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RECOVERY AND RECRYSTALLIZATION OF ZIRCONIUM AND ITS ALLOYS

PART 3

ANNEALING EFFECTS IN ZIRCALOY-2 AND ZIRCALOY-3

S. H. BUSH and R. S. KEMPER

MAY, 1961

HANFORD LABORATORIES

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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PART 3 - ANNEALING EFFECTS IN ZIRCALOY-2 AND
ZIRCALOY-3

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PART 3

ANNEALING EFFECTS IN ZIRCALOY-2 AND ZIRCALOY-3

By

S. H. Bush and R. S. Kemper

Fuels Development
Reactor and Fuels Research and Development
Hanford Laboratories Operation

May, 1961

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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ABSTRACT

Reactor-grade Zircaloy-2 and vacuum-melted Zircaloy-3V were prepared by arc melting twice under vacuum. In addition, argon-melted Zircaloy-3A was prepared by arc melting in a vacuum and then under argon. These materials were rolled into plate. Specimens of the same thickness and having the same penultimate grain size were prepared at cold work levels of 10, 25, and 50 per cent. These specimens were annealed at 300, 400, 500, 600, 700, and 800 C at 10, 100, 1000 and, in some instances, 10,000 minutes in air, helium, and vacuum (10^{-4} to 10^{-5} mm mercury) atmospheres. Recovery, recrystallization, and grain growth as a function of cold work level, composition, annealing time, temperature, and atmosphere were studied by hardness and electrical resistivity measurements, and by metallographic observations.

Recovery and secondary hardening were observed at temperatures of 300 to 500 C. Recrystallization began at 500 C and was essentially complete at 600 C after 1000 minutes or less. Recrystallization occurred by subgrain growth in material cold worked 10 per cent. Conventional nucleation and growth occurred at 25 and 50 per cent cold work.

Definite differences in response to annealing atmosphere were observed. Vacuum annealed alloys recrystallized more slowly than air- or helium-annealed alloys.

RECOVERY AND RECRYSTALLIZATION OF ZIRCONIUM AND ITS ALLOYS
PART 3
ANNEALING EFFECTS IN ZIRCALOY-2 AND ZIRCALOY-3

INTRODUCTION

Zirconium and its alloys have been used extensively in nuclear reactors because of their low neutron cross sections, fair mechanical properties and good aqueous corrosion resistance. Unfortunately, these alloys are quite sensitive to contamination by gases, acids, and organic materials, all of which deleteriously affect the corrosion resistance. In addition, many of the zirconium alloys are not readily fabricable, requiring one or more intermediate anneals during secondary cold working.

The current study was initiated to better understand the effects of annealing atmosphere, temperature, and time on the recovery and recrystallization of zirconium, Zircaloy-2, and Zircaloy-3 and to assist in predicting the bahavior of these alloys during fabrication and when exposed to elevated temperatures in-reactor.

The initial report of this series(1) dealt with preparation of the specimens prior to annealing, as well as presenting an extensive review of the literature dealing with recovery and recrystallization. Part I(1) should be read prior to reading this report.

An extensive report(2) has been issued dealing with the recovery and recrystallization of Kroll Process zirconium of the composition given in Part I.(1)

The current report describes the annealing of vacuum-melted Zircaloy-2 and Zircaloy-3, and argon-melted Zircaloy-3 in atmospheres of air, helium, and vacuum. Recovery and recrystallization are interpreted through such tests as metallography, hardness, and electrical resistivity. The effects of these annealing conditions on corrosion are described in another report.(3)

RESULTS

Recovery, recrystallization, and grain growth were followed by three methods: (1) examination of the microstructure, (2) hardness surveys, and (3) changes in the electrical resistivity ratio. These three methods were evaluated separately and then compared.

