

# PROGRESS OF RESEARCH ON P/M AND SPRAY-FORMED SUPERALLOY IN ISCPM, USTB

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## Abstract

For meeting the demands of P/M disks, ISCPM began to research work on P/M superalloys at the beginning of 21<sup>st</sup> century. The study mainly can be divided into three parts: 1. Systematic engineering research on improving argon atomization process, including refractory improvement and technological parameters optimization, and powder metallurgy route; 2. Research on spark plasma discharge (SPD) process, which is an innovative technology for making ultra-fine and clean superalloy powder; 3. Research on spray forming. We use spray-forming technology to make the FGH4095 disk alloy. The nitrogen spray-formed superalloy after HIP, isothermal forging and heat treatment, shows encouraging properties, which shows the potential for making high performance superalloy disks.

## Introduction

Research on P/M superalloys in China has made significant progress especially in recent years<sup>[1-9]</sup>. China-made P/M disks are being considered for application in a few types of airplane engines. But until now the majority of the P/M turbine disks were imported from Russia, which are afforded as components of imported high pressure gas turbines. With the purpose of reducing the dependence on imported P/M turbine disks in China, at the beginning of 21st century, our group began to research work on P/M superalloys<sup>[10,11]</sup>.

On the basis of understanding from long-term research on P/M and spray-formed high speed steels with nitrogen atomization, hot extrusion, isothermal forging and HIP, and in the absence of funding from national projects on P/M superalloy, ISCPM established cooperation with large national companies – Fu Shun Special Steel Shares Co., Ltd and Guizhou Anda Aviation Forging Co., Ltd and the group of Professor Udo Fritsching and Dr. Volker Uhlenwinkel, IWT University Bremen. With the help of these companies and research groups, ISCPM is making progress on P/M and spray-formed superalloys in following aspects. The superalloy chosen for this research is a first generation P/M superalloy with high strength - FGH4095, which is equivalent to René95. The designed chemical composition of FGH4095 is shown in Table 1.

Table 1. The designed chemical compositions of FGH4095

Element	Ti	Al	Cr	Co	W	Mo
Content (wt.%)	2.3~ 2.7	3.3~ 3.7	12.0~ 14.0	7.0~ 9.0	3.3~ 3.7	3.3~ 3.7
Element	Nb	Zr	B	C	Ni	
Content (wt.%)	3.3~ 3.7	0.03~ 0.07	0.006~ 0.015	0.04~ 0.09	Bal.	

## Improved argon atomization process and powder metallurgy route

We have improved argon atomization for the production of clean powder with high yield of fine powder (-270 mesh) and low oxygen content. To guarantee the quality of the final product, an experimental atomization facility with capacity of melting 10Kg of material, and which enables powder handling to be undertaken in vacuum (including sieving and classification, removing ceramic inclusions, sealing and welding capsules for HIP etc.) was self-designed and installed.

The systematic engineering research has also included the improvement of refractories. We selected Al<sub>2</sub>O<sub>3</sub>, MgO, BN for making liners and delivery tubes, and studied the effect of ZrO<sub>2</sub> foam filter for removing ceramic inclusions. After the argon atomization process, the superalloy powders have been produced with oxygen content of <100ppm and 35wt% yield of particles that show a diameter <53μm (-270 mesh).

After being sealed in stainless steel capsule, degassed and hot-isostatic pressed, specimens were heat treated with two schedules:

A: 1140°C×1h, furnace cooling/ 870°C×1h, furnace cooling/ 650°C×24h, air cooling.

B: 1140°C×1h, furnace cooling/ 1060°C×4h, furnace cooling/ 870°C×1h, furnace cooling/ 650°C×24h, air cooling.

After the heat treatment, the mechanical properties at room temperature and at 650°C of the prepared P/M FGH4095 superalloy are shown in Figure 1. The column graphs in Figure 1 shows that higher strength and better ductility at 650°C, are obtained with the B schedule.

This might be attributed to the growth of primary γ' and reduction of Ti, Al in surrounding substrate due to the intermediate holding at 1060°C for 4 hours with furnace cooling.

