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TECHNICAL NOTE 4082

ABNORMAL GRAIN GROWTH IN NICKEL-BASE
HEAT-RESISTANT ALLOYS

By R. F. Decker, A. I. Rush, A. G. Dano,
and J. W. Freeman

University of Michigan



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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 Waspaloy, Inconel X-550, and Nimonic 80A alloys. All of the results indicated that small reductions of essentially strain-free metal were the basic cause of abnormal grain growth. Between reductions of 0.4 and 5.0 percent, in most cases, there was a narrow range of reductions responsible for abnormal growth. 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 develop susceptibility. 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 also has relatively little 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.

The influence of alloy composition seems to be mainly in variation of the excess phases which restrict grain growth. A somewhat smaller grain size in vacuum-melted than in air-melted Waspaloy was apparently due to more grain-growth restrainers resulting from a higher carbon content. Inconel X-550 did not undergo abnormal grain growth at 1,950° F as did Waspaloy and Nimonic 80A alloys. At 2,150° F, the normal solution temperature for Inconel X-550, it did occur. Apparently the more stable columbium compounds in Inconel X-550 restrained grain growth to a higher temperature than the less stable growth restrainers in the other alloys.

INTRODUCTION

A study of the causes of abnormally large grains forming in nickel-base heat-resistant alloys during hot-working or subsequent final solution treatment was carried out. The alloys investigated were Waspaloy and Inconel X-550. Vacuum-melted Waspaloy, reputed to be less susceptible to grain growth, as well as air-melted material, was included in the investigation. A limited amount of data for Nimonic 80A alloy is included from another investigation (reported in a private communication). One previous report (ref. 1) presented preliminary results for a similar study of S-816 alloy.

The objective of the investigation was to establish the fundamental principles governing the formation of abnormally large grains during hot-working and final heat treatment in heat-resistant alloys of the types used in the gas turbines of jet engines. For purposes of this investigation, any grains larger than ASTM 1 were considered abnormally large. Furthermore, the investigation was mainly limited to normal conditions of heating for hot-working and for heat treatment, it having been well established that the abnormal grain growth of interest occurred under these conditions. However, a few experiments involving temperatures higher than normal were included.

The presence of abnormally large grains has been associated with poor properties in heat-resistant alloys, particularly with low fatigue strength and brittleness under creep-rupture conditions. The consequent necessary grain-size control is a recurring problem in making forgings and other hot-worked products from the heat-resistant alloys used in aircraft gas turbines. In practice, procedures for hot-working are eventually developed which eliminate or minimize grain-size problems. Those developed have generally been empirical and have not defined the basic principles involved. The data included in this report for Nimonic 80A alloy, for instance, represent experiments carried out to help clarify a production problem of grain-size control in an alloy which has been extensively used.

The general procedure of the investigation was to carry out controlled laboratory experiments on samples of bar stock to find conditions of heating and hot-working which resulted in abnormal grain growth. Like the investigation on S-816 alloy (ref. 1), this investigation did not disclose any conditions for abnormal grain growth other than by small amounts of critical deformation. The investigation does, however, define many conditions which lead to such critical deformation which are not so obvious as simple small amounts of deformation during hot-working.

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 procedures used to establish conditions leading to abnormal grain growth were as follows:

(1) Commercially produced bar stock was used for experimental materials. The only exceptions were small commercially produced ingots of vacuum-melted Waspaloy rolled to bar stock at the University of Michigan.

(2) Most of the as-received bar stock was not suitable for experimental research because of uneven grain growth or susceptibility to abnormal grain growth during reheating to hot-working or solution-treating temperatures. Accordingly, most stock was given an "equalizing treatment" designed to produce a uniform reasonably fine grained material for experimental purposes. This treatment usually consisted of:

- (a) A fairly heavy reduction by rolling
- (b) A heat treatment of 1 hour at the usual solution-treating temperature for the alloy

The cooling after the equalizing heat treatment was performed by either air-cooling or oil-quenching. Water-quenching made all of the alloys susceptible to abnormal grain growth on the surface of the bars. In some cases even air-cooled alloys developed some susceptibility to such growth. An equalizing heat treatment was, however, necessary. Otherwise, the initial uneven-grain-growth characteristics could mask the influence of the experimental conditions used to obtain abnormal grain growth.

The conditions of the equalizing treatment were sometimes deliberately varied, or the treatments omitted, to study the influence of such factors on abnormal-grain-growth characteristics.

(3) Repeated heating and cooling was used to study the abnormal grain growth induced by thermal stresses alone. Water-quenching, oil-quenching, and air-cooling were used to vary the cooling rate and resultant thermal stresses.

(4) The influence of amount and temperature of working was studied by rolling tapered specimens to flat bars between open rolls in a rolling mill. The tapered specimens were usually machined from the equalized stock. Two sizes of specimens (fig. 1) were used to give reductions ranging from 0 to about 15 or 29 percent. The specimens were placed in a furnace at the temperature selected for rolling and held in the furnace 1/2 hour before rolling. In most cases, only one pass through the rolls was used. The specimens were air-cooled from the rolling operation.

A few experiments were carried out using tensile specimens to obtain uniform reduction to study the comparative effects of uniform strain and the strain gradients from the tapered specimens.

(5) The rolled specimens were heated to the usual solution-treating temperatures for the usual times for grain growth to occur. In some cases, the specimens were cut into two pieces parallel to the direction of rolling before solution treatment. One half was examined in the as-rolled condition and the other, after solution treatment.

(6) The specimens were then carefully measured and the reduction in cross-sectional area computed. The specimens were sectioned and examined microscopically for grain size along the lengths of the rolled bars.

(7) The grain-size rating system used was that established by the American Society for Testing Materials (ref. 2). It was necessary to extend this system to larger sizes than number 0 through the notation -1 to -5 grain sizes. The actual grain sizes involved were as follows:

ASTM grain size	Grains/sq in. of image at 100 diam.	Approximate diam. 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 complete 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 bar stock commercially produced from air-melted heats, with the exception of the vacuum-melted Waspaloy. The information furnished by the suppliers of the test materials is given in the following sections.

Waspaloy Alloy

The air-melted Waspaloy alloy was supplied gratis by the Allegheny Ludlum Steel Corp. as 1-inch-square bar stock made from a 9-inch ingot from heat 43638. The vacuum-melted Waspaloy was supplied gratis by the Utica Drop Forge and Tool Corp. as a 2-inch-diameter ingot from heat 3-259. The chemical analyses supplied by the producers were as follows:

Chemical composition, weight percent												
C	Mn	Si	Cr	Ni	Co	Mo	Ti	Al	Fe	Cu	S	P
Heat 43638												
0.03	1.14	0.63	18.8	Balance	13.7	3.15	2.56	1.43	1.18	0.11	0.015	0.016
Heat 3-259												
0.08	0.27	0.57	19.8	Balance	13.8	3.90	3.17	1.15	0.69	<0.10	0.005	-----

The small ingots of vacuum-melted stock were hot-rolled at 1,950° F to 3/4- and 1/2-inch bars at the University of Michigan.

Inconel X-550 Alloy

The Inconel X-550 stock was furnished gratis by the International Nickel Co., Inc., as hot-rolled $2\frac{1}{4}$ - by $1\frac{1}{16}$ -inch flat bars from heat Y7180X. The only other information supplied was the following report of chemical composition:

Chemical composition, weight percent										
C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	Ta and Cb
0.05	0.73	6.59	0.007	0.28	0.03	72.63	14.97	1.16	2.50	1.03

Nimonic 80A Alloy

The Nimonic 80A alloy was in the form of 1-inch, hot-rolled, centerless, ground bar stock which had been purchased by the Continental Aviation and Engineering Corp. from commercial air-melted heat 5331B made by the International Nickel Co., Inc.

The following report of chemical composition was supplied:

Chemical composition, weight percent										
C	Mn	Fe	S	Si	Cu	Ni	Cr	Al	Ti	
0.05	0.55	0.59	0.007	0.34	0.05	Balance	20.5	0.98	2.20	

FACTORS INFLUENCING GRAIN GROWTH

A number of factors influenced observed grain-growth characteristics in the experimental materials. Because they 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 treatment 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-cooling or oil-quenching 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 variables 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, by air-cooling or oil-quenching this was kept below the critical amount required for abnormal grain growth.

(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, the following 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-cooling from the working temperature was involved.

(b) A zone covering reductions generally in the order of 0.4 to 5.0 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) A zone of still larger reductions where 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.

EXPERIMENTAL RESULTS

Grain-growth data were obtained for Waspaloy and Inconel X-550 alloys. In addition, data are reported for experiments on Nimonic 80A material from another investigation. The major experimental conditions studied were induction of abnormal grain growth by repeated heating and cooling and by deformation by rolling.

In the experiments involving rolling, tapered specimens were rolled to flat bars. 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 were also uniform on a macroscopic scale across the bar section. Recrystallization during working or after solution treatment, however, was often banded.

Air- and Vacuum-Melted Waspaloy Alloy

Grain-growth experiments were carried out on bar stock from both air- and vacuum-melted heats of Waspaloy alloy. A number of experiments were carried out on the bar stock to establish grain-growth characteristics and to develop initial treatments which would provide a reasonably uniform and fine initial grain size.

In the as-received condition, the air-melted stock was fine grained on the outside with a mixed grain size in the center (fig. 2). This material developed a nonuniform grain size when heated to 1,950° F

(fig. 3). Using temperatures higher than 1,950° F reduced the variation in grain size and did not cause large grains to form (fig. 3). It was considered, however, that it would be best to reduce the bar stock further by rolling before heat treatment in order to obtain a uniform fine grain size with the normal treatment at 1,950° F. A reduction of 70 percent from 1,950° F followed by a treatment of 1 hour at 1,950° F was applied to material used for the repeated heating and cooling experiments. This gave a grain size of 4 to 6 (fig. 3). A similar grain size was obtained by a reduction of 50 percent from 1,950° F (fig. 4) and this treatment was used for all the deformation experiments except when prior treatment was deliberately varied. Figure 4 shows that rolling at 1,950° F to a reduction of 50 percent resulted in partial recrystallization to very fine grains. However, this material had a uniform grain size of 4 to 6 after heating 1 hour at 1,950° F (fig. 4).

The vacuum-melted stock as originally rolled had a grain size of 5 to 8 (fig. 5). Heating to 1,950° F for 1 hour gave a grain size of 4 to 7 (fig. 5). The latter condition was used for all of the grain-growth experiments.

Induction of abnormal grain growth by repeated heating and cooling.-
 The experiments conducted to induce grain growth by repeated heating and cooling and the resulting grain sizes are summarized by figures 6 to 9. The observed grain-growth characteristics were:

(1) Air-cooling did not induce abnormal grain growth in air-melted stock. There was a gradual increase in grain size during four reheats so that the final grain size was 2 to 4 with few random 0 grains (fig. 6). Vacuum-melted stock was also free from abnormal grain growth as a result of repeated heating and air-cooling. The normal grain growth was less than for the air-melted stock, final grain size being 3 to 6 (fig. 8).

It should be noted that one 4-hour cycle gave nearly the same grain sizes as four cycles of 1-hour duration (figs. 6 and 8).

(2) Water-quenching between reheats did induce abnormal grain growth starting at the surface in both air- and vacuum-melted stock. The air-melted stock developed larger grains and a larger percentage of abnormal grains (figs. 6 and 8). Figures 7 and 9 show typical microstructures of the water-quenched stock.

Again it should be noted that a 4-hour reheat to 1,950° F developed just about as much abnormal grain growth as four cycles of 1 hour at 1,950° F (fig. 8).

(3) Air-melted material initially water-quenched from 1,950° F but air-cooled during four subsequent cycles to 1,950° F underwent nearly

the same abnormal grain growth as material water-quenched during each cycle. The initial water quench appeared to be the critical factor controlling abnormal grain growth (fig. 6)

(4) Oil-quenching did not induce significant abnormal grain growth in air-melted stock. The largest grains formed were of size 0 (fig. 6).

The experiments indicate that:

(1) For the sizes and shapes studied, air-cooling or oil-quenching did not induce abnormal grain growth during subsequent reheating. Water-quenching did induce abnormal grain growth in both air- and vacuum-melted materials during subsequent reheating.

(2) The governing factor in the abnormal grain growth was time of heating at the solution temperature and not the number of times the materials were reheated and quenched. One water quench was just as effective as four as long as the total time of heating was the same.

(3) The vacuum-melted material did not develop quite so large grains as did the air-melted stock.

Induction of abnormal grain growth by deformation.- All of the experiments on induction of abnormal grain growth by deformation were designed to establish the conditions which lead to abnormal grain growth during a standard final solution treatment of 4 hours at 1,950° F. Therefore, all grain-size ratings are based on material which had been solution-treated after subjection to various initial treatments possibly influencing grain growth.

The main result was that grains larger than 1 were induced in material which had been reduced between 0.4 and 5.0 percent. A limited number of special conditions resulted in abnormal grains when reductions were as small as 0.1 percent or as large as 9.7 percent. In these limited cases, abnormal grain growth did not occur over this entire range of reductions but rather did occur over some narrow reduction within this range.

There was a very sharp increase to grain sizes of -3 to -4 at the lower side of this range in reductions, usually for reductions between 0.4 and 1 percent. The grain size then diminished so that for most cases when the reduction was 5 percent, the maximum grain size was 1 or less.

The following additional features of the results can also be generalized:

(1) The range of reduction for abnormal grain growth was independent of temperature of reduction.

(2) Vacuum-melted stock underwent abnormal grain growth after the small critical reductions in the same manner as the air-melted stock. The maximum grain size was, however, less for the vacuum-melted material, the usual differential being about two sizes smaller for the vacuum-melted material.

(3) Working above the solution temperature of $1,950^{\circ}$ F generally reduced maximum grain size in the area of abnormal grain growth in air-melted stock.

(4) A number of conditions of working and heat treatment prior to critical reductions were found to have little effect on the tendency for abnormal grain growth.

(5) Uniform reductions more than the critical amount prior to a critical reduction did not completely suppress the abnormal grain growth.

(6) Uniform critical reduction in a tensile machine also induced abnormal grain growth.

(7) A very steep strain gradient from working tended to suppress maximum grain size, apparently by restricting the amount of metal subject to abnormal grain growth.

(8) A limited number of experiments were not successful in inducing abnormal grain growth as a result of partial recrystallization during working.

The details of the data which led to these summarized results are discussed as follows:

Effect of amount and temperature of reduction: Abnormal grain growth was induced at some small reduction in all specimens of air-melted stock regardless of the temperature of rolling (see table I and fig. 10). The reductions inducing this grain growth were between 0.7 and 3.0 percent. The maximum grain size in this region of reductions was -3 to -4 except for rolling at $2,000^{\circ}$ and $2,100^{\circ}$ F when it was -2. The grain size was less than 1 for all reductions larger than 1.8 to 5 percent depending on the rolling temperature. Typical microstructures for various reductions taken along a tapered specimen are shown in figure 11.

The rolling temperature had very little effect on that portion of the specimens which was not reduced, except when the rolling temperature was 80° or $2,100^{\circ}$ F. The maximum grain sizes shown by figure 10 were remarkably similar for reductions larger than the critical amount for all temperatures of rolling. Apparently, the varying degrees of recrystallization during rolling at $1,800^{\circ}$ to $2,100^{\circ}$ F did not greatly alter the

final grain size from that induced by strain-hardening at lower temperatures of rolling.

Vacuum-melted stock responded similarly to the air-melted stock (table II and fig. 12) except that the maximum grain size was -2. The overall grain size was also finer. There also was no difference in maximum grain size in the critically reduced section between samples rolled at 2,100° F and those rolled at lower temperatures. Typical microstructures of vacuum-melted stock are shown in figure 13.

It will be noted that the change in grain size in the critical section was the same for both air- and vacuum-melted material. Although this suggests change in grain size as a controlling factor in grain growth from critical reduction, it was not borne out by subsequent studies.

Influence of prior history on abnormal grain growth: A number of details in the treatments prior to rolling as tapered specimens were varied. The more important results were:

(1) Omission of the heat treatment at 1,950° F after a reduction of 50 percent at 1,950° F did not completely eliminate the susceptibility to abnormal grain growth from critical reduction although it greatly reduced maximum grain size (table I and fig. 14). This was true for specimens rolled at both room temperature and 1,600° F. The only difference for the two cases was the rather high reduction of 6 to 10 percent for abnormal growth when the specimen was rolled at 1,600° F. It had been expected that there would be no tendency for abnormal growth from critical reduction after this heavy initial reduction.

The results of the preceding discussion show that a reduction of more than 5 percent at any temperature usually restricted the grain size to less than 1. It was presumed that superimposing any further amount of reduction would not alter the tendency to produce fine grains during solution treatment. Data presented later for specimens strained uniformly in tension tend to show that the amount of deformation and not strain gradients is the controlling factor in critical deformation for abnormal grain growth. This then suggests that the superimposing of a strain gradient on material strained past the critical amount was not responsible for retention of some tendency for abnormal grain growth.

It is important to recognize in considering these possibilities that, as reduced 50 percent at 1,950° F, the metal was not susceptible to abnormal grain growth. It does, however, seem apparent that further reduction at some lower temperature can induce abnormal grain growth. It is highly probable that this susceptibility arises from partial simultaneous recrystallization leaving areas of essentially strain-free material which can subsequently be critically strained.

(2) Air-cooling from the 1,950° F treatment instead of oil-quenching did not substantially alter the maximum grain growth caused by critical reductions at 80° F (fig. 14) or 1,900° F (fig. 15). Grain growth was, however, less for the air-cooled material when it was rolled at 2,100° F (fig. 15) for reasons which do not seem explainable from the available information.

