AN EVALUATION OF SPRAY FORMED ALLOY 718

R. L. Kennedy, R. M. Davis and F. P. Vaccaro

Teledyne Allvac
Research and Development
P O Box 5030
Monroe, NC 28110
USA

Abstract

Spray deposited 718 was evaluated and its characteristics were compared with those of conventional as-cast and cast-wrought 718 material. General comparisons were made on the basis of microstructural features, microsegregation and fracture toughness. Spray forming via the Osprey[R] Process was also investigated through a computer model developed at Drexel University.

In the as-sprayed condition, 718 preforms are characterized by their refined grain structure, near-zero macrosegregation, and acceptable mechanical properties. In this condition, both intergranular and intragranular Nb-rich MC carbides are observed. Also, with elevated nitrogen levels as a result of nitrogen atomization, preforms subjected to high temperature thermal treatments undergo marked intergranular precipitation of Ti-rich carbides and carbonitrides. Microsegregation, as measured by the "segregation ratio", in as-sprayed 718 was found to be comparable to that exhibited in conventional cast-wrought material.

A computer simulation of solidification of Osprey 718 was performed to permit relating the segregation results from this study to large diameter billet size deposits. This simulation showed the majority of droplets are 75% solidified upon impingement with the substrate. Therefore, for large diameter Osprey billets, it is predicted that microsegregation will be significantly reduced and macrosegregation eliminated.

In the upset, solution treated and double aged condition, Osprey 718 displayed a fracture toughness value comparable to conventional cast-wrought material.

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Superalloy 718—Metallurgy and Applications
Edited by E.A. Loria
The Minerals, Metals & Materials Society, 1989

Introduction

The Osprey Process is a rapid solidification spray deposition technique that can generate near net-shape products in one integrated operation (1,3). During the process, a molten stream of metal is converted into a spray by means of gas atomization. The metal droplets, at various stages of solidification, are then directed towards a collecting surface where they form a dense preform (2). In general, either argon or nitrogen is utilized as an atomizing gas and depending on the deposition conditions, as-sprayed shapes are typically near 100% dense (3).

Similar to other rapid solidification processes, spray forming imparts many beneficial material properties. Typically, preforms exhibit a fine grain microstructure, near-zero macrosegregation, and acceptable mechanical properties (1). Also, Osprey reduces the number of required manufacturing operations and may potentially lead to significant economic benefits.

In the present study, spray formed 718 is evaluated and its characteristics compared with those of both as-cast and cast-wrought 718. General comparisons are made on the basis of the relative degree of microsegregation, fracture toughness and microstructural features. Also reported are the results of the application of computer modeling to the Osprey Process.

Experimental Procedure

Material used for the segregation study was obtained from three different heats of 718 representing as-cast, cast-wrought and spray formed conditions. As-cast samples were obtained from the center of a VIM/ESR 30.5 cm (12")Rd. ingot. Similarly, the cast-wrought samples were taken from the center region of a 25.4 cm (10")Rd. billet which was the product of a 50.8 cm (20")Rd. VIM/VAR ingot. The spray formed segregation study samples were taken from the mid-thickness location of a 15.2 cm (6")Rd. x 12.7 cm (5")thick Osprey preform (#392). All three preforms were the product of VIM/VAR stock converted via the Osprey Process utilizing nitrogen or argon as the atomizing gases. The chemical compositions of these materials are given in Table I.

Table I. Alloy Composition (wt.%), Ni Balance

Material	Fe	Cr	Nb	Mo	Ti	Al	O_2	N_2
As-Cast	20.09	17.63	5.01	2.88	.94	.52	.0010	.0060
Cast-Wrought	18.50	17.69	5.23	2.92	.95	.53	.0026	.0099
N_2 Atomized Preform #392	18.95	17.89	5.02	2.86	.93	.57	.0010* .0020	.0058* .0288
N_2 Atomized Preform #393	19.07	18.19	4.91	2.86	.94	.57	<.0010	.0211
Ar Atomized Preform #21	19.66	17.67	4.97	2.89	.94	.46	.0029	.0072

*Gas content prior to spray forming.

Microsegregation studies were performed on transverse specimens that were metallographically prepared and etched in a solution containing 5ml H_2O, 60ml HCl and 6g $CuCl_2$ (Modified Kalling's Etch). The electron microprobe (EMP) was utilized to evaluate each specimen quantitatively and assess the degree of microsegregation present. In this study, both line profile analyses and a statistical random point EMP method were employed to characterize microsegregation. The results from each analysis were used to determine the maximum concentration (Cmax), the minimum concentration (Cmin) and the segregation ratio (SR=Cmax/Cmin). Details of each technique are given in Table II.