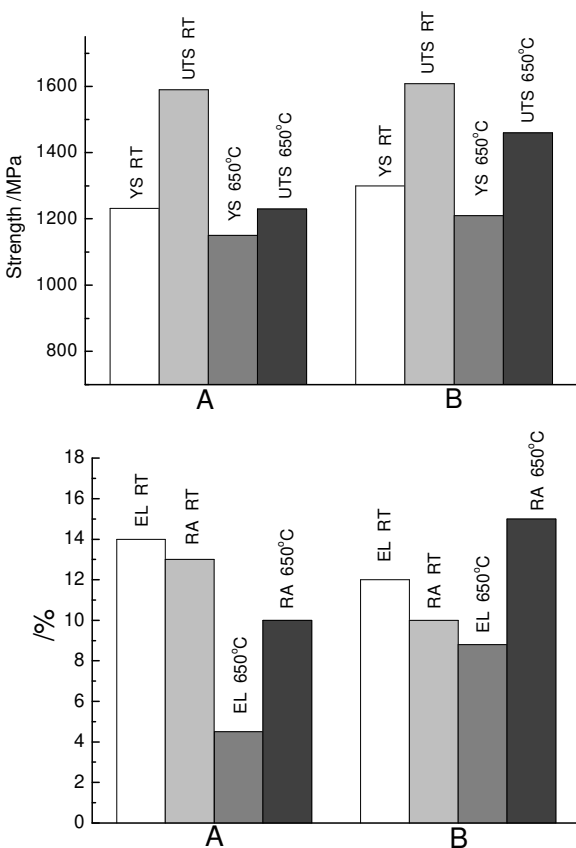


Figure 1. Mechanical properties of the prepared P/M FGH4095 superalloy with heat treatment schedule (A) and with (B)

### Research on an innovative technology for making ultra-fine and clean powder - spark plasma discharge (SPD) process

The SPD process consists of generating spark discharges between pre-alloyed charges immersed in a dielectric fluid. After a complex plasma creation and breakdown, molten and evaporated material is ejected from the electrodes and quenched in the dielectric. This material condenses as particles in the dielectric liquid. Since the powders are quenched in situ, they are extremely rapidly cooled. Comparing to the well known atomization process such as gas atomization and plasma rotating electrode process, this novel process can produce much more finer powders at much faster cooling rate ( $\sim 10^7$  K/sec).

Experiments on an existing spark erosion facility show that spherical powders with different particle sizes from several nm to 40  $\mu\text{m}$  can be adjusted through change of processing parameters. Figure 2 shows the superalloy powder made with SPD with different dielectric liquids. Spherical powders with particle size less than 40  $\mu\text{m}$  have been obtained, which are suitable for making superalloy turbine disks. The SPD powder products have wide potential market. Besides P/M superalloys, it can also be used for injection molding and rapid laser prototyping technology etc. The remaining challenges which should be solved are to reduce the oxygen content of powder and to realize pilot production. This

project on SPD is being supported continuously by National Natural Science Foundation of China (NSFC). On the principle of spark erosion for making magnetic spherical powder by A.E. Berkowitz [12, 13] in University of California, San Diego, we proposed and are self-designing a high voltage adjustable pulse-width SPD facility, as shown in Fig. 3.

