## SPRAY-FORMED HIGH-STRENGTH SUPERALLOYS

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#### Abstract

Five commercial nickel-base superalloys, Rene'95, AF115, AF2-1DA, Astroloy, and MERL 76, were spray-formed into disk preforms. Metallurgical evaluation and high-temperature deformation studies were performed on the as-sprayed materials. Spray forming offers these superalloys the advantages of rapid solidification: segregation-free, uniform structure with a fine grain size. The preforms demonstrated good forgeability even without superplasticity in the as-sprayed condition. All alloys were press-forged and supersolvus-annealed. High-temperature strengths and stress rupture properties were compared, and AF115 shows the best temperature capability.

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#### Introduction

Spray forming is a new process that combines rapid solidification and high deposition rate. This process involves atomizing a stream of molten metal into droplets with high-speed gas, with the droplets being collected on a substrate before solidifying. As compared to the conventionally cast ingot, the spray-formed deposit, or preform, has little segregation and small grain structure. The process is considered as one of the least cost methods to produce high-quality, rapidly solidified (RS) materials (1).

For the past twenty years, the advantages of rapid solidification for high-strength superalloys has been realized (2). Powder metallurgy (P/M) processing is currently the standard route to high-performance aerospace components, such as compressor and turbine disks in advanced aircraft engines. The high inherent cost of powder processing limits the applications of RS alloys. Spray forming offers less cost because of the absence of collecting and handling powders, and also results in a low oxygen content in the preforms.

In the investigation to be reported, five commercial P/M superalloys, including Rene'95, AF115, AF2-1DA, Astroloy, and MERL 76, were prepared by spray-forming. The tensile and stress rupture properties of the preforms were examined after hot die press forging. In addition to their metal processing characteristics, alloys are compared for their performance capability.

### **Experimental Procedures**

Table I lists the designation and the nominal compositions of 5 superalloys employed for this study. Also included in Table I are the density and the volume fraction of strengthening precipitates estimated for each alloy. Because of the high precipitate content (48 - 61%), these alloys can only be processed through rapid solidification (3).

ALLOY:	RENE'95	AF115	AF2-1DA	MERL76	ASTROLOY
Ni	BALANCE	BALANCE	BALANCE	BALANCE	BALANCE
Co	8.00	15.00	10.00	18.50	17.00
Cr	13.00	10.70	12.00	12.50	15.00
Mo	3.50	2.80	2.75	3.20	5.00
W	3.50	5.90	6.50		
Al	3.50	3.80	4.60	5.00	4.00
Ti	2.50	3.90	2.80	4.40	3.50
Nb	3.50	1.70	40 40 CH	1.40	
Ta			1.50	* = =	
Hf		0.75		0.40	
Zr	0.05	0.05		0.06	0.045
C	0.060	0.050	0.040	0.020	0.020
В	0.010	0.020	0.015	0.020	0.030
PPT. v%	49%	55%	52%	61%	48%
DENSITY (lb./in <sup>3</sup> )	0.298	0.301	0.301	0.283	0.289

### Table I. Chemical Compositions of Five Commercial P/M Superalloys



Figure 1 Disk preform of Rene'95 made by spray forming.

One 20 kg heat of each alloy was prepared by vacuum induction melting (VIM) and cast into a 100-mm-diameter, chilled copper mold under an argon atmosphere. The ingots were conditioned and remelted by induction heating in a magnesia crucible. A zirconia nozzle was cemented into the bottom of the crucible, and a disk of the alloy being melted was inserted into a recess in the nozzle. Spraying began when the disk melted, and nitrogen was used as the atomizing gas. The atomized stream was deposited on a rotating disk of cordierite. A typical deposit is shown in Figure 1.

Isothermal compression tests were carried out at forging temperatures to study the hot deformation behavior of spray-formed materials. A closed-loop servohydraulic machine equipped with a resistance furnace was controlled by a constant strain rate signal generator. Cylindrical coupons cut from the preforms were tested in air with a 60% reduction in height.

A billet about 75 mm by 75 mm by 90 mm was machined from the preforms for press forging. Forging was carried out on a 300-ton press with the die blocks preheated to 900°C (1652°F). The press speed was selected at 7 in/min. The forging procedures included an initial upset with 42% reduction and a final press with 43% reduction. In all cases, the forging temperature was kept below the precipitate solvus of the alloys.

