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DIRECTIONALLY SOLIDIFIED LAMELLAR EUTECTIC SUPERALLOYS BY EDGE-DEFINED, FILM-FED GROWTH

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16. Abstract
A program was performed to scale up the Edge-Defined, Film-Fed Growth (EFG) method for the γ/γ'-δ eutectic alloy of the nominal composition Ni-19.7 Cb - 6 Cr-2.5 Al. Procedures and problem areas are described. Flat bars approximately 12 × 1.7 × 200 mm were grown, mostly at speeds of 38 mm/hr. Tensile tests on these bars at 25 and 1000°C showed lower strength than expected. The Feasibility of growing hollow airfoils was also demonstrated by growing bars over 200 mm long with a teardrop shaped cross-section, having a major dimension of 12 mm and a maximum width of 5 mm.

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SUMMARY

In a previous investigation, the Edge-Defined, Film-Fed Growth (EFG) method was shown capable of processing nickel and cobalt base superalloy eutectics yielding aligned eutectic structures. In the present investigation, one of the objectives was to scale up the size capability to 12 × 1.7 mm flat bars and to carry out tensile testing of these bars. A second objective was to demonstrate the feasibility of growing hollow airfoils. This was done by growing hollow bars with a teardrop cross-section. The major dimension or chord was 12 mm and the leading edge diameter was 5 mm. Hollow and solid bars were grown over 200 mm long. All bars were produced from a nickel base alloy containing 19.7% Cb, 6% Cr, 2.5% Al and balance nickel which forms a eutectic of the γ/γ'-δ phases.

The approach to the growth of the flat bars was simply to scale-up the size of the TaC die and the crucible from the previous investigation. This scale-up was made horizontally so that the capillary rise distance was kept small. The die length was 2.5 cm. The growth rate of the bars was mainly 3.8 cm/hr. The major difficulties encountered concerned a new problem in die clogging and an apparent variability in starting alloy composition. Die clogging was found to result from solution-precipitation growth of carbide particles in the die slot and was alleviated by enlarging the slot.

Hollow teardrop shaped bars were also grown from TaC dies. These dies were constructed from two pieces. The larger piece was Vee-shaped and contained three feed slots, one in the bottom of the Vee, and one in each side. The center of the Vee was ground out for 2/3 the length to provide a blind cavity. The second die part was a half round section which fitted over the top part of the Vee. The major difficulties in growth resulted from failure of the liquid to flow over this front section (the half-round piece) or from accidental bridging of the blind cavity. The spreading problem

was solved by cutting shallow slots in the top surface of the front of the die to aid in conducting liquid from the feed slots. The problem of bridging was alleviated by either venting the cavity or by providing a non-wetting plug. Growth was carried out at 2.5 cm/hr.

Two tensile tests were carried out each at 25 and 1000°C. Maximum ultimate strengths were 124×10^7 N/m^2 and 63.7×10^7 N/m^2 respectively. Strain at failure averaged less than one percent. The low strength and ductility may have been due to not fully aligned structures and carbide precipitates.

I. INTRODUCTION

Directional solidification of eutectic superalloys is an extension of modern directional solidification of superalloys by which jet engine parts, particularly blades, can be produced with a shape close to that finally required. The processing technique itself produces an aligned structure of two or more phases which impart strengthening according to the principles of composite materials. Virtually all eutectic type alloys of potential interest are multicomponent nickel or cobalt based alloys for which large ratios of liquid temperature gradient-to-growth rate are required to produce suitable structures. This requirement in turn has led to high melt superheats, mold and core compatibility problems, and rather exotic chilling techniques in order to maintain acceptable growth rates.

The edge-defined, film-fed growth technique (EFG) is an alternative method of producing the required shaped structures and has the potential for increasing the growth speed at lower cost. The nature of the EFG process and a description of its application to eutectics processing was given in an earlier report (Ref. 1). That report also documented a study to identify die materials which could be used for the EFG growth of nickel or cobalt base systems such as the γ/γ'-δ alloy.

The objectives of the present study have been to grow larger specimens of the γ/γ'-δ alloy suitable for tensile testing, to carry out tensile tests at room temperature and 1000°C, and to grow hollow bars shaped to resemble the important characteristics of a turbine blade. In this study, no attempt was made to optimize the thermal gradient or to develop a die material more stable than tantalum carbide.

II. EXPERIMENTAL PROCEDURE

2.1 Alloy and Die Preparation

Growth trials were conducted using only the $\gamma/\gamma' - \delta$ alloy with the nominal composition Ni - 19.7 Cb - 6 Cr - 2.5 Al. Pre-alloyed feed stock for this program was prepared by the General Electric Co., Corporate Research and Development Center, Schenectady, N.Y., from the starting materials whose analyses are given in Table I. The feed stock was cast into chilled molds yielding five 3.8 cm diameter ingots. This ingot size was a convenient fit for the i.d. of the crucible used in the growth trials.

Dies were machined from tantalum carbide blanks prepared from the material whose analysis is given in Table I. The blanks were hot pressed at Fiber Materials Inc. as discs having densities approximately 96% of theoretical.

2.2 Growth Setups

Growth was carried out using machined tantalum carbide dies held by means of alumina supports. The melt was contained in an alumina crucible and heating was provided by a 450 kHz rf generator suscepting to a molybdenum susceptor. The growth furnace was a double wall water-cooled quartz chamber held in a motor driven crystal puller. This apparatus was substantially as described previously.[1]

The growth set-up was enlarged compared to that used previously in order to incorporate a wider die and an increased liquid reservoir to grow bars of the specified length. The enlargement for increased melt volume was made by increasing the crucible diameter (to 3.8 cm) rather than the height. The die length was kept at approximately 2.5 cm as before.

Table I. Purity of Charge Elements and Die Material, ppm.

	C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	Mg	Co	B	Ca	Cd	H	Hf	Mo	N	O	Pb	Sn
Nickel[1]	100	< 10		40	< 10	< 10	Bal.	< 10	-	< 10	< 10	< 10	-	-	-	-	-	-	-	-	-	-
Columbium[2]	< 30	< 20	< 50		< 50	< 40	< 20	< 20	< 20	< 40	< 20	< 10	< 1	< 20	< 50	< 5	< 50	< 20	25	70	< 20	< 10
Chromium[3]	150	-	3500	60	190	-	-	Bal.	70	-	-	-	-	-	-	-	-	-	120	470	-	-
Aluminum	-	10	10	20/40	-	-	-	-	Bal.	-	-	-	-	-	-	-	-	-	-	-	-	-
Tantalum Carbide[4]	9000	-	190	-	-	-	-	-	-	< 50	-	-	-	-	-	-	-	-	-	-	-	-

	Ta	V	W	Zr	Ga	Cb
Nickel	-	-	-	-	-	-
Columbium	200	< 20	22	< 100		Bal.
Chromium	-	-	-	-	-	-
Aluminum	-	-	-	-	100/500	-
TaC	Bal.	-	-	-	-	-

[1]Inco, Huntington Alloy Div.

[2]Dynamet, Inc., Specialty Metals Div.

[3]Union Carbide Corp., Ferroalloys Div., Mining and Metals Div.

[4]Herman C. Starck, Berlin.

2.3 Dies

Two die configurations were used. In the first part of the program, dies to produce approximately 12 × 1.7 mm rectangular cross-section bars were used. In the second part, dies were designed to produce a hollow teardrop (airfoil) shaped bar approximately 12 mm in the major dimension, with a 5 mm leading edge diameter.

Two views of the final design die used to grow flat bars are shown in Fig. 1. Externally, the die design is the same as that used earlier. However, the width and depth of the die feed slots were increased during the set-up and control runs to overcome a feeding problem.

The design of the die for hollow bar growth is shown in Fig. 2. This die is two-piece with feed slots provided only in the large Vee-shaped section. In practice no feeding difficulties were encountered but the liquid could not always be spread completely around the die top. Accordingly the rounded section of the die was modified as shown in Fig. 2c with 1.3 mm deep slots to improve the spreading characteristics.

2.4 Growth Experiments

Crucibles were charged with the pre-alloyed feed stock prior to the start of each growth run. This procedure was carried out by cutting off a slice of the feed stock the length of the crucible (~12 mm), cutting this into two half discs to fit around the die in the crucible.

Seeds were prepared by cutting feedstock into flat plates and welding to a steel bar to be held into the seed holder. In the case of the hollow bar growth trials, seeds were prepared by welding together three plates together with a rod for support to form a triangular seed which fitted over the die top. In either case, the set-up was heated to melt the liquid in the crucible after which the seed was melted back on the die top to connect to the liquid in the feed slots. Growth was initiated by withdrawing the seed, usually at 2.5 or 3.8 cm/hr, while adjusting the temperature to maintain the liquid at the edges of the die.

2.5 Tensile Tests

Duplicate tensile tests were carried out at room temperature and at 1000°C. Samples for testing were prepared using a Tensilkut machine and grinding the sides by hand. The samples were flat, half scale (25 × 6.3 mm gauge length) tensile samples. Room temperature tests were conducted at a strain rate of 0.01 min^{-1} and strain was measured with an extensometer to allow modulus calculations. Tests at 1000°C were carried out with a strain rate of 0.05 min^{-1}.