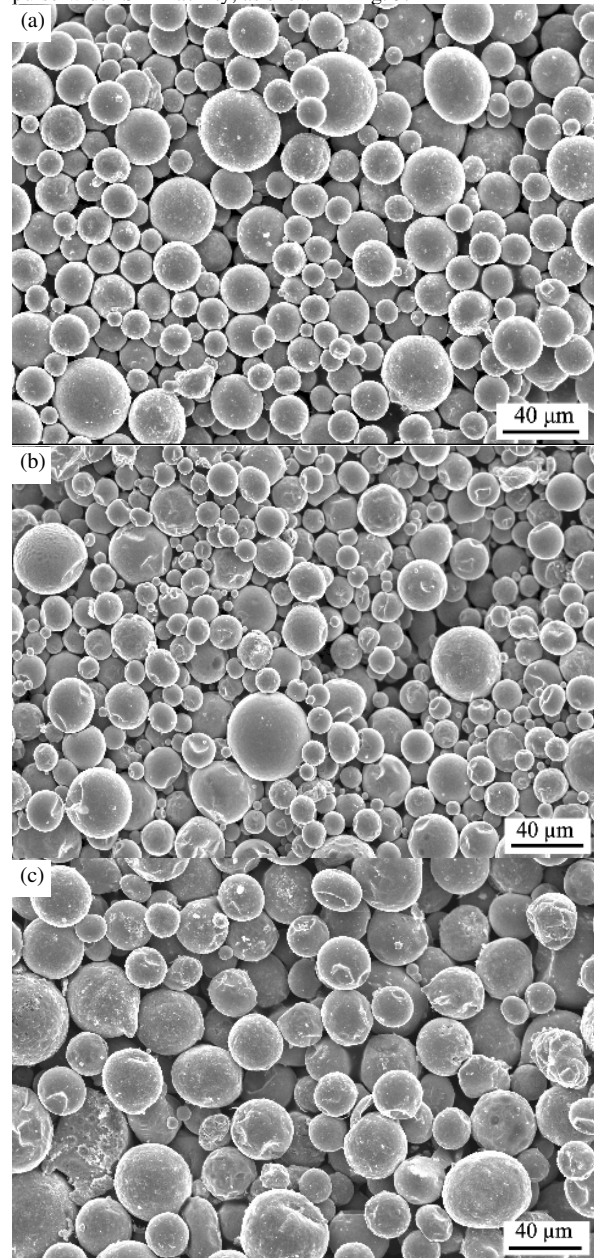


Figure 2. Fine powders prepared in different dielectric liquids, (a) kerosene, (b) alcohol with liquid argon, (c) alcohol

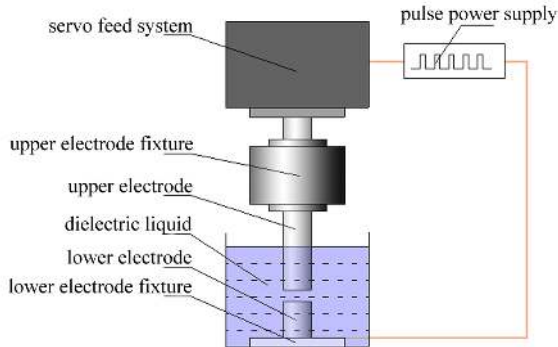


Figure 3. Schematic diagram of spark plasma discharge facility for making fine powders

### Research on spray-formed superalloy

Spray-forming (SF) is an advanced technology of rapid solidification for making bulk, densified, non-segregation high performance materials, proposed by Professor A.R.E Singer in the 1970s<sup>[14]</sup>. Spray-forming is based on powder metallurgy technology, but avoids the disadvantages of powder metallurgy, i.e. many processing steps, high cost and contamination<sup>[15]</sup>. Since 1990's, spray-forming of superalloys is developing rapidly<sup>[16-18]</sup>. Many kinds of superalloys have been studied for making rings, tubes and disks. General Electric Company and its partners have developed the clean metal spray-forming (CMSF) pilot plant<sup>[19,20]</sup>. The CMSF combined electro-slag-refining (ESR) and spray-forming is aimed at removing oxides in the ESR furnace, and improves the cleanliness by using non-ceramic water-cooled copper liner and transfer nozzles, to assure an ultra-clean superalloy production. However, the mass production of spray-forming superalloy disks has not yet been reported. Probably the properties of the spray-formed superalloy have not yet been satisfactory. On the basis of long term research on powder metallurgy and spray-forming technology, we began to study spray-forming of superalloy for making turbine disks.

The ingot stock is produced by vacuum induction melting (VIM), followed by vacuum arc remelting (VAR) from Fu Shun Special Steel Shares Co. Ltd. The spray-forming process is applied at Bremen University with spray-forming plant SK-2 shown schematically in Figure 4. First, the feedstock material was melted in a crucible by an induction coil. The crucible has a melt capacity of 20 liters. The crucible and the induction coil are located in a vacuum vessel. The vessel was evacuated and flooded with argon. At the same time, the tundish was heated by a resistance heating system. When the required temperatures (melt and tundish) were achieved, the molten metal was poured into the preheated tundish and exits through the ceramic nozzle at the bottom. The molten stream was atomized by the nitrogen stream of a scanning atomizer. The droplets were accelerated and finally impinged and solidified on the rotating substrate.