Microstructural Evaluations

Annealed Zircaloy-2, argon-melted Zircaloy-3 (hereafter designated as Zircaloy-3A), and vacuum melted Zircaloy-3 (hereafter designated as Zircaloy-3V) which had been cold worked to 10, 25, and 50 per cent reduction in thickness prior to annealing, were studied to establish the initiation of recrystallization and grain growth. A comprehensive survey of 10 per cent cold-worked alloys annealed in air, 25 per cent cold-worked alloys annealed in helium, and 50 per cent cold-worked alloys annealed in vacuum is given in Table I. The times and temperatures for the initiation and completion of recrystallization and for grain growth appear in Table II.

Some idea of the grain size at the point of complete recrystallization for the three levels of cold work is given in Figure 1 for Zircaloy-2 and Zircaloy-3A.

The preceding data were carefully compiled by examining the microstructures of the alloys after etching with a "B" etch which consisted of a mixture of 10 per cent hydrofluoric acid, 45 per cent nitric acid, and 45 per cent water, followed by anodizing 3 to 5 seconds at 20 volts in a solution of 35 ml of H_2O, 60 ml of ethyl alcohol, 5 ml of H_3PO_4, 20 ml of glycerine, 2 grams of citric acid, and 10 ml of lactic acid with a cathode-to-anode spacing of 1/8 to 1/4 inch.

Representative microstructures are given in Figures 2 through 9 for Zircaloy-2 and argon-melted Zircaloy-3. Since differences between vacuum- and argon-melted Zircaloy-3 microstructures were nominal, pictures of the vacuum-melted alloy were not presented. The effects of cold work, temperature, time, and annealing atmosphere are readily apparent on examination of the representative microstructures given at 100 and 1000 diameters. Since little or nothing can be observed during the recovery stage, most of the pictures covering this region have been deleted.

The ten per cent cold-worked Zircaloy-2 and Zircaloy-3 were found to behave quite similarly, paralleling the results of Gray[2] with Kroll Process zirconium. At this level of cold work, recrystallization does not appear to follow the classical nucleation and growth patterns; rather, duplex growth is observed with a substantial increase in grain size at temperatures of 700 and 800 C. Much of this appears to be by direct growth of subgrains in the matrix. The onset of recrystallization is related to other properties in subsequent sections of this report. As anticipated, nucleation and growth occur at lower temperatures and/or shorter times for Zircaloy-3 compared to Zircaloy-2. This would be expected on the basis of the lower alloy content of Zircaloy-3.

The behavior of 25 per cent cold-worked material, Figures 3, 4, 5, 7, and 8, adheres more closely to classical nucleation and growth theory. Here again, the effect of composition on the onset of recrystallization is seen.

The 50 per cent cold-worked metal shown in Figures 6 and 9 appears to behave anomalously, in that recrystallization occurs more slowly than in the 25 per cent cold-worked metal. An examination of all the data indicates that this is probably due to heat-treating atmosphere. The vacuum used with the higher level of cold work should result in slower heat-up times and a shorter period at equilibrium as compared to helium or air. The effect is so pronounced, as seen in hardness and electrical resistivity data, that it must be factored into all process annealing. The effect is much greater than anticipated and might mean that annealing occurred below 600 C, or that the specimen was not at temperature for the times noted.

Figures 10 and 11 present in graphical form the grain growth of Zircaloy-2 and Zircaloy-3A containing various levels of cold work, as functions of time and temperature of anneal. The difference in grain growth behavior of the 10 per cent cold-worked alloys as compared to 25 and 50 per cent cold-worked alloys, is obvious.

Hardness Surveys

Recovery and recrystallization of the cold-worked Zircaloy-2 and Zircaloy-3 was followed by measuring the Rockwell G hardness of the various specimens. Changes in hardness as functions of initial cold-work level, composition, temperature, time, and atmosphere of anneal are shown in Figures 12 through 24. Because of the number of parameters and the quantity of data, a bar graph was used rather than the more conventional continuous plots of change of hardness as functions of temperature or time. The bar graphs permitted the plotting of changes in hardness as functions of level of cold work, annealing temperature, and annealing time for any one composition and atmosphere, or for different compositions and atmospheres at any fixed time.