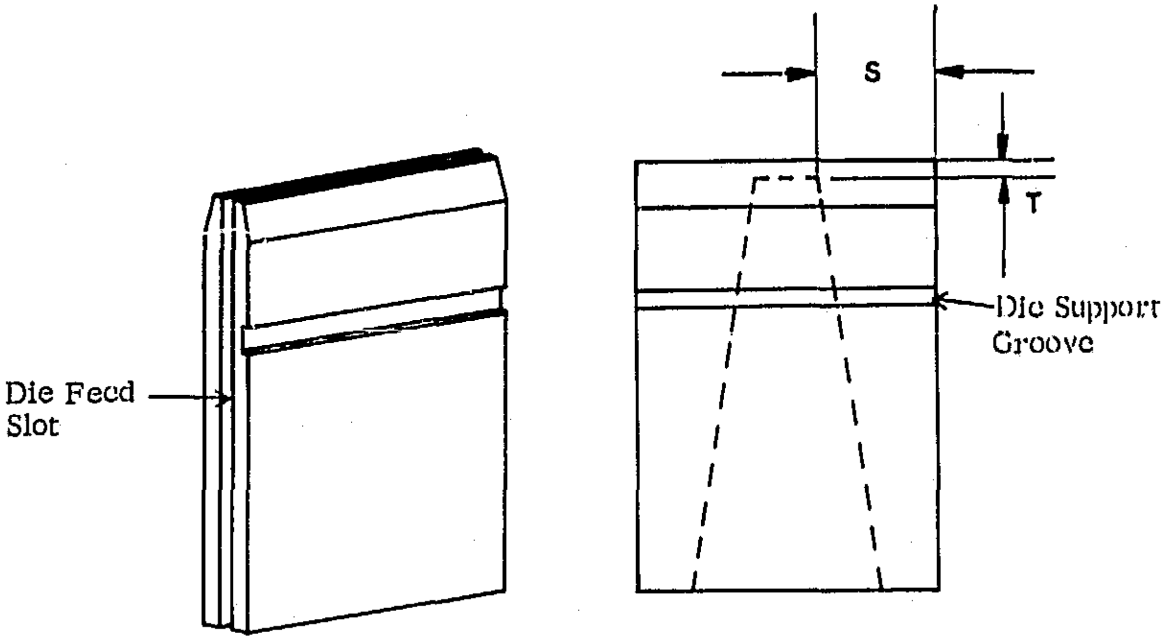

Fig. 1. Die configuration for growth of flat bars. Key dimensions S and T are referenced in Section III, Table III. Overall die dimensions were 25 × 3.9 × 12.5 mm.

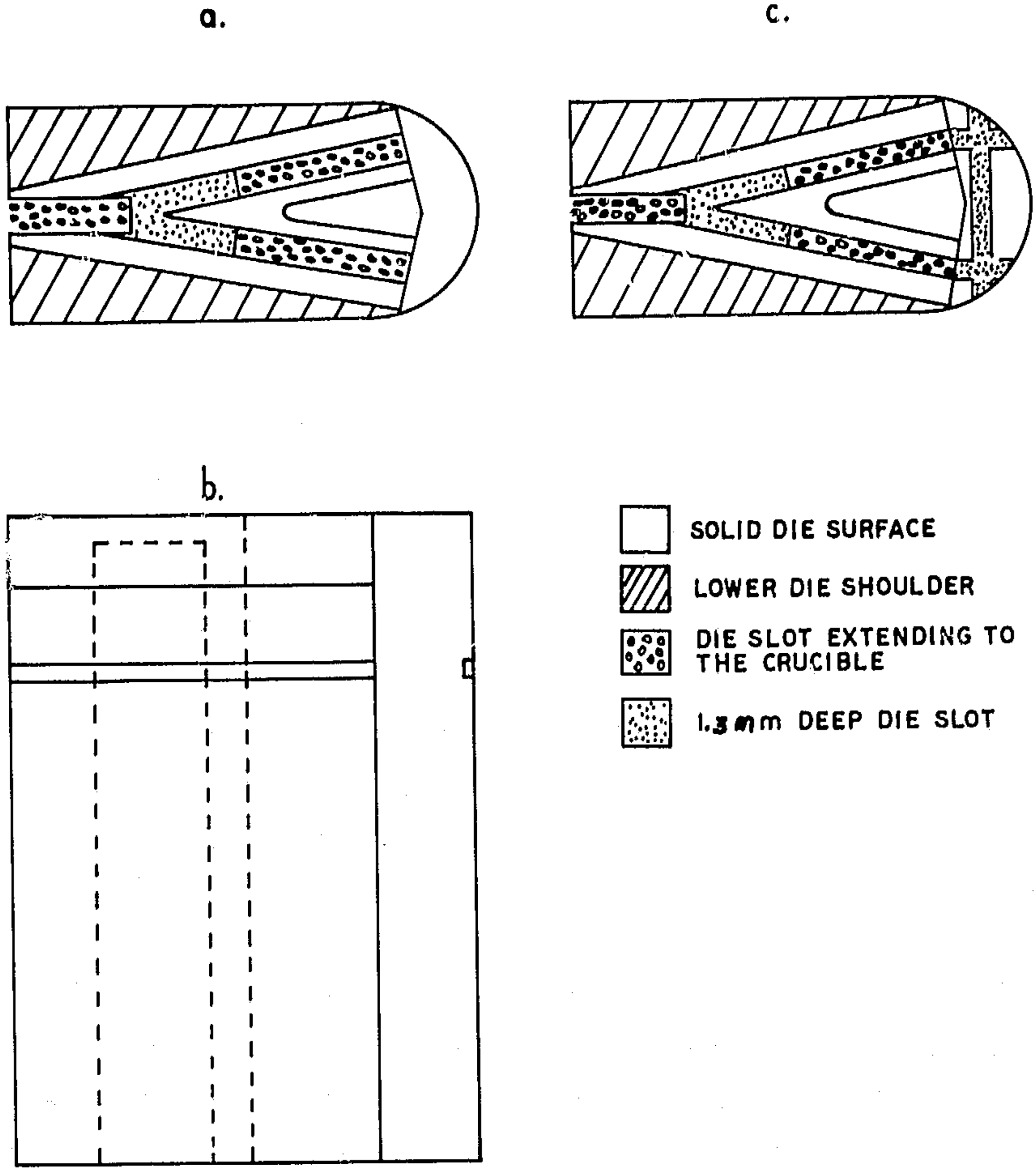

Fig. 2. Design of shaped bar die. a. and b. before modification, c. after modification. Overall die dimensions were 12 × 5 × 25 mm.

III. RESULTS

3.1 Chemical Analyses

Chemical analysis was carried out on samples selected from different locations in the pre-alloyed feedstock materials.* The quantitative analysis for Al, Cb, and Cr was carried out by atomic absorption analysis after which a qualitative analysis was used to estimate total impurities. Nickel was then taken as the difference. The X's in the diagram given in Fig. 3 indicate the location of samples taken for the first series of analyses.

The results of these first analyses are given in Table II. These values showed both discrepancies from nominal compositions and variability among samples. During the initial growth trials, certain inconsistencies in the structural characteristics of the growth samples appeared which also suggested a varying composition. A second series of samples was subsequently selected for analysis and is identified by the O's in Fig. 3. Results of the second analysis, given in Table II, were obtained by analysis of solutions containing 10% HF compared to the 3% used in the first series of samples. The results of the second series also showed compositional error as well as variability. However, the first two entries in the table were analyses of samples from a single transverse slice of an ingot. Microscopical inspection of similar slices showed seemingly homogeneous fine grained microstructure in the horizontal plane. No conclusions could be drawn without further extensive analysis including carefully prepared composition standards. Since this was beyond the scope of this contract, no further investigations of this type were carried out.

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*Chemical analysis was conducted by Jarrell Ash Division of Fisher Scientific Co., Waltham, Massachusetts 02154.