Table II. Comparison of Electron Microprobe Techniques

	Semi-Continuous Line Profile Analysis	Random Point Analysis
Source	McCrone Associates	Cameron Forge Company
Sample Size	1.3cm x 1.3cm x 1.3cm (0.5" x 0.5" x 0.5")	1.3cm x 1.3cm x 1.3cm (0.5" x 0.5" x 0.5")
Accelerating Voltage	20 KV	20 KV
Beam Diameter	0.2 microns	20 microns
Detector	EDX-Cr WDX-Nb, Al, Ti, Mo	EDX-Nb, Al, Ti, Nb, Mo, Cr
Data Correction Estimated Accuracy	None ± 20%	Standardless ZAF ± 10%
Other	Each line scan composed of 127 individual point analyses. Results used to determine Cmax, Cmin and SR	Cmax, Cmin and SR values determined from 35 random point analyses within a .63 cm x .63 cm (0.25" x 0.25") surface area region.

Microstructural characterization was carried out using optical and scanning electron microscopy. Samples of cast, cast-wrought and Osprey material were prepared by standard metallographic techniques to delineate grain boundaries and second phases.

Mechanical property testing was performed on flat discs, hot die forged by Wyman Gordon Company from preform numbers 393 and 21 supplied by General Electric Company and Osprey Metals, Ltd. respectively. An attempt was made to determine J_{IC} per ASTM-E813 for the nitrogen atomized preform #393 using a single specimen, cyclic loading method. Rapid and unstable crack growth occurred after only a few unloadings from which it was concluded that the Osprey 718 material was not fracturing with sufficient plasticity to allow J-Integral testing. Since slope, loading rate and initial crack length were also outside the requirements of ASTM E399 for a valid K_{IC}, K_Q values were calculated. In addition, K_{EE} values were determined per ASTM-E992. A fracture toughness test was also performed according to ASTM E399 to determine K_{IC} for the argon atomized preform #21. Precrack tunneling prevented obtaining a valid K_{IC}, therefore K_Q was calculated.

Results and Discussion

The general microstructural features of spray formed 718, as compared to as-cast and cast-wrought material, are shown in Figure I. The spray formed material is characterized by its equiaxed, fine grained structure and the complete lack of a prominent dendritic solidification pattern common to conventionally cast material. The average grain size was measured to be ASTM 6 with both grain boundary and intragranular precipitates appearing in the microstructure. X-ray mapping indicated these precipitates were Nb-rich and are believed to be MC carbides. The intragranular phases were on the order of 0.1 μm in size while those residing at the grain boundaries possessed a size on the order of 1 μm.

One of the more salient features of nitrogen atomized 718 is the increase in nitrogen content relative to the base material. In this case, spray forming increased the nitrogen level from 58 ppm to 288 ppm. This phenomenon has been reported by other investigators and appears not to have any detrimental effects on mechanical properties (1,3,6). However, during high temperature thermal treatments, nitrogen atomized 718 undergoes marked grain boundary precipitation as shown in Figure 2. X-ray mapping indicates the precipitating phases are titanium-rich and based on this observation, they are thought to be nitrides and carbonitrides.

A quantitative study of microsegregation in spray deposited 718 was performed using the electron microprobe. For purposes of comparison, samples from production size as-cast and cast-wrought material were also evaluated. One of the inherent difficulties in performing this analysis was that "solidification" patterns could not be defined in the spray formed material using available metallographic techniques. In contrast to a cast material, no prominent dendritic structure is observed. In fact, it has been proposed that solidification during spray deposition is cellular in nature but this has not been substantiated (7). Therefore, EMP line profiles were designed to traverse five grain diameters (≈ 250μm) in order to generate representative data. A similar technique was utilized for the evaluation of the conventional cast-wrought specimen. Because of the coarse dendritic solidification pattern in the as-cast material, line profiles were designed to traverse successive dendrite-interdendritic regions. To verify the results of the line profiles and to evaluate the method itself, an EMP random point method was employed. The difficulties encountered in performing line profiles were not of major concern using this technique.

