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TECHNICAL NOTE 2678

ABNORMAL GRAIN GROWTH IN S-816 ALLOY

By A. I. Rush, J. W. Freeman,
and A. E. White

University of Michigan

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ABNORMAL GRAIN GROWTH IN S-816 ALLOY

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SUMMARY

This report presents the results obtained to date of an investigation to establish the fundamental causes of abnormal grain growth in S-816 alloy under conditions encountered during the forging of blades for the gas turbine of jet engines. The data reported are confined to the experiments conducted to learn how to produce abnormally large grains in bar stock under controlled conditions. These are being reported because they may be useful to those concerned with the problem.

Abnormal grain growth was induced by temperature cycling alone. Water-quenching of the bar stock from 2150° or 2300° F rendered the samples susceptible to abnormal grain growth upon reheating to 2300° F. Air-cooling was sufficient to induce grain growth during subsequent solution treatment for some of the prior treatments of the stock. Certain of the prior structures were susceptible to grain growth by temperature cycling only to 2150° F.

Studies of critical deformation for abnormal grain growth on subsequent solution treatment indicated that the critical deformation by rolling lies between 0 and 2 percent. The critical deformation was independent of the temperature at which the deformation was carried out. Repeated critical deformations between reheats to 2150° F resulted in much larger grains after solution-treating at 2300° F than a single critical deformation.

To varying extents the conditions for abnormal grain growth are sensitive to the prior history of the material. Several other possible variables have not been investigated. For this reason the generality of the results remains to be established. This factor, however, will probably be of no great practical significance for solution treatment at 2300° F while it will be important to the occasional cases of abnormal grain growth when temperatures are restricted to 2150° F. Apparently avoiding abnormal grain growth will require deformations in excess of 2 percent and relatively slow cooling from the forging temperature.

INTRODUCTION

The occurrence of abnormally large grains in blades for gas turbines, particularly when segregated to certain portions of steel blades, is associated with undesirable service characteristics. Because this is a general recurring metallurgical problem in jet engines, an investigation of the fundamentals of abnormal grain growth in blade alloys for jet engines was requested by the National Advisory Committee for Aeronautics.

This report covers progress to date and is confined to the results of experiments to induce abnormal grain growth under controlled conditions, a necessary preliminary step to studying the fundamental causes. Only S-816 alloy has been included to date. A report is being issued because the results may be of use to those concerned with the problem.

In general, the conditions used for the experiments are intended to approximate those used in forging S-816 blades. This has been accomplished by using heating temperatures of 2150° F. Solution temperatures of both 2150° and 2300° F, the former because it represents current practice to reduce grain growth problems and the latter because it would be desirable if grain growth could be avoided, have been used. Of necessity the experiments have been restricted to bar stock under controlled laboratory conditions.

The investigation is being conducted as part of a general metallurgical investigation of heat-resistant alloys at the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

MATERIAL

The material used for this investigation was hot-rolled and centerless ground 3/4-inch-diameter S-816 alloy bar stock from heat 61858 supplied by the Allegheny Ludlum Steel Corporation.

The composition was reported to be as follows:

C	Mn	Si	P	S	Cr	Ni	Co	Mo	W	Fe	Cb
0.38	1.28	0.23	0.011	0.019	19.51	19.91	Bal.	3.82	4.15	3.65	3.84

The as-received bar stock exhibited a grain size of 4 to 8 on two sides around a core of finer than 8 (see fig. 1). This material was

subject to uneven grain growth when solution-treated at either 2150° or 2300° F. Solution treatment at 2150° F produced an interior region of grain size 2 to 4 between a core and rim of grain size 4 to 6. Solution treatment at 2300° F developed a similar pattern of core and rim of grain size 4 to 6, but the intermediate region showed a 1 to 3 grain size (see fig. 2).

Because the uneven grain growth characteristics of the as-received stock and the uneven grain size of the material after solution treatment confused the study of the factors causing abnormal grain growth, it was considered necessary to treat the stock so that a uniform-grain-size material could be prepared for experimental purposes. The procedure used was to roll stock to a reduction in area of 15 percent at 1000° F and then solution-treat for 1 hour at 2300° F and water-quench. This produced a grain size of 4 to 7 (see fig. 3) uniformly across the bar. Varying the time of solution treatment from 1/2 hour to 4 hours did not change the grain size. Furthermore, there are probably a number of "equalizing treatments" which would produce a uniform grain size. The reduction of 15 percent at 1000° F was the first one tried and because it worked was used for the experiments being reported.

EXPERIMENTAL PROCEDURE

During forging of gas-turbine blades, the material is subject to temperature cycling from the forging temperature, to varying reductions over a range of temperatures below the initial heating temperature, and to repeated reductions with reheating to the initial forging temperature between reductions. During the course of the investigation it became evident that all three probably affected grain growth tendencies. Each has been investigated separately to a limited extent. In addition, it was considered that the initial grain growth characteristics probably were influenced by past history. Experiments have been carried out on material believed free from uneven grain growth characteristics and on samples of known prior uneven grain growth characteristics.

A typical heating temperature for forging S-816 alloy, 2150° F, was used for the experiments. Most of the samples were reheated to 2300° F for the grain growth studies because this temperature would impart desirable strength properties at high temperatures if abnormal grain growth could be avoided. Specimens were also solution-treated at 2150° F, the present commercial solution-treating temperature, because it is understood that blades are not entirely free from abnormal grain growth at this temperature.

The experimental details are described in the following sections.

Temperature Cycling

The study of the effects of temperature cycling was undertaken because anomalous results were encountered in early critical-deformation studies. It developed that stock given the equalizing treatment would grow abnormally large grains after simply reheating to 2300° F. This phenomenon was then further investigated by varying the cooling rate. As-rolled stock was reduced 15 percent at 1000° F and then heated to 2300° F for 1 hour. Varying cooling rates were obtained by air-cooling, oil-quenching, and water-quenching. The samples were again reheated to 2300° F. The water-quenched specimen developed grains as large as (-2) at the surface whereas the oil-quenched and air-cooled samples remained uniformly 4 to 7 in grain size.

Abnormal grain sizes have been considered to be all those larger than A.S.T.M. number 1. Larger grain sizes have been designated 0, (-1), (-2), and (-3), and so forth to signify increasingly larger grains using the progression in sizes as established by the A.S.T.M. rating procedure.

Because these tests indicated that cooling rate was a factor in grain growth, the following additional tests were conducted. Four different initial conditions of bar stock were heated to 2150° F and cooled in still air or water-quenched three times. The final step involved heating to 2300° F for 1 hour and water-quenching. Samples were examined for grain size between each of the reheatings to 2150° F and after the final solution treatment at 2300° F.

The initial conditions of the bar stock used were:

(a) A uniform grain growth tendency obtained by rolling to a reduction of 15 percent at 1000° F

(b) Uniform 4 to 7 grain size obtained by the equalizing treatment of rolling to a reduction of 15 percent at 1000° F and solution-treating at 2300° F for 1 hour followed by water-quenching

(c) A mixed grain size with uneven grain growth tendencies by using the as-received stock without any prior treatment

(d) A mixed grain size obtained by solution-treating the as-received stock at 2300° F for 1 hour and water-quenching

Amount and Temperature of Deformation

Tapered bars having the dimensions shown by figure 4(a) were prepared from equalized stock - that is, stock reduced 15 percent at 1000° F,

heated for 1 hour at 2300° F, and water-quenched. Separate bars were rolled, isothermally at 1000°, 1400°, 1800°, 2000°, 2100°, 2150°, 2200°, and 2250° F to a straight bar. The resulting range in reductions was from 0 to about 12 percent. After rolling the bars were reheated to 2300° F for 1 hour and water-quenched.

In addition, another set of bars rolled at 1400°, 1800°, and 2200° F was solution-treated for 1 hour at 2150° F and water-quenched.

All bars were examined to establish the relationship between amount of deformation, temperature of deformation, and the resultant grain size.

Repeated Deformations

A bar having the dimensions shown by figure 4(b) was prepared from material reduced 15 percent at 1000° F and reheated to 2150° F for 1 hour and air-cooled. This prior treatment avoided any tendency for uneven or abnormal grain growth on reheating to 2300° F in the temperature-cycling work.

The tapered bar was rolled straight at 1400° F, reheated to 2150° F, and air-cooled. It was again tapered, rerolled at 1400° F, and again reheated to 2150° F. The same step was repeated once again. The result was that each section of the bar was deformed the same amount three times with reheats to 2150° F between each deformation. The deformations ranged from 0 to 3 percent. This range of deformation was selected because the results from the previous work indicated that the critical deformation was between 0 and 2 percent.

The bar was finally solution-treated at 2300° F for 1 hour and water-quenched. The grain size along the length of the bar was then established.

In all cases the specimens were aged at 1400° F for 16 hours to aid definition of grain boundaries. All rolling was carried out with flat rolls. Reductions of the tapered bars were made in one pass. During the equalizing operation the bars were rolled on both faces in two passes to give nearly square bars.

In every case the bars were placed in furnaces at temperature. In rolling, the bars were held at temperatures for 1/2 hour. The temperature drop during rolling is known to be less than 25° F.

In every case the reductions during rolling are expressed as percent reduction in cross-sectional area from the original cross-section area.

RESULTS

The experiments indicated that abnormal grain growth during solution-treating of S-816 alloy at 2300° F can be induced by prior temperature cycling alone and by critical deformations between 0- and 2-percent reduction. Abnormal grain growth was induced by simply cycling the bars at 2150° F with certain of the prior treatments. Repeated critical deformations between reheats to 2150° F greatly increased the grain size. Apparently the temperature of critical deformation has little effect. The detailed results are described in the following sections.

Temperature Cycling

The grain sizes observed after the various thermal treatments are summarized by figure 5. Macrographs showing the grain size distribution indicated in figure 5 are shown in figure 6. Figures 7, 8, 9, and 10 show typical microstructures after three reheatings to 2150° F and after the final solution treatments at 2300° F. The following comments apply to the results:

(1) The only material which did not undergo abnormal grain growth on final solution treatment at 2300° F was that initially reduced 15 percent at 1000° F and then air-cooled from the three heatings to 2150° F (see figs. 6 and 7).