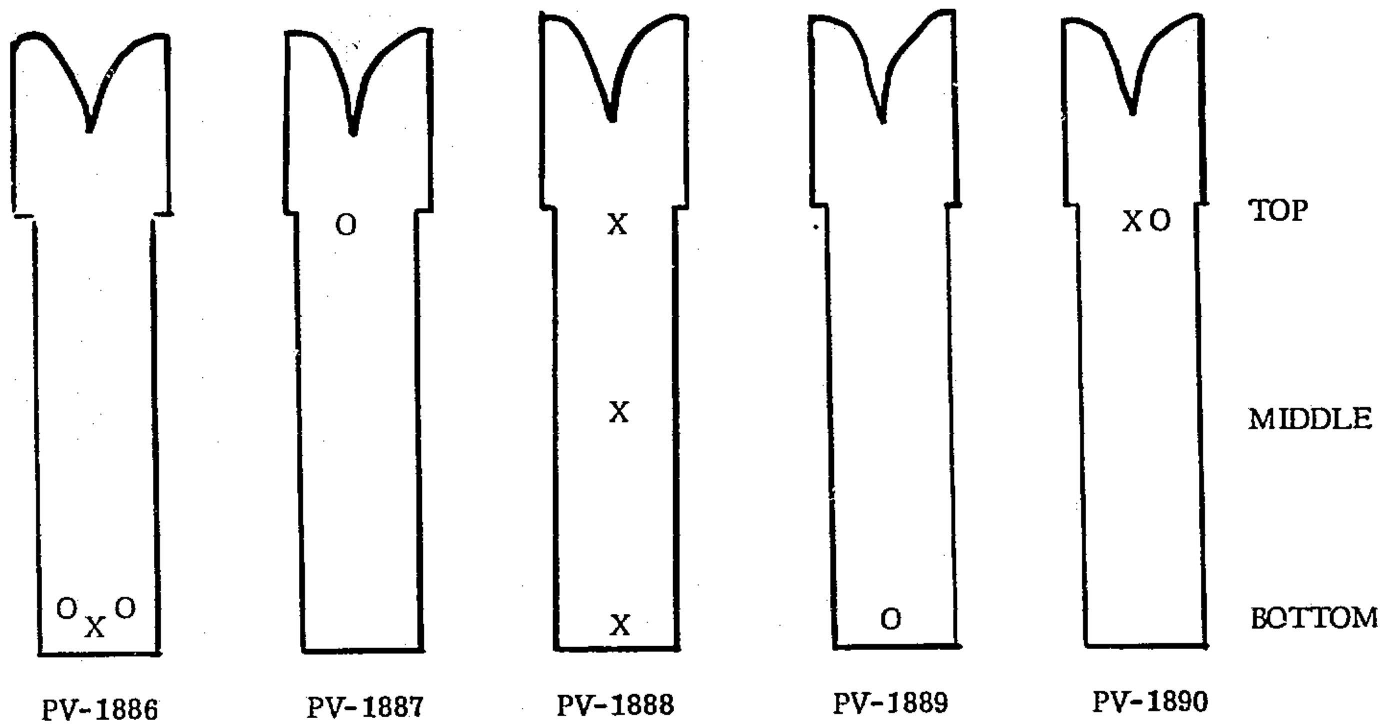


Fig. 3. First series of samples taken for chemical analysis are shown by X, second series of samples, by O

Table II. Results of Two Series of Chemical Analyses to Determine Composition of Starting Material. "X" Denotes First Series, "O" Denotes Second Series. See Fig. 2 for Sample Locations.

Sample	% Al	% Cr	% Ni	% Nb	Series
PV 1886-2 Bottom	1.6	5.8	74.8	18.5	O
PV 1886-2 Bottom Middle	1.1	5.9	75.9	19.2	O
PV 1886 Bottom	2.4	5.2	68.07	23.1	X
PV 1887-1 Top	1.2	5.6	77.9	17.2	O
PV 1888 Top	2.5	5.2	72.87	19.3	X
PV 1888 Middle	2.3	5.4	68.96	23.2	X
PV 1888 Bottom	2.1	5.9	72.36	19.5	X
PV 1889-2 Bottom	0.97	4.8	75.5	17.6	O
PV 1890-2 Top	1.4	5.9	74.9	18.6	O
PV 1890 Top	2.2	5.3	75.01	17.3	X

3.2 Growth of Flat Bars

Ten experimental growth runs were carried out to establish the best set up parameters and growth procedures for the flat bar production task. Primarily it was anticipated that this phase of the investigation would be used to establish die height and shielding adjustments to obtain proper growth conditions. After growth runs had been started, however, it was determined that the major problem area was associated with premature die failure exhibited by poor liquid feeding.

The construction of the growth set-up used in these runs is shown in Fig. 4. The alumina spacers supporting the die are nonsymmetric so that either of two die positions could be attained simply by reversing the spacers. Initial experimentation was mainly carried out using the lowest die position, but the higher position was eventually adopted as standard.

The ten experimental runs are listed in Table III. In the first run, the alumina spacers (Fig. 4) were inserted so as to position the die in the higher of two possible positions. Growth here was relatively uncontrolled but the structure was found to be aligned lamellae. In the second run, the alumina spacers were observed to lower the die, also resulting in acceptable structure, and this lower configuration was adopted as standard. Growth rate was held at 2.5 cm/hr in both cases.

In each of the first two growth runs, the sample became thinner in the thickness direction after about 10 cm had been grown. This phenomenon suggested poor feeding which was also indicated by metallographic sections of the die slots. A representative section of a failed die is shown in Fig. 5. These die sections, after use, disclosed that the feed capillaries had become clogged by rather large blocky particles within the slots. The appearance of the particles was different from pure TaC. Further, the edges of the capillary feed slots were coated with material having the same color as the blocky phase. This coating material is thin or missing at the bottom of the capillary and thickest at the top. These observations suggest that the die may interact with the liquid. The dissolved carbide is transported in the melt, and as the result of a negative temperature gradient precipitated as a blocky phase which obstructs the capillary.

The TaC used to fabricate dies for the present investigation was approximately 96% of theoretical density. The metallography carried out to determine the cause of die failure showed that the pore phase in the carbide was isolated and did not fill with metal. Also, penetration of the liquid into the grain boundaries was not evident here

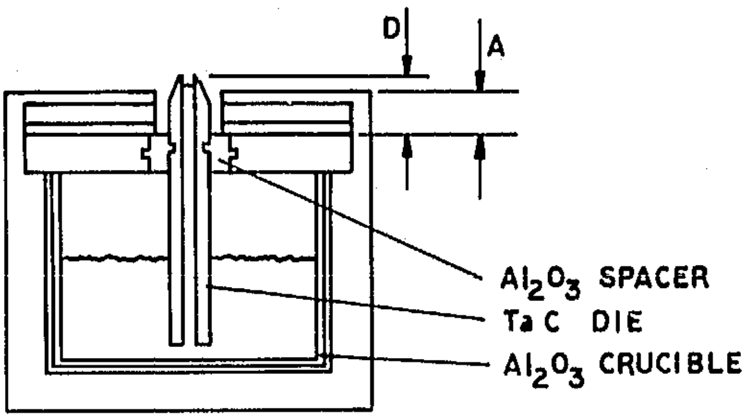


Fig. 4. Growth set-up used for flat bar growth

Table III. Set-Up and Growth Runs

Run No.	Set-Up Dimensions (cm) see Fig. 2		Die Parameters (cm) see Fig. 1		Growth Rate (cm/hr)	Die No.	Die Life (hrs)	Comments
	A	D	S	T				
RE-1	0.485	0.175	0.038	0.127	2.5	1	4.3	Growth uncontrollable
RE-2	0.269	0.157	0.038	0.127	2.5	2	5	-
RE-3	0.269	0.157	0.063	0.165	2.5, 3.8	3	-	-
RE-4	0.269	0.157	0.063	0.165	2.5	3	5	No cap shield
RE-5	0.269	0.157	0.063	0.165	2.5	4	3.5	No cap shield, H_2 cooled
RE-6	0.269	0.157	0.063	0.425*	4.4	5	-	Bar tapered after 9.5 mm, H_2 cooled.
RE-7	0.269	0.157	0.063	0.425*	5.0	5	-	Tapered
RE-8	0.269	0.157	0.063	0.425*	5.0	5	-	Tapered
RE-9	0.269	0.157	0.063	0.425*	Not recorded	5	6.75	Tapered
RE-10	0.269	0.157	0.063	0.425*	3.8	6	6	All die slots widened to 0.061 cm

*The grooves here were tapered from 0.165 cm at the bottom to 0.425 cm at the top. Sell also Fig. 4.

Fig. 5. Photomicrograph of section through the feed slot in die No. 2 (X 27)

as in the < 90% dense materials used previously. Cracks were occasionally visible and the cracked die had interacted with the liquid as evidenced by a color change from brassy yellow to white.

Since neither the die clogging problem nor the carbide color change had been noted in the previous investigation we sectioned three dies from the past program which had seen service for 5, 3, and 1.5 hrs respectively. At 1.5 hrs, no coating, per se, was visible, but a gradation in color existed from outside to inside, on each die half such that material closest to the liquid was white and the remainder grainy yellow. No particles larger than the carbide grain size were visible in the capillary. At 3 hrs and 5 hrs, the die was the uniform white color and blocky particles were formed in the capillary. Indications are that the porous structure in these dies allowed rapid formation of the white phase throughout the bulk. Since the present high density material does not allow liquid infiltration, the reaction is rate limited by diffusion across the layer as it is formed. Also, the presence of porosity in the dies used previous probably was responsible for continued feeding despite clogging in main die slots.

The analysis of the die failure suggests that the problem could be eliminated by one or more of the following:

1. Reducing the temperature gradient along the die which is assumed to be the driving force for the carbide precipitation in the feed slots.

2. Increasing the speed of growth in order to grow a longer length bar in the space of the actual die life.

3. Increasing the cross-section of the capillary feed slots in order to permit feeding despite the growth of carbide.

Since investigation of means to implement the first of the proposals would be beyond the scope of this program, the decision was made to increase the cross-section of the die slots. Since cooling by means of gas flow against the sides of the growing ribbon is known to increase the gradient, this technique was implemented to ensure that the growth rate increase did not lead to deterioration of the aligned eutectic structure.

