GRAIN REFINEMENT IN IN-706 DISC FORGINGS USING

STATISTICAL EXPERIMENTAL DESIGN AND ANALYSIS

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Abstract

Room and elevated temperature ductility, strength, fracture toughness and fatigue (low and high cycle) properties are strongly related to the grain size of a fully processed superalloy product. Variations in grain size are normally observed in a heavy section forging weighing one half ton to several tons. These variations in grain size may lead to considerable scatter in the mechanical properties. Very large IN-706 disc forgings weighing up to 30,000 lb are currently fabricated for General Electric Power Systems Division large turbine engines. In order to control the variations in grain size and to create fine grained products (grain size ASTM-5 or finer), thermomechanical processes (TMP) are being optimized using inputs from statistically designed experimental results and analysis.

A large number of variables play a significant role in the development of the final grain size in a forged product. It is extremely difficult to identify the individual effect of these 25 or more processing, as well as material variables. The synergistic and interactive effects of these variables are, however, very important in controlling the final grain size. To quantitatively determine the effects of these variables and their interaction, statistical experimental designs were adopted using a 2^{8-4} fractional factorial design. Higher orders of fractional factorial design are not statistically suitable for a more precise analysis. Several fractional factorial designs were overlapped to cover the effect of all significant processing variables. Results indicate that an ASTM-5 or finer grain size without mixed fine and coarse grains can be developed in large IN-706 disc forgings through TMP optimization; whereas, ASTM-2 or finer grain size is developed through the current conventional processing conditions. Results also indicate that thermal treatments and grain size exhibit significant influence on some of the mechanical properties.

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Introduction

Grain refinement occurs in wrought products as a result of nucleation and growth of new strain free grains (recrystallization) during the thermal exposure within the recrystallization temperature range (1). The recrystallization can be a static process as it occurs during the thermal exposure of a deformed material or it can be a dynamic process as it occurs during a deformation processing (rolling, extrusion, etc.) within the recrystallization temperature range (2). A minimum critical strain and an appropriate range of temperature compensated strain rate, Z (also known as Zener-Hollomon parameter (3)), are required for the onset of dynamic recrystallization. Small initial grain size and lower stacking fault energy in fcc metals and alloys tend to accelerate the kinetics of dynamic recrystallization. Driving force for static as well as dynamic recrystallization is provided by the stored strain-energy within the crystal structure due to hot or cold deformation. The magnitude of this stored strain energy and consequently, the nucleation kinetics, are strongly related to the deformation temperature, strain and strain rate. Besides the strain, strain rate and temperature, several other conditions exhibit significant influence on recrystallization and grain growth. A cause effect tree diagram is shown in Figure 1, which exhibits the effect of various metallurgical and processing factors influencing the recrystallization phenomena.

Although some evidence of dynamic recrystallization was observed, in this study the major objective was to investigate the effect of the deformation processing and thermal treatment variables using a static recrystallization process scheme. One reason for not investigating the effect of particle stimulated nucleation (PSN (4), Figure 1) is the difficulty of controlling this process in a large forging with relatively slow heat-up rate. This type of approach, however, has been successfully applied in IN-718 alloy through precipitation of plate type δ-phase (Ni_3Cb) prior to deformation processing, breaking up the δ-phase to small particles during deformation, followed by annealing to develop fine grained microstructure (5). Creating fine grained microstructure through precipitation of submicron size dispersoid phase may not be feasible within the bounds of the IN-706 alloy composition.

Fabrication processing of the large (62 to 92 in. diameter by 10 to 17 in. thick) disc forging requires a number of processing steps including billetizing, intermediate forming or preforming, finish forging and solution treatment followed by aging. In the billetizing stage, an ingot weighing up to 15 tons is preheated in a furnace within the hot workability range (1800° to 2150°F) followed by upset forging and redrawing. This may involve several reheating and reforging operations. The processing variables are: heat-up rate, stock temperature, hold time at temperature, upset ratio (strain), press head speed (strain rate), reheating temperature, time, redraw scheme (strain path), press speed, die temperature, redraw finish temperature and post processing cooling rate.