The results of the electron microprobe study are presented in Table III. For each of the five elements, the results of this study are presented first and are followed by data from previous investigators for comparison. As might be expected from the microstructures, the calculated segregation ratios indicate spray formed and cast-wrought 718 are very similar on the basis of the degree of microsegregation present. While both EMP methods tended to give reasonable estimates of the microsegregation present in each sample, one will note that in almost every case, the random point method yielded smaller values of SR than those calculated from line profiles. These discrepancies may have been caused by the difficulty in interpreting the line profiles in the presence of second phase x-ray peaks, usage of uncorrected data from the line profiles, and the potential averaging effect associated with the large diameter microprobe beam utilized in the random point method.

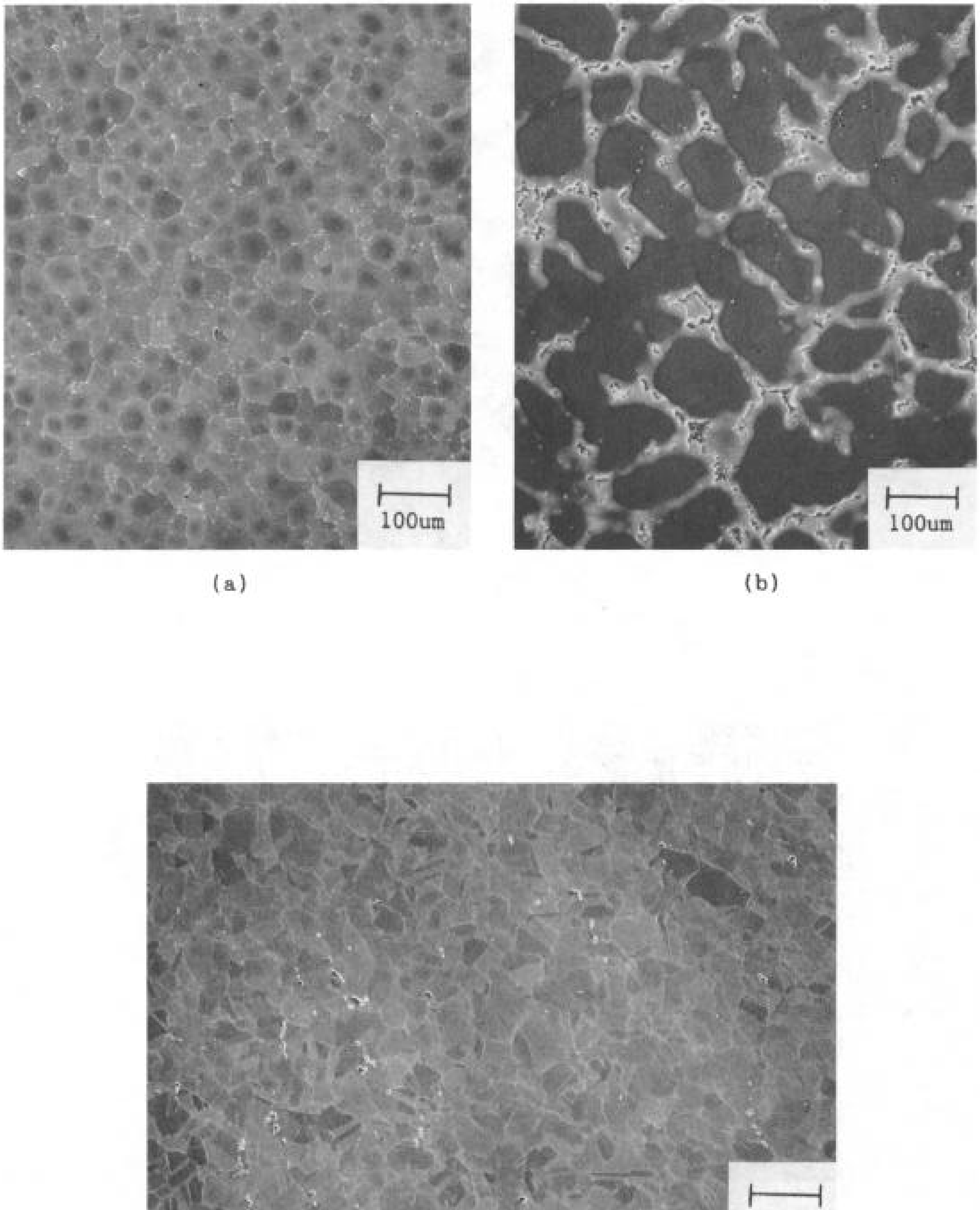
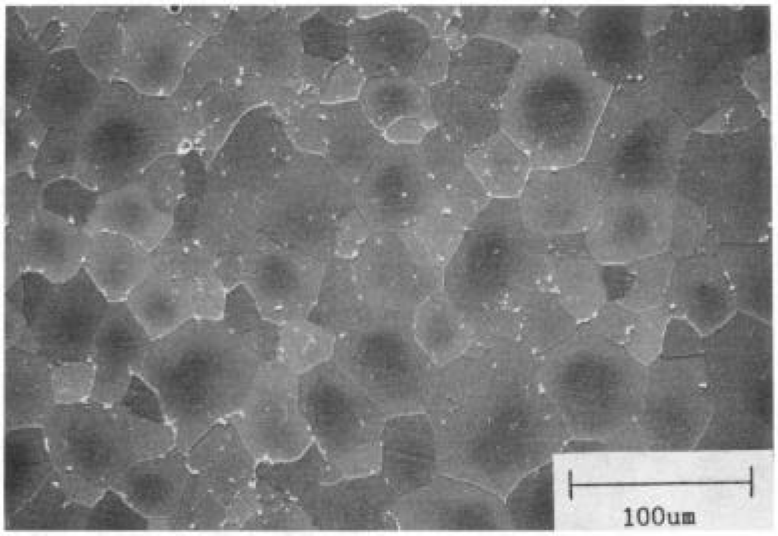
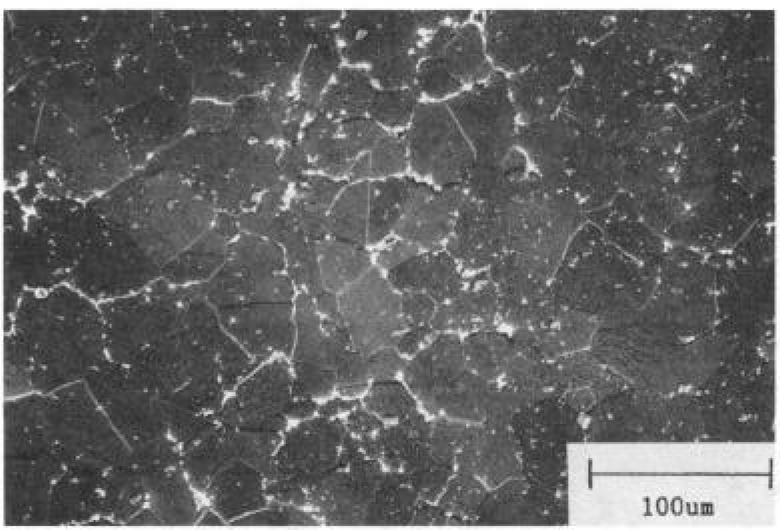


