MINIGRAIN TM PROCESSING OF NICKEL-BASE ALLOYS

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Introduction

Large improvements in fatigue strength resulting from reductions in grain size have been observed in alloy systems which plastically deform by planar slip(1). The grain size dependence of fatigue in such alloy systems is of particular interest to the gas turbine industry where nickel-base alloys are extensively used at elevated temperatures for applications in which fatigue properties are critical to performance. During a study at Pratt & Whitney Aircraft of the mechanical properties of Incoloy 901 as a function of thermo-mechanical processing, it was determined that a refinement in grain size from ASTM 2 to ASTM 5 resulted in a significant improvement in fatigue capability (2). Based on these results an experimental program was initiated to determine whether further reductions in grain size were commercially feasible and to determine if additional benefits in fatigue properties would be realized. This study, conducted on alloys Incoloy 901 and Inconel 718, resulted in the development of a process for attaining grain structures finer than ASTM 10. The process, termed the MINIGRAIN TM Process* involves the establishment of a uniform despersion of spheroidal intermetallic phases through a controlled thermomechanical processing sequence. During subsequent thermal recrystallization, grain growth is minimized by the dispersed phase resulting in an extremely fine grain microstructure. It is the purpose of this paper to describe the processing sequence used to attain the MINIGRAIN structure in Incoloy 901 and Inconel 718 along with the specific effects of grain size on the tensile, fatigue, and creep behavior of the materials.

ESSENTIALS OF MINIGRAIN PROCESSING

The MINIGRAIN process utilizes a uniform dispersion of stable intermetallic precipitates to control the recrystallized grain size of nickel-base alloys such as Incoloy 901 and Inconel 718. The second phase precipitates used for grain size control are hexagonal Ni_3Ti (eta) and orthorhombic Ni_3Cb (delta) in Incoloy 901 and Inconel 718 respectively. An understanding of the precipitation characteristics of Ni_3Ti and Ni_3Cb in these particular alloys as affected by heat treatment and processing is essential to the development of the ASTM 10-13 grain structures in these materials.

Precipitation of Ni_3Ti and Ni_3Cb occurs at the higher aging temperatures as a more stable form of the major strengthening phases Ni_3Ti (metastable FCC gamma prime) and Ni_3Cb (body centered tetragonal gamma double prime) (3). Precipitation of Ni_3Ti in Incoloy 901 occurs at significant rates in the 1500°F - 1750°F temperature range with formation occurring most rapidly between 1600°F-1650°F. Similarly, precipitation of Ni_3Cb in Inconel 718 occurs in the 1500°F-

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1800°F temperature range with formation occurring most rapidly between 1650°F-1700°F. Prior processing history influences both the rate of precipitation and the morphology of the phase. For example, in Inconel 718 approximately 10 hours at 1650°F are required to nucleate Ni_3Cb following a 2100°F(1) solution heat treatment (4) whereas only one hour is required following a 1900°F(1) solution heat treatment (Figure 1). Following a typical forging practice for Inconel 718 which results in an as-forged grain size of ASTM 4-6, a considerable amount of grain boundary Ni_3Cb forms during the 1750°F(1) solution heat treatment.

Morphologically, the precipitation characteristics of Ni_3Ti and Ni_3Cb are similar. In Inconel 718 heat treated for various times at 1650°F following an initial 1900°F(1) solution heat treatment, nucleation of Ni_3Cb occurs at grain boundaries in approximately one hour. With extended time at 1650°F growth of the needle-like phase occurs crystallographically parallel to /111/ planes forming a Widmanstatten structure after approximately 16 hours (Figure 1). Metallographically the precipitation of Ni_3Ti in Incoloy 901 is similar to Inconel 718 with the exception of an apparently lower volume percent of the phase occurring for a given processing-heat treatment history.

The MINIGRAIN process relies on controlled thermomechanical processing to alter the morphology of the Ni_3Cb - Ni_3Ti precipitates from a Widmanstatten needle structure to that of uniformly dispersed spheroidal particles. Initial hot working operations are conducted using conventional processing techniques to obtain a microstructure having a grain size in the ASTM 3-6 range prior to the final forging operation. A "conditioning" heat treatment is then performed in the Ni_3Cb, Ni_3Ti precipitation temperature range to form the Widmanstatten structure shown previously. The final forging operation is then conducted at or below the recrystallization temperature of the material using a minimum reduction of 30% (and preferably 40%) in thickness. The as-forged microstructure consists of a dispersal of the needle-like precipitates into uniformly dispersed spheroidal precipitates having an average size of 1-3 microns with an interparticle spacing of 1-5 microns. The MINIGRAIN structure is then developed using a thermal recrystallization heat treatment below the solvus temperature of the Ni_3Ti-Ni_3Cb phase. A uniform grain size of ASTM 10-13 is obtained approximately equal in grain diameter to the interparticle spacing of the Ni_3Cb-Ni_3Ti precipitates. Conventional aging heat treatments are then employed to provide full hardness response. The MINIGRAIN process is shown schematically in Figure 2.