These changes were carried out during the following 8 experimental runs. The most satisfactory die life was attained in RE-10 when the slot had been both widened and deepened.

Twelve growth runs were carried out to complete the production runs of 10 bars. These runs with pertinent details are reported in Table IV. Twelve of the last thirteen growth runs are shown photographed in Fig. 6.

Table IV. Production Run Parameters and Results

Run No.	Set-Up	Growth Speed (cm/hr)	Total Length (mm)	Width (mm) max	Width (mm) min	Thickness (mm) max	Thickness (mm) min	No. of Pieces**
PR-1	Z*	3.8	165	13.0	11.6	2.7	0.4	1
PR-2	Y†	4.4	206	13.0	11.3	2.2	0.4	1
PR-3	Y	4.4	115	11.8	9.3	1.6	0	3
PR-4	Y	3.8	229	12.7	7.1	1.9	1.2	2
PR-5	Y	3.8	290	12.8	9	1.9	1.3	1
PR-6	Y	3.8	175	13.3	11.8	2.0	1.8	1
PR-7	Y	3.8	222	13.0	12.5	2.4	2.2	1
PR-8	Y	3.8	212	12.8	12.0	2.5	1.2	1
PR-9	Y	3.8	106	13.0	12.6	2.9	1	4
PR-10	Y	3.8	228	12.8	12.5	2.6	1.7	1
PR-11	Y	3.8	207	12.6	11.8	2.5	0.6	1
PR-12	Y	3.8	192	12.7	12.3	2.5	1.3	1

*Set-up designated "Z" has the die in the lower position, below the heat shield by approximately 0.11 cm.

†Set-ups designated "Y" have the die in the higher position, above the heat shield by approximately 0.17 cm.

**No. of pieces grown from the same die.

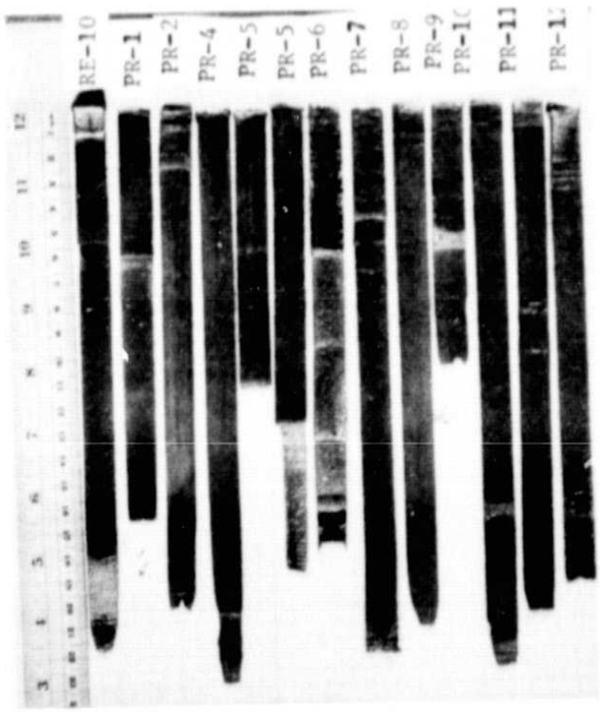


Fig. 6. Photograph of PR series growth runs

As noted above, the only significant change which occurred durir these runs was raising the die to the higher of two possible positions. This change was made for two reasons: first because die clogging considerations dictated a fast growth rate and, second because the evident variability of composition made it clear that the steepest possible gradient was required to obtain aligned structures. The higher die position is known to increase the gradient. Gas cooling was not used during these runs.

3.3 Structure of Flat Bars

Metallography was carried out on the experimental samples as a check on the effect of set-up variables and growth speeds. It was determined that proper aligned lamellar structures could be attained for growth speeds in the 2.5 to 3.8 cm/hr range. However, the results were inconsistent in that samples grown from identical set-ups at the same growth speeds failed to reproduce the same structure. Fig. 7a and b, for example, shows the structure of two samples grown under identical conditions at the same growth speed. The conclusion drawn from these results was that the alloy feedstock was not a uniform composition.

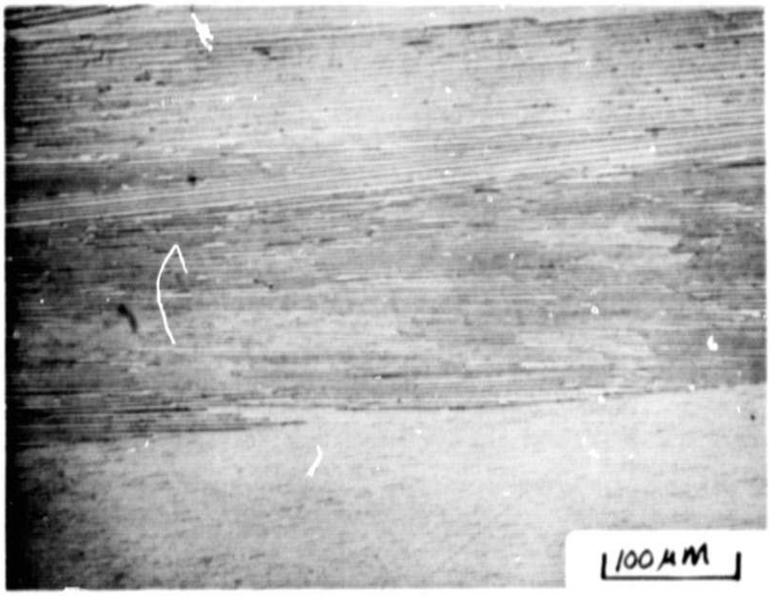
All of the PR series samples, except for PR-10, were sectioned at about 40 mm from the last-grown end, and two at about the same distance from the seed end. Inspection of these samples gave the results listed in Table V with the description indicating the major structural appearance. Most samples were either lamellar or partly cellular. Representative microstructures are shown in Figs. 8 to 10.

3.4 Tensile Testing

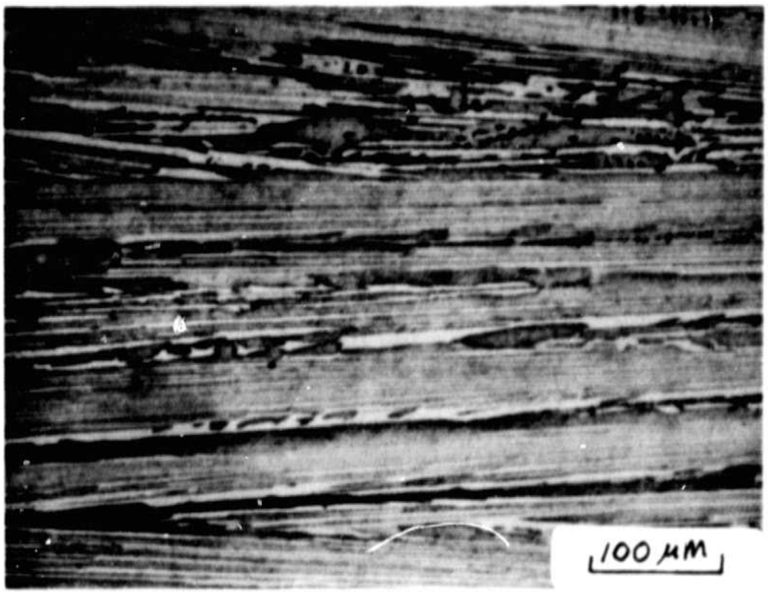
The mechanical tests were completed with results as shown in Table VI. The low ductility compared to that expected for this alloy is attributed to the TaC particles or to imperfectly aligned structures. However, fracture surface of two samples, No. PR-11 and PR-11 T were examined (Fig. 11) but did not indicate the role of TaC particles in the fracture. Note too, that PR-11 T, the worst sample, was from the first-grown end of the bar and would have been expected to contain fewer particles. Both samples appeared to contain some porosity.