(2) The material reduced 15 percent at 1000° F and then heated to 2150° F and water-quenched underwent grain growth on the surface after the first cycle at 2150° F. The surface grain size increased with each cycle and ranged from (-1) to 3 in size after the final treatment at 2300° F (see figs. 6 and 7).

(3) The material in which a uniform grain size of 4 to 7 was induced by the equalizing treatment of 15-percent reduction at 1000° F followed by 1 hour at 2300° F and water-quenching did not undergo grain growth at 2150° F when air-cooled between the three heats at 2150° F but did when water-quenched. Both the air-cooled and the water-quenched samples underwent abnormal grain growth during the final 2300° F treatment (see figs. 6 and 8).

(4) The as-received bar stock did not develop abnormally large grains when air-cooled between cycles to 2150° F. The stock water-quenched between cycles developed a surface layer of grains ranging from larger than (-1) to 2 in size during the third cycle.

Finally heat-treating the bars at 2300° F after cycling at 2150° F induced abnormally large grains, particularly in the samples which had been water-quenched from 2150° F (see figs. 6 and 9).

(5) The nonuniform-grain-sized material produced by solution-treating the as-received stock at 2300° F underwent grain growth in the fine-grained surface layer after either air-cooling or water-quenching from 2150° F. Both materials developed abnormally large grains during the final 2300° F treatment, except for a small section of the bar at the center (see figs. 6 and 10).

Amount and Temperature of Deformation

The grain sizes of the bars isothermally reduced from 0 to 12 percent at 1000° to 2250° F by rolling and subsequently solution-treated at 2300° or 2150° F are summarized in table I. These results indicate that:

(1) The critical reduction for abnormal grain growth was from 0 to about 2 percent

(2) The critical reduction was nearly independent of temperature of reduction up to 2250° F

(3) The grain size in the critically reduced section was larger than A.S.T.M. number 1 when solution-treated at 2300° F and 3 to 5 when treated at 2150° F

The leading sections of the tapered bars which had not been reduced during the rolling of the tapered bars showed abnormally large grains on the surface. There are two possible sources of induced grain growth at this point. One was the final quench from 2300° F of the equalizing treatment used to prepare the stock, as suggested by the temperature-cycling data. The other was the cold-working of the surface during the machining of the tapered bar. In either case this effect is not believed to have influenced the critical-reduction results because the grain growth was uniform through the cross section of the bar in the critically reduced sections.

Repeated Critical Deformations

The bar which was taper-rolled three times to reductions from 0 to 3 percent with reheats to 2150° F between reductions and finally solution-treated at 2300° F exhibited the grain sizes shown by figure 11. Typical photomicrographs are shown by figure 12. The main conclusion derived from this experiment was that repeated critical reductions

apparently result in much larger grain sizes than a single critical reduction. Whereas in the previous section the largest grains developed after a single critical reduction were somewhat larger than 1 in size, these repeated deformations developed grains as large as (-3) at 0.75-percent reduction.

The conclusion that repeated critical deformations lead to much larger grains must be further verified because the prior treatment used for the stock differed from that used for the study of the effect of degree and temperature of reduction in the previous section. The procedure used was the only one in the temperature-cycling studies which did not lead to abnormal grain growth - that is, 15-percent reduction at 1000° F followed by air-cooling from 2150° F. In this case, the portion of the bar which did not receive any reduction remained fine-grained as it did in the temperature-cycling studies. The difference in prior history, however, could have changed the grain size induced by a single critical reduction from that obtained in the experiments described in the previous section.

DISCUSSION

The most important results obtained were:

(1) Abnormal grain growth can be induced by temperature cycling alone

(2) Critical deformation for grain growth apparently is independent of the temperature of deformation

(3) Repeated critical deformations between reheats to 2150° F produce much larger grains than a single critical deformation

(4) Grain growth characteristics, as judged by the temperature-cycling results, are quite sensitive to prior history

(5) Abnormal grain growth can be induced by temperature cycling alone to temperatures of 2150° F with certain prior histories - a final treatment at 2300° F is not necessary to develop grains larger than 1

These results by no means cover all the possible conditions inducing abnormal grain growth in S-816 alloy. The discovery of the temperature-cycling effect, however, cleared up a number of anomalous effects initially encountered. Thus it now seems feasible to proceed with further investigation to clear up prior-history effects and study the fundamentals of the mechanism of grain growth.

From a practical viewpoint it appears that abnormal grain growth at 2300° F can be avoided only by keeping deformations in excess of about 2-percent reduction. In so doing, however, cooling rates from forging should be kept at least as slow as air-cooling of the bar stock used in this investigation, otherwise rapid cooling will, in itself, induce grain growth upon reheating to 2300° F.

The cause of the development of susceptibility to grain growth by temperature cycling from 2150° F alone remains to be definitely established. It is presumed due to critical strain induced by rapid cooling, to alteration of the grain growth restrainers, or to strain gradient effects. Orientation effects may also be a factor. The possibility also exists that the rate of strain may have an important effect on the critical deformation.

It seems very evident from the investigation that prior history has an important influence on grain growth tendencies when the material is exposed to the conditions inducing grain growth. Because this factor has been studied only briefly the generality of the results reported is open to question. It is believed, however, that the practical conditions are quite well-defined. Prior history will influence the exact cooling rate or the range of critical reductions for a given grain size upon solution treatment at 2300° F but will not change the necessity for having more than 2-percent reduction and for slow cooling after forging. Prior history, however, probably will be important to abnormal grain growth in those operations where temperatures, including solution treatment, are restricted to 2150° F.

CONCLUSIONS

The conclusions derived from this investigation of the conditions leading to abnormal grain growth in S-816 alloy are:

1. Abnormal grain growth can be induced by water-quenching and in many cases by air-cooling bar stock from 2150° or 2300° F. Such temperature cycling definitely makes the alloy susceptible to grain growth during final solution treatments.

2. The critical deformation during rolling for grain growth was between 0 and 2 percent and was independent of the temperature of deformation.

3. Repeated critical deformations between reheats to 2150° F induced much larger grains during solution treatment than a single critical deformation.

4. The prevention of susceptibility to abnormal grain growth during solution treatment apparently requires that the material be deformed more than 2 percent and that cooling rates be kept slow.

5. Abnormal grain growth can be induced under certain conditions when working and solution-treatment temperatures are restricted to 2150° F.

6. The development of susceptibility to grain growth by temperature cycling, and by critical deformation, is sensitive to prior history. It is expected that this will be important to those cases where abnormal grain growth is encountered when temperatures are restricted to 2150° F during solution treatment and only important to the details of grain growth when solution-treated at 2300° F.

Because prior history does influence grain growth tendencies and because a number of other variables have received limited attention, the conclusions require further investigation before their generality should be accepted.

Engineering Research Institute
University of Michigan
Ann Arbor, Mich., November 12, 1951

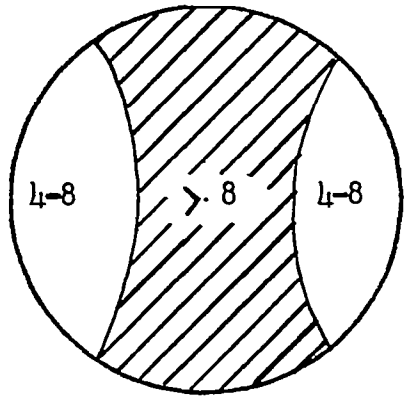
TABLE I

EFFECT OF AMOUNT AND TEMPERATURE OF DEFORMATION PRIOR TO SOLUTION TREATMENT

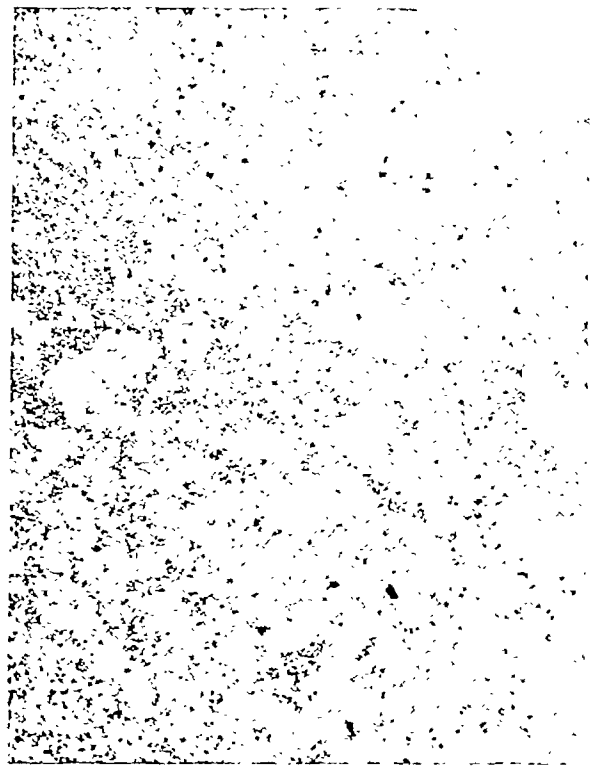
AT 2300° AND 2150° F ON GRAIN SIZE OF S-816 BAR STOCK

[Stock initially rolled 15 percent at 1000° F, solution-treated at 2300° F for 1 hr, and water-quenched]

Reduction (percent)	A.S.T.M. grain size										
	Solution-treated at 2300° F								Solution-treated at 2150° F		
	Rolling temperature (°F)								Rolling temperature (°F)		
	1000	1400	1800	2000	2100	2150	2200	2250	1400	1800	2200
Original	5-8	5-8	5-8	5-8	5-8	5-8	5-8	5-8	5-8	5-8	5-8
0 to $\frac{1}{2}$	<1	<1	<1	<1	<1	<1	<1	<1	3-4	4-5	4-5
2	<1	<1	<1	<1	<1	1-2	2-3	3-4	4-5	4-8	5-8
4	3-4	3-4	3-4	3-4	3-4	4-5	4-5	4-5	5-8	5-8	6-8
6	5-8	5-8	5-8	5-8	5-8	6-8	6-8	5-8	5-8	6-8	6-8
8	5-8	5-8	5-8	5-8	5-8	6-8	6-8	5-8	5-8	6-8	6-8
10	5-8	5-8	5-8	5-8	5-8	6-8	6-8	5-8	5-8	6-8	6-8
12	5-8	5-8	5-8	5-8	5-8	6-8	6-8	5-8	5-8	6-8	6-8

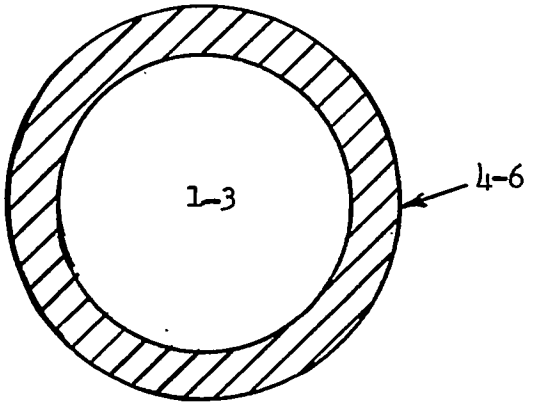


(a) Approximate distribution of grain sizes.