The preforming or intermediate processing which may require one or two steps, also includes all the variables listed under billetizing process except that no redraw operations are normally required. The finish forging operation includes preheating to the hot workability temperature range followed by upset forging. The processing variables include: heat-up rate, preheat temperature, hold time at temperature, upset ratio (strain), press head speed (strain rate), die temperature, and post upset cooling rate. Lastly, the solution treatment is conducted by heating the disk forging to a temperature within the solution treatment temperature range (for IN-706, the temperature range is 1725° to 1950°F), holding at temperature for complete solutionizing of the γ', γ'' and η or δ-phases, followed by oil quenching. The desired mechanical properties are achieved by a two step aging process (6).

The solution treatment variables include: heat-up rate, the solution treatment temperature, time at temperature and post solution cooling rate. The aging treatments are done at temperatures (1150°-1350°F) well below the recrystallization temperature range and, therefore, these treatments do not affect the recrystallized grain size.

The initial ingot characteristics such as ingot geometry and weight, the homogenization treatments, the as-cast grain size and inhomogeneities in microstructure and chemical composition may also have significant effect on the intermediate and final microstructure. However, because of the large number of material and processing variables, and their interactive (synergistic or confounding) effects on the final grain size, it would be extremely difficult to identify the individual effects of these variables. To quantitatively determine the effects of these variables and their interaction, statistical experimental designs were adopted using a 2^{8-4} fractional factorial scheme. More highly fractionated factorial designs are not statistically suitable for a precise analysis. A first set of experiments was designed using the variables from the last two process steps, i.e., the finish forging and solution treatment. Experimentally determined important variables from the first set of experiments were combined with preform processing variables (upstream variables) to design a second set of 2^{8-4} fractional factorial experiments. With this overlapping design, the effect of a large number of significant processing variables was investigated.

First Stage Experimental Procedure

The first statistical experimental design with a 2^{8-4} fractional factorial scheme using selected finish forging and solution treatment variables is shown in Figure 2. The capital letter designation of eight variables (also known as indicator variables) is also shown in Figure 2. The finish forging variables are: stock temperature (A), finish forging strain (B), strain rate (C), die temperature (D) and heat-up rate to finish forging temperature (E). The solution treatment variables are: solution treatment temperature (F), time at solution treatment temperature (G) and heat-up rate (H). A low and a high level were selected for each of these variables and are indicated by a minus (–) or a plus (+) sign in the tabular design. Actual process parameters and dimensions are proprietary and therefore, not listed here. The 16 experiments, as shown in Table 2, were conducted using the selected high or low value of all process variables.

Sixteen fully processed pancakes obtained through this scheme were sectioned for macro and microstructural evaluation. The specimen locations are shown in Figure 3. The average grain size at all three locations (A, B and C) in each forging was determined by ASTM linear intercept method. The observed grain size is reported in Table I. Macro and microstructure of a few selected specimens are exhibited in Figure 4(A). The average grain size for 16 pancakes was expressed as two separate parameters: GS1 and GS2. GS1 is the arithmetic average of grain size at all three locations (A, B and C); whereas GS2 is the arithmetic average of grain size at locations B and C only.

Statistical analyses of the experimental results were conducted to determine the relation between the response variables, designated as GS1 and GS2, and the experimental indicator variables A through H. The prediction equations for GS1 and GS2 were found to be as follows:

$$GS1 = 3.8438 - 0.7062X_F + 0.3937X_B - 0.3062X_FX_B - 0.2563X_A + 0.2188X_BX_C \quad (1)$$

and

$$GS2 = 4.0125 - 0.8250X_F + 0.2625X_B \quad (2)$$

where, $X_i = -1$ for the low level of a variable i and
$X_i = +1$ for the high level of a variable i, where the subscript i represents the indicator variables A through H.

