A UNIQUE SPRAY FORMING PROCESS FOR HIGH TEMPERATURE MATERIALS FOR AEROSPACE APPLICATIONS

Guoqing Zhang, Zou Li, Shifan Tian and Minggao Yan
Beijing Institute of Aeronautical Materials, Beijing 100095, China

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Abstract

One of the research programmes at Beijing Institute of Aeronautical Materials (BIAM) aims to study the new processing methods for high performance materials and to develop spray forming technology suitable for making sound superalloy preforms. A unique pilot low-pressure spray forming plant was established and its spray atomisation and deposition process developed.

The work performed at BIAM concluded that it was feasible to spray form high-density nickel based superalloy preforms or billets using either nitrogen or argon as atomising gas. The results indicated that high density (>99%) preforms with little gas pick-ups and with the microstructural features of rapidly solidified superalloys, i.e. refined equiaxed grains and uniform microstructure, could be achieved after the optimisation of the spray atomisation and deposition process.

The effects of subsequent hot processing on the density, microstructure and mechanical properties of the spray formed superalloy were investigated. Compared to the turbine disk made by a wrought superalloy, the spray formed superalloy disk with identical chemistry showed significantly improved metallurgical quality, higher mechanical properties, better hot workability and lower cost.

1 Introduction

Ingot metallurgy (IM) has been a conventional technology for making superalloys components, such as turbine disks and rings for aerospace applications. However, IM has recently encountered great difficulties for the processing and fabrication of compositionally complex superalloys for turbine and compressor disks, and thereby the development and applications of wrought superalloys have been limited in recent years due to their inhomogeneous chemistry, large grain size and low hot-workability. Powder metallurgy (PM) is currently a standard processing route to high-performance aircraft engine components, such as compressor and turbine disks. Since a PM route usually involves slow, multi-step processes including atomising, sieving, canning, and hot isostatic pressing (HIPping), the PM techniques do share some disadvantages, e.g. high cost, the risk of contamination and high oxygen content.

Spray forming (SF), a integrated atomisation and deposition technique, which comprises the steps of providing a source of molten alloy, converting the melt into a spray droplets by gas atomisation, directing the droplets at a collector where they re-coalesce to form a high density, semi-finished preform, may offer the metallurgical advantages of other rapid solidification techniques while avoiding the disadvantages incurred by IM and conventional PM superalloys. For the last over ten years, considerable efforts have been directed toward developing SF technique as a cost-effective production technology and potential alternative to IM and PM for the more compositionally complex superalloys for gas turbine applications [1-5].

The present research program aims to develop spray forming technology suitable for making sound superalloy preforms and to improve the mechanical properties and hot workability of compositionally complex and high performance superalloys. The microstructure and hot workability of as-deposited materials and the effect of thermal treatment (HIP, hot forging and heat treatment) were investigated in the current program.

2 Spray Forming Equipment and Process

In the early 1990's, a spray forming research facility was designed, established at BIAM and modified in the late 1990's to form a unique pilot low-pressure spray forming plant (Fig. 1).

This facility consists basically of a vacuum melting chamber and a spray chamber, as shown schematically in Figure 1. The vacuum melting chamber contains a melting crucible capable of accepting a 75 kg nickel-base superalloys, a 160 kW, 2500 Hz induction coil, a heated tundish positioned between the melting crucible and an atomizer, and thermocouples to measure the melt temperature.

The spray chamber contains water-cooled deposition collector for making billets and a hydraulic controlled motion mechanism with capability of rotational and linear travel along the collector axis, as illustrated in Fig. 2. The billet collector can also move in the direction transverse to the spray axis and be inclined and eccentrically positioned prior to and during an atomisation-deposition operation. The facility has also the capability of producing ring performs using a heated rotating mandrel as shown in Fig. 3.