Figure 1 - Scanning Electron Micrographs of (a) As-spray Formed 718, (b) VIM/ESR As-Cast 718 and (c) VIM/VAR Cast-Wrought 718.



(a)



(b)

Figure 2 - High Temperature Grain Boundary Precipitation in Spray Formed 718: (a) As-sprayed and (b) As-sprayed + 1191°C (2175°F)/WQ.

Table III. Results of the Microsegregation Study on Spray Formed, As-Cast and Cast-Wrought 718

Element	Condition	Segregation Ratio (Cmax/Cmin)
Niobium	As-Spray Formed	
	a) Line Profile	1.57
	b) Random Point	1.36
	As-Cast (Center)	
	a) Line Profile	5.68
	b) Random Point	5.58
	Cast-Wrought (Center)	
	a) Line Profile	1.23
	b) Random Point	1.24
	As-Cast (Micro-Met Lab-Ref. 4)	5.10
	As-Cast (DeVries and Mumau-Ref. 5)	5.60
Aluminum	As-Spray Formed	
	a) Line Profile	2.00
	b) Random Point	1.19
	As-Cast (Center)	
	a) Line Profile	3.00
	b) Random Point	----
	Cast-Wrought (Center)	
	a) Line Profile	3.00
	b) Random Point	1.07
	As-Cast (Micro-Met Lab-Ref. 4)	2.31
	As-Cast (DeVries and Mumau-Ref. 5)	1.80
Molybdenum	As-Spray Formed	
	a) Line Profile	2.32
	b) Random Point	1.07
	As-Cast (Center)	
	a) Line Profile	3.62
	b) Random Point	1.51
	Cast-Wrought (Center)	
	a) Line Profile	1.88
	b) Random Point	1.08
	As-Cast (Micro-Met Lab-Ref. 4)	1.13
	As-Cast (DeVries and Mumau-Ref. 5)	1.60
Titanium	As-Spray Formed	
	a) Line Profile	6.50
	b) Random Point	1.82
	As-Cast (Center)	
	a) Line Profile	3.14
	b) Random Point	2.52
	Cast-Wrought (Center)	
	a) Line Profile	1.38
	b) Random Point	1.10
	As-Cast (Micro-Met Lab-Ref. 4)	2.14
	As-Cast (DeVries and Mumau-Ref. 5)	1.70

