EFFECT OF GRAIN SIZE VARIATIONS ON THE LONG-TIME STABILITY OF ALLOY 718

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Abstract

Uniform, fine grain size is generally preferred in forgings of Alloy 718 for optimum properties. However, due to limitations imposed by forging equipment, forging size and forging configuration, it is often impossible to produce this desired grain size; instead, some grain duplexing or grain coarsening occurs in certain areas throughout the forging. Stress-rupture tests were conducted before exposure, and after exposing specimens at 1250°F, 50 ksi or 1300°F, 50 ksi for 500 hours on material having (1) uniform fine grain size (ASTM 7 or finer), (2) duplex grain size and (3) uniform coarse grain size (ASTM 5 or coarser). These grain sizes were obtained in forgings with a simple pancake configuration using varying forging techniques. The results of the rupture tests conducted before exposure showed an excellent correlation with grain size and Ni_3Cb plate size. After exposure, rupture ductility increased and strength decreased due to overaging. In duplexed samples the controlling factor appears to be the amount and size of the coarse grain. The optimum grain size for the best balance of rupture properties appears to be ASTM 6 to 7.

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INTRODUCTION

Alloy 718 is a wrought nickel-base alloy for moderately high temperature applications developed by H. L. Eiselstein of International Nickel Company.(1) In addition to possessing high strength and good ductility, it is readily weldable. Currently it is being utilized as a sheet alloy and as forgings for compressor and turbine parts and in cryogenic applications.

In recent years there has been an increased emphasis on the production of larger forgings from superalloys. A uniform, fine grain size is generally preferred in these forgings for optimum properties. However, due to limitations imposed by equipment, forging size and forging configuration, it is often impossible to produce this desired grain size; instead, some grain duplexing or coarsening occurs in certain areas throughout the forging.

Recently Barker, Ross and Radavich evaluated the long-time stability of Alloy 718.(2) The results of their evaluation showed that 718 has excellent stability at 1200°F through 10,000 hours. Although phase changes occur rapidly at 1300°F, these changes did not embrittle the alloy but did cause considerable loss in strength between 200 and 800 hours exposure.

The present investigation was undertaken to study the effect of the grain size of Alloy 718 on its stress-rupture properties both before and after stressed exposure at 1250° and 1300°F. These exposure conditions were selected to simulate accidental over-temperature operation of a gas turbine engine. The grain size was varied through the use of different controlled forging techniques.

The study was divided into two phases. Phase I was concerned with the evaluation of the influence of 13 different combinations of forging temperature and reduction on stress-rupture behavior. In Phase II, the stability of six of the original forgings was evaluated by means of stress-rupture testing and extensive metallographic studies.

EXPERIMENTAL PROCEDURE

Phase I

In Phase I of this study, thirteen 3-inch thick slugs were obtained from a 4-1/2-inch RCS billet of Lescalloy 718 TIVA, Heat C50615-1. This heat was vacuum-induction melted and subsequently vacuum-consumable-electrode remelted into a 20-inch ingot. The composition of the billet selected for study is listed in Table I. This billet met all the requirements of the leading current aerospace specifications. Mechanical properties of this billet and of forgedowns made from billet slugs are presented in Table II.

The 3-inch lengths were upset forged on a 3-ton steam hammer by the procedures shown in Table III to 3/4-inch thick pancakes. These upsetting procedures were selected because they simulate the ranges of temperatures and reductions used in the forging of Alloy 718 components. Pancakes A and B were made similarly except that in forging Pancake A a pause of about two seconds was used between consecutive hammer blows. The procedure minimized any tendency toward temperature build-up in the forging and, therefore, gave lower finishing temperatures. The same holds true for Pancakes F and G and Pancakes H and I. Finishing temperatures, more accurately off-die temperatures, were measured with an optical pyrometer.

A 1/2-inch slice was cut diametrically from each pancake. This slice was used to determine the grain size of the pancake at 1/2-inch intervals across its entire width.

Stress-rupture specimen blanks were removed from radial locations of each pancake such that the center of each specimen was the same distance from the center of the forgedown. These blanks were heat treated as follows: 1750°F/1 hr./AC + 1325°F/8 hrs./FC 100°F/hr. to 1150°F/8 hrs./AC and then machined into .252-inch gauge diameter combination stress-rupture specimens (K_t=3.7). Testing was done at 1200°F and 110,000 psi.

Phase II

To evaluate the effect of grain size on the stability of this material, six of the upset pancakes were selected. The basis for the selection of these pancakes was grain size and rupture ductility. Two pancakes each were selected to represent:

a) fine grain size - high rupture ductility (G and L)

b) duplex grain size - intermediate rupture ductility (D and H)

c) coarse grain size - low rupture ductility (B and J)

Four stress-rupture blanks were cut radially from each pancake and heat treated as before. The blanks were machined into .375-inch smooth bars and then two blanks were exposed at 1250° and two at 1300°F, 50,000 psi for 500 hours. The specimens were then finish machined into .252-inch combination bars (K_t=3.7) and tested at 1200°F and 110,000 psi.

Samples for microstructural examination were obtained from (1) the as-forged pancakes, (2) the solution-treated blanks, (3) the heat-treated blanks, and (4) the stress-rupture specimens at two locations, adjacent to the fracture and in the threaded end, both before and after exposure. Optical metallography was conducted by conventional techniques. Negative and extraction replicas were examined by electron microscopy, both in our laboratory and by John Radavich at Micromet Laboratories, Incorporated. X-ray diffraction of extracted residues was also conducted both in our laboratory and by Dr. Radavich. The procedures used for electron microscopy and X-ray diffraction are outlined in the Appendix.

RESULTS AND DISCUSSION

Phase I

This portion of the study deals with an evaluation of the influence of forging variables and grain size on the stress-rupture properies of Alloy 718.

Grain Size of the As-Forged Pancakes

The grain sizes of the as-forged pancakes are listed in Table III. As mentioned previously, grain size measurements were made at 1/2-inch intervals across the width of the pancake. The results of these measurements showed that for a given pancake the grain size was the same throughout the pancake except at the extreme edges. From the grain sizes given in Table III one will note that reheating results in a duplexed grain structure with two exceptions, J and K. These latter samples were essentially a uniform cold worked structure indicating the final reduction and finishing temperature had been low enough to prevent recrystallization during the forging cycles. Direct reduction from 3-inch to 3/4-inch in all cases produced a uniform grain structure. For pancakes made without reheating, the grain size is essentially determined by the finishing temperature of the forging as shown by the data plotted in Fig. 1.

Microstructures typical of the as-forged samples are shown in Fig. 2. In general, Pancakes C, E, H, I, and M, all of which had duplexed grain structures, exhibited microstructures similar to that of Pancake D, except for varying amounts of fine and coarse grains. Pancakes A, F and G had fully recrystallized (equiaxed) structures similar to that of B, except that the actual grain size varied, whereas Pancakes J, K and L had grain structures that exhibited little or no recrystallization.

Additional metallographic work was conducted on the as-forged samples that were selected for the stability study. The results of this work and of phase identification studies are presented in Phase II of this investigation.

Stress-Rupture Properties and Grain Size Before Exposure

The results of duplicate stress-rupture testing of combination bars from each of the 13 pancakes after heat treatment are presented in Table IV. Also included in this table are the ASTM grain sizes of the heat treated specimens reported both conventionally and as a "weighted" ASTM grain size. The "weighted" grain sizes were calculated by multiplying the grain diameters by the percentage of that grain size present and then converting this calculated grain diameter to an ASTM grain size. There was no significant change in the grain size during heat treatment, except that L recrystallized during solutioning at 1750°F.

Graphical presentations of the influence of forging procedure on the grain size and notch rupture sensitivity of the specimens are shown in Fig. 3 and 4, respectively. In Fig. 3 a line has been drawn to separate specimens having grain sizes of ASTM 6 and finer from those having grain sizes coarser than ASTM 6. The forging parameters represented by the upper left-hand corner of this graph are those that tend to produce a fine grain size. When one draws a line to separate specimens that were notch ductile from those that exhibit a tendency toward notch brittleness, Fig. 4, the line is in a position identical to that separating grain size, Fig. 3., indicating a strong relationship between grain size and notch rupture ductility. Plotting the average rupture ductility of the duplicate specimens against the actual or, in the case of duplexed specimens, the weighted ASTM grain size does indeed show excellent correlation, Fig. 5. Specimens from B and J are not included on this plot because they exhibited notch failures and an accurate rupture-elongation value could not be assigned to these specimens. A regression analysis of these data is being conducted to relate rupture properties to both the forging variables and the grain size.*

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*These results will be presented at the symposium in September.

Interrupted-low-cycle-fatigue tests were also conducted on specimens from all 13 pancakes. The preliminary results of this work indicate that interrupted-low-cycle-fatigue life does not have a simple relationship to stress-rupture properties. This is being further investigated.

Microstructures typical of the heat-treated specimens are shown in Fig. 6. Additional metallography work was conducted on heat-treated specimens prior to exposure as a portion of Phase II of this study.