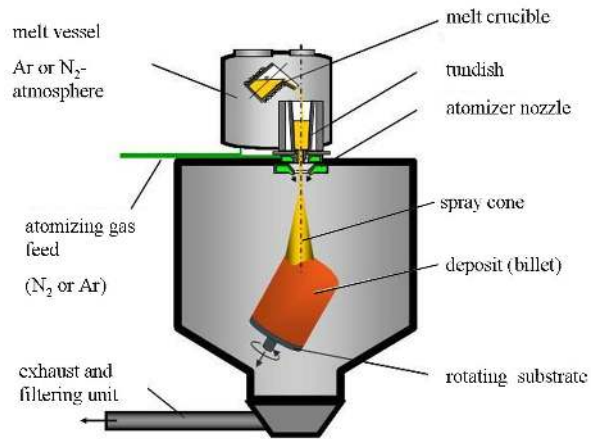


Figure 4. Sketch of the spray-forming plant

Figure 5 shows the photos of the deposit during the spray run. The as spray-formed superalloy shows high density of ~99% with about 1% of porosity and ceramic inclusions measured by image analysis as shown in Figure 6(a). The as-spray-formed specimens have an average grain size about 20 $\mu$ m and the primary  $\gamma'$  about 400~500nm, which can be observed in Figure 6(b) and (c). The photos showing the grain boundaries were etched with Kalling's #2 after polishing and observed by optical microscopy, and showing the precipitations were etched with 9g CrO<sub>3</sub>+90mL H<sub>3</sub>PO<sub>4</sub>+30mL H<sub>2</sub>SO<sub>4</sub> under 2~5volt for 1~5s after electrical polishing with 20mL H<sub>2</sub>SO<sub>4</sub>+80mL CH<sub>3</sub>OH under 25~30 volt for 30~60s and observed by field emission scanning electron microscopy with secondary electron signal model.

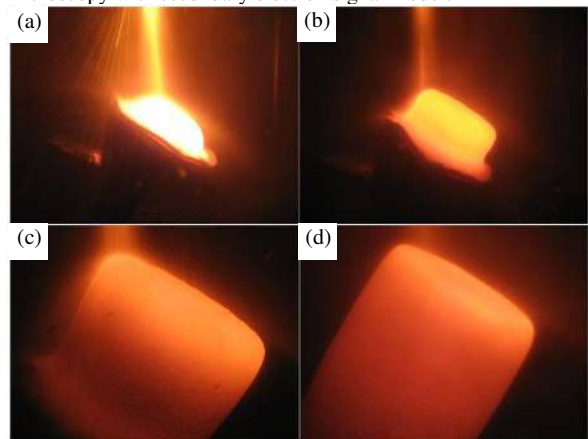


Figure 5. Photos of the deposit during the spray run  
a) t = 41 s, b) t = 88 s, c) t = 135 s, d) t = 270 s.