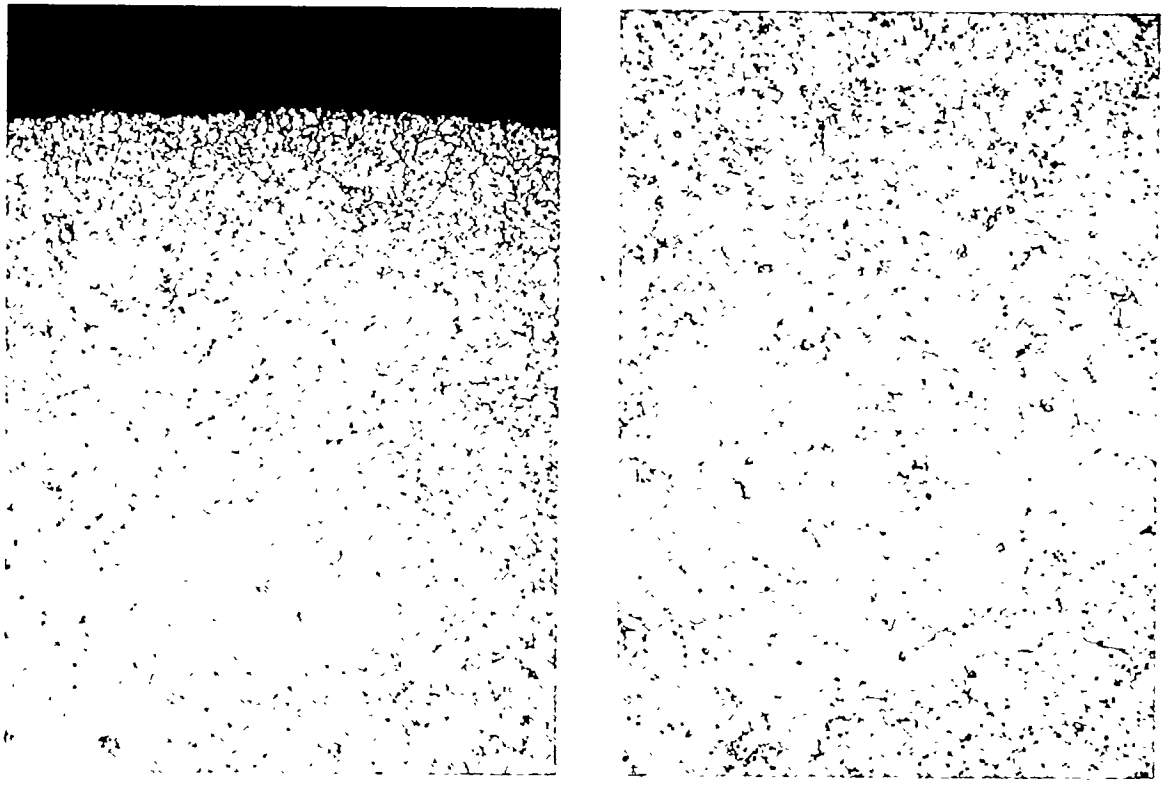


(b) Microstructure at junction of coarse- and fine-grained areas. X50.

Figure 1.- Grain size of as-received S-816 bar stock.



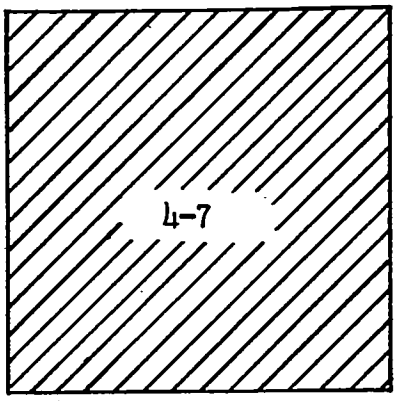
(a) Approximate distribution of grain sizes.



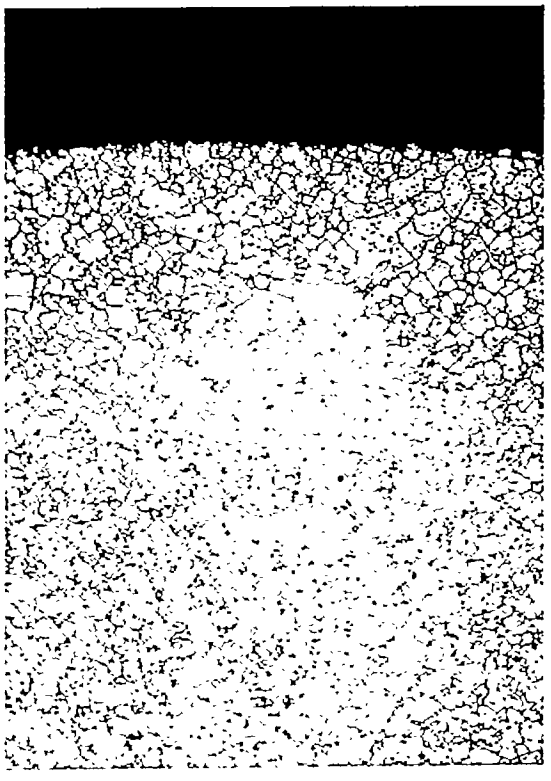
Surface Interior

(b) Microstructure of cross section of bar at surface and interior. X50.

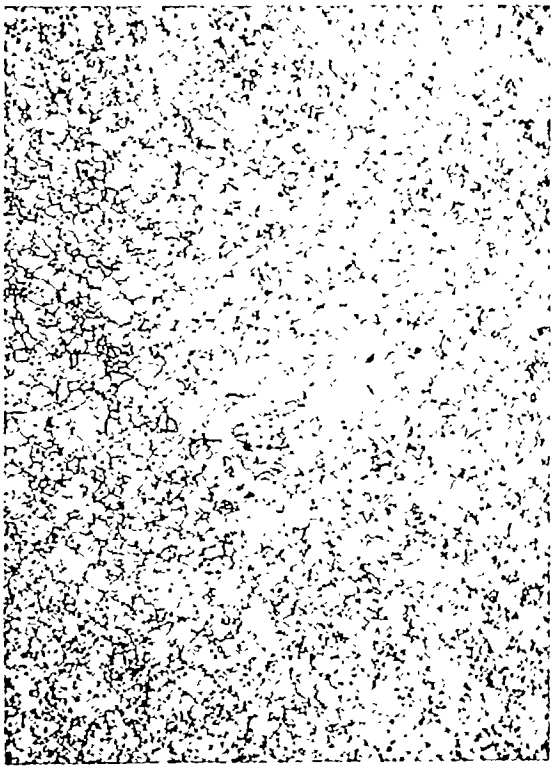
Figure 2.- Grain sizes resulting from solution-treating as-received S-816 stock at 2300° F for 1 hour and water-quenching.



(a) Uniform distribution of 4 to 7 grain size across bar.