(3) It was noted that the grain growth after solution treatment was considerably greater in that part of the tapered specimens which received no reduction when the rolling was carried out at room temperature (figs. 12 and 14). Apparently, the 1/2 hour of heating for rolling at 1,400° F or higher restricted general grain growth during the final solution treatment in material which did not receive any further reduction.

(4) The inclusion or omission of the equalizing heat treatment at 1,950° F before rolling at 2,100° F had practically no effect on grain growth (see table I and fig. 16). Heating first at 2,100° F and then dropping the temperature to 1,600° F may have increased the amount of reduction to initiate abnormal grain growth from 0.7 to 2.5 percent.

These results suggest that the tendency for the critical reduction to increase for rolling at 2,100° F is due to heating to 2,100° F and not to working at that temperature. This was carried over in the specimen cooled to 1,600° F before rolling.

(5) As-received material reduced 25 percent at 1,600° or 1,950° F had about the same growth characteristics for maximum grain size (fig. 17) as material reduced 50 percent at 1,950° F when all were equalized at 1,950° F and oil-quenched prior to rolling at 1,600° F. This result is support for the general conclusion that prior history has relatively little effect on abnormal grain growth unless there is a large reduction without opportunity for substantial recrystallization.

Abnormal grain growth induced by tensile straining: Uniform critical reduction by limited straining in a tensile machine was used to obtain an indication as to the relative importance of the amount of reduction and a strain gradient. Specimens were stretched: (1) 1 percent at 1,400° F; (2) 1 percent at 1,600° F; and (3) 2.5 percent at 1,600° F. The grain sizes after subsequent solution treatment are given in figure 18.

The results of these tests show:

(1) The 1-percent elongation at 1,400° F developed a maximum grain size of only -1.

(2) The 1-percent elongation at 1,600° F gave very nearly the same result as 1-percent reduction by rolling of tapered specimens, the maximum grain size being -4.

(3) An elongation of 2.5 percent at 1,600° F strained the gage section more than the critical amount so that abnormal grain growth was restricted to the fillets where the strain was the smaller critical amount.

These data are interpreted to show that the critical strain is the controlling factor and not the strain gradient. The absence of appreciable grain growth after straining 1 percent at 1,400° F apparently was due to the strain being below the minimum amount required.

Influence of recrystallization during working: The apparent confinement of induction of abnormal grain growth to a small critical reduction raised questions as to whether the same condition could be attained by partial recrystallization during working. Since recrystallization leaves a relatively strain-free condition, there must be strain gradients between the recrystallized and the unrecrystallized zones.

The method of study selected was to roll tapered specimens so as to obtain reductions from 0 to about 29 percent. This would provide a wider range of recrystallization than was obtained in the bars reduced a maximum of 15 percent. Temperatures of 1,850°, 1,950°, and 2,050° F were used to vary further the recrystallization characteristics during rolling. The grain sizes obtained (table I and fig. 19) along with typical microstructures (fig. 20) indicate the following things:

(1) The reduction for abnormal grain growth remained approximately the same as that which had previously been found, 0.4 to 5.8 percent. The maximum grain size, however, was -1 to -2 instead of -3 to -4. The narrower zone of critical reduction in the bars with the greater taper apparently restricted the maximum grain size. This seemed to be due to an insufficient volume of metal being critically deformed to provide enough material for larger grains.

(2) No abnormal grains were found in the regions reduced more than the critical amount in spite of a wide range in degree of recrystallization during rolling.

(3) The material reduced more than 10 percent at 1,850° F did show grains having a size of 1 to 2 in bands between finer grained areas (fig. 20(c)). This appeared to be due to grain-boundary migration from the few very small recrystallized grains which formed during rolling. These apparently grew preferentially at the expense of surrounding grains.

(4) The material reduced over 7 percent at 1,950° F underwent extensive partial simultaneous recrystallization (figs. 20(d), 20(e), 20(f), and 22). However, upon subsequent final solution treatment, a uniform

fine-grained structure was obtained in the regions of the tapered specimen which received the heavier reductions (figs. 20(d), 20(e), and 20(f)).

These specimens were solution-treated at 1,975° F in accordance with more recent commercial practice. There is no reason to believe that this increase from 1,950° F appreciably affected the abnormal-grain-growth characteristics.

The investigation of the possible induction of abnormal grain growth through partial recrystallization was too limited to allow definite conclusions. The results, however, point to certain probable fundamentals which suggest that it would be very difficult to induce abnormal grain growth in this manner. Any strain gradients between recrystallized and unrecrystallized areas would be very steep. As discussed in the previous section, this would probably limit the maximum grain size because of the small amount of available metal subject to abnormal grain growth. In addition, there is good reason to believe that the unrecrystallized grains adjacent to recrystallized grains are deformed more than the critical amount for abnormal grain growth. This would result in a very narrow zone or even the absence of critical deformation with little or no tendency for abnormal grain growth.

Influence of rate of heating on abnormal grain growth: The occurrence of grains with a size of 1 to 2 in the sample rolled at 1,850° F to reductions of 10 to 20 percent, as described in the preceding section, suggested the possibility of abnormal grain growth from a few small simultaneously recrystallized grains (figs. 20(a), 20(b), and 20(c)). If a slow rate of heating was used, the few very small grains which formed during rolling at 1,850° F (figs. 20(a) and 20(b)) might have an opportunity to grow even larger. Very large grains can be grown in metals when only a few small grains form by recrystallization and are given time enough to grow at a relatively low temperature to large grains at the expense of the surrounding strained metal.

Accordingly, a sample was prepared and taper-rolled to include a considerable region of reduction between 10 and 20 percent. When it was heated from 1,400° to 1,950° F in 3 hours, the grain sizes found (table I and fig. 19) were no different from those found when it was placed in a furnace at the maximum temperature.

As far as could be ascertained, the slow rate of heating had little effect on the abnormal grain growth at the critically deformed section. It certainly did not increase grain size in this area.

It will be noted, however, that the critical reduction for abnormal grain growth was only 0.1 percent. This low value suggests that the critical reduction for abnormal grain growth is sensitive to heating rate.

If so, unrecognized variations in heating rate could account for some of the apparently inexplicable variations in the minimum reduction for abnormal grain growth observed throughout the investigation.

Degree of recrystallization during working: During the investigation, estimates were made of the amount of recrystallization in as-rolled structures. These are summarized in figure 21. For some reason, the larger reductions gave more recrystallization at 1,950° than at 2,050° F. A very small amount occurred at 1,850° F and none at lower temperatures.

Inconel X-550 Alloy

A number of heat treatments were carried out on the as-received Inconel X-550 bar stock to establish initial grain-growth characteristics. It was subject to uneven grain growth at 1,900° and 2,000° F and to abnormal grain growth at 2,100° F (see fig. 22). The tendency for abnormal grain growth was reduced at 2,200° F. Typical microstructures are shown in figure 23.

Induction of abnormal grain growth by repeated heating and cooling.- The Inconel X-550 stock was reduced 64 percent from 2,150° F and then reheated as shown in figure 24. Four and five cycles to 2,150° F with air-cooling did induce some abnormal grain growth. This growth occurred during the first reheat after an initial water quench and became more extensive during succeeding cycles (fig. 25).

The Inconel X-550 material was quite sensitive to abnormal grain growth from the surface if water-quenched and reheated to 2,150° F. It was far less sensitive when air-cooled. However, repeated air-cooling or, more probably, increased heating time at 2,150° F resulted in some abnormal grain growth. This, together with the experiments carried out on the as-received stock, indicates that abnormal grain growth can occur in 1 to 4 hours of heating at temperatures above 2,000° and below 2,200° F for either air-cooled or water-quenched material.

Apparently Inconel X-550 was somewhat more susceptible to abnormal grain growth from repeated heating and cooling than Waspaloy. At least, abnormal grain growth was not induced in the latter material by repeated air-cooling from its solution temperature.

Induction of abnormal grain growth in Inconel X-550 alloy by deformation.- As-received Inconel X-550 stock was heated for 2 hours at 1,900° F, air-cooled, and machined into tapered specimens having a uniform grain size of 7 to 8. Abnormal grain growth occurred during final solution treatment at 2,150° F in regions reduced 2.6 to 10.5 percent during rolling at 1,600°, 1,800°, and 2,000° F (table III and fig. 26(a)). There was no abnormal grain growth after rolling at 2,200° F. Increasing the temperature of rolling from 1,800° to 2,000° F reduced the range

of reductions subject to grain growth. It will also be noted that there was no tendency for abnormal grain growth in the section of the specimens which received no reduction. Evidently the heat treatment at 1,900° F or the removal of surface metal in machining the specimens eliminated the susceptibility to abnormal grain growth at 2,150° F originally present in the stock.

Tapered specimens were prepared from stock solution-treated 2 hours at 2,100° F. The machining removed the surface material which underwent abnormal grain growth during treatment at 2,100° F and left material with a uniform grain size of 0 to 5.

The critical reduction for abnormal grain growth in the Inconel X-550 material was between 0.4 and 1.4 percent. Again, rolling at 2,200° F practically eliminated abnormal grain growth. The tendency was also slightly reduced by rolling at 2,000° F in comparison with rolling at 1,600° or 1,800° F (fig. 26(b)).

Some stock was reduced 50 percent at 1,950° F and then machined into tapered specimens. When heated to 2,100° F for 0.5 hour and rolled, it was subject to abnormal grain growth for a reduction of 4 to 7.8 percent (fig. 26(c)). Figure 27 shows typical microstructures of these specimens. When the specimen was cooled to 1,600° F before rolling, the range of reduction for abnormal grain growth was somewhat higher. This latter material also developed grains as large as -1 when reduction was very small.

Apparently the recrystallization during working at 1,950° F plus that on heating to 2,100° F changed the amount of reduction for critical grain growth. Possibly recrystallization and grain growth during heating at 2,100° F for rolling left less residual strain from prior history and therefore required more deformation to induce critical strain. Possibly grain-size differences were involved in the change in critical deformation.

Nimonic 80A Alloy

The as-received Nimonic 80A stock had a grain size of 6 to 8. Figure 28 shows the influence of various heating conditions on grain growth in this material. Heating for 4 hours at 1,950° F developed a grain size of 1 to 3. Higher temperatures resulted in larger grains, including abnormal grains.

Experiments involving various conditions of rolling.- Experiments were carried out involving various conditions of rolling. Both as-received stock and stock reduced 50 percent at 1,950° F followed by a 1-hour treatment at 1,950° F were utilized. The original grain structure

for both materials is shown by figure 29. It will be noted that a treatment of 1 hour at 1,950° F resulted in a structure of fairly coarse grains with bands of very fine grains.

Induction of abnormal grain growth by deformation.- In general the Nimonic 80A stock underwent abnormal grain growth in the same manner as the other alloys. One outstanding difference was the tendency for grain sizes of 1 to 0 to develop after large reductions.

Effect of temperature and amount of reduction: Material which had been reduced 50 percent at 1,950° F and then reheated for 1 hour at 1,950° F and air-cooled and prepared as tapered specimens and rolled at 1,750°, 1,850°, and 1,950° F and reheated under the conditions outlined in table IV and figure 30. Abnormal grain growth to sizes greater than 0 took place for reductions between 0.1 and 8.5 percent when the specimens were reheated by being placed in a furnace at 1,975° F. The larger grain size was never smaller than 3 and usually was 1 or 0 for the larger reductions. So far as the effect of temperature of reduction was concerned, there were only minor variations of doubtful significance in the maximum size of the grains formed by abnormal growth.

Reworking after prior deformation: A specimen equalized by a 50-percent reduction at 1,950° F followed by a 1-hour treatment at 1,950° F was rolled once as a tapered specimen, reheated to 1,950° F for 10 minutes, and again passed through the rolls. Because of springback of the rolls the second pass imposed about 1-percent additional reduction on the specimen. The observed grain sizes after final solution treatment are given in table IV and figure 30. It is important to recognize that the reductions shown are the combined reduction from the two passes. The observed grain sizes are interpreted as follows:

(1) During the 10-minute reheat to 1,950° F, considerable relief of strain from the original reduction took place. At small total reductions, the combined effect of the relatively small further reduction from the second pass and the initial reduction did not become effective for abnormal grain growth until the point where the total reduction was 1.6 percent.

(2) For all total reductions between about 8 percent and about 23 percent, the strain relief from the 10-minute reheat was probably insufficient to bring the residual strain below the critical amount. Therefore, the second pass did not induce abnormal grain growth.

(3) For total reductions between 23 and 28 percent, extensive recrystallization occurred during the original pass. This recrystallization probably left the material essentially strain free. When given the second pass it probably was then critically deformed and became susceptible to the observed abnormal grain growth.

Another specimen with less taper was made from as-received stock and subjected to the same sequence of operations (see table IV and fig. 31). The behavior was very similar to that of the previously discussed specimen over the comparable range of total reductions. This second specimen did develop considerably coarser grains in the regions where there was more than a critical reduction than a similar specimen given only one pass. The grain size in this region was similar to that of the first rerolled specimen discussed. In fact, it was similar to that of all the specimens first equalized at 1,950° F. This suggests that the common factor of two heatings to 1,950° F was responsible for the relatively large grain size for a more than critical reduction.

The most important features of the results of these experiments are:

- (1) Evidence of considerable strain relief in 10 minutes at 1,950° F without a cool to room temperature
- (2) Further indication that extensive recrystallization during working leaves alloys susceptible to abnormal grain growth from small additional deformation
- (3) Evidence that repeated heating to 1,950° F increases grain size to near abnormally large sizes for metal not critically reduced

From a practical viewpoint, the changes in critical reduction were too small to be significant.

Effect of heating rate on abnormal grain growth: Reducing the heating rate to solution temperature after rolling at 1,850° F (fig. 30) practically eliminated abnormal grain growth and restricted grain size for larger reductions. Bringing the specimen up slowly in the furnace from 1,400° to 1,950° F in comparison with placing the specimen in a furnace at 1,950° F could be expected to give considerable strain relief before the temperature was high enough for grain growth. The opportunity for precipitation and agglomeration at the lower temperatures may also have increased grain-growth restrainers. Both factors would favor the observed reduction in grain growth.

Slow-heating material from 1,950° to 2,300° F after reduction at 1,950° F increased grain size for all reductions (fig. 32) when compared with material rapidly heated to 2,300° F. In this case, grain growth could occur at all temperatures involved and the increased grain size was probably due to increased time in the grain-growth range.

Influence of temperature of solution treatment after reduction at 1,950° F: Several conditions of heating were used after reduction of tapered specimens at 1,950° F with the following results (table IV and fig. 32):

(1) One hour at 2,100° F gave the same critical grain growth as 4 hours at 1,950° F. The main difference was the larger grain size for the larger reductions with the 2,100° F treatment (grain size of 0 as compared with 3 (fig. 31)).

(2) One hour at 2,200° F gave a maximum grain size of -1. The maximum grain size varied between -1 and 0 along the length of the bar. Apparently solution-treating at 2,200° F erased any effect of critical reduction.

(3) One hour at 2,250° F was very similar except that a grain size of -3 developed for 1.6-percent reduction.

(4) One hour at 2,300° F was similar to 1 hour at 2,250° F except that the grain sizes along the bar varied between -1 and -2.

The most important feature of these results was the relatively little effect of extremely high temperature treatments on abnormal grain growth. Inadvertent high temperatures apparently are not a major factor in abnormal grain growth provided it can occur at normal heating temperatures. The absence of abnormal grain growth in heating at 2,200° F suggests that there are intermediate conditions of strain relief, solution of grain-growth restrainers, and grain-growth characteristics which restrict abnormal grain growth.

DISCUSSION

The investigation provides considerable information regarding the conditions which can cause abnormal grain growth in heat-resistant alloys of the types 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

All of the results indicate that small amounts of deformation applied to essentially strain-free metal are responsible for the development of abnormal grain growth. When such critically strained metal is reheated to the usual hot-working or solution-treating temperatures, abnormal grain growth may occur. The main problem in preventing abnormal grain growth seems to be the anticipation and avoidance of the often complex conditions which can induce critical strain. The usual temperatures and time periods of solution treatment are sufficient for abnormal grain growth. Heating

conditions for hot-working may or may not develop abnormal grains depending on the temperatures and time periods used.

The amount of strain required to induce susceptibility to abnormal grain growth was rather small. In most cases studied, it was a small portion of the reduction in the range between 0.4 and 5.0 percent. Over all the experiments this reduction was as low as 0.1 percent and as high as 9.7 percent. This means that, if the metal is reduced at least 10 percent in all parts during any one working operation, it should be free from abnormal grain growth; in most cases a reduction of 5 percent is adequate. The only exception to this rule noted was the case where a small reduction was applied after fairly large amounts of reduction caused recrystallization during working. The recrystallization left the material essentially strain free and the small amount of further straining induced susceptibility. This apparently does not occur when reduction is continued at essentially constant temperature after recrystallization starts if the last pass is heavy. A reheat after such recrystallization followed by a small critical reduction definitely induces susceptibility. Continued reduction with a falling temperature after recrystallization at higher temperatures is a less obvious but important source of critical reduction.

The data clearly showed that susceptibility can be reduced by rapid cooling from a high temperature. In this case, the thermal stresses critically deform the surface of the stock. It should be clearly recognized that this is a case where the dimensions of the metal piece together with the cooling rate are combined variables governing the amount of thermally induced strain. Air-cooling can induce abnormal growth in some cases. In other shapes, water-quenching may be required. If temperature gradients or the restraint to contraction is sufficiently small even water-quenching will not critically strain the metal. This source of critical strain is favored by high thermal expansion and low thermal conductivity. It should be noted that cooling rate after a reduction larger than the critical amount will have no effect because the thermally induced strain will be merely superimposed on the strain already present.