EXPERIMENTAL FORGING PROCEDURE

Incoloy 901

MINIGRAIN Incoloy 901 pancake forgings having dimensions of 15" diameter by 1.5" thickness were produced by closed die hammer forging. Mults were cut from 8" diameter billet having a grain size of ASTM 2-3. The initial forging operation was conducted from 1800°F reducing the initial 5" height to 3". The as-forged grain size at the intermediate size was ASTM 5-6. Prior to the final forging operation a conditioning heat treatment of 1650°F(8) was employed to precipitate a Widmanstatten Ni_3Ti structure. The final forging operation was conducted from 1750°F using a 50% reduction.

The as-forged microstructure consisted of a uniform dispersion of spheroidal Ni_3Ti precipitates in an unrecrystallized matrix. A recrystallization heat treatment of 1750°F(1) transformed the worked structure into a MINIGRAIN structure having a grain size of ASTM 12. The material was then given an aging cycle of 1325°F (4) + 1200°F(12) to provide full hardness. Two additional pancake forgings, one having a grain size of ASTM 5 and one having a grain size of ASTM 2 were also produced using the same forging conditions followed by solution heat treatment at

1800°F(1) and 2000°F(1) respectively followed by the previous aging heat treatment. These forging were used for mechanical property comparisons with the MINIGRAIN Incoloy 901 forging. Microstructures at several stages of the processing sequence for the Incoloy 901 MINIGRAIN forgings are shown in Figure 3.

Inconel 718

The processing sequence used to produce MINIGRAIN Inconel 718 material was similar to that used for the Incoloy 901 forgings. Twelve inch diameter billet having a grain size of ASTM 4-5 was extruded at a 4:1 reduction to a 6" diameter from 1900°F, providing an as-extruded grain size of ASTM 5-6. A conditioning heat treatment of 1650°F(8) was employed prior to the final extrusion operation to precipitate the Widmanstatten Ni_3Cb structure. The final extrusion operation was conducted from 1800°F using a 4:1 reduction. The as-extruded microstructure consisted of an unrecrystallized matrix containing a uniform dispersion of spheroidal Ni_3Cb precipitates. A recrystallization heat treatment of 1775°F(1) transformed the worked structure to a uniform ASTM 12-13 grain size. An aging heat treatment of 1325°F(8) FC 100°F/Hr to 1150°F + 1150°F(8) AC was employed to provide full hardness. Sections of the as-extruded 3" diameter bar were solution heat treated at 1850°F(1) and 1900°F(1) (resultant grain size - ASTM 5 and ASTM 2 respectively) followed by the previous aging cycle to provide coarse grain material for mechanical property comparisons. Microstructures at several stages of the processing sequence are shown in Figure 4.

Effect of Grain Size On Mechanical Properties

Room temperature and elevated temperature tensile tests were conducted along with high cycle fatigue and low cycle fatigue tests on the Incoloy 901 and Inconel 718 material each processed to three grain sizes. Creep tests were conducted on the Inconel 718 material only. Results of the tensile tests are given in Table 1.

TABLE I

The Effect Of Grain Size On
The Tensile Properties Of
Incoloy 901 and Inconel 718

Material	Test Temperature °F	Grain Size, ASTM	UTS KSI	0.2%Y.S. KSI	Elong. %	R.A. %
Incoloy 901	70	2	177	126	19	21
"	"	5	185	130	20	22
"	"	12	195	139	15	19
"	1000	2	140	111	13	18
"	"	5	148	115	27	36
"	"	12	167	130	19	35
"	1200	2	128	102	20	34
"	"	5	135	107	26	37
"	"	12	151	125	18	37
Inconel 718	70	2	191	162	26	43
"	"	5	201	170	25	41
"	"	12	230	187	17	37
"	1000	2	166	126	20	44
"	"	5	172	145	21	43
"	"	12	192	155	14	36
"	1200	2	150	131	21	39
"	"	5	167	139	19	31
"	"	12	180	151	18	36

Significant increases in both ultimate and yield strengths occurred for both materials as grain size was reduced. The room temperature ultimate and yield strengths for the MINIGRAIN Inconel 718 were 235 KSI and 188 KSI respectively accompanied by excellent ductility. Improvements in tensile properties with decreasing grain size were noted throughout the temperature range investigated.

