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TECHNICAL NOTE 4084

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ABNORMAL GRAIN GROWTH IN M-252 AND S-816 ALLOYS

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SUMMARY

A laboratory study was carried out to establish the basic causes of abnormal grain growth in air- and vacuum-melted M-252 and S-816 alloys. The results were in general agreement with a previous study of Waspaloy, Inconel X-550, and Nimonic 80A alloys. Results of tests on the five alloys indicated that small reductions of essentially strain-free metal were the basic cause of abnormal grain growth. In most cases, there was a narrow range of reductions responsible for abnormal growth between reductions of 0.4 and 5.0 percent. In a few special cases the responsible reductions were as low as 0.1 percent and as high as 9.7 percent.

The prevention of abnormal grain growth clearly requires avoidance of small critical reductions. The main problem is to anticipate and to avoid conditions leading to critical deformation. Insuring that all parts of a metal piece receive more than 5- to 10-percent reduction will prevent it. Nonuniform metal flow during hot-working operations is probably the major source of abnormal grain growth. Any small reduction, particularly if it includes a strain gradient so that the critical reduction will definitely be present, is a common source. Strains arising from thermal stresses during rapid cooling can cause susceptibility to abnormal grain growth. Removal of strain by recrystallization during working followed by a small further reduction can, in certain cases, induce abnormal grain growth in the presence of large reductions.

The phenomenon of abnormal grain growth is remarkably independent of temperature of working and of heating temperatures. If the heating temperature and time are sufficient for abnormal grain growth, higher temperatures increase the grain size only slightly. Prior history of the alloys before critical straining has a relatively minor effect, provided the prior treatment reduces strain below the critical amount. Certain conditions of working or heating seemed to minimize abnormal grain growth. These, however, do not appear dependable for controlling abnormal grain growth because of the probability that their effectiveness is dependent on prior history of the alloy.

The influence of alloy composition seems to be mainly in variation of flow characteristics during working and variation in excess phases

which restrict grain growth. Waspaloy and Nimonic 80A alloys readily underwent grain growth at 1,950° F. Because of higher carbide content, the M-252 alloy had marginal growth at 1,950° F. While S-816 and Inconel X-550 alloys did not undergo abnormal growth at 1,950° F, at 2,150° F, the normal solution temperature for these two alloys, abnormal growth did occur. However, in S-816 alloy, 2,150° F was marginal and temperatures of 2,200° to 2,300° F were required for rapid growth. Apparently the more stable columbium compounds in S-816 and Inconel X-550 alloys restrained grain growth to a higher temperature than the less stable growth restrainers in the other alloys.

INTRODUCTION

An experimental investigation was carried out to study causes of abnormal grain growth in heat-resistant alloys of the type used for the blades in the rotors of aircraft gas turbines. The present report covers the results obtained for M-252 and S-816 alloys. A preliminary report (ref. 1) has previously been issued for S-816 alloy. A similar report (ref. 2) presents the results of the studies of the phenomenon for Waspaloy, Inconel X-550, and Nimonic 80A alloys. The present report includes results of tests on four normal air-melted arc-furnace heats of M-252 and S-816 alloys and two vacuum-melted heats of M-252 alloy. One of the vacuum-melted heats was very low in manganese and silicon.

The primary purpose of the investigation was to determine the cause of abnormal grain growth in typical heat-resistant alloys and in aircraft gas turbines. The research was undertaken because abnormally large grains sometimes develop in forged blades during fabrication and heat treatment with a consequent deleterious effect on properties of the blades. The conditions and causes for the phenomenon as well as the principles for avoiding the difficulty have not been understood.

For purposes of the investigation, abnormal grain growth was defined as the development of grains larger than ASTM 1. It had been well established that the abnormal grain growth of interest occurred at normal working and heat-treating temperatures. Therefore, for the most part, normal temperatures of solution treatment were used to allow grain growth after susceptibility to abnormal grain growth was developed by various experimental conditions.

The investigations previously reported (refs. 1 and 2) disclosed no source of abnormal grain growth other than small critical deformations. The problem appeared to be mainly the identification and avoidance of the often complex conditions under which such small deformations could occur. The two alloys covered by the present report differ from the alloys covered by reference 2 mainly in that their structure contains large numbers of dispersed carbides. These serve to act as grain refiners and generally cause a finer grained structure for a given heat treatment. The lower carbon alloys covered by reference 2 have far fewer dispersed phases in their structures and their grains become coarse at lower temperatures. The Inconel X-550 alloy included in reference 2 was an exception in that it had a higher coarsening temperature even though its carbon content was low.

The investigation was carried out by the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics. The members of the NACA Subcommittee on Power Plant Materials assisted in the planning of the experimental program, particularly by defining conditions of working where grain-growth problems had been troublesome.

PROCEDURES

The general procedure involved the following steps:

(1) Commercially produced bar stock was procured for use as experimental materials. In the case of M-252 alloy, stock from three different heats melted in air and two different heats melted in vacuum was used. The air-melted materials were said to vary in grain-growth sensitivity and the vacuum-melted material was said to be relatively immune. The inclusion of several heats also served as a check on the generality of the findings.

(2) The as-received stock could not be relied upon to be free of uneven or abnormal grain-growth tendency. Therefore, in most cases, stock was initially "equalized" through a heavy reduction by rolling and a heat treatment for 1 hour at the normal solution temperature. The heat treatment was necessary to produce an essentially strain-free material which would not obscure the experimental results through the influence of prior strain. In a few limited cases, the equalizing treatment consisted of only a heat treatment.

(3) Repeated heating and cooling were used to study the induction of abnormal grain growth by thermal stresses alone. Air-cooling, oil-quenching, and water-quenching were used.

(4) The influence of the amount and temperature of deformation was studied by rolling tapered specimens to flat bars between open rolls in a rolling mill. The tapered specimens were machined from equalized stock. Two types of specimens (fig. 1) were used to vary the range of reduction. One bar (fig. 1(a)) gave about 0- to 15-percent reduction when rolled

flat in one pass. A second type (fig. 1(b)) was repeatedly reduced, giving 0- to 5-percent reduction per pass. The specimens were placed in a furnace at the desired working temperature, held 1/2 hour, rolled, and air-cooled. In the case of S-816 alloy, the influence of cooling rate from the rolling mill was also studied to obtain an indication of the importance of this factor in view of the ease of introducing susceptibility to abnormal grain growth by rapid cooling.

The rolled specimens were reheated to the usual solution-treating temperatures for the usual times during which grain growth occurred. The specimens were carefully measured for reduction of area during rolling. The bars were then split lengthwise, polished, and examined metallographically and the grain size was measured as a function of percent reduction of area.

(5) Additional study of the effect of amount and temperature of deformation was carried out using tensile specimens to obtain uniform small reductions to induce abnormal grain growth.

(6) The grain-size rating system used was that established by the American Society for Testing Materials (ref. 3). It was necessary to extend this system to sizes larger than 0 by using the notation -1 to -5 grain sizes. The actual grain sizes involved were as follows:

ASTM grain-size number	Grains per sq in. of image at 100 diameters	Approximate diameter of grains, in.
8	128	0.0009
7	64	.0012
6	32	.0018
5	16	.0025
4	8	.0035
3	4	.005
2	2	.007
1	1	.010
0	.5	.014
-1	.25	.020
-2	.125	.028
-3	.0625	.040
-4	.0312	.056
-5	.0156	.080

In reporting grain sizes, the range is given in the tables of data. The graphical presentations are generally limited to the maximum size.

EXPERIMENTAL MATERIALS

The experiments were carried out on commercially produced bar stock. The information furnished by the suppliers of the alloys is given in the following sections.

M-252 Alloy

Air-melted M-252 stock was supplied gratis by the General Electric Co. from three heats. Heats 43482 and 63674 had been made by the Allegheny Ludlum Steel Corp. in arc furnaces. The bars were 1 inch and 7/8 inch square, respectively. Heat A6891 had been made by the Universal-Cyclops Steel Corp. in an arc furnace and was in the form of 7/8-inch-square bars.

The chemical analyses of these heats were reported to be:

Heat	Chemical analysis, percent by weight											
	C	Mn	Si	Cr	Ni	Co	Mo	Ti	Al	Fe	S	P
43482	0.17	1.30	0.62	19.0	Balance	9.97	10.2	2.08	0.62	3.14	-----	-----
63674	.12	1.28	.59	19.0	Balance	11.3	9.44	2.88	1.14	1.63	0.016	0.014
A6891	.18	1.26	----	19.0	Balance	10.3	9.85	2.67	.95	.84	-----	-----

Stock from two vacuum-melted and cast heats was supplied gratis by the General Electric Co. The heats had been made by their Carboloy Division. The bars were 7/8 inch square. Complete chemical analyses were not supplied. The information given indicated the following differences between the two heats:

(a) Heat A-41: Silicon and manganese were omitted from the alloy

(b) Heat B-29: Normal M-252 composition

S-816 Alloy

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

The composition was reported to be as follows:

Chemical composition, percent by weight											
C	Mn	Si	Cr	Ni	Co	Mo	Fe	W	Cb	S	P
0.38	1.28	0.23	19.5	19.9	Balance	3.82	3.65	4.15	3.84	0.019	0.011

FACTORS INFLUENCING GRAIN GROWTH

A number of factors influenced observed grain-growth characteristics in the experimental materials. Because these factors were fairly complicated, consideration of the following discussion of some of these factors will help in understanding the results of the studies:

(1) The experimental materials in the as-received condition had been hot-worked to bar stock under unknown conditions. In some cases the grain sizes were initially mixed. The grain-growth characteristics when reheated to normal hot-working or solution-treating temperature indicated susceptibility to abnormal or uneven grain growth in most cases. Usually this tendency varied along the bar-stock lengths.

(2) These varied and uncertain prior-history effects were minimized in most experiments by an equalizing treatment. This was a fairly heavy reduction by rolling combined with a heat treatment for 1 hour at the normal solution temperature. This gave a uniform grain structure in material with uniform response to subsequent experimental variables. The cooling rate from the heat treatment had to be restricted to that of air- or oil-quenching in order to avoid susceptibility to abnormal grain growth on the surface during subsequent reheating.

It should be recognized that there are certain important considerations involved in these equalizing treatments:

(a) The best way to avoid uneven or abnormal grain growth during any subsequent heating is to introduce more than a minimum amount of uniform work into the stock. As discussed later, this should be a reduction larger than at least 5 percent. Material given such reductions would, however, be unsuitable for the experimental program because the initial reduction would mask the experimental variable to be studied.

(b) The equalizing treatments do not make the material independent of prior history. The actual grain size is influenced by the prior working and heating conditions. It can be postulated that if the prior working results in a material which undergoes recrystallization and grain growth to uniform reasonably fine grain size it is then in a condition suitable for study of abnormal grain growth. The recrystallization reduces prior strain-hardening to a minimum. As far as is known, some other sequence of treatments could have resulted in a different initial grain structure. This, however, would alter the results of the experiments only in detail.

(c) First, the heat-treatment step probably did not attain the equilibrium grain size for the temperature of heating. Second, the degree of solution of excess phases was probably variable. Third, the cooling from the heat treatment introduced a small strain in the surface of the metal. However, the affected zone was probably completely removed when the tapered specimen was machined.

(3) The equalized material when reheated for working might or might not have undergone further alteration of grain structure as a result of the additional heating before working actually started.

(4) When the tapered specimens were rolled, a range of conditions was set up in the specimens:

(a) A zone of no reduction where any change should have been only that induced by reheating.

(b) A zone of increasing amounts of strain resulting from the increasing reduction.

(c) If the temperature of reduction was too low for any recrystallization for the range of reductions, the whole length of the specimen was strain-hardened. This was dependent on the amount and temperature of reduction and the opportunity for recovery during cooling.

(d) If the temperature of working was sufficiently high for recrystallization during working, there was a zone of increasing strain-hardening followed by a zone at the larger reductions where strain-hardening had been reduced by the recrystallization. In general, the zone of cold-worked material decreased with increasing temperature of reduction. The zone of recrystallization was reduced in strain-hardening in proportion to the degree of completeness of recrystallization. In general, this increased with both temperature and amount of reduction.

(e) The air-cooling from working introduced some surface strain from the thermal stresses.

(5) When the tapered specimens were reheated for solution treatment, the reaction was characterized by zones as follows:

(a) A zone of no or very small reduction where the grain growth was mainly dependent on the further growth to be expected from unstrained material. Presumably the machining of the tapered specimen removed any surface metal strained during cooling from the equalizing treatment. Consequently, only the air-cool from the working temperature was involved.

(b) A zone, covering reductions generally in the order of 0.5 to 4.4 percent, which was critically strained, resulting in a few grains growing to abnormal sizes.

(c) A zone of higher reductions where deformation resulted in more grains growing in competition to prevent abnormal final grain size.

(d) At still larger reductions recrystallization definitely occurred in the more severely strain-hardened metal during reheating unless it occurred during working. In the latter case grain growth occurred. Many of the specimens showed partial recrystallization at the heavier reductions. Presumably recrystallization occurred during reheating in those locations where it did not occur during rolling. The zones of recrystallization presumably underwent grain growth.

RESULTS

Grain-growth characteristics of M-252 and S-816 alloys were studied. Repeated heating and cooling, deformation by rolling, and tensile straining were used to induce grain growth.

In the experiments involving rolling, tapered specimens were rolled to flats. In the regions of small reductions causing abnormal grain growth, as discussed in subsequent sections, the grain growth was remarkably uniform across the entire section of the specimens. The line of demarkation at the smallest reduction causing such growth was very sharp. The recrystallization and grain growth was also uniform on a macroscopic scale across the bar section. Recrystallization during working or after solution treatment, however, was often banded.

M-252 Alloy

Induction of abnormal grain growth in M-252 alloy by repeated heating and cooling and by rolling was studied. A final solution temperature of 1,950° F was used to promote grain growth.

The M-252 experimental materials all had uniform fine grain sizes after the equalizing treatments (figs. 2 and 3). Two of the air-melted materials had a grain size of 6 to 8 and the third had a grain size of 8 or finer. The grain size of the vacuum-melted heat with low silicon and manganese content was 7 to 8 and that of the normal-composition vacuum-melted heat was 5 to 8.

Induction of abnormal grain growth by repeated heating and cooling.- The experiments conducted on the induction of abnormal grain growth by repeated heating and cooling and the resulting grain sizes are summarized in figures 4, 5, and 6. The major points observed were:

(1) Air-cooling from 1,950° F did not induce abnormal grain growth in either air- or vacuum-melted stock.

(2) Water-quenching induced grain growth on the surface of all heats equalized by rolling at 1,950° F. Equalizing by rolling at 2,150° F suppressed growth in the vacuum-melted heat A-41 with low silicon and manganese content.

The maximum grain size developed was larger than 1 for only air-melted heats 63674 and A6891 and vacuum-melted heat B-29.

In reviewing these data it should be recognized that it was shown in reference 2 that the time at the solution-treating temperature and not the number of reheats was the controlling factor in grain growth. Therefore the data in figure 6 for the vacuum-melted heats should be comparable with the data for the air-melted heats where repeated heating and cooling was used.

The absence of grain growth during 5 hours of heating without a water quench (fig. 6) shows the necessity for the more drastic thermal stressing of water-quenching for grain growth.

Photomicrographs (fig. 5) show typical grain sizes after various amounts of reheating subsequent to a water quench.

These results indicate the following general observations:

(1) The solution-treating temperature of 1,950° F is marginal for grain growth in M-252 alloy. The grain sizes were not so large nor the

growth so extensive as those observed for the lower carbon alloys of reference 2.

(2) The vacuum-melted heats were just as susceptible to grain growth as the air-melted heats.

(3) Either hot-working or exposure to 2,150° F reduced the susceptibility to grain growth of the vacuum-melted material.

(4) Lower carbon content was the only apparent difference between heat 63674, which was slightly more susceptible to grain growth, and the slightly more resistant heats 43482 and A6891. The low silicon and manganese content in vacuum-melted heat A-41 may have been related to its being somewhat less susceptible to growth after working at 2,150° F. In the absence of actual analyses, variation in carbon content could be postulated, however.

Induction of abnormal grain growth by rolling.- A sharp increase in grain size occurred at some critical reduction between 0.5 and 3.4 percent in all the experiments carried out on the induction of abnormal grain growth by rolling. (See table I and figs. 7 to 9.) The grain size diminished as the amount of reduction was increased further. The grain size was 1 or less for reductions greater than 0 to 4.4 percent, depending on the working conditions. The range in reductions was obtained by rolling tapered specimens and was limited to a maximum reduction of approximately 15 percent.

Minor variations in the critical reduction and the maximum grain size resulted from varying the rolling temperature and the equalizing treatments and by heating to a high temperature before rolling. There was also only slight variation between air- and vacuum-melted materials. The effects of these variables are as follows:

Effect of temperature of reduction: There was a slight tendency for the critical reduction to increase with increasing temperature of rolling. This was not consistent or pronounced.

The maximum grain size did not vary much with temperature of reduction. In most cases rolling at 1,600° F produced the largest grains.

Effect of variation in equalizing treatment: Again there was very little effect from the variables studied. The reduction for critical deformation was generally increased slightly by prior rolling at 2,100° F over that obtained when the material was equalized by rolling at 1,950° F. There was, however, little difference in maximum grain size.

The critical reduction appeared to increase slightly for rolling at any temperature except 2,100° F when the stock was first treated at 2,100° F. Either rolling at 2,100° F followed by a treatment at 1,950° F or heating to 2,100° F and then cooling to a lower temperature seemed to have this effect. This supports the indication of the data in reference 2 that the apparent increase in critical reduction at higher temperatures was the result of heating to those temperatures and not the temperature of working.

Influence of vacuum-melting: There was little difference in the critical reduction between stock melted in air or in vacuum (fig. 9). The critical reduction perhaps tended to be a little larger for the material made in vacuum. There was no consistent difference in maximum grain size (fig. 9). The low-silicon and manganese heat A-41 tended to have a slightly smaller maximum grain size than the normal-composition vacuum-made heat B-29.

S-816 Alloy

The induction of abnormal grain growth in S-816 alloy by repeated heating and cooling, by rolling, and by tensile straining was studied. A heating temperature of 2,150° F was extensively used because it is a common temperature for heating, for working, and for final solution-treating. A solution treatment of 2,300° F was also extensively used to intensify grain growth.

The original bar stock was subject to uneven grain growth in the as-received condition (fig. 10). Most of the experiments were carried out on material equalized by a reduction of 15 percent at 1,000° F to remove this uneven-grain-growth tendency. When solution-treated at 2,150° or 2,300° F, this treatment resulted in uniform grain sizes of 5 to 8 and 4 to 7, respectively (fig. 11). The reduction at 1,000° F was used in early experiments simply because it was the first one tried and it gave the necessary grain uniformity. Other equalizing treatments were subsequently used, as indicated in the results of the individual experiments.

Induction of abnormal grain growth by repeated heating and cooling.- The induction of abnormal grain growth by quenching was discovered when unexpected abnormal grain growth occurred during early experiments on water-quenched S-816 alloy. Consequently, the subject was quite extensively studied to clear up the factors involved (see fig. 12).

The grain sizes which developed in stock equalized by the 15-percent reduction at 1,000° F and in as-received stock after a number of conditions of heating and cooling are shown by figure 12(a). Macrographs of the specimens of figure 12(a) are shown by figure 13. The significance of these figures can be summarized as follows:

(1) When air-cooled from repeated heating to 2,150° F, abnormal grain growth did not occur during subsequent treatments to 2,150° and 2,300° F in stock equalized by a reduction of 15 percent at 1,000° F. When water-quenched, however, it did occur on the surface.

(2) The as-received material did not undergo abnormal grain growth from repeated heating to 2,150° F and air-cooling or water-quenching but did when finally heated to 2,300° F. The degree of abnormal grain growth at 2,300° F was much greater in the water-quenched samples. Unlike that in the equalized stock, abnormal grain growth tended to occur first at points intermediate between the surface and center.

(3) When treated at 2,300° F and water-quenched after an equalizing reduction of 15 percent at 1,000° F, abnormal grain growth did not occur during reheats to 2,150° F with air-cooling. When finally reheated to 2,300° F, it did occur extensively. Water-quenching from reheats to 2,150° F did start growth on the surface.

(4) The as-received stock when solution-treated at 2,300° F did undergo grain growth on the surface during reheats to 2,150° F with air-cooling. This growth was more extensive when the stock was water-quenched from 2,150° F. When they were finally heat-treated at 2,300° F, extensive abnormal grain growth occurred in materials both air-cooled and water-quenched after reheating to 2,150° F.

The general conclusion from these experiments seems to be that a water quench somewhere in the history is required to obtain abnormal-type grain growth at 2,150° F. It is presumed that the tendency for abnormal grain growth in the as-received stock even after air-cooling is related to the prior history which caused uneven grain growth.

