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Metallurgy and Ceramics

THE RECRYSTALLIZATION AND GRAIN GROWTH OF INCONEL

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INTRODUCTION

The importance of a stable, metallurgical microstructure in the realization of the optimum strength of alloys at elevated temperatures is well known. Experiments at this laboratory have shown that residual cold work in metal specimens may induce recrystallization during service with the result that the creep rate is accelerated and the rupture life is shortened. Since many reactor components are fabricated by pressure-forming methods which introduce various amounts of cold work into the material, an investigation into the recrystallization characteristics of a common high-temperature alloy was initiated. This report discusses the effects of cold work, annealing time, and initial grain size on the recrystallization temperature of Inconel; and presents some preliminary data on the grain-growth characteristics of this alloy.

PROCEDURE

The cold work was introduced by rolling 1/4-in.-thick sheet to reductions ranging from 5 to 80%. The annealing was performed by placing the material in a pre-heated furnace with the temperature controlled to ± 10°F. The estimated heating rate was 190°F/min. Determination of the recrystallization temperature was made by hardness measurements and confirmed by metallographic examination. As an illustration, Fig. 1 is a plot of hardness versus annealing temperature for material with 20 and 50% cold work; the dashed lines indicate the recrystallization temperature. The specimens were held for 15 min at the annealing temperature.

RESULTS

The effect of cold work on the temperature of recrystallization of fine-grain material, held 30 min at temperature, is shown in Fig. 2. It is apparent that this phenomena can occur at temperatures as high as 1750°F when only 5% cold work is present, and as low as 1240°F with 80% cold work. The time the material is held at the annealing temperature can also have a significant effect on the recrystallization, and Fig. 3 illustrates this effect for three different hold times on fine-grain material. Figure 4 depicts the results for identical hold times on coarse-grain material. It can be seen that recrystallization will occur about 100°F lower after being held 60 min at temperature as compared to similar material held for 15 min. A comparison of Figs. 3 and 4 indicate that when 10%, or more, cold work is introduced, initially fine-grain material will recrystallize at lower temperatures than similarly treated coarse grain stock. This is probably due to the greater number of nucleation sites, grain boundaries, and precipitates present in the fine-grain material.

EFFECT OF TEMPERATURE ON GRAIN SIZE

In addition to recrystallization, numerous other metallurgical factors are sensitive to temperature. Two of these, which are somewhat interrelated, are the grain size and the stability of the carbide particles. Since it has been reported that both constant-stress and dynamic-stress properties will be affected by the grain size of Inconel, it is important that some degree of control of this variable be achieved. Therefore, grain-size determinations were made in conjunction with the recrystallization study to determine the stable grain size at annealing temperatures of 1600, 1700, 1800, and 1900°F. Table I shows the mean-grain diameter tabulated with respect to the amount of cold work and time at temperature. The finely dispersed carbide particles are the main agents inhibiting the growth of the grains so that the temperature at which they begin to coalesce, or dissolve, will determine the maximum temperature that Inconel can be subjected to for any extended period without a major change in grain size. Figure 5 is a plot of hardness versus temperature for Inconel with deformations ranging from 0 to 80%. In addition to the sharp drop in hardness characteristic of the recrystallization process, which of course occurs at various temperatures, there is a second drop occurring between 1750 and 1800°F where all specimens begin to soften rapidly. This change in the slope of the hardness curves represents the temperature range in which the carbides are beginning to coalesce, and defines the upper temperature limit where a small grain size can be maintained. The only sharp change in the hardness curve for 5% worked metal occurs at about this same temperature, which makes it difficult to determine if recrystallization

TABLE I

MEAN GRAIN DIAMETER RESULTING FROM INDICATED TREATMENTS

	Rolled Fine-Grain Material				Rolled Coarse-Grain Material			
% Cold Work	Annealed 1800°F 15 min	Annealed 1800°F 120 min	Annealed 1900°F 15 min	Annealed 1900°F 120 min	Annealed 1800°F 15 min	Annealed 1800°F 120 min	Annealed 1900°F 15 min	Annealed 1900°F 120 min
0	2.0 ± 1	2.0 ± 1	3.5 ± 2	6.0 ± 4	6.0 ± 3	3.0 ± 3	6.0 ± 3	8.0 ± 5
5	2.5 ± 1	2.0 ± 1	2.0 ± 1	5.0 ± 3	5.0 ± 3	6.0 ± 3	6.0 ± 3	6.0 ± 4
10	1.5 ± 1	1.5 ± 1	2.0 ± 1	4.0 ± 3	3.0 ± 2	4.0 ± 3	4.5 ± 3	6.0 ± 4
20	1.0 ± 1	2.0 ± 1	2.5 ± 1	4.0 ± 2	2.5 ± 2	3.0 ± 2	4.5 ± 3	4.5 ± 3
80	1.0 ± 0.5	1.0 ± 0.5	2.0 ± 1	2.0 ± 1	1.0 ± 0.5	2.0 ± 1	3.5 ± 2	4.0 ± 3

*The grain diameters are given in thousandths of an inch. The indicated variation is also in thousandths of an inch and represents the spread in grain size in forty measurements. Ninety per cent of the grains were within the indicated size limits.

actually occurs, or whether this is merely softening due to the change in carbide dispersion. Figure 6 shows the grain structure of initially fine-grain material after 5, 10, 20, 50, and 80% deformation. Figures 7 through 10 are photomicrographs of the microstructure resulting from anneals at 1600, 1700, 1800, and 1900°F for 30 min, and Figs. 11 through 14 are a similar series held at the annealing temperature for 2 hr. A comparison of these two series of pictures shows that the grain size reached in the first 30 min did not increase appreciably during the next 90 min. It also appears that a relatively fine-grain size can be maintained after 2 hr at 1800°F but that a coarse-grain size will result from only 30 min at 1900°F. This agrees fairly well with the results of the hardness tests discussed previously. Although the final grain size is markedly affected by the prior cold work for lower annealing temperatures, at 1900°F only a slight refinement in grain size will result from prior cold work. Figure 15 shows the initial structure for coarse-grain material with various amounts of deformation, and Figures 16 through 23 represent the microstructure resulting from a similar annealing treatment to that given the fine-grain stock. A study of this series reveals the following important points:

1. Deformations of less than 20% do not appreciably refine the grain structure.
2. The shape of the deformed coarse-grain remains outlined by the carbide precipitates after recrystallization occurs. The effect of this psuedo-grain structure on mechanical properties is not known, but one may assume that it will not be beneficial.
3. Although cold work in the range of 50 to 80% results in a fine-grain size after 2 hr at 1800°F, this is probably not a stable grain size and a further increase in size approaching that shown by the metal given 5 or 10% cold work seems likely. This will occur as the carbide particles begin to coalesce and dissolve with time, thus unblocking the boundaries and allowing further grain growth to occur.
4. At 1900°F no significant difference in final grain size is obtained regardless of their previous amount of deformation. The

grain size reached in 30 min is essentially the same as that resulting from a 2-hr anneal.

CONCLUSIONS

The results of this investigation show the wide-temperature range over which recrystallization of Inconel is possible. It appears that a temperature of 1700°F will obtain recrystallization of all worked structures regardless of the severity, but that the resulting final grain size, at least for short-exposures, will depend on the degree of prior cold work.

When the material has a coarse-grain size prior to deformation, a somewhat higher temperature for recrystallization is required. Also, the carbides in the grain boundaries of the worked, coarse-grain material remain fixed when recrystallization occurs so that the new grains do not receive the usual contribution to their creep resistance from such particles. Thus it can be assumed that the mechanical properties of such a structure would be inferior to material of a similar grain size, but with the carbides more favorably situated. This mixed structure is still found in some cases after a 2-hr anneal at 1800°F, and therefore is not completely eliminated until treated at 1900°F.

Finally, it appears to make little difference to the grain size resulting from a 1900°F anneal, whether the material was initially fine or coarse grain, or the degree of prior cold work to which it was subjected.

ACKNOWLEDGEMENT

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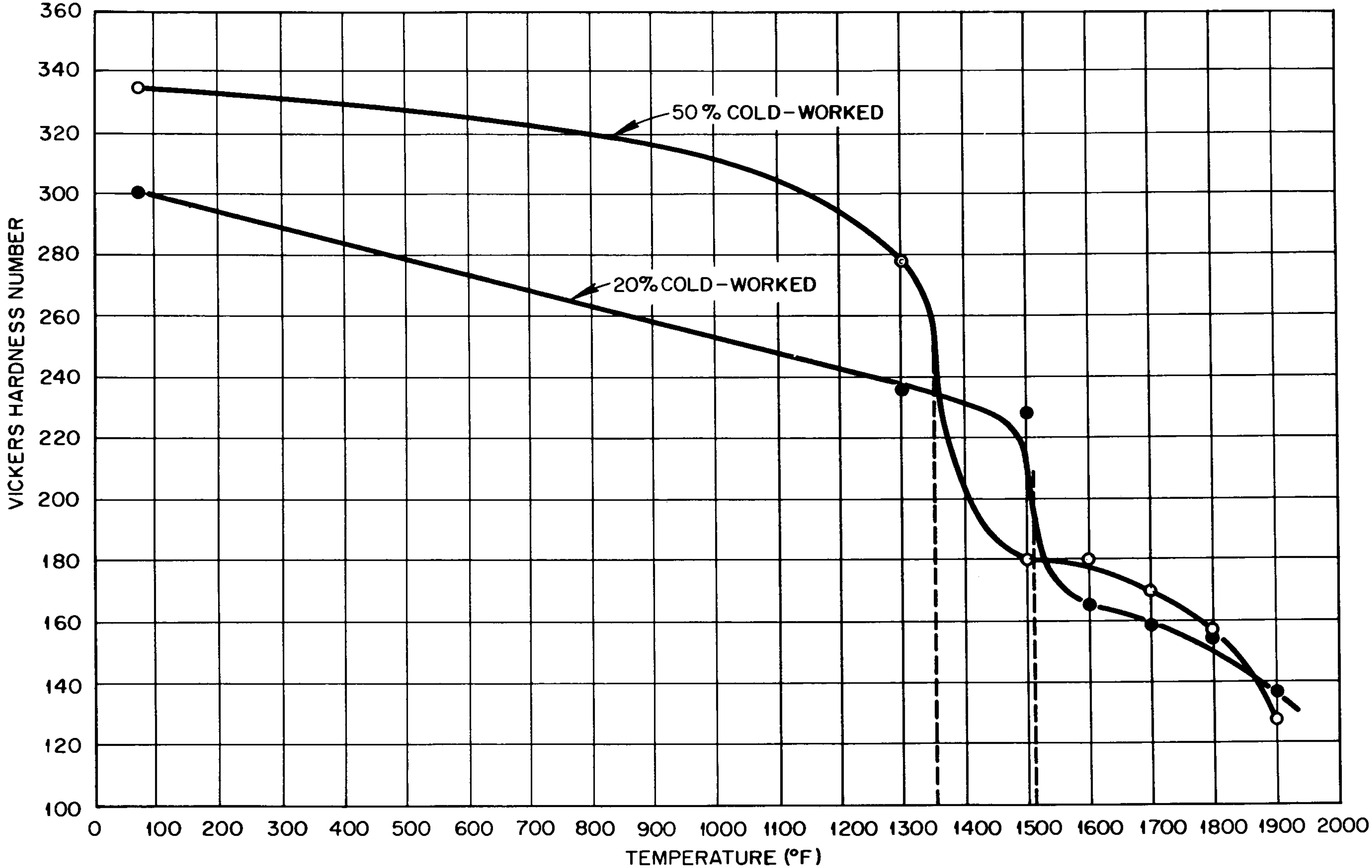

Fig. 1. Illustration of the Method Used to Determine the Recrystallization Temperature.

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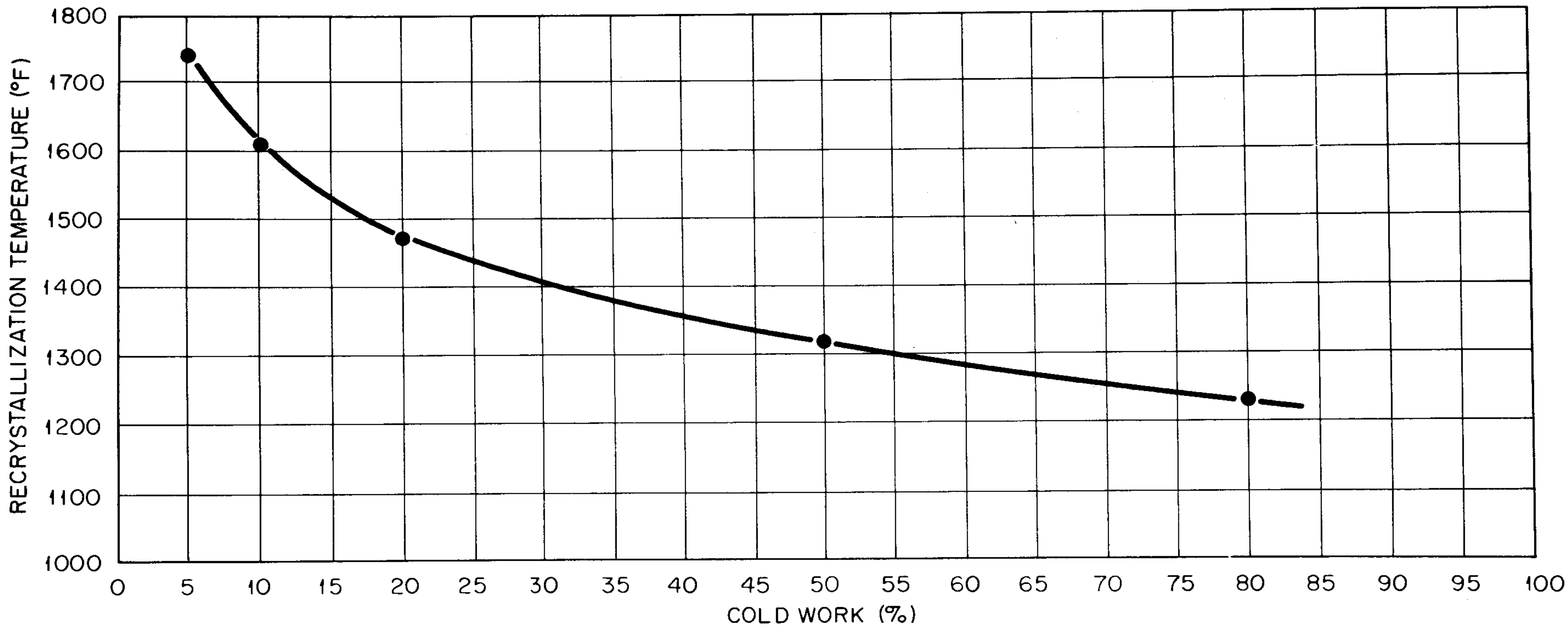

Fig. 2. Effect of Cold Work on the Recrystallization Temperature of Fine Grain Inconel.