These results clearly indicate the following principles necessary to avoid abnormal grain growth:

- (1) Rapid cooling from an essentially strain-free condition must be avoided. Thus, if metal is heated under conditions which remove strain from prior working and then cooled rapidly enough to deform the surface critically by thermal stressing, it will undergo abnormal grain growth when reheated to usual solution or hot-working temperatures. This condition could probably be encountered on cooling from hot-working only if the reduction conditions were such that recrystallization to an essentially strain-free condition occurred during the working operation. Likewise, any section of a part which received no reduction would be susceptible after such cooling.

(2) The critical reduction for developing sensitivity to abnormal grain growth is essentially independent of temperature of straining. Thus, if metal is annealed and cold-straightened by methods which introduce small strains, those sections of the metal receiving critical deformations will be susceptible.

(3) Any reduction 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 free from strain from prior reduction.

(4) In hot-working in dies, it is essential that the deformation in all parts of the piece be more than the critical amount. This means that the dies must be designed to insure more than the critical amount of metal movement in every step. Common sources of difficulty include die "hang up" where the metal does not move, incorrect proportioning of the sequency of dies so that some parts of the piece receive little or no reduction in some steps, and flash preventing dies from closing thereby restricting metal flow to small amounts in some parts. Trimming of flash from a forging after a reheat without any other reduction is a common source of critical reduction if the reheat relieves prior strain because the operation introduces a strain gradient certain to include critical deformation.

(5) Abnormal grain growth can occur during reheats if the time and temperature of reheating are sufficient for the grain growth. Even though initial reductions may be larger than the critical reduction, it can occur during subsequent reductions if reheating relieves prior strain. Repeated steps involving critical strain and grain growth during reheats are almost certainly the cause of extremely large grains sometimes encountered in forging turbine blades. The repeated sequence causes additional growth during each reheat.

(6) The possibility of recrystallization during working rendering metal susceptible to abnormal grain growth from an additional small reduction seems to be a fairly important principle. This recrystallization is probably a common source of fairly large grains (grain sizes of the order of 0 to -1. It probably explains why many successful forging operations for gas-turbine blades require the limiting of the forging blows to one per heat. The first blow at a relatively high temperature induces recrystallization with little or no strain-hardening. The temperature falls rapidly and additional blows probably result in small deformations which critically deform the relatively strain free metal. If multiple operations per heat are to be used, care must be exercised to be sure that recrystallization is not followed by small critical reductions at a lower temperature where recrystallization stops.

(7) Forging experience indicates that temperature and time of heating and the capacity of hot-working equipment are important practical variables. The reason for this apparently involves several factors. The most important factor probably is the uniformity of metal flow in a die as influenced by the temperature-sensitive flow characteristics of the metal. Time of heating probably governs amount of relief from prior strain and grain-growth effects during reheats. Small parts and thin edges cool rapidly in a die and may become so resistant to deformation that the deformation possible with the equipment being used is limited to the critical amount.

(8) Inadvertent abnormally high temperatures of heating for hot-working or solution treatment appear to be a relatively unimportant feature of abnormal grain growth. The major exception to this appears to be the case where the normal hot-working or solution temperatures and times were too low for abnormal grain growth to occur. In these cases, an abnormally high temperature will permit abnormal grain growth after critical deformation when there would be no evidence of it from normal heating conditions. The size of abnormal grains increases only slightly with temperature. If abnormal grain growth can occur during normal heating, the increase from abnormally high temperatures is relatively small.

(9) These generalities are restricted to the formation of abnormally large grains under conditions which normally do not develop excessively large grains. The investigation did not consider the causes of mixed grain sizes where the largest grains are of the order of 1 or 0. Several instances of this type of grain growth were, however, noted in the experiments. The development of a very few recrystallized small grains during working at a relatively low temperature was one source. Partial recrystallization causing bands of strained and recrystallized small grains was another. In some cases, certain conditions of prior heating caused relatively large grains to form during a subsequent reheat. Excessively high temperatures of heating frequently caused uniformly coarse grains to form.

(10) The investigation showed that the degree of reduction and not strain gradients was the major cause of abnormal grain growth. Abnormal grain growth is usually associated with strain gradients only because the critical deformation is usually present in the gradient. The critical-deformation range is so small that it can easily be missed in uniform reduction.

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

Metallurgical and Compositional Effects

The influence of metallurgical variables and compositional effects on abnormal grain growth was not studied in detail. However, certain observations and deductions can be made from the experimental results:

(1) The vacuum-melted Waspaloy alloy did not develop abnormal grains so large as did the air-melted stock and tended to be generally finer grained. In theory, the vacuum-melted material should have fewer oxides, nitrides, and other dispersed phases which act as grain-growth restrainers than the air-melted stock. If this were the governing factor, the temperatures and heating time for grain growth ought to be reduced. This, therefore, can hardly account for the restriction of the abnormal grain growth. The vacuum-melted heat had a carbon content of 0.08 percent while the air-melted heat had only 0.03 percent carbon. This is a sufficient difference so that the larger amount of carbides in the vacuum-melted material should restrict grain growth appreciably more than those in the lower carbon air-melted material. While this investigation did not demonstrate that the carbon content was the controlling factor between the air- and vacuum-melted stock, it alone could be responsible for the observed differences.

(2) The composition of the alloys was related to the temperatures and time for abnormal grain growth. For the three alloys considered, Nimonic 80A had the least resistance to grain growth. It underwent rapid grain growth at 1,950° F. Waspaloy required considerable time for abnormal grain growth at 1,950° F. Inconel X-550 required a temperature above 2,000° F and still required some time at 2,150° F for abnormal grain growth.

The comparatively high coarsening temperature for Inconel X-550 alloy was probably due to the grain-growth-restraining effect of columbium compounds. The main difference between the Nimonic 80A and the Waspaloy alloys presumably was the higher titanium and aluminum contents of the latter material. This presumably increased resistance to grain growth.

(3) There were many aspects of the details of the observed grain-growth variations which seem to be due to differences in grain-growth restraint from dispersed phases. The usual solution temperatures apparently are on the lower side of the temperature range for solution of such phases. The grain growth was relatively slow at the temperatures used. Under such conditions the critical deformation could be expected to be sensitive to the conditions of the grain-growth restrainers and possibly to the size of the abnormal grains. This fact seemed to be involved in the restraint of grain growth by prior heating in the precipitate-agglomeration range of 1,400° to 1,700° F. It probably was a factor in

the variable effect on abnormal grain growth due to working at or above the usual solution temperature.

(4) Deformation of multigrained materials is not uniform on a microscopic scale even though it may be on a macroscopic scale. The microscopic flow characteristics probably vary depending on the temperature and method of working. Consequently, it could be expected that the rather narrow range of reductions inducing abnormal grain growth could be sensitive to details of the mechanisms of flow during working. Furthermore, such metallurgical factors as compositional differences, grain size, dispersed phases, and degree of solution of dispersed phases could influence flow characteristics and thereby influence critical deformation details.

(5) The size of grains and extent of abnormal grain growth were remarkably insensitive to increased temperatures of heating above the lowest temperature at which abnormal grain growth would occur. There were, however, certain intermediate higher temperatures which in some cases restricted abnormal grain growth. The explanation of these effects is not clear from this investigation. There are probably several interrelated effects. Increased temperature should intensify grain growth. On the other hand, very large grains require initiation of growth from only a few centers. Increasing the temperature would increase the number of centers of grain growth and thereby restrict grain growth through competition for surrounding grains. An increase in temperature would also tend to reduce grain-growth restrainers by solution and thereby increase the centers nucleated for growth. Some strain relief probably occurs during heating before grain growth starts, thereby influencing critical strain. This could be expected to be variable depending on temperature and heating rate. Possibly this would result in variation in the amount of initial strain required to leave a residual critical strain at the time the metal attained a temperature sufficient for abnormal grain growth.

Strain relief during heating to the higher temperatures of rolling may well be the cause for the general increase in the amount of strain required for critical grain growth.

(6) In general it appeared that abnormal grain growth was fairly independent of initial grain size. In those cases where there was an apparent grain-size effect, it is probable that variation in grain-growth restrainers was the controlling factor.

(7) There was considerable variation in the amount of deformation required to initiate abnormal grain growth. The reasons were not clear. Varying strains from cooling were probably a factor. Also, as previously discussed, variation in grain-growth restrainers could have been involved.

There was also some evidence that the amount of deformation was related to rate of heating to the solution temperature. The most important effect, however, was probably unrecognized variations in the strain from the conditions of manipulation used in the experiments.

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. 3 and 4) 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) The conditions at the critical deformation are discussed above.
- (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. 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.

In the discussion of methods of avoiding abnormal grain growth it was pointed out that recrystallization followed by critical reduction could be a source of abnormal grain growth under conditions where a more than critical reduction was apparently being required. The mechanism involved apparently was no different from that for the case of small reduction of initially strain-free material, although there might be differences in the temperatures and times required for abnormal grain growth due to unusually small grain size of the recrystallized metal.

The data predominately indicated that the amount of strain and not the strain gradients was the controlling factor. This would be in accordance with the theory involved.

The influence of the presence of abnormally large grains in the structure before working was not studied. Such grains could be present because of lack of refinement from ingot structure or because of allowing abnormal growth to occur during prior processing. General experience indicates that it is difficult to break up such isolated large grains. It is doubtful, however, that they would contribute to abnormal grain growth except in the case where large-scale critical deformations were superimposed over the large grains. Apparently, where larger than critical deformations are involved the proper strain gradient does not develop, or the amount of critically strained metal between the large grain and surrounding fine grain is so small that no appreciable grain growth occurs.

Since rate of grain growth increases as heat-treating temperature increases, abnormal grain growth can occur in less time at the higher than at the normal solution-treating temperatures.

Metallurgical variables had relatively little effect on abnormal grain growth. Various prior-history effects caused only minor variations so long as the prior history did not include more than critical deformation without an opportunity for strain relief. Grain-growth restrainers as influenced by composition and heat treatment had minor effects. There was some evidence that metal-flow characteristics as influenced by temperature and metallurgical variables caused minor changes in critical reduction and grain size. Certain heating rates and temperatures apparently can restrict abnormal grain growth.

The major difference between the three alloys studied was the temperature and time periods for abnormal grain growth. Inconel X-550 alloy required a higher temperature than did the other two alloys. Presumably this was due to the grain-growth-restraining tendency of the columbium compounds present in the alloy. The vacuum-melted Waspaloy developed smaller grains than did the air-melted, possibly because of the grain-growth restraint of a higher carbon content.

CONCLUSIONS

The following results and conclusions were obtained from an investigation to determine the basic causes of abnormal grain growth in air- and vacuum-melted Waspaloy, Inconel X-550, and Nimonic 80A alloys:

1. Abnormal grain growth was found to occur in Waspaloy, Inconel X-550, and Nimonic 80A alloys only when small deformations caused very large grains to grow during subsequent heating. The deformations inducing abnormal growth usually were within a range of reductions of 0.4 to 5.0 percent and were within a reduction range of 0.1 to 9.7 percent when all variables were considered. Normal solution temperatures and times are sufficient for the abnormal grain growth.

2. Abnormal grain growth can be avoided if care is exercised to be sure that all parts of the metal are deformed more than 10 percent in any one working step before reheating. In most cases a reduction of 5 percent will be sufficient. The only exception to this is the case where large reductions cause recrystallization during working and working is continued under conditions which will critically deform the strain-free recrystallized metal.

3. The main problem in avoiding abnormal grain growth seems to be in recognizing and avoiding conditions leading to critical deformation. It can be induced by the thermal stresses of rapid cooling. Nonuniform metal movement during hot-working leaving certain sections critically deformed is a major source of critical deformation. Attention must be given to the

design and metal flow to avoid critical deformation. Particular care must be taken to avoid small deformations and deformation gradients which are sure to include a critical deformation. Recrystallization during working and reheating can remove the effect of previous deformation so that it is important to obtain a more than critical deformation in every hot-working operation.

4. The development of susceptibility to abnormal grain growth was remarkably independent of temperature of working. Deformation at room temperature had the same effect as at hot-working temperatures. Heating temperature had relatively little effect on abnormal grain growth provided the temperature was high enough for the growth to occur at all. Because it could occur at the normal solution temperatures for the alloys, inadvertent excessively high temperatures are not necessary for it to occur. In addition, these temperatures do not cause substantially larger grains to form.

University of Michigan,
Ann Arbor, Mich., September 19, 1957.

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TABLE I.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF AIR-MIXED MARPALOY

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 provide abnormal grains, percent		
			Reduction	Grain size	0	0.7	2.2	3.7	6.5	8.9	11.5	12.4	12.2	12.6	13.6	13.6			13.6	13.6
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, OQ	80	1/2 hr at 1,950° F, OQ	Reduction 0	0.7	2.2	3.7	6.5	8.9	11.5	12.4	12.2	12.6	13.6	13.6	13.6	13.6	13.6	13.6	0.7	3.7
Do-----	1,400	-----do-----	Reduction 0	0.8	2.0	3.1	5.5	8.0	9.0	12.2	12.2	12.2	12.2	12.2	12.2	12.2	12.2	12.2	.8	3.1
Do-----	1,600	-----do-----	Reduction 0	0.7	1.9	3.2	4.4	5.9	8.3	10.9	12.6	13.6	13.6	13.6	13.6	13.6	13.6	13.6	.7	3.2
Do-----	1,800	-----do-----	Reduction 0	0.5	0.7	1.6	2.9	4.2	7.2	9.3	11.1	12.8	13.6	13.6	13.6	13.6	13.6	13.6	.7	2.9
Do-----	1,950	-----do-----	Reduction 0	0.5	1.0	2.0	3.9	5.0	6.4	8.8	10.6	13.0	13.6	13.6	13.6	13.6	13.6	13.6	1.0	5.0
Do-----	2,000	-----do-----	Reduction 0	0.9	1.0	1.8	2.8	3.8	4.9	6.9	10.2	12.1	13.6	13.6	13.6	13.6	13.6	13.6	1.0	1.8
Do-----	2,100	-----do-----	Reduction 0	1.6	2.7	3.0	3.7	4.9	7.5	9.6	11.0	11.9	13.6	13.6	13.6	13.6	13.6	13.6	3.0	4.9
1 hr at 1,950° F, OQ	1,600	-----do-----	Reduction 0	0.6	1.5	2.2	3.4	6.2	8.4	10.6	12.6	14.6	13.6	13.6	13.6	13.6	13.6	13.6	1.5	2.2
Rolled 25 percent at 80° F plus 1 hr at 1,950° F, OQ	1,600	-----do-----	Reduction 0	0.7	2.0	3.4	6.1	8.6	11.1	13.0	14.7	13.6	13.6	13.6	13.6	13.6	13.6	13.6	.7	2.9
Rolled 25 percent at 1,600° F plus 1 hr at 1,950° F, OQ	1,600	-----do-----	Reduction 0	1.3	2.5	3.5	4.9	6.3	8.9	11.3	13.0	14.9	13.6	13.6	13.6	13.6	13.6	13.6	1.3	3.5
Rolled 25 percent at 1,950° F plus 1 hr at 1,950° F, OQ	1,600	-----do-----	Reduction 0	0.8	2.4	3.9	5.3	6.8	8.8	11.7	13.1	14.4	13.6	13.6	13.6	13.6	13.6	13.6	.8	2.4
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	80	-----do-----	Reduction 0	0.4	1.5	2.7	5.8	8.2	10.8	12.2	13.6	13.6	13.6	13.6	13.6	13.6	13.6	13.6	.4	2.7
Do-----	1,900	-----do-----	Reduction 0	0.2	0.6	0.8	1.3	2.4	3.6	4.8	7.3	8.8	9.8	11.5	13.6	13.6	13.6	13.6	.8	3.1
Do-----	2,100	-----do-----	Reduction 0	1.1	2.2	3.4	4.9	7.2	8.9	10.6	12.2	13.6	13.6	13.6	13.6	13.6	13.6	13.6	1.1	3.4
Rolled 50 percent at 1,950° F plus 1/2 hr preheat at 2,100° F	2,100	-----do-----	Reduction 0	0.2	1.8	2.5	2.9	4.2	5.2	7.2	9.0	10.9	11.8	13.6	13.6	13.6	13.6	13.6	2.5	4.2
Rolled 50 percent at 1,950° F, then transferred to 1,600° F furnace for 1/2 hr	1,600	-----do-----	Reduction 0	0.4	1.8	2.5	3.2	4.2	5.2	7.2	9.5	10.6	11.9	13.6	13.6	13.6	13.6	13.6	2.5	7.2
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,850	Slow-heated from 1,400° F to 1,950° F in 3 hr, held 4 hr at 1,950° F, AC	Reduction 0	0.1	0.9	2.9	4.8	6.1	7.4	10.3	13.3	16.3	22.5	13.6	13.6	13.6	13.6	13.6	.1	4.8
Rolled 50 percent at 1,950° F plus 1 hr at 1,975° F, AC	1,850	1/2 hr at 1,975° F, AC	Reduction 0	0.4	1.4	3.6	5.6	7.4	8.3	10.2	11.8	14.7	17.7	27.0	13.6	13.6	13.6	13.6	.4	4.6
Do-----	1,950	-----do-----	Reduction 0	0.4	1.4	3.9	6.1	6.9	8.9	9.3	9.8	12.2	16.6	27.5	13.6	13.6	13.6	13.6	.4	5.9
Do-----	2,050	-----do-----	Reduction 0	1.0	2.9	5.8	6.8	8.9	10.0	11.0	12.8	15.8	28.3	13.6	13.6	13.6	13.6	13.6	1.0	5.8
Rolled 50 percent at 1,950° F	80	1/2 hr at 1,950° F, OQ	Reduction 0	0.4	0.7	1.0	4.8	7.7	9.8	10.5	13.6	13.6	13.6	13.6	13.6	13.6	13.6	13.6	.4	7.7
Do-----	1,600	Slow-heated from 1,400° F to 1,950° F in 3 hr, held 4 hr at 1,950° F, OQ	Reduction 0	1.6	3.2	4.7	6.2	8.4	9.7	11.5	21.1	13.6	13.6	13.6	13.6	13.6	13.6	13.6	6.2	9.7

TABLE II.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF VACUUM-DETERGED MARBLOY

Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °C	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 provide abnormal grains, percent
			Reduction	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size		
3/4-in. stock rolled at 1,990° F from 2-in. ingot plus 1 hr at 1,990° F, AC	1,400	4 hr at 1,990° F, OQ	0	0.1	0.5	1.2	2.0	3.8	5.1	6.7	---	---	---	0.5	1.6
	1,600	do	0	0.1	0.5	1.2	2.0	3.4	5.3	6.2	---	---	---	.5	2.0
	1,900	do	0	0.3	0.6	0.8	1.2	1.5	3.4	5.2	6.1	7.3	---	.6	2.5
	2,100	do	0	0.5	1.2	2.1	4.0	6.2	7.6	9.3	---	---	---	2.1	3.5

^aAC, air-cooled; OQ, oil-quenched.