Material equalized by a reduction of 15 percent at 1,400° F appeared to be considerably more susceptible to abnormal grain growth (fig. 12(b)). This material developed grains as large as -2 when reheated to 2,150° F after a water quench from 2,150° F and as large as -4 after 4 hours at 2,150° F. Material equalized by a reduction of 70 percent at 2,150° F was less susceptible to abnormal grain growth (fig. 12(b)) although it developed grains as large as -2 in 3 hours at 2,150° F after an initial water quench from 2,150° F. Typical photomicrographs of the material equalized by rolling at 1,400° F are shown in figure 14.

Material oil-quenched or air-cooled from 2,150° F after a 70-percent reduction at 2,150° F was not so susceptible to abnormal grain growth as when water-quenched (fig. 12(b)). Three hours at 2,150° F did, however, result in grains as large as 1 on the surface.

Apparently total time at temperature after a water quench is the controlling factor in abnormal grain growth and repeated heating and cooling has very little additional effect. (See the data in figure 12(b) for material equalized by rolling at 1,400° F.) This confirms the same finding for the data in reference 2.

All the results of repeated heating and cooling may be summarized as follows:

(1) Water-quenching from either 2,150° or 2,300° F introduced susceptibility to abnormal grain growth. The susceptibility was prevented or greatly reduced when air-cooling or oil-quenching was used.

(2) The susceptibility to abnormal grain growth varied considerably with the conditions of the initial equalizing treatment.

(3) Total time of reheating is the controlling factor in abnormal grain growth while repeated heating and cooling have little additional effect.

Induction of abnormal grain growth by rolling.- A range of reductions was obtained by rolling tapered specimens. A number of conditions of rolling were used. The grain sizes after subsequent final solution treatment were then measured as a function of degree of reduction.

As in all alloys studied, abnormal grain growth started abruptly at some critical reduction. This was between 0.5 and 2.8 percent for S-816 alloy. The maximum grain size always developed at the smallest reduction inducing abnormal grain growth. The grain size fell off rapidly with further reduction so that the maximum grain size was 1 or smaller for reductions of 3.3 percent or less. The maximum grain size decreased rapidly with further deformation so that it was quite small for deformations of 6 to 8 percent.

The experimental conditions included a number of variables as described in the following sections.

Influence of rolling temperature: Rolling temperature between 1,000° and 2,250° F had very little effect on the critical deformation for abnormal grain growth (fig. 15(a)). The total range of critical reductions for the experimental conditions used was from 0.7 to 1.5 percent with the highest temperatures of reduction tending to require the most deformation and to have the smallest range of critical deformation.

Influence of solution-treating temperature: The maximum grain size at the critical reduction was either -1 or -2 when the solution-treating temperature was 2,300° F (fig. 15(a)). When the solution-treating temperature was 2,150° F the maximum grain size was 3 (fig. 15(b)). In considering these grain sizes it is important to recognize that the samples were heated only 1 hour during the solution treatments. As was indicated in the studies of quenching, grains larger than 1 would have developed if longer heating times at 2,150° F had been used, and possibly grains larger than -2 would have developed for longer times at 2,300° F.

The temperature required for abnormal grain growth to occur in 1 hour after critical deformation appears to be between 2,000 and 2,100° F (figs. 16 and 17). A temperature higher than 2,150° F was required to produce grains larger than 1. Microstructures of the sample solution-treated at 2,200° F have been included in figure 18 as typical of those of the tapered specimens examined.

The reliability of the exact values of critical reduction for grain growth and the grain sizes developed is uncertain. The general trends, however, appear to be valid. The reason for questioning the reliability of the exact values is the apparent variation with prior history.

Influence of prior history: When the equalizing treatment included a water quench from 2,300° F, the maximum grain size at the critical reduction for rolling at 1,400° F was 3 for a final solution treatment at 2,150° F (fig. 15(b)). When the equalizing heat treatment was 2,150° F, this maximum grain size was 1 (fig. 16). When the final solution temperature was 2,300° F, the respective grain sizes were -1 and 0 (figs. 15(a) and 16). It will also be recalled that there was an indication of prior-history sensitivity in the samples quenched to induce abnormal grain growth.

The apparent influences of prior history suggest that there may be considerable variation in the minimum temperature for abnormal grain growth in 1 hour (fig. 17) as well as in the maximum grain sizes.

Influence of working at or near the final-solution-treatment temperature: In a number of experiments, indications were found suggesting that abnormal grain growth could be suppressed by working at or just above the final-solution-treatment temperature. This has not been found to be reliable in either this report or reference 2. Figure 19 shows that rolling at 2,250° F did not suppress grain growth at the critical reduction very much.

Development of very large grains.- One of the disturbing features of the experiments was the inability of the S-816 alloy to develop the very large grains which had been experienced in forging of blades. Some special experiments were therefore undertaken to attempt to produce such grains.

The first method used was to heat and cool the specimens repeatedly after rolling as tapered specimens. Grains as large as -2 were produced by four cycles to 2,150° F (fig. 20) as compared with a maximum grain size of 1 for one cycle for material with the same equalizing treatment (fig. 16). As has previously been discussed this was probably due to the increase in time at temperature rather than to the repeated heating and cooling. Superimposing a final treatment at 2,300° F increased the maximum grain size to -4. This is considerably coarser than that obtained from just 1 hour at 2,300° F in any test.

The second experiment involved repetitive rolling in the critical deformation range with intermediate reheats to 2,150° F (fig. 21). A specimen with only a small amount of taper (fig. 1(b)) was used. Figure 21 differs from the other figures in that it shows the range in grain size. The maximum grain size developed was -3 as compared with a maximum of -1 when rolled only once (fig. 15(a)) with a final treatment at 2,300° F. It is uncertain, however, whether the increase was due to the repetitive critical deformation or to the increased exposure to 2,150° F during the heatings between rolling cycles. Material with the same equalizing treatment given one pass through the rolls and reheated to 2,150° F for 1 hour developed a maximum grain size of only 3 (fig. 15(b)). The experiments involving heating and cooling, however, indicated that longer time at 2,150° F resulted in larger grains.

It seems evident from both experiments that repetitive exposure to conditions inducing abnormal grain growth leads to larger grains. The data cited do not, however, conclusively indicate whether increased time at temperature for grain growth or repeated critical deformation is the controlling factor.

<u>Influence of cooling rate after rolling</u>.- The discovery that abnormal grain growth was induced by rapid cooling raised a question as to the effect of cooling rate after rolling. To obtain information on this point a series of samples were prepared using the following steps:

(1) The stock was equalized by a reduction of 15 percent at 1,000° F.

(2) Bars were reheated to 2,150° F and given reductions of 0, 1, 4, and 7 percent. Two bars were used for each reduction, one being air-cooled and the other water-quenched from the rolling mill. Two samples were cut from each bar, one being solution-treated at 2,150° F and the other, at 2,300° F.

(3) The remainder of the bars were again reheated and rolled samples cut off and solution-treated as in step (2).

(4) The remainder of the bars were again reheated, rolled, and samples cut off and solution-treated as in step (2).

Examination of the samples for grain size revealed the following results:

(1) No abnormal grain growth occurred in the samples reduced 4 or 7 percent during each pass. The reduction was more than the critical amount. There was likewise no effect on the grain size from cooling rate from the rolling mill.

(2) Samples given no reduction did not undergo abnormal grain growth when air-cooled but did when water-quenched, as expected.

(3) Samples given a reduction of 1 percent per pass did undergo abnormal grain growth whether air-cooled or water-quenched. The slightly larger grain size for water-quenching may or may not be significant. Apparently the reduction of 1 percent combined with the thermal-stressing effect carried the deformation slightly past the critical amount on two faces with a consequent slight reduction in grain size on those faces in comparison with that of samples simply quenched. It is evident in figure 22 that the reduction of 1 percent influenced grain size completely through the bar stock.

(4) The general conclusion seems to be that cooling rate from the rolling mill has little effect unless the rolling reduction is at or below the critical amount for abnormal grain growth. Apparently the deformations from thermal stresses on cooling are additive to those from rolling and would therefore have some effect when working deformations are very small. For instance, it is possible that if a part was receiving critical reduction during working, a water quench after working might increase the deformation past the critical amount.

Induction of abnormal grain growth by tensile straining.- Tensile specimens deformed small amounts at 1,400° and 1,600° F and solution-treated at 2,300° F developed grain sizes shown by figure 23. The small deformations definitely induced growth similar to that induced by critical reductions by rolling. The smaller grain size in the undeformed threaded ends shows that temperature alone was not responsible.

Comparison can be made of the tensile-straining data at 1,400° F with rolling data at 1,400° F with the same equalizing and final treatments (fig. 16). While the critical reduction by rolling was 1.2 percent, resulting in a maximum grain size of 0, tensile straining 1 percent resulted in size 1 grains after final solution treatment.

The uniform tensile deformation did not cause quite so large grains to form as did the critical reduction by rolling of tapered specimens. In two of the specimens size 0 grains formed in the fillets where the exact critical deformation must have occurred. These grains were smaller than those obtained in analogous tapered specimens. Possibly the slower cooling of the tensile specimens was involved. However, similar data for Waspaloy (ref. 2) showed closer agreement between tensile and rolling deformation. The general conclusion of reference 2 that the amount of deformation is the controlling factor in abnormal grain growth and not the method of deformation or strain gradient seems valid.

Deformation required to refine large grains.- As-received stock was heated to 2,300° F for 2 hours and air-cooled. This produced grains ranging in size from -2 to 5. This material was rolled at 2,150° F to give reductions of 9 to 56 percent. The recrystallization during rolling and the grain-size ranges after subsequent solution treatments of 2,150° or 2,300° F are summarized by figure 24.

While a reduction of 9 percent gave very little recrystallization during rolling, the grain-size range after subsequent solution treatment at 2,150° F was 4 to 7 and after treatment at 2,300° F was 3 to 6. Thus, as little reduction as 9 percent at 2,150° F broke up the initial large -2 grains. A reduction between 40 and 56 percent was required to refine the grain structure completely by recrystallization during rolling. It will be noted that the grain size after solution treatment decreased with increasing amounts of recrystallization during rolling. Very little was gained, however, by reductions of more than 20 percent; the largest effect was between 13 and 21 percent.

DISCUSSION

The investigation provides considerable information regarding the conditions which can cause abnormal grain growth in heat-resistant alloys of the type studied. Many, if not most, of the conditions of working to be avoided for freedom from abnormal grain growth can be specified. The basic mechanisms involved in many of the interrelated variables can also be postulated from the theory of grain growth.

Prevention of Abnormal Grain Growth

The only means found for inducing abnormal grain growth in any of the alloys investigated including Waspaloy, Inconel X-550, and Nimonic 80A alloys (ref. 2) was by small deformations of strain-free material. The phenomenon seemed to be independent of the temperature of deformation. The most important conclusion therefore is that abnormal grain growth can occur only during reheating to grain-growth temperatures after small critical deformations, usually between 0.5 and 5.0 percent, of initially strain-free material. Apparently, if all metal in any part is deformed more than 5 to 10 percent, abnormal grain growth will be prevented during a subsequent reheat.

The major problem in preventing abnormal grain growth in practice is to identify and anticipate the possible sources of critical deformation. It is obvious that small deformations and especially deformations causing strain gradients, both common procedures in straightening methods, should be avoided. Critical deformations can occur in the presence of large overall deformations due to uneven metal flow in dies. Die design and operation must insure that all parts of the metal piece move more than the critical amount.

In working operations involving reheating, the reheats can remove the effects of large prior reductions. It is therefore important to recognize that critical deformation must be avoided in every working operation. For instance, trimming a forging without a reheat will probably simply superimpose deformation on material already deformed more than the critical amount. If the forging is reheated for trimming with the reheat removing the strain from prior deformation, susceptibility to abnormal grain growth is sure to develop because trimming introduces a strain gradient certain to include the critical deformation.

If nearly complete recrystallization occurs during working, the resulting strain-free condition of the metal leaves it susceptible to critical deformation by a small amount of further deformation. Thus multiple-blow forging can lead to abnormal grain growth even with large total deformations if initial deformations cause recrystallization and subsequent blows deform some parts only the critical amount. This probably frequently occurs when the final operation involves small deformations to obtain desired size. Avoidance of abnormal grain growth in multiple-blow forging requires that complete recrystallization be avoided in any one blow or that more than critical deformation be used in all parts with every blow.

The critical deformation must be applied to essentially strain-free materials to induce subsequent abnormal grain growth. If small deformations superimposed on unrelieved prior deformation result in more than critical deformation, abnormal growth will not occur. This apparently is the reason why sensitivity to abnormal grain growth does not often occur as a result of cooling from the working operations. Conditions involving a rapid cool after simple heating without mechanical deformation followed by reheating do not often occur. It is important to recognize, however, that thermal stressing is a function of the degree of restraint as well as of the cooling rate. There may be shapes and sizes in which slower cooling rates than water-quenching could induce critical strain. Likewise, in other shapes, water-quenching might not do it.

The amount of reduction required to break up abnormal grains once they are formed was studied slightly for S-816. It appeared that 5- to 10-percent reduction by rolling would be sufficient prior to subsequent solution treatment. It is suspected, however, that in practice metal flow characteristics may retard deformation of large grains and prevent these grains from receiving sufficient deformation to cause recrystallization. While it was not studied, it may be that large grains retained from solidification of ingots may be more difficult to break up than those formed by heat treatments.

The results clearly indicate the principles necessary to avoid abnormal grain growth. They are

(1) Rapid cooling from an essentially strain-free condition must be avoided. This strain-free condition might be present before quenching in parts which had received no deformation or in parts which had been heavily reduced, resulting in extensive simultaneous recrystallization.

(2) Any reductions should be more than the critical amount. Thus a reheat followed by a small finishing reduction should be avoided if the reheat conditions leave the metal essentially strain free.

(3) When multiple operations are used between reheats, care must be exercised to be sure that extensive simultaneous recrystallization is not followed by a final small critical reduction before reheat.

(4) Working at or above the normal solution temperature cannot be depended on to reduce abnormal grain growth.

Metallurgical and Compositional Effects

The use of abnormally high temperatures had relatively little effect on abnormal grain growth (ref. 2). It appears that increased temperatures only slightly increase the size of the grains, unless the usual temperatures and heating times are marginal for grain growth. In S-816 alloy 2,150° F was marginal. Therefore increasing the temperature to 2,200° F considerably increased grain growth. Increasing the temperature from 2,200° to 2,300° F, however, had little effect (fig. 17), apparently because 2,200° F for 1 hour was sufficient for nearly complete grain growth. In reference 2, alloys which underwent nearly complete grain growth under normal heat-treating conditions showed little further effect from higher temperatures.

In practice temperatures and heating times are marginal for complete recrystallization or grain growth. Consequently the grain-growth characteristics can be sensitive to prior history. Therefore variations in grain-growth characteristics between heats may result in wide differences in abnormal-grain-growth sensitivity for a fixed working and treatment schedule. For instance, it was found that S-816 alloy equalized by a reduction of 15 percent at 1,000° F plus a 1-hour treatment at 2,300° F would not develop grains larger than 3 during a 1-hour treatment after critical deformation. Abnormally large grains did form during the same final treatment after other equalizing treatments. It would seem that the practical differences between heats involve variations which change the grain-growth rates under the marginal conditions for grain growth usually used. So far as the experiments carried out were concerned, this showed up as a variation in time to attain a given grain size after critical deformation.

There are differences between alloys in the temperatures and times required for abnormal grain growth after critical deformation. These temperatures were not well established for the alloys studied. Two of the alloys studied in reference 2, Nimonic 80A and Waspaloy, were very sensitive to abnormal grain growth when heated to 1,950° F. Inconel X-550 alloy required a higher temperature with the grain growth occurring readily at 2,150° F. M-252 alloy required more than 1 hour at 1,950° F for abnormal grains to grow. The same was true for S-816 alloy at 2,150° F for 1 hour. Larger abnormal grains grow in S-816 alloy in less time at 2,300° F.

It is probable that variation in grain-growth restrainers was a major reason for the difference in grain-growth characteristics between alloys. S-816 alloy contains large numbers of refractory carbide particles because of its high carbon and columbium content. These apparently raise the temperatures and increase the time required to obtain a given grain growth. The higher carbon content of M-252 alloy apparently increased the resistance to grain growth in comparison with that of Nimonic 80A or Waspaloy. Inconel X-550 apparently required a higher temperature and longer time periods for abnormal grain growth because of the grain-growth-restraining characteristics of refractory columbium carbides and nitrides. In the studies made it was noted that higher carbon heats of the various alloys were slightly more resistant to grain growth. High-temperature treatments giving more solution of grain-growth restrainers seemed to increase the severity of abnormal grain growth somewhat.

In practice it is probable that differences in flow characteristics between alloys are very important variables. For a given working operation one alloy might be far more susceptible than another to nonuniform flow, thereby providing opportunity for critical deformation. Recrystallization temperature and time differences between alloys might also be very important. An alloy resistant to recrystallization may be much easier to keep strained above the critical amount than one which readily recrystallizes.

Temperatures of heating for working and heating times are often found important in controlling grain structure in practice. It appears from this investigation that these factors probably have their major effect through the way the metal moves. Certain temperatures probably are conducive to more uniform metal flow, in particular hot-working operations. Heating times, other than their possible influence on the temperature attained in the metal, probably influence strain recovery between operations. As is evident in the data, recrystallization and grain growth at usual hot-working temperatures are time dependent. Control of the heating time may prevent recovery effects from dropping the strain below the critical amount. This would prevent the metal from becoming susceptible to abnormal grain growth in parts which receive a small deformation in subsequent working.

Heating temperatures for working may be important if the temperature is sufficiently high and the time long enough for abnormal grain growth. This would allow grain growth during hot-working which would have to be broken up by subsequent working to be eliminated from the structure. It might allow repeated critical deformation and grain growth under the right conditions. This may be the source of the extremely large grains sometimes encountered.

There was no great difference found among air-melted heats or between air- and vacuum-melted heats of a given alloy. Yet in practice these melting conditions are often found to be important variables. The investigation did not disclose the reasons for this. The most probable reason is differences in flow characteristics which for a given procedure result in variation in the way the metal moves during working. This, in turn, sets up critical deformation conditions in some heats and not in others.

The method of deformation does not seem to be important to abnormal grain growth. Approximately equal effects were obtained by rolling, tensile straining, and thermal stressing by quenching. Strain gradients are not required for abnormal grain growth. The greater chance for critical deformation in the presence of a strain gradient explains the frequent association of abnormal grain growth with strain gradients.

The overall data did not indicate any particular effect of initial grain size on abnormal grain growth. The degree of growth possibly decreased as very large grains were formed and were subsequently again critically deformed. Apparently treatment at various temperatures had some effect on the rate of abnormal grain growth. This probably was due to variation in solution or precipitation of particles acting as grain-growth restrainers.

Mechanism of Abnormal Grain Growth

There are two basic mechanisms resulting in grain growth: (1) Absorption of surrounding grains by grain-boundary migration, and (2) formation of new grains by recrystallization followed by grain-boundary migration. Both mechanisms require a difference in energy between grains such that those at a higher energy level are absorbed by those at a lower energy level. In the first case, some factor sets up a condition such that some grains are at a higher energy level than others. It is the common mechanism for growth of larger grains from smaller grains. In the second case, relief of strain due to deformation causes a small new grain to form. This grain then grows at the expense of the surrounding metal which is at a higher energy level by virtue of the strain present. If there are many centers at which the small new grains form in relation to the original grain size, there will be more grains after recrystallization is complete and grain refinement will have occurred. If there are few centers strained enough to recrystallize, growth of only a few grains will occur, resulting in grain coarsening.

The literature (refs. 4 and 5) does not clearly define whether abnormal grain growth occurs by grain-boundary migration of existing grains or by growth of a very few small grains formed by recrystallization. In either case the essential feature would seem to be nonuniformity of strain within the individual original grains. Grain-boundary migration would require that a few grains receive very little strain in relation to their neighboring grains. Recrystallization followed by grain growth would require sufficiently large deformations at a very few centers initiating new grains.

Regardless of this initial mechanism, it can be postulated that the characteristic shape of the curves of grain size versus percent reduction by rolling results from the following sequence of conditions:

(1) In regions of no reduction or smaller reductions than the critical amount, there is not a sufficient contrast in energy levels to make only a few grains grow at the expense of surrounding grains. Grain growth that occurs is the normal uniform growth.

(2) At the critical reduction, the straining leaves only a very few low-energy grains. The energy difference is great enough to allow them to grow during subsequent heating, leaving a few large abnormal grains.

(3) At somewhat larger amounts of strain than the critical, apparently there are more grains in a condition to absorb their neighbors than at the critical strain. The increase in the number results in competition for available surrounding grains. The grain size is then restricted because there are not enough grains available for any one to become large.