High cycle fatigue (Kt=1) tests were conducted at 850°F in fully reversed bending for all materials. In addition, notched (Kt=3.5) HCF tests were conducted on Inconel 718 material processed to ASTM 5 and ASTM 12-13 grain sizes. The runout stress was established at 10^7 cycles. The results of the HCF tests are given in Table 2.

TABLE 2

Effect of Grain Size On The High Cycle Fatigue Properties Of Incoloy 901 and Inconel 718 at 850°F

Material	Grain Size	K_t	Runout Stress (10^7 cycles), KSI	Fatigue Ratio (FS/UTS)
Incoloy 901	ASTM 2	1	46	0.32
"	ASTM 5	1	64	0.42
"	ASTM 12	1	91	0.55
Inconel 718	ASTM 2	1	55	0.33
"	ASTM 5	1	80	0.45
"	ASTM 5	3.5	42	-
"	ASTM 12	1	115	0.59
"	ASTM 12	3.5	55	-

A dramatic improvement in HCF properties was observed for both materials as grain size was reduced, with the runout stress being doubled with refinement of the grain size from ASTM 3 to ASTM 12-13. Of greater significance is the effect of grain refinement on the fatigue ratio (fatigue strength/ultimate tensile strength) presented in Table 2 and Figure 5 whereby refinement of the grain size from ASTM 3 to ASTM 12 provided an increase in fatigue ratio of from 0.33 to 0.59. For a given grain size the fatigue ratio appeared to be similar for both materials. (The fatigue ratio was determined on the basis of tensile and fatigue tests conducted at 850°F). Results of the notched HCF tests indicated the same percentage increase in fatigue strength observed in the smooth HCF tests.

Uniaxial push-pull low cycle fatigue tests were conducted at 850°F at several stress levels using a cyclic rate of 1800 cpm. The results of these tests are given in Table 3.

While the results indicate a significant grain size effect on LCF life, the magnitude of the effect is not as pronounced as that observed in the HCF tests.

The results of the creep tests conducted on the Inconel 718 material are given in Table 4.

The date indicates a significant reduction in 1300°F creep capability for the MINIGRAIN material whereas an improvement in the 1100°F creep properties was observed. Interpolation of the data indicates equivalent creep properties for the materials at 1150°F independent of grain size.

TABLE 3

Effect of Grain Size on the Low
Cycle Fatigue Properties of Incoloy 901
and Inconel 718

Material	Grain Size	Stress KSI	Temperature °F	Cycles to Failure (1)
Incoloy 901	ASTM 2	30±65	850	9,000
"	ASTM 5	"	"	26,000
"	ASTM 12	"	"	200,000+
"	ASTM 2	30±77	"	5,000
"	ASTM 5	"	"	16,000
"	ASTM 12	"	"	137,000
Inconel 718	ASTM 2	40±80	"	14,000
"	ASTM 5	"	"	24,000
"	ASTM 12	"	"	53,000

(1) Average of 8 tests

TABLE 4

Effect of Grain Size On The
Creep Properties of Inconel 718

Grain Size	Temperature °F	Stress KSI	Time to, % Creep, Hrs. 0.1	0.5
ASTM 5	1100	120	32	188
"	"	100	62	480
"	1200	100	22	62
"	"	80	305	1000+
"	1300	65	58	310
"	"	40	542	1000+
ASTM 12	1100	120	52	425
"	"	100	348	1000+
"	1200	100	7	53
"	"	80	16	231
"	1300	65	19	51
"	"	40	107	363

Discussion

Nickel-base alloys find considerable application in gas turbine engine components which operate in the intermediate temperature range of 900°F to 1200°F. The limiting design properties of these alloys for intermediate temperature application are generally tensile and fatigue. The creep capability of nickel-base alloys such as Incoloy 901 and Inconel 718 in this temperature range is rarely utilized fully. While the Minigrain process does degrade the creep properties of these alloys at the higher temperatures, the accompanying improvements in tensile and fatigue capability offer a more balanced combination of properties useful in component design.