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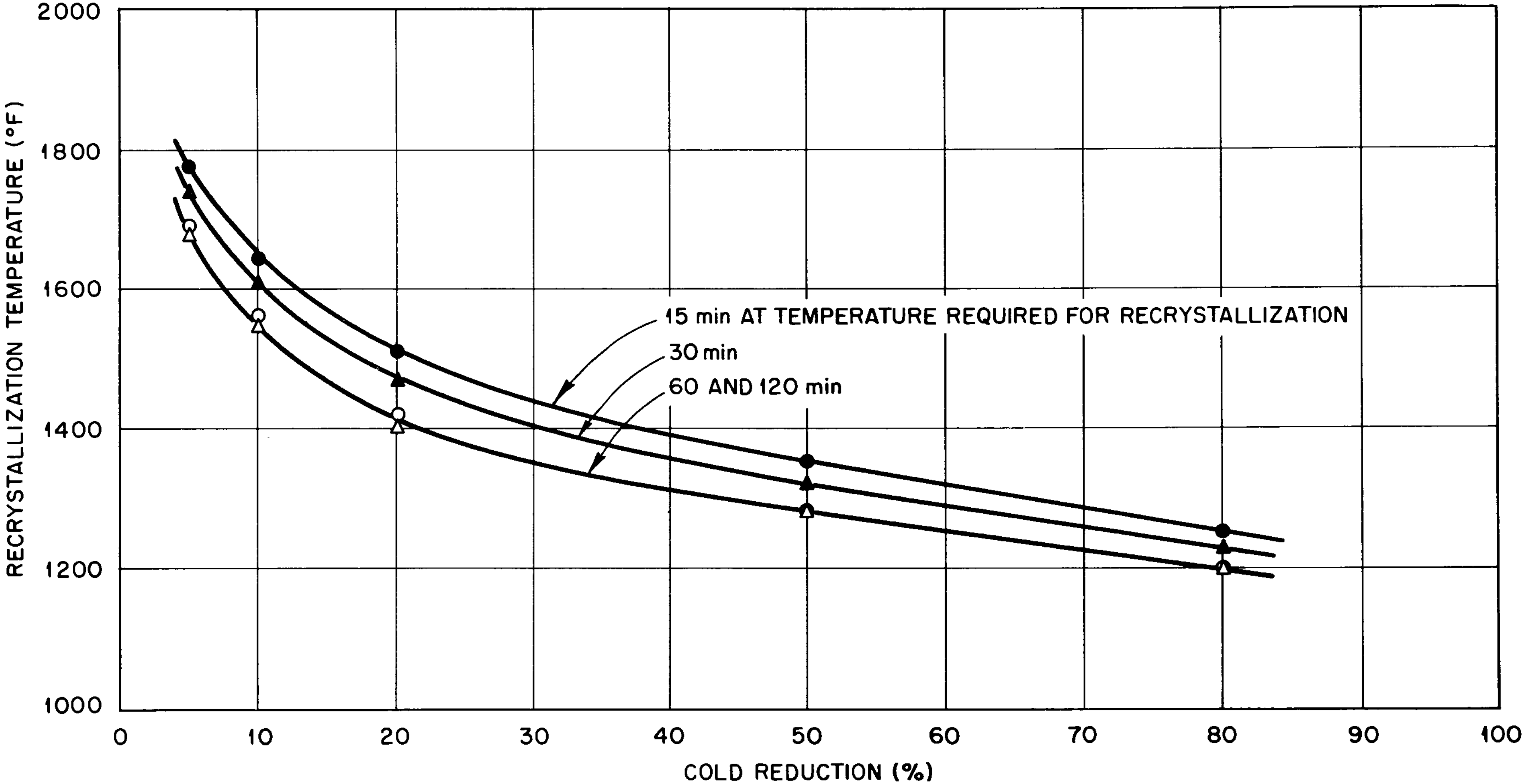

Effect of Cold Reduction on Recrystallization Temperature of Fine – Grained Inconel.

Fig. 3.

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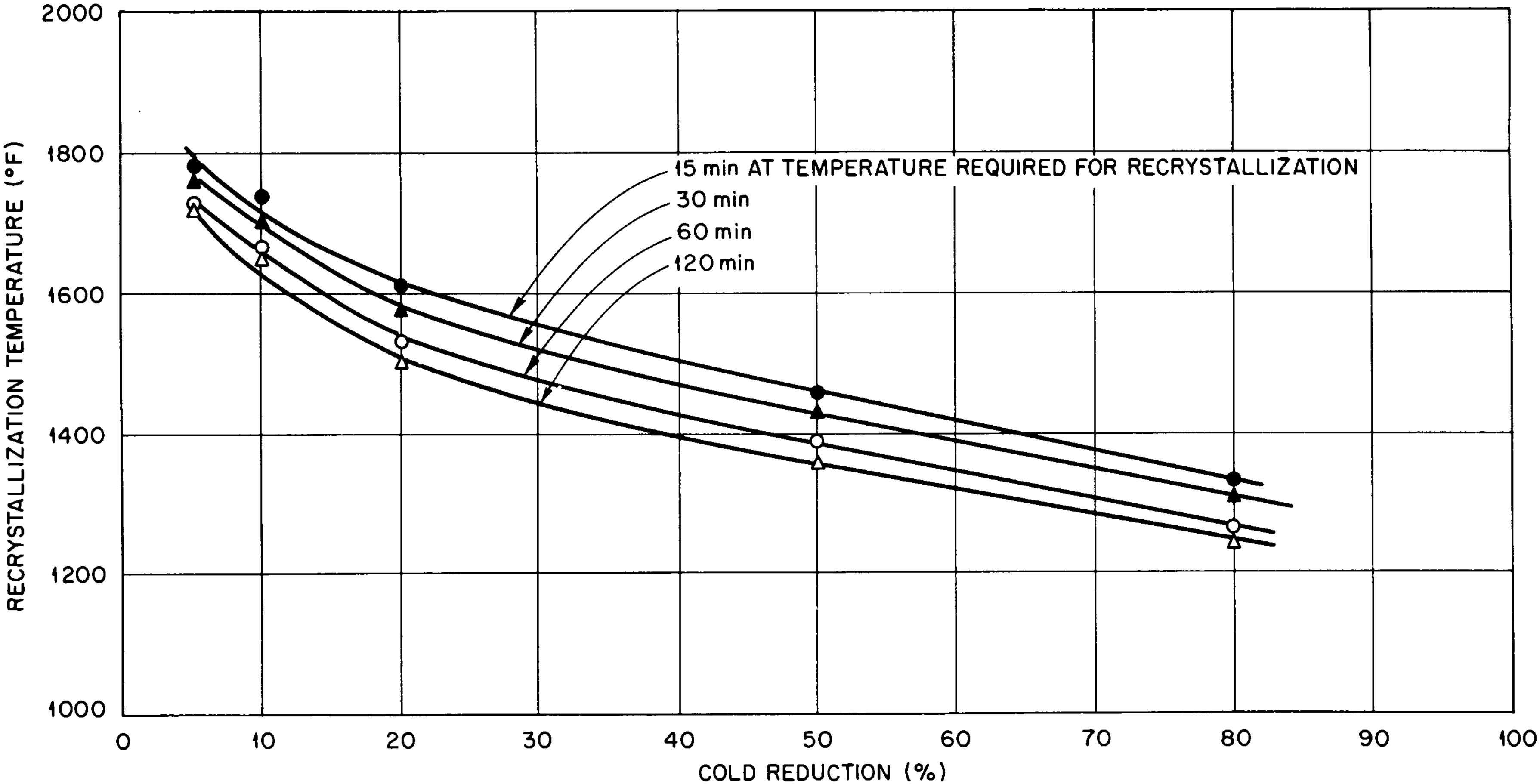

Effect of Cold Reduction on Recrystallization Temperature of Coarse-Grained Inconel.

Fig. 4.

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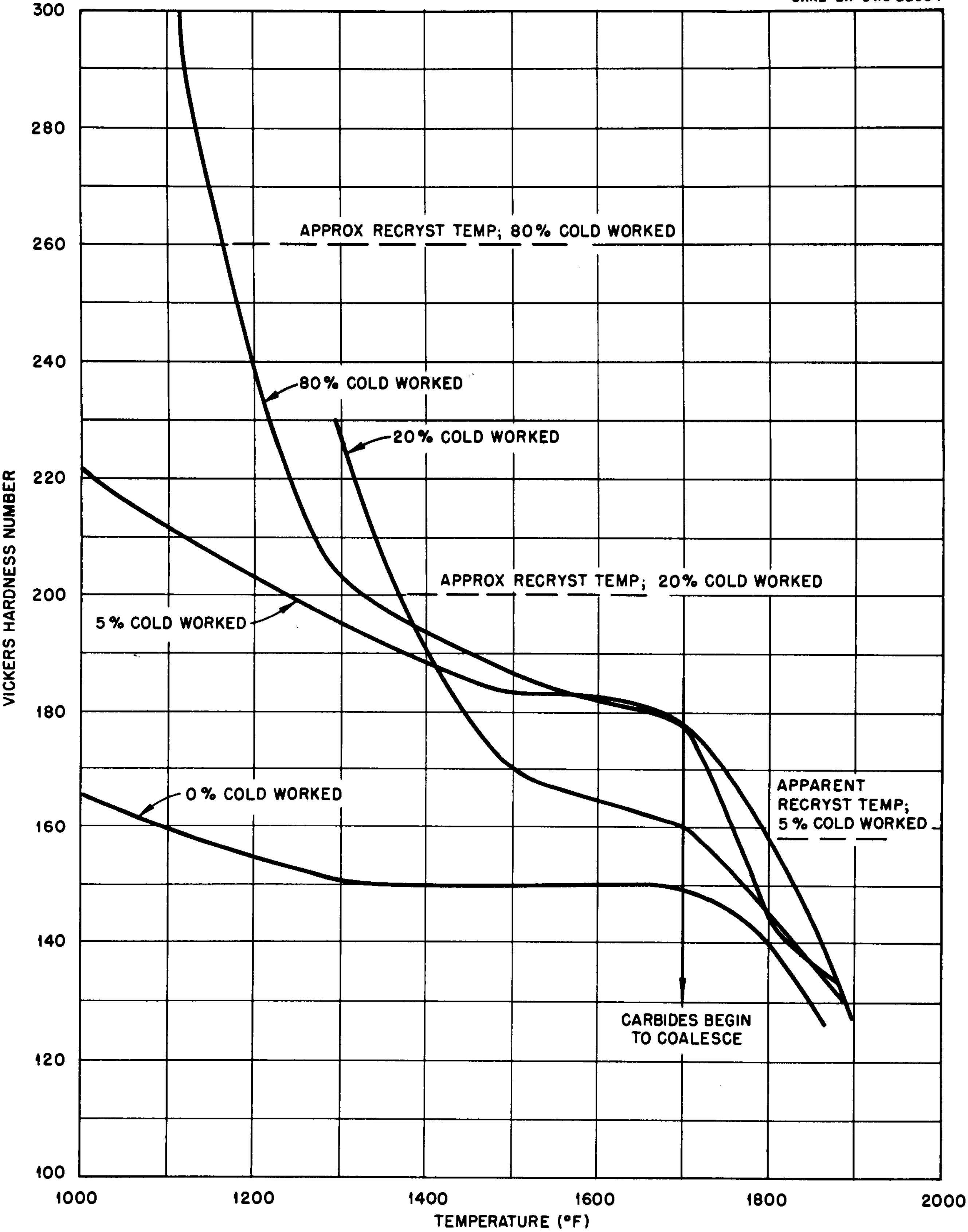


Fig. 5. Fine Grain Inconel Held 30 min at Temperature.

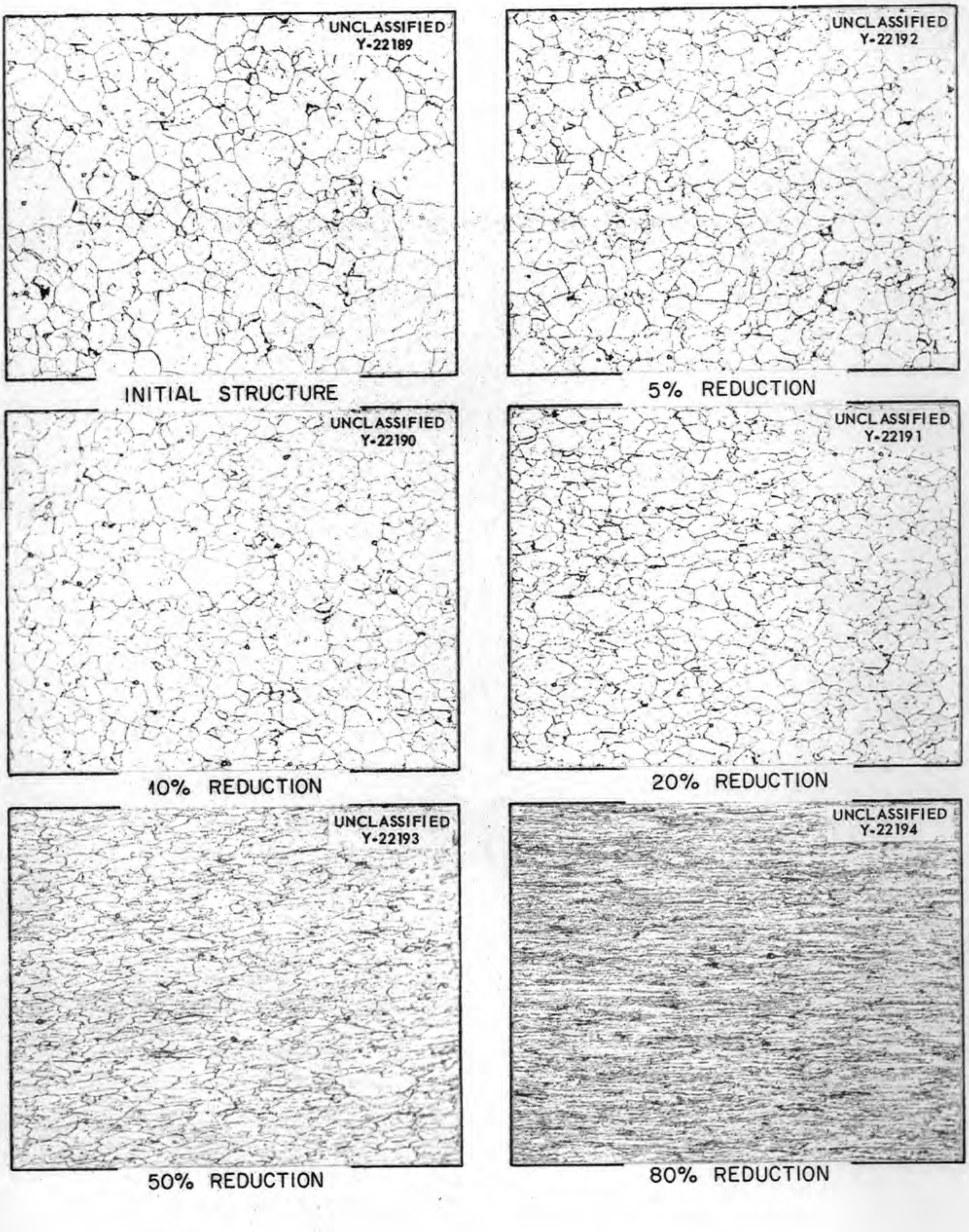
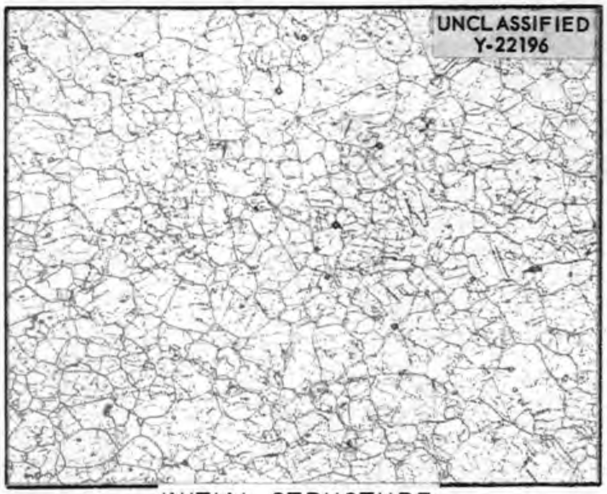


FIG. 6 INITIALLY FINE GRAIN AS WORKED.