(4) At still larger amounts of strain, normal recrystallization and grain growth most certainly take place. The effects at larger amounts of strain are, however, complicated if simultaneous recrystallization occurs during working. It appeared from the data that there was little difference in the grain size in either case except when a very small amount of recrystallization occurred. Mixed grain sizes resulted during reheating in this case, apparently by the few initial small grains growing at a

faster rate than those which formed by recrystallization. In the experiments conducted, this mechanism did not develop abnormally large grains although it was theoretically possible. The mechanism, however, seemed to be mainly responsible for mixed fine and coarse grains.

CONCLUSIONS

An investigation of abnormal grain growth in M-252 and S-816 alloys led to the following results and conclusions:

1. In a study of abnormal grain growth in M-252 and S-816 alloys, as well as in a prior study of Waspaloy, Inconel X-550, and Nimonic 80A alloys, only one cause for such grain growth was found. Small critical deformation of essentially strain-free metal is required. In the experimental work on M-252 and S-816 alloys, these deformations were in the range of 0.5 to 4.4 percent. Considering the data from all five of these heat-resistant alloys, the deformations inducing abnormal grain growth were usually within the range of 0.4 to 5.0 percent and were within the range of 0.1 to 9.7 percent for all variables considered. Normal solution-treating temperatures and times were sufficient for abnormal grain growth, although those commonly used for some alloys were marginal for growth.

2. The main problem in avoiding abnormal grain growth seems to be identification and avoidance of the often complex conditions which lead to critical deformation. In addition to small deformations themselves, nonuniform metal flow during working leaving part of the metal critically deformed appears to be a common source. Attention must be given to design of metal-working operations to insure more than critical deformation throughout the part being worked. Recrystallization during working leaves metal susceptible to critical deformation and care must be exercised to avoid small deformations after such recrystallization since abnormal grain growth can occur in such cases even with large overall reductions.

3. Rapid cooling of nearly strain-free material from the heating temperature can be the source of critical deformation leading to abnormal growth during subsequent heating.

4. Critical deformation and abnormal grain growth were remarkably independent of temperature of working. Abnormally high temperatures do not contribute very much to abnormal grain growth. Metallurgical variables such as grain size and initial heat treatment generally had very little effect. The major difference between alloys appears to be differences in temperatures and times for grain growth. The presence of extensive excess-phase precipitates restrained grain growth, tending to increase the temperatures and times for grain growth. The slight effects of prior

temperatures of heating probably resulted from the variations in solution of grain-growth restrainers.

5. In practice, a number of variables are often found important. Such variables as heating temperature, equipment used, heat-to-heat differences, melting practice, and alloy composition usually involve differences in metal flow characteristics. Thus, for a fixed operation, the flow characteristics may govern whether or not critical deformation occurs. When marginal temperatures and times for grain growth are present, the small inherent differences in grain-growth rates may also considerably influence the final grain size in a fixed hot-working operation. Experimentally it was found that, although many of the variables mentioned altered the details and extent of growth, the same mechanism of abnormal growth occurred in air- and vacuum-melted M-252 and Waspaloy alloys, and in air-melted S-816, Inconel X-550, and Nimonic 80A alloys.

University of Michigan,
Ann Arbor, Mich., June 15, 1956.

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TABLE I.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF M-252 ALLOY

Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °F	Final treatment for grain growth (a)	Percent reduction by rolling and ASTM grain size after final treatment as measured along tapered specimens												Critical reduction, percent	Minimum reduction to prevent abnormal grains, percent
Heat 43482																
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,600	4 hr at 1,950° F, AC	Reduction . . .	0	0.5	1.1	2.4	3.8	6.3	8.9	10.9	12.7	----	----	0.5	1.1
			Grain size . . .	6-8	(-1)-4	1-4	3-6	4-7	5-7	6-8	7-8	7-8	----	----		
Do	1,800	do	Reduction . . .	0	0.2	1.3	2.5	3.6	4.8	7.0	9.4	11.1	12.6	----	1.3	1.9
			Grain size . . .	6-8	6-8	0-4	2-5	3-6	4-7	5-7	5-8	6-8	6-8	----		
Do	1,950	do	Reduction . . .	0	0.2	0.5	2.0	3.2	4.5	5.9	8.0	9.8	11.8	13.5	2.0	0
			Grain size . . .	5-8	5-8	5-8	1-4	3-5	4-7	4-7	5-7	5-8	6-8	6-8		
Do	2,000	do	Reduction . . .	0	1.7	3.4	5.2	8.4	10.5	12.6	13.8	----	----	----	1.7	0
			Grain size . . .	6-8	1-2	2-4	3-5	5-7	6-8	6-8	6-8	----	----	----		
Do	2,100	do	Reduction . . .	0	1.7	3.4	5.5	8.4	10.0	11.4	12.7	----	----	----	1.7	0
			Grain size . . .	6-8	1-3	2-4	3-5	5-6	5-7	6-8	6-8	----	----	----		
Rolled 50 percent at 1,950° F plus $\frac{1}{2}$-hr preheat at 2,100° F then transferred to 1,600° F furnace for $\frac{1}{2}$ hr	1,600	do	Reduction . . .	0	2.0	2.7	4.5	6.8	8.4	9.7	12.1	----	----	----	2.0	2.7
			Grain size . . .	5-7	(-1)-1	1-3	2-5	3-6	5-7	5-7	5-8	----	----	----		
Rolled 50 percent at 1,950° F plus $\frac{1}{2}$ hr preheat at 2,100° F	2,100	do	Reduction . . .	0	1.3	2.3	4.6	6.9	9.6	10.1	12.4	----	----	----	1.3	0
			Grain size . . .	5-8	1-3	2-5	2-5	4-6	5-8	5-8	6-8	----	----	----		
Heat 63674																
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,600	4 hr at 1,950° F, AC	Reduction . . .	0	0.8	1.1	2.9	5.3	6.6	8.1	9.3	----	----	----	1.1	2.9
			Grain size . . .	6-8	6-7	(-1)-1	1-3	2-3	3-5	5-6	6-8	----	----	----		
Do	2,100	do	Reduction . . .	0	1.8	4.7	6.8	7.5	8.4	10.8	----	----	----	----	1.8	3.2
			Grain size . . .	6-8	0-1	2-3	3-4	4-5	5-7	6-8	----	----	----	----		
Heat A6891																
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,600	4 hr at 1,950° F, AC	Reduction . . .	0	0.7	2.6	5.3	6.6	8.1	9.8	----	----	----	----	0.7	2.6
			Grain size . . .	7-8	0-1	1-3	3-5	5-6	6-7	6-8	----	----	----	----		
Do	2,100	do	Reduction . . .	0	0.8	1.0	4.0	6.0	7.4	8.2	11.8	----	----	----	1.0	4.0
			Grain size . . .	6-8	6-8	0-2	1-3	2-4	3-5	5-7	6-8	----	----	----		

[a] AC, air-cooled.

TABLE I.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF M-252 ALLOY - Concluded

Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °F	Final treatment for grain growth (a)	Percent reduction by rolling and ASTM grain size after final treatment as measured along tapered specimens												Critical reduction, percent	Minimum reduction to prevent abnormal grains, percent
Heat A-41																
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,400	4 hr at 1,950° F, AC	Reduction . . .	0	0.7	1.9	3.6	5.7	7.3	8.2	10.1	----	----	----	1.9	0
			Grain size . . .	5-8	5-8	1-3	5-5	3-5	4-6	5-8	6-8	----	----	----		
Do	1,600	do	Reduction . . .	0	0.6	1.2	1.8	2.2	4.3	6.2	7.9	----	----	----	1.8	2.2
			Grain size . . .	6-8	6-8	6-7	0-1	1-3	4-6	5-8	6-8	----	----	----		
Do	1,900	do	Reduction . . .	0	0.7	1.5	3.6	6.0	7.6	8.7	10.5	----	----	----	1.5	2.5
			Grain size . . .	5-8	5-8	0-2	2-4	3-5	4-6	5-6	5-8	----	----	----		
Do	2,100	do	Reduction . . .	0	0.6	2.3	3.4	4.4	5.9	7.7	8.9	----	----	----	3.4	4.4
			Grain size . . .	4-6	4-6	4-6	0-1	1-3	3-5	4-6	4-7	----	----	----		
Rolled 50 percent at 2,100° F plus 1 hr at 1,950° F, AC	1,400	do	Reduction . . .	0	1.0	2.2	3.6	5.4	7.7	8.2	9.6	----	----	----	2.2	2.9
			Grain size . . .	4-7	4-6	0-1	2-4	3-5	4-6	5-6	6-8					
Do	1,600	do	Reduction . . .	0	0.6	1.2	2.0	2.8	4.8	5.7	7.9	----	----	----	2.0	0
			Grain size . . .	5-7	4-6	4-6	1-3	2-4	3-5	5-6	6-8	----	----	----		
Do	1,900	do	Reduction . . .	0	0.7	1.4	2.5	3.3	6.0	7.0	8.3	10.1	----	----	2.5	0
			Grain size . . .	4-8	4-8	4-7	1-2	2-4	3-5	4-7	5-7	5-8	----	----		
Do	2,100	do	Reduction . . .	0	0.6	1.7	2.8	3.9	5.9	7.2	8.3	----	----	----	2.8	0
			Grain size . . .	4-7	4-6	4-6	1-2	2-4	3-6	4-6	4-6	----	----	----		
Heat B-29																
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,400	4 hr at 1,950° F, AC	Reduction . . .	0	0.7	1.2	3.3	5.6	7.9	9.3	10.4	----	----	----	0.7	1.2
			Grain size . . .	6-8	0-1	1-3	3-5	5-6	5-7	6-8	6-8	----	----	----		
Do	1,600	do	Reduction . . .	0	1.2	1.4	1.7	3.2	5.6	7.4	----	----	----	----	1.4	1.7
			Grain size . . .	5-8	5-8	(-1)-1	1-3	3-4	5-7	6-8	----	----	----	----		
Do	1,900	do	Reduction . . .	0	0.8	1.0	3.0	4.8	6.5	8.0	9.4	----	----	----	.8	1.0
			Grain size . . .	6-8	(-1)-1	1-3	2-4	4-5	5-7	6-8	6-8	----	----	----		
Do	2,100	do	Reduction . . .	0	0.6	1.7	2.8	3.9	5.8	7.7	8.4	----	----	----	2.8	3.3
			Grain size . . .	5-7	5-6	4-6	0-2	2-3	3-5	5-7	6-8	----	----	----		
Rolled 50 percent at 2,100° F plus 1 hr at 1,950° F, AC	1,400	do	Reduction . . .	0	0.7	1.2	1.7	3.9	5.6	6.9	8.1	10.0	----	----	1.2	1.5
			Grain size . . .	5-7	5-7	0-1	2-3	3-5	4-6	4-6	5-7	6-8	----	----		
Do	1,600	do	Reduction . . .	0	0.6	1.2	2.0	2.8	4.3	5.7	8.9	----	----	----	2.0	2.5
			Grain size . . .	5-6	4-6	4-6	(-1)-2	2-3	3-5	4-6	5-6	----	----	----		
Do	1,900	do	Reduction . . .	0	1.1	2.0	3.0	3.9	7.2	7.6	9.3	11.8	----	----	2.0	2.3
			Grain size . . .	5-6	5-6	0-1	3-5	3-5	4-6	4-6	5-3	6-8	----	----		
Do	2,100	do	Reduction . . .	0	0.6	1.1	2.5	3.9	5.9	7.2	7.9	----	----	----	2.5	2.9
			Grain size . . .	4-6	4-6	4-7	0-2	3-4	4-5	5-6	5-7	----	----	----		

[a] AC, air-cooled.

TABLE II.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF S-816 ALLOY

Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °F	Final treatment for grain growth (a)	Percent reduction by rolling and ASTM grain size after final treatment as measured along tapered specimens											Critical reduction, percent	Minimum reduction to prevent abnormal grains, percent
Rolled 15 percent at 1,000° F plus 1 hr at 2,300° F, WQ	1,000	1 hr at 2,300° F, WQ	Reduction . . .	0	0.9	1.1	1.5	2.0	4.0	6.0	8.0	10.0	12.0	0.9	2.0
			Grain size . . .	5-8	(-1)-1	0-3	0-3	1-3	3-4	5-8	5-8	5-8	5-8		
Do-------------	1,400	------------do------------	Reduction . . .	0	0.8	1.8	2.0	4.0	6.0	8.0	10.0	12.0	----	.8	2.7
			Grain size . . .	5-8	(-1)-1	0-1	0-1	3-4	5-8	5-8	5-8	5-8	----		
Do-------------	1,800	------------do------------	Reduction . . .	0	0.7	1.3	2.0	4.0	6.0	8.0	10.0	12.0	----	.7	2.0
			Grain size . . .	5-8	(-1)-1	0-2	1-2	2-4	3-4	5-8	5-8	5-8	----		
Do-------------	2,000	------------do------------	Reduction . . .	0	0.7	1.0	2.0	3.6	4.8	6.0	8.0	10.0	12.0	1.0	2.8
			Grain size . . .	5-8	3-1	(-1)-0	0-1	2-4	3-4	5-8	5-8	5-8	5-8		
Do-------------	2,100	------------do------------	Reduction . . .	0	0.8	1.5	2.4	3.8	6.0	8.0	10.0	12.0	----	.8	2.9
			Grain size . . .	5-8	(-2)-0	0-1	0-1	3-4	5-8	5-8	5-8	5-8	----		
Do-------------	2,150	------------do------------	Reduction . . .	0	1.3	1.6	2.6	3.9	4.8	6.0	8.0	10.0	12.0	1.3	2.6
			Grain size . . .	5-8	(-2)-1	0-1	1-2	4-5	5-6	6-8	6-8	6-8	6-8		
Do-------------	2,200	------------do------------	Reduction . . .	0	1.0	1.4	2.6	4.4	5.5	6.8	8.0	10.0	12.0	1.0	2.2
			Grain size . . .	5-8	1-2	(-1)-0	2-3	4-5	4-5	6-8	6-8	6-8	6-8		
Do-------------	2,250	------------do------------	Reduction . . .	0	0.5	1.5	2.0	3.1	4.7	6.5	8.0	10.0	12.0	1.5	1.8
			Grain size . . .	5-8	3-5	(-1)-1	3-4	4-5	4-5	5-8	5-8	5-8	5-8		
Do-------------	1,400	1 hr at 2,150° F, WQ	Reduction . . .	0	0.8	1.7	2.6	4.0	6.0	8.0	10.0	12.0	----	.8	0
			Grain size . . .	5-8	3-4	3-4	4-5	5-8	5-8	5-8	5-8	5-8	----		
Do-------------	1,800	------------do------------	Reduction . . .	0	0.8	1.6	2.4	4.4	6.0	8.0	10.0	12.0	----	.8	0
			Grain size . . .	5-8	4-5	4-6	4-8	5-8	6-8	6-8	6-8	6-8	----		
Do-------------	2,200	------------do------------	Reduction . . .	0	1.3	2.6	4.1	6.0	8.0	10.0	12.0	----	----	1.3	0
			Grain size . . .	5-8	3-5	5-8	6-8	6-8	6-8	6-8	6-8	----	----		

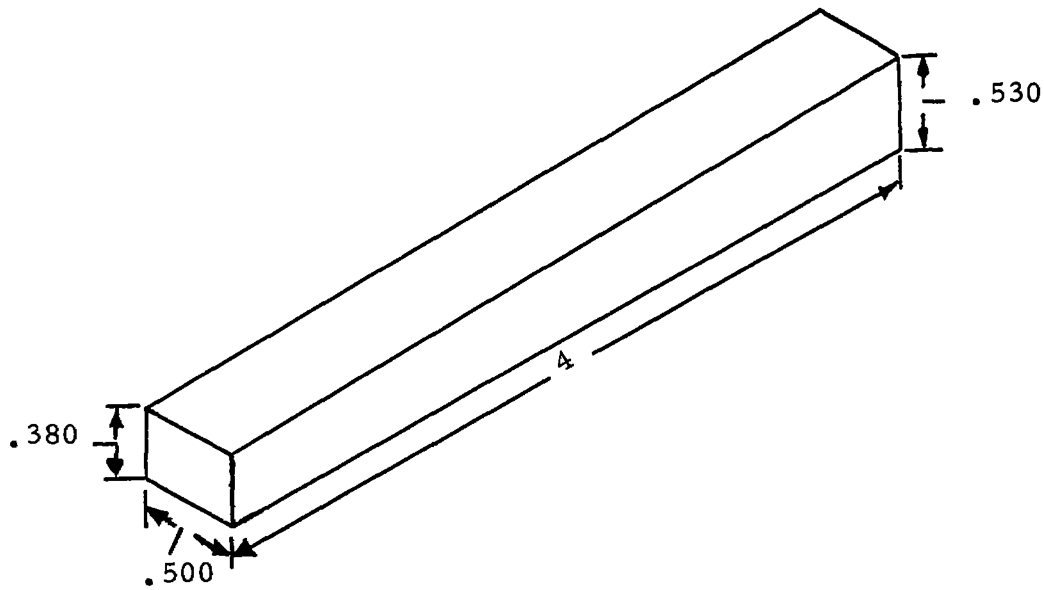
[a] WQ, water-quenched.

TABLE II.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF S-816 ALLOY - Concluded

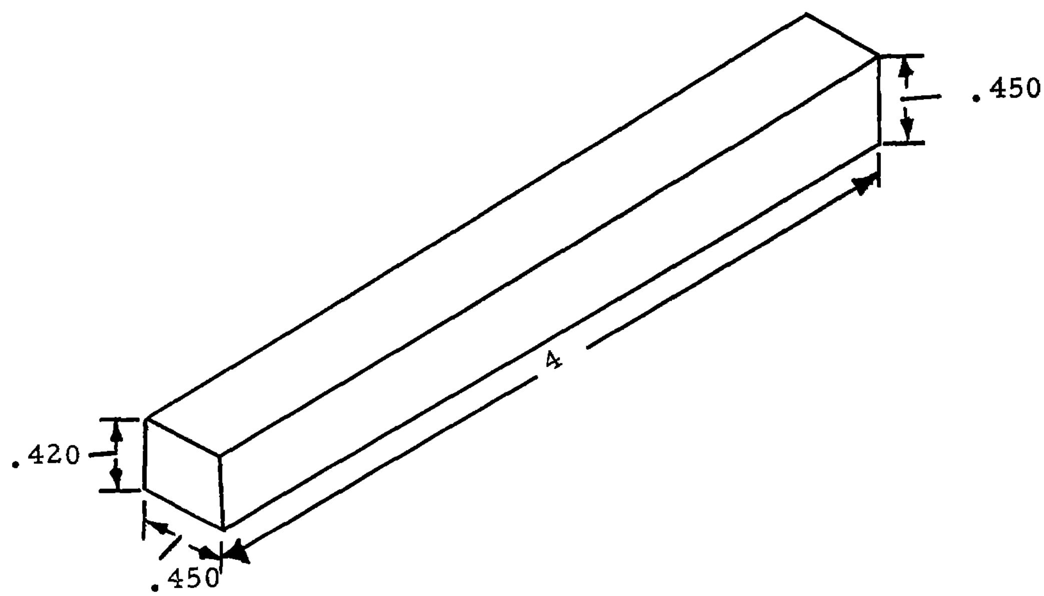
Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °F	Final treatment for grain growth (a)	Percent reduction by rolling and ASTM grain size after final treatment as measured along tapered specimens											Critical reduction, percent	Minimum reduction to prevent abnormal grains, percent
Rolled 15 percent at 1,000° F plus 1 hr at 2,150° F, AC	1,400	1 hr at 1,800° F, AC	Reduction . . .	0	0.2	0.7	3.1	3.2	7.5	8.7	9.5	----	----	None	0
			Grain size . . .	6-8	6-8	5-7	5-7	6-8	6-8	6-8	6-8	----	----		
Do------------	1,400	1 hr at 1,900° F, AC	Reduction . . .	0	0.2	0.7	2.2	4.0	5.0	7.4	8.8	----	----	None	0
			Grain size . . .	6-8	6-8	6-8	6-8	6-8	5-7	5-7	6-8	----	----		
Do------------	1,400	1 hr at 2,000° F, AC	Reduction . . .	0	1.4	3.2	4.0	5.8	6.9	8.1	----	----	----	None	0
			Grain size . . .	6-8	6-8	5-7	5-6	6-7	6-8	6-8	----	----	----		
Do------------	1,400	1 hr at 2,100° F, AC	Reduction . . .	0	0.3	2.2	4.4	5.3	6.5	7.6	----	----	----	2.2	0
			Grain size . . .	6-8	4-6	1-3	2-5	4-6	5-7	6-8	----	----	----		
Do------------	1,400	1 hr at 2,150° F, WQ	Reduction . . .	0	1.2	3.7	6.2	8.0	9.6	11.1	12.4	----	----	1.2	0
			Grain size . . .	5-8	1-3	2-4	3-7	5-8	5-8	6-8	6-8	----	----		
Do------------	1,400	1 hr at 2,200° F, WQ	Reduction . . .	0	0.7	1.7	3.6	5.6	7.5	9.5	9.8	----	----	.7	2.9
			Grain size . . .	6-8	(-1)-2	(-1)-2	2-4	4-6	5-7	5-7	6-8	----	----		
Do------------	1,400	1 hr at 2,250° F, WQ	Reduction . . .	0	2.8	4.0	6.3	8.3	9.7	11.7	----	----	----	2.8	3.5
			Grain size . . .	4-6	(-1)-2	4-6	5-6	5-7	6-8	6-8	----	----	----		
Do------------	1,400	1 hr at 2,300° F, WQ	Reduction . . .	0	1.2	1.7	4.2	6.6	8.3	10.2	11.8	13.2	----	1.2	2.6
			Grain size . . .	4-7	0-3	0-3	3-6	5-7	5-8	5-8	5-8	5-8	----		
Do------------	2,250	1 hr at 2,200° F, WQ	Reduction . . .	0	0.7	1.3	2.8	4.6	6.8	8.7	9.7	10.8	----	1.3	2.0
			Grain size . . .	4-7	3-5	(-1)-3	3-5	5-6	5-6	5-7	6-8	6-8	----		
Do------------	2,250	1 hr at 2,250° F, WQ	Reduction . . .	0	0.5	1.0	2.8	5.0	7.2	8.4	10.1	10.6	----	.5	1.6
			Grain size . . .	3-5	1-6	0-2	3	5-6	5-6	5-6	5-7	5-7	----		
Do------------	1,400	3 cycles of 1 hr at 2,150° F, AC, plus 1 hr at 2,150° F, WQ	Reduction . . .	0	0.9	1.7	4.6	6.4	8.1	9.8	11.8	13.0	----	.9	3.2
			Grain size . . .	5-8	(-2)-3	0-3	2-4	4-7	5-8	5-8	5-8	6-8	----		
Do------------	1,400	3 cycles of 1 hr at 2,150° F, AC, plus 1 hr at 2,300° F, WQ	Reduction . . .	0	0.8	1.2	1.9	4.6	6.8	8.7	10.2	11.9	13.0	.8	3.2
			Grain size . . .	2-7	(-4)-0	(-2)-0	0-3	2-5	4-6	4-6	4-6	4-6	4-6		
Do------------	[b]1,400	1 hr at 2,300° F, WQ	Reduction . . .	0	0.2	0.5	0.8	1.0	1.5	2.0	2.5	3.0	3.2	0-1.5	2.5
			Grain size . . .	3-7	0-5	(-2)-3	(-3)-2	(-2)-3	(-1)-3	0-4	1-4	2-5	2-5		

[a] WQ, water-quenched; AC, air-cooled.