3.5 Growth of Hollow Airfoil Shaped Bars

Growth runs were carried out with the basic set-up used for the flat bar growth, modified in two respects. The upper most heat shield in Fig. 4 was omitted to provide better visibility. In addition, the two-piece die required an additional



(a) RE-2



(b) RE-3

Fig. 7. Longitudinal sections of $\gamma/\gamma' + \delta$ solidified at 2.5 cm/hr

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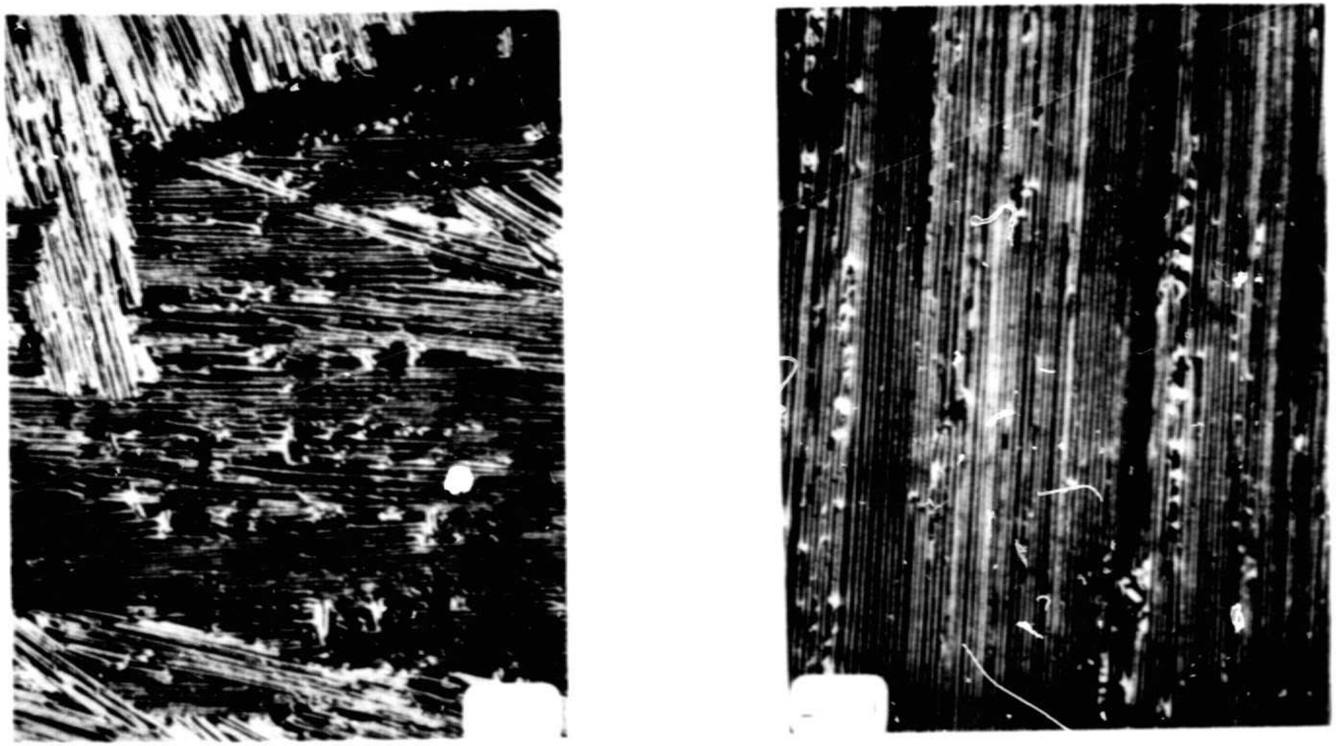
Table V. Results of Metallographic Inspection of PR Series Flat Bars

Sample No.	Location	Results
PR-1	Bottom*	Lamellar
PR-2	Bottom	Lamellar
PR-2	Top*	Lamellar, one side not aligned
PR-3	Bottom	Lamellar
PR-4	Bottom	Partly cellular
PR-5	Top	Partly cellular
PR-5	Bottom	Cellular
PR-6	Bottom	Partly cellular
PR-7	Bottom	Cellular
PR-8	Bottom	Lamellar
PR-9	Bottom	Cellular
PR-11	Bottom	Cellular
PR-12	Bottom	Partly cellular

*Bottom or top refer to last-grown or seed ends, respectively.



a. Transverse

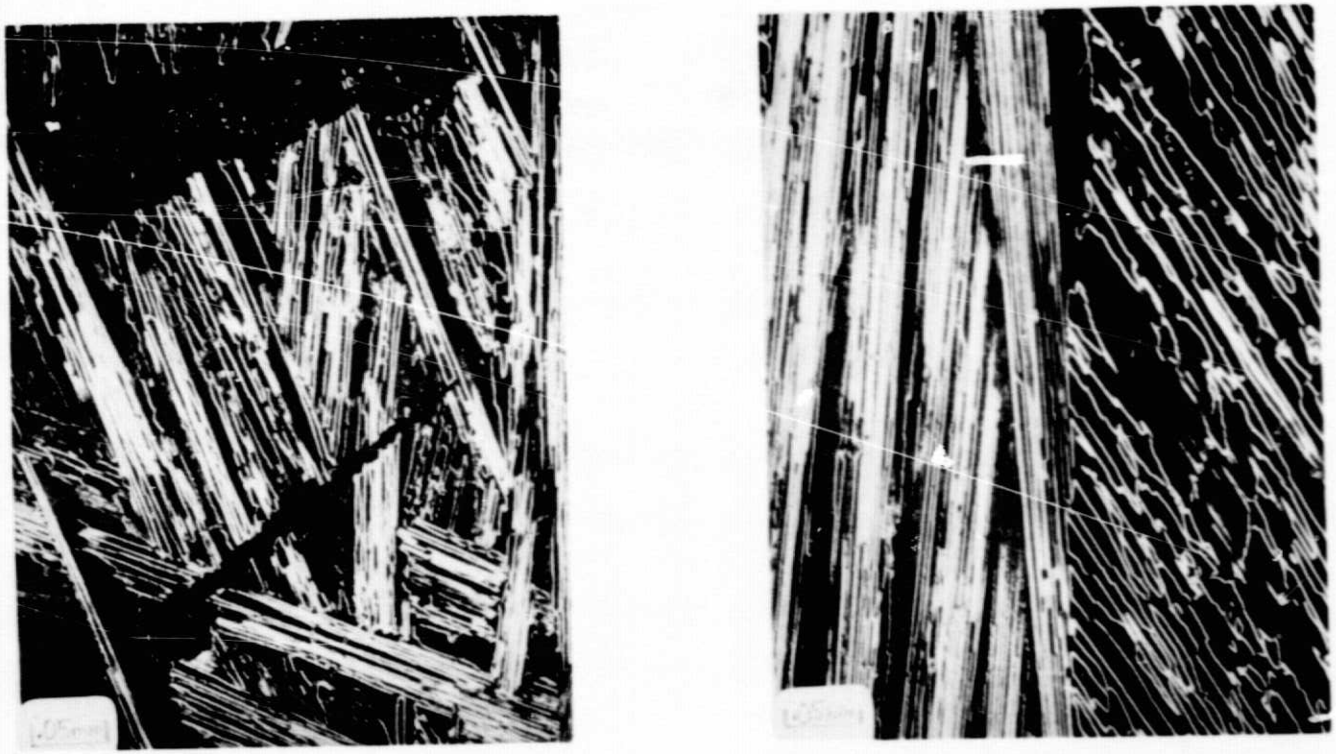
b. Longitudinal

Fig. 8. Sample No. PR 1B. Aligned structure

a. Transverse

b. Longitudinal

Fig. 9. Sample No. 6B. Partly cellular structure



a. Transverse

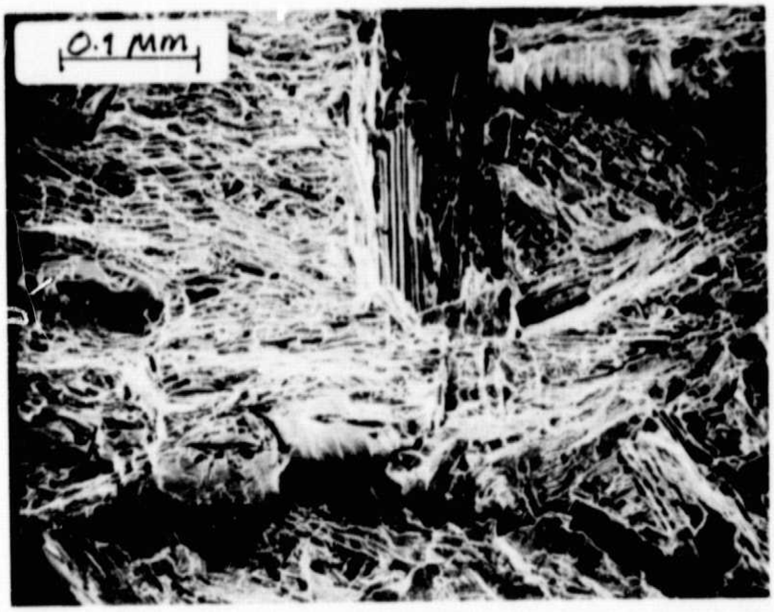
b. Longitudinal

Fig. 10 Sample No. 9B. Cellular structure

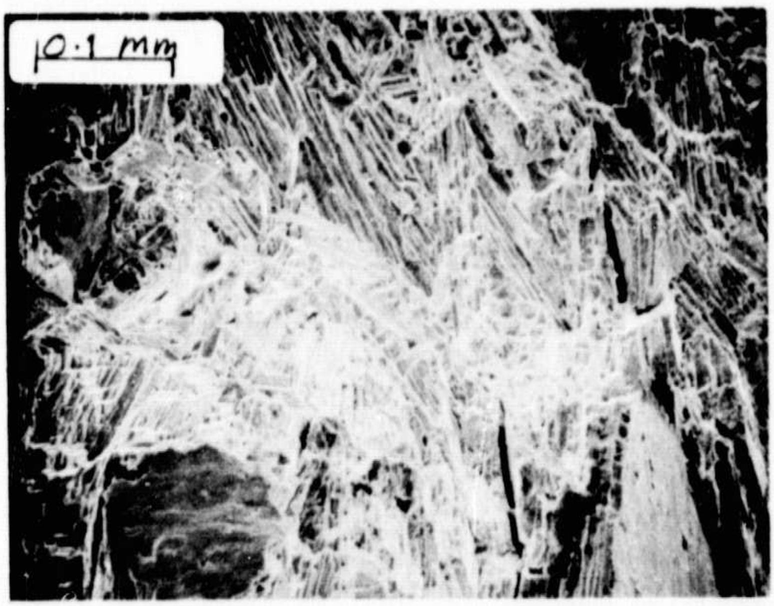
Table VI. Tensile Test Results. Room Temperature Tests were Carried Out at 0.01 min^{-1} and Elevated Temperature Tests at 0.005 min^{-1}