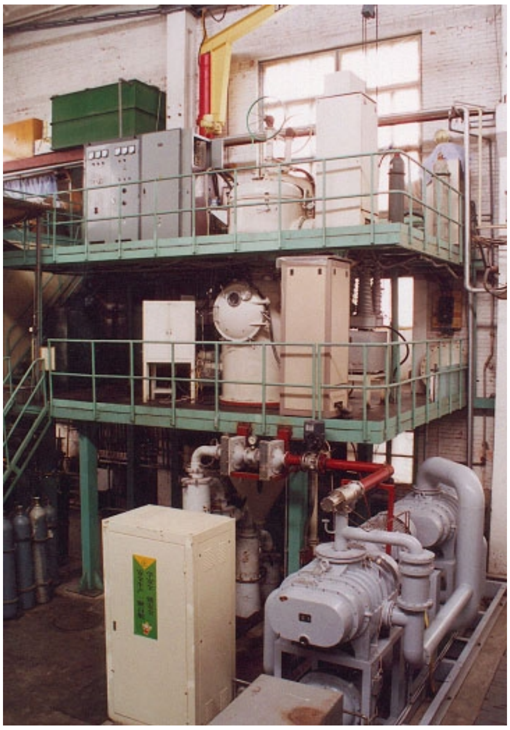

Fig. 1. Low-pressure spray forming plant

This spray-forming unit is equipped with an atomising gas control system, allowing the use of either argon or nitrogen as the atomising gas, an instrument to control and measure the gas flow and video systems to observe atomisation and deposition process. The spray forming facility is also capable of producing powder when the preform collector is removed from the spray chamber.

The spray forming plant includes a vacuum exhaust system with capability of drawing a vacuum in the spray chamber to a pressure lower than 0.02 MPa, or approximately 0.2 atm.

The new system is also equipped with a

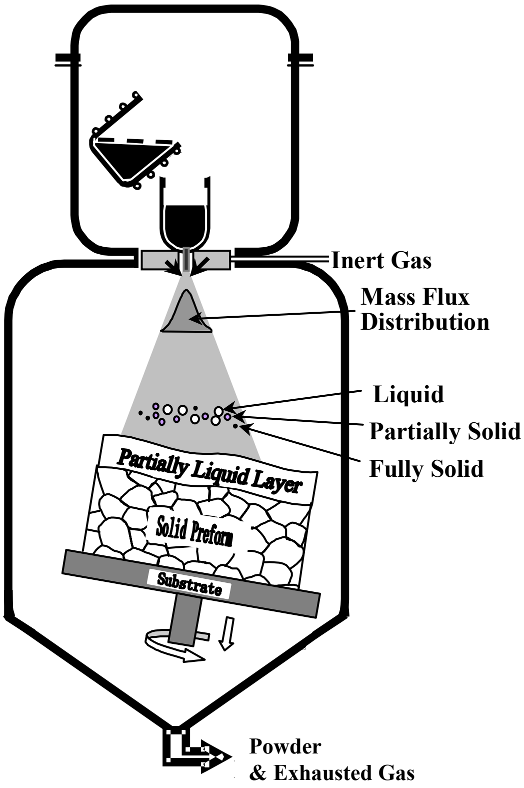

Fig.2. Schematic of spray forming process for billet performs.

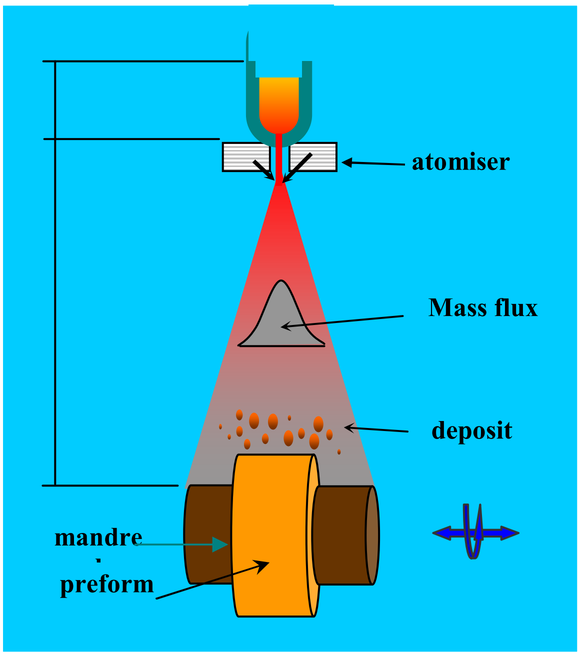

Fig.3. Schematic of spray forming process for ring performs.

pressure control system which allows the spray forming proceed at a constant pressure during an entire atomisation and deposition process.