Metallographic samples were prepared by using conventional mechanical grinding and polishing procedures. An etchant consisting of 10 ml HCl, 10 ml HNO<sub>3</sub>, and 30 ml  $H_2O_2$  revealed the grain structure. Subsize round tensile specimens of 0.10-in.-gauge diameter were machined and low-stress ground for both tensile and rupture testing. Tensile tests were performed in a vacuum chamber with an initial strain rate of 0.05 per minute. Stress rupture tests were run under an argon atmosphere by using a lever-arm machine with a constant load.

## Results

## Microstructure

All compositions were spray-formed successfully except AF2-1DA. When cut into halves, a large crack was observed at the center of the AF2-1DA preform. The crack developed during the cooling after solidification. A second attempt with a careful control of cooling still resulted in a crack. The other four alloys that were processed with the same spraying condition did not show any cooling crack.

The as-deposited microstructure (Figure 2) consists of equiaxed grains with no indication of chemical segregation. The grain size is about 25 to  $40\mu$ m, depending upon the alloy composition. A certain degree of annealing after rapid solidification is expected to cause some grain growth before the preform is cooled. The cooling rate is also reflected by the formation of precipitate particles inside the grains and along the grain boundaries (Figure 2.b).

Figure 3 shows the relative density values of a slab cut through the center of the Rene'95 preform. Low density regions are found along the bottom horizontal row and the center line of rotation. Porosity as seen in Figure 2.a is responsible for the low density in both regions, but the formation mechanism may differ. One porosity is caused by the deposition on the cold surface of collector; the other is a consequence both of centrifugal forces and of nonuniform deposition parallel to the face of the collector disk. All pores are isolated and do not connect with the free surface. The forging brought the preforms essentially to full density; the porosity can also be removed by hot isostatic pressing (HIP). All spray-formed preforms contained less than 35 ppm oxygen. There is a substantial pickup of nitrogen that is, the atomizing gas, to the amount of 100 to 150 ppm, depending on the alloy being atomized.

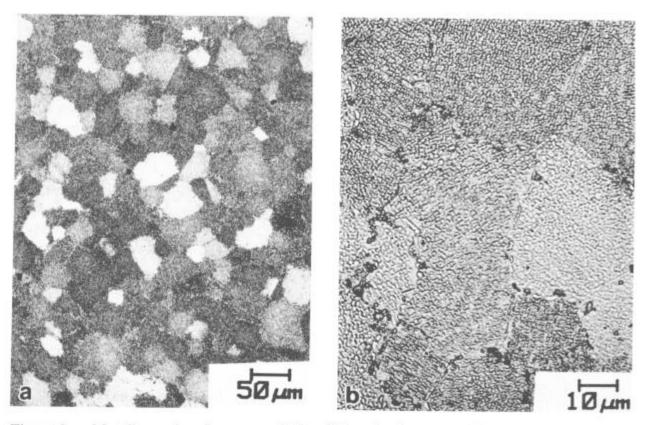


Figure 2 Metallography of as-sprayed Rene'95: a.) a low magnification showing the uniform equiaxed grain structure with some porosity; b.) a high magnification showing the cooling precipitates inside grains and along grain boundaries.

$\begin{array}{c ccccccccccccccccccccccccccccccccccc$			T	T			
99.8       99.8       99.8       99.8       99.6         26       20       14       8       2         99.8       99.8       99.7       99.4         27       21       15       9       3         99.8       99.8       99.6       99.5         28       22       16       10       4         99.7       99.8       99.7       99.7         29       23       17       11       5				<u> </u>			
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99.8         99.8         99.8         99.7         99.4           27         21         15         9         3           99.8         99.8         99.6         99.5           28         22         16         10         4           99.7         99.8         99.8         99.7         99.7           28         22         16         10         4           99.7         99.8         99.8         99.7         99.7           29         23         17         11         5		99.8	99.8	99.8	99.8	99.6	
99.8         99.8         99.7         99.4           27         21         15         9         3           99.8         99.8         99.6         99.5           28         22         16         10         4           99.7         99.8         99.7         99.7           29         23         17         11         5		26	20	14	8	2	1
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	1	99.8	99.8	99.8	-	99.4	
28         22         16         10         4           99.7         99.8         99.7         99.7           29         23         17         11         5		27	21	15	9		1
99.7         99.8         99.7         99.7           29         23         17         11         5	1	99.8	99.8	99.8	99.6	99.5	
29 23 17 11 5	/	28	22	16	10	4	1
	/	99.7	99.8	99.8	99.7	99.7	
998998998998998997		29	23	17	11	5	
		99.8	99.8	99.8	99.8	99.7	
30 24 18 12 6		30	24			6	
99.3 99.4 99.4 99.4 99.3		99.3	99.4	99.4	99.4	99.3	

Figure 3 Density measurements on the cross section of a Rene'95 disk preform. 100% density equals 8.30 g/cc.