[b] Rolled at 1,400° F, plus 1 hr at 2,150° F, AC, plus rolled at 1,400° F, plus 1 hr at 2,150° F, AC, plus rolled at 1,400° F.

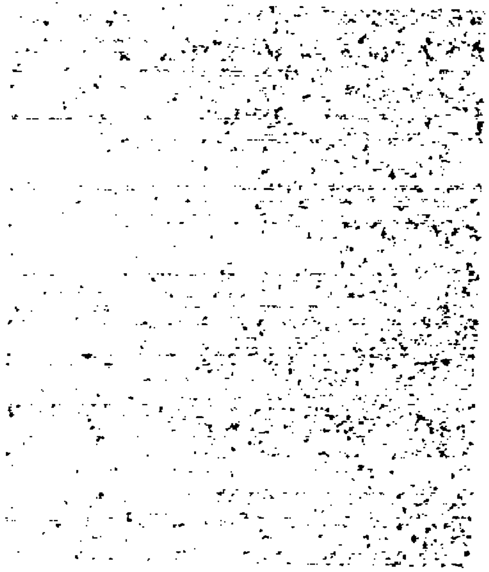


(a) Tapered specimen used to obtain approximately 0- to 15-percent reduction in one pass.



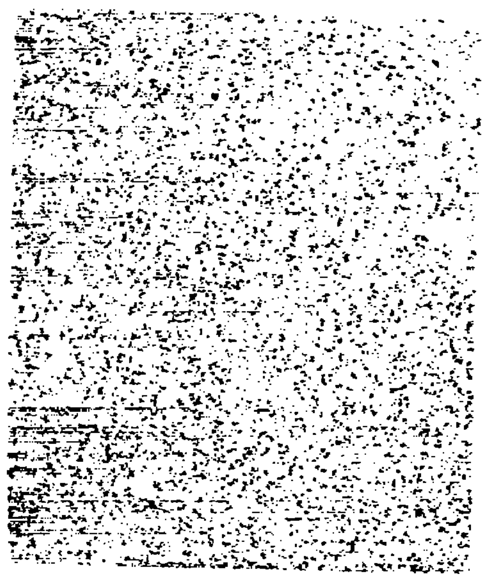
(b) Tapered specimen giving reductions of 0 to 5 percent per pass used to study effect of repeated critical reductions. Specimen remachined between passes to obtain same range of reductions.

Figure 1.- Tapered specimens used to obtain range of percent reduction by rolling to flat bars (dimensions in inches).



(a) Heat 43482; grain size, 6 to 8.

(b) Heat 63674; grain size, 6 to 8.



(c) Heat A6891; grain size, 8 to less than 8.

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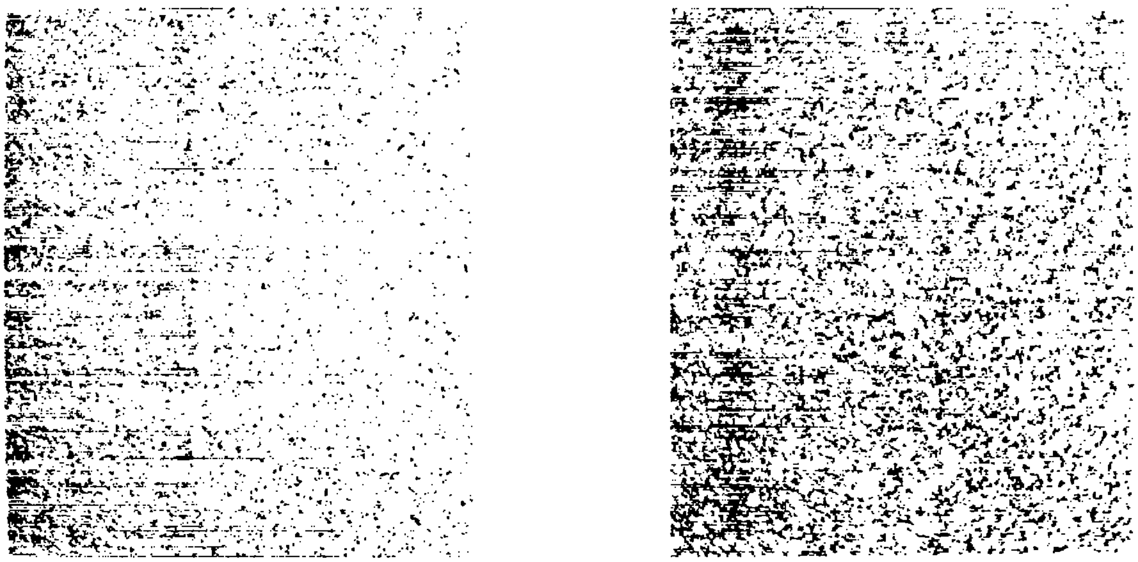
Figure 2.- Microstructures of air-melted M-252-alloy bar stocks after equalizing treatment of 50-percent reduction at 1,950° F plus 1 hour at 1,950° F, then air-cooled. Magnification, X50.



(a) Heat A-41; grain size, 7 to 8; low silicon and manganese content.

(b) Heat B-29; grain size, 5 to 8; normal composition.

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Figure 3.- Microstructures of vacuum-melted M-252-alloy bar stock after equalizing treatment of 50-percent reduction by rolling at 1,950° F plus 1 hour at 1,950° F, then air-cooled. Magnification, X50.

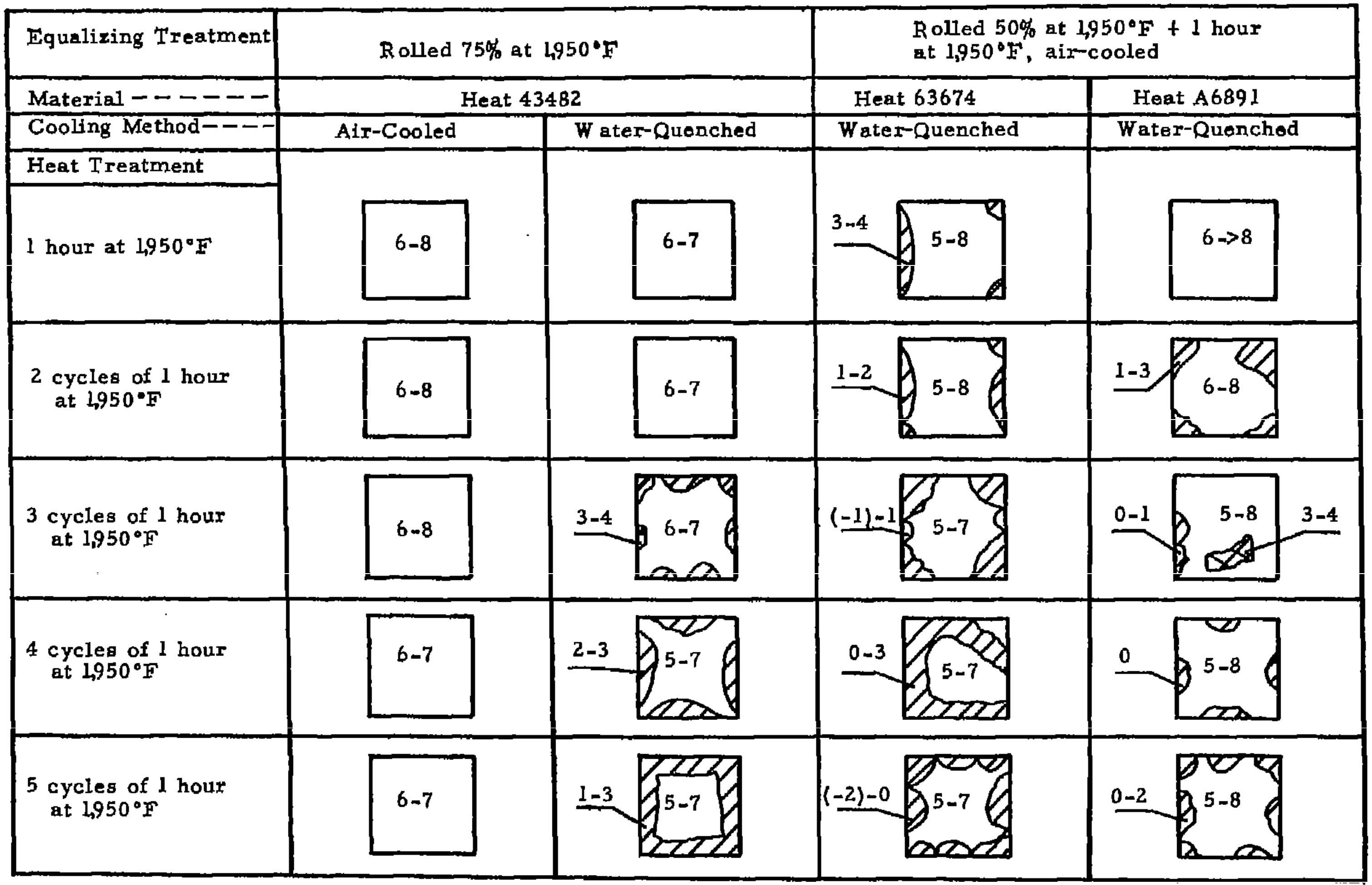
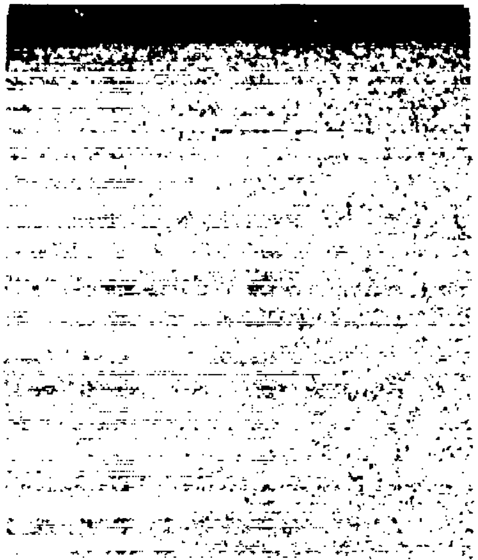
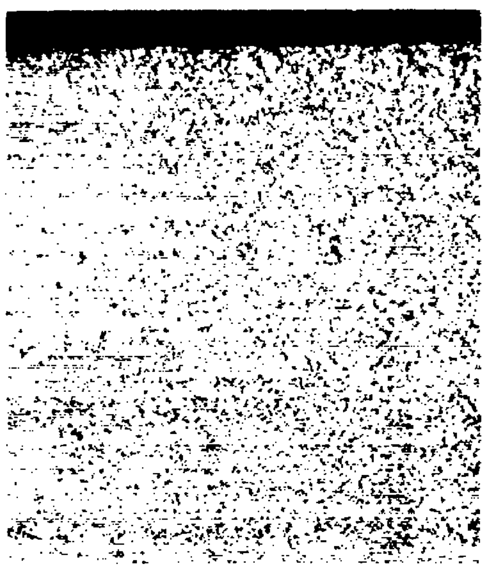


Figure 4.- Effect of repeated heating and cooling upon grain sizes of transverse sections of air-melted M-252-alloy bar stock.



(a) 1 hour at 1,950° F, then water-quenched.

(b) Two cycles of 1 hour at 1,950° F, then water-quenched.



(c) Four cycles of 1 hour at 1,950° F, then water-quenched.

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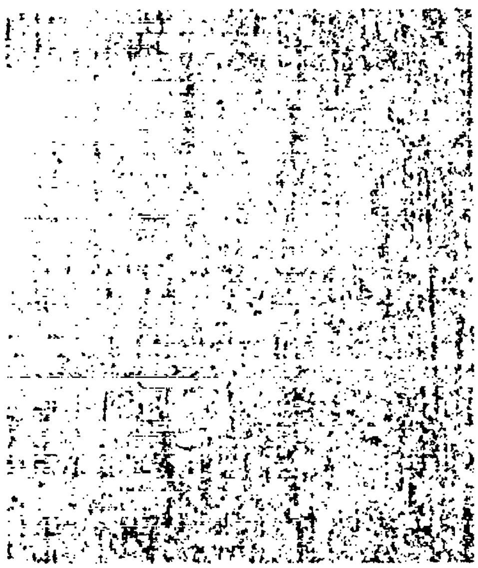
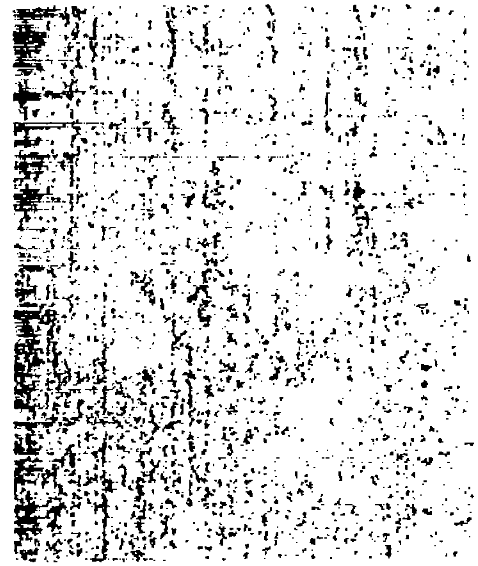
Figure 5.- Effect of repeated heating and cooling upon microstructure of transverse sections of heat 43482 of air-melted M-252-alloy bar stock. Equalizing treatment was 75-percent reduction at 1,950° F. Magnification, X50.

Equalizing Treatment	Rolled 50% at 1,950°F + 1 hour at 1,950°F, air-cooled		Rolled 50% at 2,100°F + 1 hour at 1,950°F, air-cooled	
Material-------- / Heat Treatment	Heat A-41	Heat B-29	Heat A-41	Heat B-29
1 hour at 1,950°F, air-cooled	8-10	6-8	6-8	5-7
1 hour at 1,950°F, air-cooled + 4 hours at 1,950°F, air-cooled	5-6	4-7	5-7	5-6
5 hours at 1,950°F, air-cooled	6-8	4-5	5-8	4-7
1 hour at 1,950°F, water-quenched	7-8	6-8	6-8	5-7
1 hour at 1,950°F, water-quenched + 4 hours at 1,950°F, air-cooled	1-2, 2-4	0-3, 2-4	5-7	0-4, 4-6

Figure 6.- Effect of equalizing treatment and repeated heating and cooling upon grain growth of vacuum-melted heats A-41 and B-29 of M-252 alloy.

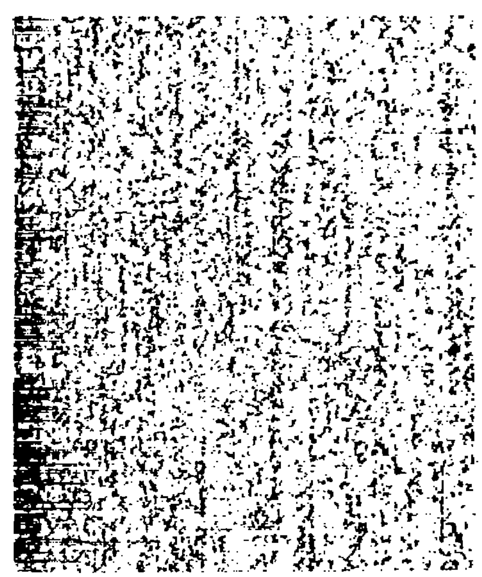


(a) Zero-percent reduction.



(b) 2.0-percent reduction.

(c) 2.2-percent reduction.