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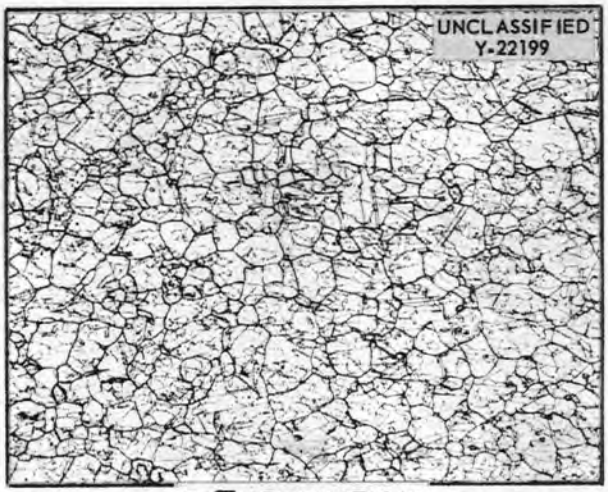

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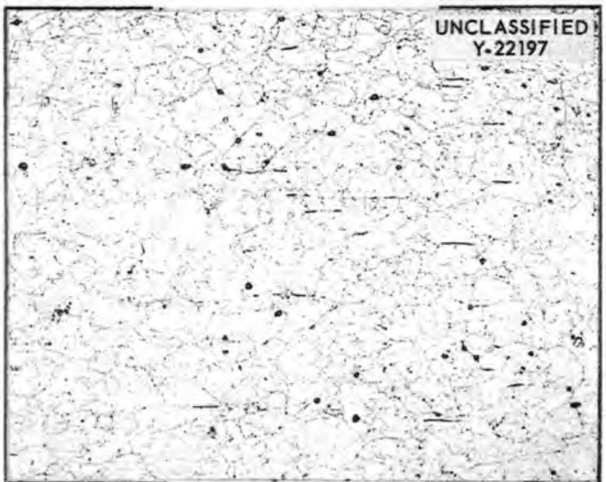

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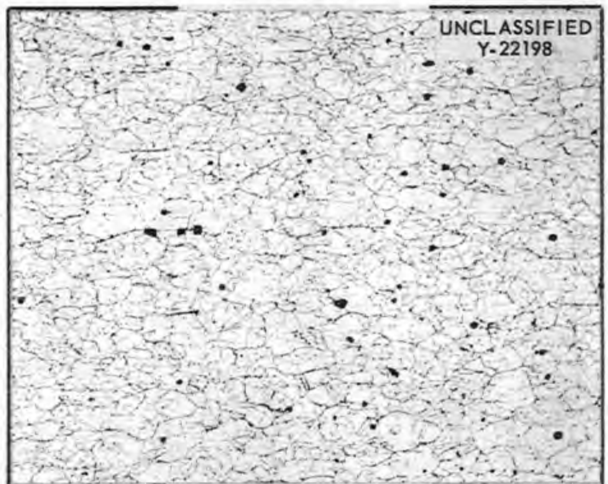

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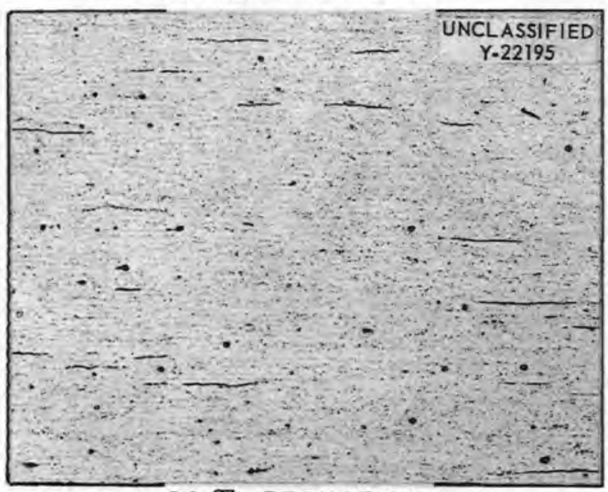
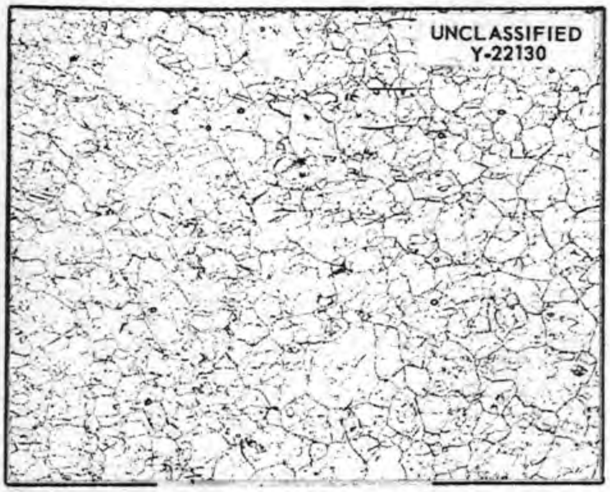


80 % REDUCTION

FIG. 7 INITIALLY FINE GRAIN ANNEALED 1600°F FOR 30 MIN.



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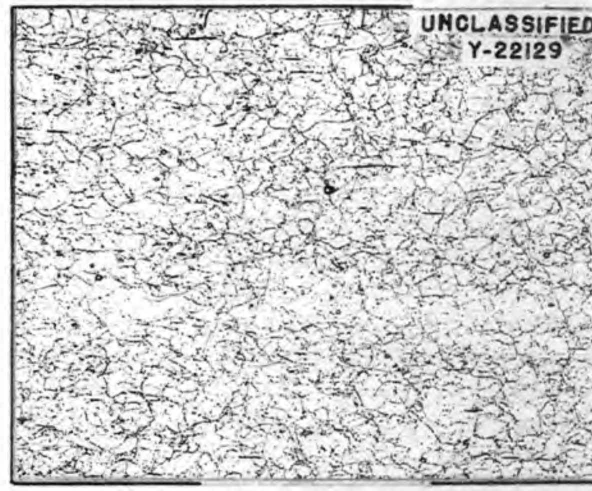

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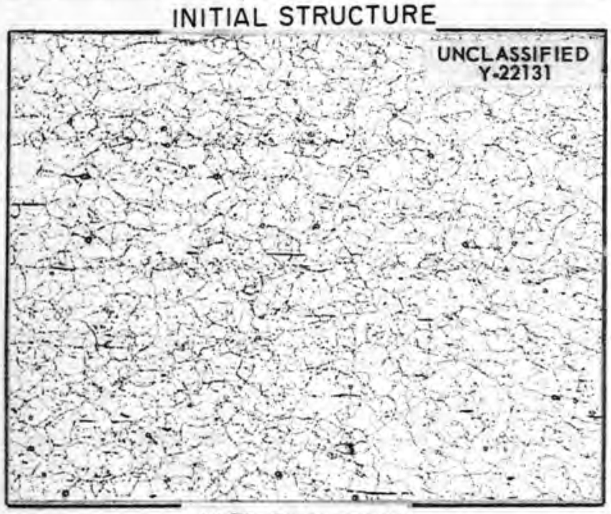

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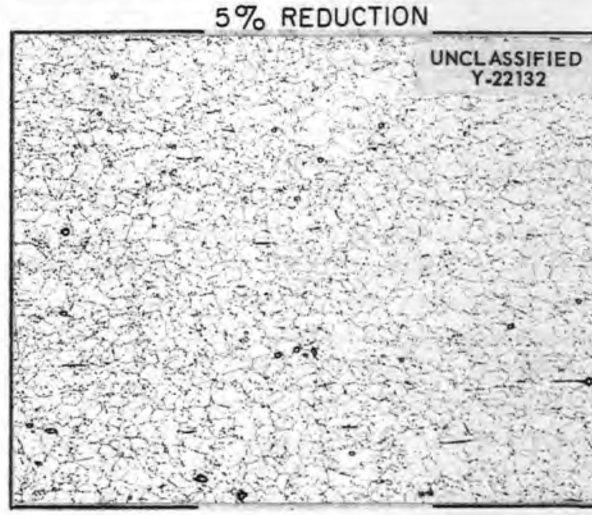

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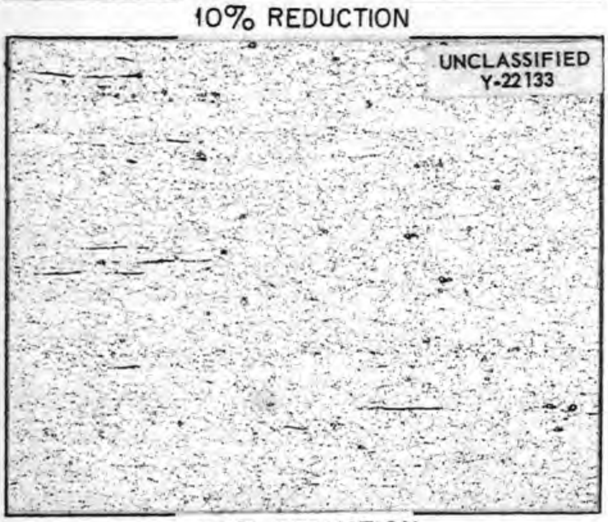

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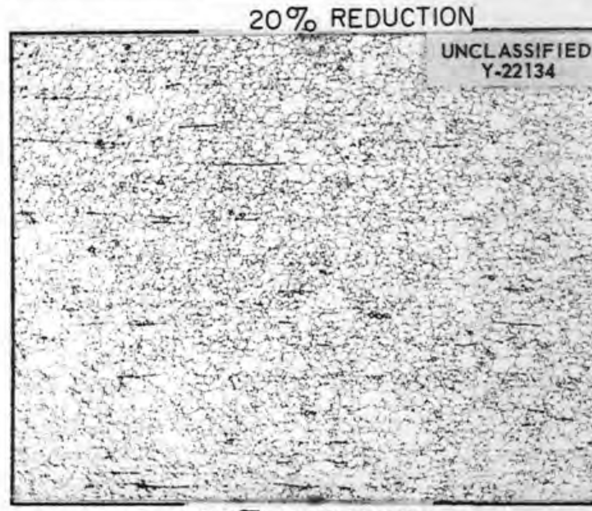


80% REDUCTION

FIG. 8 INITIALLY FINE GRAIN ANNEALED 1700°F FOR 30 MIN.

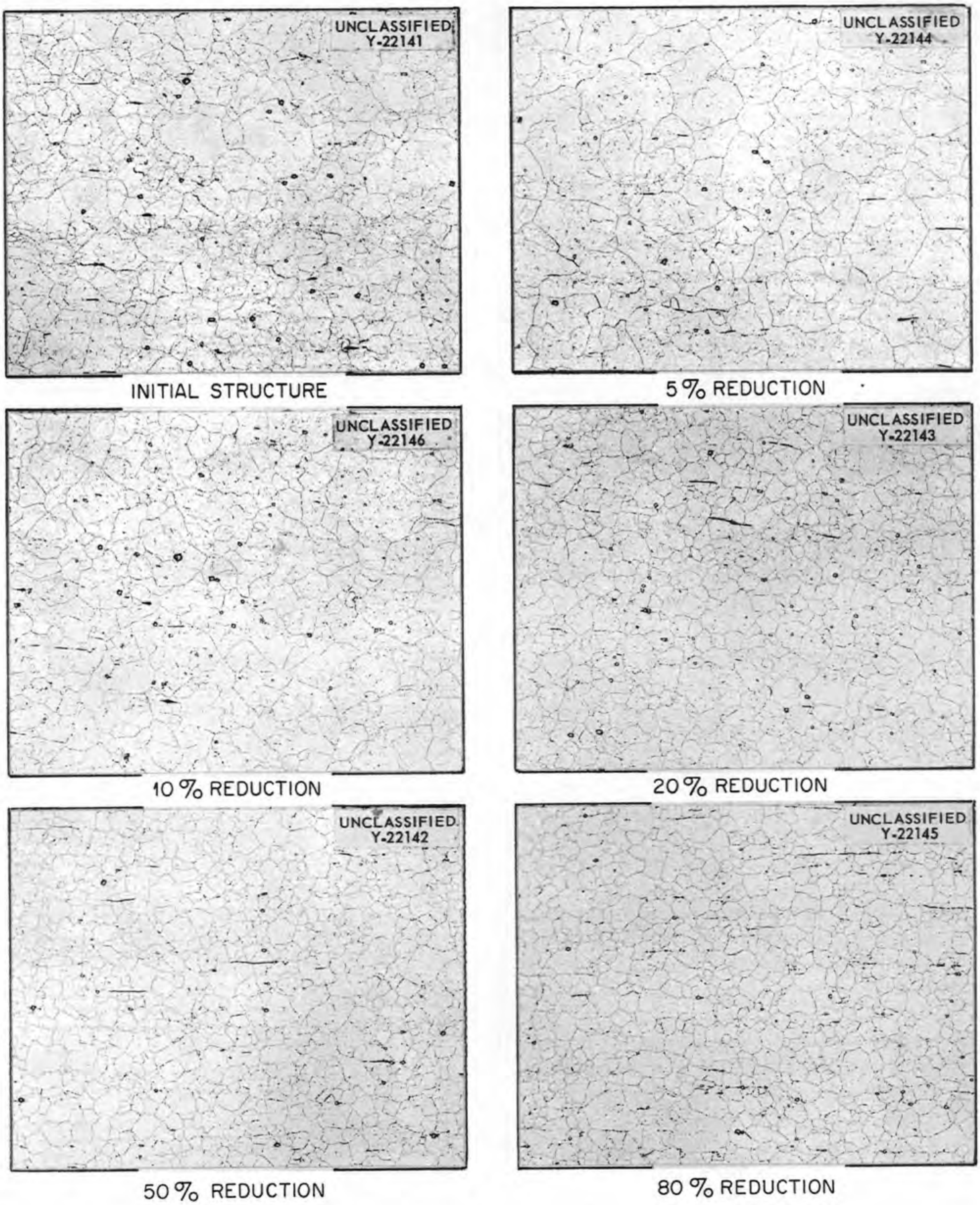
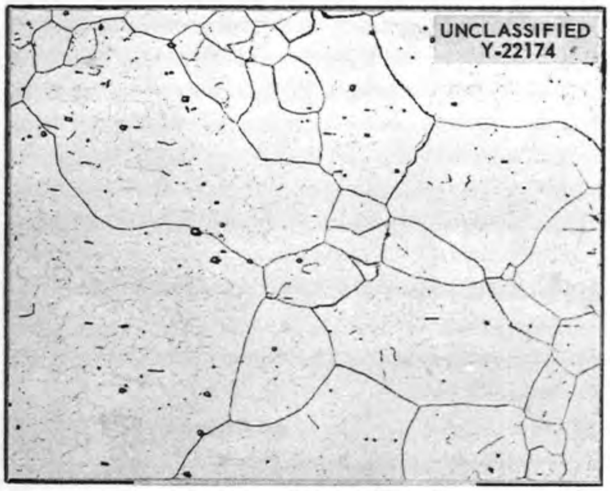


FIG. 9 INITIALLY FINE GRAIN ANNEALED 1800°F FOR 30 MIN.