Sample	Growth Rate	Test Temp.	σ YS $\times 10^7$ N/m^2	σ YS KSI	σ UTS $\times 10^7$ N/m^2	σ UTS KSI	Plastic Strain at Failure	Modulus $\times 10^{10}$ N/m^2	Modulus $\times 10^6$ psi
PR-11	3.8 cm/hr	20°C	-	-	124	180	0.1%	26.6	38.6
PR-12	3.8 cm/hr	20°C	-	-	75.9	110	0.02%	26.8	38.9
PR-11T	3.8 cm/hr	1000°C	-	-	54.4	78.8	0.4%	-	-
PR-8	3.8 cm/hr	1000°C	61.5	89.1	63.7	92.3	1.2%	-	-
UARL*	3 cm/hr	20°C	119	172	123	179	3.6%	25.1	36.4
UARL	3 cm/hr	1000°C	73.5	107	75.5	110	10%	7.5	10.9

*Average values (Ref. 2).



(a)



(b)

Fig. 11. Fracture surfaces of a) PR-11T, and b) PR-11

alumina support to hold the rounded die portion in the proper vertical plane while two more pieces were used to hold the two die parts together. A top view of an assembled set up is shown photographed in Fig. 12.

Then growth runs were carried out and resulted in three bars over 200 mm long two of which were hollow. In addition, several shorter growth runs were carried out in which partly hollow, or partly spread but hollow, bars were grown as summarized in Table VII.

The various set-up parameters which were experimentally adjusted are listed in Table VIII and in the last column of Table VII. The first growth run was successfully seeded with material spread completely over the die top. However, the liquid bridged the blind die cavity so the bar was not hollow. An alumina tube was subsequently fixed to the seed, extending into the die cavity to vent the hold during seeding. This was later modified to an alumina plug which stayed in the die cavity and extended slightly higher than the die top. This latter modification was preferred since longer vents could be pulled away during growth, occasionally leading to filling in of the cavity.

After introducing the alumina vent, a second problem was found in the reluctance of the liquid completely to wet the top die surface leading to a gap over part of the bar circumference. Slots in the blank part leading edge of the die top (Fig. 2c) successfully overcame this difficulty.

The first hollow bar of over 200 mm length was grown during the fourth growth run. This sample, the longer of the two shown in Fig. 13, exhibited poor surface quality due to a number of pocked areas on the surface. An extreme example of this fibrous-appearing growth is shown in Fig. 14. At high magnification these sections can be seen to be comprised of many fractures perpendicular to the pulling direction. This phenomenon seems to be the result of the liquid freezing on the outer part of the die with concomitant hot tearing. The most effective remedy was found to be increasing the temperature. Radiographs of the hollow and solid bars showed that these airfoil shapes could be produced without microshrinkage. An edge-enhanced radiograph (Ref. 3) of sections of two hollow bars is shown in Fig. 15.

Die life was not a problem during this second phase of the program. In contrast to the flat bar runs which normally exhibited die clogging within 5 to 6 hrs, none of the dies used here failed due to clogging even though the longest runs lasted for close to twelve hr.

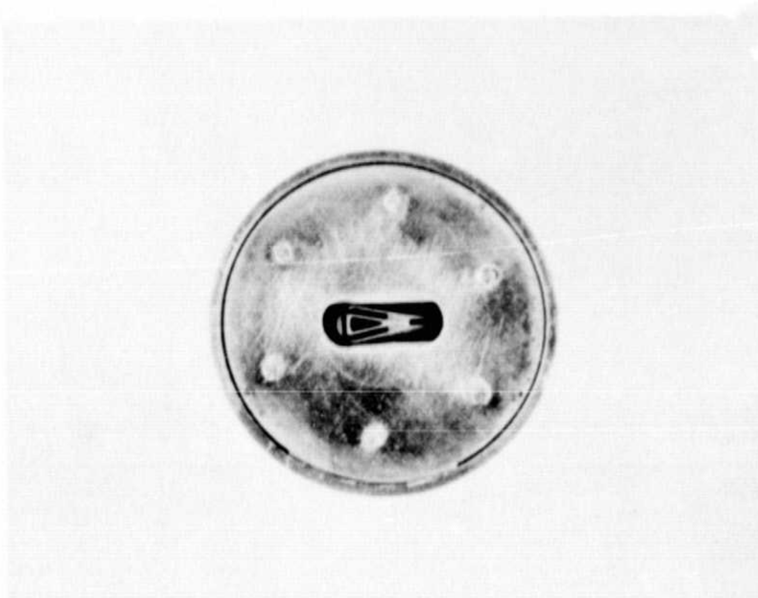


Fig. 12. Photograph of set-up for growth of hollow airfoil shaped bar

Table VII. Summary of Hollow Airfoil Shaped Bar Growth Runs

Run No.	Set-Up†	Growth Speed (cm/hr)	Length (mm)	Width (mm) max	Width (mm) min	Thickness* (mm) max	Thickness* (mm) min	Cavity‡	Other
F1	Q	2.5	220	11.7	10.0	5/1.2	4.2/1	Flooded	-
F2	Q	2.5	85	-	-	-	-	Incomplete	Vent used.
F3	Q	2.5	135	-	-	-	-	Incomplete	Vent used.
F4	R	2.5	235	11.5	10.5	5/1	4.5/1.5	Complete	Plug used.
F5	S	2.5	40/27	-	-	-	-	Incomplete	Plug used.
F6	R	2.5	180	-	-	-	-	Flooded after 47 mm	Plug used.
F7	R	2.5	60	-	-	-	-	Incomplete	Plug used.
F8	R	2.5	88/65	-	-	-	-	Incomplete	Plug used.
F9	R	2.5	210	12	10.5	5.1/1.5	4.1/1.1	Complete	Plug used.
F10	R	2.5	185	11	11	5.3/2	4.5/.8	Flooded after 25 mm	Plug used.

*5/1 indicates sample was 5 mm thick at max thickness, and 1 mm at the trailing edge.

†See Table VIII.

‡"Flooded" indicates no cavity.
"Incomplete" indicates opening in cavity wall.
"Complete" indicates properly formed bar.

Table VIII. Identification of Airfoil Shaped Bar Set Up Types

Type	Dimension "D",* (cm)	No. of Heat Shields	Dimension "A",* (cm)	Die
Q	0.22	2	0.3	Die as in Fig. 2a
R	0.22	2	0.3	Die as in Fig. 2b
S	0.095	2	0.3	Die as in Fig. 2b

*See Fig. 4.