TABLE III.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF INCOVEL X-550 ALLOY

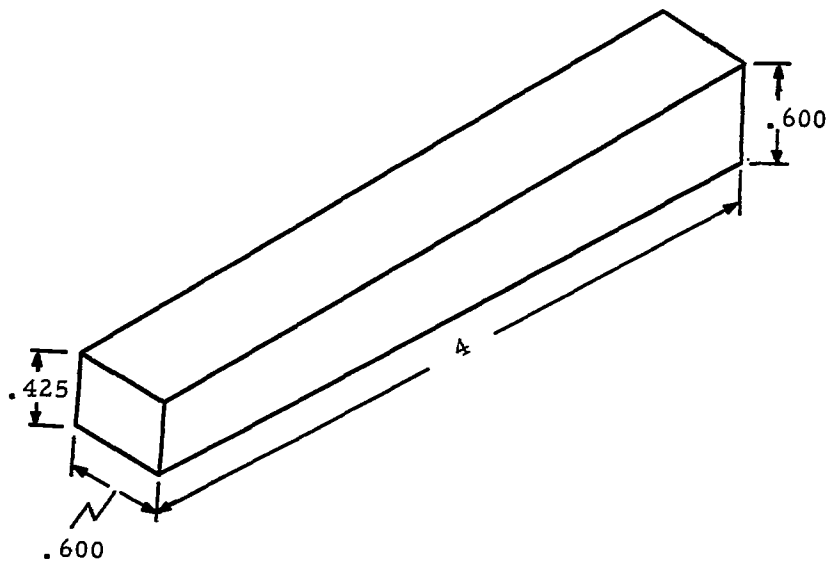
Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °C	Final treatment for grain growth (a)	Percent reduction by rolling and ASTM grain size after final treatment as measured along tapered specimens											Critical reduction, percent	Minimum reduction to prevent abnormal grains, percent	
			Reduction	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size	Grain size			
2 hr at 1,900° F, AC	1,600	1 hr at 2,150° F, AC	0	1.4	3.0	4.5	5.7	8.0	10.5	11.1	13.7	---	---	---	2.6	8.1
	1,800	do	0	1.4	3.0	4.5	5.7	8.0	10.5	11.1	13.7	---	---	4.5	10.5	
	2,000	do	0	0.5	1.4	3.1	4.4	5.8	7.8	9.3	10.1	12.5	---	---	3.1	5.8
	2,200	do	0	1.3	2.7	4.0	5.4	6.8	8.5	9.7	10.9	11.9	---	None	0	
2 hr at 2,100° F, AC	1,600	do	0	0.2	1.4	1.8	3.4	5.0	6.7	9.1	11.4	12.4	14.4	1.4	5.0	
	1,800	do	0	0.4	2.0	3.6	4.9	6.2	8.1	9.6	10.1	12.5	---	.4	4.9	
	2,000	do	0	0.4	2.0	3.6	5.1	6.7	8.2	9.8	11.0	12.1	---	.4	3.6	
	2,200	do	0	0.5	2.0	3.7	4.8	6.0	8.2	9.2	11.5	11.7	---	None	1.2	
Rolled 50 percent at 1,990° F plus 1/2 hr preheat at 2,100° F, then transferred to 1,600° F furnace for 1/2 hr	1,600	do	0	0.1	1.0	2.1	3.5	4.4	5.6	7.5	8.9	11.1	---	4.4	7.5	
	2,100	do	0	2.0	4.0	5.1	6.2	7.8	9.2	11.1	12.4	---	---	4.0	7.8	

^aAC, air-cooled.

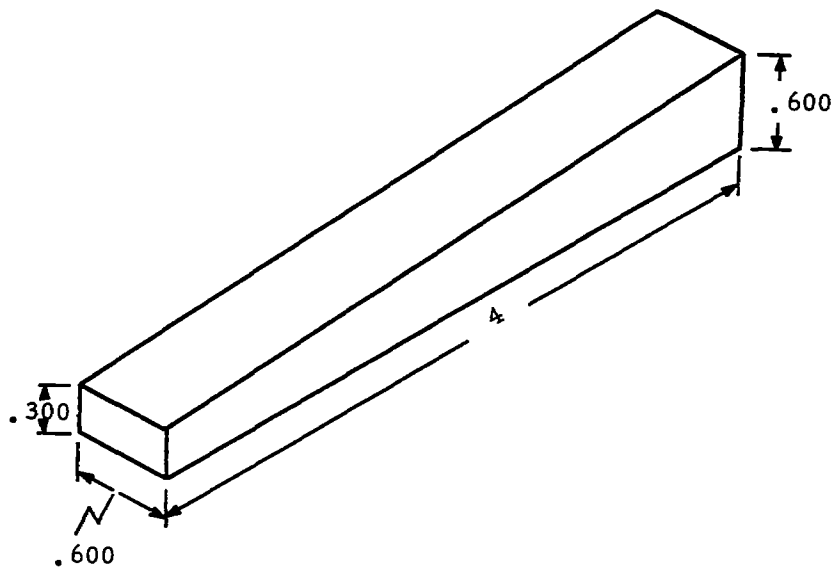
TABLE IV.- GRAIN-SIZE DATA FROM ROLLED TAPERED SPECIMENS OF 7075-T6 ALUMINUM ALLOY

Equalizing treatment of as-received stock (a)	Rolling temperature for tapered specimen, °F	Number of passes	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 grain, percent	
				Reduction	0	0.1	1.0	3.6	7.0	8.2	9.3	10.3	12.5	14.2	16.5	25.0				
Rolled 50 percent at 1,950° F plus 1 hr at 1,950° F, AC	1,750	1	1/2 hr at 1,975° F, AC	Reduction	0	0.1	1.0	3.6	7.0	8.2	9.3	10.3	12.5	14.2	16.5			0.1	12.5	
Do	1,850	1	do	Reduction	0	0.1	1.9	3.9	5.8	7.1	8.4	9.9	11.4	12.6	15.2	25.7		1	None	
Do	1,850	1	Slow-heated from 1,400° F to 1,950° F in 5 hr plus 1/2 hr at 1,950° F, AC	Reduction	0	0.7	2.9	5.2	6.6	8.1	9.7	11.5	14.6	22.0				2.9	5.2	
Do	1,950	1	1/2 hr at 1,975° F, AC	Reduction	0	0.1	1.0	3.8	5.6	6.8	8.0	9.5	10.6	14.0	16.3	26.8		1	26.8	
Do	1,950	2	1/2 hr at 1,950° F, AC	Reduction	0	0.5	1.6	2.6	4.7	6.8	8.6	10.4	12.7	15.0	17.7	19.2	22.0	27.5	1.6	None
None	1,950	1 (light taper)	do	Reduction	0	0.5	0.7	1.0	1.2	1.8	2.7	3.7	5.2	6.8	7.6	8.6		1.0	1.2	
None	1,950	1 (heavy taper)	do	Reduction	0	1.6	3.1	4.6	5.0	6.7	7.4	9.2	10.9	16.5	24.2			3.1	4.2	
None	1,950	2	do	Reduction	0	0.4	0.7	1.2	1.6	1.9	2.8	4.0	5.2	6.8	8.0	9.0	9.6	1.6	None	
None	1,950	1	1 hr at 2,100° F, AC	Reduction	0	0.4	1.0	1.8	3.7	6.0	7.4	9.0	11.7	14.6	18.0	24.3		1.0	24.3	
None	1,950	1	1 hr at 2,200° F, AC	Reduction	0	0.4	1.5	2.5	4.5	6.6	8.0	9.2	12.5	15.2	18.1	25.6		1.5	None	
None	1,950	1	1 hr at 2,250° F, AC	Reduction	0	0.4	1.1	1.6	4.0	6.0	7.6	9.2	12.0	14.0	18.3	24.5		1.6	None	
None	1,950	1	1 hr at 2,300° F, AC	Reduction	0	0.4	0.6	1.0	1.4	2.0	2.5	3.5	4.7	10.0	16.0	18.0	25.0	1.4	None	
None	1,950	1	Slow-heated from 1,950° F to 2,300° F in 1 hr plus 1 hr at 2,300° F, AC	Reduction	0	0.4	0.9	1.0	1.2	3.1	3.0	6.4	7.8	10.5	12.5	17.0	25.0	0.1-2	None	

*AC, air-cooled.

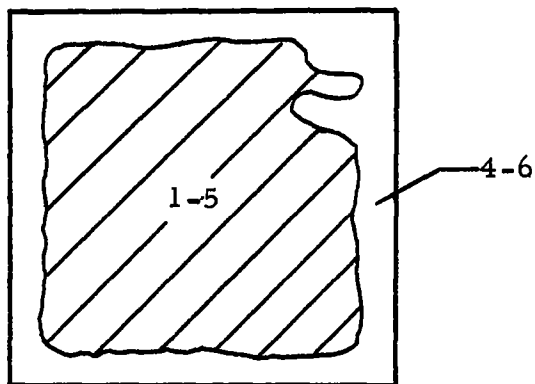


(a) Tapered specimen used to obtain approximately 0- to 15-percent reduction.

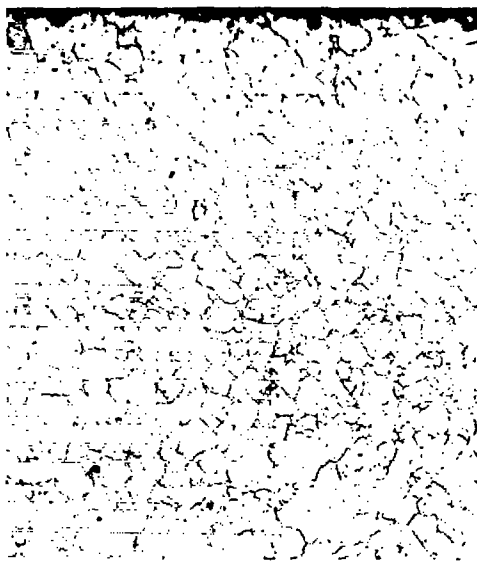


(b) Tapered specimen used to obtain approximately 0- to 29-percent reduction.

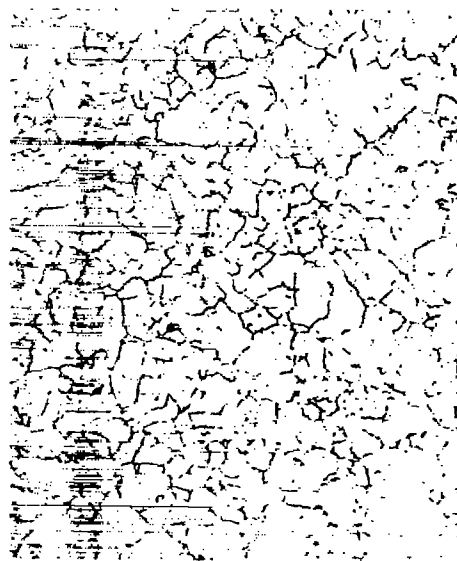
Figure 1.- Tapered specimens used to obtain indicated range of percent reduction by being rolled to flat bars (dimensions in inches).



(a) Approximate distribution of grain sizes.



(b) Microstructure near bar surface. Magnification, X50.



(c) Microstructure in bar center. Magnification, X50.

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Figure 2.- Microstructures and grain sizes of transverse section of as-received air-melted Waspaloy bar stock.

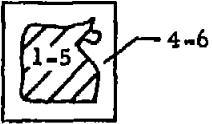
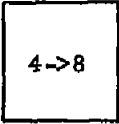
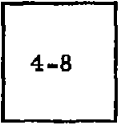
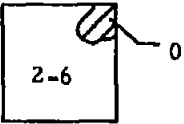

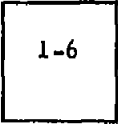


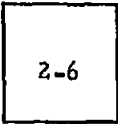
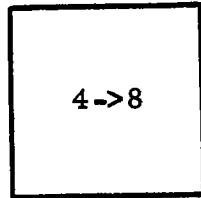
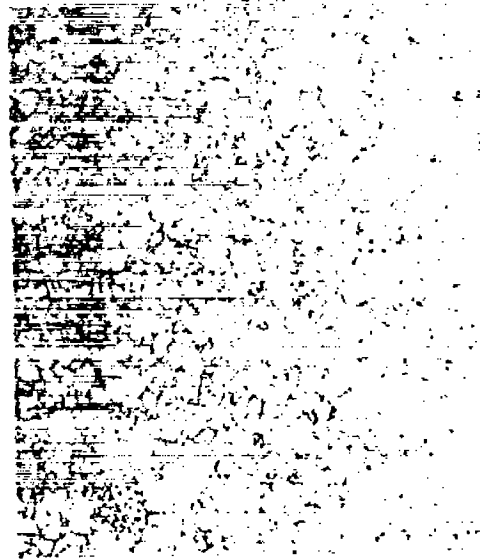
Heat Treatment	Equalizing Treatment of As-Received Stock	
	None	Rolled 70% at 1950°F
		
1 hour at 1900°F		
1 hour at 1950°F		
4 hours at 1950°F		
1 hour at 2000°F		
1 hour at 2100°F		

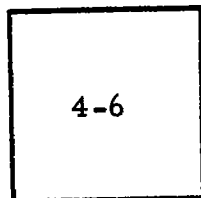
Figure 3.- Effect of heat-treating time and temperature upon grain size of transverse sections of air-melted Waspaloy bar stock.



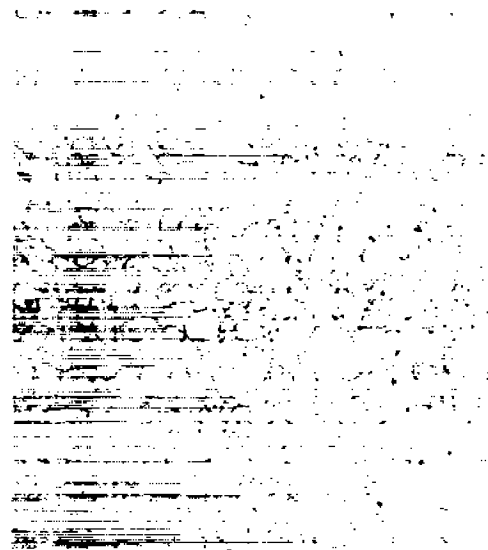
(a) Approximate distribution of grain sizes as rolled 50 percent at 1,950° F.



(b) Microstructure as rolled 50 percent at 1,950° F. Magnification, X50.



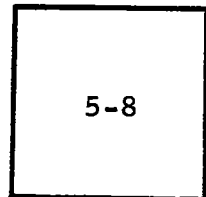
(c) Approximate distribution of grain sizes as rolled 50 percent at 1,950° F, plus 1 hour at 1,950° F, then air-cooled.



(d) Microstructure as rolled 50 percent at 1,950° F, plus 1 hour at 1,950° F, then air-cooled. Magnification, X50.

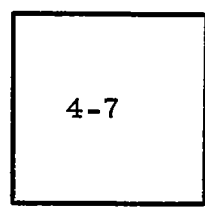
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Figure 4.- Microstructures and grain sizes of transverse sections of equalized air-melted Waspaloy bar stock.



(a) Approximate distribution of grain sizes as rolled.

(b) Microstructure as rolled. Magnification, X50.



(c) Approximate distribution of grain sizes as rolled, plus 1 hour at 1,950° F, then air-cooled.

(d) Microstructure as rolled, plus 1 hour at 1,950° F, then air-cooled. Magnification, X50.

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Figure 5.- Microstructures and grain sizes of transverse sections of vacuum-melted Waspaloy bar stock.

Heat Treatment	Equalizing Treatment of As-Received Stock			
	Rolled 70% at 1950°F			Rolled 25% at 80°F
	+ 1 hour at 1950°F, air-cooled 4-6	+ 1 hour at 1950°F, water-quenched 4-6		+ 1 hour at 1950°F, oil-quenched 4-8
	Cooling Method, Air-Cooled	Cooling Method, Air-Cooled	Cooling Method, Water-Quenched	Cooling Method, Oil-Quenched
1 cycle of 1 hour at 1950°F, cooled	4-6	(-2) 2	3-6 (-2) -1	2-8 5-8
2 cycles of 1 hour at 1950°F, cooled	3-5	(-2) 2	(-2)-1 3-4	
3 cycles of 1 hour at 1950°F, cooled	3-4	(-3) (-1)	(-2)-1 2-4	
4 cycles of 1 hour at 1950°F, cooled	2-4 0	(-3) (-1)	(-3)-1 1-2	
1 cycle at 1950°F, cooled		(-4) 4hr	3-6	0-8 3 hr 5-8

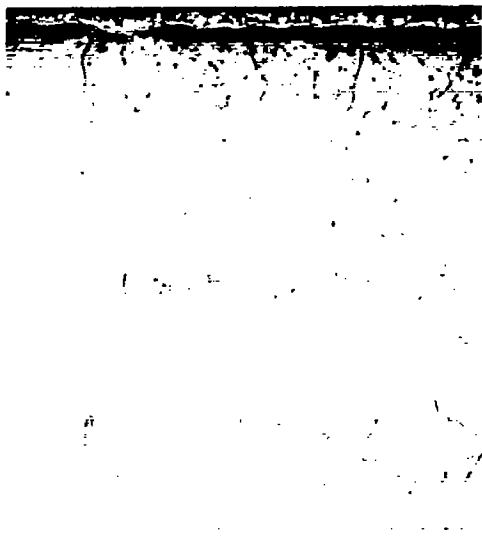
Figure 6.- Effect of repeated heating and cooling upon grain size of transverse sections of air-melted Waspaloy bar stock.