Figures 12 through 15 contain hardness data for Zircaloy-2, where the parameter differentiating Figures 12, 13, and 14 is atmosphere (vacuum helium, or air). Figure 15 permits a comparison of the effects of annealing Zircaloy-2 in the various atmospheres for 100 minutes. Increases in hardness occur at recovery temperatures of 300 to 500 C, probably due to a precipitation of material from the matrix. At higher temperatures, pronounced decreases in hardness are noted with the changes becoming more marked at the longer times. Results in hardness tend to duplicate the effects noted in microstructural changes in that the vacuum-annealed specimens decreased in hardness to a lesser extent than did helium- or air-annealed specimens for equivalent conditions of anneal. Differences between air and helium anneals were minor. The similarities and differences in response to annealing conditions are quite apparent in Figure 15, where the effects of atmosphere are compared at 100 minutes over annealing temperatures of 300 to 800 C.

Figures 16 through 19 contain data covering conditions similar to those discussed for Zircaloy-2; however, these figures deal with Zircaloy-3A.

In essence, over-all performance is comparable for Zircaloy-3A and Zircaloy-2 insofar as time, temperature, and atmosphere effects are concerned. Greater changes in hardness occur with Zircaloy-3A than in the case of Zircaloy-2. In addition, the changes initiate somewhat more rapidly. This isn't surprising, considering the differences in chemical composition.

Figures 20 through 23 present data for Zircaloy-3V. Essentially, the results for Zircaloy-3V duplicate those for Zircaloy-3A. It would be possible to use one set of data for both materials without perceptible error.

A limited number of long-time anneals were conducted with Zircaloy-2, Zircaloy-3A, and Zircaloy-3V. The maximum time investigated was 10,000 minutes. Data are presented in Figure 24 covering the effect of temperature, atmosphere, composition, and time of anneal. Apparently longer annealing times have a limited effect at 400 C when air is used as an atmosphere. Changes from 1000 to 10,000 minutes are nominal and both increases and decreases in hardness were observed in going from 1000 to 10,000 minutes. In the case of vacuum anneals at 600 C, the changes were much more pronounced in going from 1000 to 10,000 minutes. This was particularly apparent in the case of Zircaloy-2. Again as at 400 C, the changes were quite nominal in an air atmosphere when comparing 1000- and 10,000-minute anneals of Zircaloy 3V. Contamination by oxygen or nitrogen in the extended anneals could increase the hardness somewhat, minimizing the effect of annealing.

In the case of the vacuum atmosphere, the lack of convective heat transfer may play a more marked role than originally assumed. Temperatures were carefully checked at equilibrium conditions using thermocouples ballasted with a small piece of metal to minimize cycling. It is interesting to note that 10,000 minutes in a vacuum for Zircaloy-3A in the 10 and 25 per cent cold-worked states result in hardness comparable to those of Zircaloy-3V after 1000 minutes in air. Recrystallization is a time-temperature dependent process, so it isn't possible to say whether there is an apparent temperature effect, differing from the true temperature in the case of vacuum-annealed specimens; it does not seem possible that the thermal lag could be greater than a few minutes in the case of vacuum-annealed specimens as compared to helium- or air-annealed specimens.

Electrical Resistivity Measurements

Changes in electrical resistivity constitute a sensitive method of detecting the recovery and recrystallization of cold-worked metals. The conventional method is to plot the ratios of resistivity where the denominator is the electrical resistivity of the specimen in the cold-worked state. While this method is quite sensitive to changes in recovery and recrystallization, it is also sensitive to gaseous contamination of the metal, specimen size, lead resistance, and temperature. Because of these factors, the results presented in Figures 25 through 36 should be considered qualitative, not quantitative. The avidity with which zirconium alloys getter gaseous contaminants is a major source of error.

Figures 25 through 28 contain the electrical resistivity data for Zircaloy-2. Changes at 300 of 500 C are comparable for the different atmospheres. The effect of contamination becomes quite apparent at higher temperatures in air or helium, where substantial increases occur in the resistivity ratio. The vacuum-annealed specimens continue to decrease in resistivity ratio at the higher temperatures, indicating a lower degree of contamination in this instance. The preceding effects are much more apparent at the lower level of cold work, as can be seen in Figure 28.