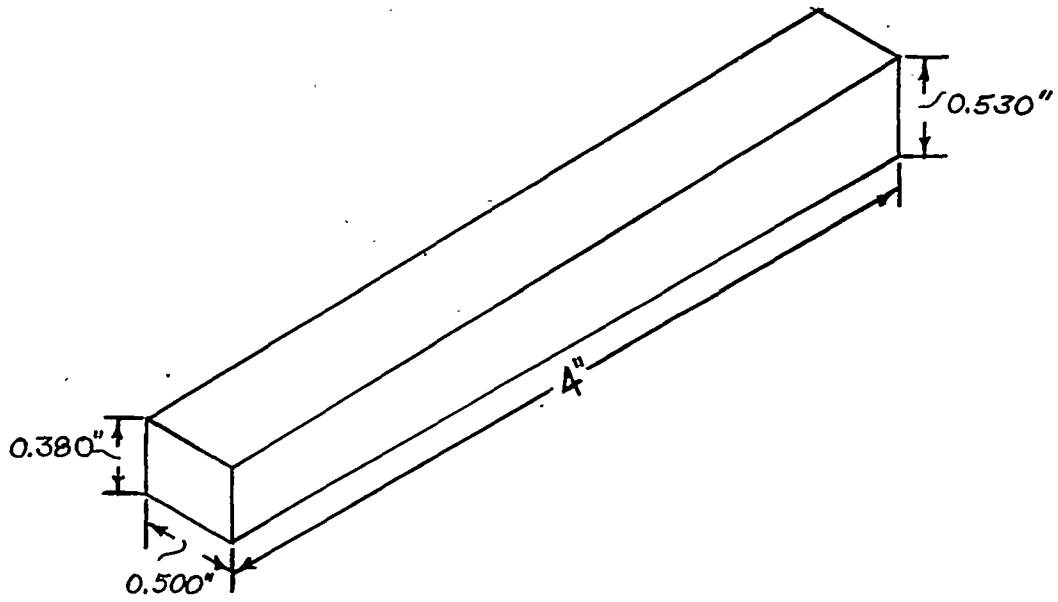
Surface



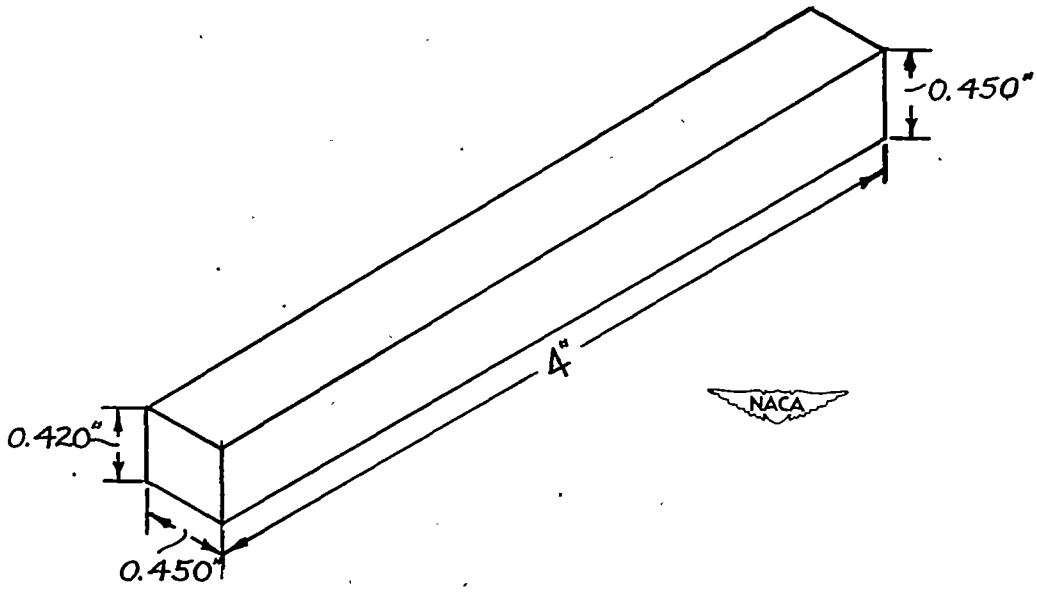
Interior

(b) Microstructure of cross section of bar at surface and interior. X50.

Figure 3.- Grain size resulting from rolling as-received bar stock to 15-percent reduction at 1000° F and solution-treating for 1 hour at 2300° F.



(a) Bar used for study of effect of amount and temperature of reduction.



(b) Bar used for study of repeated critical deformations.

Figure 4.- Specimens to obtain varying reduction by rolling the indicated tapered bars to straight bars.

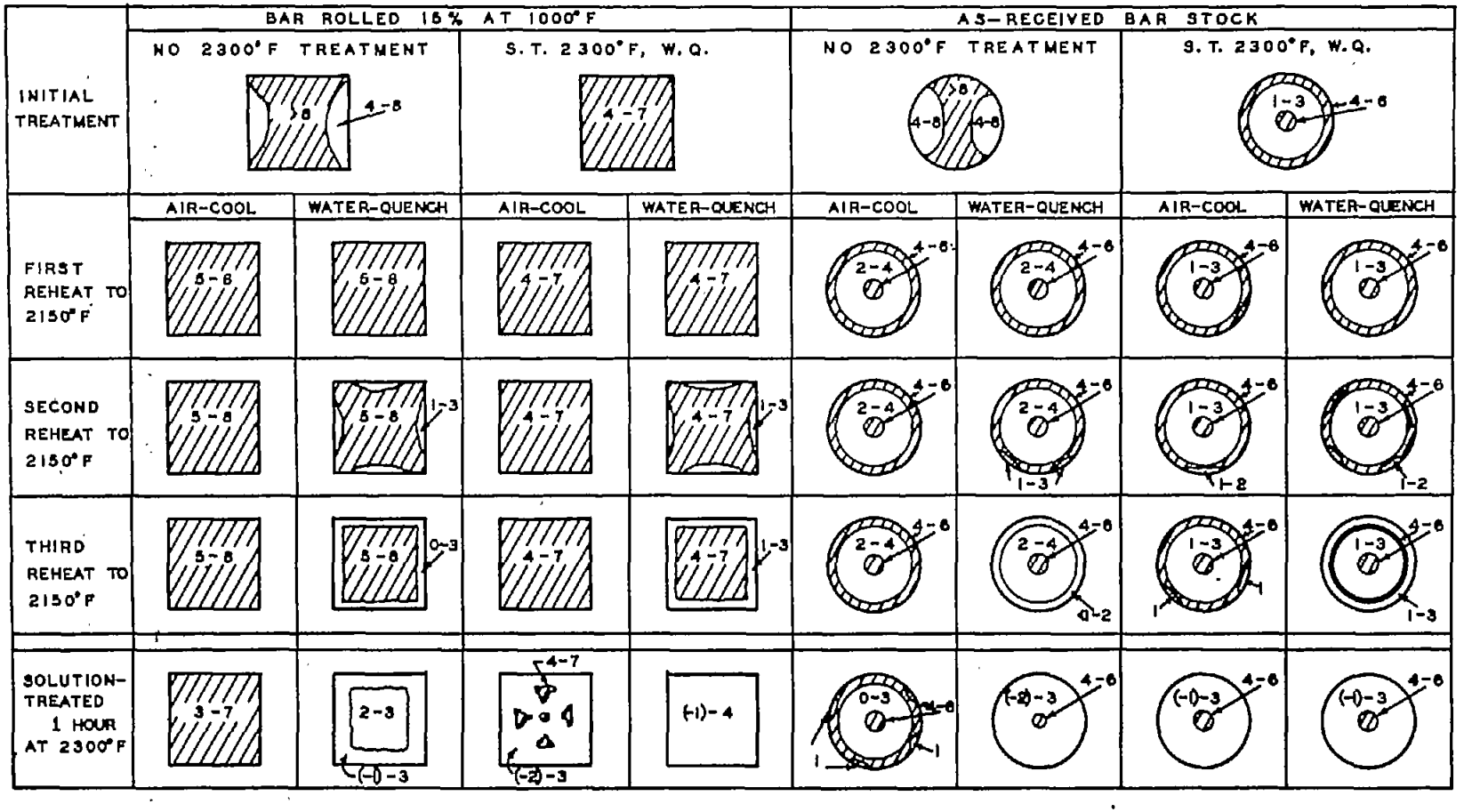
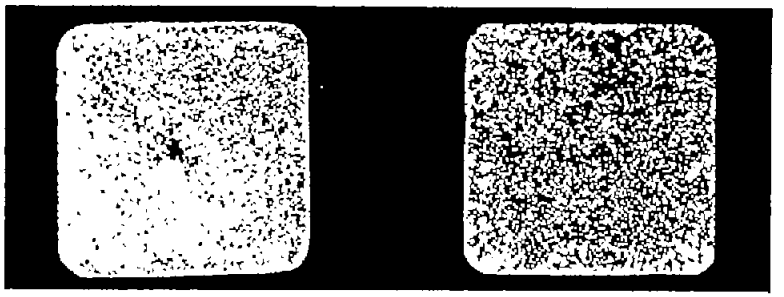
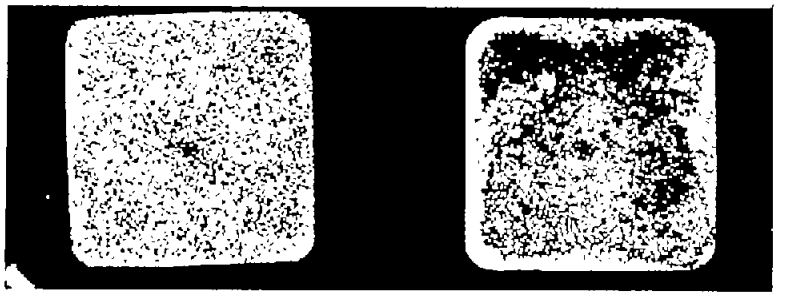


Figure 5.- Effect of temperature cycling on grain growth in S-816 bar stock. (S.T., solution-treated; W.Q., water-quenched.)



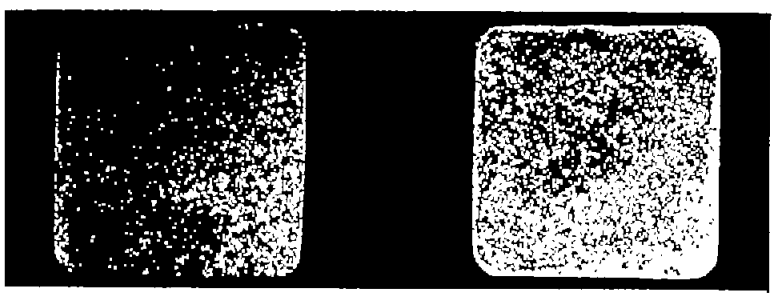
Air-cooled Water-quenched

(a) Reduced 15 percent at 1000° F and reheated to 2150° F three times and cooled as indicated.



Air-cooled Water-quenched

(b) Reduced 15 percent at 1000° F, reheated to 2150° F three times and cooled as indicated, and finally solution-treated 1 hour at 2300° F.



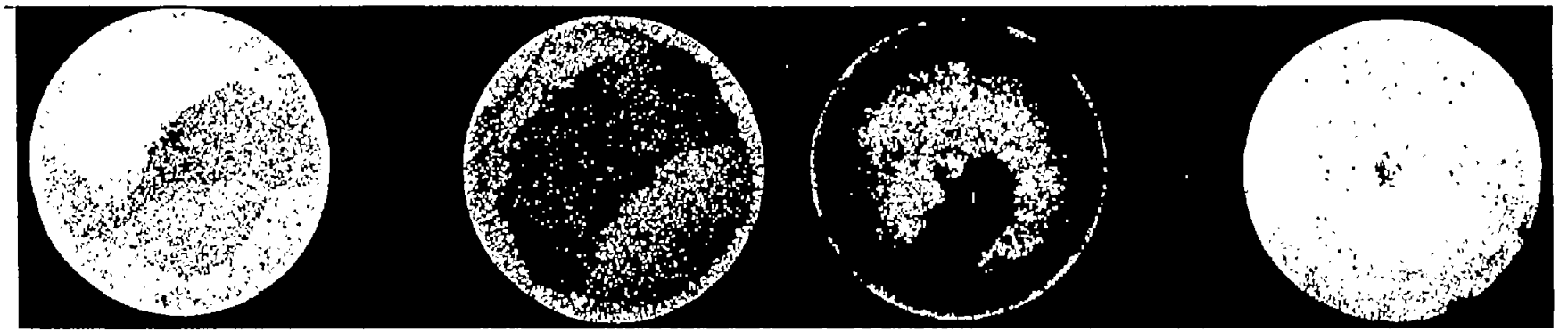
Air-cooled Water-quenched

(c) Reduced 15 percent at 1000° F, solution-treated 1 hour at 2300° F and water-quenched, and then reheated to 2150° F three times and cooled as indicated.