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



(b) 1 hour at 1,950° F,
water-quenched, plus
2 cycles of 1 hour at
1,950° F, then air-cooled.



(c) 1 hour at 1,950° F, water-
quenched, plus 4 cycles of
1 hour at 1,950° F, then
air-cooled.



(d) 1 hour at 1,950° F, water-
quenched, plus 1 cycle of
4 hours at 1,950° F, then
air-cooled.

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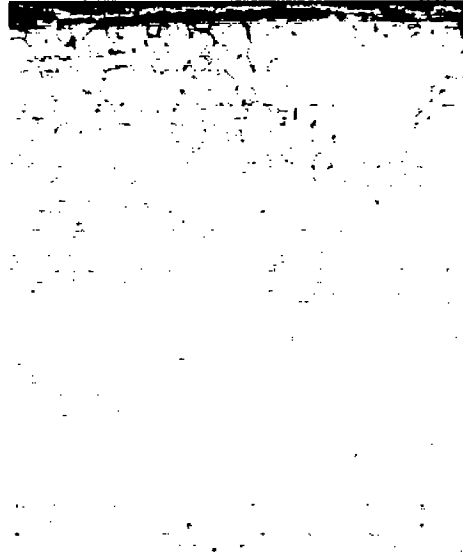
Figure 7.- Effect of repeated heating and cooling upon microstructure of air-melted Waspaloy which had been equalized by a 70-percent reduction at 1,950° F. (Transverse section at bar-stock surface.) Magnification, X50.

Heat Treatment	Equalizing Treatment of As-Rolled Stock	
	Rolled at 1950°F from 2-inch ingot to 1/2-inch bar stock	
	+ 1 hour at 1950°F, air-cooled 4-7	+ 1 hour at 1950°F, water-quenched 4-7
	Cooling Method, Air-Cooled	Cooling Method, Water-Quenched
1 cycle of 1 hour at 1950°F, cooled	4-7	0-2 → 4-7
2 cycles of 1 hour at 1950°F, cooled	4-7	0-2 → 3-6
3 cycles of 1 hour at 1950°F, cooled	4-7	(-2)-(-1) → 3-6
4 cycles of 1 hour at 1950°F, cooled	3-6	(-2)-(-1) → 3-6
1 cycle of 4 hours at 1950°F, cooled	Cooling Method, Air-Cooled	Cooling Method, Air-Cooled
	3-6	(-2)-(-1) → 3-6

Figure 8.- Effect of repeated heating and cooling upon grain size of transverse sections of vacuum-melted Waspaloy bar stock.



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



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



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



(d) 1 hour at 1,950° F,
water-quenched, plus
1 cycle of 4 hours at
1,950° F, then air-
cooled.

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Figure 9.- Effect of repeated heating and cooling upon microstructure of vacuum-melted Waspaloy which had been rolled at 1,950° F from a 2-inch ingot to $\frac{1}{2}$ -inch bar stock. (Transverse section at bar-stock surface.) Magnification, X50.

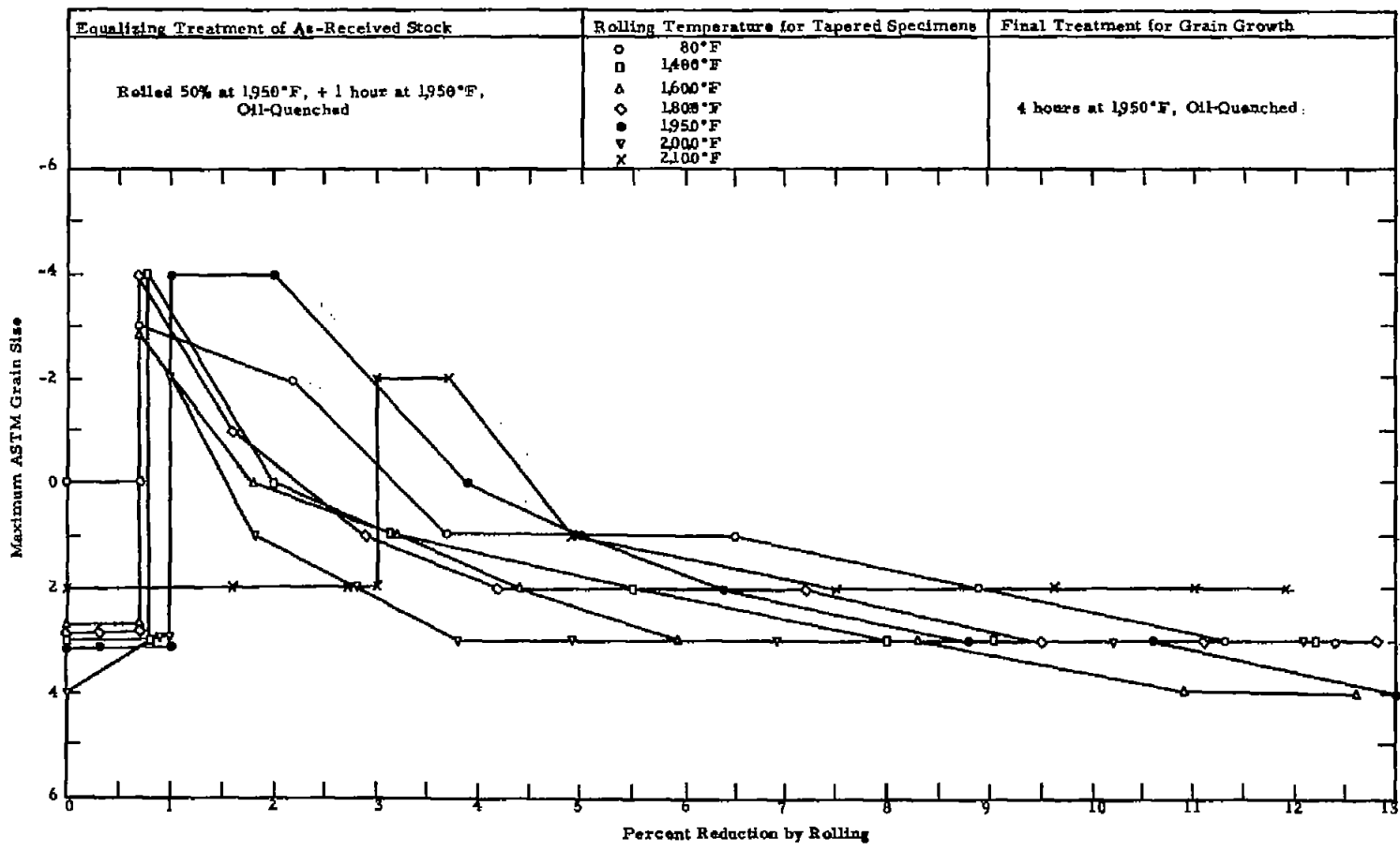


Figure 10.- Effect of rolling temperature and percent reduction upon maximum grain size of air-melted Waspaloy after final solution treatment.



(a) Zero-percent reduction.



(b) 1.0-percent reduction.



(c) 1.5-percent reduction.



(d) 13.0-percent reduction.

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Figure 11.- Effect of percent reduction by rolling at 1,950° F upon microstructure of equalized air-melted Waspaloy 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 oil-quenched. Final solution treatment was 4 hours at 1,950° F, then oil-quenched. Magnification, X50.

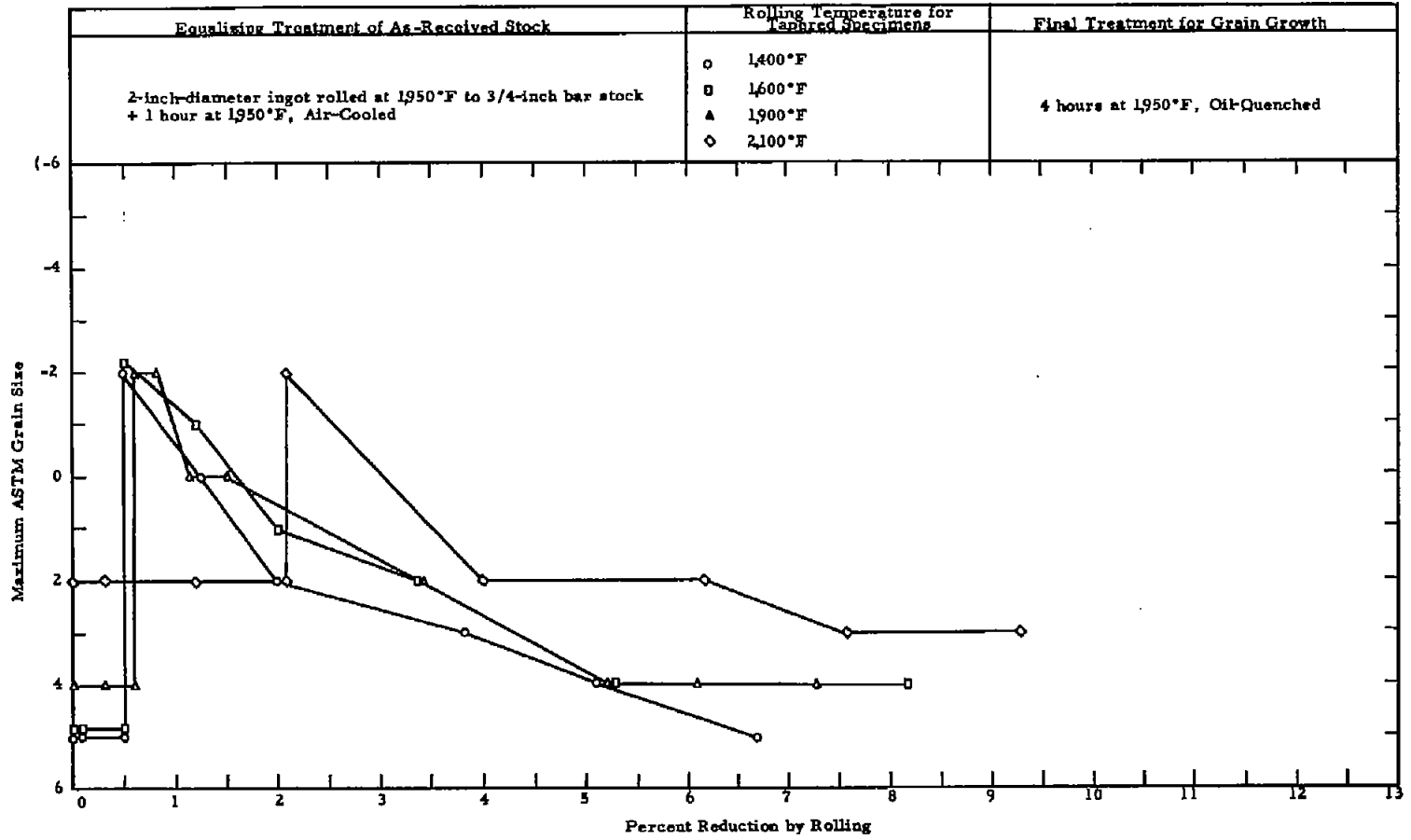


Figure 12.- Effect of rolling temperature and percent reduction upon maximum grain size of vacuum-melted Waspaloy after final solution treatment.



(a) Zero-percent reduction.



(b) 0.6-percent reduction.



(c) 0.8-percent reduction.



(d) 6.1-percent reduction.

Figure 13.- Effect of percent reduction by rolling at 1,900° F upon microstructure of vacuum-melted Waspaloy after final solution treatment. Equalizing treatment of as-rolled stock was 1 hour at 1,950° F, then air-cooled. Final solution treatment was 4 hours at 1,950° F, then oil-quenched. Magnification, X50.

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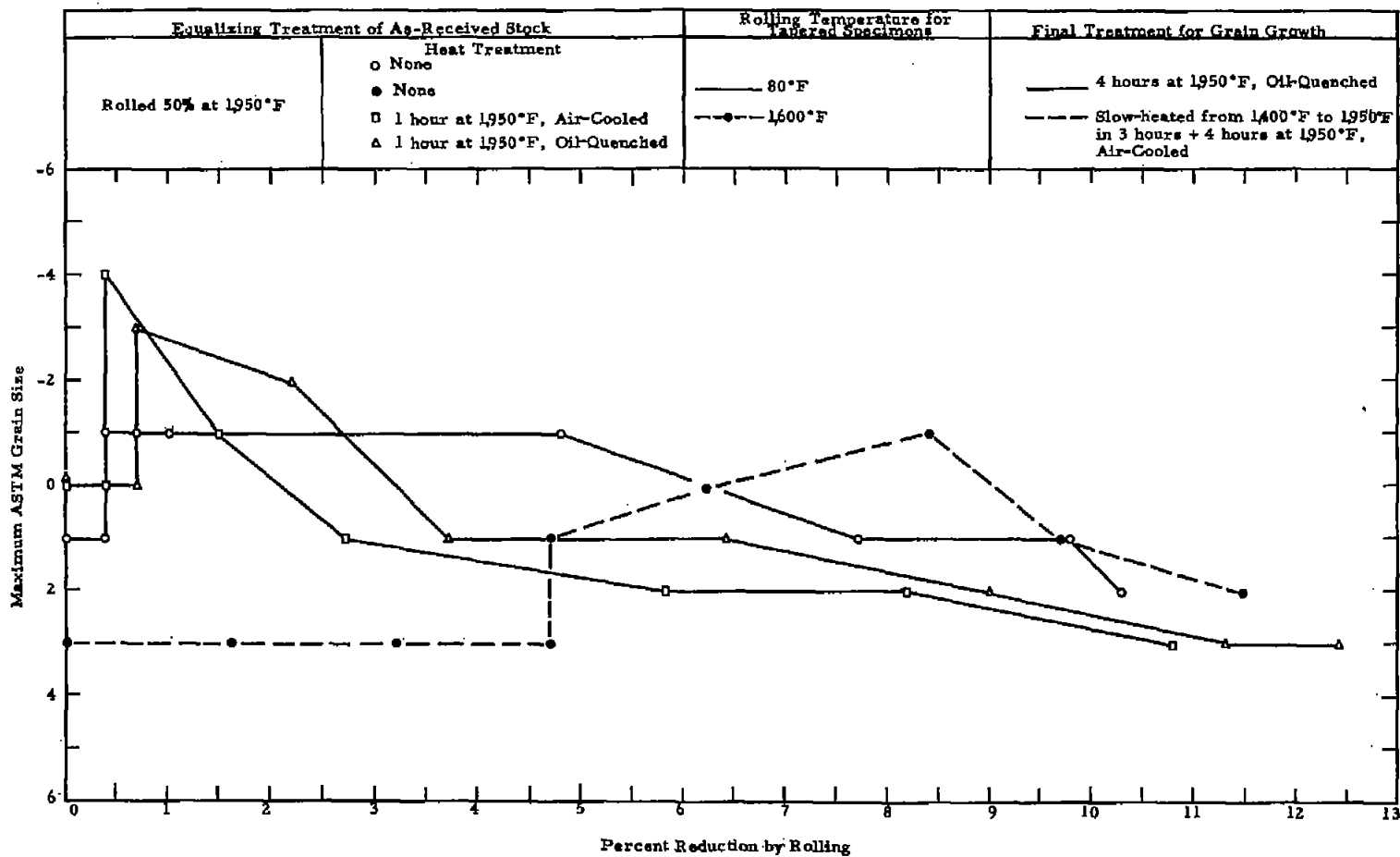


Figure 14.- Effect of equalizing treatment, rolling temperature, percent reduction, and heating rate before final solution treatment upon maximum grain size of air-melted Waspaloy after final solution treatment.