In the above predictive equations, a finer grain size is achieved when the value of the response variables GS1 and GS2 increases. The equations predict that a low solution treatment temperature has the predominant effect on achieving finer grain size. A high finish forging strain, a low finish forging temperature and a high strain rate would also have significant effect in terms of grain refinement. It is interesting to observe from Equation (1) that a high finish forging strain along with a low solution treatment temperature would have an interactive beneficial effect in terms of grain refinement. Similarly, a high finish forging strain along with a high strain rate would also have a beneficial effect on grain refinement.

Metallographic observation of grain size (Table I) indicates that ASTM-5 or finer grain size was achieved through several experimental processing conditions, viz., process run numbers 3, 4 and 13. The common features in these processes are: a low solution treatment temperature and a high value of finish forging strain. A low finish forging and a low solution treatment temperature, a high finish forging strain and a high strain rate are very beneficial in grain refinement; however, from industrial fabrication considerations, these may not be readily achievable. It was, therefore, decided to study the effect of other upstream processing conditions such as preforming operations. Experimental design to study the combined effect of preforming, finish forging and solution treatment is outlined in the next section.

Second Stage Experimental Procedure

The second stage statistical experimental design using a 2^{8-4} fractional factorial scheme and variables from preforming, finish forging and solution treatment processes is shown in Figure 5. The variables and their capital letter designation (indicator variables) are also listed in Figure 5. The preforming variables are: hold time at preforming stock temperature (A), first step % reduction (B), reheat time at preforming stock temperature (C) and second step % reduction (D). The preform process stock temperature was selected to provide adequate hot workability for IN-706 alloy. The finish forging variables are: finish forging metal temperature (E), time at temperature prior to finish forging (F) and finish forging % reduction (G). The only solution treatment variable is: solution treatment temperature. Again, a low and a high level were selected for each of these variables and are indicated by a minus (–) or a plus (+) sign in the tabular design (Figure 5). Again, actual processing conditions are considered proprietary and not reported here. Sixteen processing experiments, as shown in Figure 5, were conducted using selected but fixed values of these variables.

A second set of sixteen pancakes fully processed through this scheme were sectioned for macro and microstructural observations using specimens from locations shown in Figure 3. Average grain size at these locations was determined using the procedure described earlier and is reported in Table II.

Second Stage Experimental Results and Discussion

Microstructures of a few selected specimens are shown in Figure 4(B). Some of these microstructures exhibit a duplex type of structure with regions of coarse and fine grains. For these microstructures, it was decided to estimate the average grain size by three separate procedures represented by parameters GS1, GS2 and GS3. GS1 was estimated with an assumption that the average grain size is the average of the size of coarse grains only. GS2 was estimated with an assumption that the average grain size is the average of the size of all grains, and GS3 was estimated with the assumption that the average grain size is the average of the size of fine grains only. The values of these parameters are also shown in Table II.

Statistical analyses of the experimental results were conducted to determine the relation between the response variables designated as GS1, GS2 and GS3 and the experimental indicator variables A through H. The prediction equations were determined to be as follows:

$$GS1 = 3.0187 - 0.2188X_C - 0.5938X_E + 0.3687X_C \cdot X_E - 0.2687X_F + 0.3312X_H \quad (3)$$

$$GS2 = 3.5250 - 0.3375X_E - 0.1625X_F - 0.1500X_E \cdot X_F - 0.6125X_G + 0.3625X_H + 0.1750X_F \cdot X_H \quad (4)$$

$$GS3 = 4.7750 - 0.05X_C - 1.55X_G - 0.375X_C \cdot X_G + 0.0625X_E - 0.3625X_E \cdot X_G \quad (5)$$

where $X_i = -1$ for the low value of a variable i, and
$X_i = +1$ for the high value of a variable i, where the subscript i represents the indicator variables A through H.