Superalloy structures are determined by the forging conditions and the subsequent heat treatments. A supersolvus solution treatment was applied to all preforms so that the property comparison will be predominantly the chemistry effect. Fully recrystallized grain structures were developed for every alloy.

# Forgeability

One of the significant advantages of RS materials is the hot workability provided by their fine and homogeneous structure. Figure 4 shows various Rene'95 coupons tested in compression with a 60% reduction. The conventionally cast coupon suffers a serious cracking problem, while both P/M and spray-formed coupons can sustain the deformation satisfactorily.

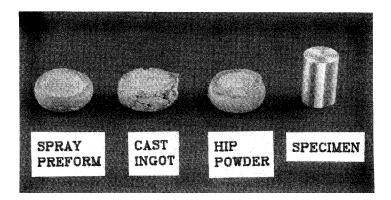


Figure 4 Improved forgeability of spray formed materials as illustrated by isothermal compression testing.

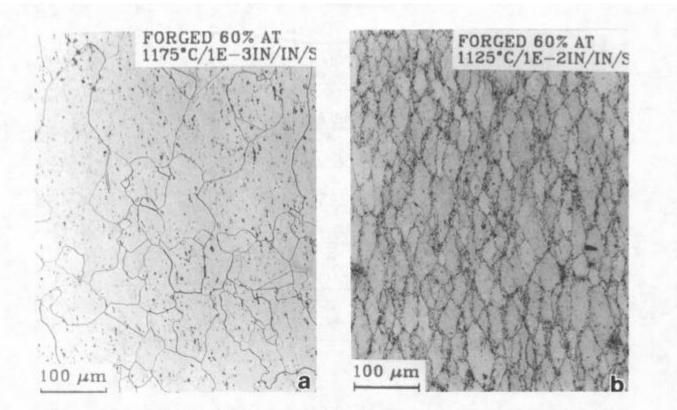


Figure 5 Metallography of as-forged Rene'95 preforms forged at: a.) supersolvus; b.) subsolvus.

The hot deformation behavior of sprayed preforms was studied at different temperatures and different strain rates. The flow curves indicate that the as-deposited preforms never can reach a steady state, i.e., a constant stress for a given strain rate at a fixed temperature. Metallographic examination agrees with the above observation. Figure 5 shows the asdeformed microstructures of Rene'95 after 60% reduction in height. When the preform is deformed above the precipitate solvus of the alloy, the grains grow continuously during the deformation, and the slow strain rate generates a large grain size. In contrast, when the temperature is below the precipitate solvus, dynamic recrystallization takes place along the grain boundaries. However, the recrystallization can never be completed within a 60% reduction. As a result, a duplex grain structure was developed during forging below the precipitate solvus. Such a grain structure has been called the "necklace" structure, which shows some unique properties.

The stress for hot working relates to the grain size directly, illustrated by Figure 6, in which the stress at 30% strain is plotted as a function of strain rate for different Rene'95 materials tested at 1100°C. The lower working stress required for RS materials means better forgeability. The strain rate sensitivity, defined as the slope of the curves in Figure 6, can suggest the occurrence of superplasticity (4). Unlike the P/M material, which shows a strong strain rate dependent flow stress at slow strain rates, the spray-formed materials behaves similar to the cast material. No superplastic regime is found in the as-sprayed preform; the grain size is not fine enough as suggested by the metallography (Figure 5.b). However, superplasticity can be easily achieved for the spray-formed superalloys with some thermal mechanical processing (TMP).