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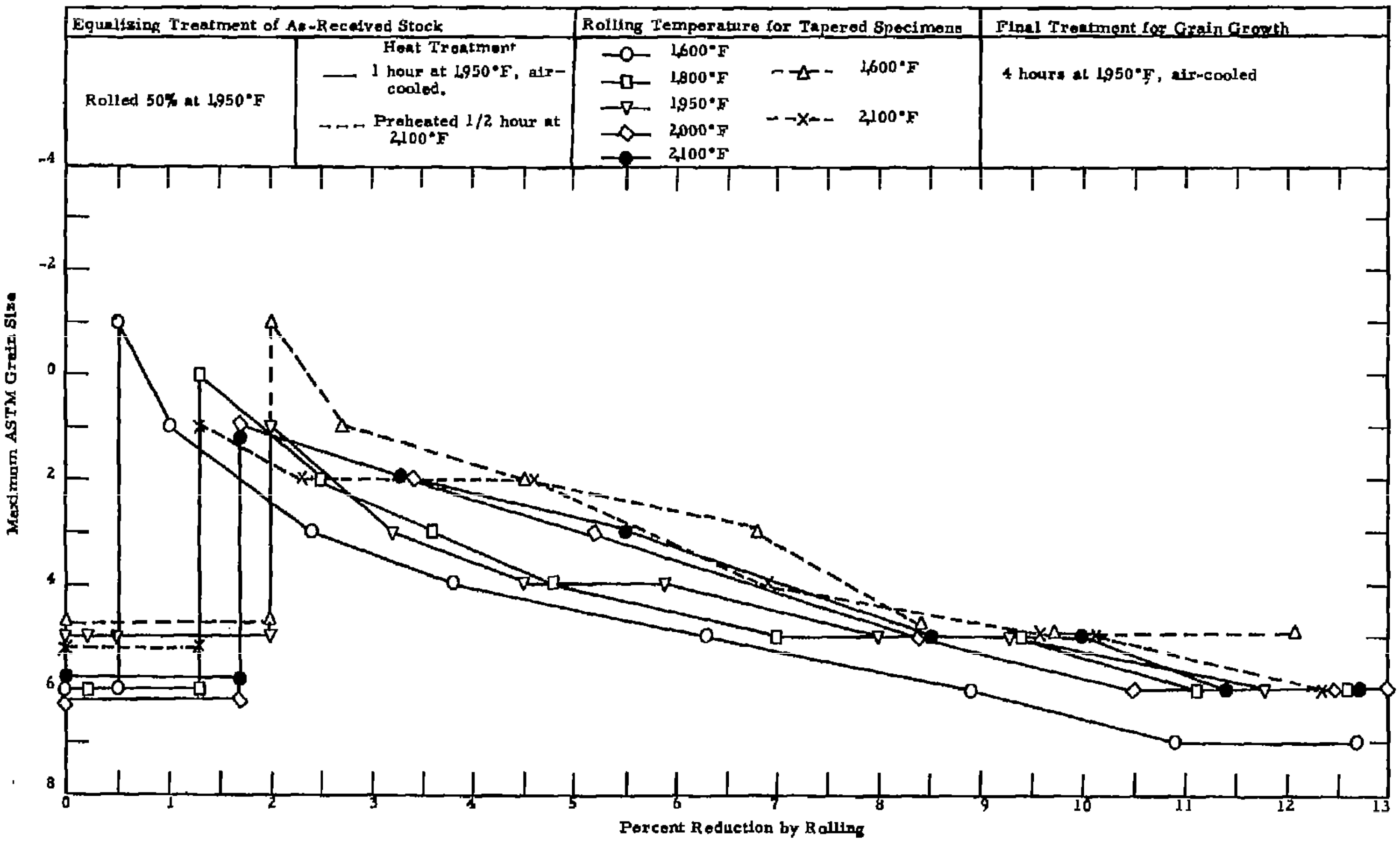
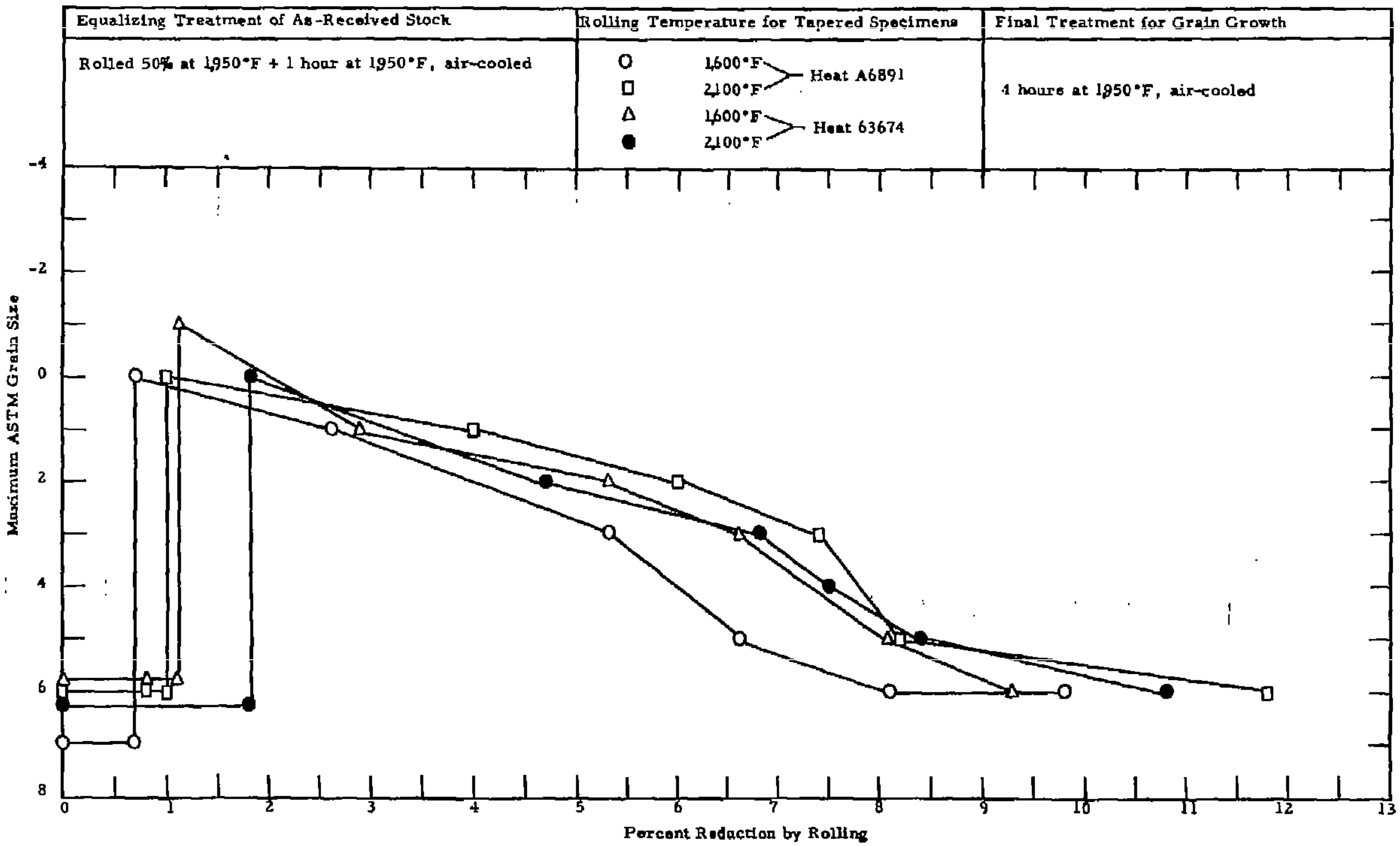
(d) 13.0-percent reduction.

Figure 7.- Effect of percent reduction by rolling at 1,950° F upon microstructure of heat 43482 of air-melted M-252 alloy after final solution treatment. Equalizing treatment of as-received stock was a 50-percent reduction at 1,950° F plus 1 hour at 1,950° F, then air-cooled. Final solution treatment was 4 hours at 1,950° F, then air-cooled. Magnification, X50.



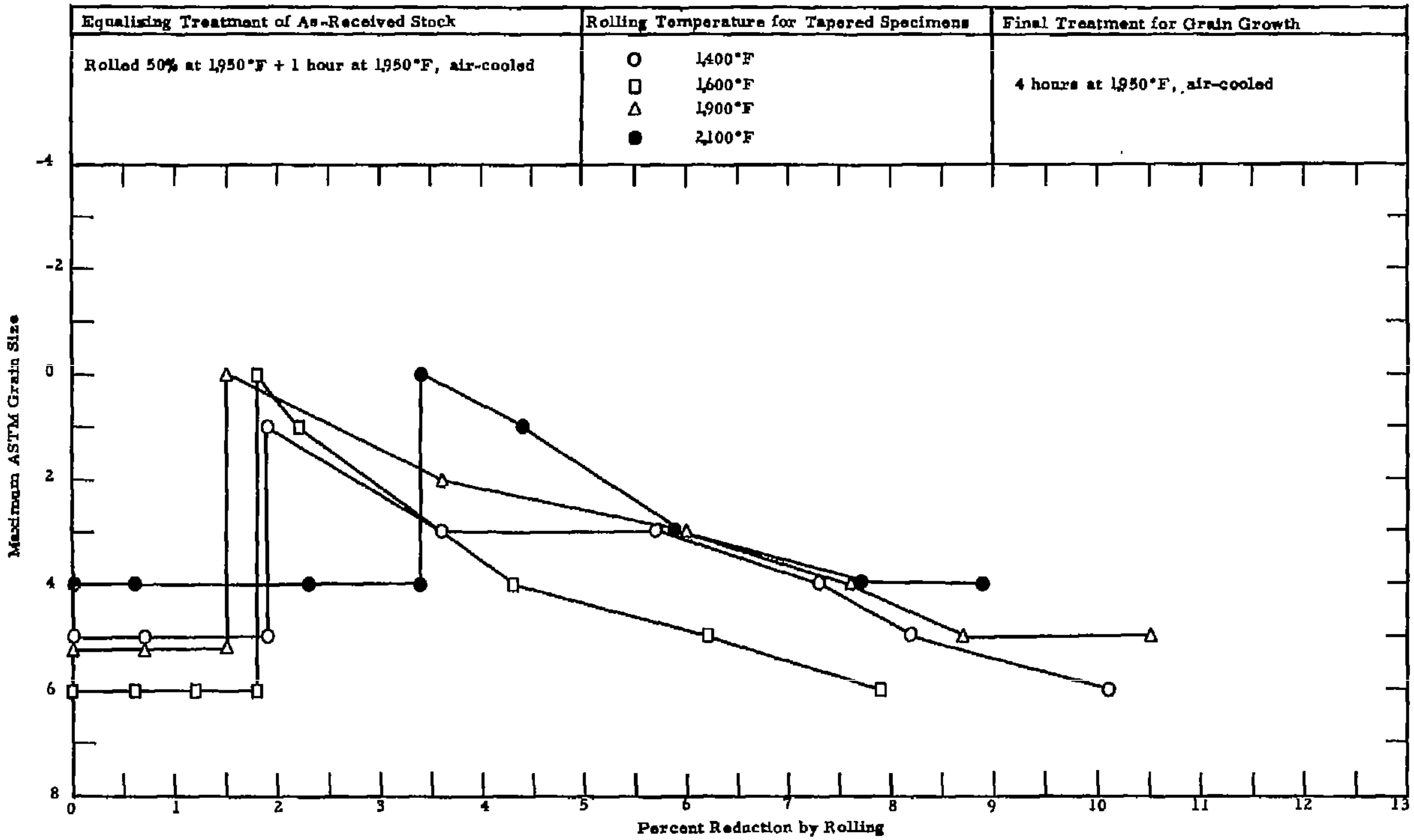
(a) Heat 43482 of air-melted alloy.

Figure 8.- Effect of rolling temperature and percent reduction upon maximum grain size of M-252 alloy after final solution treatment.



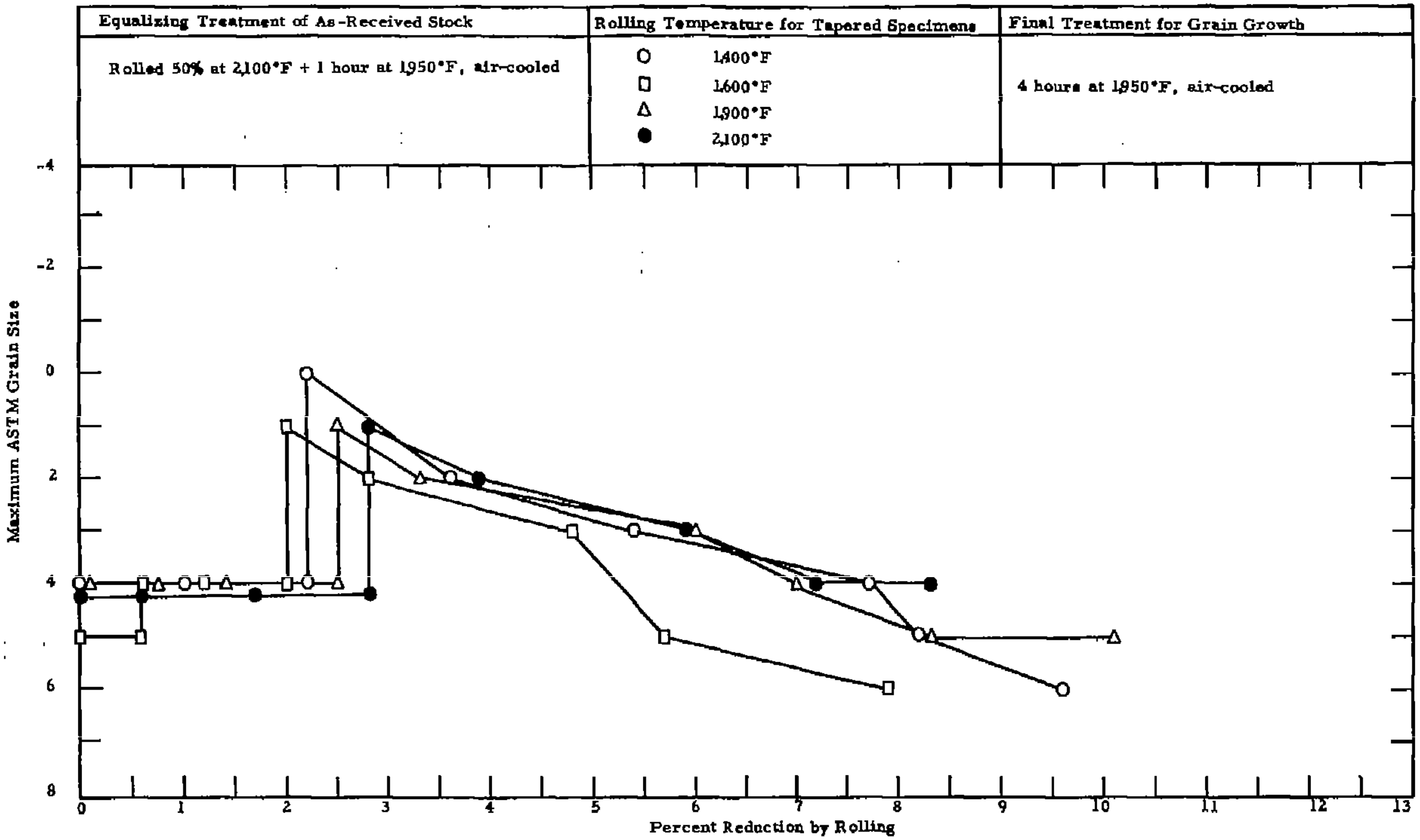
(b) Heats 63674 and A6891 of air-melted alloy.

Figure 8.- Continued.



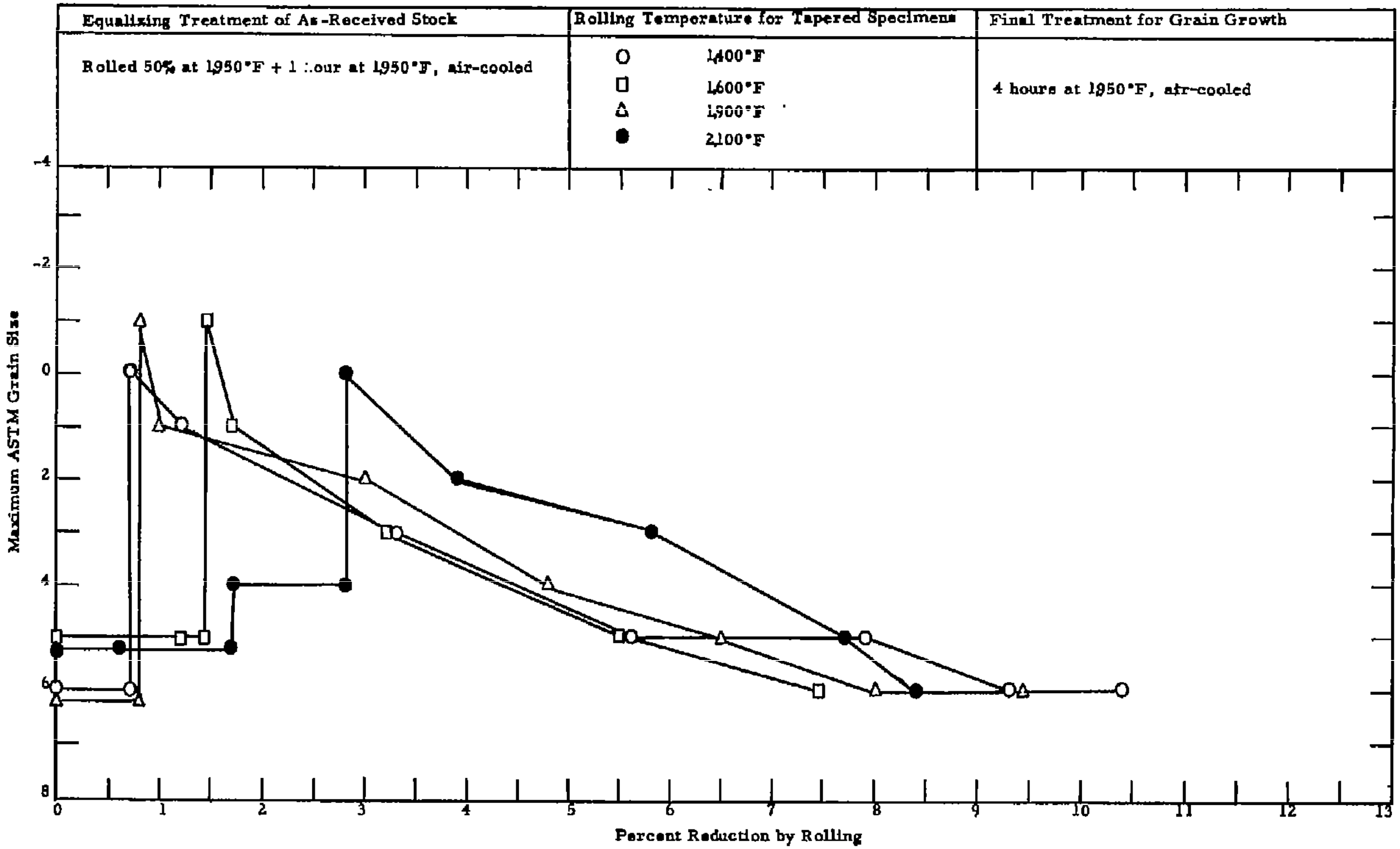
(c) Low manganese and silicon heat A-41 of vacuum-melted alloy. Equalizing treatment included rolling at 1,950° F.

Figure 8.- Continued.



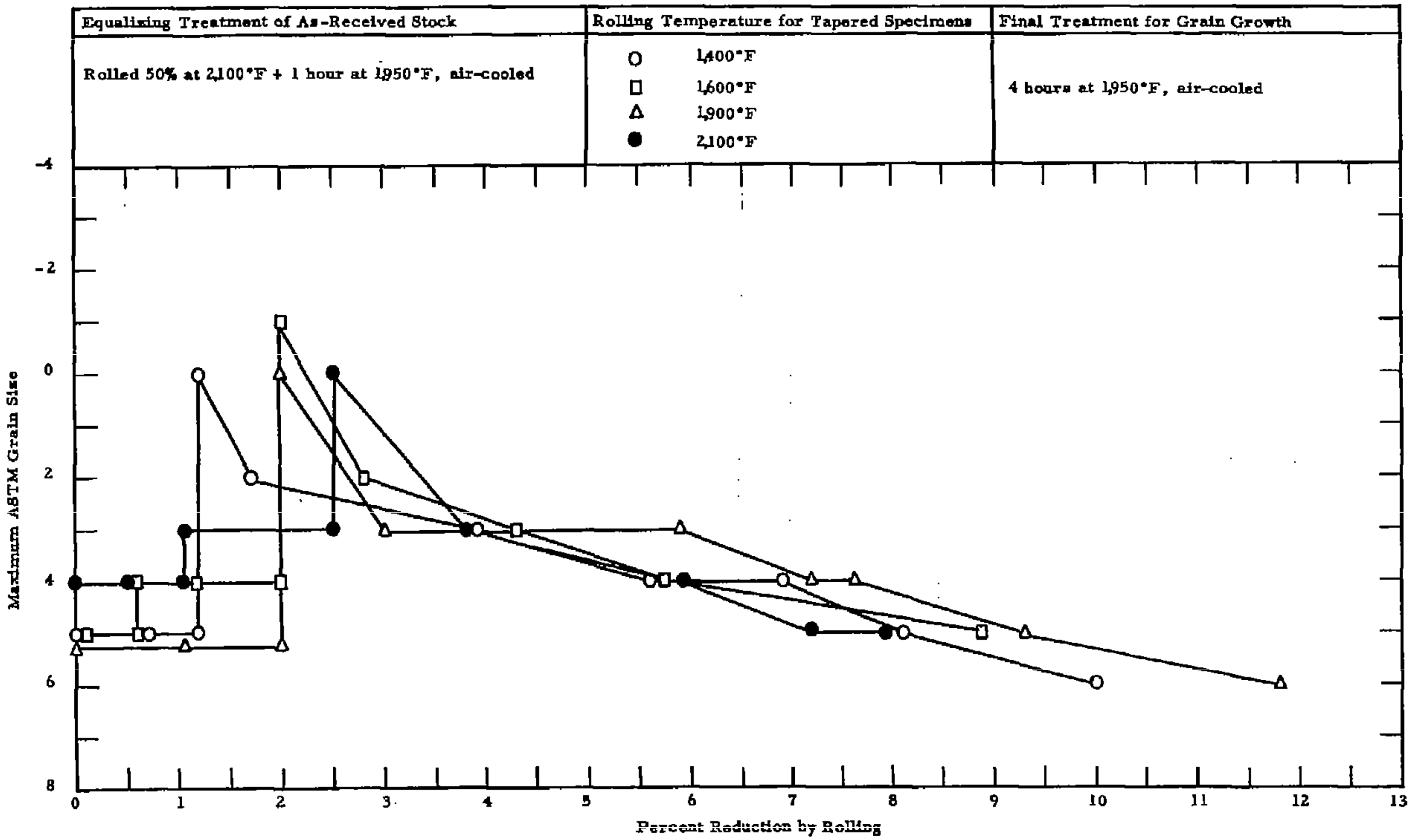
(d) Low manganese and silicon heat A-41 of vacuum-melted alloy. Equalizing treatment included rolling at 2,100° F.

Figure 8.- Continued.



(e) Normal manganese and silicon heat B-29 of vacuum-melted alloy. Equalizing treatment included rolling at 1,950° F.