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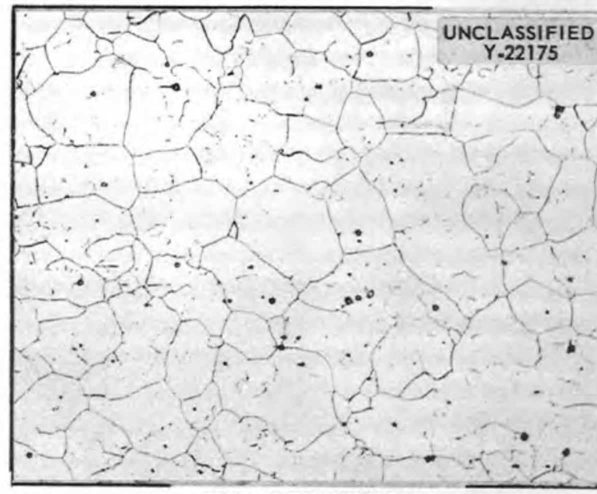

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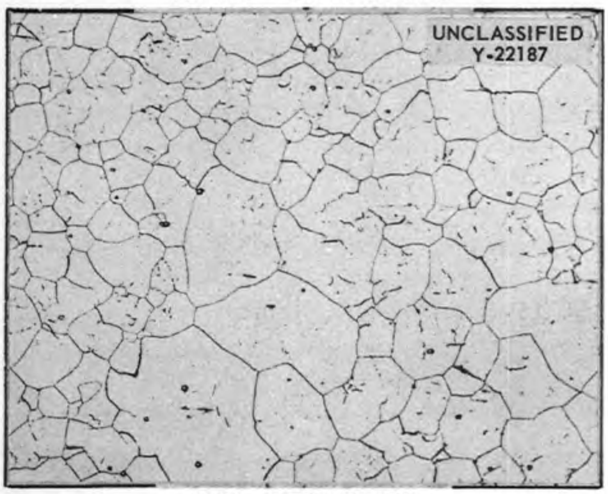

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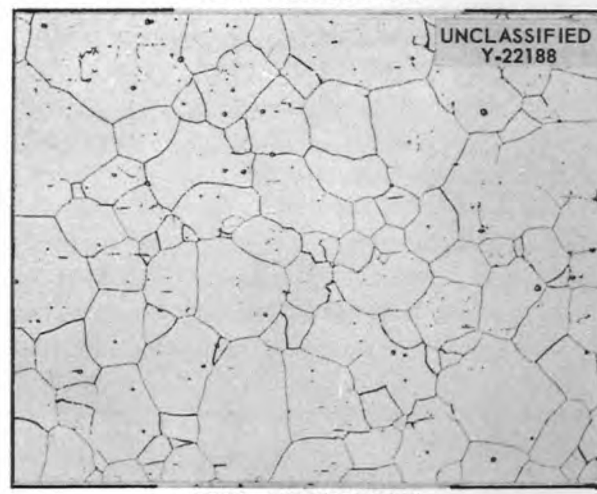

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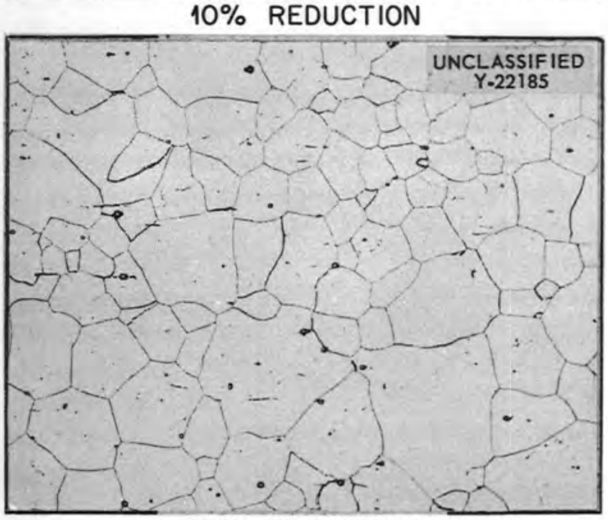

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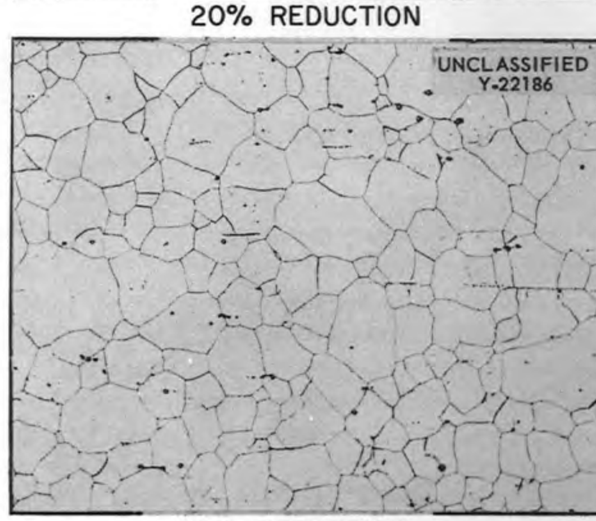

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FIG. 10 INITIALLY FINE GRAIN ANNEALED 1900°F FOR 30 MIN.

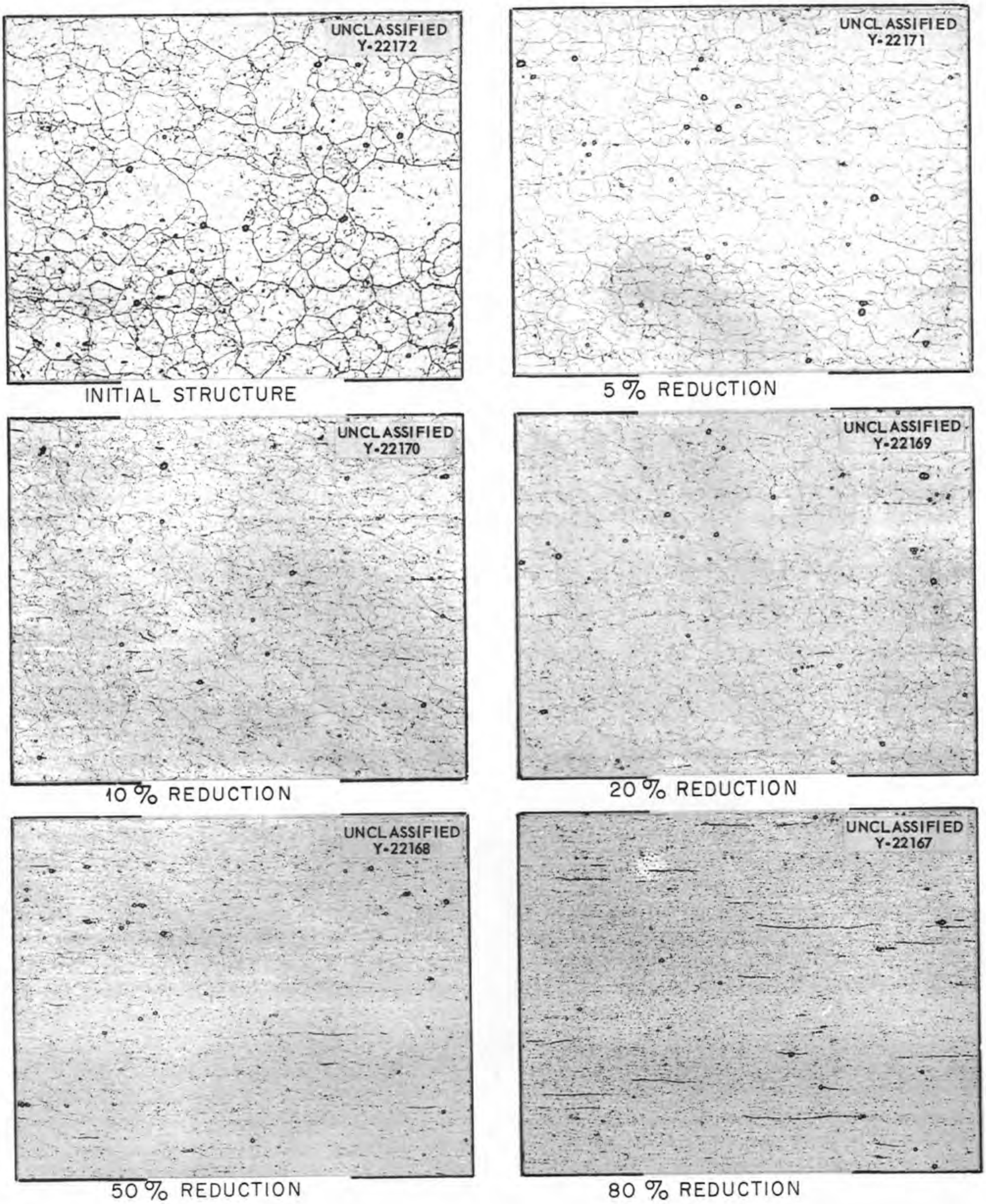
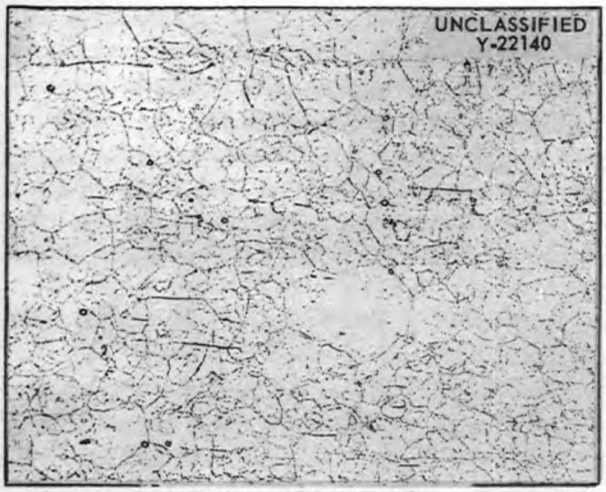


FIG. II INITIALLY FINE GRAIN ANNEALED 1600°F FOR 2 HR.



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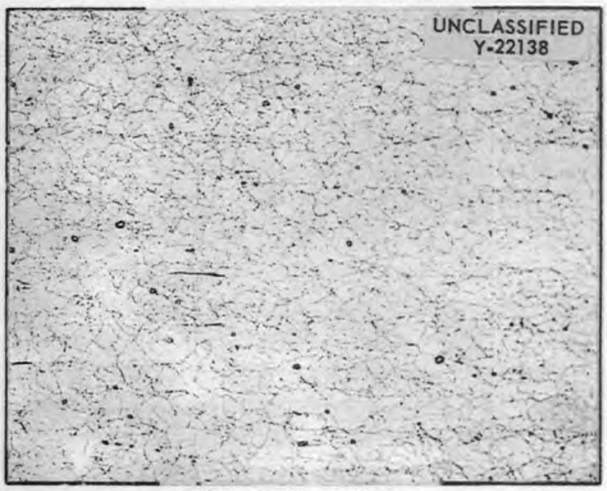

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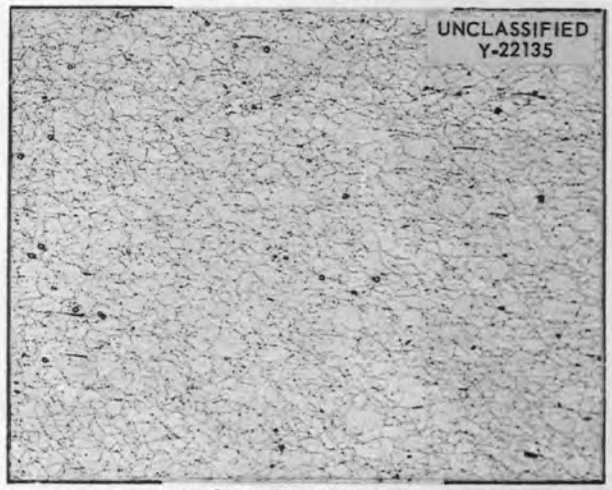

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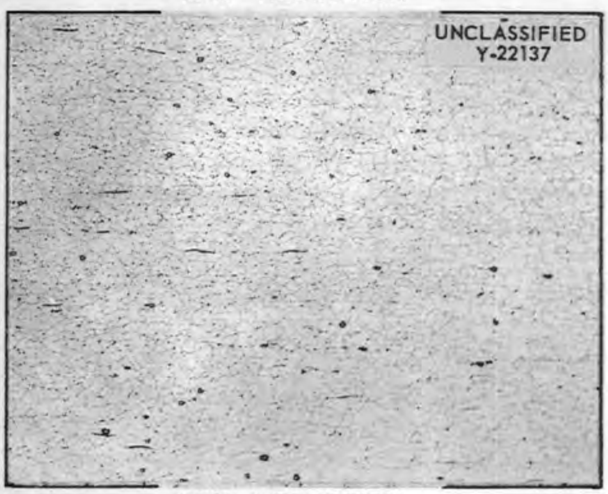

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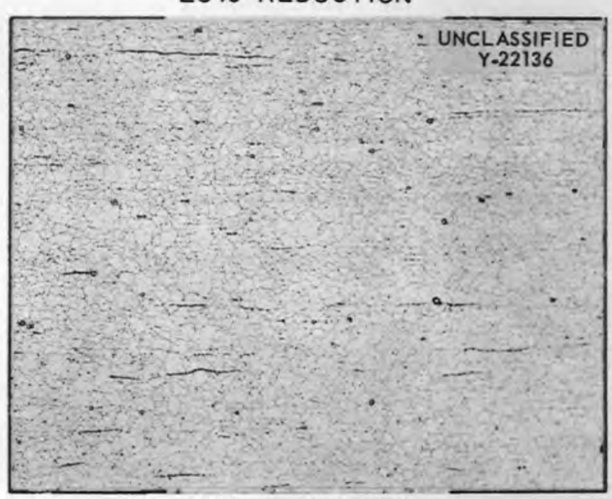

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FIG. 12 INITIALLY FINE GRAIN ANNEALED 1700°F FOR 2 HR.