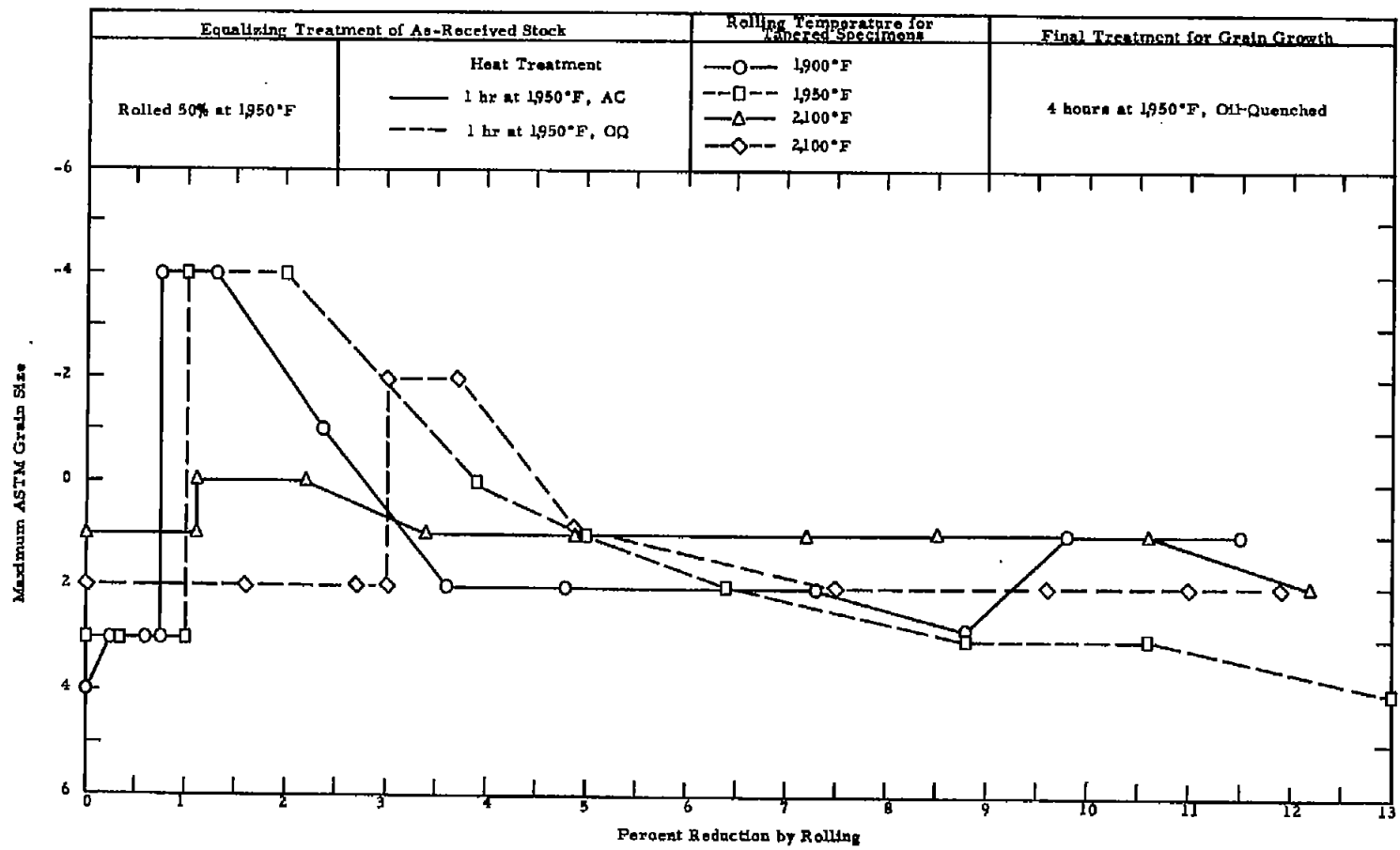


Figure 15.- Effect of equalizing treatment, rolling temperature, and percent reduction upon maximum grain size of air-melted Waspaloy after final solution treatment. AC, air-cooled; OQ, oil-quenched.

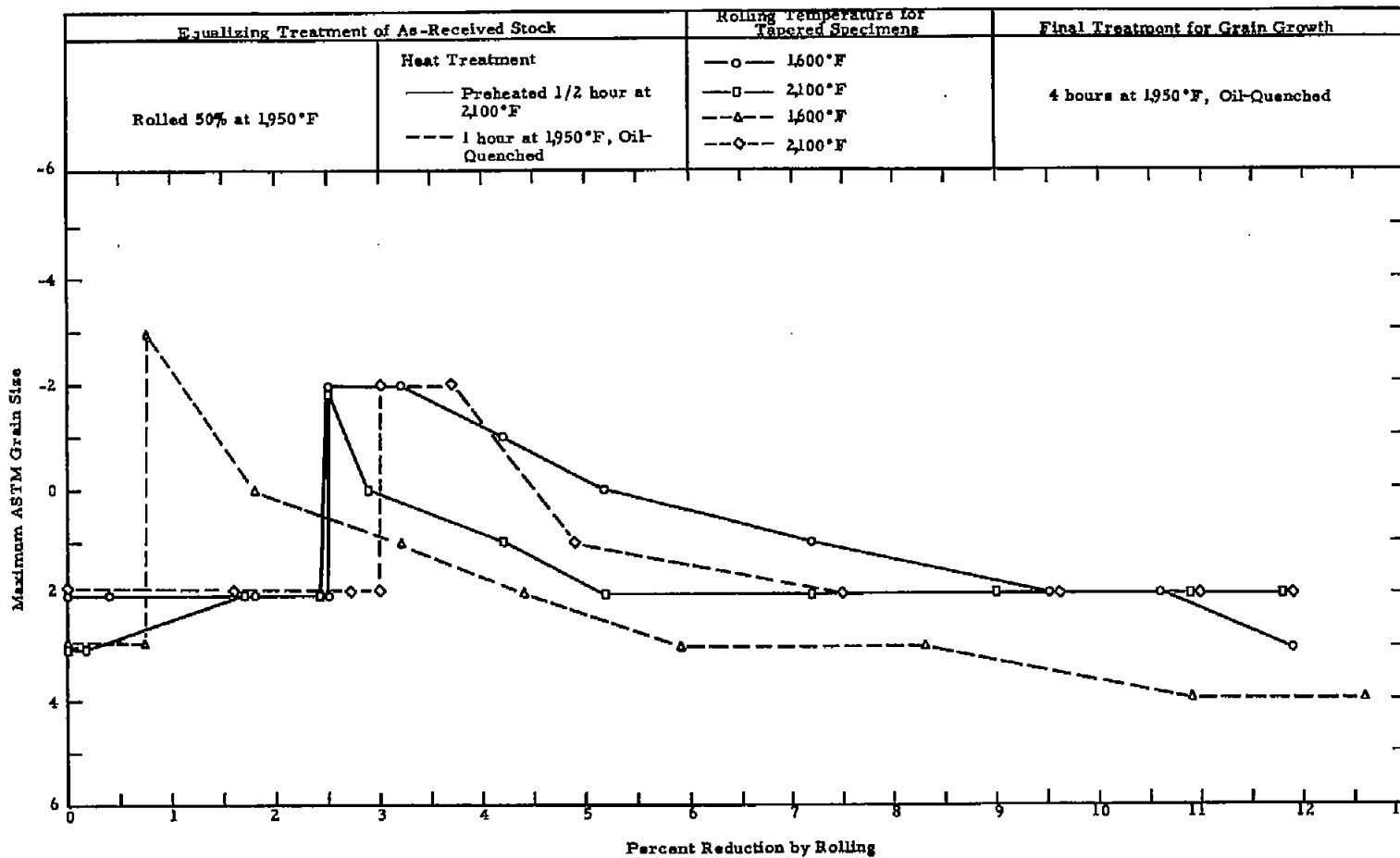


Figure 16.- Effect of equalizing treatment, rolling temperature, and percent reduction upon maximum grain size of air-melted Waspaloy after final solution treatment.

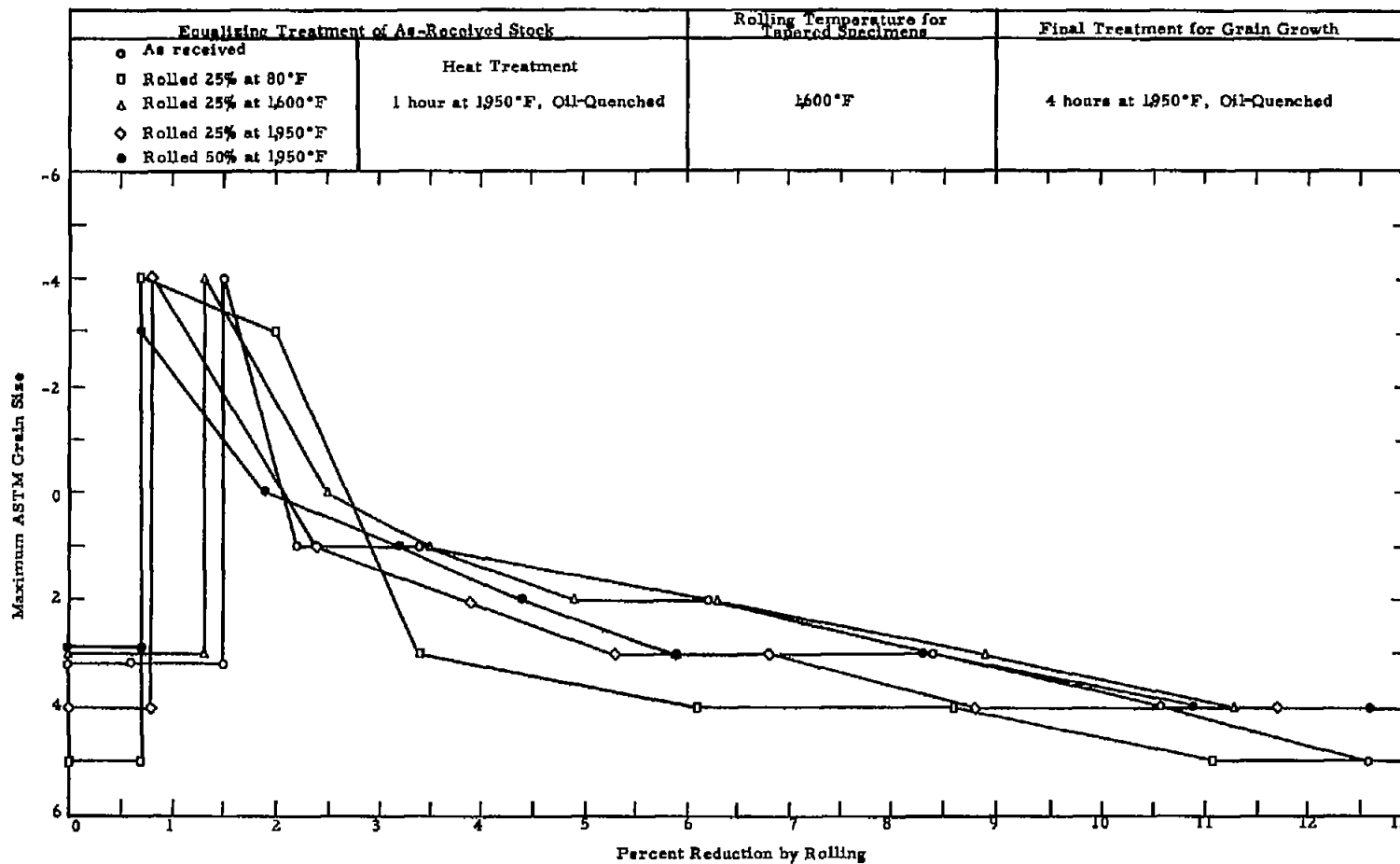


Figure 17.- Effect of equalizing treatment and percent reduction by rolling at 1,600° F upon maximum grain size of air-melted Waspaloy after final solution treatment.

<p>Equalizing Treatment of As-Received Stock</p>
<p>1 hour at 1,950°F, oil-quenched, + 25% reduction at 80°F, + 1 hour at 1,950°F, oil-quenched</p>

Tensile Test Temperature, °F	Elongation, percent	Approximate Distribution of Grain Sizes After Final Solution Treatment
1,400	1.0	
1,600	1.0	
1,600	2.5	

<p>Final Treatment for Grain Growth</p>
<p>4 hours at 1,950°F, oil-quenched</p>

Figure 18.- Effect of temperature and percent elongation by tensile testing upon grain size of air-melted Waspalloy after final solution treatment.

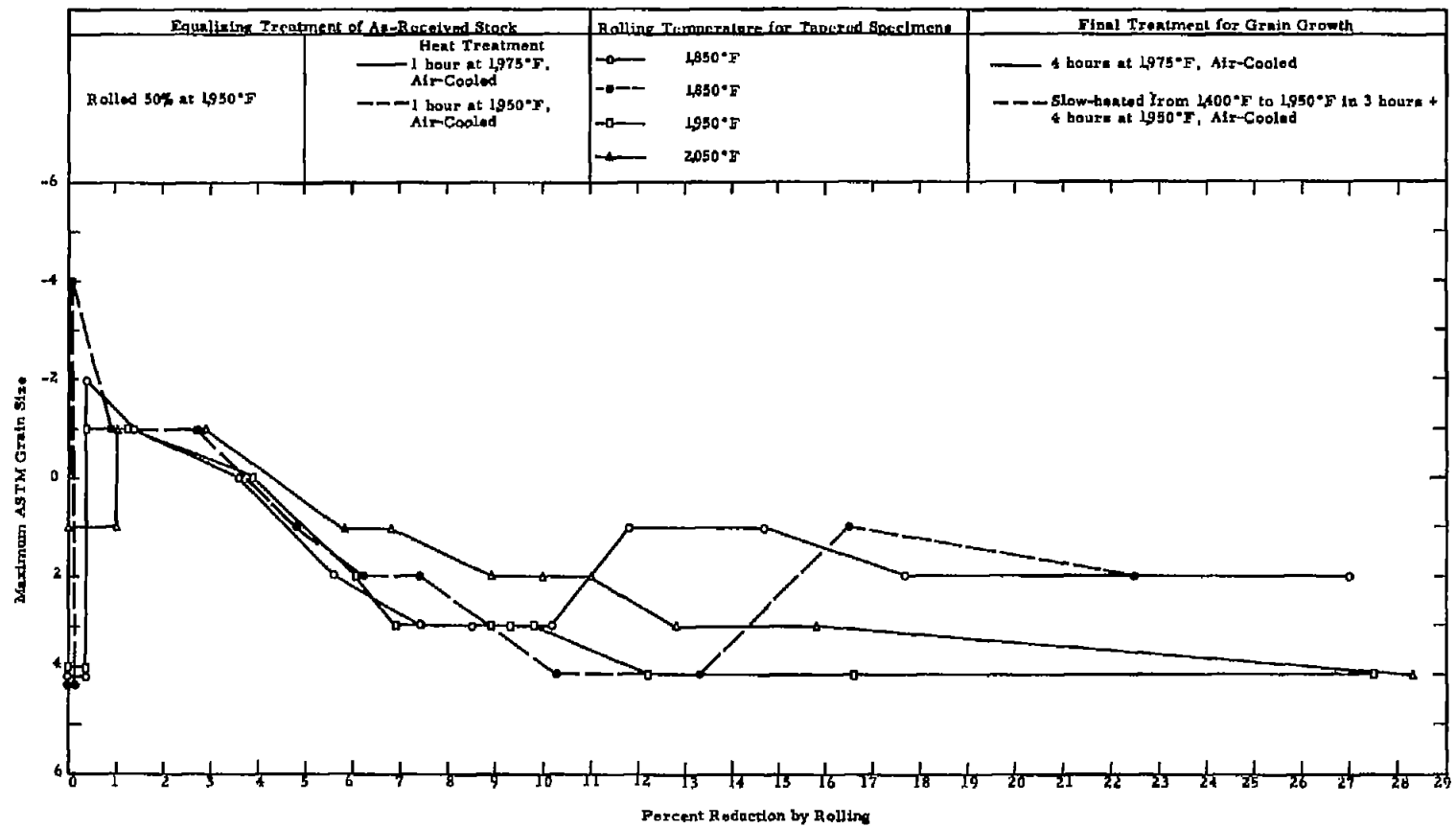


Figure 19.- Effect of rolling temperature and percent reduction upon maximum grain size of air-melted Waspaloy after final solution treatment.



(a) Microstructure as rolled.
14-percent reduction;
rolled at 1,850° F.
Magnification, X50.

(b) Microstructure as rolled.
14-percent reduction;
rolled at 1,850° F.
Magnification, X50.



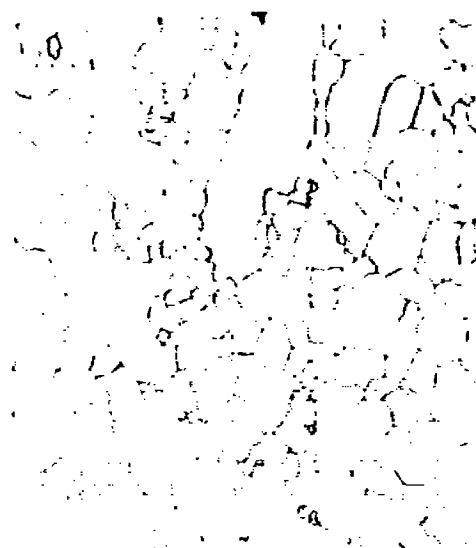
(c) Microstructure after final solution treatment. 14-percent reduction;
rolled at 1,850° F. Magnification, X50.

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Figure 20.- Effect of partial simultaneous recrystallization during reduction by rolling at 1,850° and 1,950° F upon grain size of air-melted Waspaloy after final solution treatment of 4 hours at 1,975° F, then air-cooled.



(d) Microstructure as rolled.
16-percent reduction;
rolled at 1,950° F.
Magnification, X50.



(e) Microstructure as rolled.
16-percent reduction;
rolled at 1,950° F.
Magnification, X500.

(f) Microstructure after final solution treatment. 16-percent reduction;
rolled at 1,950° F. Magnification, X50.

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Figure 20.- Concluded.