Phase II

Based on the grain size and before-exposure rupture data previously presented, Pancakes B, D, G, H, J and L were selected for stability and extensive microstructural studies. The grain sizes of these forgings are shown in Table V. Pancakes B and J exhibit a coarse grain size with one important difference; B is fully recrystallized, while J has less than 5% recrystallized grains. This was expected from the parameters used to produce these forgings. Pancakes G and L exhibit recrystallized, uniform, fine-grained structures that were produced by combinations of low furnace temperatures, low finishing temperatures and large reductions. Forgings D and H have duplex grain sizes due primarily to the amount of final reduction given to them.

Rupture Properties After Exposure

The results of 1200°F - 110,000 psi stress-rupture testing before and after 500-hour 50-ksi exposure at 1250° and 1300°F also are listed in Table V and presented graphically in Fig. 7 and 8. In almost all cases the exposure conditions increased rupture ductility and decreased rupture life with the samples exposed at 1300°F having the higher ductilities and the shorter lives. It is interesting to note that the material which was most notch brittle before exposure (B) exhibited excellent properties after exposure.

There were several exceptions to the above general observations. Both of the 1250°F and one of the 1300°F specimens from D broke in the threads during exposure suggesting extreme notch brittleness problems. One of the 1300°F specimens from L failed in the smooth portion of the combination bar during exposure while the second 1300°F test specimen failed in less than 2 hours of testing at 1200°F - 110,000 psi after exposure.

Grain Size Effect

Examining the results presented in Table V with reference to grain size, the familiar effect of an increase in rupture ductility and a decrease in rupture strength with increasing fineness of grain size is seen in the uniform, fully recrystallized grain specimens - B, G and L. The most desirable uniform grain size for optimum properties before and after exposure appears to be approximately ASTM 6-7. Of course the method used to produce the grain sizes in B, G, and L also produced other differences in the microstructure which contribute to the differences in properties; these will be discussed later.

Looking at the duplexed structures it becomes more difficult to correlate grain size and properties. As previously shown in Fig. 5, plotting the weighted grain size versus rupture ductility before exposure for all the forgings examined in Phase I shows excellent correlation. Looking further at the properties before exposure on Pancakes D, H and J, it would appear that a certain amount of duplexing can be tolerated as in the case of H. However, when the large grain size in the duplexed structure becomes coarser i.e., ASTM 3 in D as compared to ASTM 4 in H, or when a large percentage of coarse grain is present, i.e., 90% ASTM 3 in D and virtually 100% 3.5 in J, notch brittleness occurs. The weighted grain size values also indicate that D and J are coarser. Values obtained for the duplexed samples were ASTM 5.7 for H, 3.2 for D and 3.5 for J.

Looking at the behavior of D, H, and J during and after exposure, it would seem that again both the coarsest grain size and the percentage of coarse grain have an influence. D exhibited the coarsest grain size (ASTM 3.2) and showed the most brittle behavior during exposure. Pancake J with the next coarsest grain size (ASTM 3.5) was better than D but was less notch ductile than the remainder of the forgings. Pancake H, with 40% of the grains being ASTM 4, was more notch ductile than B which was a uniform ASTM 5; however, H also contained 60% fine grain to give a weighted grain size of ASTM 5.7 which, using grain size as a criteria, indicates that H should be more notch ductile than B.

Using the weighted grain sizes, one will note from Table V that the rupture ductility after stressed exposure increases linearly as the grain size becomes finer. However, the correlation is not as good as that obtained before exposure (Fig. 5).

Again, as mentioned previously for the uniform grain forgings, other microstructural changes also contribute to the differences observed. This is discussed in the next section.

Microstructural and X-Ray Diffraction Analysis

As-Forged. Microstructural and X-ray diffraction analysis of the as-forged pancakes disclosed the presence of MC (probably CbC), TiN and possibly Ti_2CS in all six forgings in the unheat-treated condition. In addition, considerable Ni_3Cb was found in Pancake L. This was attributed to the working temperatures used for this forging. The X-ray diffraction data are presented in Table VI.

Fig. 9 is an electron micrograph showing the clean grain-boundaries of Pancake B in the as-forged condition. This was also typical of Forgings D, G, H and J. Fig. 10 illustrates the structure of L in the as-forged condition. Based on the X-ray data, many of the visible particles in the micrograph of L are identified as Ni_3Cb.

Heat-Treated Before Exposure. During solution treating at 1750°F, Ni_3Cb precipitated at the grain-boundaries in all the samples. This was readily apparent both microstructurally and from X-ray diffraction analysis. The X-ray diffraction data (Table VI) further indicated that the samples having the finer grain sizes (G and L) contained the most Ni_3Cb. This analysis also suggested a further increase in the amount of Ni_3Cb upon subsequent aging in all samples. No Laves phase was found in any of the specimens.

In addition, micrographs (Fig. 11) of the extracted residues used in the X-ray diffraction analysis revealed two significant facts:

1) The Ni_3Cb plate size becomes larger as grain size increases.

2) In the duplex grain size specimens, the Ni_3Cb was found as a mixture of large and small plates.

Microstructural observations based on the examination of microstructures and numerous negative replicas from each sample are presented in Table VII. The following observations were made regarding the interaction of grain size, microstructure, rupture properties, and forging parameters:

1) The size of Ni_3Cb plates is directly proportional to grain size; larger plate sizes are associated with larger grain sizes regardless of whether they are recrystallized or non-recrystallized grain.

2) Increasing Ni_3Cb plate size associated with increasing grain size decreases rupture ductility and promotes an increasing susceptibility to notch brittleness.

3) Decreasing the volume of <u>large</u> plates further increases the tendency toward notch brittleness.

4) Higher forging temperatures promote a larger grain size (increased Ni_3Cb plate size) and a decreased Ni_3Cb plate volume. Thus higher forging temperatures promote lower rupture ductility and a greater tendency toward notch brittleness.

5) Grain boundary films were most noticeable in those specimens forged from the higher temperatures, Pancakes B and D. This appears to confirm Eiselstein's findings that film formation is favored by high annealing temperatures.(3) This film was identified as CbC by Eiselstein.(3)

6) None of the forgings showed depleted zones adjacent to grain-boundaries.

7) In duplex-grained samples, the largest percentage of microcracking was observed at the boundaries of the larger grains suggesting fracture could initiate at these boundaries at much lower stresses than were necessary at the boundaries of the fine grains. These microcracks also were two to four times longer than those formed at fine grain-boundaries, thereby being much closer to the critical crack length necessary for complete fracture.

Light micrographs at 100X and electron micrographs of negative replicas at 7500X of the heat-treated structures illustrating the above observations are shown in Fig. 12 through Fig. 17.

Fig. 12 from Forging B illustrates a uniform, somewhat coarse grain size and a large Ni_3Cb plate size.

The micrographs for D (Fig. 13) show the very severe duplexing in this sample. A mixture of large and small Ni_3Cb plates is apparent in the electron micrograph.

Fig. 14 shows the uniform fine grain size of Forging G along with medium size Ni_3Cb plates.

In the micrographs for H (Fig. 15) note a structure similar to D except that it contains more fine grain. A mixed Ni_3Cb plate size is evident.

In J, Fig. 16, the light micrograph shows that this sample consists of essentially 100% worked grain, i.e., almost no recrystallization has occurred. The electron micrograph shows primarily large plates.

Fig. 17 shows the very fine uniform grain size in L. A large volume of small Ni_3Cb plates is evident.

In Phase I of this study it was found that stress-rupture ductility increases as grain size becomes finer. Extensive metallographic studies carried out on the before-exposure specimens in Phase II of the investigation indicate that Ni_3Cb plate size decreases as grain size becomes finer.

The question now arises as to whether Ni_3Cb plate size or plate volume is more influential on rupture ductility. Relating all the microstructural evidence, it appears that the plate size may be more important than plate volume. All the forgings having large Ni_3Cb plates were notch brittle regardless of volume except H; however, as discussed earlier, H had only 40% coarse grain as compared to 90% coarse grain in D and 100% in B and J. Furthermore H, although not notch brittle, was still considerably less notch ductile than G or L. The effect of a small plate volume as illustrated by B is to make the material even more notch brittle as indicated by the very short rupture lives of this sample. In other work done in our laboratory and by others(4) it also has been found that 718 structures having boundaries free of Ni_3Cb plates are very notch brittle.

After Exposure. Examination of the microstructures of the six pancakes after stressed exposure at 1250° and 1300°F indicated the following changes had occurred during exposure.

1) The gamma-prime had begun to coarsen and form a plate-like morphology in the matrix. This was tentatively identified as the gamma-prime → $Ni_{(x)}Cb$ transformation discussed by Barker, Ross and Radavich.(2) These effects were more pronounced in the 1300°F samples and for each temperature were most advanced in Sample L.

2) The samples also showed Ni_3Cb plates which had formed during exposure. (This was proven by the different etching characteristics of this Ni_3Cb as compared to that which formed during the original solution treatment.) Preliminary X-ray diffraction results indicate that sigma is present in L after the 1300°F exposure. Based on the work of Barker et. al.,(2) it was also considered possible that body-centered-cubic alpha-prime and Laves might be present in L.*

3) A grain boundary depletion effect was evident in all the specimens exposed at 1300°F and all except possibly B and H at 1250°F whenever Ni_3Cb was formed in the grain boundaries. Careful electron microscopy study showed that these zones were not depleted in the gamma-prime that formed during aging before exposure. Instead, during exposure plate-like growth of Ni_3Cb occurred in these grain boundary zones at the expense of the gamma-prime creating the denuded appearance.

