alloy. It can be seen from figure 6 that the powder corresponding to the two different methods has reached a good density, with no pore residues found. At the mean time, a small amount of pink original granules are observed, and the larger particles of size remain close to the outline of the spherical shape. The existence of large particles is related to the deformation during the density process. Plastic flowing and creep supplies the main derivation mechanism during the SPS sintering process. Among them, the creep mechanism controlled by the Inconel 718 alloy is the main factor. As the vibration and relative density of spherical powder reached 65%, there are particles of different sizes in the powder. Compared with the powder of large particles, small particles are subject to greater contact stress during the deformation process, which will produce greater than deformation and filling in the middle of the pores of large particles and small particles during the deformation process. Because of the shear effect, the destructive surface as fresh surface is not conducive to other phase precipitation and grown. Large particles are smaller due to less deformation. The appearance is more likely to be retained. From figure 6(a), it can be seen that there are many small particles in A powder. Because the small particles have deformed more after sintering, the size of the particles is significantly larger, which is close to the size of the large particles. For P powder, it can be seen from figure 6(b) that basically the change is not much, which has a lot to do with the thickness of the powder particles.

In the microstructure of Inconel 718 alloy in SPS state, a small amount of precipitates appear at the boundary. After solid solution and aging, there are obvious changes in the microstructure of the alloy corresponding to the powder of the two different methods. The precipitates at the alloy grain boundary corresponding to A powder are significantly increased, and a large number of precipitates also appear in the grain. However, the precipitates of the alloy corresponding to P powder are mainly concentrated at the grain boundary. Compared to that in SPS state, The size of acicular precipitates is obviously larger. The structure evolution diagram during the SPS and heat treatment for A powder and P powder can be seen in figure 7. In order to further clarify the composition distribution of the precipitated phase, EDS line scanning analysis was carried out, and the results are demonstrated in figure 8.

From the line scanning results, the precipitates of Inconel 718 alloy corresponding to A powder are apparently different from the matrix in C, Mo, Nb, Ti and other elements. The precipitates of Inconel 718 alloy corresponding to P powder are obviously different from the matrix in C, Nb, Al, Ti and other elements. In other words, MC carbides containing Nb and Ti elements are more likely to occur at the branch segregation. The acicular phase in the grain is $\delta$ phase, because the precipitated phase with similar composition of only $\gamma''$, $\gamma'$ and $\delta$ phase. Though $\gamma''$, $\gamma'$ are the main strengthening phase of the alloy, they are also metastable phases. When the size increases to a certain extent, it loses the lattice with the matrix and turns into a stable phase with an oblique structure $\delta$ phase.

Figure 9 presents the TEM analysis of Inconel 718 alloy after solution aging. After heat treatment, strengthening phases $\gamma'$ and $\gamma''$ are found in the corresponding alloys of A and P powder. As shown in figures 9(b) and (d), this is because the presence of Nb in nickel base alloys produces $\gamma''$-Ni$_3$Nb, which forms a...
Figure 6. SEM photos of SPS and solution aging (a: A powders SPS, a1: A powders SPS + SA, b: P powders SPS, b1: P powders SPS + SA).

Figure 7. The structure evolution diagram during the SPS and heat treatment for A powder and P powder.
coherent phase with the austenite matrix. At the same time, acicular \(\delta\) phase, as shown in figure 8(c), which is consistent with the results of SEM. In addition, MC type carbides or M\(_{23}C_6\) type carbides are found in the alloys. In addition, \(\text{Al}_2\text{O}_3\) and \(\text{TiO}_2\) particles were also found on MC carbides in the alloys corresponding to A powder with high C and O contents, because Ti and C of the powder migrate to specific oxides on the surface of the particles during heat treatment, thus forming stable titanium oxide carbides along the PPB. The existence of phase of strip carbides and acicular carbides \(\delta\) can reduce the properties of Inconel 718 alloy.