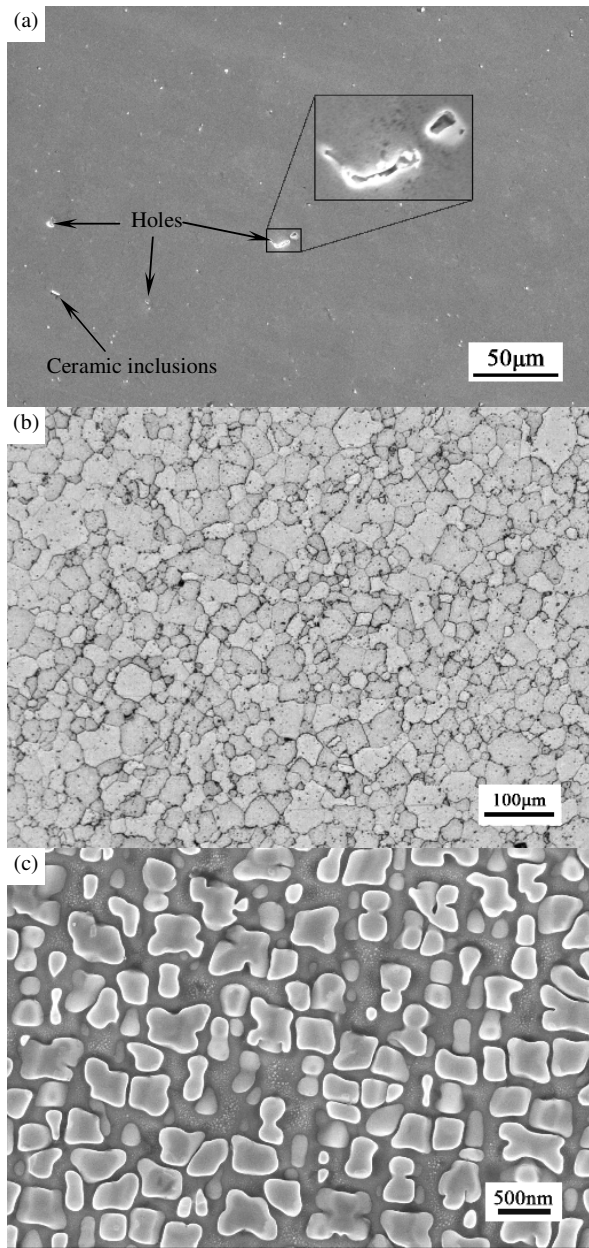


Figure 6. As spray-formed superalloy FGH4095  
 (a) Un-etched secondary electron image observed with SEM,  
 (b) the grain structure, (c)  $\gamma'$  phase

After solution heat treatment at 1140°C for 3h, most primary  $\gamma'$  dissolved (Figure 7 (b)). After hot isostatic pressing (HIP) at 1140°C for 3h, the billets have been fully densified. The grain size remains unchanged (Figure 7(a)). Since the HIP temperature of 1140°C is just under the  $\gamma'$  solvus temperature (about 1170°C), and the cooling rate is slow in the HIP furnace, the retained primary  $\gamma'$  has coarsened. (Figure 7 (c))

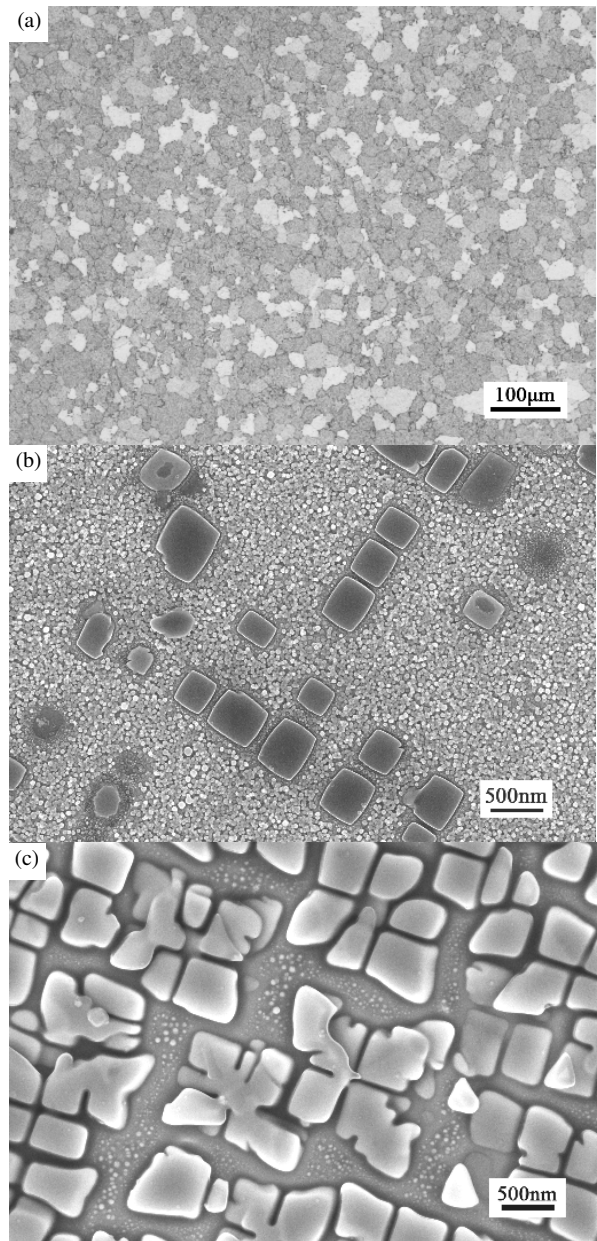


Figure 7. Microstructure of spray-formed FGH4095  
 (a) the grains after HIPing, (b) the  $\gamma'$  phase after solution heat treatment at 1140°C and oil quenched, (c)  $\gamma'$  phase after HIPing.

After hot isostatic pressing, the billets have been isothermal forged in Guizhou Anda Aviation Forging Co., Ltd with 25% height reduction. The last process is heat treatment. The heat treatment schedule is 1140°C×3h, oil quench/870°C×1h, air cool/650°C×24h, air cool. As we can see in Figure 8(a), after isothermal forging and heat treatment, the average grain size is refined to about 13µm, and the ALA(as large as) grain size is about 125µm due to the abnormal grain growth. The primary  $\gamma'$  phase size is about 100nm as shown in Figure 8(b).