These changes are illustrated in the light and electron micrographs shown in Fig. 18 through Fig. 20.

Fig. 18 taken from Forging J illustrates the darkening of the microstructure which was typical in all the specimens after both the 1250°F and 1300°F exposures due to gamma-prime coarsening. Interestingly enough, hardness surveys suggested that little or no overaging had occurred in any of the specimens except L. While most of the specimens showed either no change or a 1-2 point gain in Rockwell A hardness, L had an approximate 2 point Rockwell A loss in hardness as a result of the 1300°F exposure.

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*This is presently being checked by means of X-ray diffraction.

The electron micrographs from B (Fig. 19 and Fig. 20) serve to illustrate in detail some of the microstructural changes which occurred to varying degrees in all the samples during stressed exposure at 1300°F. Fig. 19 shows the $\gamma' \rightarrow Ni_xCb$ transformation; the Ni_xCb appears as the very short matrix plates. This figure also illustrates the depletion effect adjacent to the Ni_3Cb plates at the grain boundaries.

Fig. 20 illustrates both grain boundary and matrix Ni_3Cb which presumably formed during the stressed exposure. Note the depleted zones adjacent to the particles.

Relating microstructure after exposure to rupture properties produces the following general observations.

1) With the exception of D, rupture ductilities improved significantly after exposure. It appears this can be attributed primarily to the weakening of the matrix by the $\gamma' \rightarrow Ni_xCb \rightarrow Ni_3Cb$ transformation.

2) The depletion effect adjacent to Ni_3Cb plates formed at grain boundaries or in the matrix during exposure did not promote notch brittleness.

3) The generally poor notch behavior of D during exposure may be related to the very large Ni_3Cb plates found in this specimen before exposure.

SUMMARY

In this work an attempt has been made to study quantitatively the interaction of certain forging parameters, grain size, and microstructure and their effect upon the stress-rupture properties of Alloy 718 before and after stressed exposure. It is very clear that a given sample of Alloy 718 can be forged to produce stress-rupture properties ranging from very good to very poor, i.e., excellent strength and good notch ductility to poor strength and/or notch brittle behavior. It is also apparent that the finest grain is not necessarily the best for optimum properties or is a duplex grain size necessarily detrimental just because it is duplex.

A good correlation was established between weighted grain size values and the notch rupture ductilities of the forgings. A regression analysis is being carried out to establish the relationships between rupture ductility and various forging parameters.

The work on uniform grain size specimens before exposure suggests that a grain size of ASTM 6-7 is optimum for the best balance of stress-rupture properties. At coarser grain sizes, ASTM 5 or coarser, the risk of notch brittle behavior becomes serious and at grain sizes in excess of ASTM 7 the reverse occurs - a serious loss in strength.

In duplex-grained structures the two major facts to be considered are the percentage and size of the coarse grains. It appears that duplexing can be tolerated if the percentage of coarse grains is not in excess of 50-60% and if the coarse grains are not appreciably larger than ASTM 4. Therefore, it might be concluded that an essentially fine-grained structure having a few coarse grains, or any duplex structure for that matter, would have a probability for notch failure which is proportional to the size and number of the coarse grains.

The results of microstructural studies indicate that increasing Ni_3Cb plate size, associated with increasing grain size, decreases rupture ductility. Of somewhat lesser influence, a decreasing volume of Ni_3Cb plates appears also to decrease rupture ductility. Grain-boundary films were most noticeable in those forgings made with the higher working temperatures but appeared to have no real influence on stress-rupture properties.

The exposures to simulate over-temperature operation, with the exception of one forging (D), improved notch ductility with a general sacrifice in rupture strength. This was attributed primarily to the weakening of the matrix by the $\gamma' \rightarrow Ni_xCb \rightarrow Ni_3Cb$ transformation, the degree of which determined the loss in rupture strength and gain in notch ductility. Excepting Forging D, it would appear that accidental over-temperature exposure up to 1250°F could be tolerated without fear of catastrophic failure. The coarse grained forgings, (B and J) showed excellent rupture properties after 1250°F exposure but, unfortunately, were notch brittle before exposure. Use of a different heat treatment undoubtedly would remedy this problem.

Examining the behavior of Forging D suggests that it was so embrittled before exposure that it failed in a brittle manner during exposure. This seems a likely possibility since D contained 90% ASTM 3 grain size which was the coarsest grain size studied. As a further correlation, this specimen contained the largest Ni_3Cb plates found; these would serve as paths for rapid micro-crack propagation. The main conclusion to be derived from the behavior of D is that if the structure is too abnormal, it will not become more notch ductile during exposure but, instead will act in a notch brittle manner.

Exposure at 1300°F for 500 hours produced a severe drop in resultant rupture strength which would seriously affect the life of a component. It would seem therefore that the maximum over-exposure limit for a 500 hour exposure is 1250°F.

REFERENCES

1. H. L. Eiselstein, "Age-Hardenable Nickel Alloy", U. S. Patent 3,046,108, July 24, 1962.
2. J. F. Barker, E. W. Ross, and J. F. Radavich, "Long-Time Stability of Inconel 718", General Electric Company Report No. R67FPD 379, January 5, 1968.
3. H. L. Eiselstein, "Metallurgy of a Columbium-Hardened Nickel-Chromium-Iron Alloy" ASTM STP No. 369 (1965).
4. J. F. Radavich, Private communication.

TABLE I. Composition of Alloy 718 Material Used in Investigation

Heat	C	Si	Mn	S	P	Cr	Ni	Mo	Co	Al	Ti	Cb	T
C50615	.03	.07	.02	.004	.003	17.64	53.00	2.90	.10	.43	1.04	5.10	.0

TABLE II. Mechanical Properties of 4-1/2" RCS Billet Ht. C50615 Used in This Investigation

Test Conditions	UTS ksi	.2YS ksi	Elong. %	R.A. %	Life Hrs.	BHN
			Transverse Billet Testing			
R.T.	207	174	14	24		415
	210	181	17	28		
	209	180	16	27		415
	208	179	16	21		
1200°F	174	157	19	30		
	171	154	19	32		
1200°F/100 ksi			16		227	
			10		204	
1300°F/75 ksi			14		38	
			20		41	
			3:1 Upset Testing			
R.T.	206	178	19	37		429
1200°F	167	148	15	20		
1200°F/110 ksi			6		337	
			7		65	
1300°F/75 ksi			7		125	

NOTE: All samples given 1750°F/1 hr./AC + 1325°F/8 hrs./FC 100°F/hr. to 1150°F/8 hrs./AC heat treatment.

TABLE III. Forging Procedures Used in Investigation

Pan-cake Ident-ity	Heating Temp.°F	Reduction	Re-Heating Temp.°F	Reduction	Finish-ing Temp.°F
A	2050	3" to 3/4" (75%)*	-	-	1840
B	2050	3" to 3/4" (75%)	-	-	1930
C	2050	3" to 1-1/4" (42%)*	2050	1-1/4" to 3/4" (40%)*	1820
D	2050	3" to 1-1/4" (42%)*	1950	1-1/4" to 3/4" (40%)*	1740
E	1950	3" to 1-1/4" (42%)*	2050	1-1/4" to 3/4" (40%)*	1820
F	1950	3" to 3/4" (75%)*	-	-	1780
G	1950	3" to 3/4" (75%)	-	-	1830
H	1950	3" to 1-1/4" (42%)*	1950	1-1/4" to 3/4" (40%)*	1770
I	1950	3" to 1-1/4" (42%)	1950	1-1/4" to 3/4" (40%)*	1780
J	1950	3" to 7/8" (71%)*	1900	7/8" to 3/4" (14.5%)*	1720
K	2000	3" to 1" (67%)*	1900	1" to 3/4" (25%)*	1670
L	1850	3" to 3/4" (75%)	-	-	1680
M	1950	3" to 1-1/2" (50%)	1900	1-1/2" to 3/4" (50%)	1640

*Paused several seconds between consecutive hammer blows.
>Denotes "finer than".

TABLE IV. Grain Size and Stress-Rupture Properties

Pancake Identity	Heat Treated ASTM Grain Size*	Weighted ASTM Grain Size	1200°F/110,000 psi Life Hrs	Elong. %	R.A. %
A	6	-	127	7.0	19.5
			71	10.5	12.5
B	5	-	0.4	NF	-
			0.7	NF	-
C	7 (50) & 4	5.1	121	5.5	12.5
			91	8.0	12.5
D	10 (10) & 3	3.2	24	4.0	10.0
			61	NF	-
E	8 (60) & 3	4.9	103	10.0	14.5
			70	7.0	15.0
F	8	-	24	12.0	26.5
			117	16.0	33.0
G	7	-	138	10.5	24.0
			174	8.0	24.0
H	8 (60) & 4	5.7	203	5.5	18.0
			156	7.0	21.5
I	8 (15) & 3	3.4	82	5.5	11.5
			149	NF	-
J	3.5 few 8 (1)	3.5	87	NF	-
			152	NF	-
K	6	-	107	9.0	12.5
			151	7.5	15.5
L	9	-	70	22.0	35.5
			84	13.0	32.0
M	10 (75) & 5	7.8	41	10.0	25.5
			90	21.0	25.5

*Number in parenthesis is the percentage of the finer grain size that is present in the structure.
Note - NF indicates a notch failure.