As pointed out in the first stage experimental results and discussion section, a finer grain size would be achieved when the value of the response variables GS1, GS2 and GS3 increase. It can be predicted from Equation 3 that a low value of reheat time in preforming and finish forging metal temperature would have a beneficial effect on grain refinement. Equations 3, 4 and 5 exhibit different predictive relations (and they do not point out a common sign (+ or –) of the experimental variables for grain refinement) which can be rationalized in terms of the procedure that is adopted to calculate the response variables GS1, GS2 and GS3.

It is interpreted from these equations that a low preheat or reheat time at any temperature and a low metal temperature in finish forging would have beneficial effect in grain refinement. A small variation in the solution treatment temperature of the order of 50°F may not have a significant effect on the grain size. Contrary to the predictions in Equations 4 and 5, a high finish forging strain (% reduction) should have a beneficial effect on grain refinement as was indicated in the first set of designed experiments. It is possible that a higher order interaction analysis needs to be conducted to unravel the existence of possible confounding effects.

Average grain size of ASTM-5 or finer were achieved through two experimental processing conditions, viz., process run numbers 6 and 10. Based on these experimental results, it is predicted that a combination of low exposure time at temperatures prior to the intermediate and finish forging operations, a higher % reduction in height (strain) during finish forging and a low solution treatment temperature would develop ASTM-5 or finer grain size at an intermediate finish forging temperature within the hot workability range.

Mechanical Property Evaluations

Room temperature tensile and Charpy V-notch tests were conducted on standard specimens from all 32 pancakes from first stage and second stage designed experiments. A few typical test results are shown in Table III. It appears that the tensile yield and ultimate strengths are lower for pancakes solution treated at a higher temperature. However, on a closer examination, it is found that a higher solution treatment temperature also developed a coarser grain size (Table III) in these specimens. It is most likely that the tensile strength properties (TYS and UTS) are closely related to the grain size rather than the solution treatment temperatures and this grain size dependence of tensile yield and ultimate strength can be well rationalized in terms of Hall-Petch relation. Results also indicate that the fine grained materials exhibit a higher ductility in terms of %RA at failure. Two different aging treatments (A vs. B) did not exhibit significant difference in tensile properties. Limited data in Table III also show that the CVN value is not affected by a coarse grain size.

Conclusions

ASTM-5 or finer grain size can be achieved in IN-706 pancake forgings through a combination of lower finish forging temperature, higher strain, higher strain rate and lower solution treatment temperatures. Fine grain size may also be created through the control of upstream processing conditions such as lower hold time at temperatures prior to preform forging, lower solution treatment temperature and possibly higher finish forging strain at a moderately high finish forging temperature.