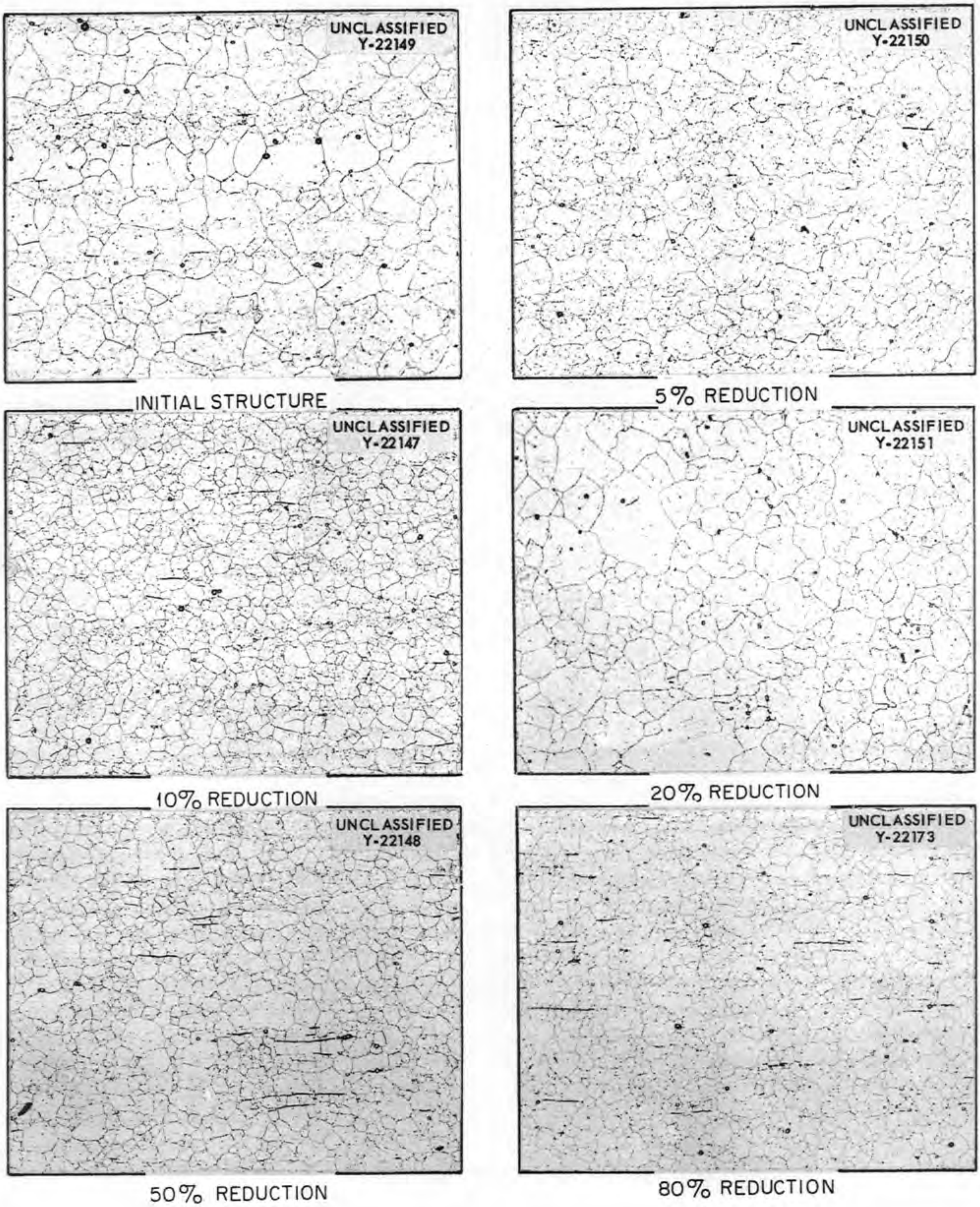
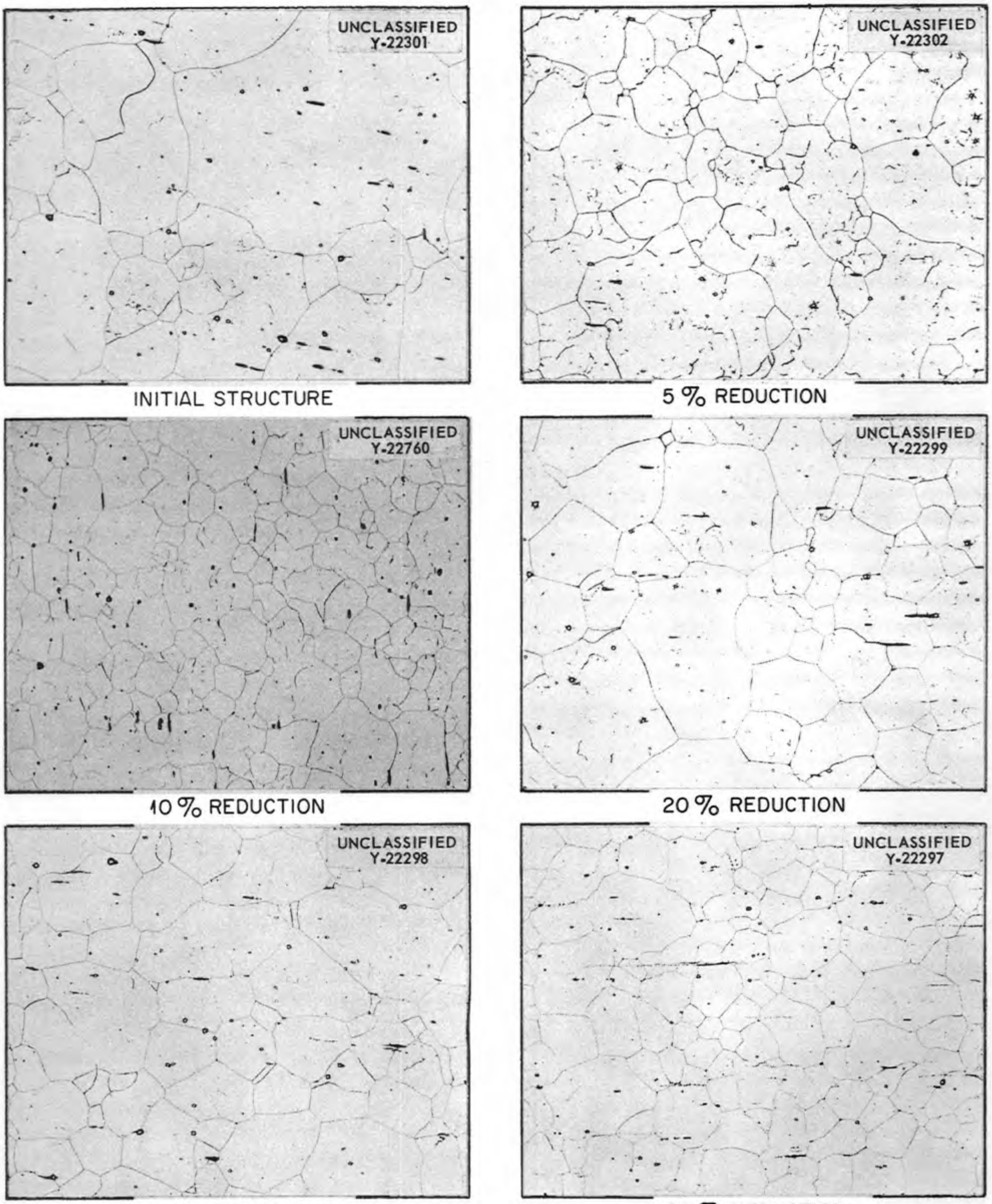


FIG. 13 INITIALLY FINE GRAIN ANNEALED 1800°F FOR 2 HR.



INITIAL STRUCTURE

5 % REDUCTION

10 % REDUCTION

20 % REDUCTION

50 % REDUCTION

80 % REDUCTION

FIG. 14 INITIALLY FINE GRAIN ANNEALED 1900°F FOR 2 HR.

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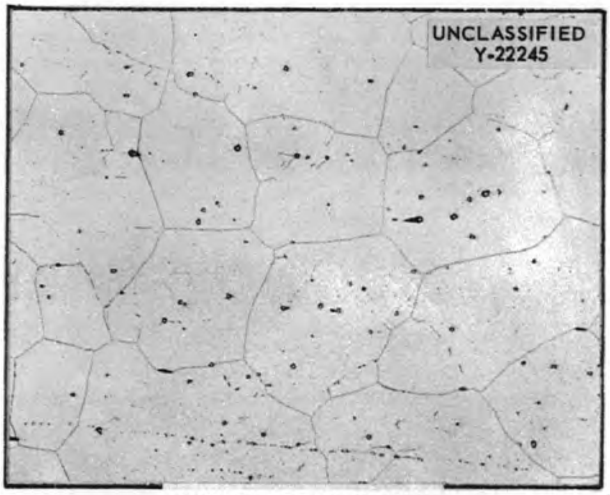

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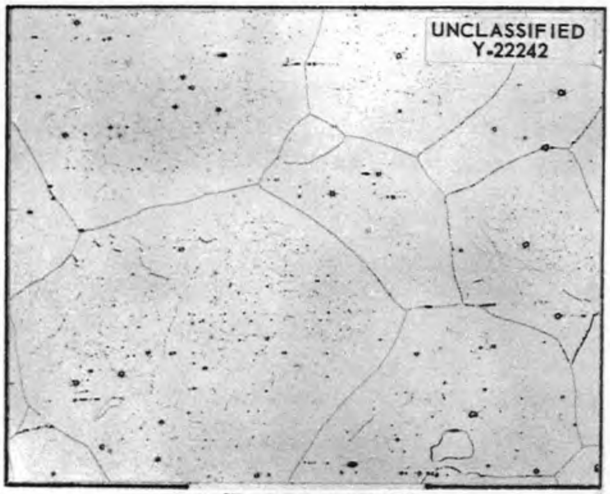

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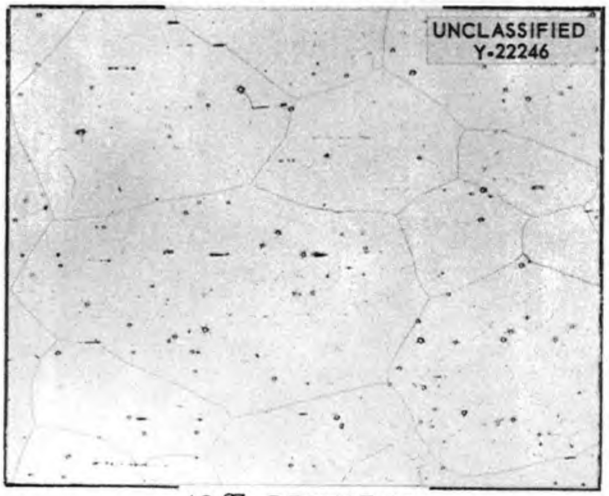

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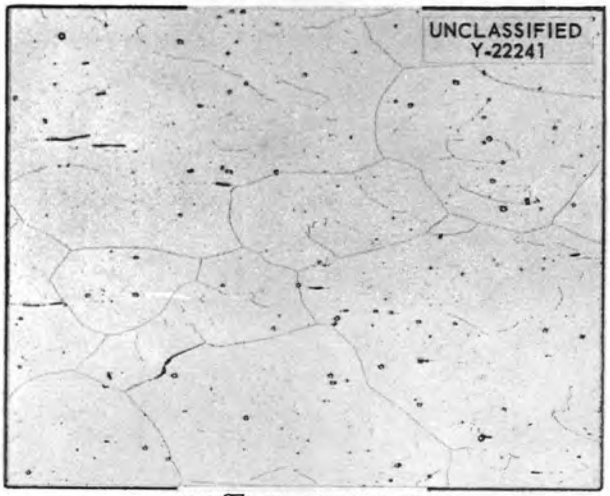

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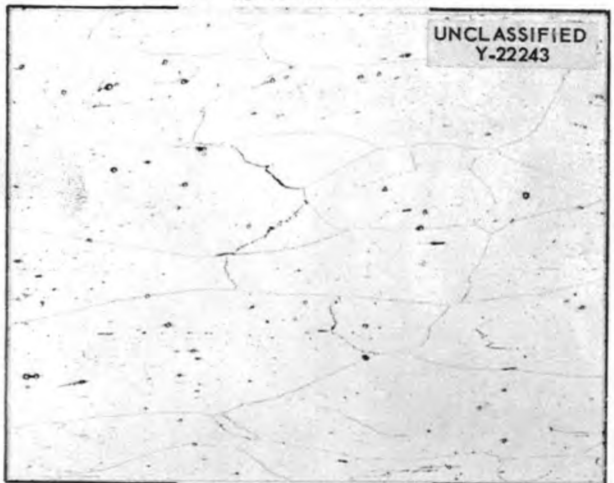

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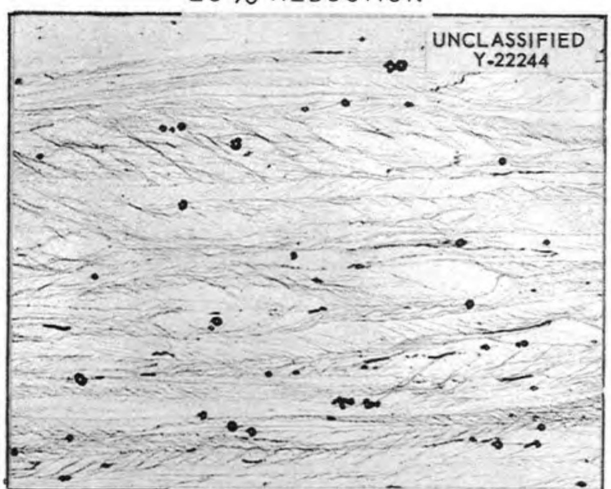
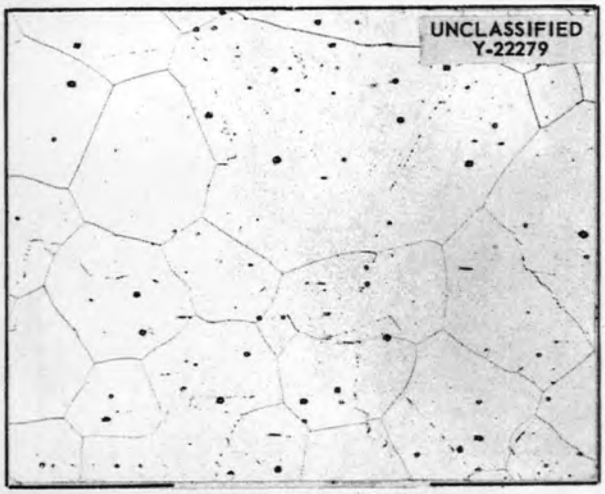


80 % REDUCTION

FIG. 15 INCONEL INITIALLY COARSE GRAIN AS WORKED.