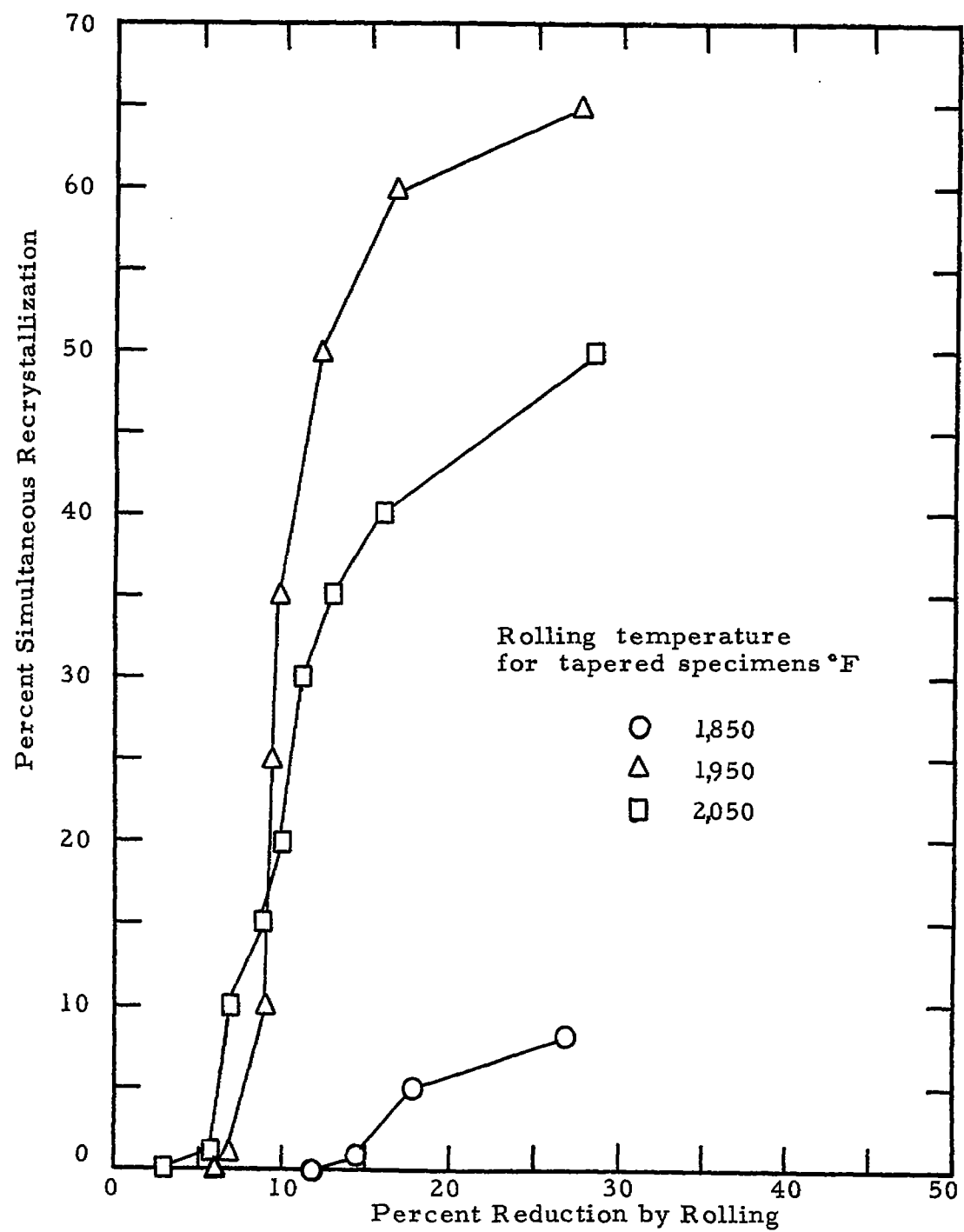
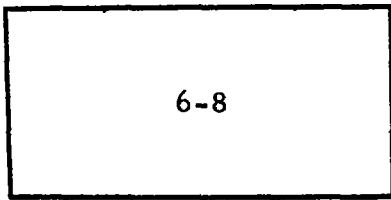


Figure 21.- Effect of percent reduction by rolling upon percent simultaneous recrystallization of air-melted Waspaloy.

As-Received
 6 - 8

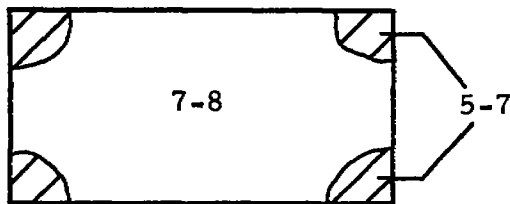
Heat-Treating Temperature, °F	Heat Treating Time, hr		
	1	4	6
1900			
2000			
2100			
2200			

Graph showing the effect of heat-treating time and temperature upon grain size of transverse sections of as-received Inconel X-550 bar stock.



(a) Approximate distribution of grain sizes as received:

(b) Microstructure as received. Magnification, X50.

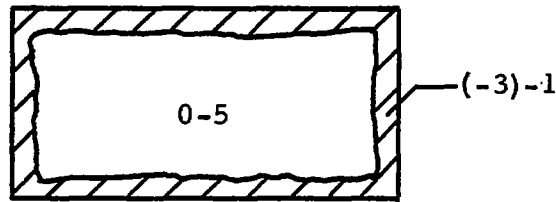


(c) Approximate distribution of grain sizes as received plus 2 hours at 1,900° F, then air-cooled.

(d) Microstructure as received plus 2 hours at 1,900° F, then air-cooled. (Center of bar stock.) Magnification, X50.

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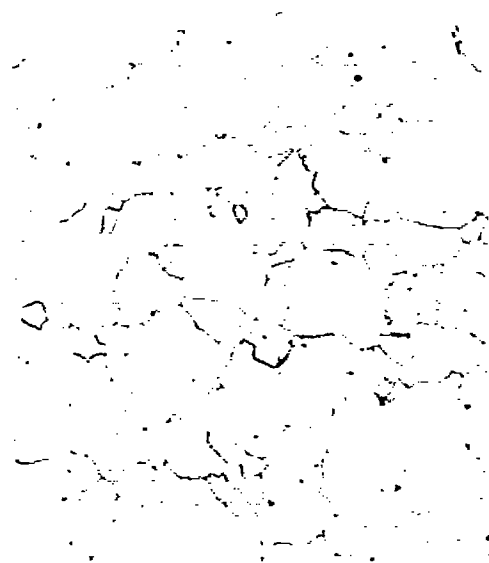
Figure 23.- Microstructure and grain sizes of transverse sections of Inconel X-550 bar stock.



(e) Approximate distribution of grain sizes as received plus 2 hours at 2,100° F, then air-cooled.



(f) Microstructure as received plus 2 hours at 2,100° F, then air-cooled. (Junction between fine and coarse grains.) Magnification, X50.



(g) Microstructure as received plus 2 hours at 2,100° F, then air-cooled. (Center of bar stock.) Magnification, X50.

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Figure 23.- Concluded.

As-Equalized

5 - 8

89



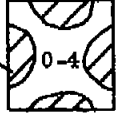

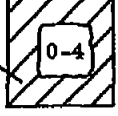

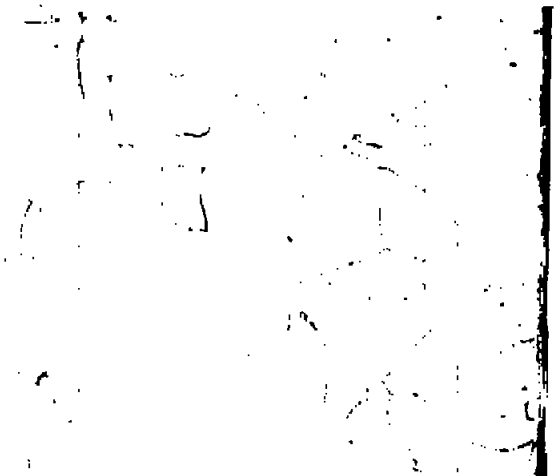
Heat Treatment	Cooling Method	
	Air-Cooled	Water-Quenched
One cycle of 1 hour at 2,150°F, cooled	0 - 4	0 - 4
Two cycles of 1 hour at 2,150°F, cooled	0 - 4	(-3)-2  0 - 4
Three cycles of 1 hour at 2,150°F, cooled	0 - 4	(-1)-2  0 - 4
Four cycles of 1 hour at 2,150°F, cooled	(-1)-2  0 - 4	(-3)-2  0 - 3
Five cycles of 1 hour at 2,150°F, cooled	(-2)-2  0 - 4	(-4)-(-1)  2 - 3

Figure 24.- Effect of repeated heating and cooling upon grain size of transverse sections of Inconel X-550 bar stock which had been equalized by a 64-percent reduction at 2,150° F.

NACA TN 14082



(a) 1 hour at 2,150° F,
then air-cooled.



(b) 5 cycles of 1 hour at
2,150° F, then air-cooled



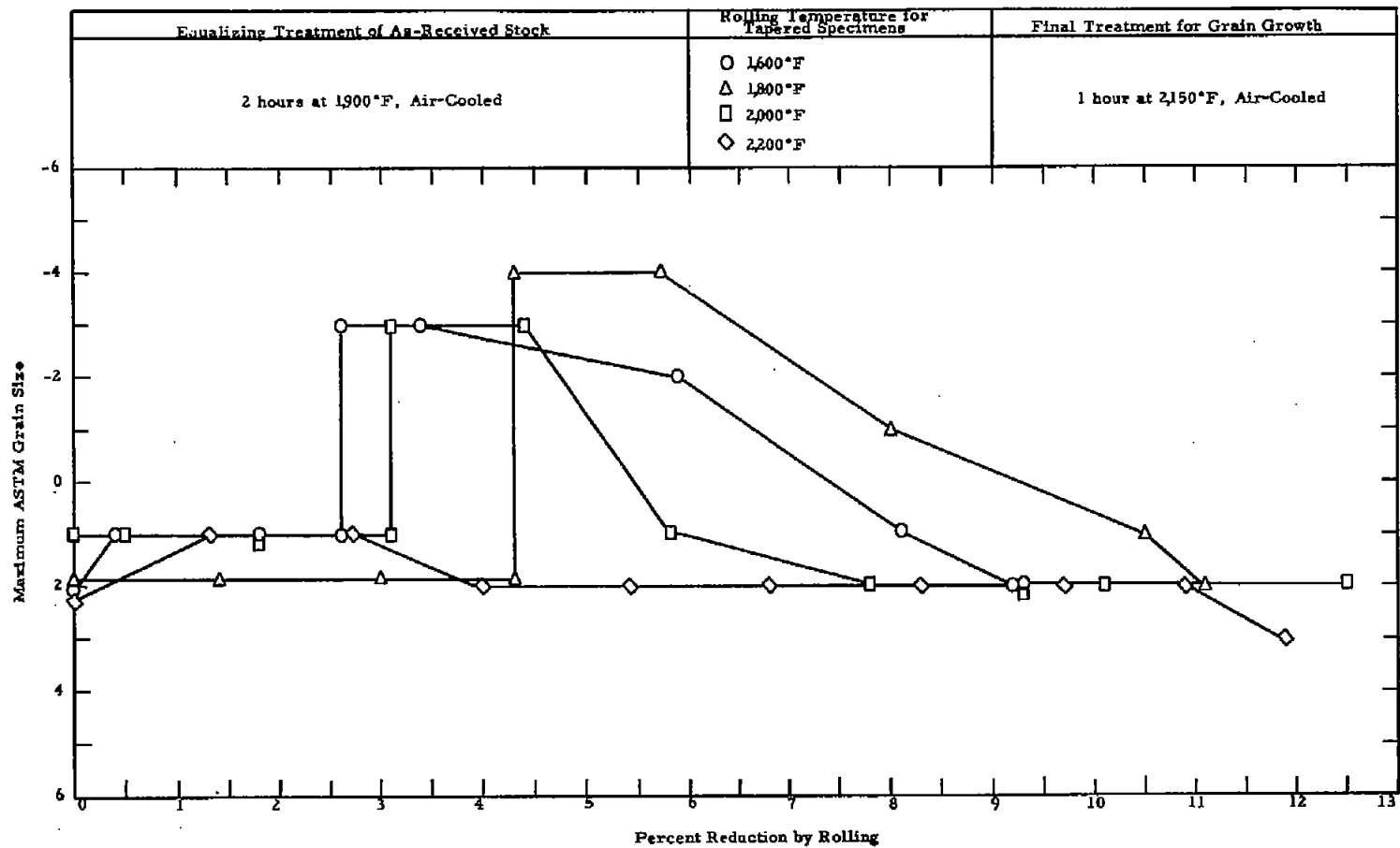
(c) 1 hour at 2,150° F,
then water-quenched.



(d) 5 cycles of 1 hour at
2,150° F, then water-
quenched.

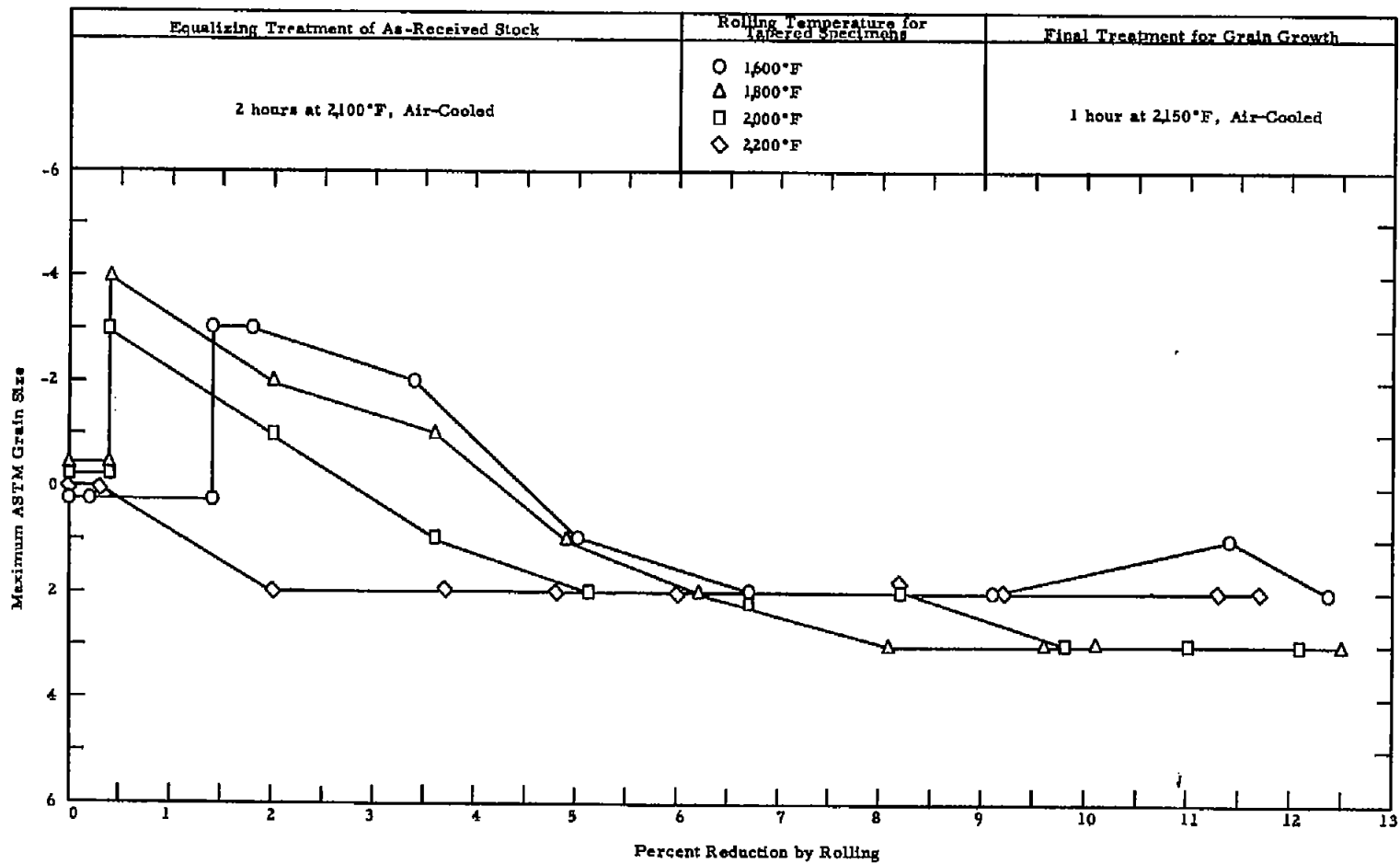
Figure 25.- Effect of repeated heating and cooling upon microstructure of Inconel X-750 bar stock which had been equalized by a 64-percent reduction at 2,150° F. (Transverse section at bar-stock surface.) Magnification, X50.