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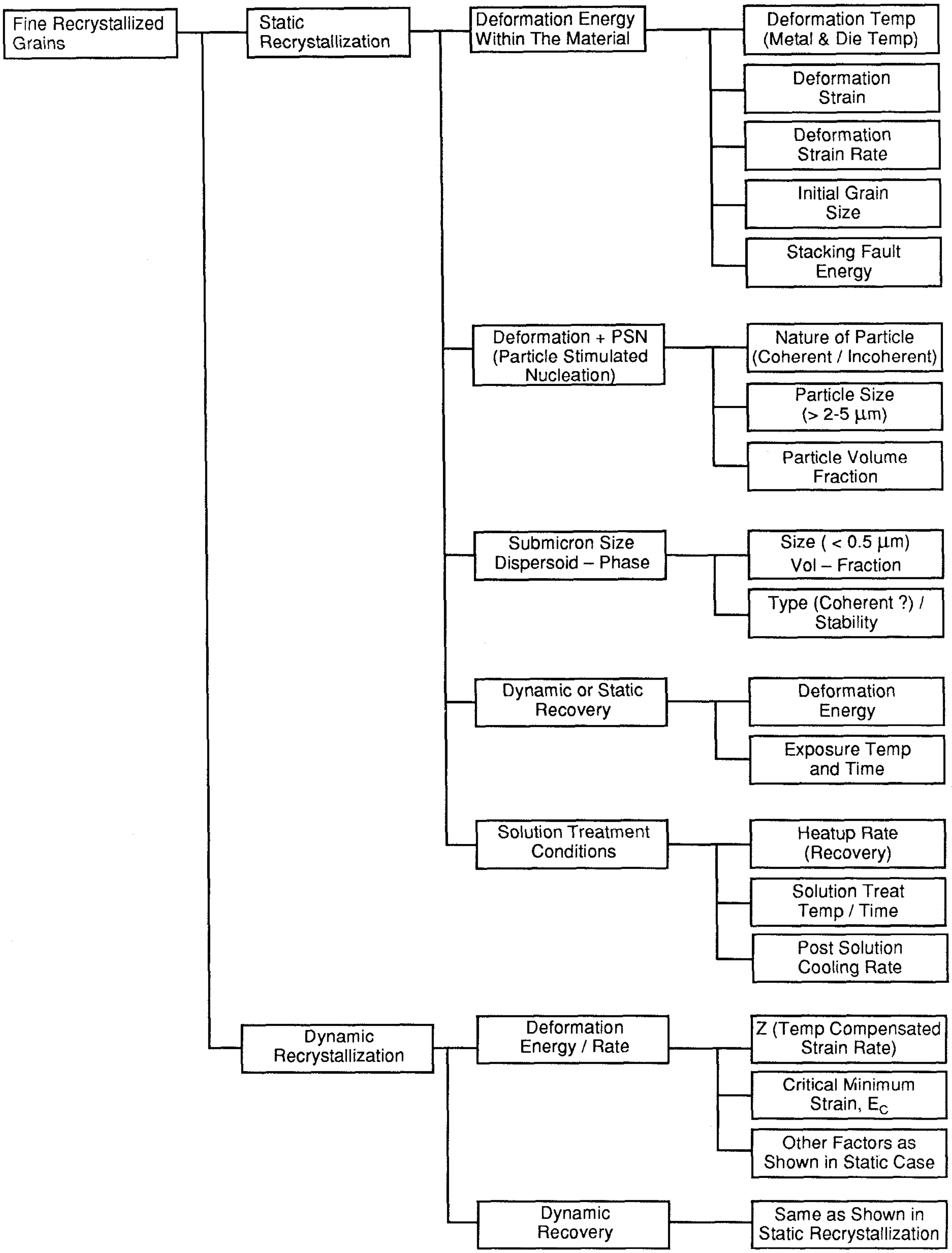


Figure 1. Cause-Effect Tree Diagram for Development of Fine Grained Recrystallized Microstructure

2^{8-4} Fractional Factorial Design:

Run #	A	B	C	D	E	F	G	H
1	+	–	+	+	–	+	–	–
2	+	+	+	–	–	–	+	–
3	–	+	+	+	+	–	–	–
4	–	+	–	–	+	–	+	+
5	–	–	–	+	+	+	–	+
6	+	–	–	–	–	+	+	+
7	–	–	+	+	–	–	+	+
8	–	+	–	+	–	+	+	–
9	+	+	–	–	+	+	–	–
10	–	–	–	–	–	–	–	–
11	–	+	+	–	–	+	–	+
12	–	–	+	–	+	+	+	–
13	+	+	–	+	–	–	–	+
14	+	–	–	+	+	–	+	–
15	+	–	+	–	+	–	–	+
16	+	+	+	+	+	+	+	+

Process Step	Variables	Levels Low	Levels High
Finish Forging	A. Temperature	–	+
	B. Strain (% Upset)	–	+
	C. Strain Rate	–	+
	D. Die Temperature	–	+
	E. Heat-up Rate	–	+
Solution Treatment	F. Temperature	–	+
	G. Time at Temperature	–	+
	H. Heat-up Rate	–	+

Figure 2. 2^{8-4} Fractional Factorial Statistical Experimental Design Including a Few Selected Finish Forging and Solution Treatment Variables.