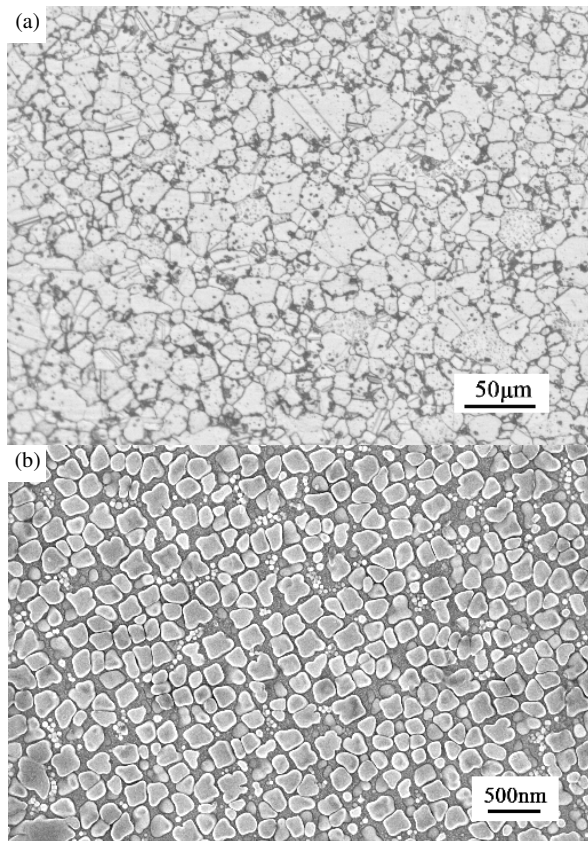


Figure 8. the microstructure of spray-formed FG4095 end product  
(a) the grains (b) the  $\gamma'$  phase

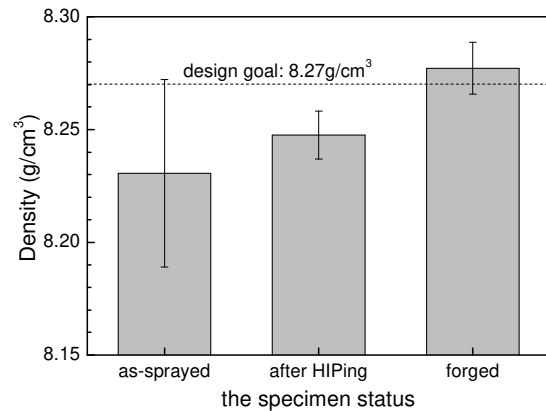


Figure 9. The density of superalloy specimens at different processing status. Measured from (per status) 8 pieces of 10mm×10mm×5mm specimens from similar location.

As show in Figure 9, after sprayforming, the average density is about 8.23g/cm<sup>3</sup>. After HIP, the density increased to near 8.25g/cm<sup>3</sup>. After forging, the average density is above the design goal 8.27g/cm<sup>3</sup>.

The properties of spray-formed FG4095 after HIPing, forging and heat treatment are listed in Table 2, the design goal is to achieve the property levels specified in FG4095 material specification. From the table, we can see that most of design goals have been reached. These encouraging results indicate that this pilot study of spray-forming superalloy has confirmed the potential of the process for producing components.

Table 2. The properties of spray-formed FG4095 end product

property		design goal	maximum	average	minimum
Room temperature tensile	UTS/MPa	1520	1565	1463	1380
	YS/MPa	1170	1231	1211	1195
	EL/%	10	10.9	8.1	6.7
	RA/%	8	13.5	12.2	10.9
High temperature tensile 650°C	UTS/MPa	1280	1552	1511	1490
	YS/MPa	1055	1130	1108	1095
	EL/%	8	12.2	11.4	10.1
	RA/%	10	14.3	12.4	11.3
Creep rupture 650°C, 1034MPa	life/h	≥50h		88.67	
	EL /%	≥3%		3.4	
	RA /%	—		5.7	
Creep 593°C, 1034MPa,100h	Creep Strain/%	≤0.2		0.06	
Low cycle fatigue 538°C, Strain Ratio=0.95±0.02	life/N	>5000	75081	48000	20951

### Conclusions

1. The P/M FGH4095 superalloy prepared from argon atomization powder has been studied with two heat treatment variants. The scheme with 1060°C×4h isothermal treatment shows better strength and better ductility at 650°C.
2. Ultra fine superalloy powders were produced by spark plasma discharge (SPD) process. The powder size can be controlled by adjusting the electrical parameters and changing the kind of the dielectric liquids.
3. The processing of the spray-formed FGH4095 has been studied. The spray formed superalloy after HIPing, forging and heat treatment shows properties approaching the level required by the P/M FGH4095 standard.