Air-cooled Water-quenched

(d) Reduced 15 percent at 1000° F, solution-treated 1 hour at 2300° F and water-quenched, reheated to 2150° F three times and cooled as indicated, and finally re-solution-treated at 2300° F.

Figure 6.- Macrographs showing grain-size distribution in S-816 alloy bar stock after cyclic heating to 2150° F. X2.4.

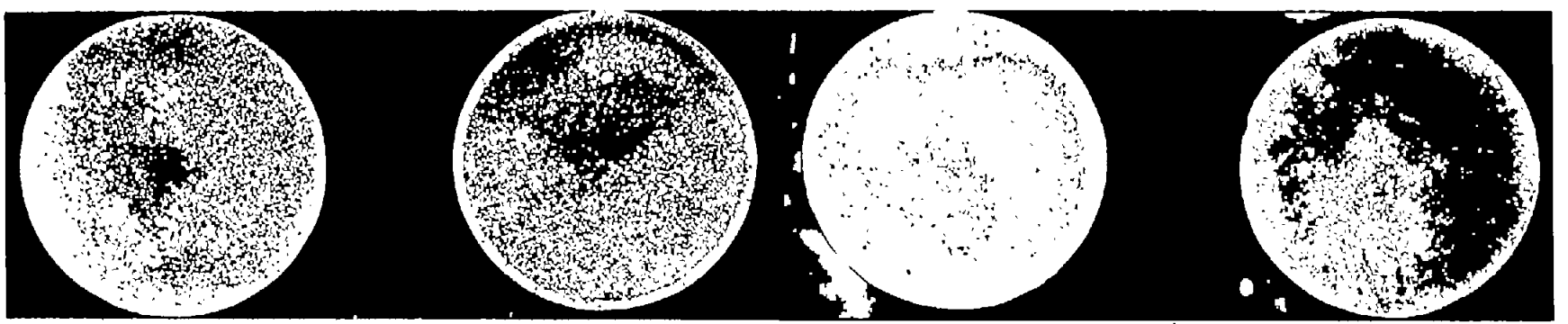


Air-cooled Water-quenched

(e) As-received bar stock heated three times to 2150° F and cooled as indicated.

Air-cooled Water-quenched

(f) As-received bar stock heated three times to 2150° F and cooled as indicated and finally solution-treated for 1 hour at 2300° F.



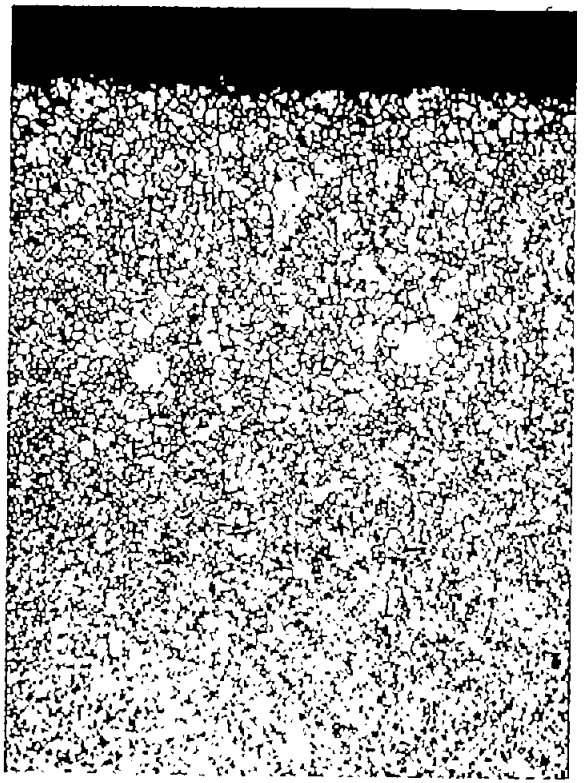
Air-cooled Water-quenched

(g) As-received bar stock solution-treated for 1 hour at 2300° F and water-quenched; reheated to 2150° F three times and cooled as indicated.

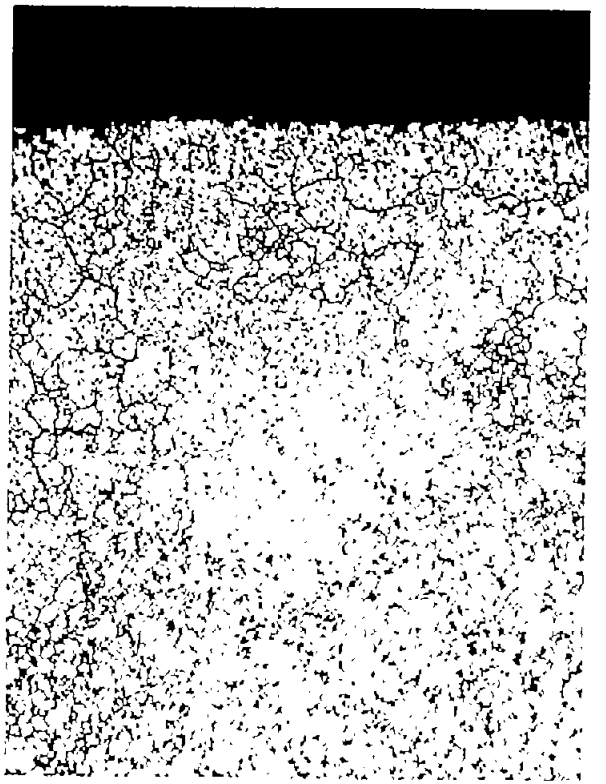
Air-cooled Water-quenched

(h) As-received bar stock solution-treated for 1 hour at 2300° F and water-quenched, reheated to 2150° F three times and cooled as indicated, and finally re-solution-treated at 2300° F.

Figure 6.- Concluded.

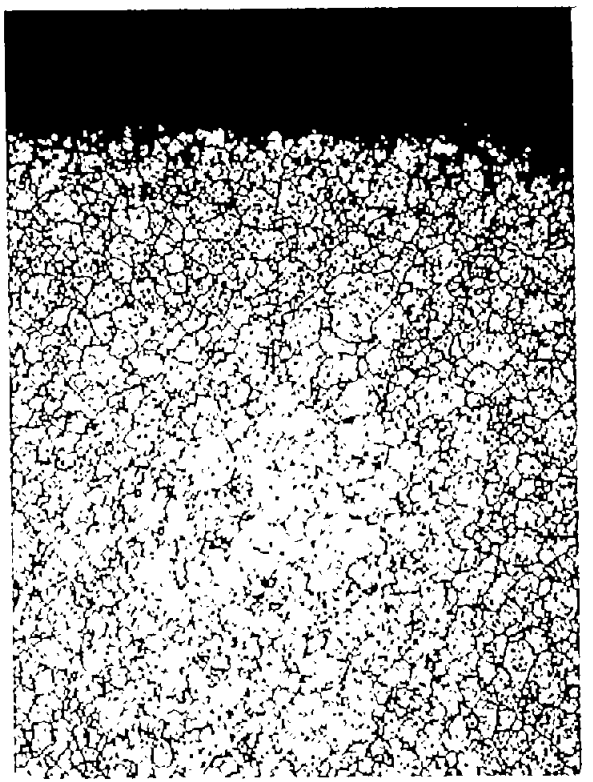


(a) Heated to 2150° F and air-cooled three times.

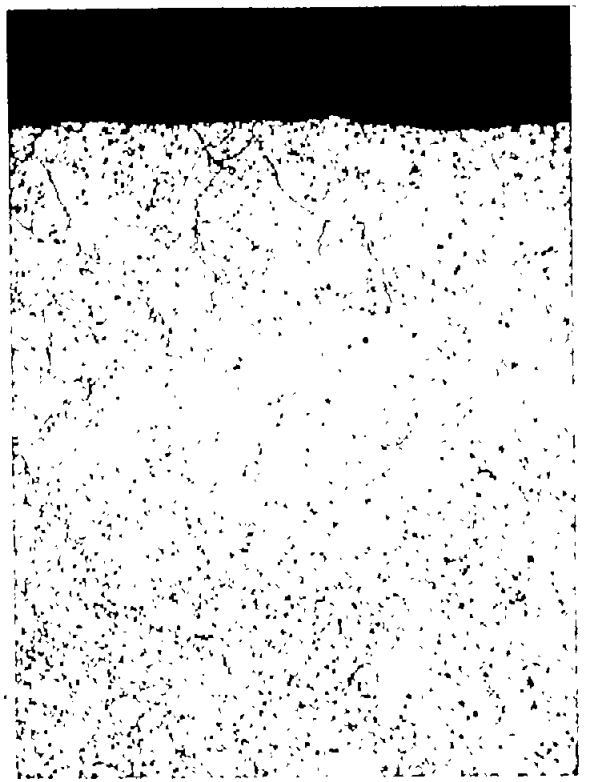


(b) Heated to 2150° F and water-quenched three times.

Figure 7.- Effect of temperature cycling to 2150° F on grain size of S-816 bar stock reduced 15 percent at 1000° F. X50.