3 Experimental Procedure

3.1 Materials

The superalloys studied in this program were representatives of compositionally complex nickel based superalloys for aerospace disk and ring applications. The melt stocks were prepared using a vacuum induction melting (VIM) furnace at BIAM. The nominal chemical composition (wt.%) of the nickel base VIM ingot for disk applications can be given as 14Cr, 10Co, 5Mo, 2.6Ti, 2.6Al, 2.6Nb, and 0.06C.

3.2 Spray forming experiments

Spray forming is a three-stage processing approach that involves atomisation, droplet flight and deposition. The spray-forming unit at BIAM has been adjusted and modified to meet the requirements of this research program. The spray forming facility is capable of operating at a constant chamber pressure lower than an atmospheric pressure. Cylindrical billets of approximately 150mm diameter and 200-300 mm height, and ring preforms were produced using optimised spray atomisation and deposition parameters. Numerous atomisation processing variables, including gas type, melt superheat, gas/metal flow ratio and chamber pressure, were considered to study their effects on the porosity and microstructures of spray-deposited materials. The major processing variables used in this investigation are listed in Table 1.

Table 1. Process parameters used in the SF experiments.

Atomising gas	Spray height (mm)	Chamber pressure (MPa)
Nitrogen	350 – 400	0.02 - 0.1
Melt superheat (°C)	Melt pour rate (kg/min)	Atomisation gas pressure (MPa)
100 – 200	10 - 20	2.0 - 2.5

3.3 Hot deformation test

Laboratory forging simulations were performed using a Gleeble 3500 thermal/mechanical testing system. Compression tests were run at constant true strain rates of 10/s, 1/s, 0.1/s and 0.01/s to evaluate the workability of the spray formed superalloys, i.e., the ability to resist cracking and the flow stress for deformation. The size of the cylindrical specimen was 8mm diameter by 10mm long. The hot ductility of the spray formed alloy was determined by the measuring the reduction in height after compression deformation at various temperatures. The heating rate for the specimen was 8°C/s and the specimen was soaked at the designated temperature for 3 minutes.

3.4 Subsequent thermal processing

Spray formed billets were further consolidated by HIPping at 1150°C and 150 MPa for 4 hours in argon to seal the porosity.

HIPped billets were hot upset forged (HF) at 1000-1100°C to make disk forgings; HIPped spray formed rings were rolled to the designed sizes.

Forged pancakes were subsequently heat treated (HT) by subsolvus solution followed by single ageing to optimise the mechanical properties [8].

3.5 Microstructural examination

Metallographic samples were prepared by using conventional mechanical grinding and polishing procedures and examined by optical microscopy (OM), transmission electron microscope (TEM) and scanning electron microscope (SEM).

4 Experimental Results and Discussion

4.1 Spray Formed Preforms

After the optimisation of the spray atomisation and deposition process, process parameters were established to produce high-density (higher than 99%) superalloy billets and rings at a low chamber pressure. Fig. 4 shows the spray formed billet, forging of a high performance superalloy for the manufacture of aerospace disks.

The spray formed superalloy exhibited excellent forgeability. Ring preforms of compositionally complex nickel based superalloys were also produced by spray forming. Spray formed and HIPped preforms were directly ring rolled to evaluate the workability of the spray formed "non-hot workable" materials. Both qualitative and quantitative results obtained from ring roll operation indicated that the spray formed ring performs of superalloys were easier to ring roll, as compared to their wrought counterparts of the same alloys. The improved hot workablility of spray formed alloys was a result of the improved homogeneity and refined microstructure. Examples of spray formed ring perform and rolled rings are shown in Fig. 5.