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### Reference

1. Wang Xu-Qing, Luo Xue-Jun and Zou Jin-Wen, "Effects of HIP temperature on microstructure of FGH96 superalloy," *Journal of Aeronautical Materials*, 26(3) (2006), 293-294.
2. Zhou Xiao-Ming et al., "Character of artificial non-metallic inclusions in HIPed FGH96 alloy," *Journal of Aeronautical Materials*, 25(4) (2005), 1-5.
3. Jia, Jian et al., "Effects of HIP temperature on microstructures of a new P/M superalloy," *Journal of Aeronautical Materials*, 28(3) (2008), 20-23.
4. Zhang Ying et al., "Heat treatment processes and microstructure and properties research on P/M superalloy FGH97," *Journal of Aeronautical Materials*, 28(6) (2008), 5-9.
5. Wang Xu-qing, Luo Xue-jun and Zou Jin-wen, "Effects of HIP temperature on microstructure of FGH96 superalloy," *Journal of Aeronautical Materials*, 26(3) (2006), 293-294.
6. Tao Yu, Jia Jian and Liu Jian-tao, "Microstructure Characterization and Mechanical Properties of FGH 95 Turbine Blade Retainers," *Journal of Iron and Steel Research International*, 17(9) (2010), 73-78.
7. Zhang Ying et al., "Effect of powder particle size on microstructure and mechanical property of Ni-based P/M superalloy product," *Journal of Iron and Steel Research International*, 10(3) (2003), 71-74.
8. Tian Gao-feng et al., "Cooling precipitation and strengthening for different locations of a powder metallurgy nickel-base superalloy disk," *Transactions of Materials and Heat Treatment*, 29(3) (2008), 126-130.
9. Wu Kai et al., "Characterization of hot deformation behavior of a new Ni-Cr-Co based P/M superalloy," *Materials Characterization*, 61(3) (2010), 330-340.
10. Yu Jun et al., "Superalloy fine powders prepared by a novel spark plasma discharge process," *Acta Metallurgica Sinica*, 44(7) (2008), 892-896.
11. Ge Chang-chun et al., "Improving argon atomization of superalloys," (Invited paper presented at the 4th International Conference on Spray Deposition and Melt Atomisation. Bremen , Germany. 7-9 September 2009)
12. A. E. Berkowitz and J. L. Walter, "Spark erosion: A method for producing rapidly quenched fine powders," *Journal of Materials Research*, 2(1987), 277-288.
13. A.E. Berkowitz et al., "Amorphous soft magnetic particles produced by spark erosion," *Journal of Magnetism and Magnetic Materials*, 254(2003), 1-6.
14. A.R.E. Singer, "Principles of spray rolling of metals," *Metal Materials*, 4(6) (1970), 246-250, 257.
15. P.S. Grant, "Spray forming," *Progress in Materials Science*, 39(1995), 497-545.
16. R.L. Kennedy et al., "Superalloys made by conventional vacuum melting and a novel spray forming process," *Vacuum*, 47(1996), 819-824.
17. J. Mi et al., "Multiphysics modelling of the spray forming process," *Materials Science and Engineering: A*, 477(2008), 2-8.
18. J.Mi and P.S.Grant, "Modelling the shape and thermal dynamics of Ni superalloy rings during spray forming Part 1: Shape modelling – Droplet deposition, splashing and redeposition," *Acta Materialia* 56 (7) (2008), 1588-1596.
19. W.Carter Jr., M.Benz and F.Muller, "Electroslag refining as a liquid metal source for spray forming," (Paper presented at European Conference on Advanced PM Materials, Birmingham, UK, 23-25 October 1995).
20. R.S. Minisandram, R.M. Forbes Jones and K.M. Kelkar, "Prediction of the thermal history of preforms produced by the clean metal spray forming process," *Materials Science and Engineering A*, 326(2002), 184-193.