TABLE V. Stress-Rupture Properties Before and After Stressed Exposure

Pan-cake Ident.	ASTM Grain Size		Before Exposure			After 500-Hour Exposure Conditions Indicated				
						1250°F/50,000 psi			1300°F/50	
	Actual	Weighted	Life	Elong.	R.A.	Life	Elong.	R.A.	Life	El
B	5.0		0.4	NF	-	172	10.5	12.5	62	15
			0.7	NF	-	191	10.0	15.5	38	21
D	10 (10) & 3	3.2	24	4.0	10.0	Broke at threads during exposure (72 hrs)			Broke at during ex (387	
			61	NF	-	Broke at threads during exposure (243 hrs)			24	14
G	7.0		138	10.5	24.0	100	19.0	30.5	15	27
			174	8.0	24.0	70	15.0	34.5	21	22
H	8 (60) & 4	5.7	203	5.5	18.0	49	15.0	26.0	19	18
			156	7.0	21.5	102	15.0	25.5	18	23
J	3.5, few 8(1)	3.5	87	NF	-	130	8.0	8.5	21	24
			152	NF	-	133	6.5	9.0	24	12,
L	9.0		70	22.0	35.5	18	15.5	24.0	Broke in during exp hrs, 8.7% R.A.)	
			84	13.0	32.0	26	24.0	43.0		
									1.8	24

NOTE: All stress-rupture tests conducted at 1200°F and 110,000 psi. NF indicates failure.

TABLE VI. X-Ray Diffraction Study

Sample Identity		Condition	MC	Ni_3Cb	TiN	Ti_2S,C
B	1	As Forged	m	-	m	w
	2	AF + 1750°F	m	m		
	3	AF + 1750°F + age	s	s		
D	4	As Forged	s	-	m	vw
	5	AF + 1750°F	s	s		
	6	AF + 1750°F + age	s	vs		
G	7	As Forged	m	-	m	?
	8	AF + 1750°F	m	s		
	9	AF + 1750°F + age	m	vvs		
H	10	As Forged	s	-	m	vw
	11	AF + 1750°F	s	s		
	12	AF + 1750°F + age	s	vs		
J	13	As Forged	s	-	m	vw
	14	AF + 1750°F	s	s		
	15	AF + 1750°F + age	s	vs		
L	16	As Forged	m	s	?	?
	17	AF + 1750°F	m	vvs		
	18	AF + 1750°F + age	m	vvs		

Notes:
s = strong
m = medium
w = weak
v = very
? = questionable identification

TABLE VII. Relative Abundance of Second Phases in Microstructure Before Exposure

Sample	Ni_3Cb Plate Size	Ni_3Cb Plate Volume	Grain Boundary Film	ASTM Grain Size	Stress-Rupture Ductility % Elong.*
B	Large	Small	Some	5.0	Notch Failures
D	Mixed	Large	Some	10 (10%) & 3	2.0
G	Medium	Medium	Little	7.0	9.2
H	Mixed	Medium	Little	8 (60%) & 4	6.2
J	Large	Large	Little	3.5 few 8 (1%)	Notch Failures
L	Small	Large	Little	9.0	17.5

*Average of 2 tests

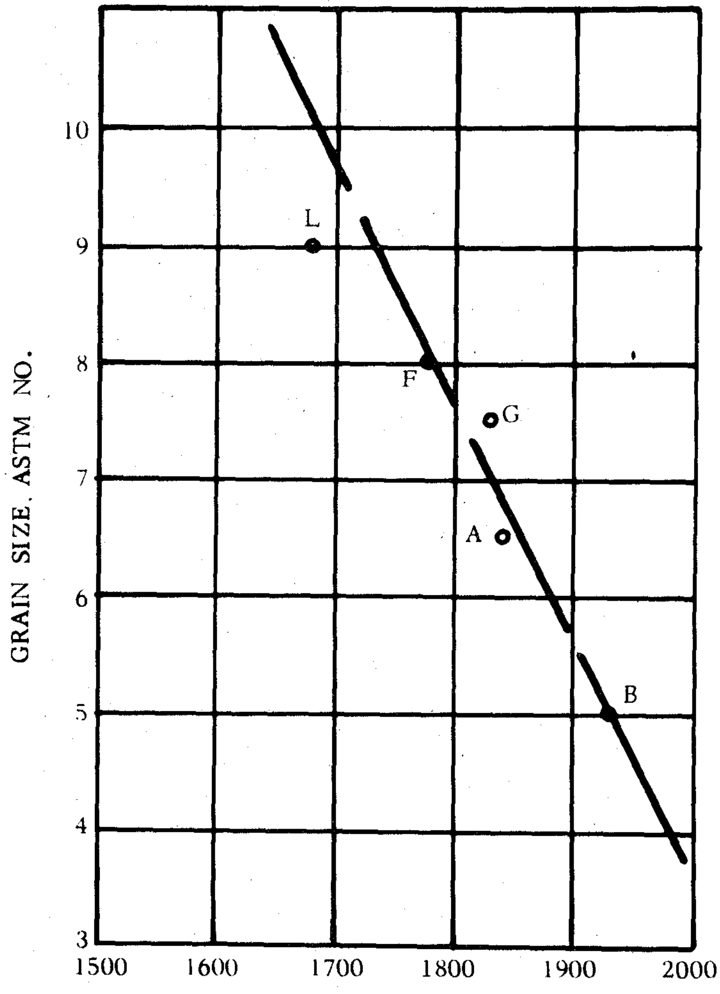

Fig. 1. Influence of Finishing Temperature on the As-Forged Grain Size of Pancakes Made Without Reheating

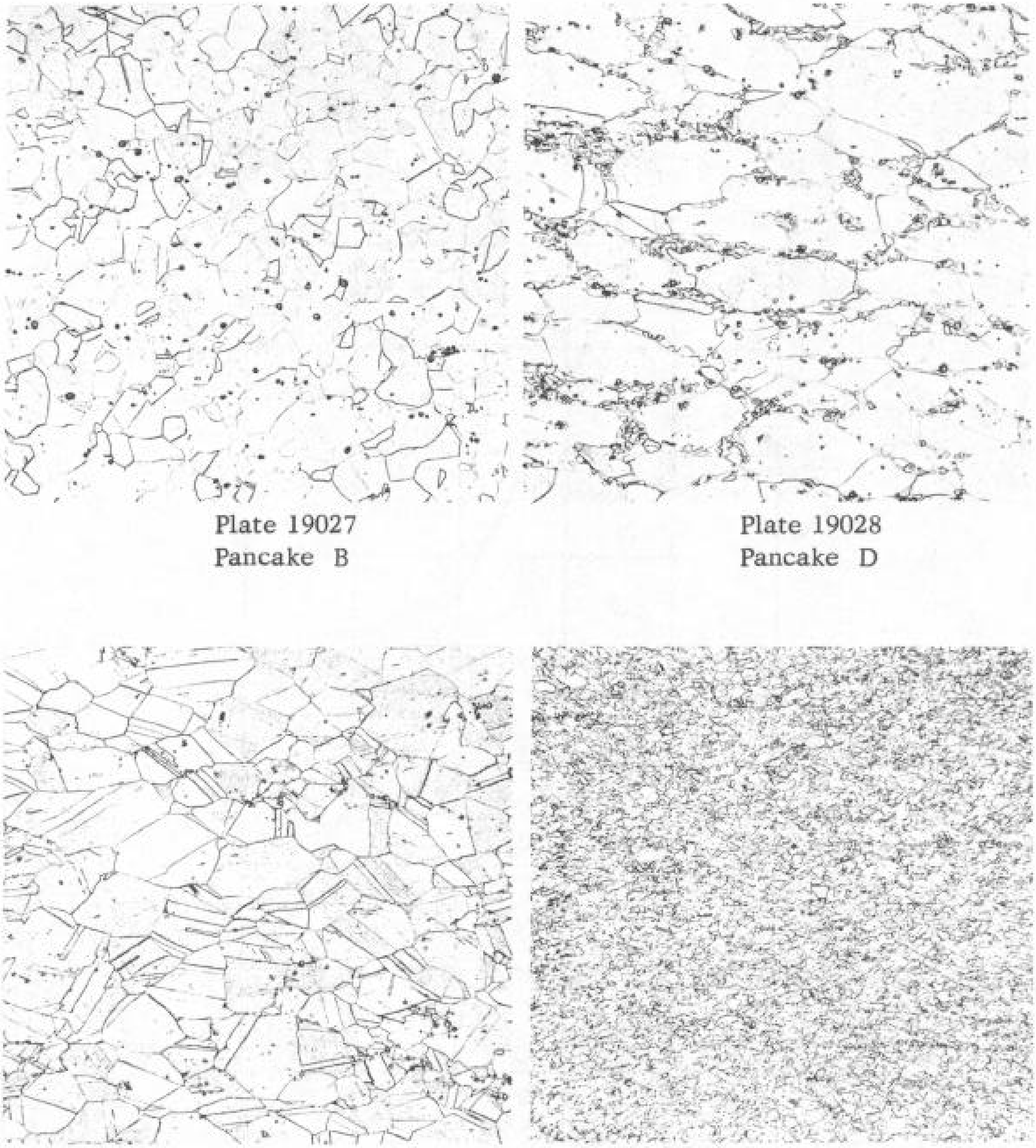

Plate 19027
Pancake B

Plate 19028
Pancake D

Plate 19207
Pancake J

Plate 19031
Pancake L

Fig. 2. Microstructures Typical of Those Exhibited by the As-Forged Pancakes
Etchant: HCl + H_2O_2 100X