Table I. ASTM Grain Size at Three Locations Within Pancake Forgings

Process No.	ASTM - Grain Size Locations A	B	C	GS1 A+B+C/3	GS2 B+C/2	Avg. Dia. (μm)
1	2.9	2.9	2.9	2.9	2.9	120
2	4.7	5.0	4.7	4.8	4.8	70
3	5.6	5.6	5.6	5.6	5.6	53
4	5.0	5.3	5.1	5.2	5.2	61
5	3.7	4.0	3.9	3.8	3.9	93
6	2.8	3.0	2.8	2.8	2.9	120
7	4.3	4.5	4.2	4.3	4.4	76
8	2.5	2.8	2.8	2.7	2.8	129
9	2.7	3.0	2.8	2.8	2.9	120
10	3.6	5.1	4.9	4.5	5.0	65
11	4.1	4.0	4.0	4.0	4.0	90
12	2.8	2.5	2.7	2.7	2.7	138
13	5.3	5.6	5.3	5.4	5.5	55
14	2.3	4.5	4.2	3.6	4.3	81
15	1.2	4.8	3.1	3.0	3.9	93
16	3.3	3.4	3.5	3.4	3.4	111

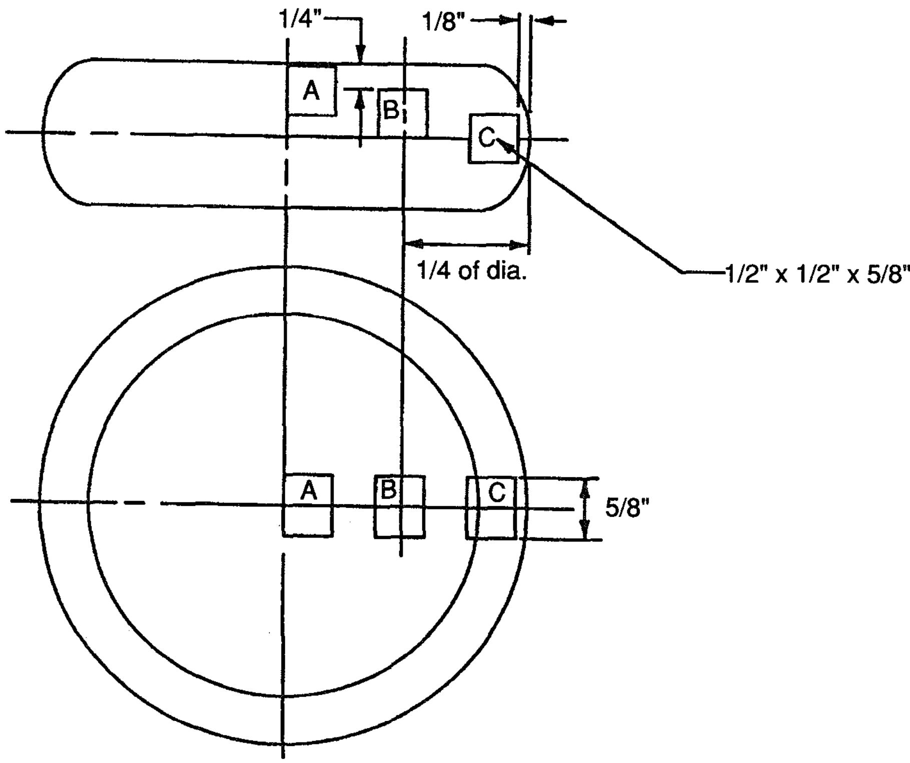
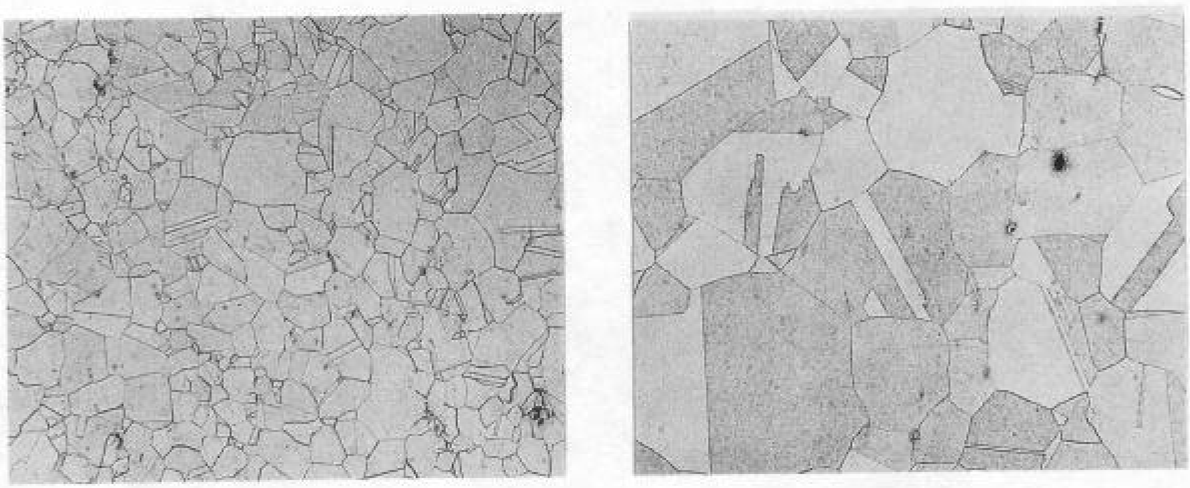