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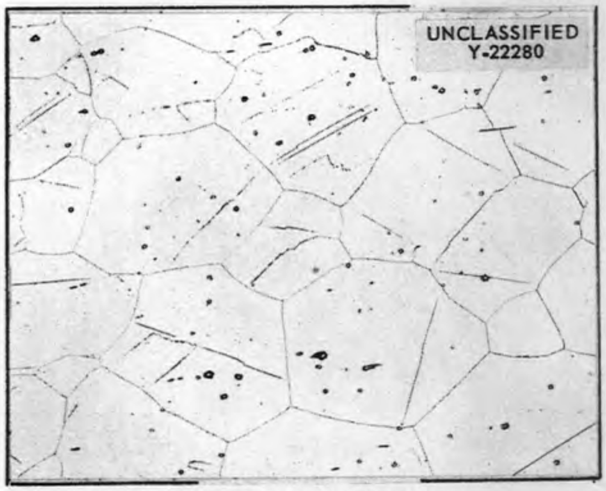

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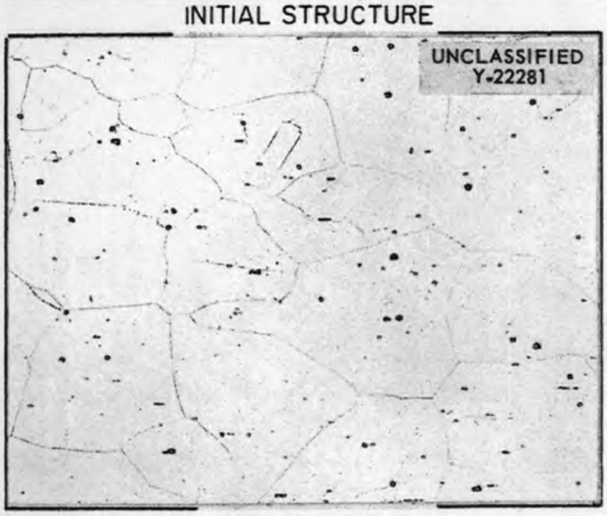

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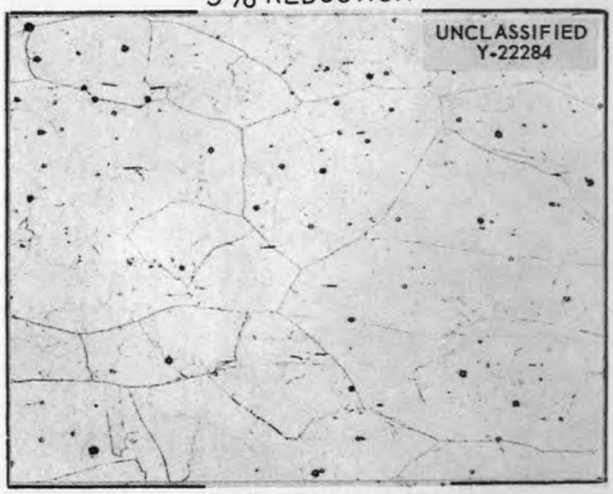

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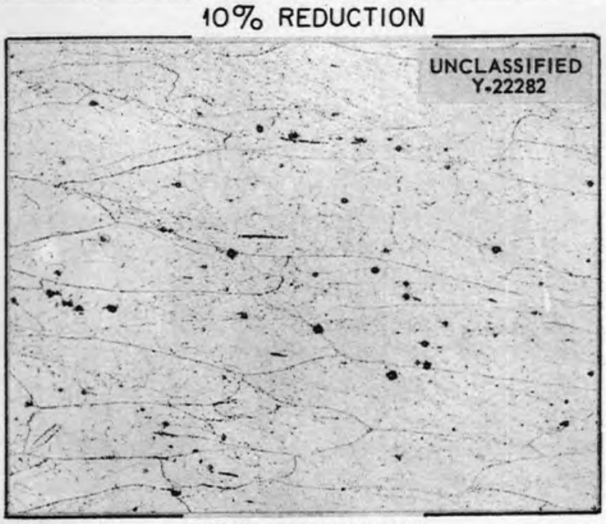

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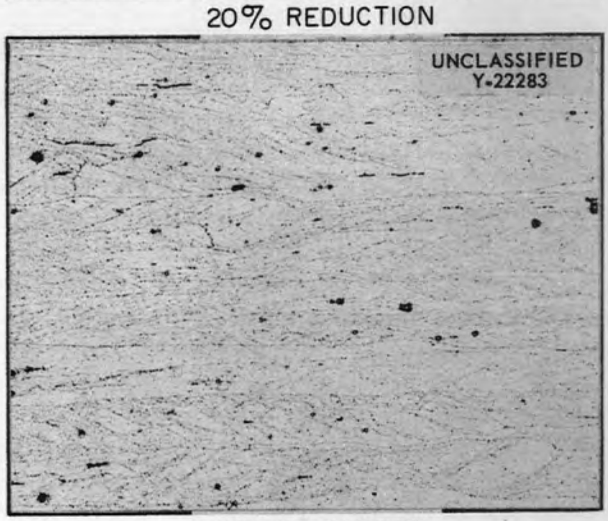
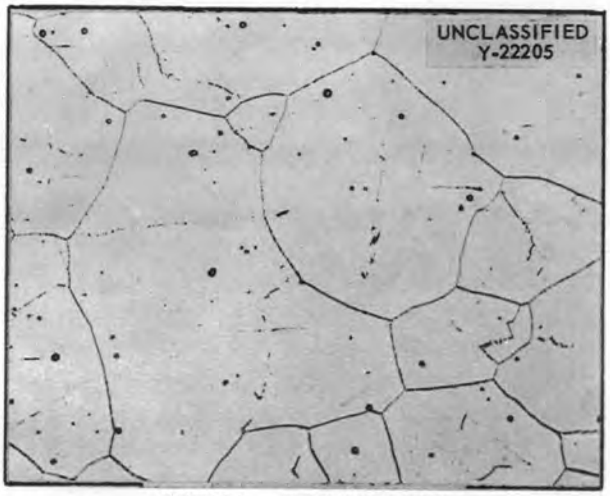


80% REDUCTION

FIG. 16 INITIALLY COARSE GRAIN ANNEALED 1600°F FOR 30 MIN.



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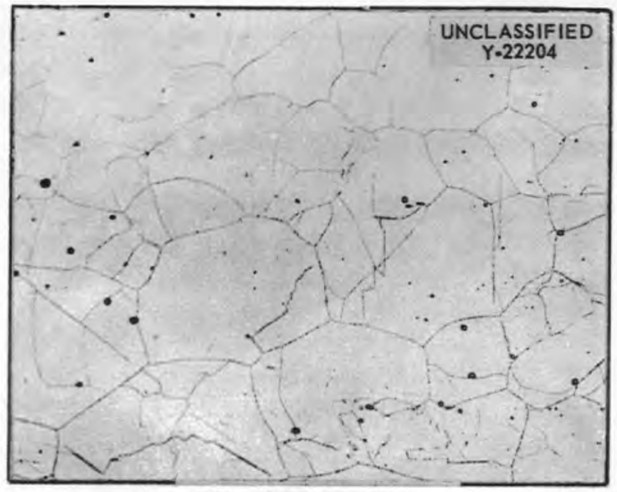

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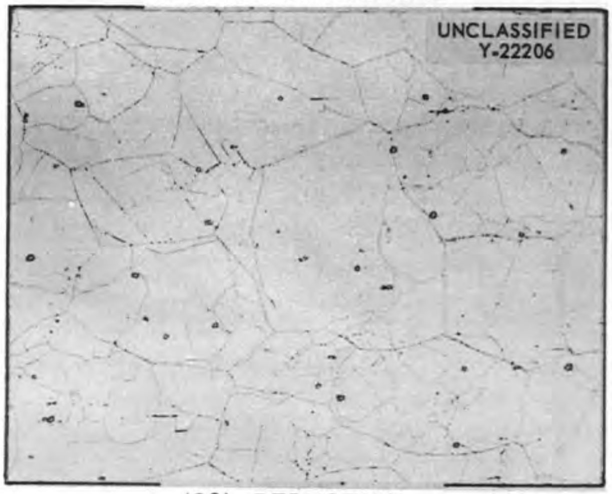
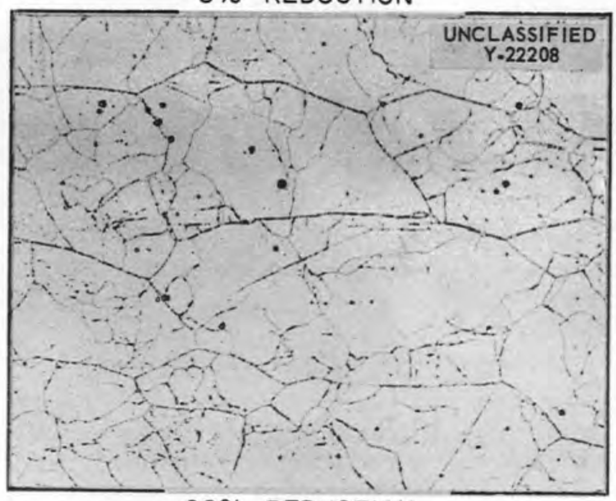


10% REDUCTION



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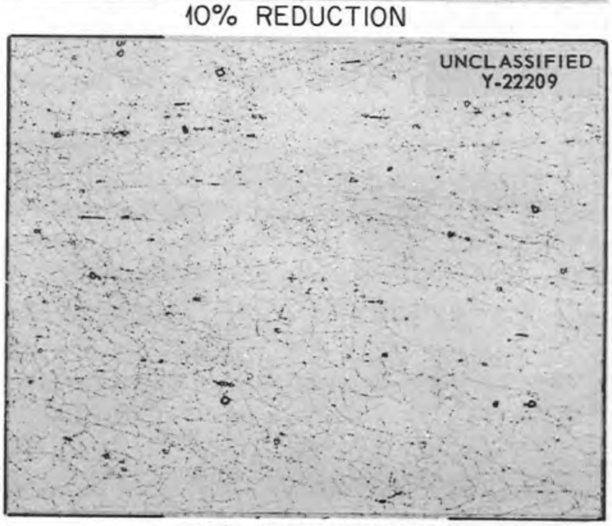

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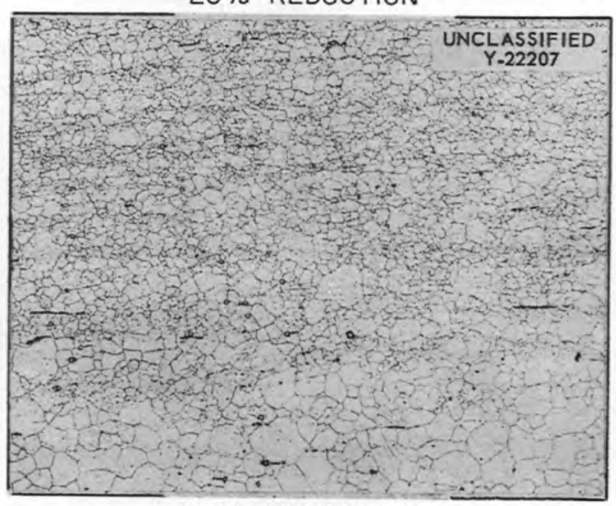
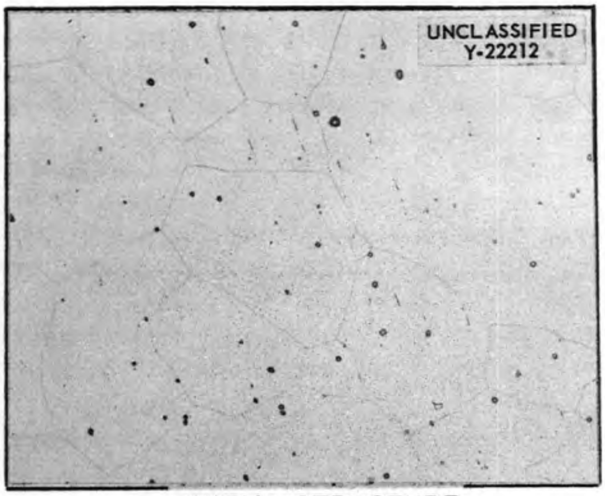
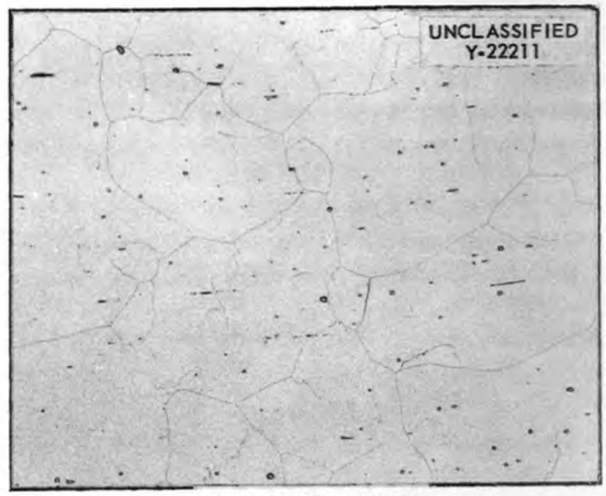


80% REDUCTION

FIG. 17 INITIALLY COARSE GRAIN ANNEALED 1700°F FOR 30 MIN.