H-72140

H-72140

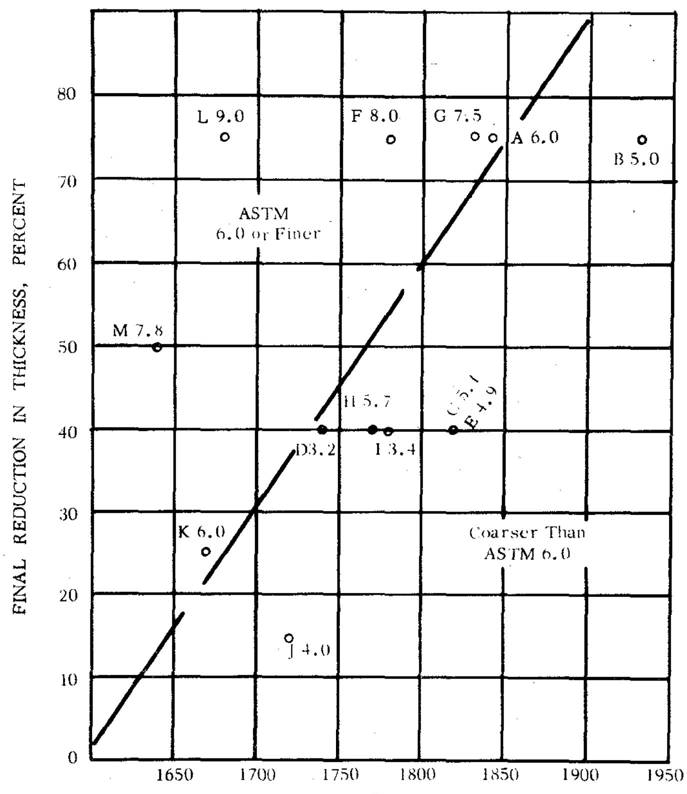

Fig. 3. Influence of Final Reduction and Finishing Temperature on Heat-Treated Grain Size

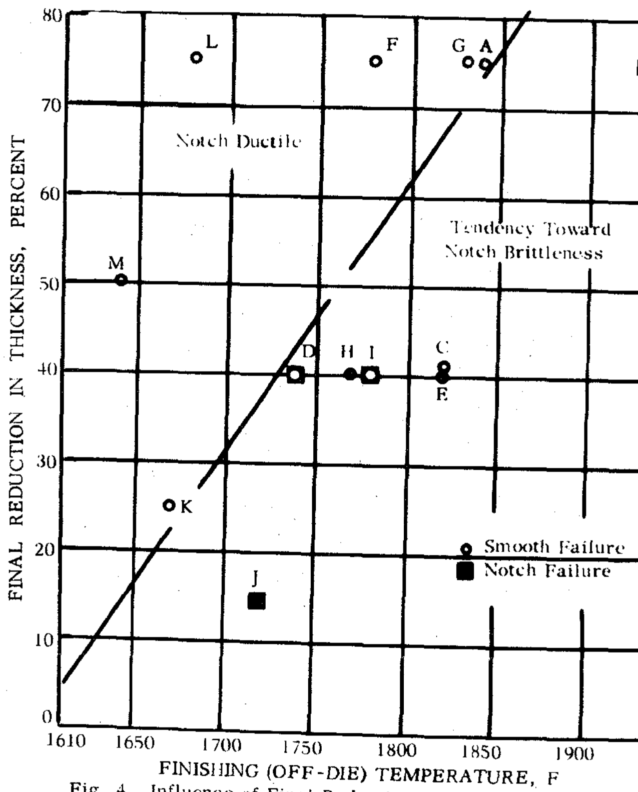

Fig. 4. Influence of Final Reduction and Finishing Temperature on Notch Sensitivity