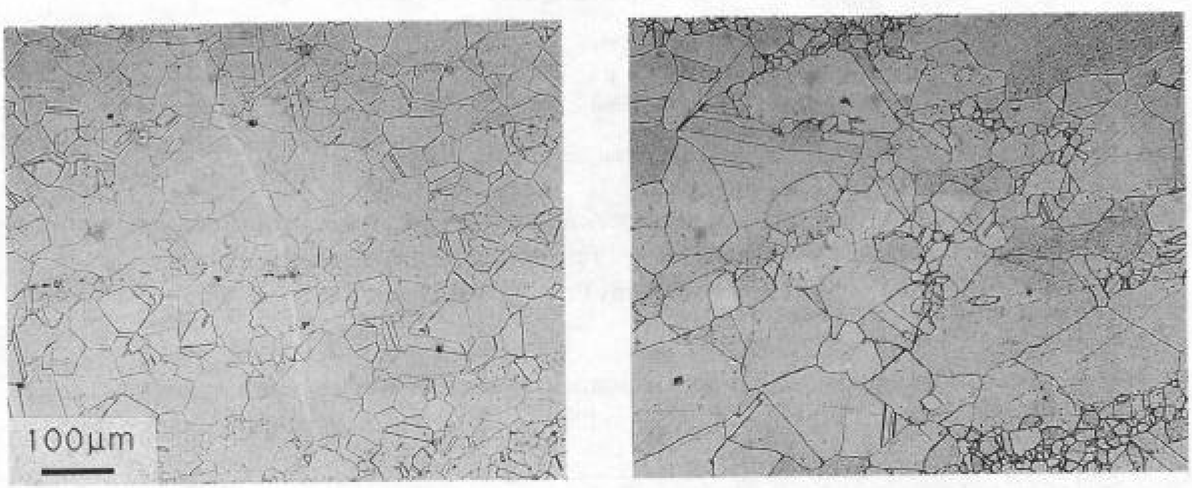
Figure 3. Location of Macro- and Microstructural Specimens Within a Pancake Forging.

Table II. Grain Size Observation of IN-706 Pancake Forgings from Second Stage Statistical Design Including Preforming, Finish Forging and Solution Treatment Processing Variables