Figures 29 through 32 present comparable data for Zircaloy-3A, covering vacuum, air, and helium anneals. While the changes in resistivity ratio are more pronounced for Zircaloy-3A than for Zircaloy-2, the trends are comparable. Zircaloy-3A does appear to be more sensitive to contamination by air at 1000 minutes than helium, or than was observed with Zircaloy-2 in helium, or than was observed with Zircaloy-2 in helium and air.

Figures 33 through 36 present the electrical resistivity data for Zircaloy-3V. The same trends observed in Zircaloy-2 and Zircaloy-3A recur in Zircaloy-3V. A direct comparison of Zircaloy-3A and Zircaloy-3V reveals nominal differences in a few cases. Otherwise, they are virtually the same.

DISCUSSION

Microstructures

A history of the microstructural evaluation of Zircaloy-2, Zircaloy-3A, and Zircaloy-3V is presented in Table I. A complete microstructural evaluation was not made because of the large number of specimens. However, it was felt that the selection was representative.

Some definite changes were observed in the zirconium alloys during the recovery stage of annealing extending from 300 to 500 C. An increase in precipitate, ranging from nominal to extensive, occurred with increases in time and temperature of anneal; at higher temperatures (600 to 800 C), there appeared to be some redissolution of the precipitate. This precipitate was normally random, but in some instances it occurred selectively at the grain boundaries. Accompanying the increase in precipitate was a general sharpening of grain boundaries. It is felt that these microstructural changes are related to the decreased corrosion rates observed in specimens given a recovery anneal compared to the as-cold-worked material.[3] The microstructural effects are observable in Figure 3; they appear to be independent of annealing atmosphere, composition, and level of cold work.

The onset of nucleation was quite difficult to detect and there may be some error in the values cited. All pictures were scanned closely at various levels of magnification to determine where recrystallization first began. Table II lists the times and temperatures where recrystallization was initiated and completed. In some instances, recrystallization was noted at 500 C. This was particularly true on 10 per cent cold-worked specimens. The recrystallization in the 10 per cent cold-worked material appeared to occur by subgrain growth rather than by conventional nucleation. In every instance, recrystallization was essentially complete at 600 C and 1000 minutes. Recrystallization was somewhat slower in the 50 per cent cold-worked vacuum-annealed alloys than in other atmospheres and at lower cold-work levels. This lag is attributed in part to the thermal lag in reaching temperature in the vacuum. This postulate is born out in the hardness results given in Figure 15 which will be discussed later. The recrystallization of the zirconium alloys containing 25 and 50 per cent cold work appears to occur by conventional nucleation from the cold-worked matrix.

The as-recrystallized grain sizes cited in Figure 1 become finer with increasing levels of cold work. Zircaloy-3A has a consistently smaller grain size than Zircaloy-2, although their penultimate grain sizes were comparable. The slight tendency for recrystallization to be initiated and completed at lower temperatures and/or shorter times for Zircaloy-3A, as compared to Zircaloy-2, might account for the finer grain size.

No attempt was made to determine activation energies for the various recrystallization processes on the basis of reaching some finite degree of recrystallization. The variations introduced by annealing, coupled with the difficulty in fixing a precise level of recrystallization, militated against an activation energy approach.

The grain growth of Zircaloy-2 and Zircaloy-3 is given in Figures 10 and 11. It is obvious that 10 per cent cold-worked alloys are much more susceptible to growth than are the 25 and 50 per cent cold-worked alloys. Zircaloy-3A grows to a greater extent than Zircaloy-2. On the 10 per cent cold-work level, particularly at 600 and 700 C, the lower level of alloy additions could account for these differences; however, no quantitative data are available covering the effect of 1 to 3 per cent alloy additions on grain growth of zirconium. No attempt was made to establish a rate equation for the grain growth of the alloys because the data were too limited to permit establishment of a valid equation. The pronounced difference in growth of the 10 per cent zirconium alloys compared to higher levels of cold work could be related to the difference in nucleation mechanism. Apparent differences between 25 and 50 per cent cold-worked Zircaloy-2 could be due to the different response noted in vacuum annealing of the 50 per cent cold-worked material. This is apparent in Figure 10.