Figure 8.- Continued.



(f) Normal manganese and silicon heat B-29 of vacuum-melted alloy. Equalizing treatment included rolling at 2,100° F.

Figure 8.- Concluded.

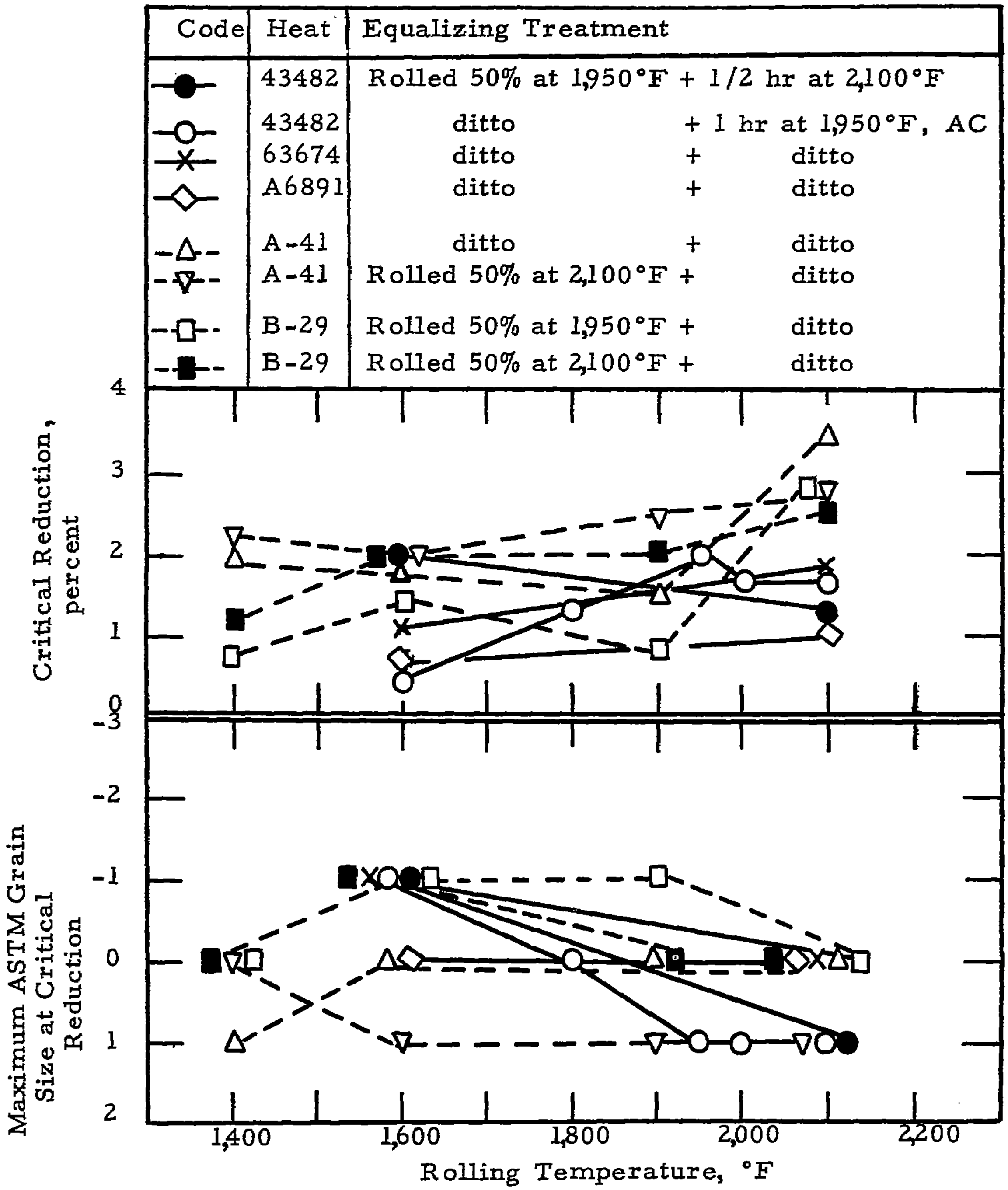
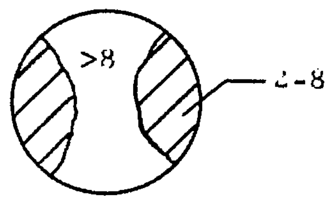
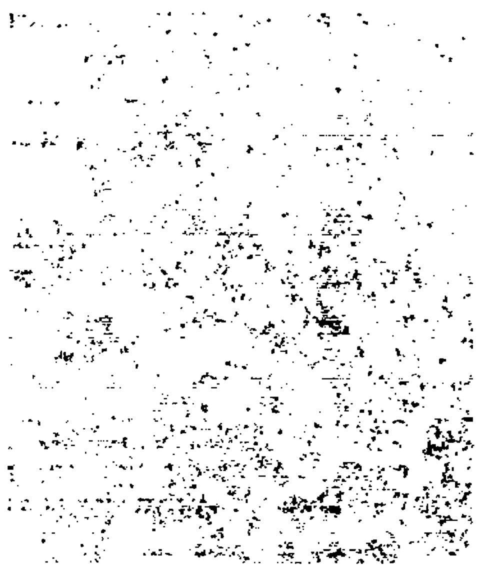


Figure 9.- Summarized comparison of critical reductions and maximum grain sizes for air- and vacuum-melted M-252 alloy. AC, air-cooled.



(a) Approximate distribution of grain sizes.



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

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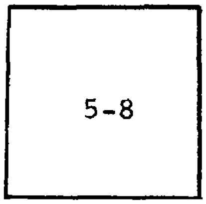
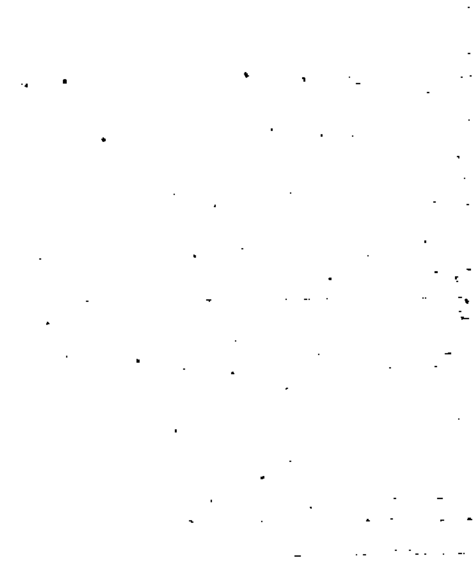
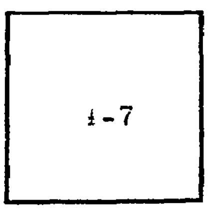
Figure 10.- Microstructure and grain sizes of transverse section of as-received S-816-alloy bar stock.



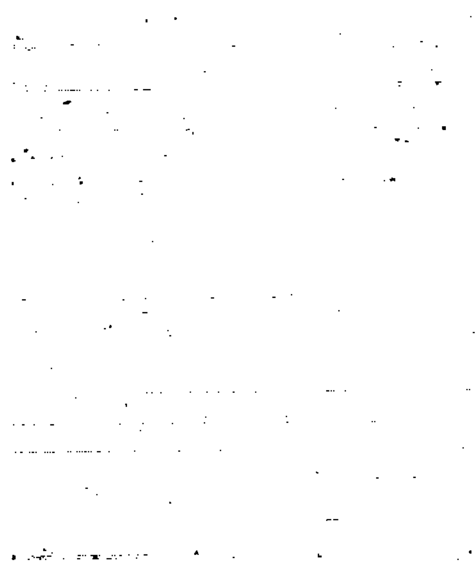
(a) Approximate distribution of grain sizes after 15-percent reduction at 1,000° F plus 1 hour at 2,150° F.



(b) Microstructure after 15-percent reduction at 1,000° F plus 1 hour at 2,150° F. Magnification, X50.



(c) Approximate distribution of grain sizes after 15-percent reduction at 1,000° F plus 1 hour at 2,300° F.



(d) Microstructure after 15-percent reduction at 1,000° F plus 1 hour at 2,300° F. Magnification, X50.

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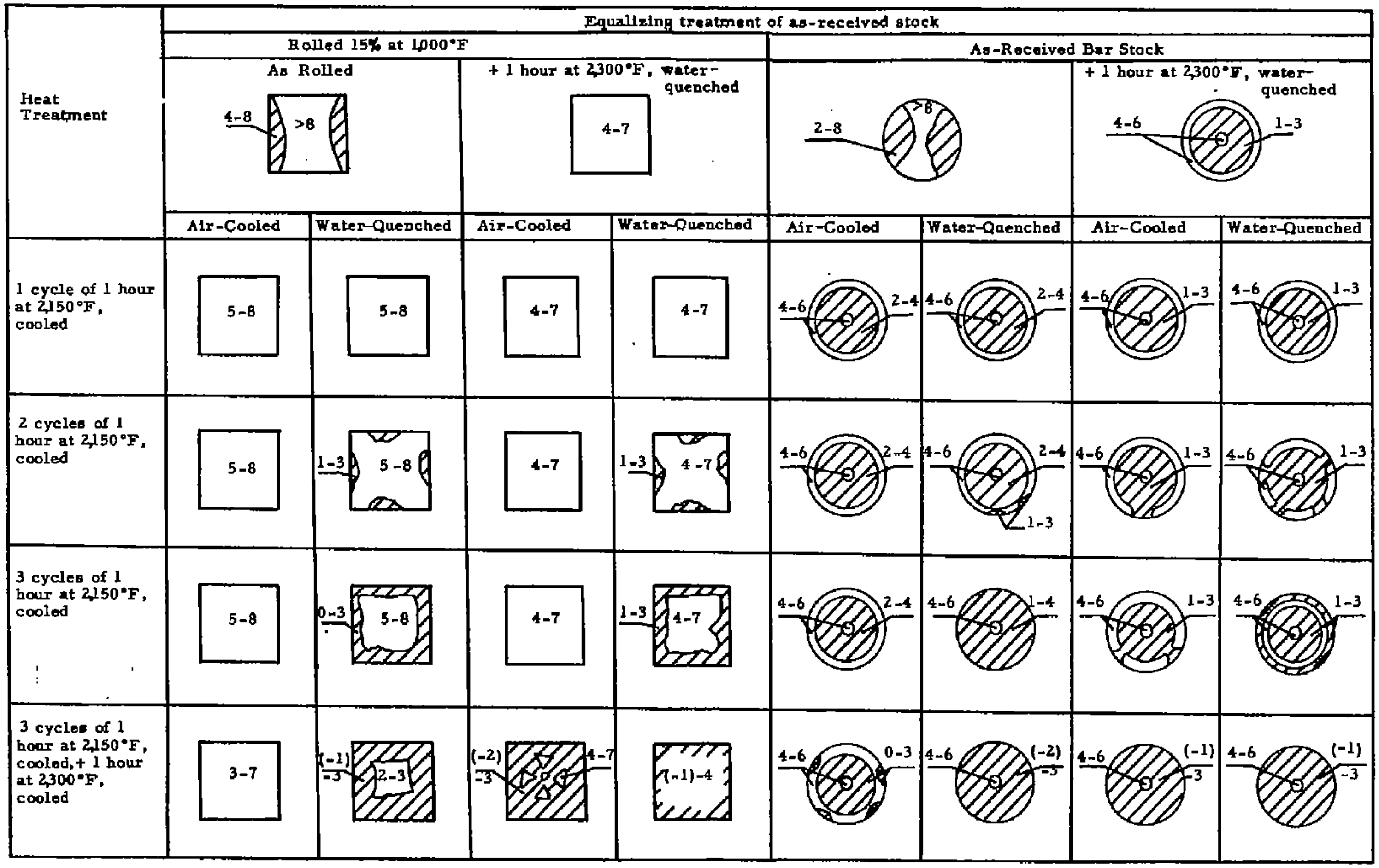
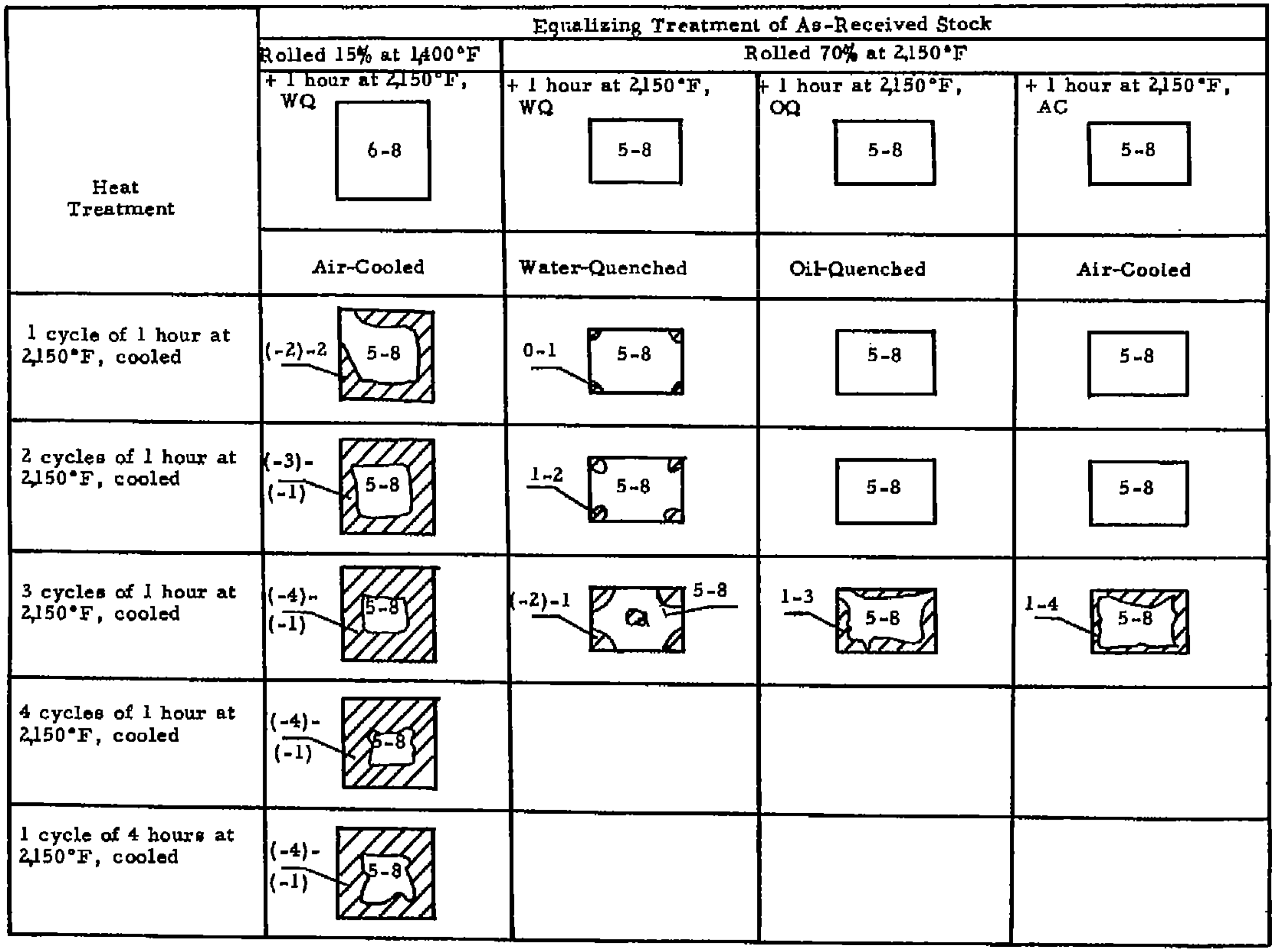
Figure 11.- Microstructures and grain sizes of transverse sections of equalized S-816-alloy bar stock.



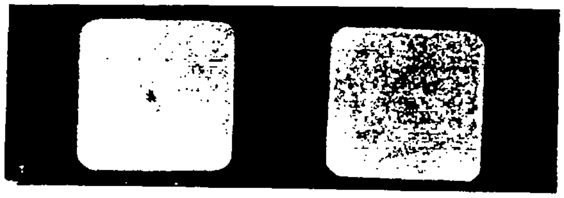
(a) As-received bar stock and stock rolled 15 percent at 1,000° F.

Figure 12.- Effect of repeated heating and cooling upon grain sizes of transverse sections of S-816-alloy bar stock.



(b) Stock rolled 15 percent at 1,400° F and then rolled 70 percent at 2,150° F. WQ, water-quenched; OQ, oil-quenched; AC, air-cooled.

Figure 12.- Concluded.

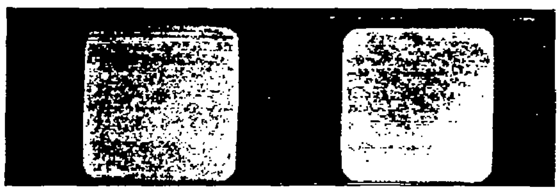


Air-cooled Water-quenched

(a) Reduced 15 percent at 1,000° F, reheated to 2,150° F three times, and cooled as indicated.

Air-cooled Water-quenched

(b) Reduced 15 percent at 1,000° F, reheated to 2,150° F three times, and cooled as indicated, and finally solution-treated 1 hour at 2,300° F.



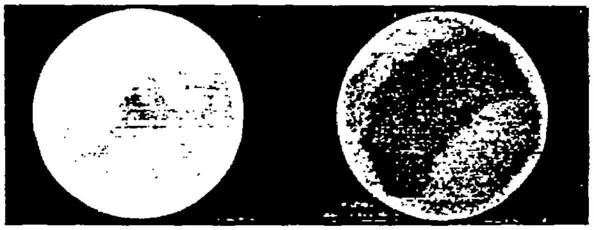
Air-cooled Water-quenched

(c) Reduced 15 percent at 1,000° F, solution-treated 1 hour at 2,300° F and water-quenched, and then reheated to 2,150° F three times and cooled as indicated.

Air-cooled Water-quenched L-57-4033

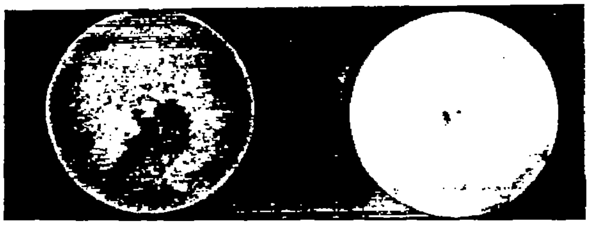
(d) Reduced 15 percent at 1,000° F, solution-treated 1 hour at 2,300° F and water-quenched, reheated to 2,150° F three times and cooled as indicated, and finally solution treated again at 2,300° F.

Figure 13.- Macrographs showing grain-size distribution in S-816-alloy bar stock after repeated heating to 2,150° F and cooling. Magnification, X1.5.



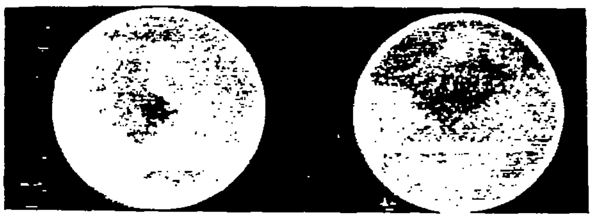
Air-cooled Water-quenched

(e) As-received bar stock heated three times to 2,150° F and cooled as indicated.



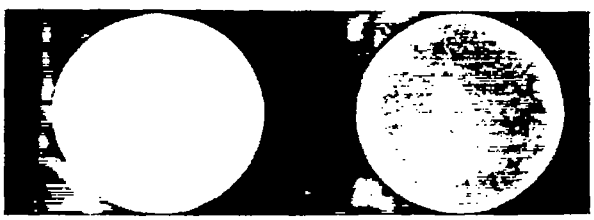
Air-cooled Water-quenched

(f) As-received bar stock heated three times to 2,150° F and cooled as indicated and finally solution-treated for 1 hour at 2,300° F.



Air-cooled Water-quenched

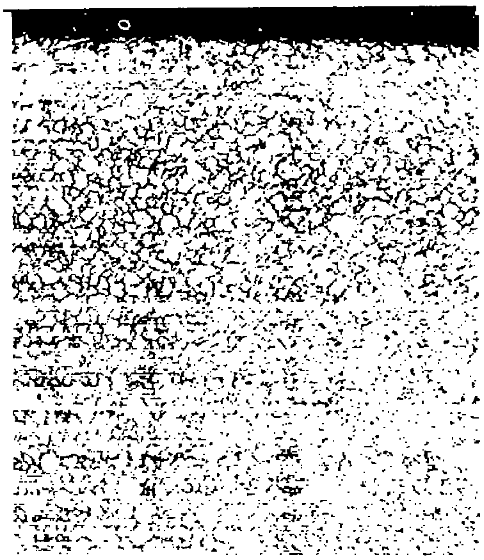
(g) As-received bar stock solution-treated for 1 hour at 2,300° F and water-quenched and reheated to 2,150° F three times and cooled as indicated.