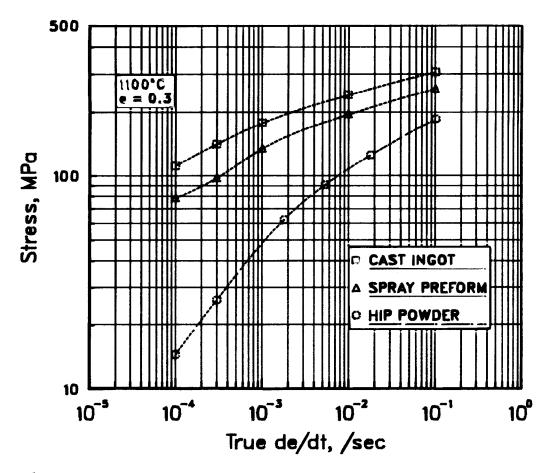


Figure 6 Comparison of strain rate dependence of flow stress for Rene'95 made from different processes.

### **Property Comparison**

To eliminate the influence of forging operation on the alloy properties, alloy forgings were solutioned above their precipitate solvus. The solution temperatures are listed in Table II. A fully recrystallized, equiaxed grain structure was developed for every alloy. A standard aging treatment, 760°C for 16 hours, was then applied subsequently.

Table II lists the tensile properties of four spray-formed superalloys measured at 650 and 760°C, respectively. AF115 stands out as the strongest alloy, followed by Rene'95, MERL 76, and then Astroloy. The difference becomes pronounced for the 760°C tensile strength. The alloy strength order can be rationalized by the volume fraction and the chemical composition of strengthening precipitates. A high precipitate content, as well as a high level of refractory alloying addition, are the important factors for high-temperature strength (5).

Alloy temperature capability was evaluated by the stress rupture test at  $663^{\circ}C/758$ MPa and at  $760^{\circ}C/620$ MPa. The stress rupture test data for each preform forging was normalized by the Larson-Miller parameter, (absolute temperature × [log(rupture life) + 20]), and the results are plotted in Figure 7. The same relationship as found for alloy strength is observed for temperature capability. However, the chemistry of alloy matrix is believed to play a role as important as that of precipitates, especially at high temperatures.

Figure 7 also includes a data point of P/M Rene'95 tested at 760°C/620MPa. This P/M alloy was HIP'ped and forged under conditions similar to the spray-formed preform. The

ALLOY:	RENE'95	AF115	MERL76	ASTROLO
650C				
YS (MPa)	1040	1080	1007	903
TS (MPa)	1471	1464	1327	1347
EL (%)	12	15	21	27
760C		× .		
YS (MPa)	1008	1049	1007	887
TS (MPa)	1152	1242	1102	1040
EL (%)	26	30	30	30
Anneal				
Temp.	1175C	1200C	1200C	1150C

 Table II.
 High-Temperature Tensile Properties of Supersolvus Annealed

 Superalloys Prepared by Spray-Forming

same type of supersolvus annealing and the standard aging were applied. The inferior rupture life of P/M material is attributable to the grain size effect (6). A coarser grain structure associated with the spray forming process is another advantage in high-temperature applications.

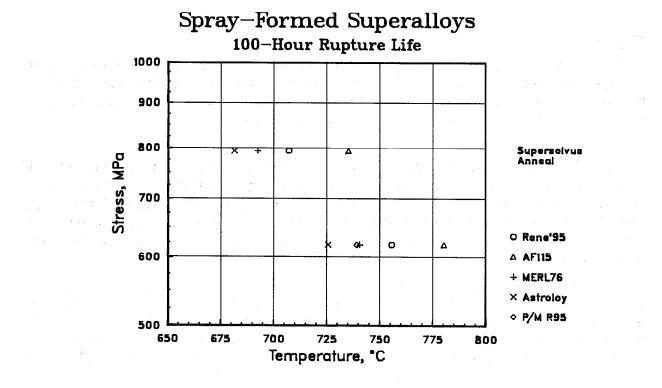


Figure 7 Comparison of stress rupture capability measured in spray-formed, high-strength superalloys.

# Conclusions

- A. Spray forming has been successfully applied to high-strength superalloys and offers an economical route for RS materials. The preforms show good forgeability and a homogeneous structure.
- B. The spray-formed superalloys exhibit a unique structure with beneficial characteristics. The smaller grain size of the spray-formed alloy relative to a cast structure improves forgeability, and the coarser grain size relative to the P/M structure improves the high-temperature capability.
- C. From the results of five commercial alloys studied, the alloy containing high refractory metal additions like AF115, show the most potential for high-temperature applications.

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