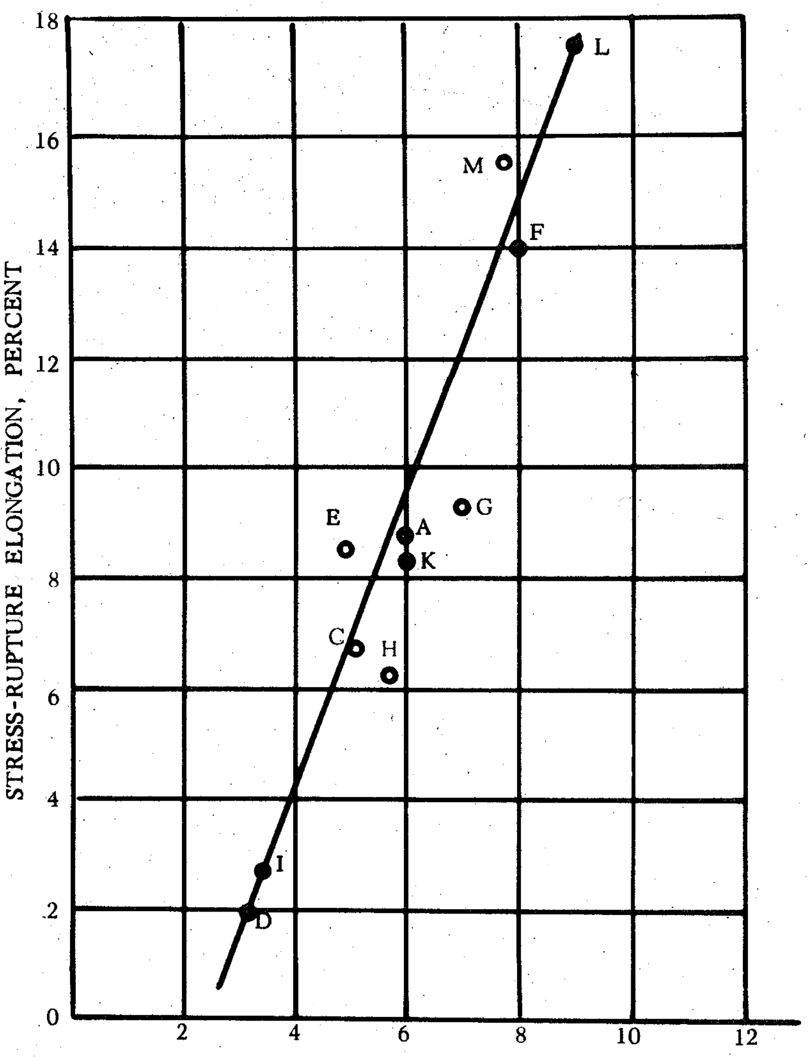

Fig. 5. Influence of ASTM Grain Size on Stress-Rupture Ductility

H-72141

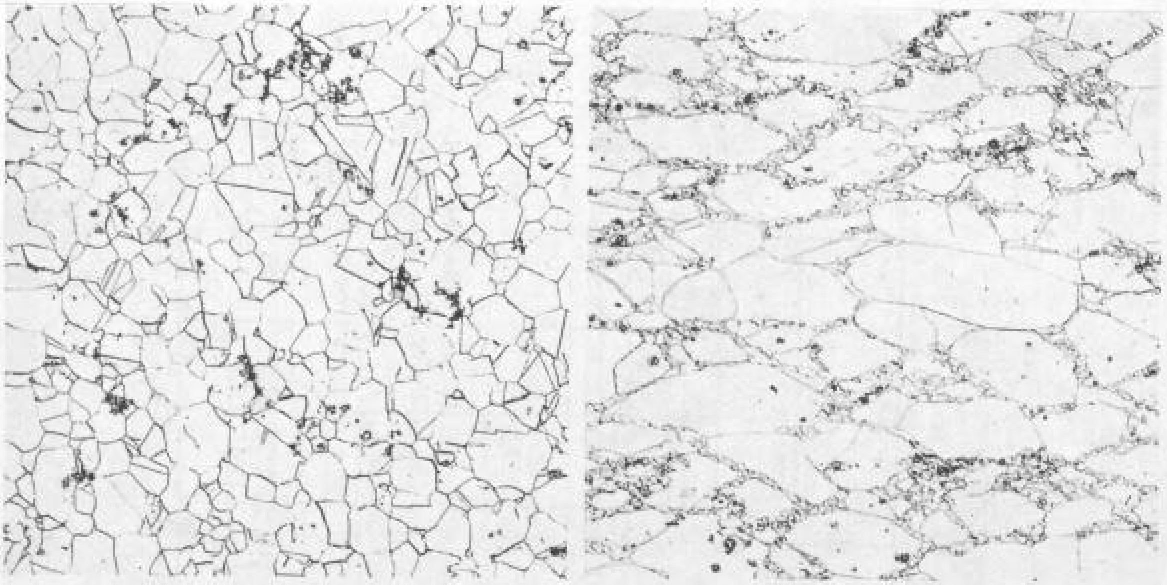

Plate 19138
Pancake B

Plate 19141
Pancake D

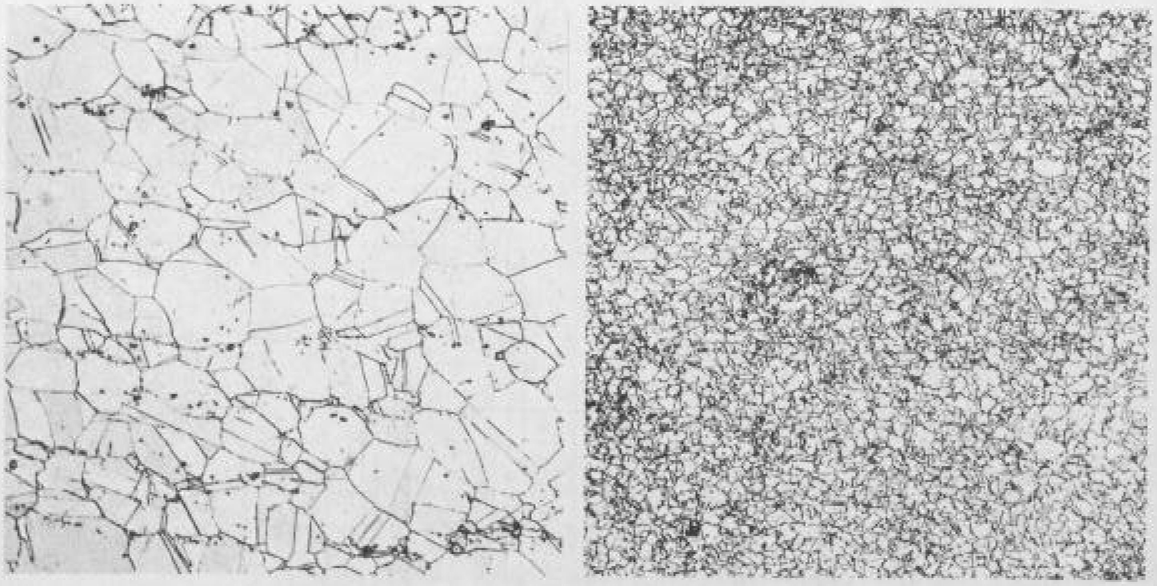

Plate 19135
Pancake J

Plate 19137
Pancake L

Fig. 6. Microstructures Typical of those Exhibited by Heat-Treated Specimens
Etchant: $HCl + H_2O_2$ 100X

H-72141

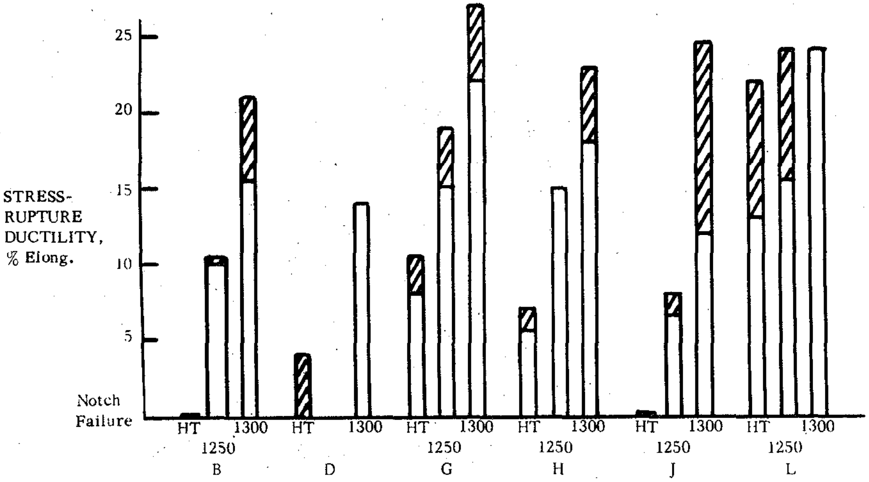

Fig. 7. Stress-Rupture Ductility Before and After 500 Hour Exposure at 1250F-50 Ksi and 1300F-50 Ksi

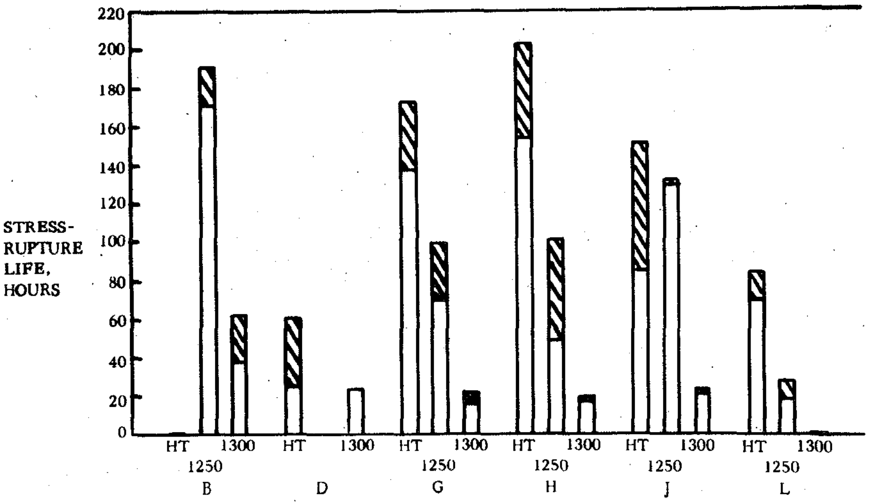

Fig. 8. Stress-Rupture Life Before and After 500 Hour Exposure at 1250F-50 Ksi and 1300F-50 Ksi