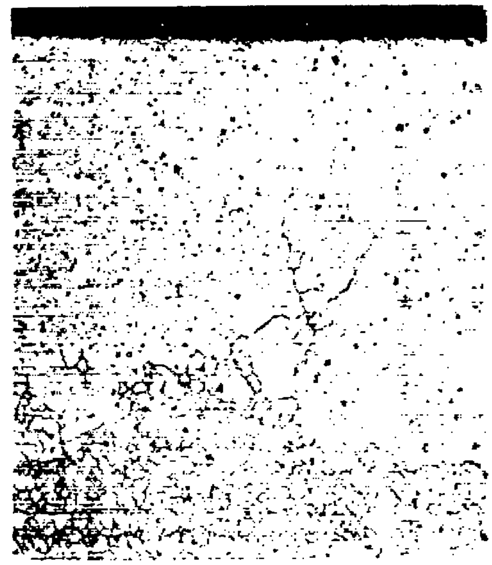
Air-cooled Water-quenched L-57-4034

(h) As-received bar stock solution-treated for 1 hour at 2,300° F and water-quenched, reheated to 2,150° F three times and cooled as indicated, and finally solution-treated again at 2,300° F.

Figure 13.- Concluded.

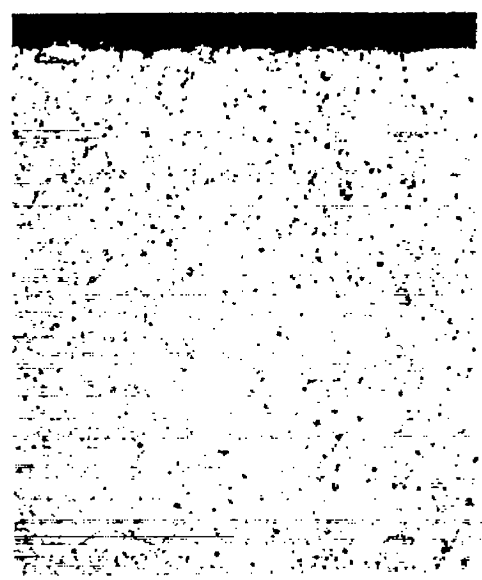


(a) 1 hour at 2,150° F, then water-quenched.



(b) 1 hour at 2,150° F, then water-quenched, plus 1 hour at 2,150° F, then air-cooled.

(c) 1 hour at 2,150° F, then water-quenched, plus four cycles of 1 hour at 2,150° F, then air-cooled.



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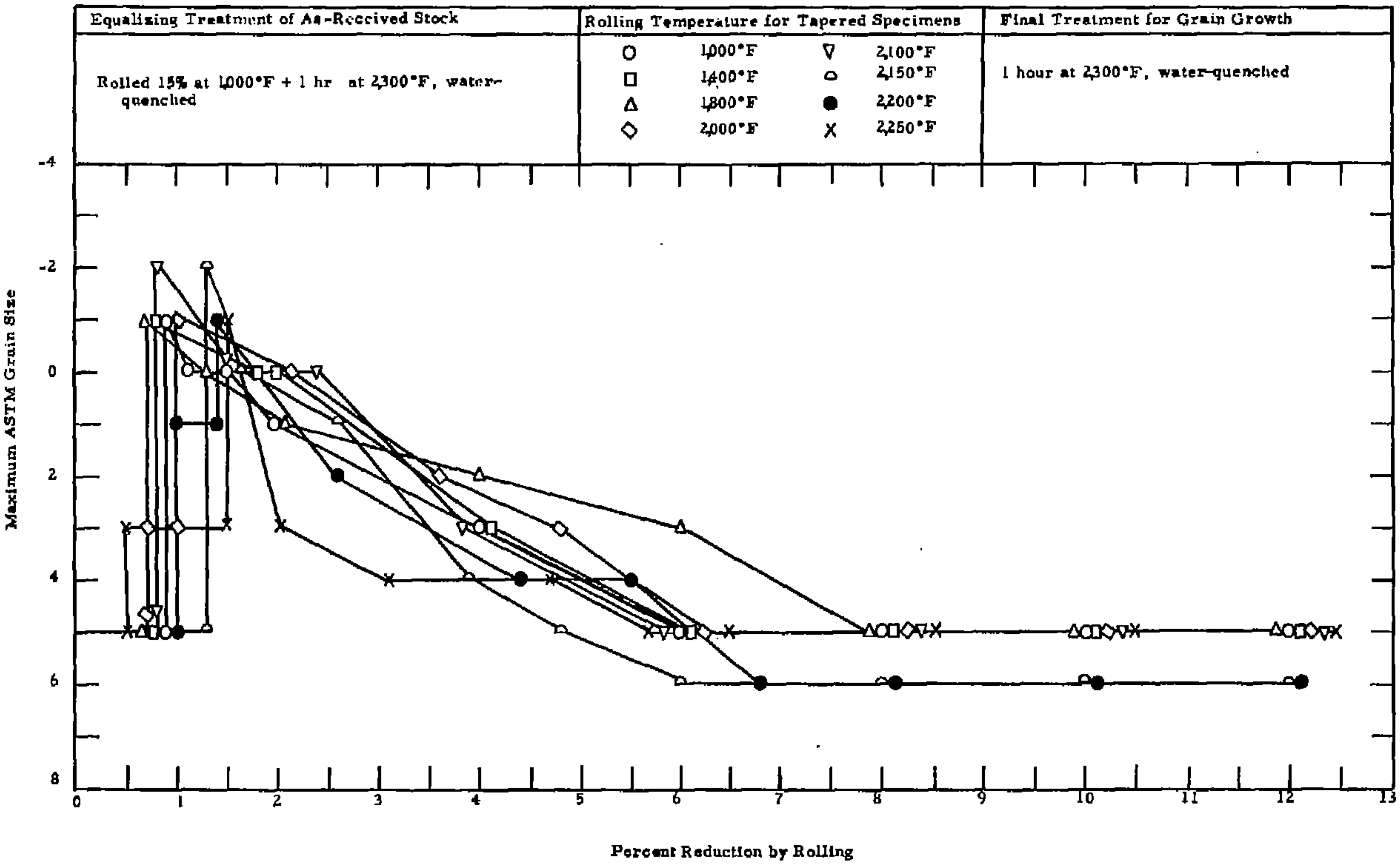
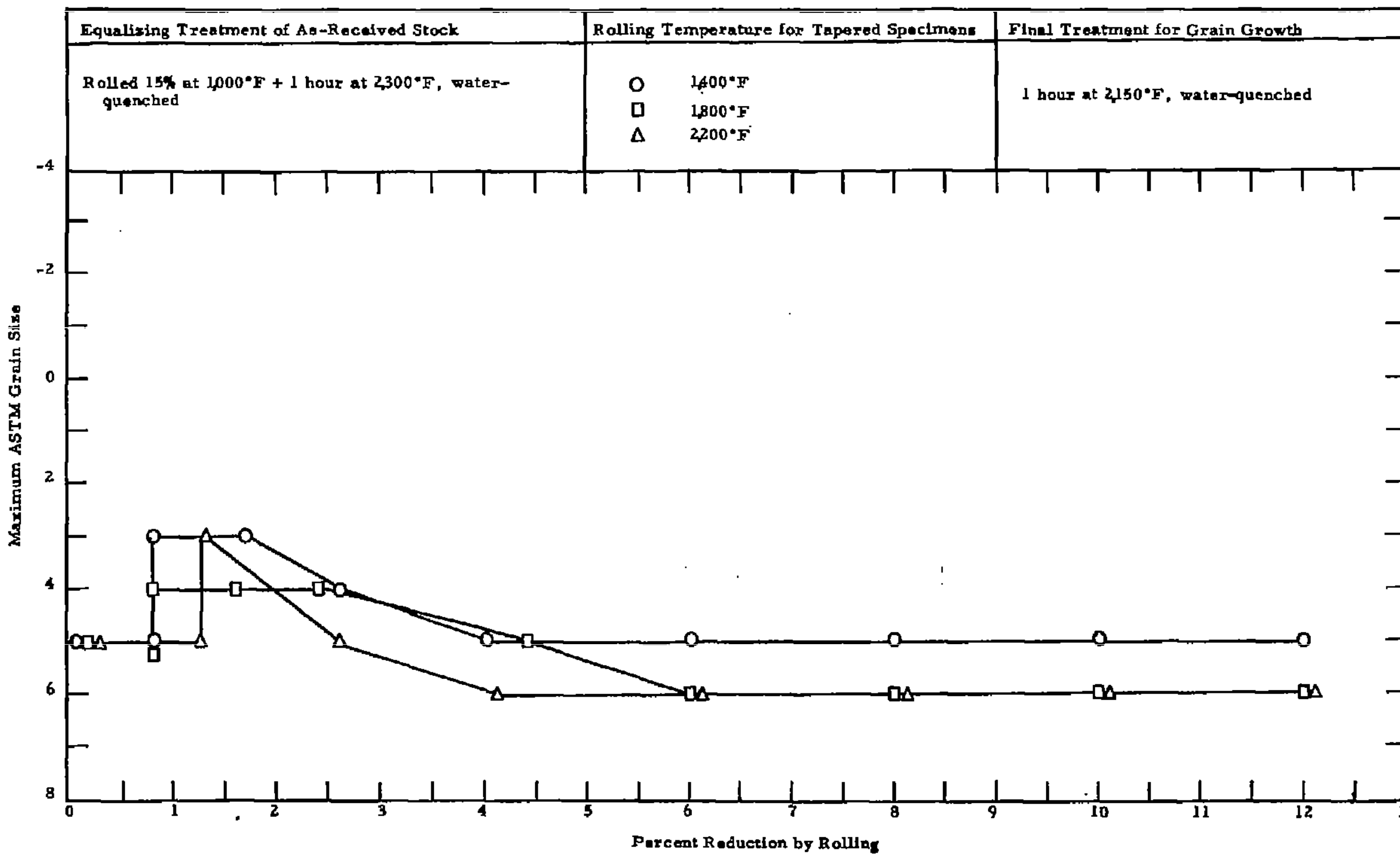
(d) 1 hour at 2,150° F, then water-quenched, plus one cycle of 4 hours at 2,150° F, then air-cooled.

Figure 14.- Effect of repeated heating and cooling upon microstructure of S-816 alloy which had been equalized by a 15-percent reduction at 1,400° F. (Transverse section at bar surface.) Magnification, X50.



(a) Final solution treatment at 2,300° F.

Figure 15.- Effect of rolling temperature and percent reduction upon maximum grain size of S-816 alloy after final solution treatment.



(b) Final solution treatment at 2,150° F.

Figure 15.- Concluded.

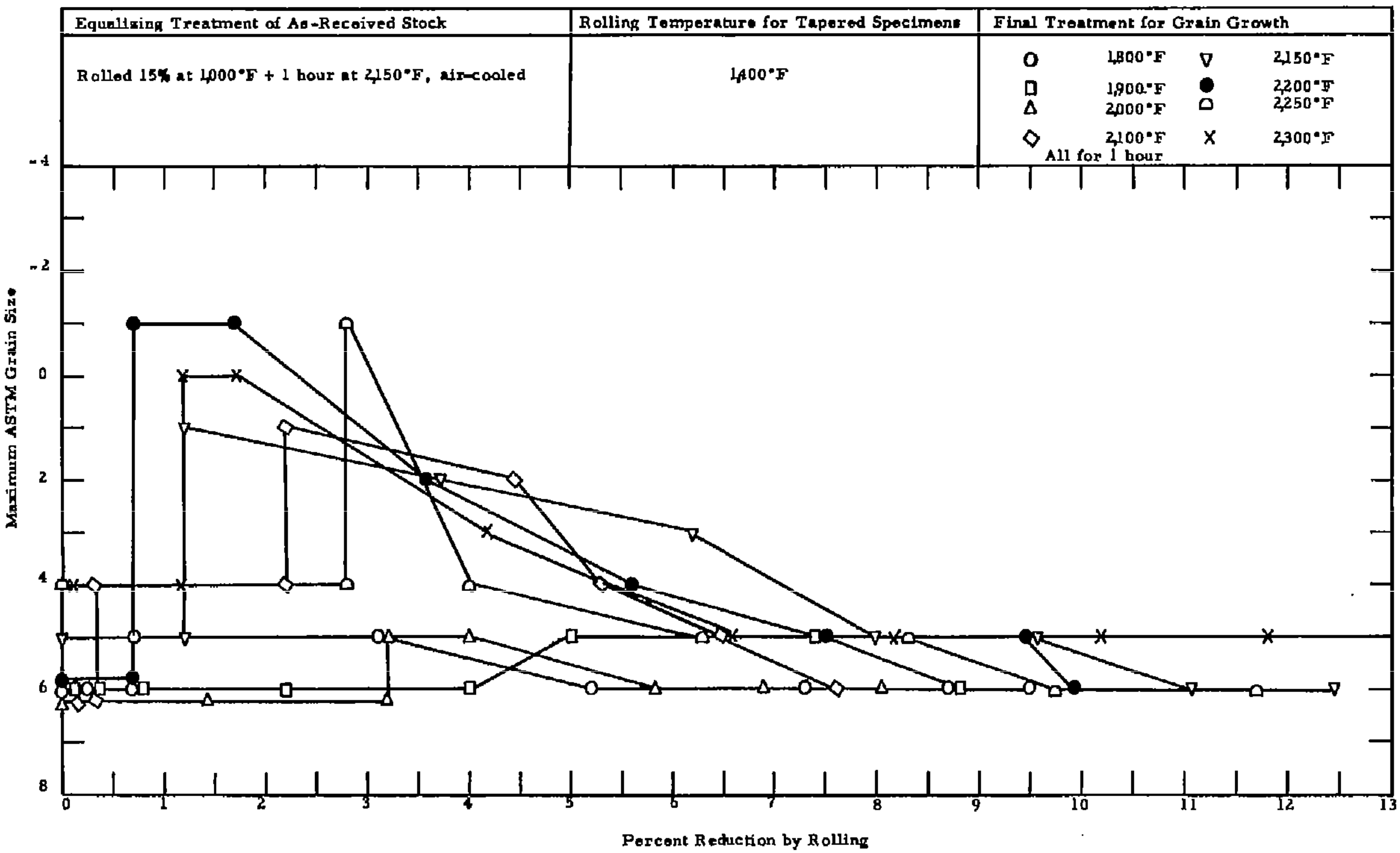


Figure 16.- Effect of percent reduction by rolling at 1,400° F and final solution treatment upon maximum grain size of S-816 alloy after final solution treatment.

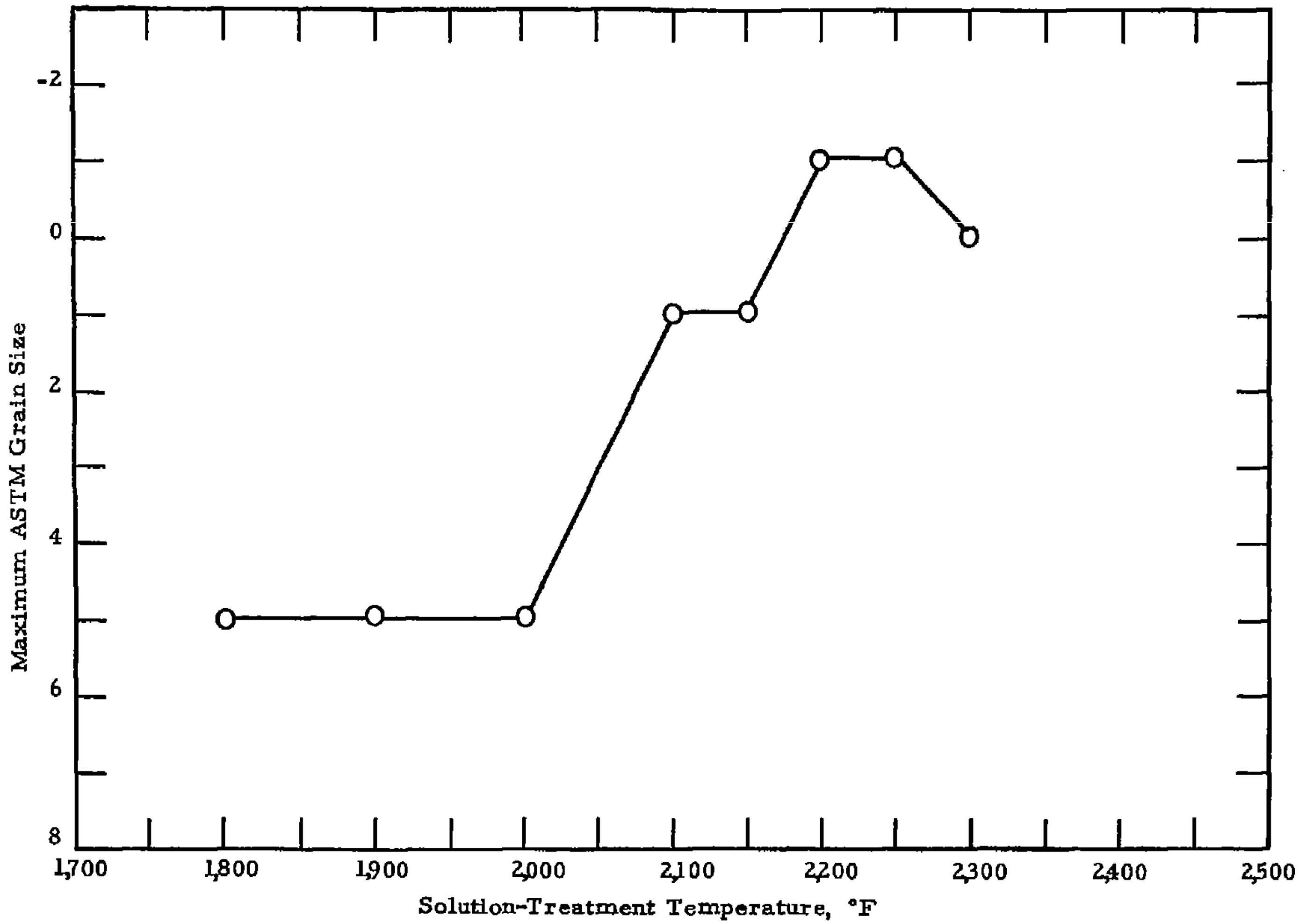
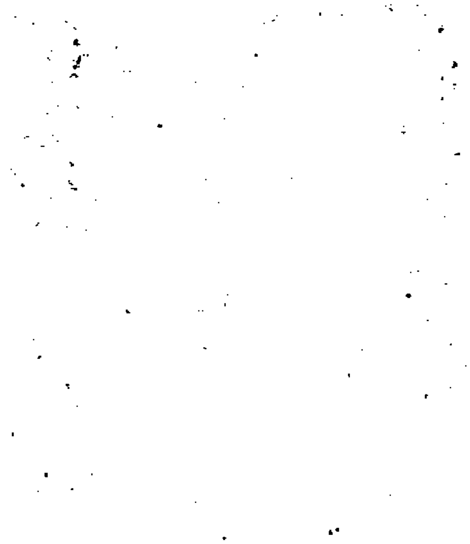


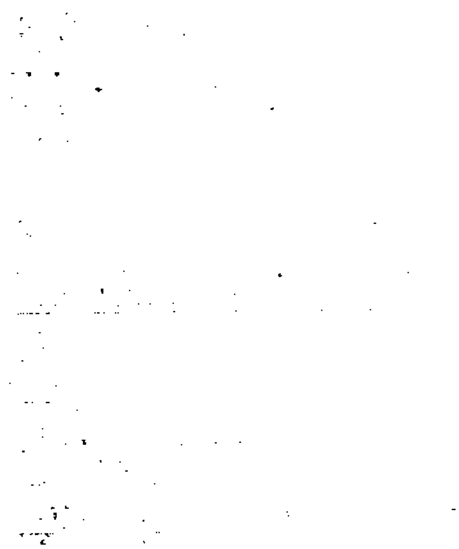
Figure 17.- Maximum grain size at critical reduction as a function of final solution-treating temperature for S-816 alloy. Equalizing treatment of as-received stock was a 15-percent reduction at 1,000° F plus 1 hour at 2,150° F, then air-cooled. All solution treatments were 1 hour.

(a) Zero-percent reduction.



(b) 0.7-percent reduction.

(c) 1.6-percent reduction.



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(d) 10.0-percent reduction.

Figure 18.- Effect of percent reduction by rolling at 1,400° F upon microstructure of equalized S-816 alloy after final solution treatment. Equalizing treatment of as-received stock was a 15-percent reduction at 1,000° F plus 1 hour at 2,150° F, then air-cooled. Final solution treatment was 1 hour at 2,200° F, then water-quenched. Magnification, X50.