The x-ray diffraction patterns of Inconel 718 powders and heat-treated alloys are presented in figure 10. The diffraction peaks of \(\gamma\), \(\gamma'\), and \(\gamma''\) phases could hardly be distinguished because the positions of diffraction peaks on the \((111)\), \((200)\), and \((220)\) planes of \(\gamma/\gamma'\) phases are close to those on the \((112)\), \((004)\), and \((220)\) planes of \(\gamma''\). It should be noticed that the \(\gamma, \gamma',\) and \(\gamma''\) phases are the main phases for powders. When the alloy was heat-treated the \(\delta\) phase and MC type carbides are detected which is consistent with TEM analysis.

Figure 11(a) shows the compressive stress-strain curve of Inconel 718 alloy before and after heat treatment. Figure 11(b) displays the compressive yield strength and compressive strength. Because of the excellent compression plasticity of Inconel 718 alloy prepared by SPS, there is no obvious compression failure. However, a plateau stage is found at the place with large deformation on the stress-strain curve. That is, at the stage when cracks occur and expand on the sample surface, the corresponding stress value is the compressive strength of Inconel 718 alloy. It is found that the compressive yield strength of the alloy corresponding to A powder is significantly higher than that of the alloy corresponding to P powder. After heat treatment, the yield strength of the two alloys increases by more than 2 times, and the compressive yield strength and compressive strength of P powder are higher than those of A powder, which indicates that the plasticity of P powder corresponding alloy is better than that of A powder after heat treatment.

**Figure 8.** EDS analysis of Inconel 718 alloy after solution and aging (a: A powders SPS + SA, b: P powders SPS + SA).
Figure 9. TEM images of Inconel 718 alloy after solution and aging (a,b: A powders SPS + SA; c,d: P powders SPS + SA).

Figure 10. The x-ray diffraction patterns of Inconel 718 powders and alloys (a: powders, b: heat-treated alloy).
4. Discussion

From the analysis listed above, it is found that particle discharge, conductive heating and pressurization are the three main characteristics of SPS process. Heating and pressurization are the traditional features of promoting sintering. The plasmons generated by discharge between powder particles have very high temperature, which can produce local high temperature on the powder surface, causing local melting of particles and peeling off of surface oxides/carbides, so that the powder particles can be purified at the initial stage of sintering. With the combined action of applied pulse current and pressure, the volume diffusion and grain boundary diffusion of grains are strengthened, the densification speed of the material is accelerated [21].

Figure 11. a: Compressive stress-strain curve of Inconel 718 alloy before and after heat treatment; b: Compressive yield strength and compressive strength.

Figure 12. Morphology of Inconel 718 SPS at different stages during sintering a:1050 °C; b:1150 °C for 2 min; c,d: 1150 °C for 10 min.
Figure 12 shows the morphology of sintered samples obtained at different stages. At the initial stage of sintering, the local high temperature of the powder causes evaporation and melting on the surface of Inconel 718 powder particles, and the evaporated substances deposit near the particle contact point to form a sintering neck. As shown in figure 12(a), most of the particles have formed a sintering neck at the initial stage of sintering. With the further increase of sintering temperature and pressure, the densification of Inconel 718 powder is further improved. After 2 min of insulation, the density has reached more than 90%. As shown in figure 10(b), the sintered holes are mainly concentrated at the boundary of the particles. With the extension of the holding time, the powder pores gradually disappear. It is difficult to find holes after holding for 10 min.

5. Conclusion

(1) In the microstructure of Inconel 718 alloy in SPS state, the microstructure of A powder is similar to that of P powder. But after solid solution and aging, the precipitates at the alloy grain boundary corresponding to A powder significantly increased, and a large number of precipitates appear in the grains, while the precipitates of the alloy corresponding to P powder are mainly concentrated at the grain boundary, and the size of acicular precipitates is significantly larger.

(2) The compressive yield strength of Inconel 718 alloy in SPS state corresponding to A powder is significantly higher than that corresponding to P powder. However, after heat treatment, the yield strength of both alloys increases by more than 2 times, and the compressive yield strength and compressive strength of P powder are higher than those of A powder.

(3) At the initial stage of SPS, Inconel 718 powder is partially in high-temperature, resulting in evaporation and melting on the surface of powder particles. The evaporated materials deposit near the particle contact point to form a sintering neck, thus reducing the original particle boundary of the powder.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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