Fig. 4. As-spray formed billet (upper left), machined pre-forge cylinder (upper right) and forged disk part (lower) of a high performance superalloy.

Fig. 5. Spray formed ring perform (upper) and rolled ring (lower).

4.2 Metallurgical quality of spray formed superalloys

The quality of spray formed preforms was evaluated in four ways: (1) oxygen and nitrogen contents, (2) grain size, (3) other microstructural features and (4) density (or porosity). The most important process variables identified were chamber pressure, metal flow rates, atomising gas pressure, spray distance, superheat and collector settings.

Table 2. Nitrogen, oxygen and hydrogen contents in the spray formed disk alloy (wt. ppm).

Gas	N_2	O_2	H_2
VIM melt stock	10	13	<1
Preforms	160 ~ 270	13 ~ 17	<1

As revealed in Table 2, there was very low pick up of the oxygen and hydrogen during spray forming with nitrogen atomisation. It is also evident that the nitrogen pickup was considerable with nitrogen atomisation. Since the solubility of nitrogen in nickel-base superalloys is low, nitrogen levels in excess of its saturation level would result in precipitation of TiN or more complex (Ti, Nb)(C, N) both at grain boundaries and within the grains [3].

The density measurements for the spray formed preforms were obtained from the average measurements of the density samples taken from the slabs of the preforms. After the optimisation of processing parameters, the porosity of the nitrogen-atomised preforms could be less than vol.1% in every section of the preforms.

The microstructure of as-deposited materials is shown in Fig. 6. The typical microstructure consisted of equiaxed nondendritic structure with an average grain size ranging from ASTM 7 to 8, depending on the process conditions. Due to the rapid solidification during the spray atomisation, essentially no chemical segregation nor macro-segregation were evident. Additionally, no prior particle or splat boundaries were observed in the spray formed materials.

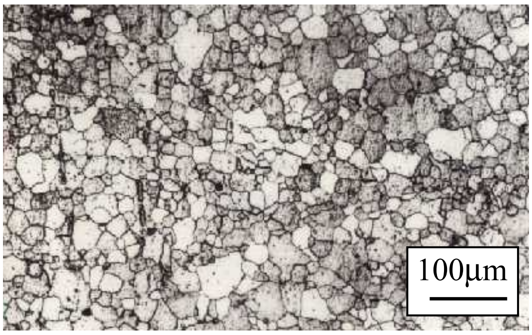

Fig. 6. Optical microstructure of the spray formed superalloy.

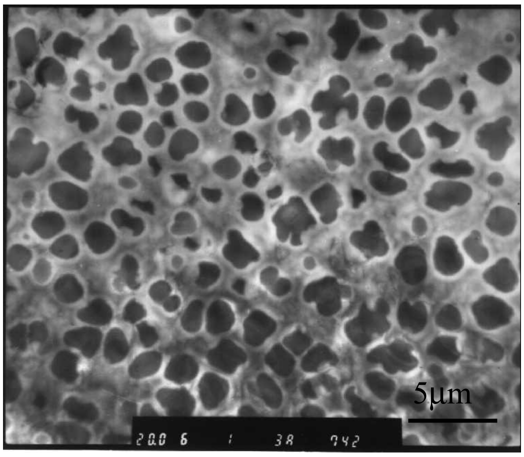

Fig. 7. TEM microstructure of the spray formed superalloy.

The microstructure of the as-deposited material shown in TEM micrograph (Fig. 7) at higher magnification indicated the variety of precipitates formed during sequential steps of atomisation and deposition process.

4.3 Microstructural and porosity evolution during thermal processing

The residual porosity (<1%) in the as-deposited preforms, which was isolated and not interconnected, was eliminated by HIPping and a density in excess of 99.9% of theoretical density was achieved after HIPping. Microstructural observations revealed that HIP did not coarse the grain size of spray formed materials. Hot forging refined the grain size of spray formed materials from ASTM 7-8 to ASTM 9-10, as shown in Fig. 8. The preforms were successfully consolidated to a density of 100% by using HIPping and hot forging.