The most significant property improvement resulting from MINIGRAIN processing is in fatigue particularly at the low end of the stress range. It is not the purpose of this discussion to exhaustively explain the strong grain size effect of fatigue and other strength properties. However, the grain size effect can be rationalized in terms of the slip mode in the plastic deformation of nickel-base alloys. Johnston and Feltner [5] have indicated that grain boundary strengthening is intimately related to strain hardening which is affected by slip mode, i.e., the number of slip systems and the ability to cross slip. In copper where cross slip is easy and wavy slip the consequence, the cyclic flow stress is independent of grain size while the monotonic flow stress shows a moderate grain size dependence. Conversely, copper- 7.5 weight percent aluminum alloys where cross slip is restricted because of a low stacking fault energy resulting in planar slip shows a strong grain size dependence for both monotonic and cyclic tests. Precipitation hardening nickel-base alloys fall into the class of materials characterized by the planar slip mode. Therefore, the MINIGRAIN material fully exploits the grain boundary as a principal barrier to dislocation motion and the operation of dislocation sources in this class of materials.

MINIGRAIN Inconel 718 extrusions up to 5" diameter have been produced successfully to date indicating production feasibility for the process. However, low final forging temperatures (below the Ni_3Cb-Ni_3Ti solvus temperature) are required to attain the desired microstructure, resulting in high forging pressures and increased die wear compared to conventional processing techniques. Alloy chemistry modifications of Incoloy 901 exhibiting increased Ni_3Ti solvus temperature are currently being developed to allow higher processing temperatures. Studies on Incoloy 901 indicate that the Ni_3Ti solvus temperature increases with increasing titanium, chromium and molybdenum content. Experimental alloy compositions having an Ni_3Ti solvus temperature as high as 1925°F have been successfully processed to the MINIGRAIN structure using a final forging temperature of 1900°F. A reduction of pressure was noted for the modified alloy compositions. In addition, improvements in both strength and fatigue properties were obtained on these forgings owing to the increased titanium content. The results of this study will be presented in a future paper.

SUMMARY AND CONCLUSIONS

The MINIGRAIN process was developed as a method of providing improved fatigue resistance in wrought nickel-base alloys for intermediate temperature gas turbine component application. The process, applicable to nickel-base alloys such as Incoloy 901 and Inconel 718, utilizes thermomechanical processing techniques to develop a uniform dispersion of spheroidal Ni_3Ti-Ni_3Cb precipitates throughout the alloy structure. Grain growth during subsequent thermal recrystallization heat treatment is minimized by the dispersed phase, providing a very fine grain size of ASTM 10-13. The tensile, high cycle fatigue, and low cycle fatigue properties of material processed to the MINIGRAIN condition are improved dramatically compared to conventionally processed material. Of greater significance, the fatigue ratio (fatigue strength/ultimate tensile strength) increases from 0.33 to 0.59 with refinement in the grain size from ASTM 12. A degradation in creep properties was observed for MINIGRAIN Inconel 718 in the 1200°F-1300°F temperature range although improvements in the 1100°F creep properties occurred. However, the overall balance in mechanical properties resulting from MINIGRAIN processing offers advantages in the design of many intermediate temperature gas turbine engine components.

Although production capability for the MINIGRAIN process has been established, the low processing temperatures required offer disadvantages from the standpoint of high forging pressures and increased die wear. Alloy modifications aimed at increasing the Ni_3Ti-Ni_3Cb solvus temperature appear to offer increased processing temperatures to alleviate this problem with accompanying improvements in mechanical properties.