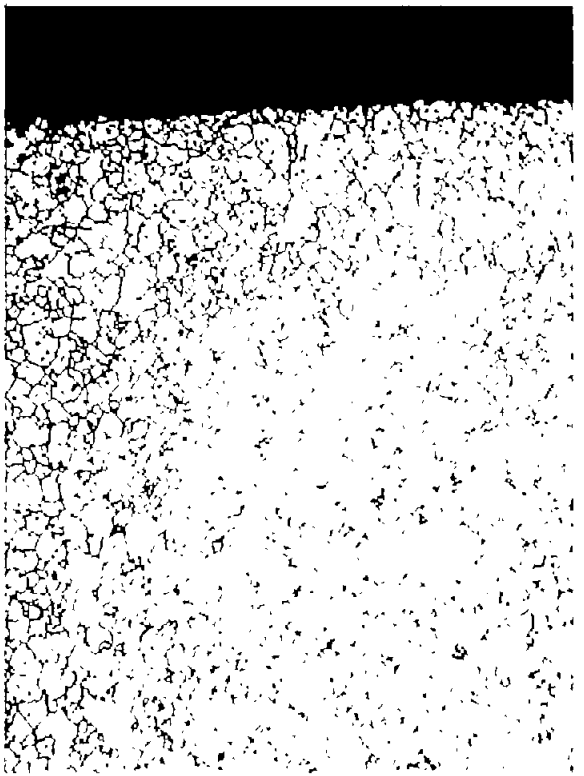


(c) Heated to 2150° F and air-cooled three times and finally solution-treated at 2300° F.

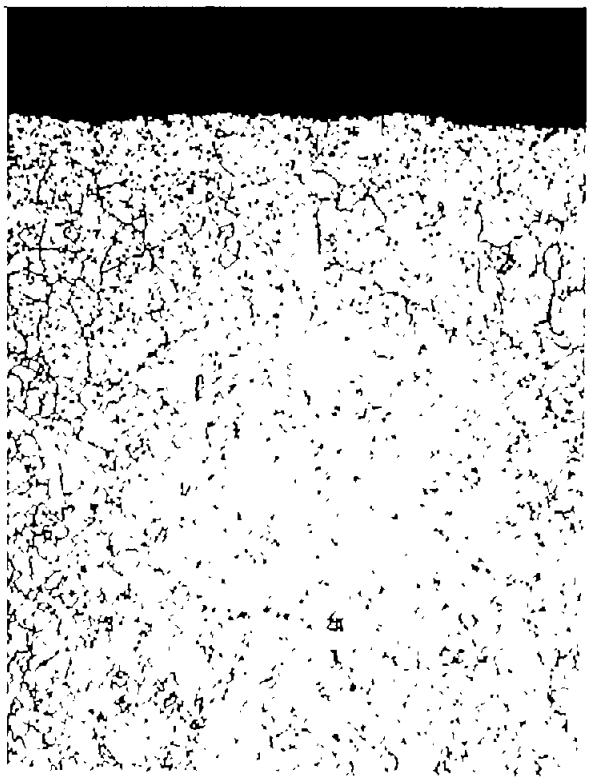


(d) Heated to 2150° F and water-quenched three times and finally solution-treated at 2300° F.

Figure 7.- Concluded.

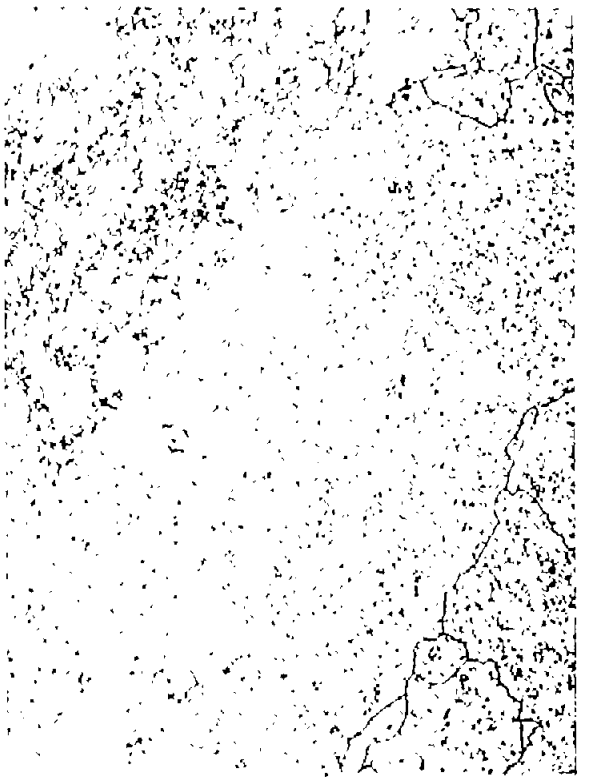


(a) Heated to 2150° F and air-cooled three times.

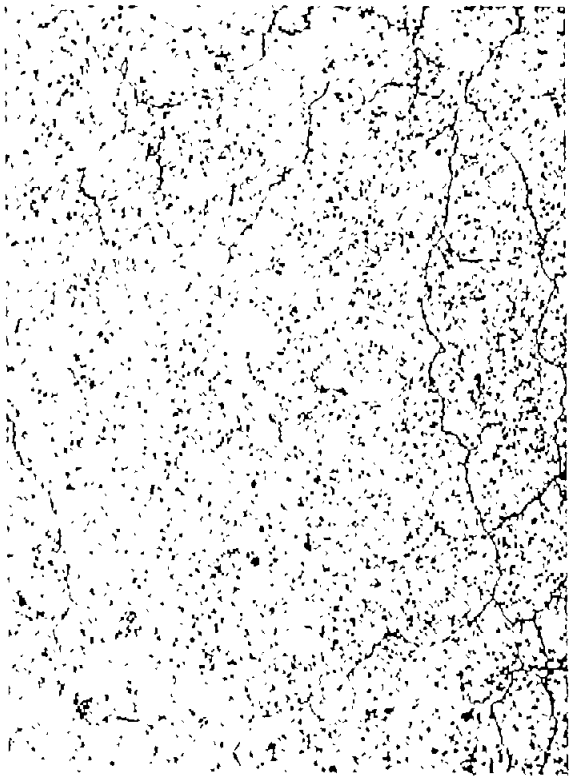


(b) Heated to 2150° F and water-quenched three times.

Figure 8.- Effect of temperature cycling to 2150° F on the grain size of S-816 bar stock reduced 15 percent at 1000° F followed by solution treatment at 2300° F for 1 hour. X50.

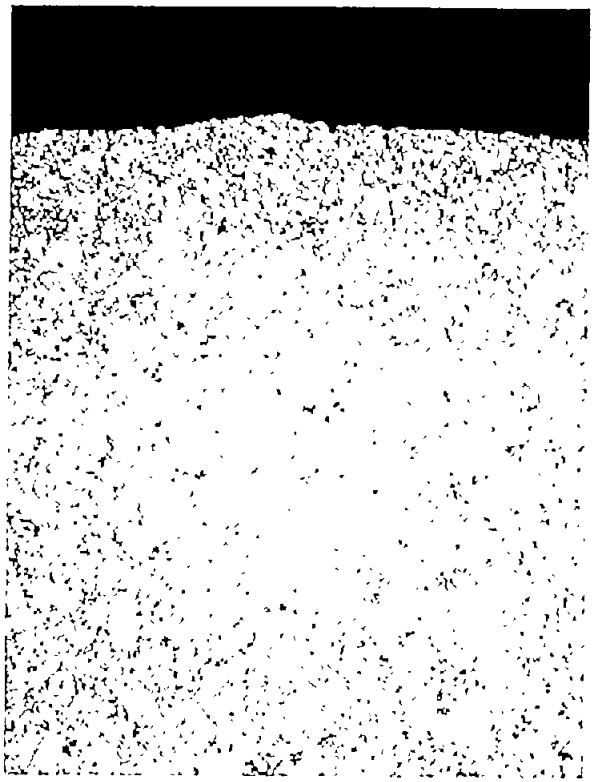


(c) Heated to 2150° F and air-cooled three times and finally solution-treated at 2300° F.

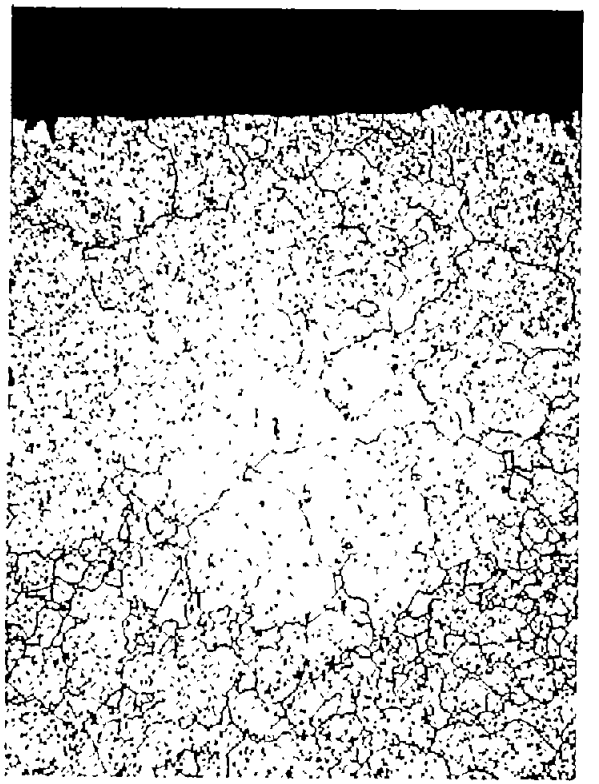


(d) Heated to 2150° F and water-quenched three times and finally solution-treated at 2300° F.

Figure 8.- Concluded.