The only known studies of recovery and recrystallization of Zircaloy-2 and Zircaloy-3 are those of Richards,[4] Goodwin and Goldman,[5] Johnson,[6] et al.[7] Results obtained in this study paralleled the work reported by these authors. A more precise evaluation of the mechanism of recrystallization is possible with unalloyed zirconium. These data are presented by Gray[2] and Gray, et al.[1]

No relevant work was found dealing with the growth of Zircaloy-2 and Zircaloy-3. Part I of this series is a more general discussion of the literature of recrystallization and growth.[1]

Hardness Surveys

An examination of Figure 12, 13, and 14 reveals the existence of competing mechanisms during the 300 to 500 C anneals. The first process is recovery, accompanied by a slight decrease in hardness. The second process is aging or secondary hardening phenomena denoted by general increases in hardness. It is interesting to note that the secondary hardening effect is much more pronounced in the vacuum-annealed material than in either air- or helium-annealed material. An examination of the microstructural results presented in Table I, reveals a greater quantitiy of precipitate in the vacuum-annealed specimens. The cause of the increased level of precipitate is not known. Herenguel, et al.,[7] reported a similar secondary hardening at 450 C in 60 per cent cold-worked Zircaloy-2 annealed for 24 hours in air.

Analysis of Figures 12, 13, and 14 reveals another major difference--the degree of hardening at any given combination of time and temperature as a function of annealing atmosphere. While there is little or no difference between air and helium anneals, the difference is quite marked when vacuum-annealed hardening results are compared to those of helium- or air-annealed hardening. The decreases in hardness, particularly at or above 600 C, are much more marked in air or helium. It may be that the specimens were annealed at lower-than-cited temperatures. However, the temperatures were checked with thermocouples in the central zone of the furnace, where most specimens were held, and no such differences were noted. It is difficult to conceive that the differences in response time due to a lack of convective heat transfer could be more than a few minutes with this specimen geometry at 500 C and above. Tests are planned utilizing a larger furnace with greater capacity to establish the cause of this phenomenon.

Hardness test results for Zircaoy-3A and Zircaloy-3V presented in Figures 16 through 23 display the same trends that were noted in Zircaloy-2. Both recovery and secondary hardening is observed, but to a lesser degree than in Zircaloy-2. The same effect of atmosphere is also noted.

Where metallographic data are not available, it is possible to predict the time and temperatures for the complete recrystallization of the alloys on the basis of hardness along. For example, the hardness trends indicate partial recrystallization at 500 C and 1000 minutes in Zircaloy-2. Hardnesses below 77 Rockwell G indicate complete recrystallization of Zircaloy-2 in air or helium; however, this is not true in vacuum anneals. The effect of atmosphere is similar in Zircaloy-3A and Zircaloy-3V. Rockwell G 55 can be taken as the point of complete recrystallization in air or helium, while Rockwell G 60-62 is used with vacuum anneals. The effect of longer (10,000-minute) anneals at 400 and 600 C is portrayed in Figure 24. Changes in hardness of Zircaloy-2, Zircaloy-3A, and Zircaloy-3V are quite nominal in going from 1000 to 10,000 minutes in air at 400 C. At 600 C, there is little difference in air-annealed Zircaloy-3V at 1000 and 10,000 minutes. A greater difference is observed in vacuum-annealed Zircaloy-3A and Zircaloy-2. The effect of the longer time is particularly pronounced in Zircaloy-2 which contains 50 per cent cold work.