Element	Condition	Segregation Ratio (Cmax/Cmin)
Chromium	As-Spray Formed	
	a) Line Profile	1.05
	b) Random Point	1.07
	As-Cast (Center)	
	a) Line Profile	1.46
	b) Random Point	1.23
	Cast-Wrought (Center)	
	a) Line Profile	1.12
	b) Random Point	1.05
	As-Cast (Micro-Met Lab-Ref. 4)	1.18
	As-Cast (DeVries and Mumau-Ref. 5)	1.80

Niobium line profiles of the as-cast material revealed periodic concentration maxima and minima with an average spacing of approximately 80µm. For the cast-wrought and spray formed specimens, the periodicity was measured to be approximately 9µm and 2µm, respectively. It is interesting to note that even across grain boundary regions, the elemental profiles remained relatively flat. This observation tends to contradict the idea of heavy grain boundary segregation in spray deposited materials as reported by Leatham, et al (8), perhaps because of the small size of the preforms. In their model, solidification originates from either dendrite fragments or solid particles and occurs in a non-dendritic mode. Due to the turbulence in the liquid caused by the atomizing gas and impacting particles, local temperature and compositional homogeneity is maintained ahead of the advancing solidification front making dendritic growth difficult. However, as solidification continues, some degree of solute enrichment in the liquid occurs and ultimately manifests itself as grain boundary segregation.

In order to extend these microsegregation results to relate to large diameter billet size preforms, a computer simulation was performed at Drexel University based on an analytical model (9). Preliminary calculations were performed to select deposition conditions for manufacturing a 61 cm (24") diameter preform. Assuming a mean droplet diameter of 80 µm and a log-normal droplet size distribution, the percent liquid in the spray was calculated to be 25 weight percent for the selected operative flight distance of 30 cm (11.8"). On the basis of these calculations and prior experience, the conditions in Table IV were chosen for conducting the computer simulation.

Table IV. Osprey Deposition Conditions for Computer Simulation of 24" Diameter Alloy 718 Preforms

Melt Superheat = 75°C (135°F)
Gas:Metal Ratio = 1.0 Kg/m^3 (6.24 x 10^{-2} lbs/per ft^3)
Metal Deposition Rate = 0.5 Kg/sec. (1.1 lbs/sec.)
Nozzle height = 30 cm (11.8")
Spray Diameter ≈ 10 cm (3.94")
Substrate Rotational Frequency = 200 rpm
Mean Droplet Size = 80µm
Spray Liquid Content = 25%
Spray Temperature = 1271°C (2320°F)

Results of the modeling study suggest that for the conditions chosen and with a single nozzle, only one deposition pass was possible which gave a preform height of about 9 cm (3.5"). This resulted from the requirement to maintain incremental solidification (i.e. the solidification of the prior deposited layer cannot be completed before the subsequent layer is deposited) and thus minimize the formation of porosity.

The model also suggests that with increasing preform length and diameter, the centerline solidification times would continue to increase to a point limited by the preform diameter. However, macro and microsegregation should be severely restricted since 75% of the droplets at impingement are already solidified.

The tensile properties and fracture toughness values for Osprey 718 are presented in Tables V and VI respectively. Tensile results for argon and nitrogen sprayed preforms are similar to data reported elsewhere (3).

Although no valid K_{IC} results were obtained, the data are useful for comparison purposes with conventional cast-wrought 718. Based on discussions with test personnel (18) it is felt that for preform #393 K_{EE} values better represent the actual toughness of the material since K_Q values may be considered conservative under these circumstances, and thus a valid K_{IC} value for the nitrogen deposited alloy would be between 127.6 MPa $\sqrt{m}$ (115.2 ksi $\sqrt{in}$) and 197.9 MPa $\sqrt{m}$ (178.7 ksi $\sqrt{in}$). In contrast for the argon atomized preform #21, the K_Q value determined is slightly higher than K_{IC}, although probably by no more than 5.5 MPa $\sqrt{m}$ (5 ksi $\sqrt{in}$).