H-72016

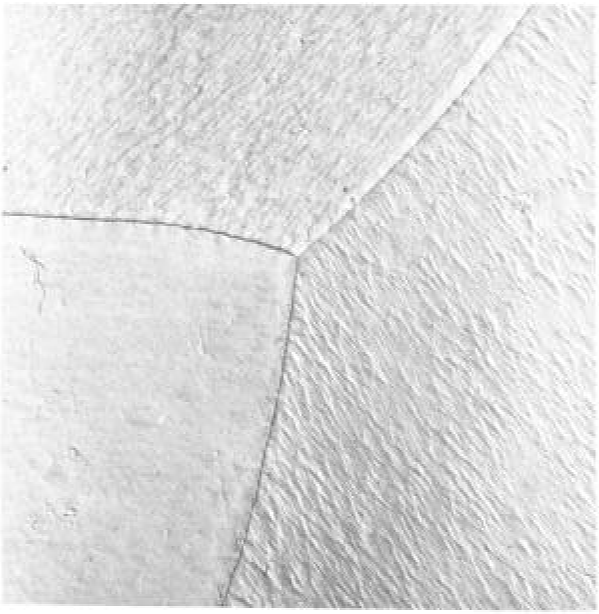

Plate 654-4

Fig. 9. Electron Micrograph of Pancake B in the Forged Condition

7500X

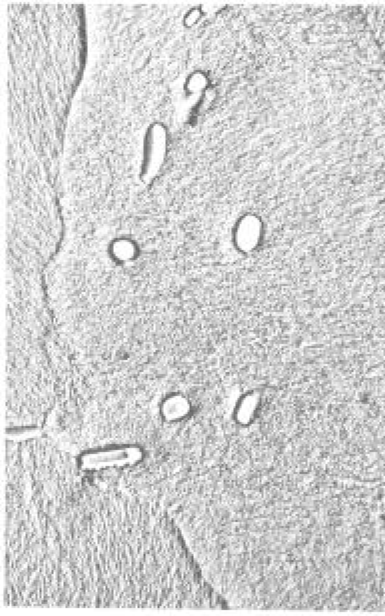

Plate 6533-4

Fig. 10. Electron Micrograph of
Forged Condition

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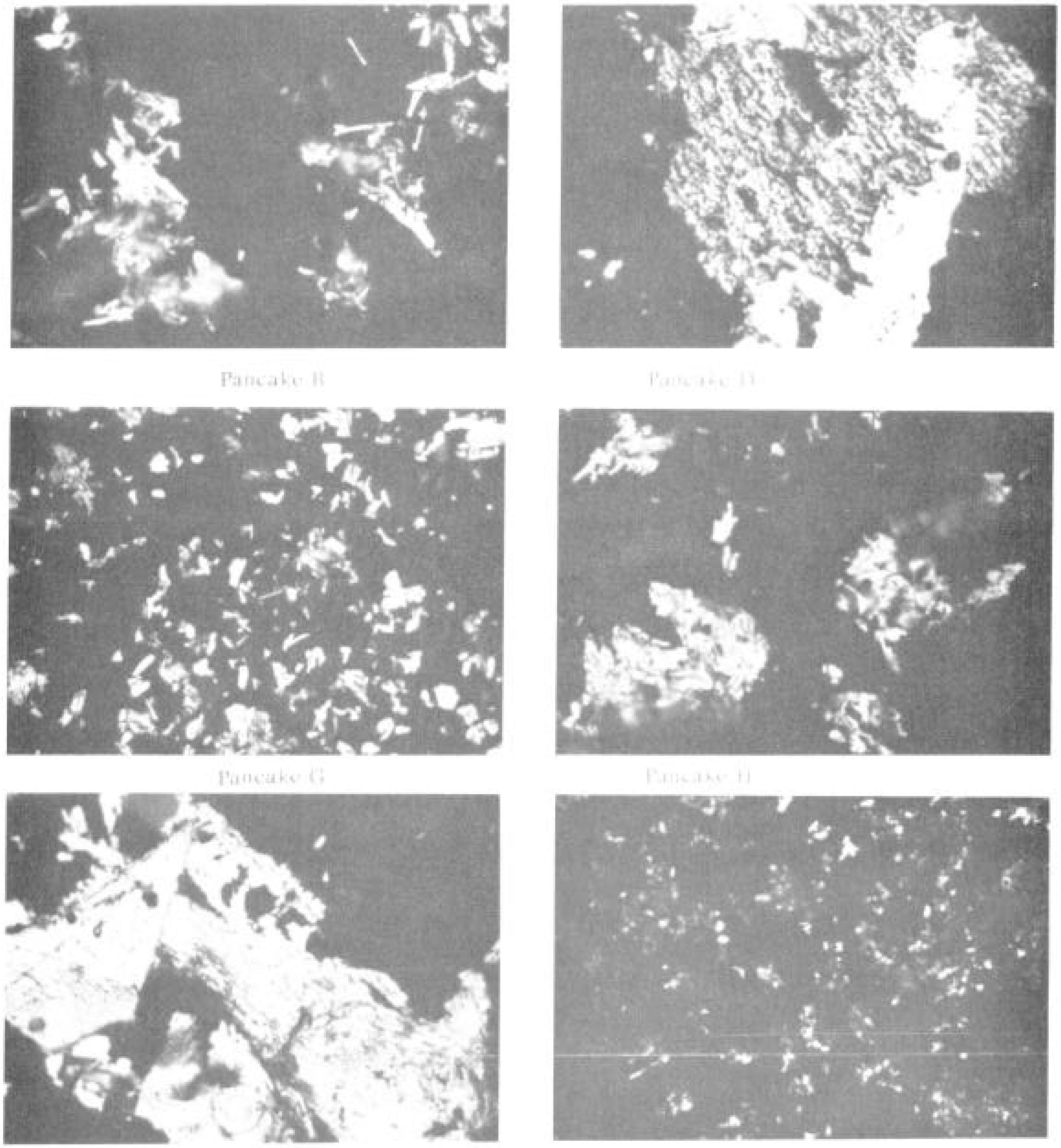