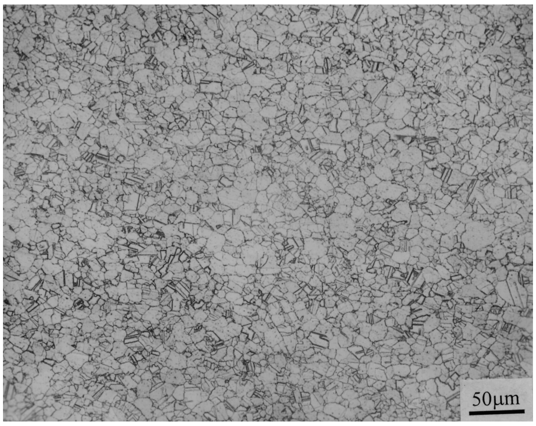

Fig. 8. Optical microstructure of the SF + HIP + forged superalloy.

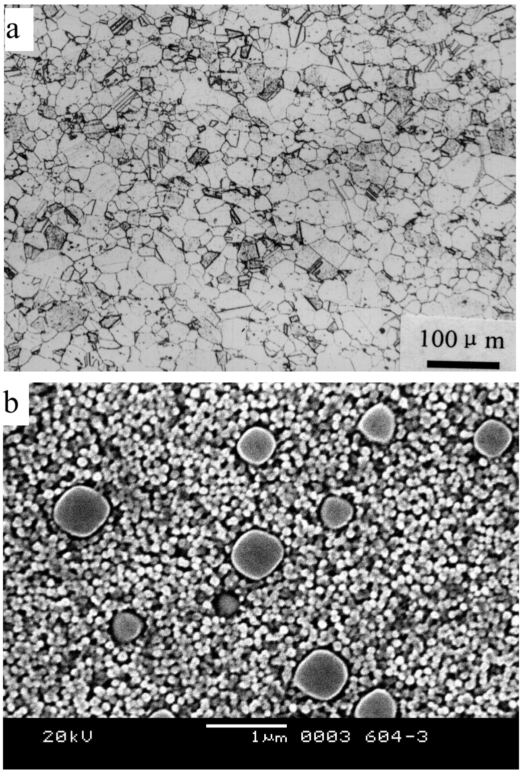

Fig. 9. Optical and SEM microstructures of the spray formed materials after heat treatment.

Prior to the mechanical testing, heat treatment studies were performed on the spray formed superalloy to optimise the mechanical properties of this alloy [8]. A unique heat treatment cycle developed for the spray formed superalloy resulted in improved tensile and stress rupture properties of the SF + HIP + forged materials. Fig. 9 shows the microstructure of the heat treated materials. A recrystallised, equiaxed and fine microstructure was obtained after the heat treatment. Gamma prime phase of two different sizes precipitated during the heat treatment (Fig. 9b).