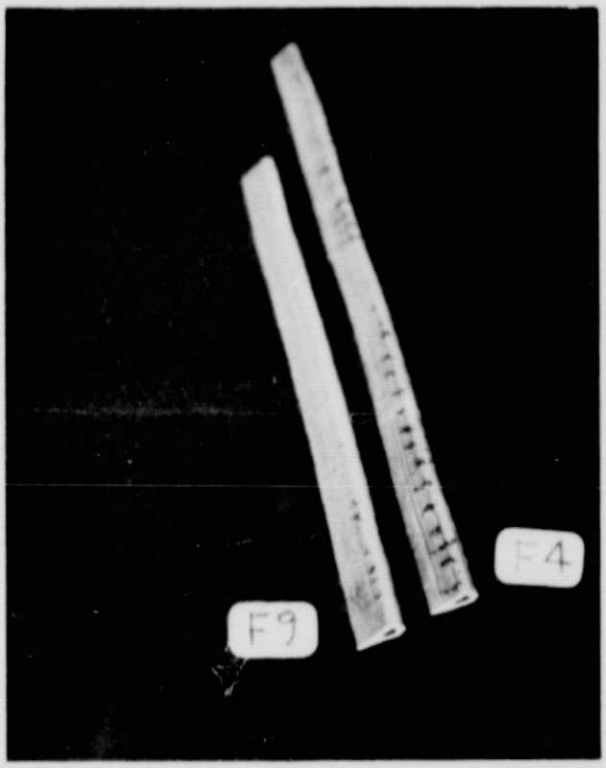
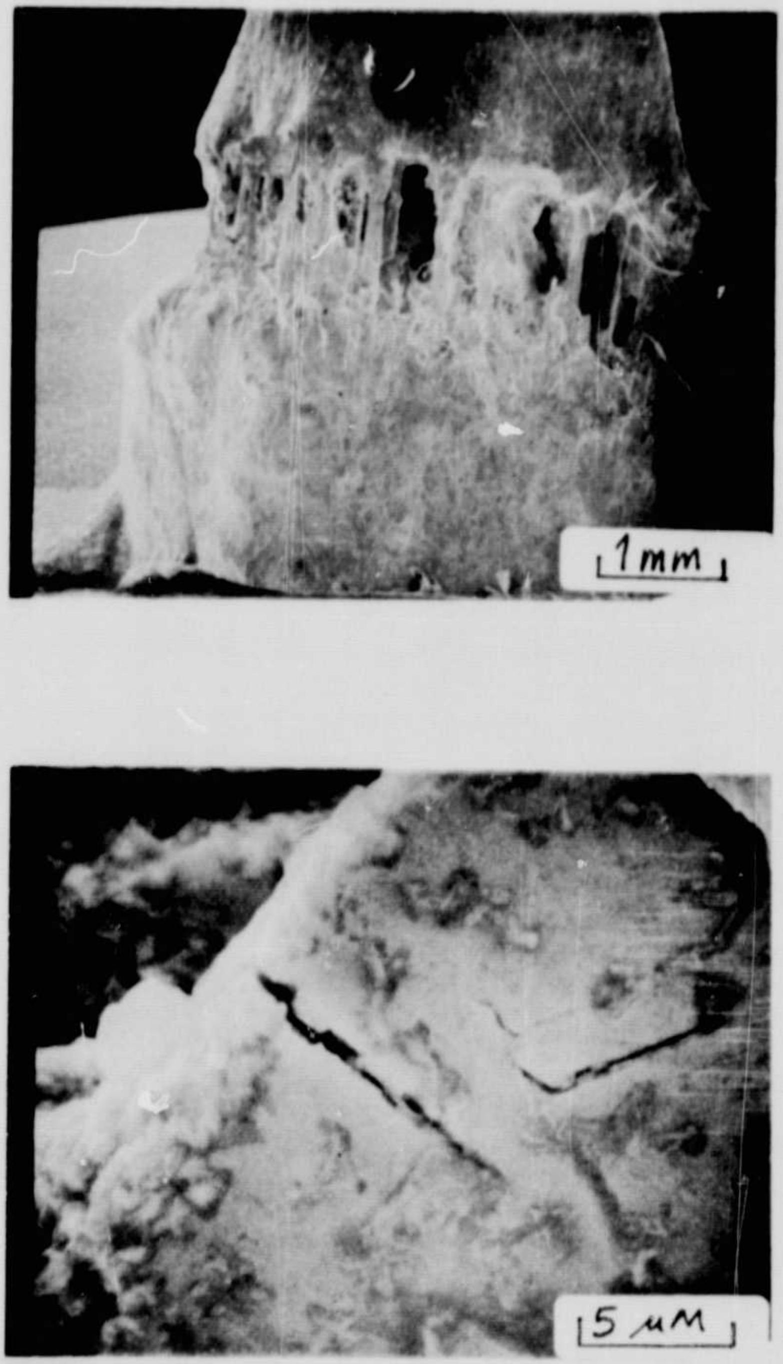


Fig. 13. Two airfoil shaped hollow bars over 200 mm long. Hot tearing in F-4 was all but eliminated in F-9 by increasing the temperature



(a)

(b)

Fig. 14. Fibrous growth morphology resulting from too cold a die top

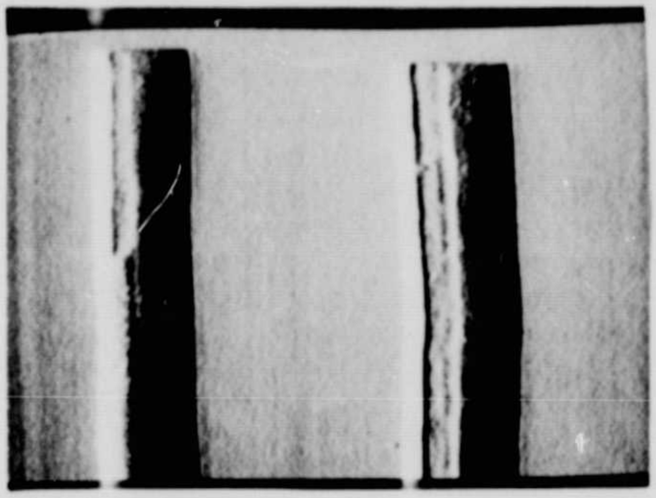


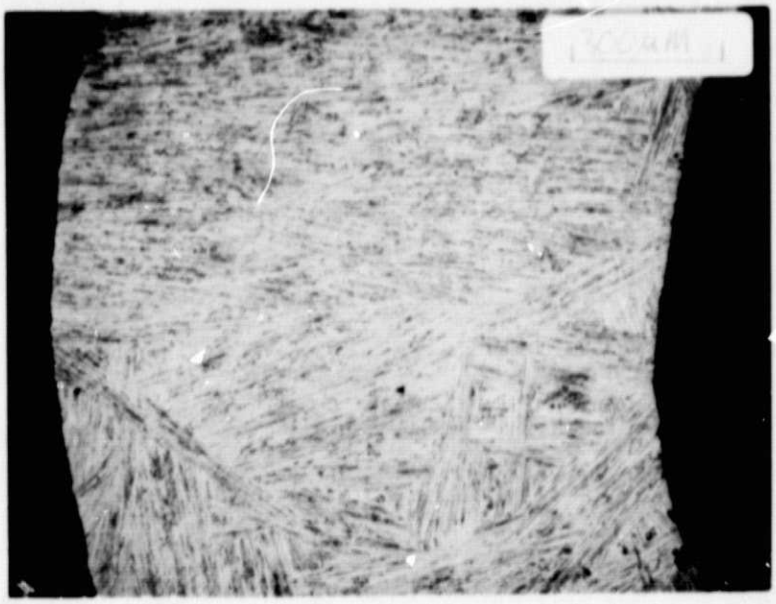
Fig. 15. Radiograph of sections first formed of hollow bars 9 (right) and 10 (left). Note that cavity of bar 10 has filled after 25 mm of growth (Radiography and edge enhancement - Ref. 3 - performed by NASA).

3.6 Microstructure of Hollow Airfoil Shaped Bars

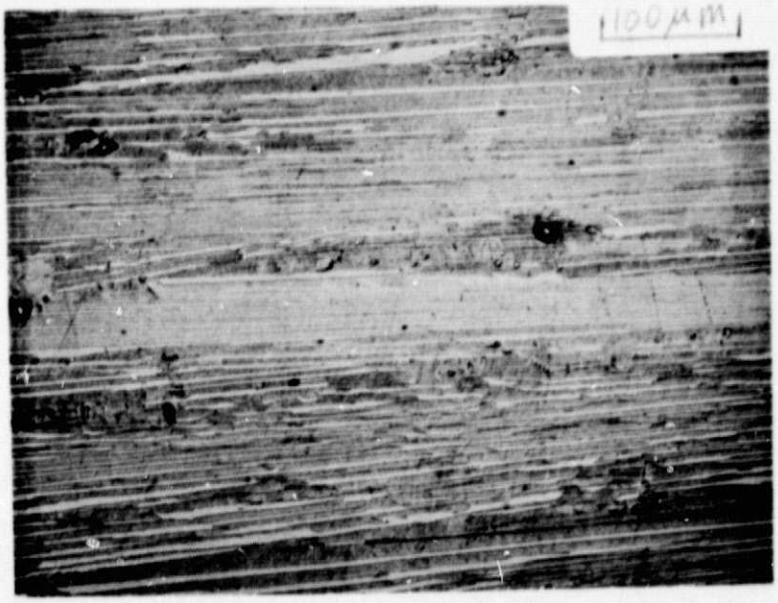
Samples F4 and F9 (full length hollow bars) as well as Fl (solid, full length) F3 and F7 (incompletely spread) were sectioned metallographically to determine their structure. All were grown under essentially identical conditions with respect to die position and shielding, and with the same growth rate. Results of the metallographic inspection are given in Table IX, and were in fact similar both in quality and variability to the flat bars. Representative micrographs of the samples represented by Table IX are shown in Figs. 16 to 18. Photographs of the top and bottom cross-sections of both full lengths hollow bars are shown in Fig. 19. The bottom section of F9 intersected one of the cold regions which had produced a pocked region in the surface.