References

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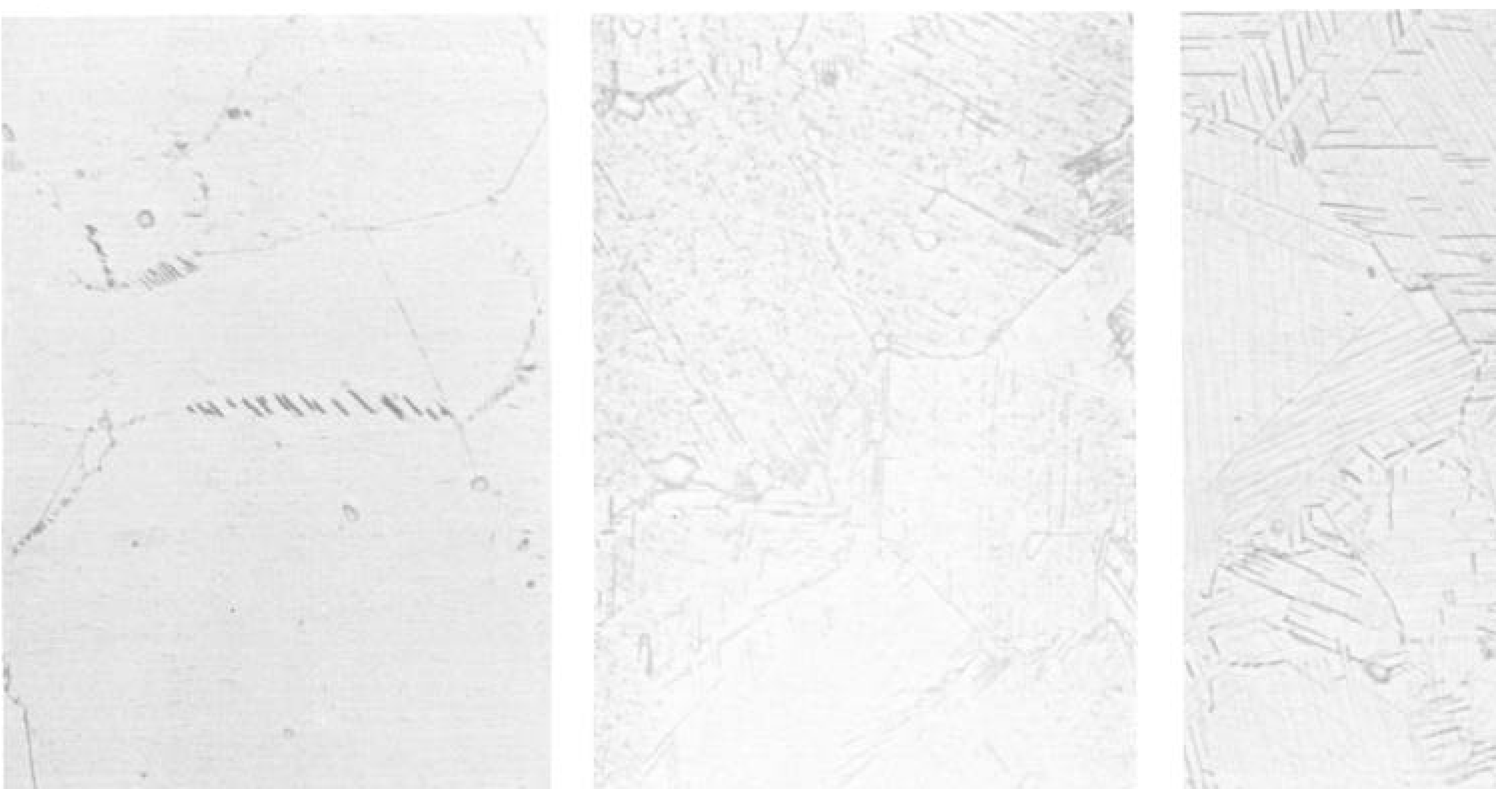


Figure 1

Microstructure of Inconel 718 material solution heat treated at 1900°F (1) followed by Ni_3Cb precipitation heat treatments at 1650°F for one hour (left), 8 hours (center) and 16 hours (right). Magnification - 500x