Process No.	ASTM Grain Size			Grain Size Measurements for Statistical Analysis		
				GS1 (CG) A+B+C/3	GS2 (Avg) A+B+C/3	GS3 (FG) A+B+C/3
1	1	2.5	2.5	2	2.0	2
2	2/6	3/7	2/7	2.3	3.9	6.7
3	3	3.5	3	3.2	3.0	3.2
4	3	5	4	4.0	3.8	4.0
5	3	2.5	3	2.8	2.7	2.8
6	2/6	5/7	2/7	3.0	5.2	6.7
7	3	3.5	3	3.2	3.2	3.2
8	2	5/9	4.5/7	3.8	4.3	6.0
9	1/2	4/9	3/6	2.7	3.7	5.6
10	5	5.8	5.0	5.3	5.0	5.3
11	3/8	4/9	2.5/7	3.2	4.3	8.0
12	4.5	3.5	3	3.7	2.7	3.7
13	1/8	2/8	1.5	1.5	3.0	6.0
14	2/4	3/4	2/3	2.3	3.0	3.9
15	3	3	3	3.0	3.0	3.0
16	2/5	2/7	3/7	2.3	3.7	6.3



A. Micrographs of specimens from process numbers 4 (left) and 9 (right) at the location B from the first stage of statistical experimental design.



B. Micrographs of specimens from process numbers 10 (left) and 11 (right) at the location B from the second stage of statistical experimental design.

Figure 4. Microstructures of a Few Selected Specimens from the Pancakes Fabricated via First and Second Stage of the Statistical Experimental Design (All at 100X Magnification).

2^{8-4} Fractional Factorial Design:

Run #	A	B	C	D	E	F	G	H
1	–	–	+	–	+	+	–	+
2	–	–	–	+	+	+	+	–
3	+	+	+	–	–	–	–	+
4	+	–	–	–	–	+	+	+
5	+	+	+	+	+	+	+	+
6	–	+	+	–	–	+	+	–
7	–	–	+	+	–	–	+	+
8	–	–	–	–	–	–	–	–
9	+	–	+	+	–	+	–	–
10	+	+	–	+	–	–	+	–
11	+	–	+	–	+	–	+	–
12	–	+	–	+	–	+	–	+
13	+	+	–	–	+	+	–	–
14	+	–	–	+	+	–	–	+
15	–	+	–	–	+	–	+	+
16	–	+	+	+	+	–	–	–

Process Step	Variables	Levels: Low	Levels: High
Preforming (First Upset)	A. Time at Temp	–	+
Preforming (First Upset)	B. First Upset % Reduction	–	+
Preforming (Second Upset)	C. Reheat Time	–	+
Preforming (Second Upset)	D. Second Upset % Reduction	–	+
Finish Forging	E. Metal Temp (°F)	–	+
	F. Time at Temp	–	+
	G. Finish Forging % Reduction	–	+
Solution Treatment	H. Solution Treat Temp (°F)	–	+

Figure 5. Second Stage Statistical Experimental Design for Grain Refinement in IN-706 Disc Forgings Using Preforming, Finish Forging and Solution Treatment Process Variables.

Table III. Room Temperature Mechanical Properties of Pancake Forgings Fabricated via First and Second Stage Designed Experiments

Experimental Stage	Process Run No.	Solution Treat Conditions	Average ASTM Grain Size	Tensile Properties TYS (ksi)	UTS (ksi)	%El	%RA	Charpy V-Notch (ft-lb)
First Stage	3(A)	1750/1 hr	5.8	155	190	21	44	–
	4(A)	1750/5 hr	5.5	157	190	22	44	–
	8(A)	1870/5 hr	2.7	153	183	19	30	–
	8(B)	1870/5 hr	2.7	149	180	19	30	–
	12(A)	1870/5 hr	3.1	152	180	21	33	–
Second Stage	1(A)	1800/4 hr	2.0	156	181	23	41	70
	6(A)	1750/4 hr	5.2	161	190	24	49	56
	8(A)	1750/4 hr	4.3	163	191	21	42	45
	10(A)	1750/4 hr	5.3	161	190	24	50	60
Minimum Goal	–	–	5.0	135	155	12	15	25

NOTE: A - Aging Condition: 1350°F/16 hr, cool to 1150°F @ 100°F/hr + 1150°F/24 hr, air cool.
B - Aging Condition: 1350°F/12 hr, cool to 1150°F @ 100°F/hr + 1150°F/12 hr, air cool.