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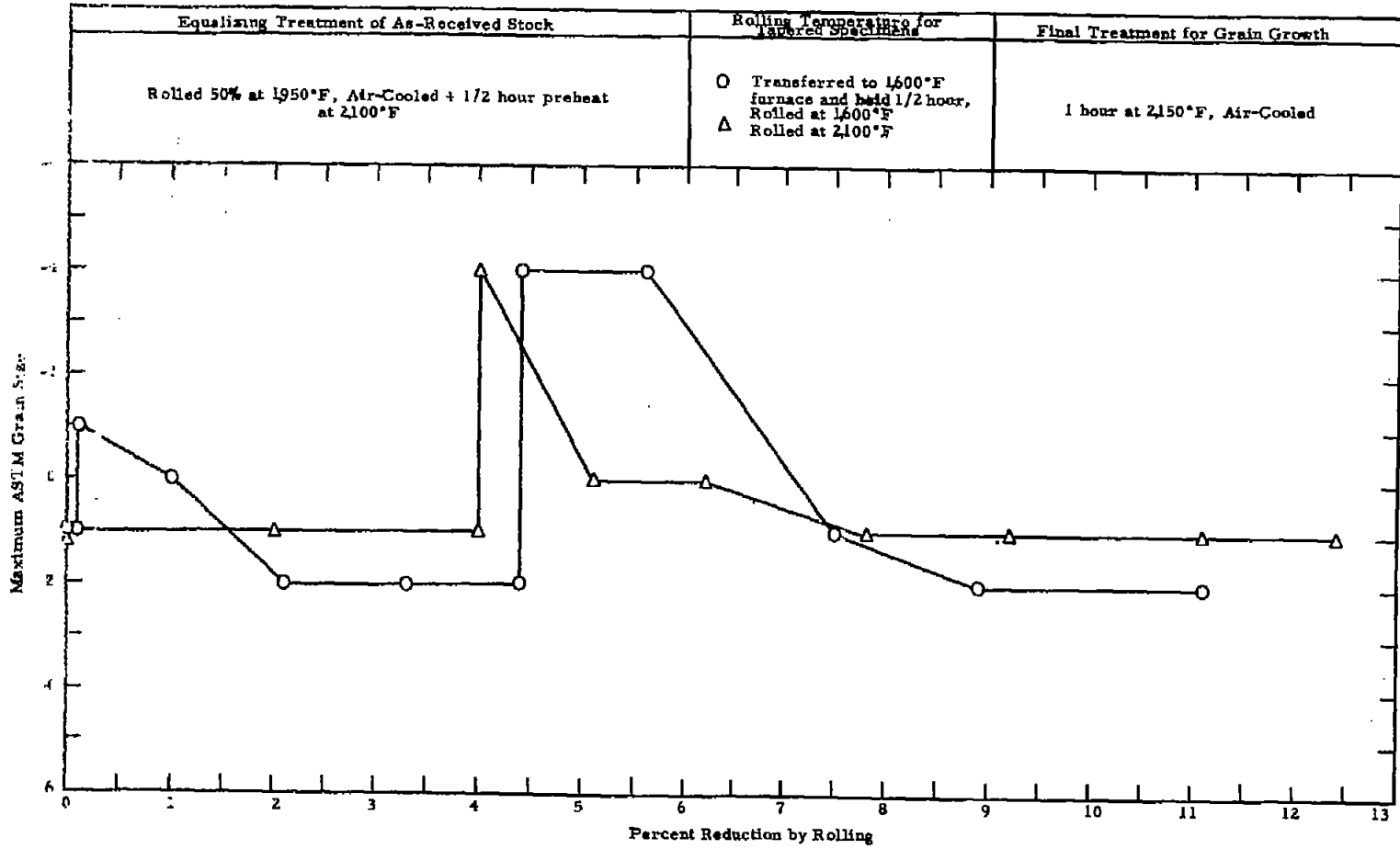
(a) Grain size after equalizing treatment was 7 to 8.

Figure 26.- Effect of rolling temperature and percent reduction upon maximum grain size of Inconel X-550 after final solution treatment.



(b) Grain size after equalizing treatment was 0 to 5.

Figure 26.- Continued.



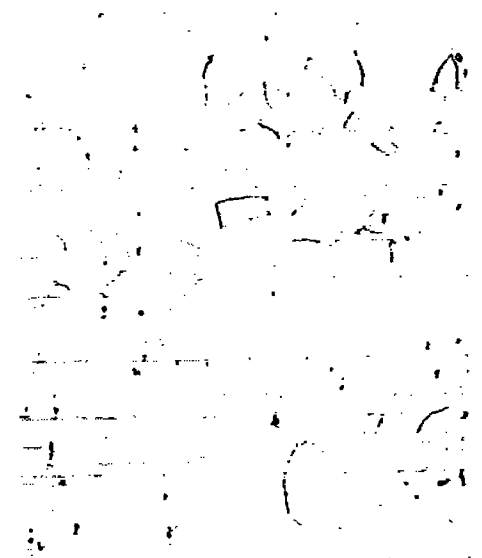
(c) Equalizing treatment included rolling at 1,950° F and preheat at 2,100° F.

Figure 26.- Concluded.



(a) Zero-percent reduction.

(b) 4.0-percent reduction.

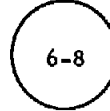


(c) 11.1-percent reduction.

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Figure 27.- Effect of percent reduction by rolling at 2,100° F upon microstructure of equalized Inconel X-550 after final solution treatment. Equalizing treatment of as-received stock was a 50-percent reduction at 1,950° F plus $\frac{1}{2}$ -hour preheat at 2,100° F before rolling. Final solution treatment was 1 hour at 2,150° F, then air-cooled. Magnification, X50.

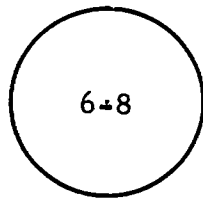
As-Received



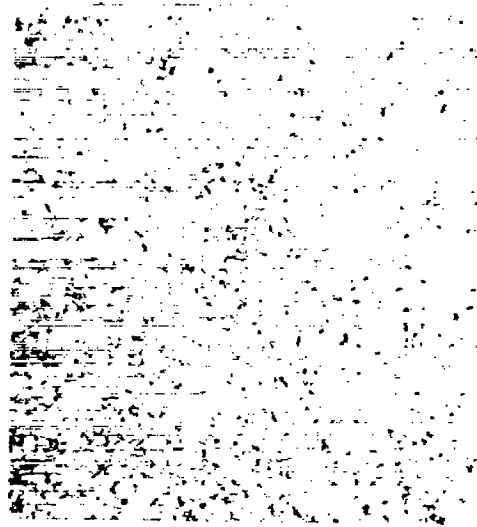
66

Heat-Treating, Temperature °F	Heat-Treating Time, hr		
	1	4	6
1950			
2100			
2150			
2200			
2300			

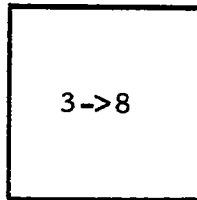
Figure 28.- Effect of heat-treating time and temperature upon grain size of transverse sections of as-received Nimonic 80A bar stock.



(a) Approximate distribution of grain sizes as received.



(b) Microstructure as received. Magnification, X50.



(c) Approximate distribution of grain sizes after equalizing with 50-percent reduction at 1,950° F plus 1 hour at 1,950° F, then air-cooled.

(d) Microstructure after equalizing with 50-percent reduction at 1,950° F plus 1 hour at 1,950° F, then air-cooled. Magnification, X50.

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Figure 29.- Microstructures and grain sizes of transverse sections of as-received and equalized Nimonic 80A bar stock.

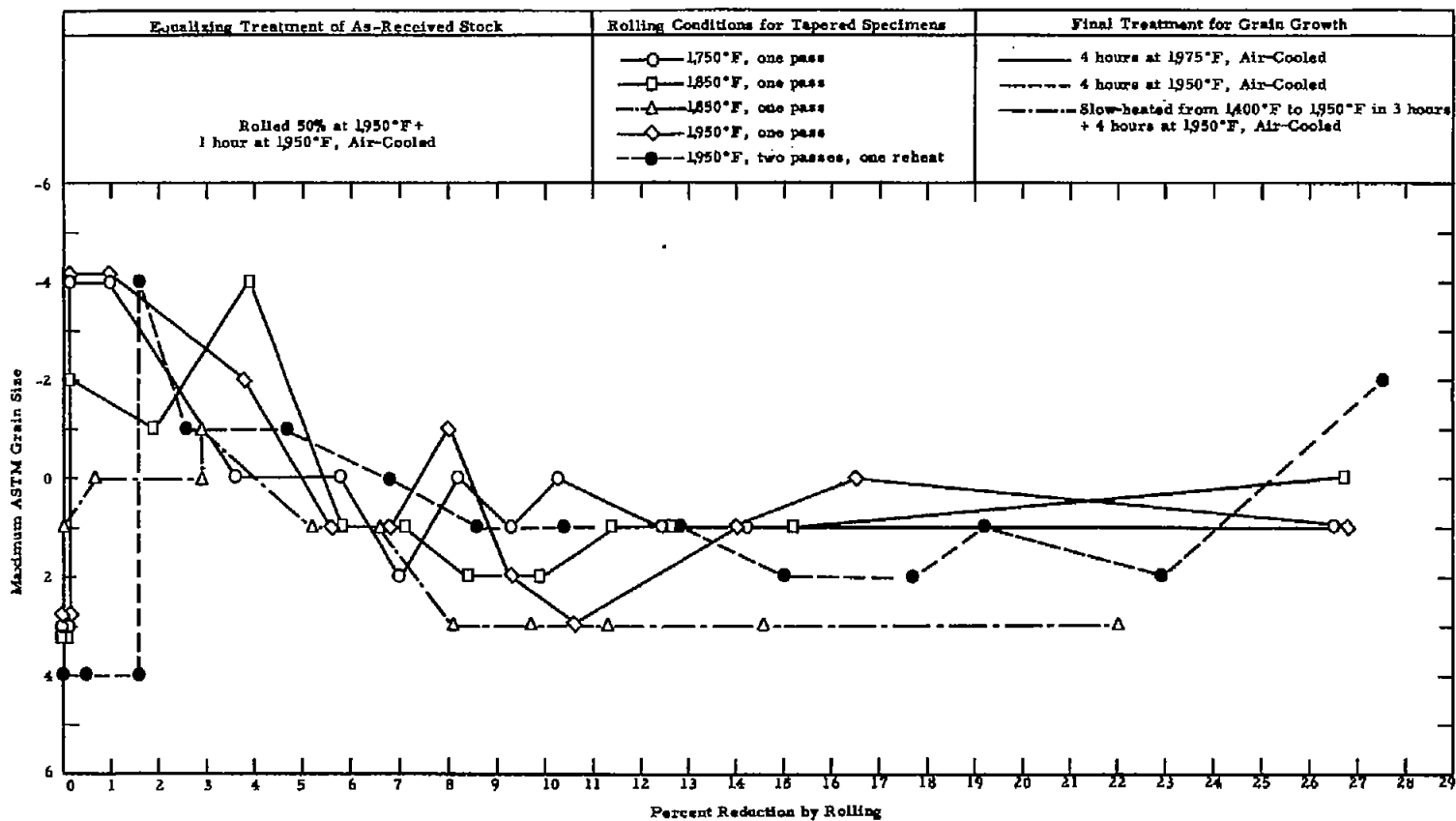


Figure 30.- Effect of temperature and percent reduction by rolling, repeated deformation by rolling, and heating rate before final solution treatment upon maximum grain size of Nimonic 80A alloy after final solution treatment.

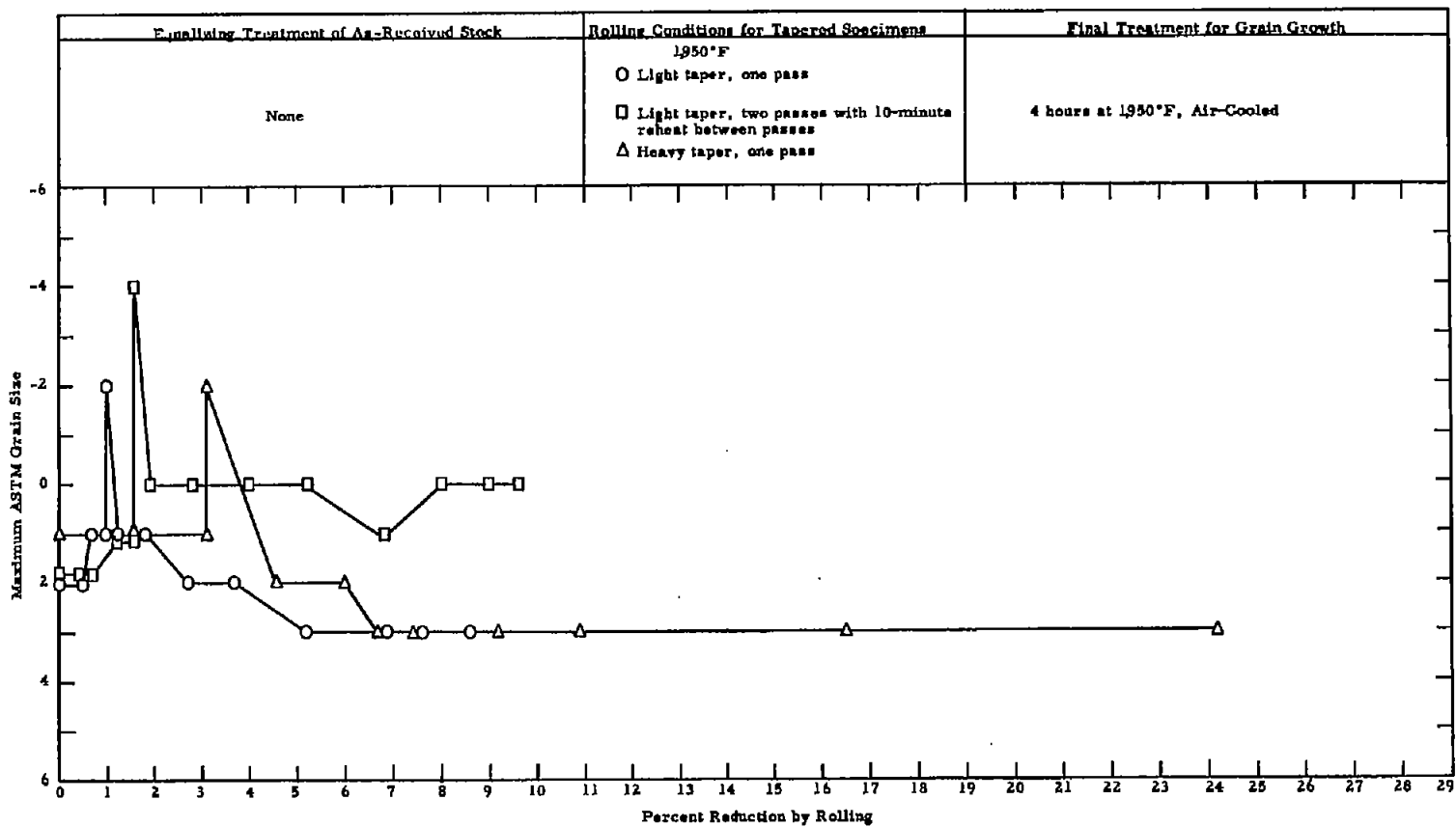


Figure 31.- Effect of degree of taper, repeated deformation, and percent reduction by rolling upon maximum grain size of Nimonic 80A alloy after final solution treatment.

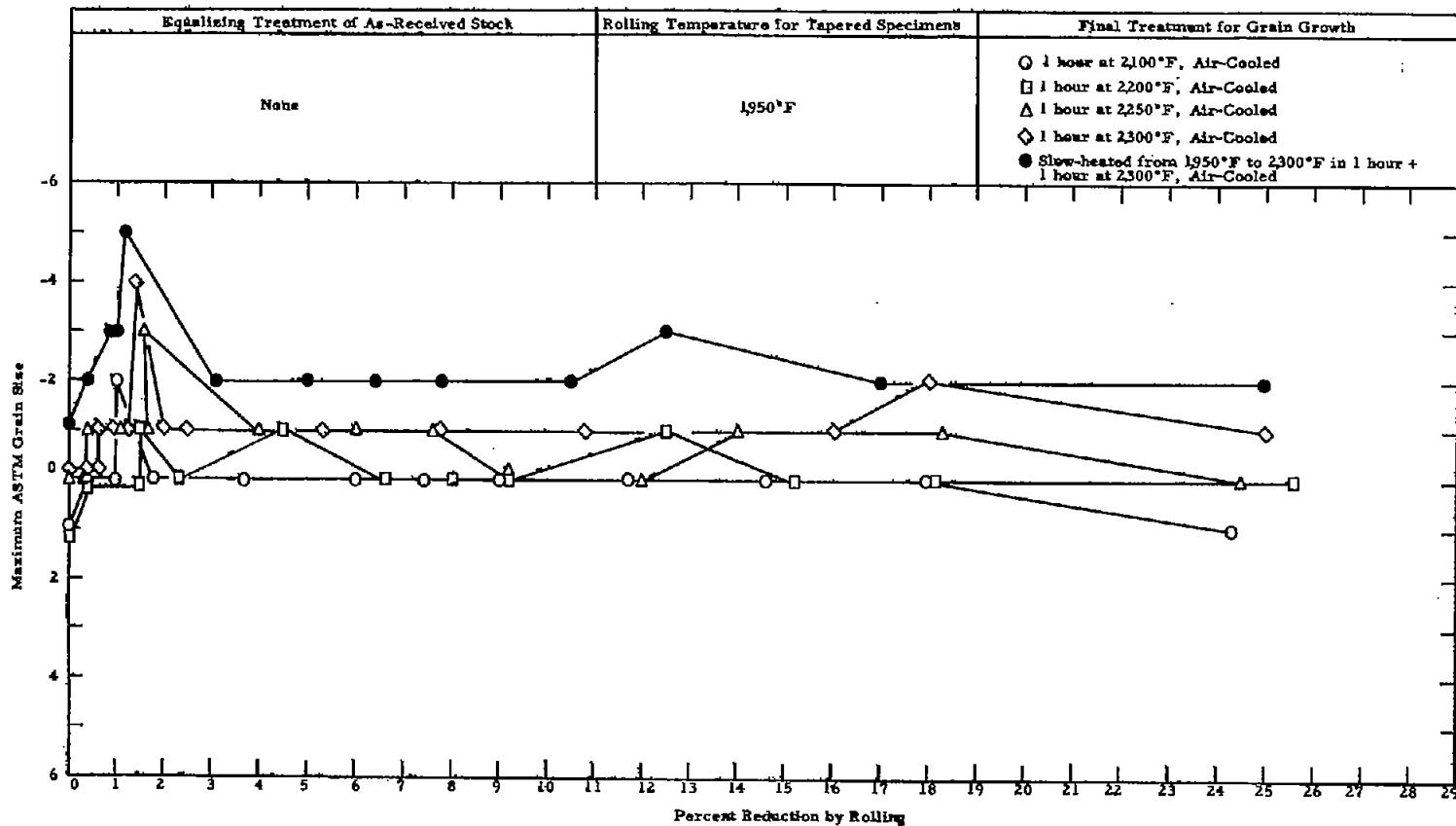


Figure 32.- Effect of final solution treatment and percent reduction by rolling upon maximum grain size of Nimonic 80A alloy.