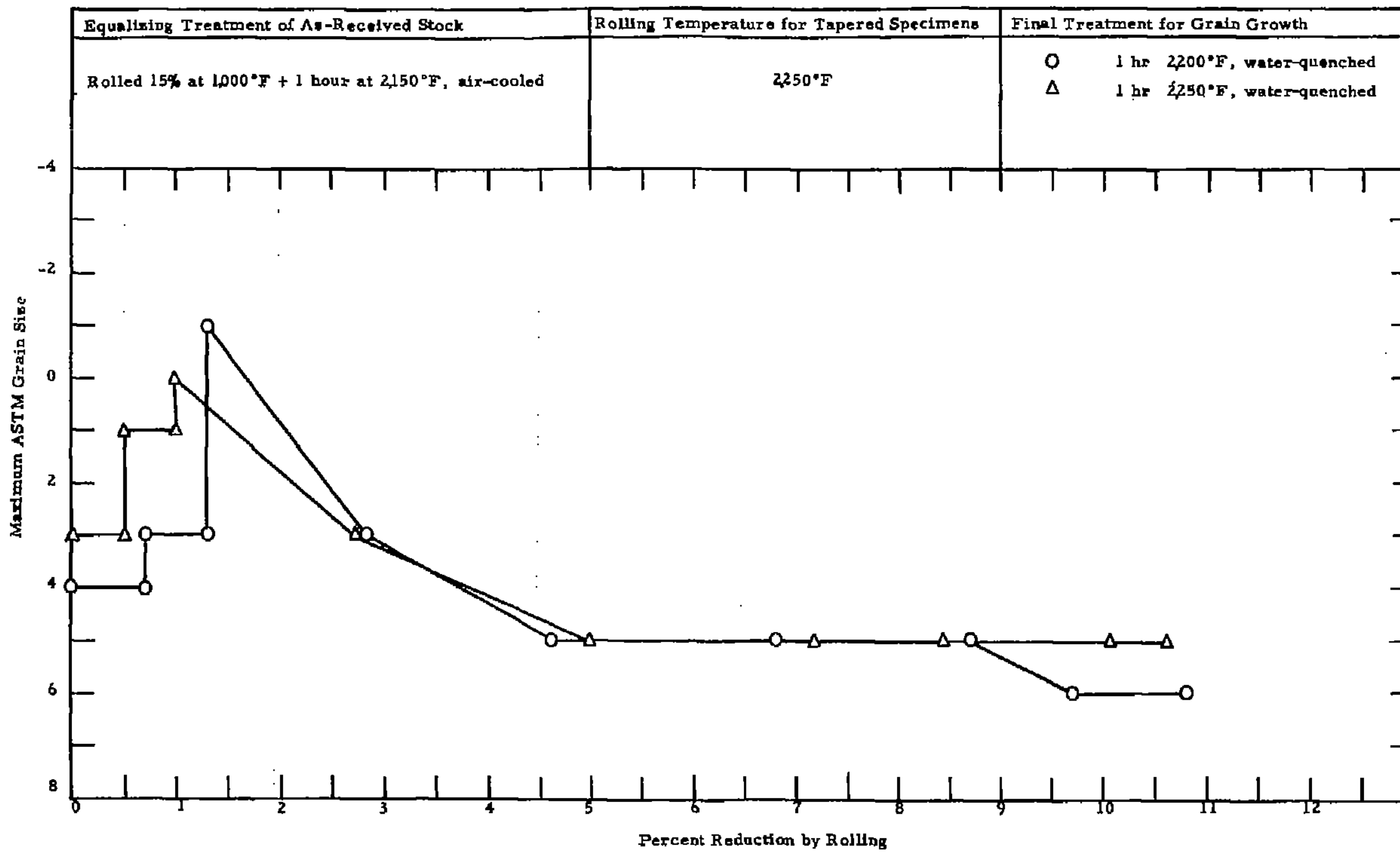


Figure 19.- Effect of percent reduction at 2,250° F with final solution treatment at 2,200° F and 2,250° F upon maximum grain size of S-816 alloy after final solution treatment.

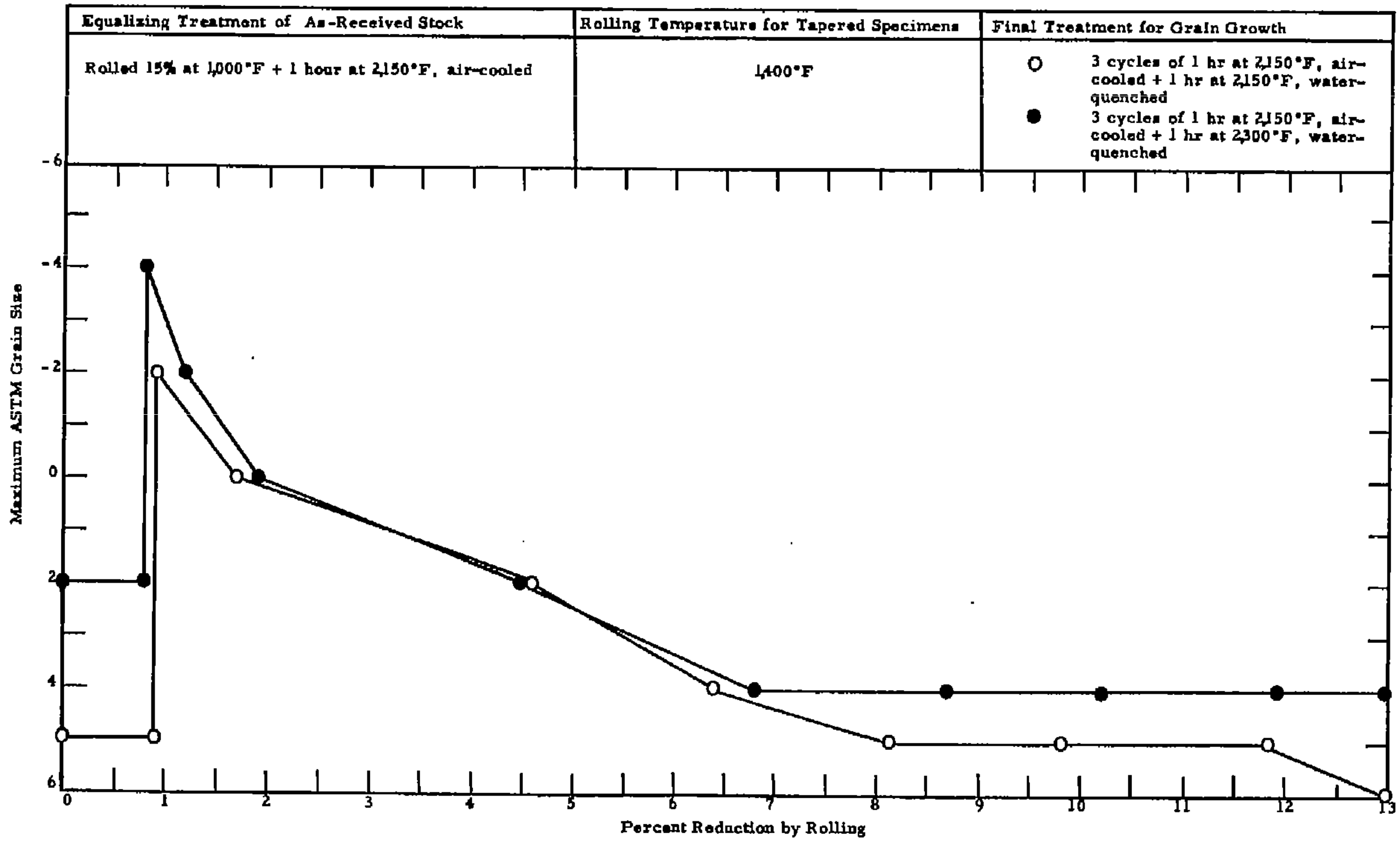


Figure 20.- Effect of percent reduction at 1,400° F and repeated solution treatment at 2,150° F and 2,300° F upon maximum grain size of S-816 alloy after final solution treatment.

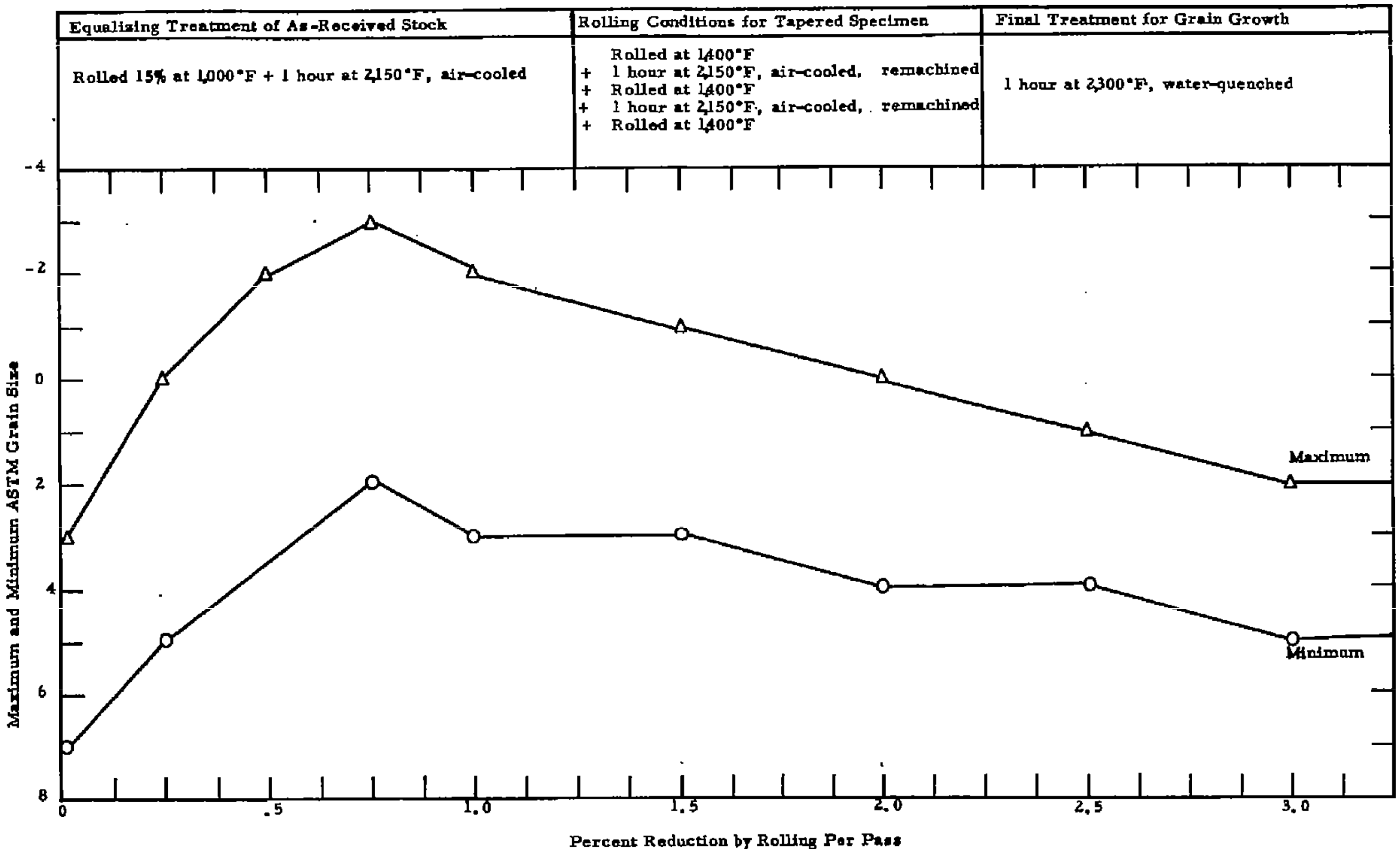


Figure 21.- Effect of repeated reduction by rolling with intermediate heat treatment upon grain size of S-816 alloy after final solution treatment.

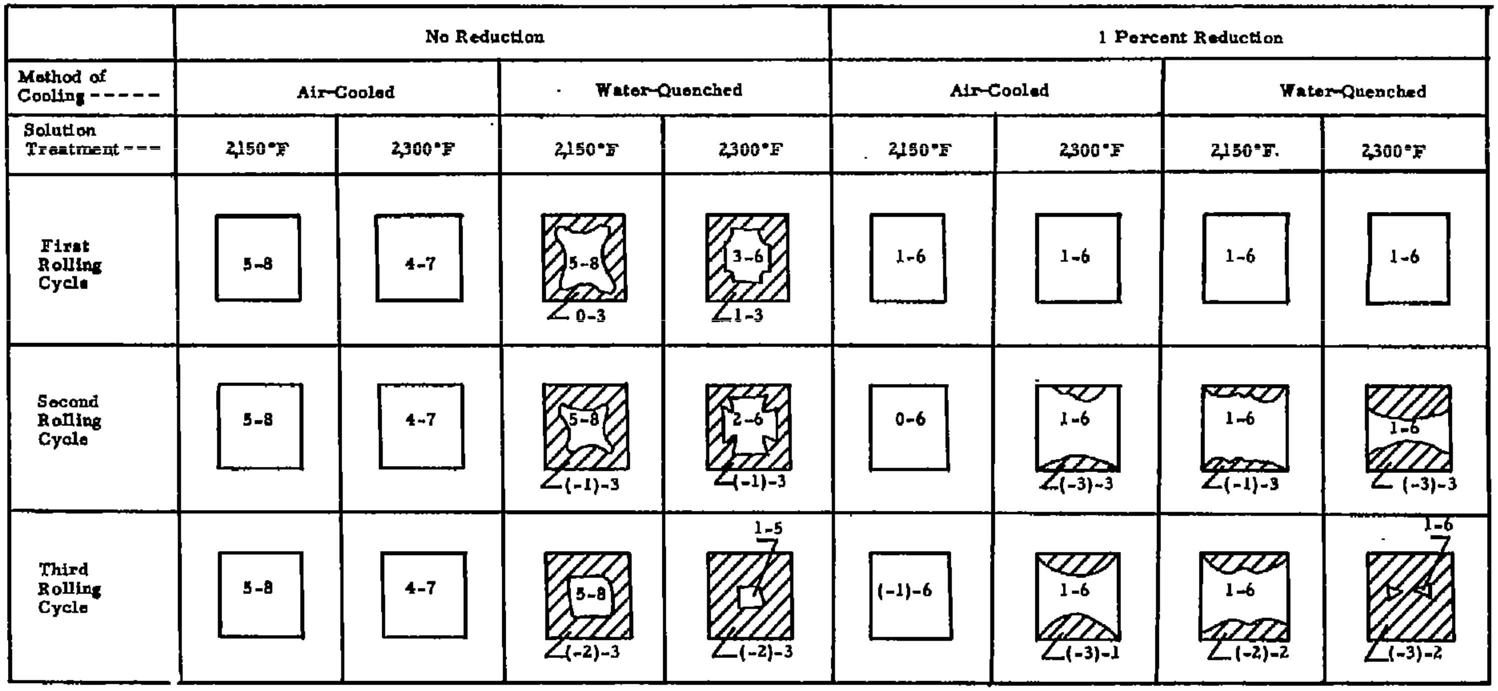
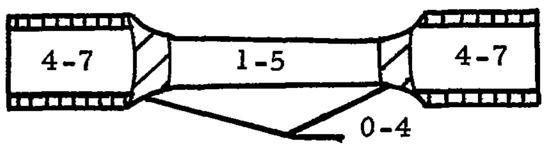
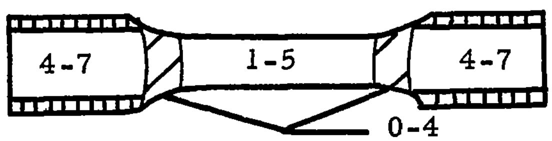
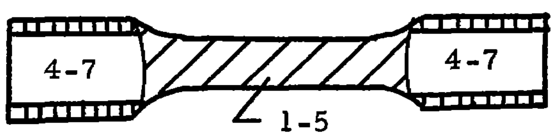


Figure 22.- Effect of cooling rate after rolling at 2,150° F on grain size of S-816 alloy after solution treatment at 2,150° and 2,300° F. (Equalizing treatment was 15-percent reduction at 1,000° F.)

Equalizing Treatment of As-Received Stock
15% Reduction at 1,000°F + 1 hour at 2,150°F, air-cooled

Tensile Test Tempera-ture, °F	Elongation, percent	Approximate Distribution of Grain Sizes After Final Solution Treatment
1,400	1.0	
1,600	0.8	
1,600	1.5	

Final Treatment for Grain Growth
1 hour at 2,300°F, water-quenched

Figure 23.- Grain-growth characteristics of S-816 alloy after critical deformation by tensile straining followed by final solution treatment.

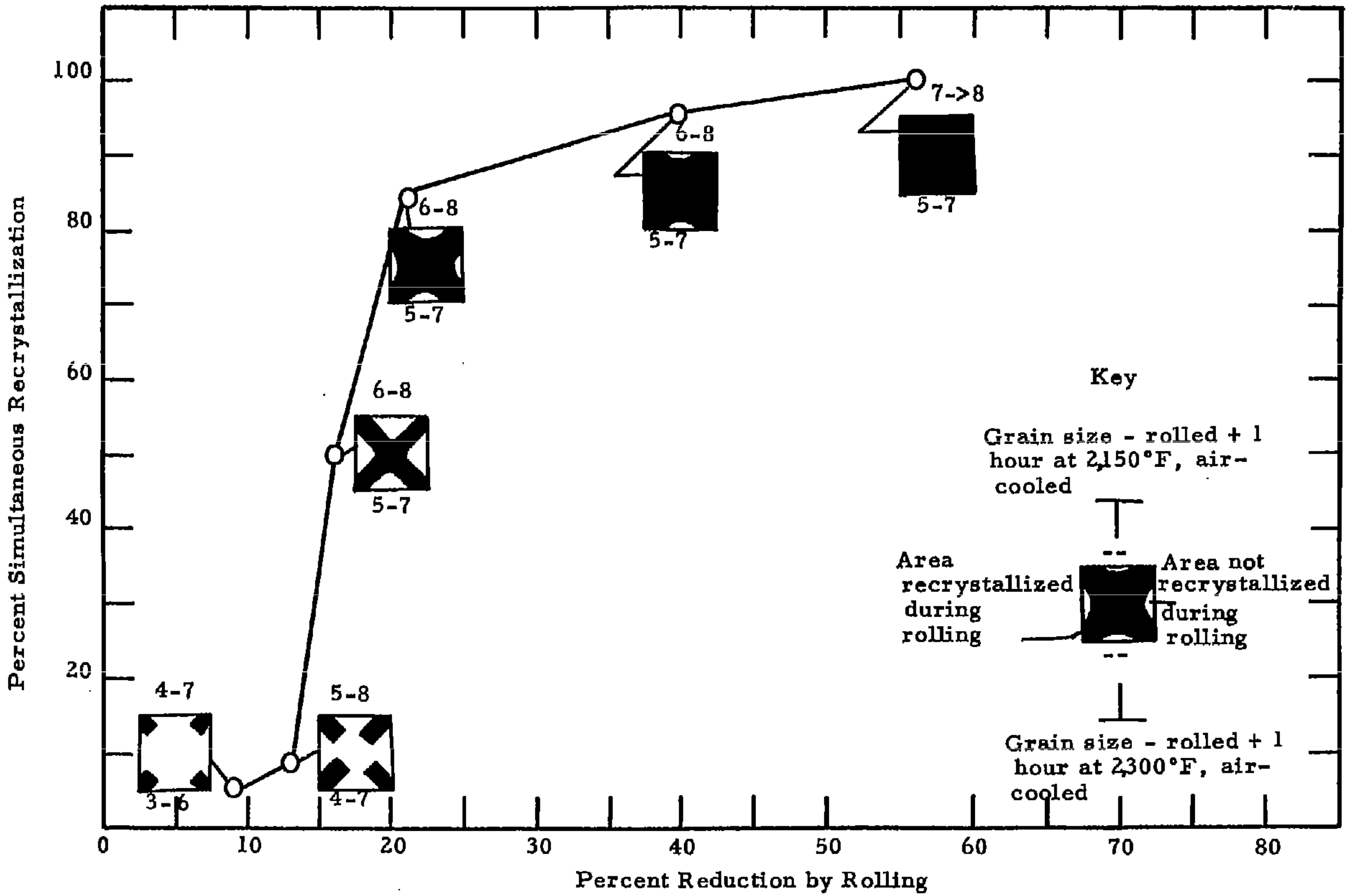


Figure 24.- Influence of amount of reduction at 2,150° F on grain size of S-816 alloy with an initial grain size of -2 to 5. Extent of recrystallization during rolling is shown as well as uniform grain size developed during subsequent solution treatment at 2,150° and 2,300° F. Initial grain size of -2 to 5 obtained by heating as-received stock 2 hours at 2,300° F.

NACA - Langley Field, Va.