Table IX. Results of Metallographic Inspection of F-Series Airfoil Shaped Hollow Bars

Sample No.	Location	Results
F1	Middle	Lamellar
F3	Middle	Cellular
F4	Top	Lamellar
F4	Bottom	Partly Cellular
F7	Middle	Cellular
F9	Top	Partly Cellular
F9	Bottom	Partly Cellular

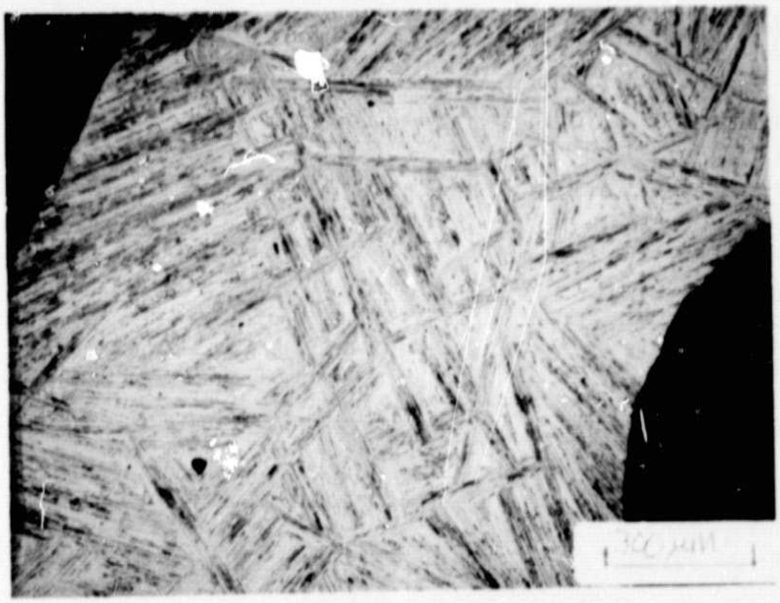


a. Transverse



b. Longitudinal

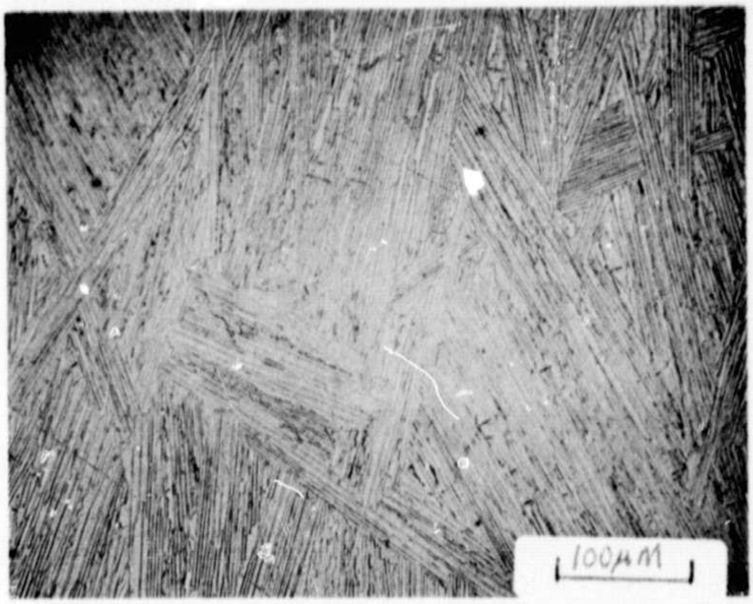
Fig. 16. Microstructure of F4-top (seed end).



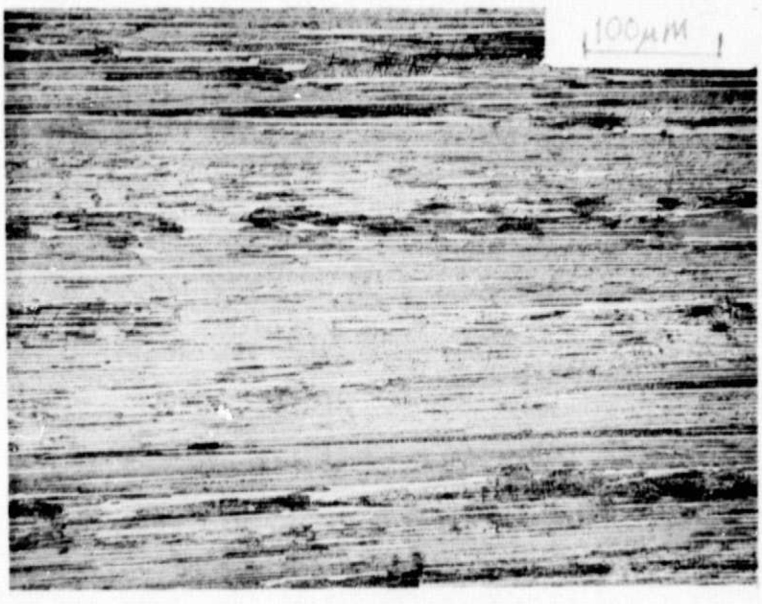
a. Transverse

b. Longitudinal

Fig. 17. Microstructure of F4-bottom (last grown end)

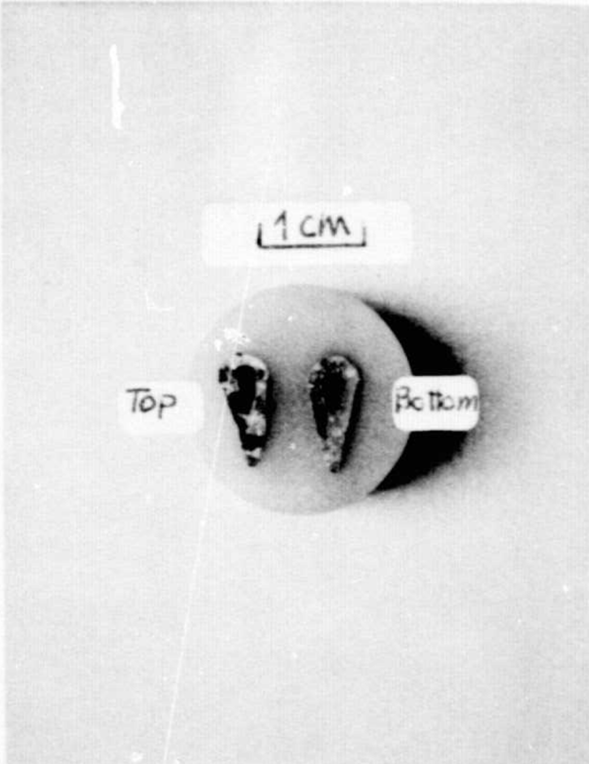


a. Transverse

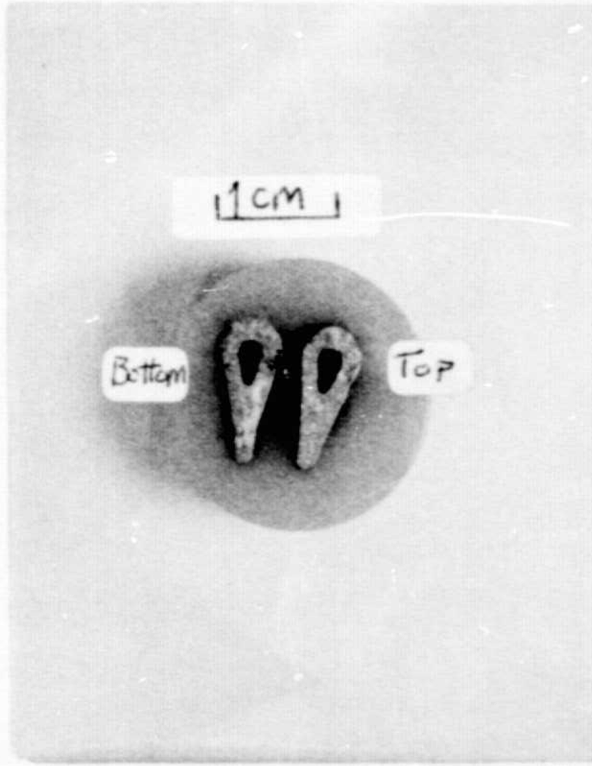


b. Longitudinal

Fig. 18. Microstructure of F1



a. F4



b. F9

Fig. 19. Macrographs of full length hollow airfoil shaped bar cross sections

IV. CONCLUDING REMARKS

The objectives of this program were successfully met in that conditions appropriate for the EFG growth of lamellar $\gamma/\gamma'-\delta$ alloy flat bars and a hollow airfoil shape were established. Thus, 12 × 1.7 mm flat bars were grown as well as hollow airfoil shapes with a 12 mm chord and a 5 mm leading edge diameter. The growth rates which were attained, 2.5 to 3.8 cm/hr do not represent the potential of this processing method.

The experience gained in this program with tantalum carbide dies emphasizes the need for further work to establish an optimum die material. The inspection of failed dies showed two types of interaction which had not been previously recognized. First, the die material itself changed color from a dark to a lighter brassy color. Second, a negative temperature gradient from melt to die top was evidently responsible for the solution growth of large blocky carbides which reduce feeding near the top of the die slot. It is evident from the microstructures of the bars grown that imperfect alignment and precipitated carbides could be causes of the reduced mechanical properties.

Future work should be along one or more of the following approaches:

1. Reduction of superheat in the crucible. The effect of this would be to reduce the amount and rate of dissolution in the melt.

2. Search for better die materials. Previous work (Ref. 1) suggested several feasible alternatives.

3. Use of composite dies. Here one would simply limit the area of active die materials (e.g., TaC) exposed to the liquid.

4. Use of a $\gamma/\gamma'-\delta$ alloy containing both Ta and C.

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4. Use of a γ/γ'-δ alloy containing both Ta and C.

The thermal gradient attained in the present work was not determined. However, in future studies, it is clear that this factor is an important one. Any improvements in the normally slow rates of growth required for multicomponent eutectic alloys would be an important stimulus to their use and high growth rates require steep thermal gradients.

V. REFERENCES

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2. F. D. Lemkey. Eutectic Superalloys Strengthened by δ, Ni_3Cb Lamellae and γ', Ni_3Al Precipitates. NASA CR-2278, Nov. 1973.

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