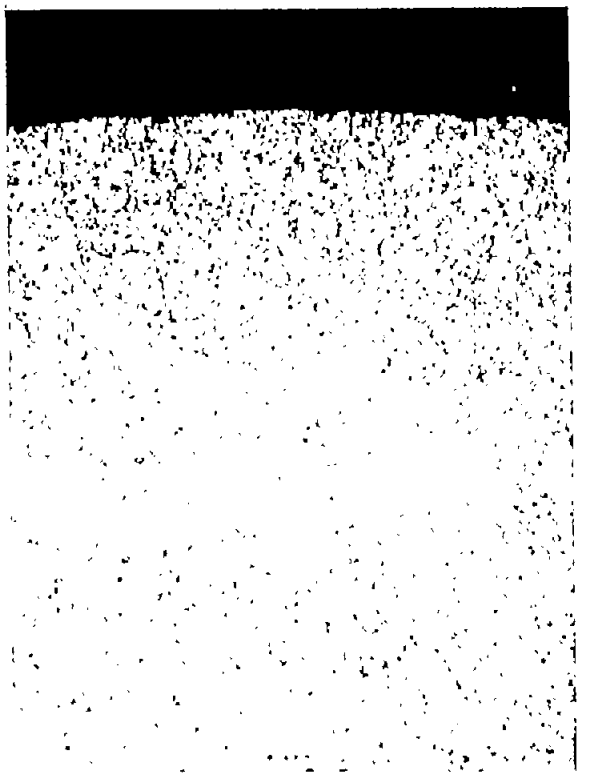


(a) Heated to 2150° F and air-cooled three times.

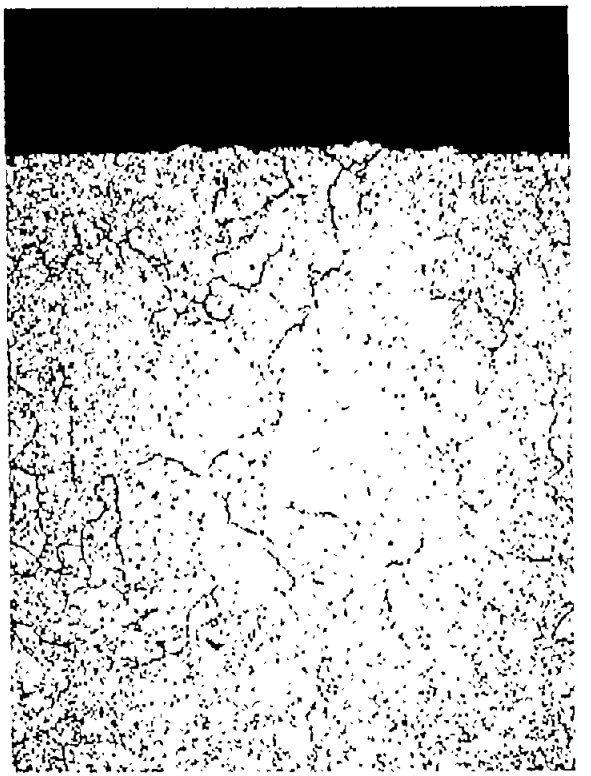


(b) Heated to 2150° F and water-quenched three times.

Figure 9.- Effect of temperature cycling to 2150° F on the grain size of the as-received S-816 bar stock. X50.

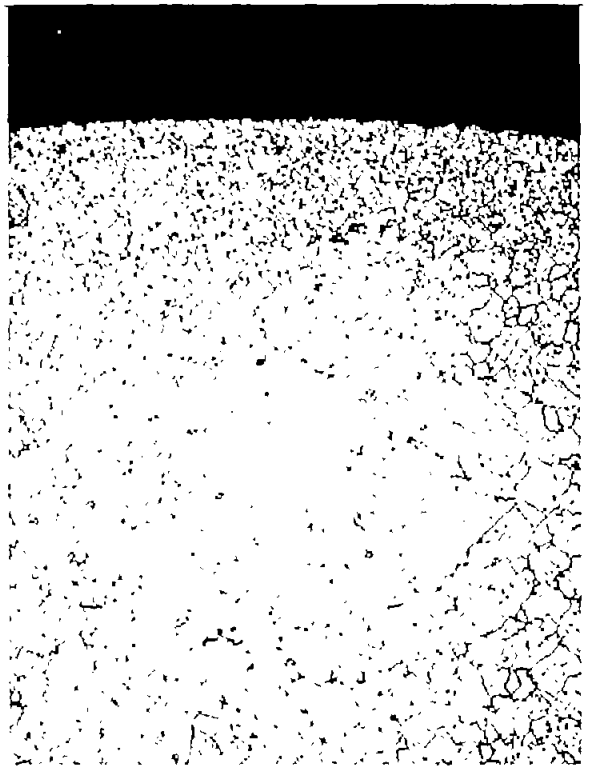


(c) Heated to 2150° F and air-cooled three times and finally solution-treated at 2300° F.

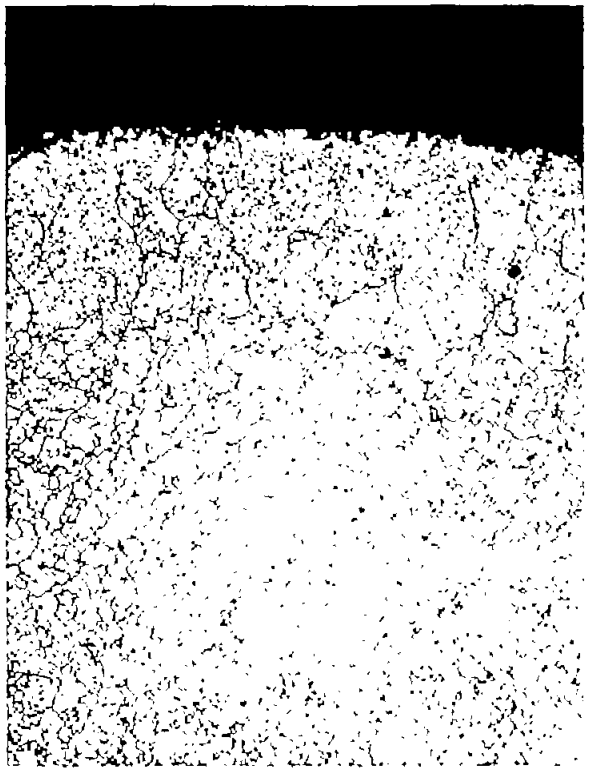


(d) Heated to 2150° F and water-quenched three times and finally solution-treated at 2300° F.

Figure 9.- Concluded.

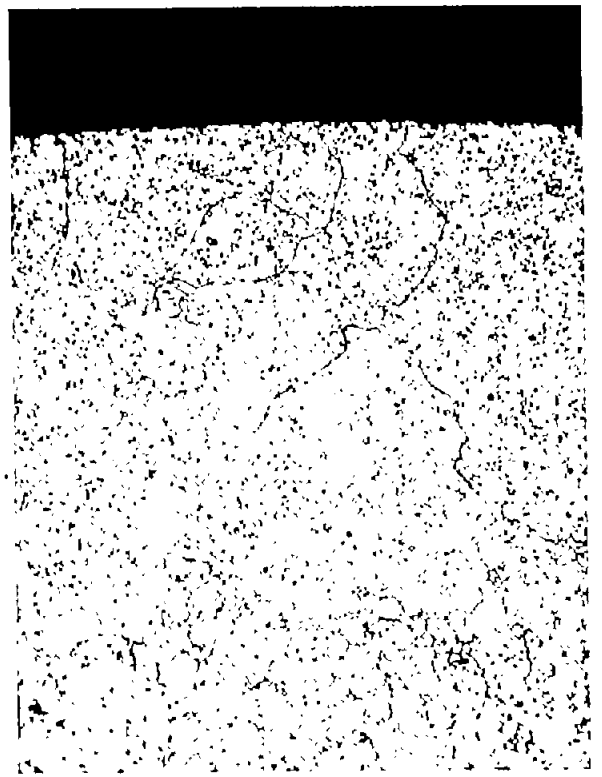


(a) Heated to 2150° F and air-cooled three times.

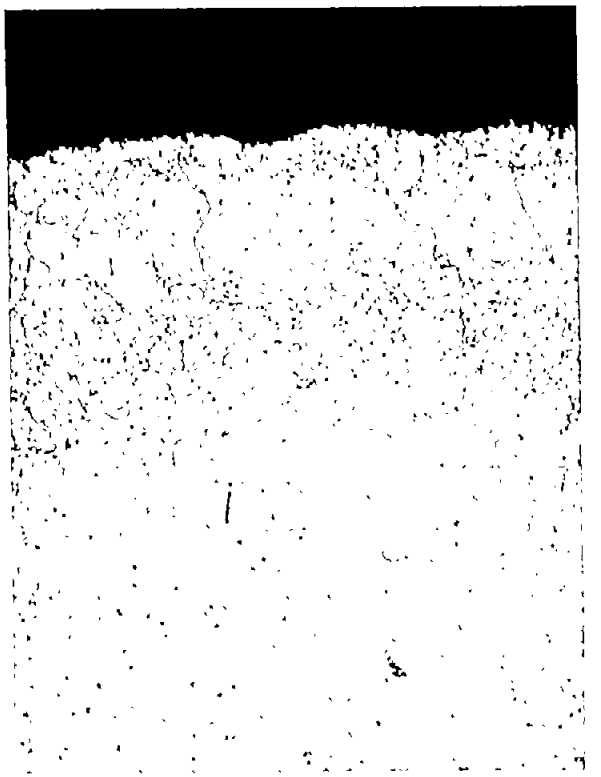


(b) Heated to 2150° F and water-quenched three times.

Figure 10.- Effect of temperature cycling to 2150° F on the grain size of as-received S-816 bar stock solution-treated 1 hour at 2300° F and water-quenched. X50.



(c) Heated to 2150° F and air-cooled three times and finally solution-treated at 2300° F.



(d) Heated to 2150° F and water-quenched three times and finally solution-treated at 2300° F.

Figure 10.- Concluded.