Pancake B

Pancake D

Pancake G

Pancake H

Pancake J

Pancake L

Fig. 11. Micrographs of Residues Extracted in 10% HCl - Methanol Solution

800X

H-72142

H-72142

H-72011

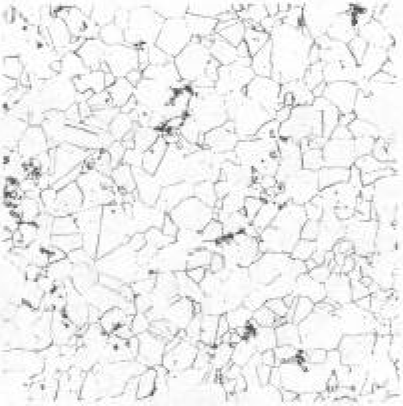

Plate 19138
100X

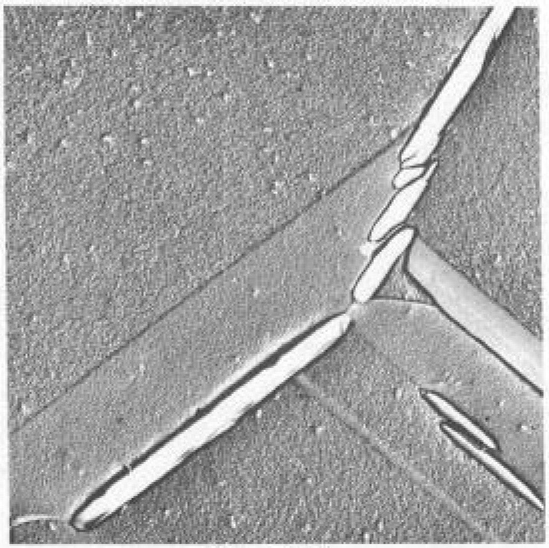

Plate 6534-2
7500X

Fig. 12. Micrographs of Pancake B in the Heat-Treated Condition

Plate 19141
100X

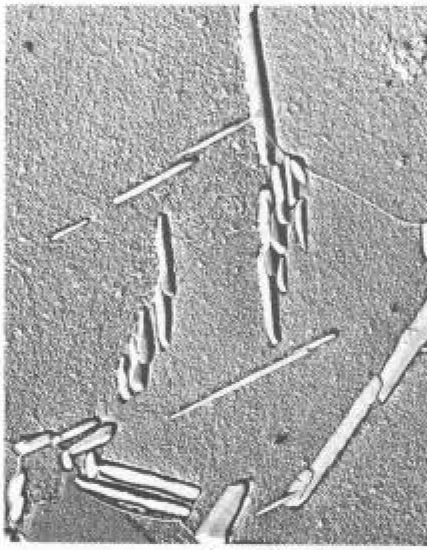

Plate 6836-1
7500X

Fig. 13. Micrographs of Pancake D in th
Condition

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H-72011

H-72012

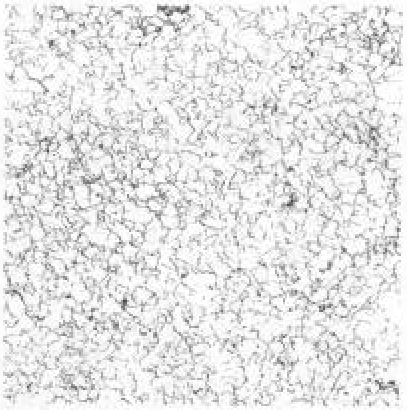

Plate 19131
100X

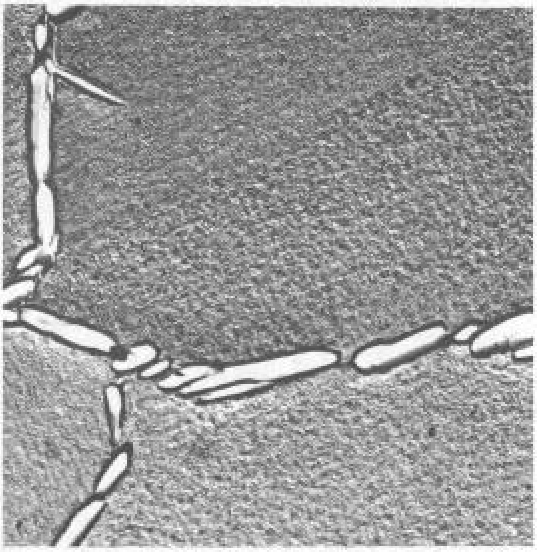

Plate 6538-2
7500X

Fig. 14. Micrographs of Pancake G in the Heat-Treated Condition

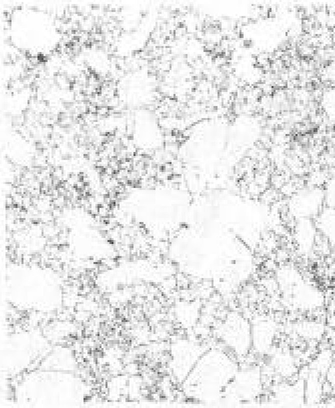

Plate 19132
100X

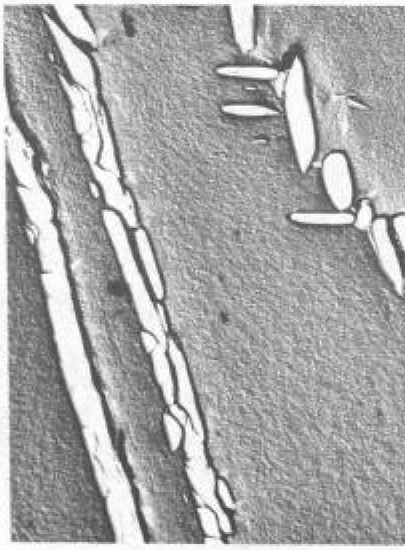

Plate 6543-2
7500X

Fig. 15. Micrographs of Pancake H in
Condition

H-72012

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H-72013

Plate 19135
100X

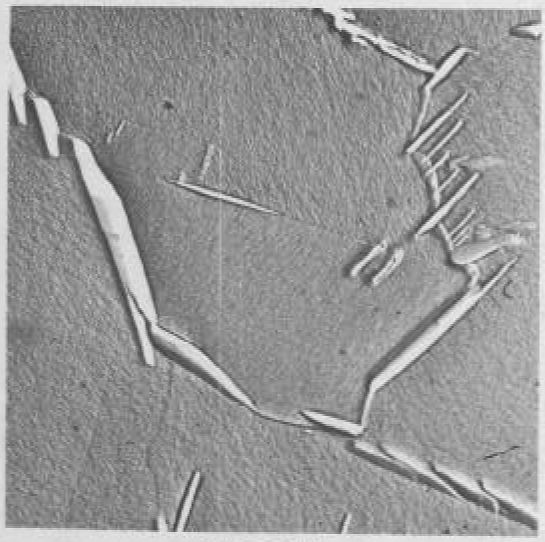

Plate 6544-1
7500X

Fig. 16. Micrographs of Pancake J in the Heat-Treated Condition

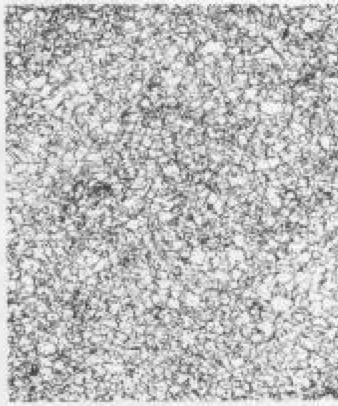

Plate 19137
100X

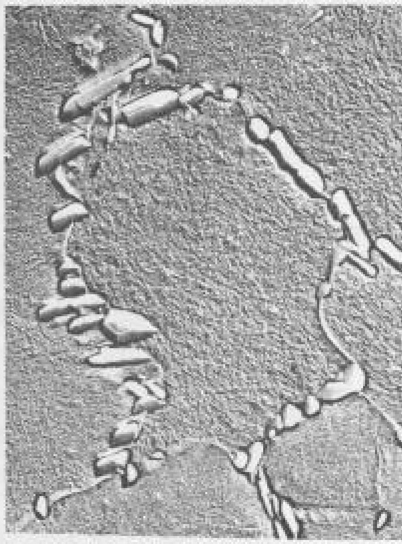

Plate 6541-3
7500X

Fig. 17. Micrographs of Pancake L in
Condition

Reduce 30% for publication

H-72013

Plate 19175
1250F -- 50 ksi

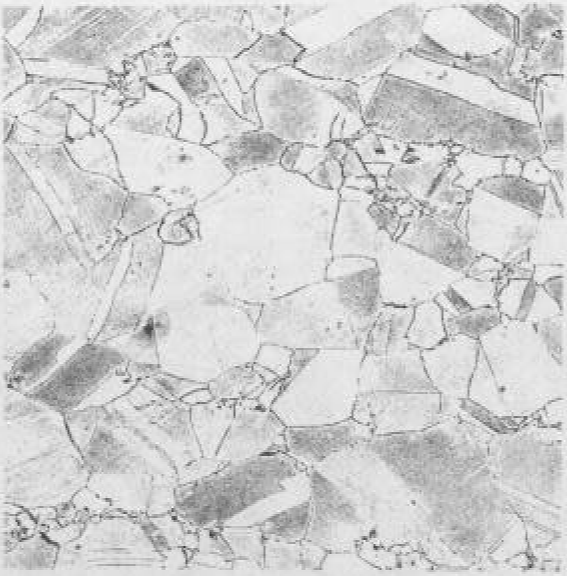

Plate 19173
1300F -- 50 ksi

Fig. 18. Light Micrographs of Pancake J After 500 Hour Stressed Exposure as Indicated and Subsequent Testing at 1200F and 110,000 psi

100X

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H-72143

H-72014

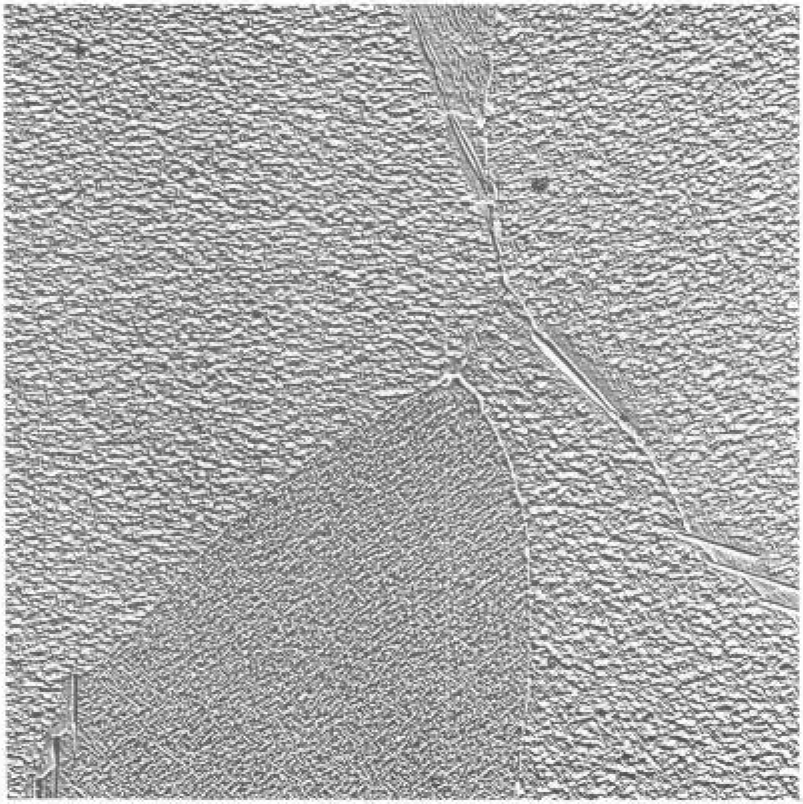

Plate 6573-3

Fig. 19. Electron Micrograph of Pancake B After 500 Hour Exposure at 1300^{O}F and 50 Ksi Showing Gamma-Prime to Ni_xCb Transformation and Depleted Zones Adjacent to Ni_3Cb Grain-Boundary Plates.

7500X

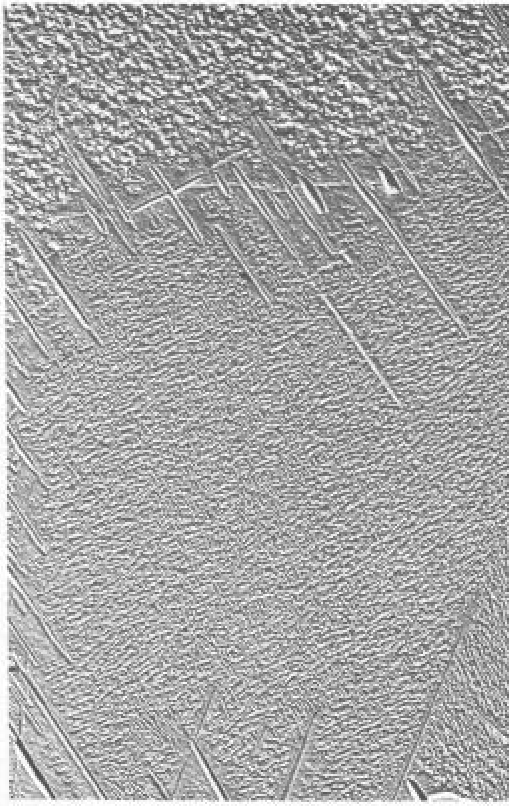

Plate 6574-3

Fig. 20. Electron Micrograph of Pa
500 Hour Exposure at 130
Ksi Showing Grain-Bounda
Ni_3Cb Plates with Adjacen

Reduce 30% for publication

H-72014

APPENDIX

Metallographic Preparation for Electron Metallography

Samples were prepared by two different techniques to show the variations in microstructure.

Procedure A - Grind through 600 grit paper and polish with Linde A alumina abrasive -- emersion etch with 2:1 HCl - H_2O_2 (3%) -- repolish and re-etch as required.

Procedure B - Grind through 600 grit paper and polish with Linde A alumina abrasive -- electropolish for two to five seconds in 10% HCl - methanol solution and -- emersion etch in HCl - methanol - H_2O_2 solution -- repolish and re-etch as required.

The polished surfaces were replicated with collodion, shadow-cast with chromium and backed with a thin layer of carbon. Electron micrographs were taken at magnifications ranging between 1750X to 3500X and optically enlarged to the magnification indicated in each figure.

X-Ray Analyses of Phases

The phases found in Alloy 718 were extracted from test specimens electrolytically in either a 10% HCl -- methanol or a 10% HCl -- water solution using a low current density. The residue was washed with fresh alcohol after decanting the acid solution. The X-ray patterns were obtained using a Philip's diffractometer using Cu radiation with a monochromator and a pulse height analyzer.

The particle shapes and sizes were studied by extracting with a 10% HCl -- methanol to obtain the second phase particles. These particles were scrapped off the surface into fresh alcohol to minimize the dissolution in the HCl solution and also to gain a better view of the smaller particles. These particles were then viewed with the optical microscope to obtain a relationship of size, shape and distribution of the relative particles.