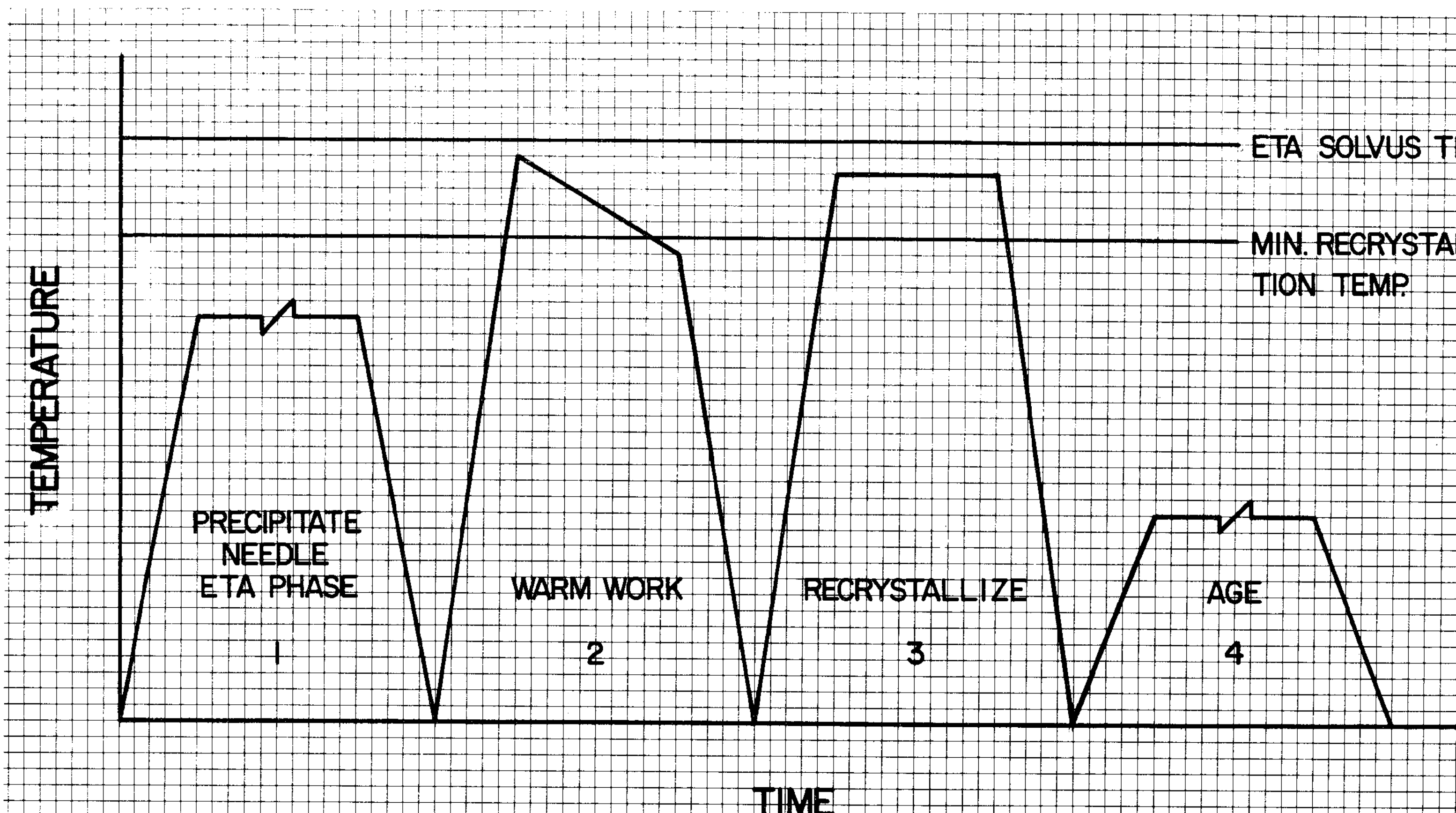
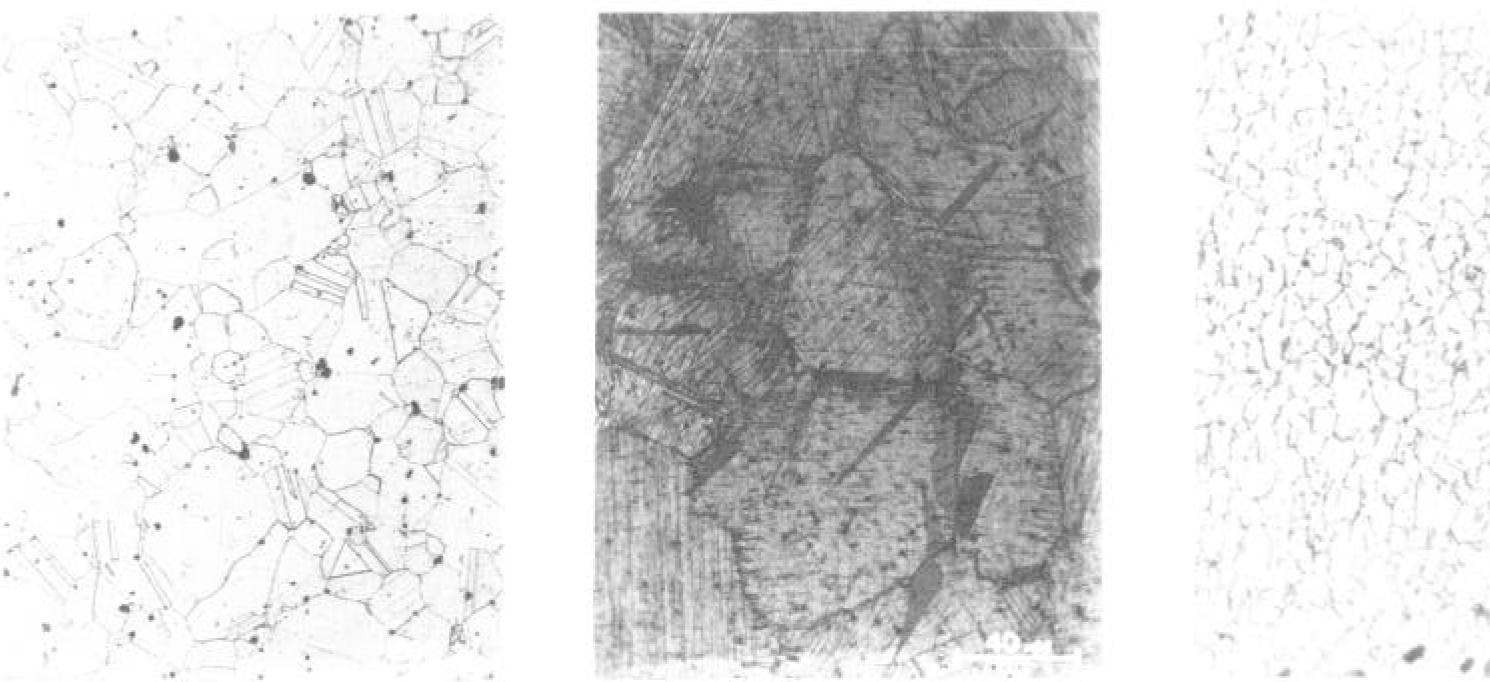