Cast-wrought alloy 718, processed according to aerospace heat treatments, typically displays K_{IC} values between 61.5 MPa $\sqrt{m}$ (55.6 ksi $\sqrt{in}$) and 188 MPa $\sqrt{m}$ (171 ksi $\sqrt{in}$) (10-16). In spite of this large variation in reported K_{IC} values, specified values of 109.9 MPa $\sqrt{m}$ (100 ksi $\sqrt{in}$) for aircraft engine applications are routinely achieved (17). Therefore, it may be concluded that the fracture toughness of Osprey formed alloy 718 is comparable to that of conventional cast-wrought material. Furthermore, there was little variation in K_Q or K_{EE} with specimen orientation for preform #393 indicating the fracture toughness of Osprey 718 is isotropic.

Table V. Room Temperature Tensile Properties: Osprey Alloy 718

Preform	Dir*	UTS (MPa)	UTS (ksi)	0.2% Y.S. (MPa)	0.2% Y.S. (ksi)	% EL	% RA	Rc
#393: N_2 Atomized	R	1,437	208.4	1,197	173.6	19.9	37.8	44
	R	1,440	208.8	1,215	176.2	20.4	33.2	
	C	1,436	208.2	1,202	174.3	19.8	34.3	
	C	1,448	209.9	1,221	177.0	17.8	29.8	
#21: Ar Atomized	R	1,422	206.2	1,163	168.6	15.3	18.6	42
	R	1.428	207.0	1,162	168.4	21.0	33.8	

Hot die forged 66% reduction at 1037°C (1900°F). Heat Treatment: 954°C (1750°F)/1 Hr/Oil Quenched; Double aged at 718°C (1325°F)/8 Hrs/Furnace Cooled @ 55.6°C (100°F)/Hr to 621°C (1150°F)/8 Hrs/Air Cooled.
*R = Radial C = Circumferential

Table VI. Fracture Toughness of Osprey Alloy 718

Preform	Specimen Orientation	Specimen Type*	K_Q MPa $\sqrt{m}$	K_Q ksi $\sqrt{in}$	K_{EE} MPa $\sqrt{m}$	K_{EE} ksi $\sqrt{in}$
#393: Nitrogen Atomized	C-R	1.000 C(T)	135.0	121.9	166.8	150.6
	R-C	1.000 C(T)	141.3	127.6	178.3	161.0
	R-L	0.5 S(B)	127.6	115.2	197.9	178.7
#21: Argon Atomized	C-R	1.500 C(T)	123.4	112.3	--	--

* C(T) = Compact tension specimen; S(B) = Standard bend specimen

Conclusions

1. Spray deposited 718 exhibits a unique combination of characteristics including a refined equiaxed grain structure, near-zero macrosegregation, and good mechanical properties.

2. In the as-sprayed condition, nitrogen atomized alloy 718 preforms possess a fine distribution of both intergranular and intragranular second phase precipitates. X-ray mapping indicates these precipitates are Nb-rich and are thought to be MC-type carbides.

3. Atomizing alloy 718 with nitrogen resulted in a pickup of 230 ppm nitrogen. This increased nitrogen content appears to promote intergranular precipitation of Ti-rich carbides and carbonitrides during high temperature thermal treatments. As expected, the nitrogen content of the argon atomized material was lower (72 ppm), comparable to cast-wrought product.

4. Microsegregation, as measured by the "segregation ratio", in as-sprayed 718 was found to be comparable to that exhibited by conventional cast-wrought material.

5. In the upset and double aged condition, fracture toughness of Osprey 718 is equivalent to aerospace quality cast-wrought material, and is isotropic.

Acknowledgements

The authors would like to acknowledge Dr. Lori Streit of McCrone Associates, Mr. Ed Raymond and Dr. S. V. Thamboo of Cameron Forge Company and Mr. Mickey Gregory of the NCSU Analytical Instrumentation Facility for their contributions involving electron microscopy. We are also grateful to Dr. J. F. Radavich of Micro-Met Labs for his consultation on metallography and structure. Special thanks goes to Dr. H. C. Fiedler of the General Electric Company and Dr. Alan Leatham of Osprey Metals, Ltd. for providing spray deposited 718, Red Couts, Wyman-Gordon, for forging and heat treatment, Professor D. Apelian and Dr. P. Mathur of Drexel University for computer modeling of the Osprey Process, and Mssrs. J. D. Rossi and T. S. Fedor of Westmoreland Mechanical Testing and Research, Inc. for performing fracture toughness tests and for their enlightening discussions.

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