INITIAL STRUCTURE



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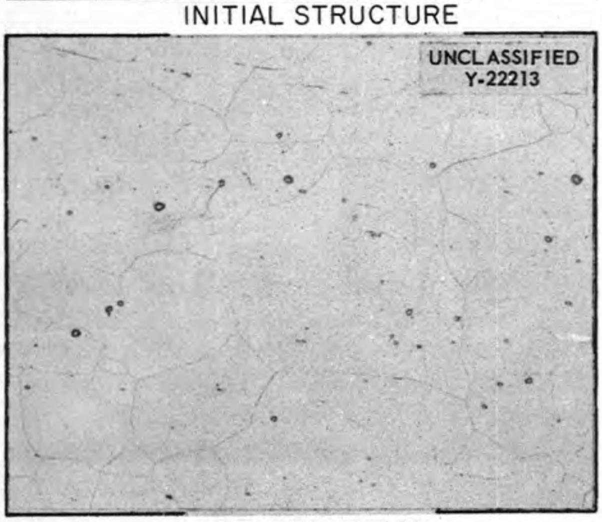

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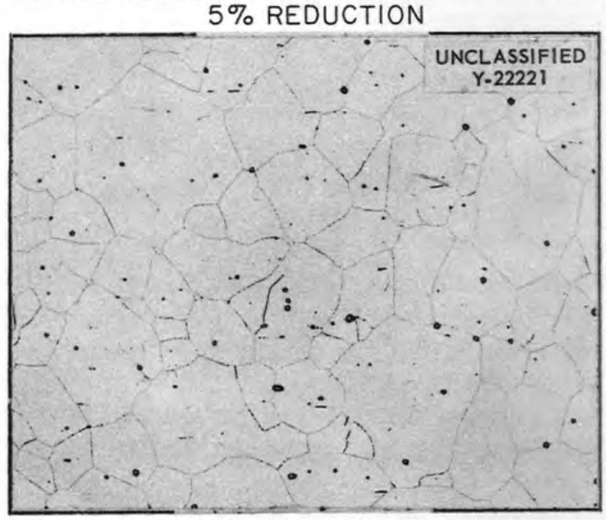

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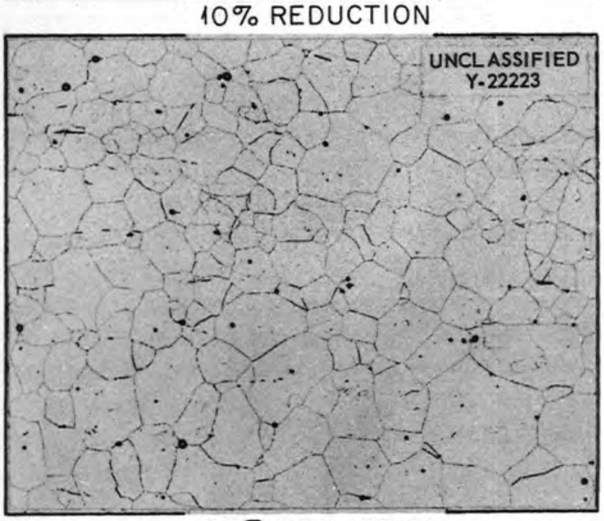

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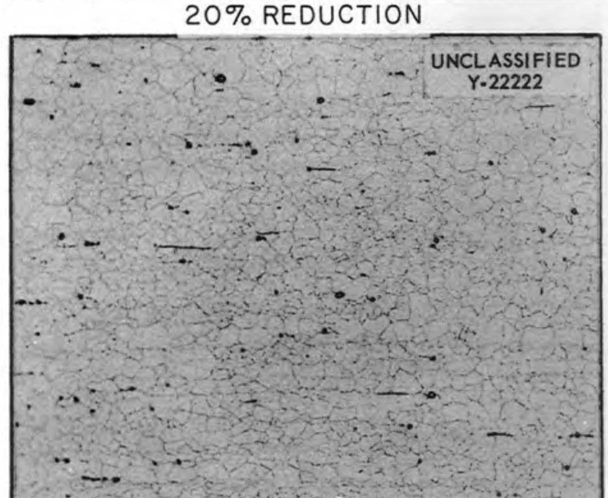
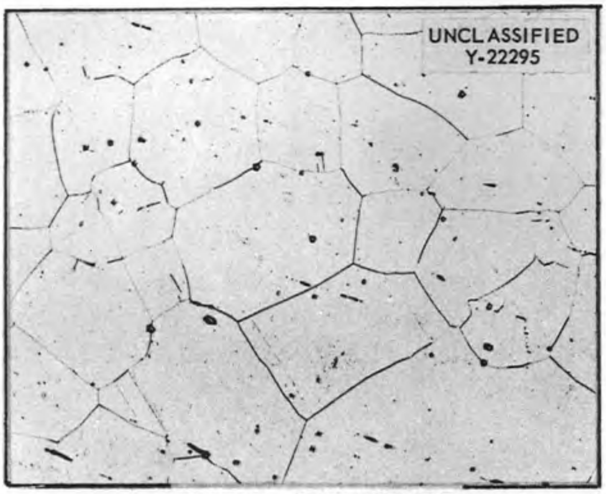


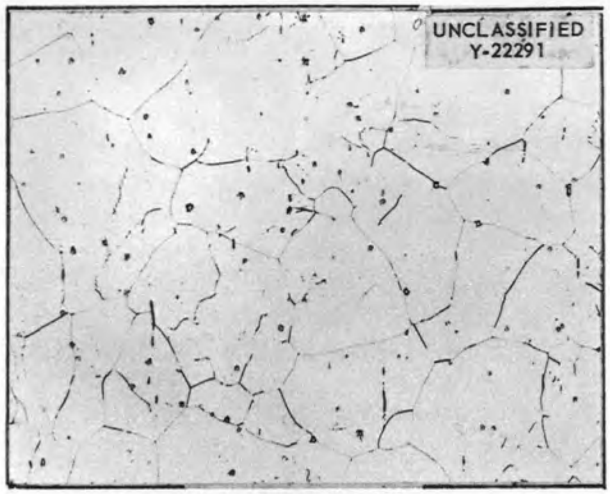
80% REDUCTION

FIG. 18 INITIALLY COARSE GRAIN ANNEALED 1800°F FOR 30 MIN.

Y-23620



INITIAL STRUCTURE



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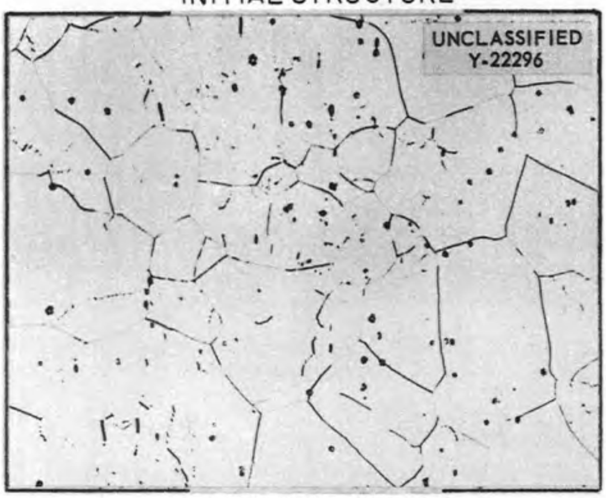

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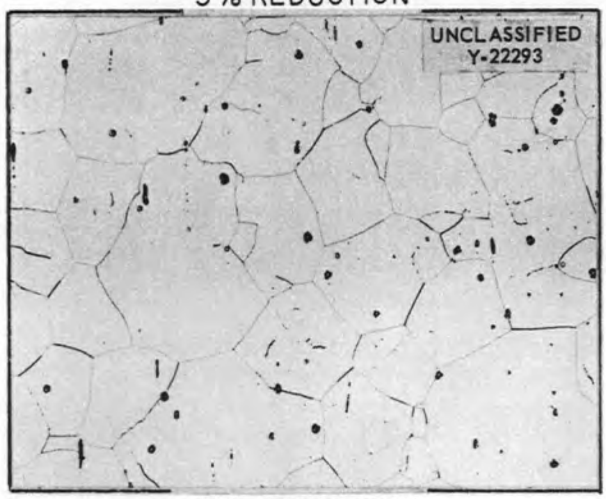

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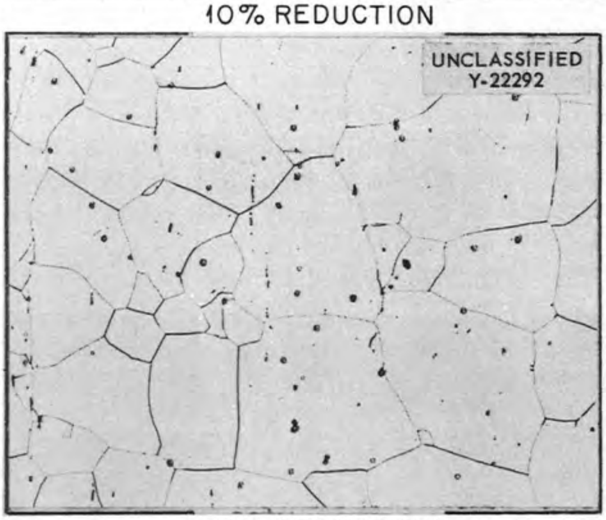

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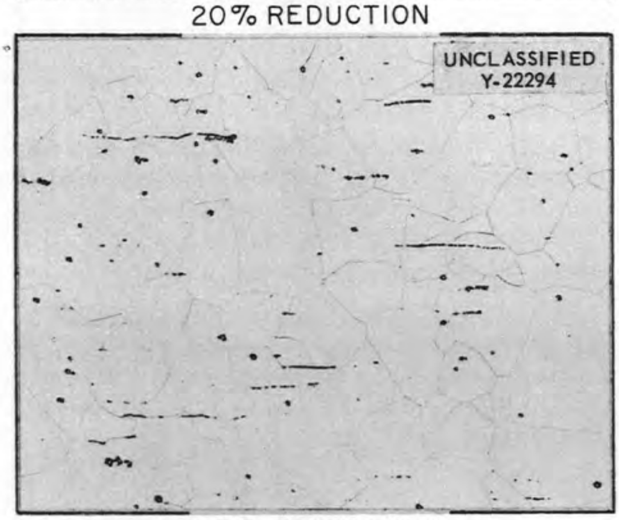

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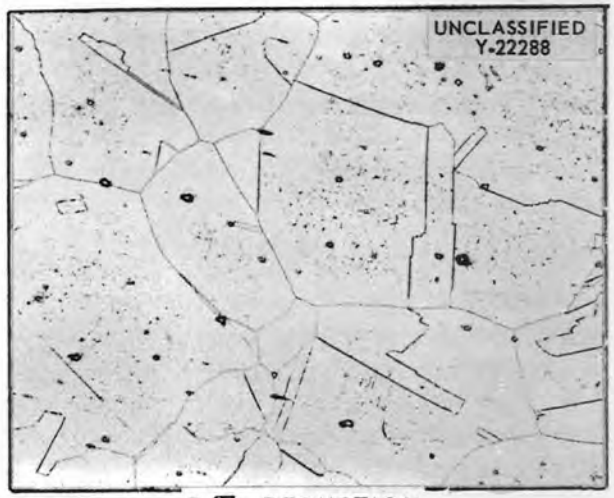
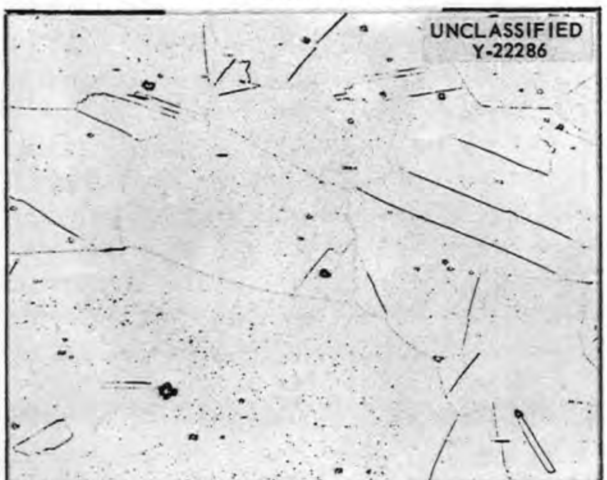
FIG. 19 INITIALLY COARSE GRAIN ANNEALED 1900°F FOR 30 MIN.

INITIAL STRUCTURE



5 % REDUCTION



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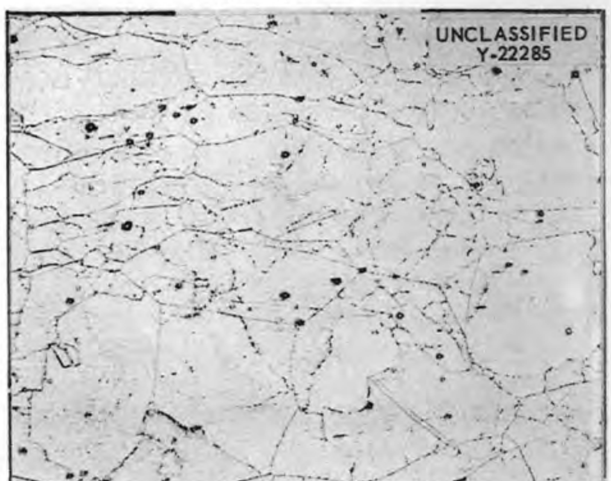
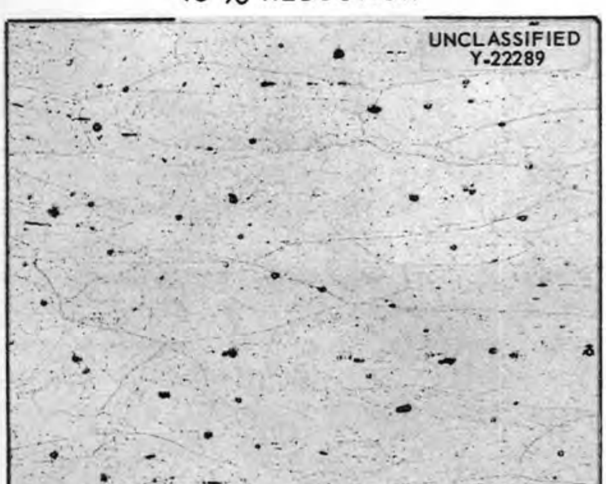
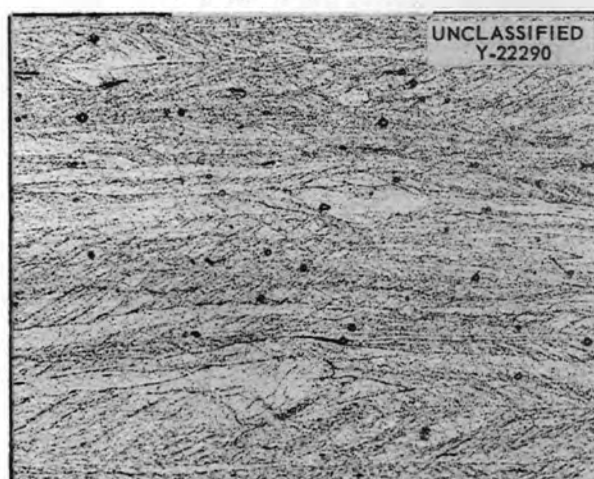


20 % REDUCTION



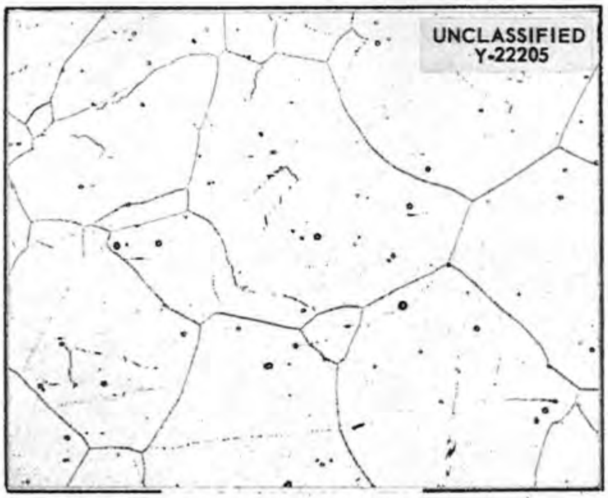
50 % REDUCTION



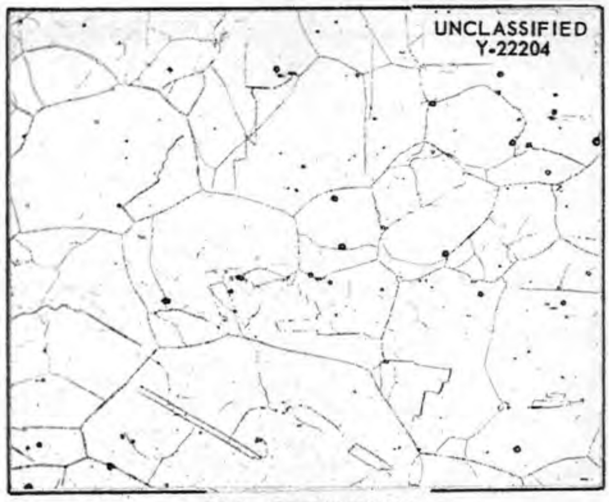
80 % REDUCTION

FIG. 20 INITIALLY COARSE GRAIN ANNEALED 1600°F FOR 2 HR.