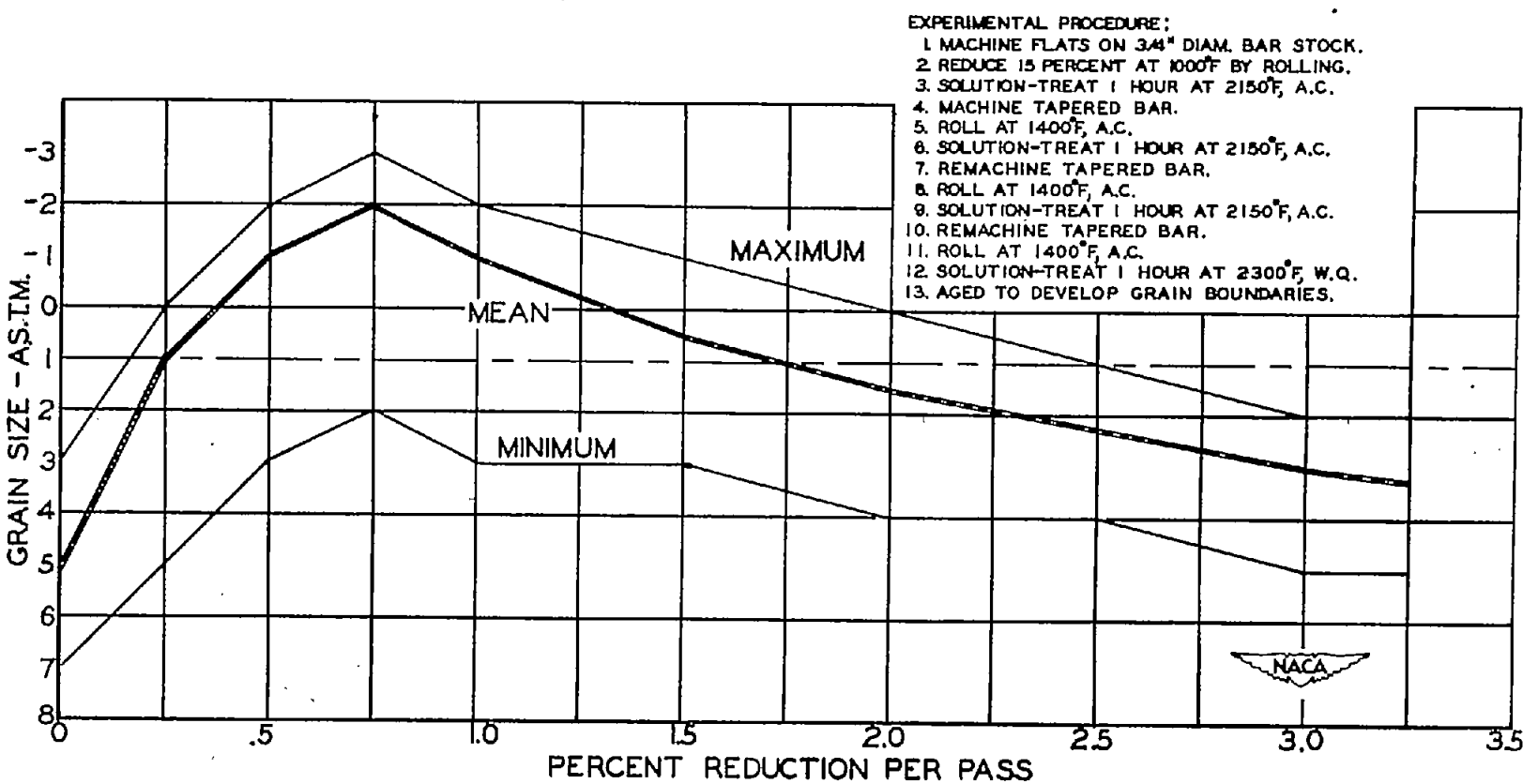
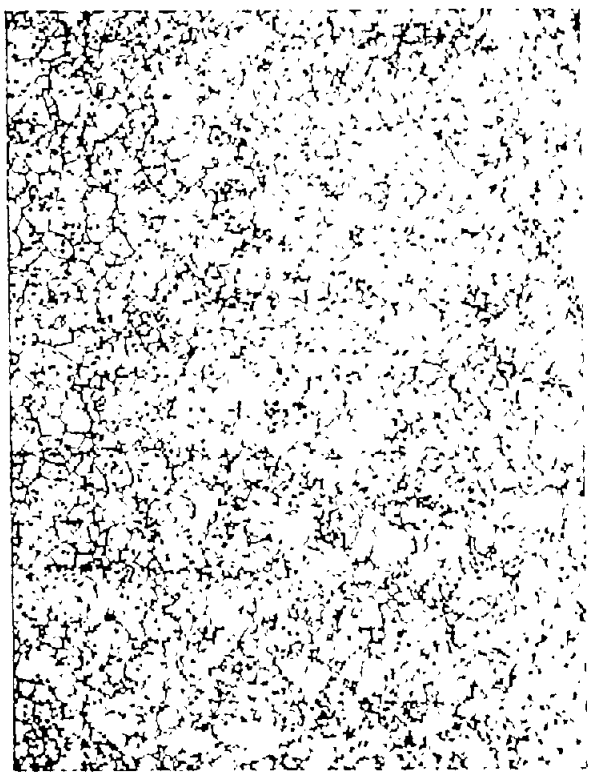
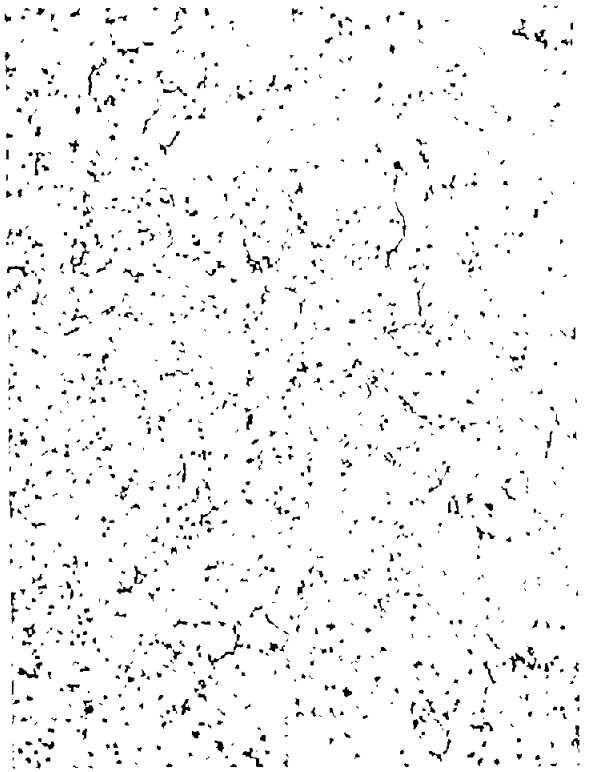


Figure 11.- Effect of repeated prior critical deformation on grain size of S-816 bar stock after final solution treatment at 2300° F. (A.C., air-cool; W.Q., water-quench.)



(a) No reduction. Grain size: 3 to 7.



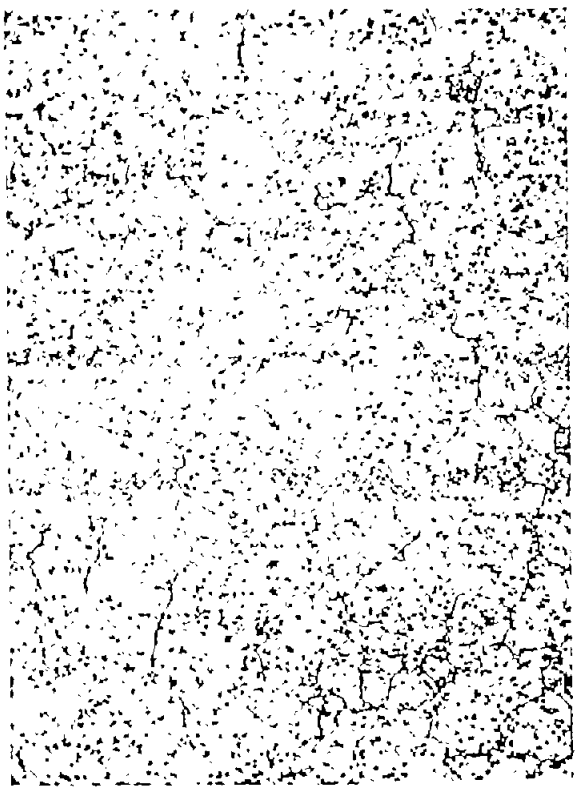
(b) 0.25-percent reduction. Grain size: 0 to 5.

Figure 12.- Effect of repeated critical reductions on grain size of S-816 bar stock. Stock was reduced 15 percent at 1000° F, solution-treated 1 hour at 2150° F, and air-cooled. A tapered bar was rolled to produce reductions from 0 to 3 percent, reheated to 2150° F and the process repeated twice more, and then finally solution-treated at 2300° F. X50.

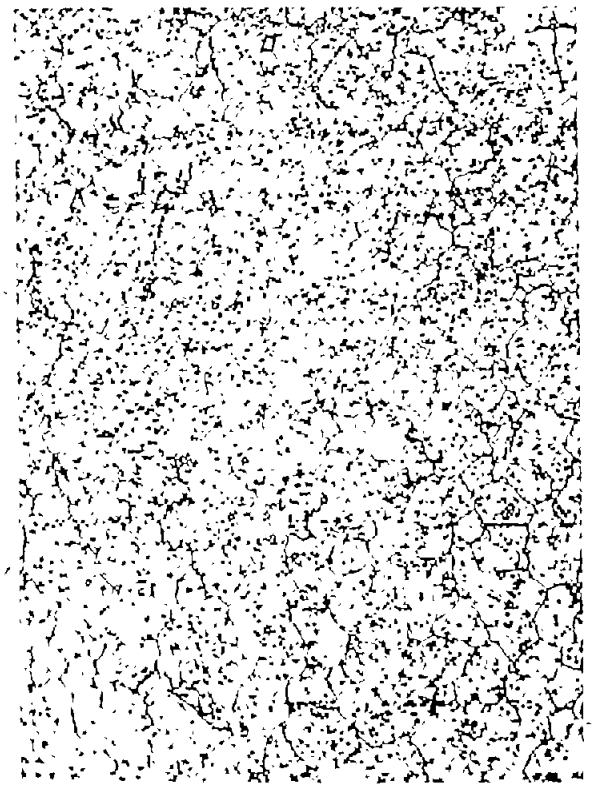
(c) 0.75-percent reduction. Grain size: (-3) to 2.

(d) 1.5-percent reduction. Grain size: (-1) to 3.

Figure 12.- Continued.



(e) 2.0-percent reduction. Grain size: 0 to 4.



(f) 3.0-percent reduction. Grain size: 2 to 5.

Figure 12.- Concluded.

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