Figure 2

Schematic representation of the MINIGRAIN Process



<u>Figure 3</u>

Microstructures of MINIGRAIN Incoloy 901 pancake forging at three stages of the process. Microstructure prior to conditioning heat treatment (left), microstructure after conditioning heat treatment (center), microstructure after full process (right). Magnifications 100x (left), 200x (center), 1000x (right).

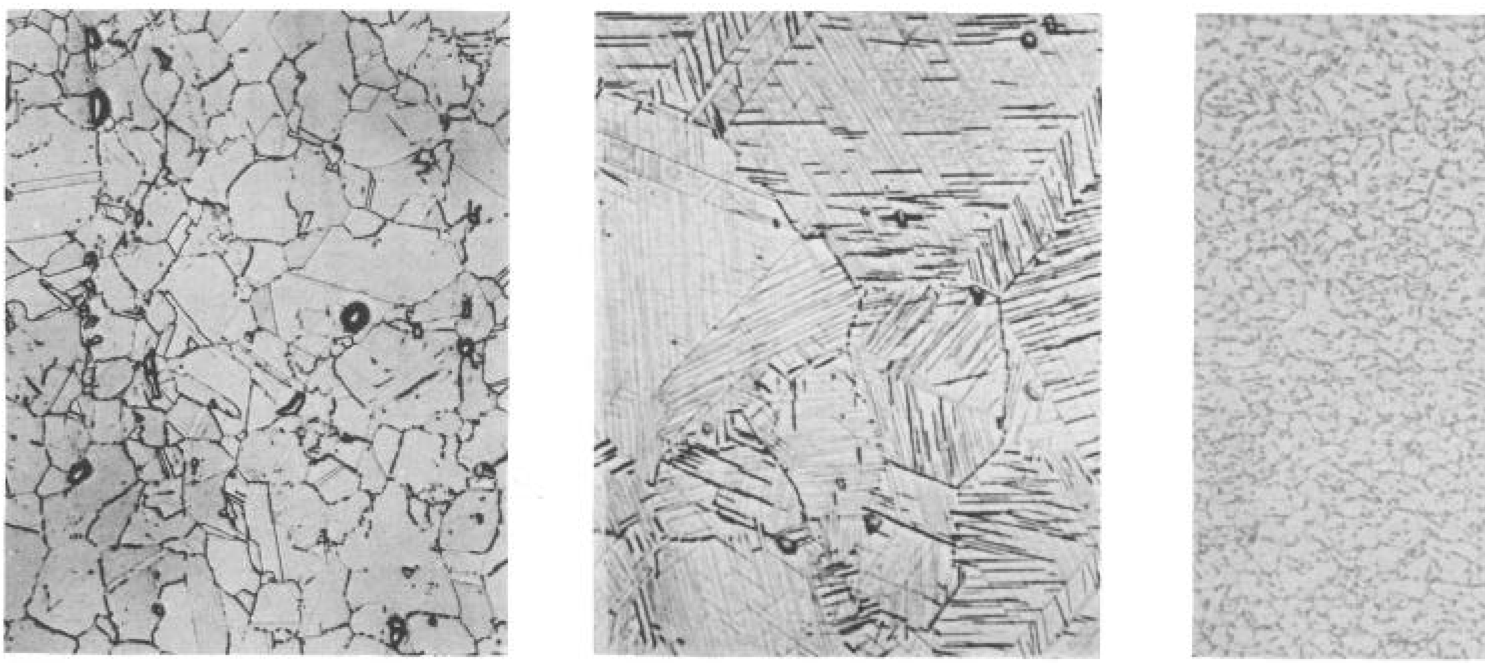


Figure 4

Microstructures of MINIGRAIN Inconel 718 extrusion at three stages of the process. Microstructure prior to conditioning heat treatment (left), microstructure after conditioning heat treatment (center), microstructure after full process (right). Magnifications 100x (left), 500x (center), 1000x (right).