Y-23622



INITIAL STRUCTURE



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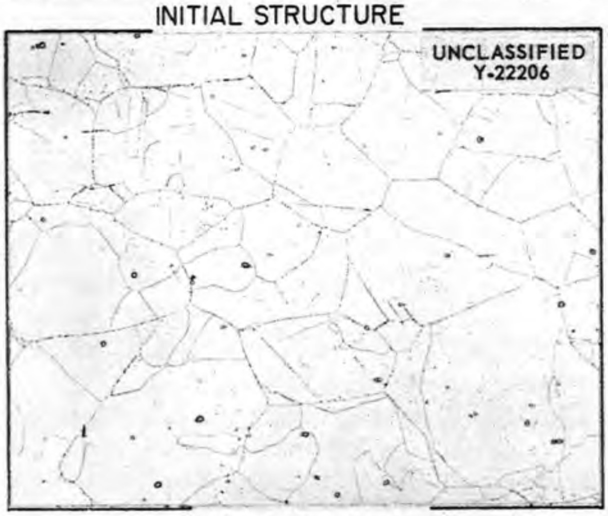

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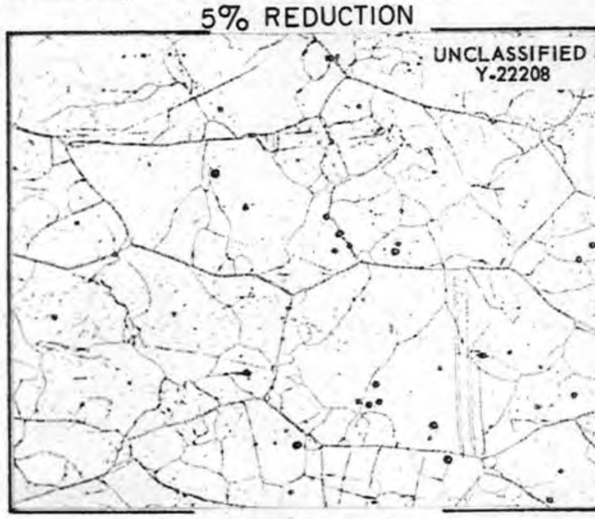

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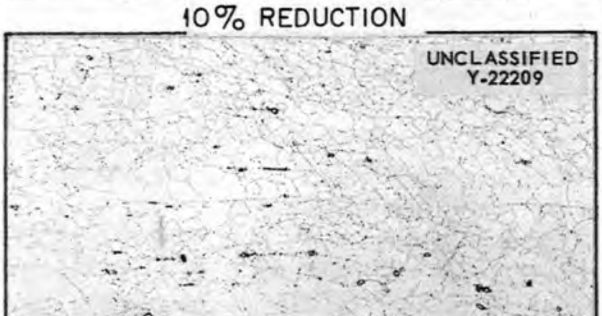

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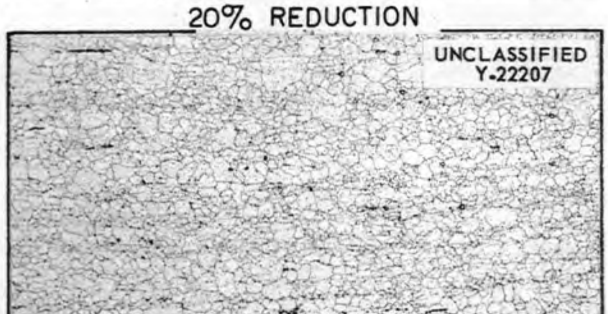
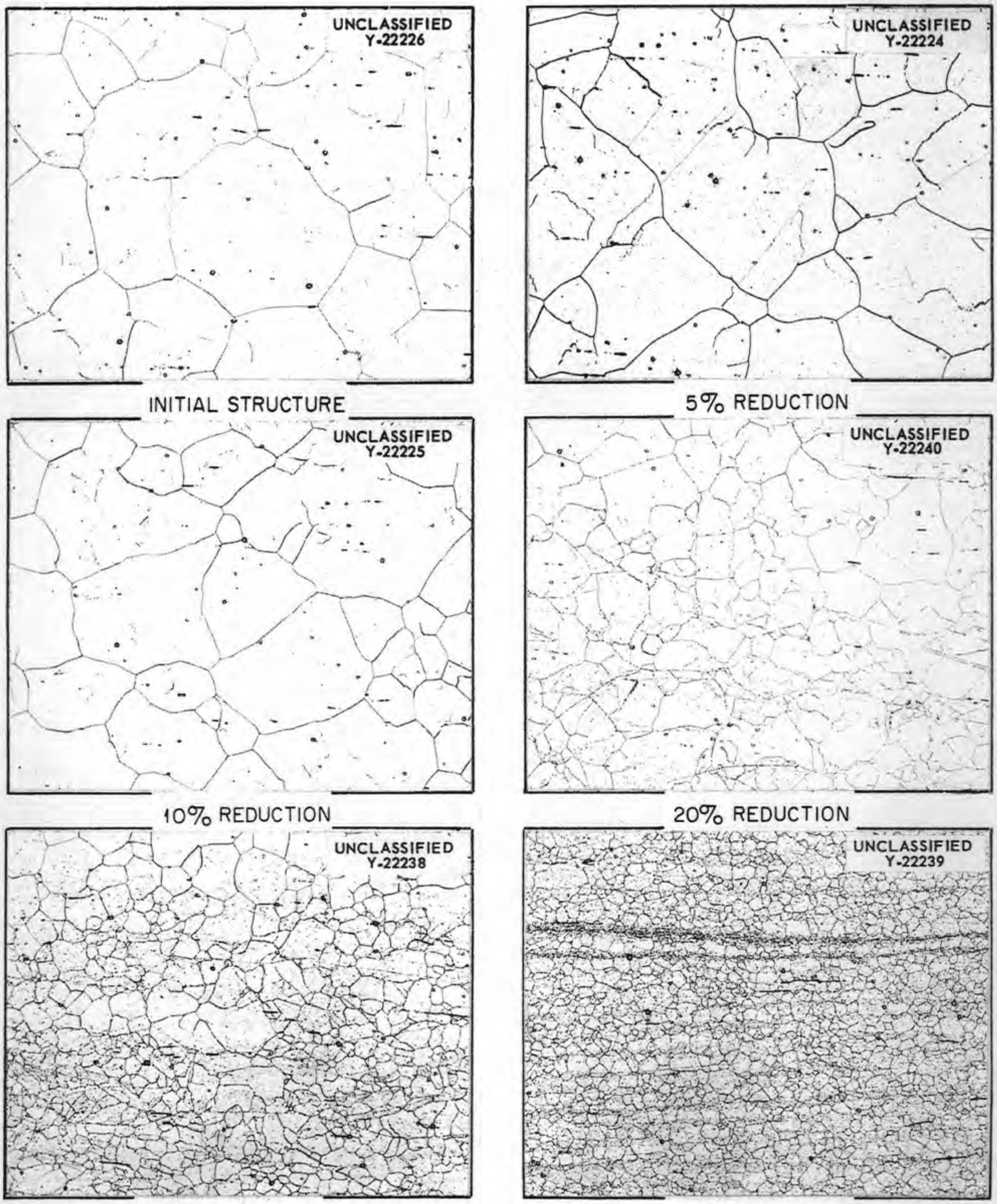


80% REDUCTION

FIG. 21 INITIALLY COARSE GRAIN ANNEALED 1700°F FOR 2 HR.

Y-23623



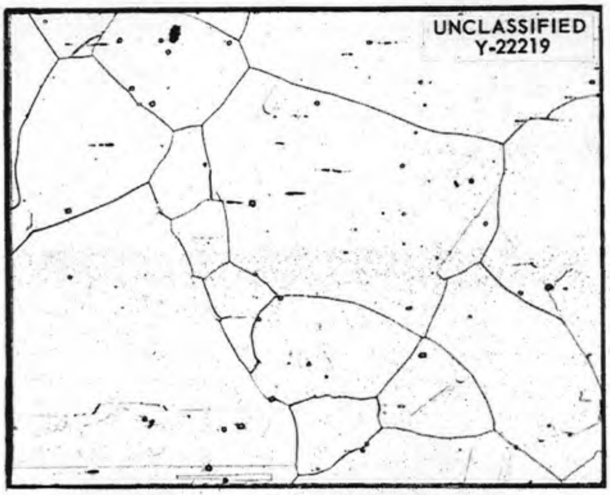
INITIAL STRUCTURE — 5% REDUCTION

10% REDUCTION — 20% REDUCTION

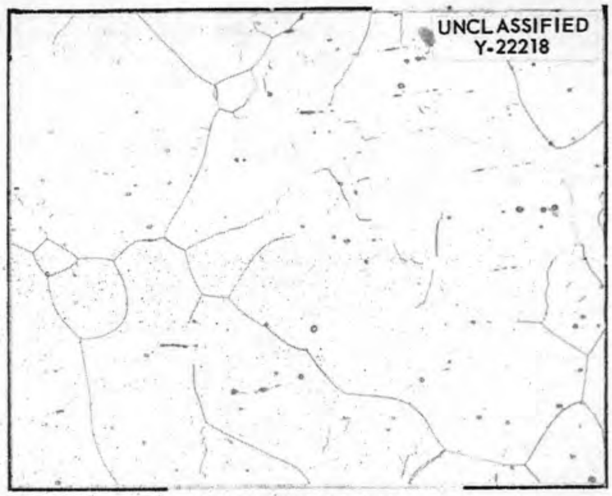
50% REDUCTION — 80% REDUCTION

FIG. 22 INITIALLY COARSE GRAIN ANNEALED 1800°F FOR 2 HR.

Y-23624



INITIAL STRUCTURE



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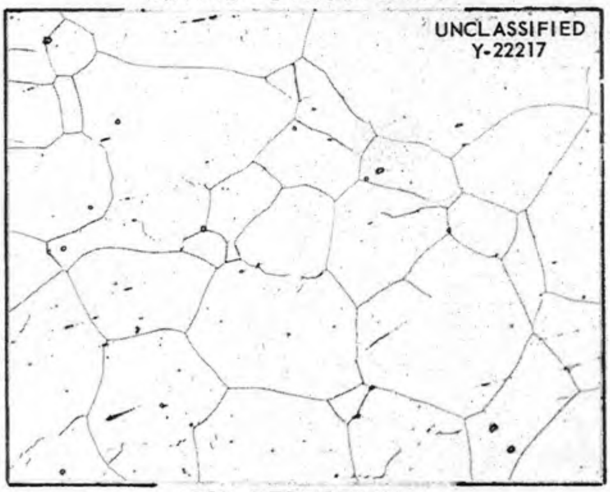

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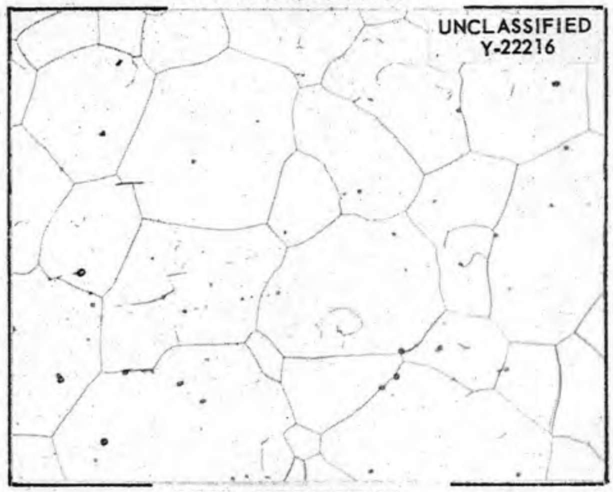

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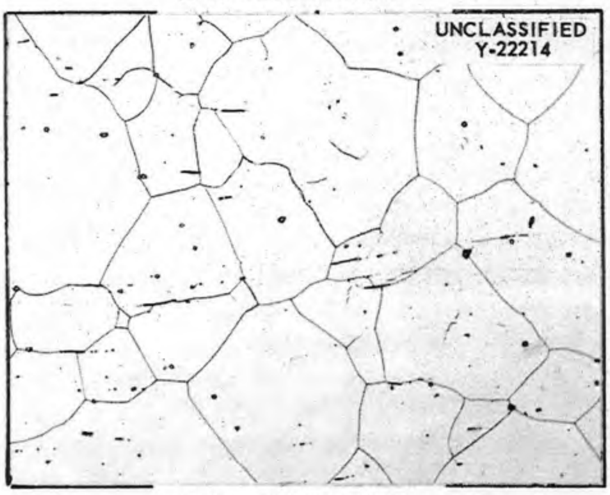

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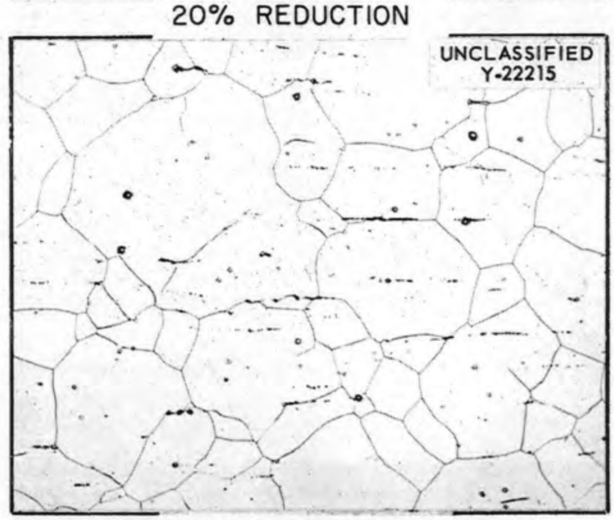

80% REDUCTION

FIG. 23 INITIALLY COARSE GRAIN ANNEALED 1900°F FOR 2 HR.

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