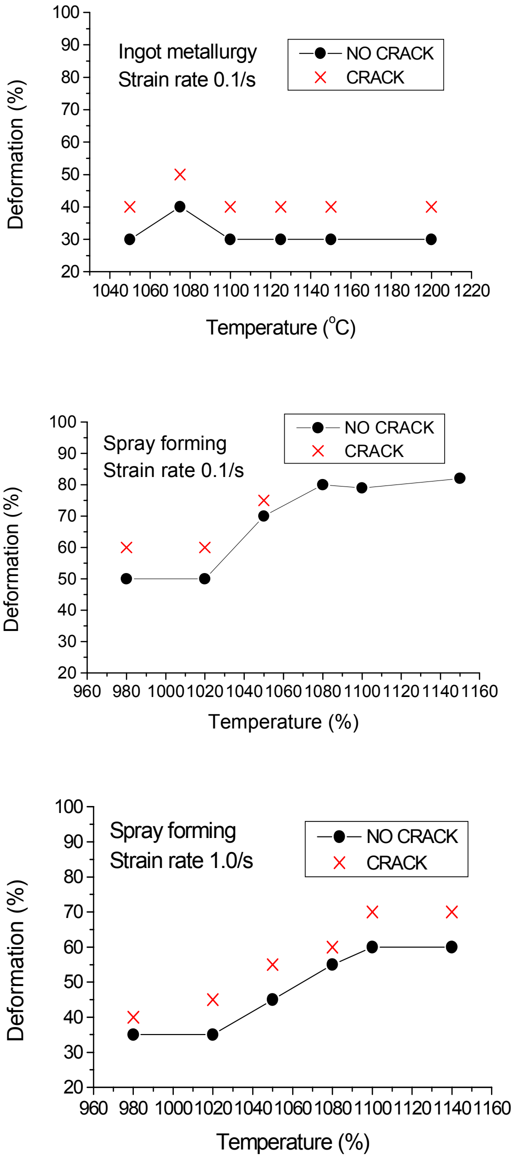

Fig. 10. Workability of the spray formed and IM superalloys at strain rates of 0.1/s and 1.0/s.

4.4 Hot workability of the spray formed superalloy

The workability of as-sprayed materials was dependent on the deformation temperature and increases approximately in proportion to the increment of deformation temperature (Fig. 10). The hot ductility of the spray formed alloy at 1100°C could be greater than 60%. The forgeability of spray formed materials was significantly higher than that of the IM alloy of identical composition to the SF alloy. Higher strength has traditionally been offset by lower hot workability in conventionally processed superalloys. The spray forming technology provides improved hot workability because of improved homogenisation of alloy chemistry and refined grain structure inherent in gas atomisation processing.

4.5 Mechanical properties of the spray formed superalloy

Tensile properties of spray formed and hot processed materials after the heat treatment were determined at room temperature, 650°C and 750°C. As listed in Table 3, upset forging resulted in considerable increase of room temperature and elevated strength. The tensile properties of spray formed materials were significantly improved when compared to IM processed materials. The improvement was because of the homogeneous and refined structure.

Table 3. Tensile properties of the spray formed superalloy.

Condition	Test temp.	0.2%YS MPa	UTS MPa	EL %	RA %
SF + HT	20°C	898	1372	26.3	36.6
SF + HF + HT		1060	1449	23.6	41.2
IM + HF + HT		864	1329	17.7	18.9
SF + HT	650°C	762	1193		
SF + HF + HT		957	1326		
SF + HT	750°C	822	1050		
SF + HF +HT		952	1059		

As shown in Table 4, stress rupture properties of the spray formed superalloy at 650°C and 750°C were comparable to the property levels of the wrought counterpart of the same alloy. Thermal processing refined the microstructure of the as-deposited materials, and thereby resulted in significant improvement on the stress rupture at 650°C but slightly reduced the stress rupture life at 750°C.

Table 4. Stress rupture life (hour) at 650°C and 750°C

Condition	650°C/834MPa	750°C/539MPa
SF + HT	165	81
SF + HF + HT	404	63
IM + HF + HT	>50	>50

5 Conclusions

1) A unique pilot low-pressure spray forming plant was established and its spray atomisation and deposition process developed to produce billets and rings for aerospace applications.

2) It was feasible to spray form high-density nickel based superalloy preforms p with little gas pick-ups using either nitrogen or argon as atomising gas. Higher density (>99%) superalloy billets could be spray formed at a low chamber pressure using nitrogen as the atomisation gas.

3) Microstructural features of rapidly solidified superalloys, i.e. refined, equiaxed grains and homogeneous microstructure, were achieved after the optimisation of the spray atomisation and deposition process.

4) Thermal-mechanical processing could consolidate the preforms. and further refine the microstructure of the spray formed materials.

5) The hot workability of the spray formed superalloy was significantly improved compared to the wrought counterpart of the same alloy. The improved hot workability by spray forming technology was due to the refined and homogenous structure.

6) The tensile properties of spray formed and thermal processed materials were

significantly higher than those of wrought superalloy with identical chemistry.

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