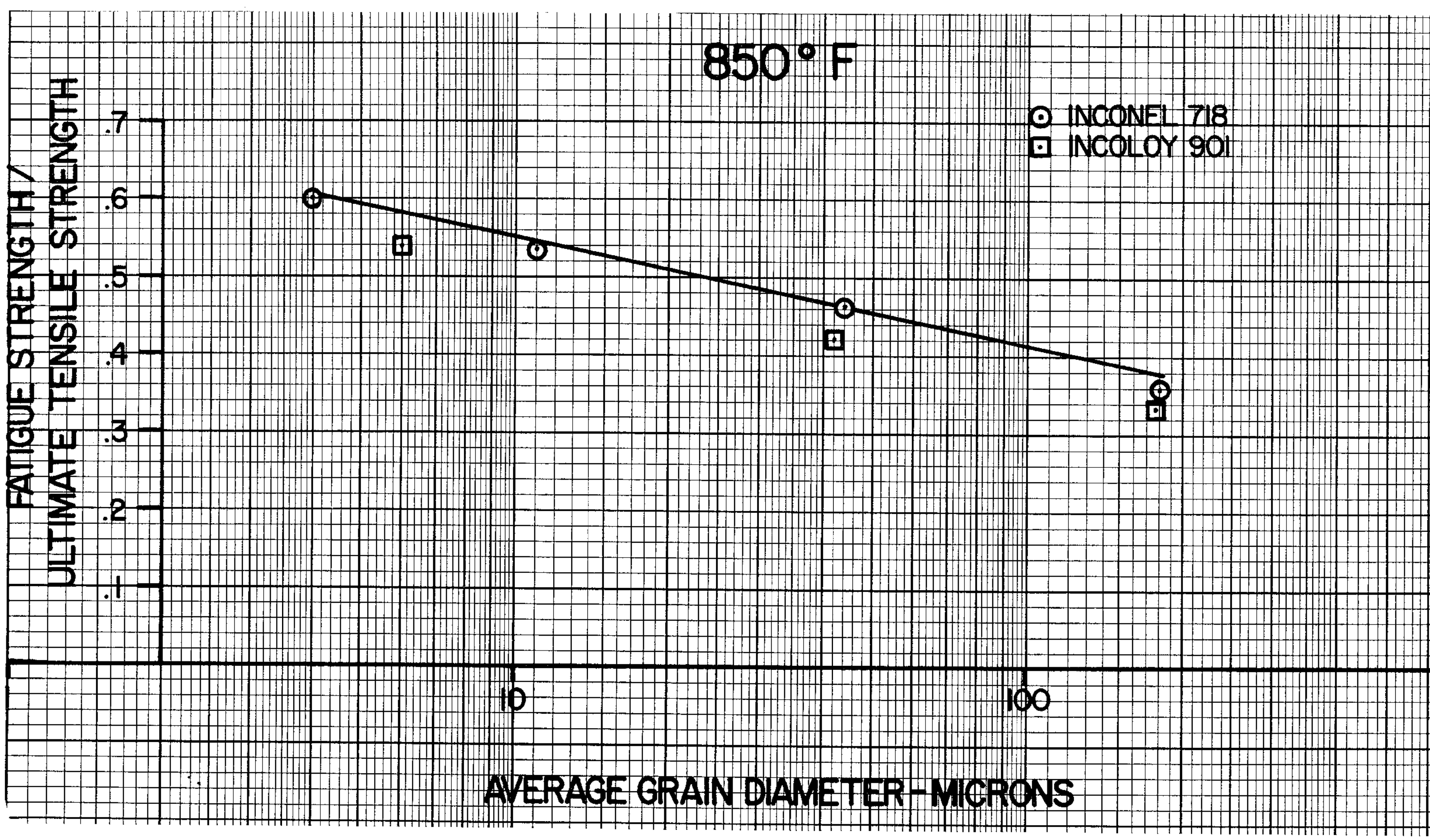


Figure 5

Effect of grain refinement on the fatigue ratio of Incoloy 901 and Inconel 718.