Electrical Resistivity

Electrical resistivity measurements do not appear a particularly sensitive method of detecting recovery and recrystallization of the zirconium alloys. Zircaloy-2 is less sensitive than Zircaloy-3A or Zircaloy-3V. In addition, the secondary hardening is not detectable at 300 to 500 C. No marked changes are noted in electrical resistivity in the early stages of nucleation. There is some evidence of recrystallization at 500 C, but the changes are not marked. At 700 C, the decrease in resistivity resulting from recrystallization is masked by increases in resistivity due to a pickup of gaseous contaminants. This competing process offsets the decreases to the extent that electrical resistivity ratios of greater than one are obtained. The effect of atmosphere is quite pronouned at 700 C--the vacuum-annealed specimens have low resistivity ratios; helium has higher values; and air anneals exceed resistivity ratio values of one at longer times. It is felt that electrical resistivity is an excellent method for following the pickup of contaminants, but it is not satisfactory for following recovery and recrystallization due to the small differences in values and the competing process of contamination.

General Comments

The response of the zirconium alloys to vacuum annealing introduces some doubt of the accuracy of temperature measurement and some question about the underlying cause for the marked differences. Even so, the data were concluded to indicate the possibility of differences, considering the care taken in establishing temperature conditions. The results are distinctly anomalous. There is no reason to believe that such errors exist in the air or helium anneals. In fact, results of these anneals compare with the work of others.[5, 7]

CONCLUSIONS

Reactor-grade Zircaloy-2 and Zircaloy-3V were prepared by arc melting twice under vacuum. In addition, Zircaloy-3A was prepared by arc melting in a vacuum, then under argon. These materials were rolled into plate. Specimens of the same thickness and having the same penultimate grain size were prepared at cold work levels of 10, 25, and 50 per cent. These specimens were annealed at 300, 400, 500, 600, 700, and 800 C at 10, 100, 1000, and, in some instances, 10,000 minutes in air, helium, and vacuum (10^{-4} to 10^{-5} mm mercury) atmospheres. Recovery, recrystallization, and grain growth as a function of cold work level, composition, annealing time, temperature, and atmosphere were studied by hardness and electrical resistivity measurements, and by metallographic observations.

The following conclusions were derived from this study of recovery, recrystallization, and grain growth. Observations pertaining to the cold-worked material are given in Part I of this series.[(1)]

Recovery and Recrystallization

a. Recovery of Zircaloy-2 and Zircaloy-3 occured between 300 and 500 C, as detected by hardness and electrical resistivity.

b. Definite evidence of secondary hardening, probably due to precipitation of insoluble phases from the matrix, was observed in Zircaloy-2 and Zircaloy-3 at 300 to 500 C.

c. Recrystallization of the 10 per cent cold-worked alloys appeared to occur by subgrain growth rather than by conventional nucleation.

d. Recrystallization of 25 and 50 per cent cold-worked Zircaloy-2 and Zircaloy-3 occurred by nucleation and growth of new strain-free grains from the matrix.

e. Recrystallization was responsible for the major recovery in hardness or electrical resistivity.

f. Recrystallization temperature did not appear greatly influenced by the level of cold work. However, an increase in time of anneal decreased the temperature of recrystallization.

g. Zircaloy-3 recrystallized at lower temperatures and in shorter times than Zircaloy-2, other conditions being comparable.

h. The as-recrystallized grain sizes of Zircaloy-2 and Zircaloy-3 were:

Zircaloy-2	10 per cent cold work - 0.030 mm,
	25 per cent cold work - 0.018 mm,
	50 per cent cold work - 0.013 mm, penultimate grain size - 0.025 mm;
Zircaloy-3	10 per cent cold work - 0.024 mm,
	25 per cent cold work - 0.015 mm,
	50 per cent cold work - 0.010, penultimate grain size - 0.026 mm.

Grain Growth

a. Grain growth was most pronounced in alloys containing 10 per cent cold work. The final grain size was 0.055 to 0.060 mm. Higher levels of cold work resulted in grain sizes of 0.030 to 0.040 mm at 800 C and 1000 minutes.

b. There did not appear to be a marked difference in rate of grain growth in Zircaloy-2 and Zircaloy-3 over the range of conditions investigated.

Effects of Annealing Atmosphere

a. The major processes which occurred during annealing were recovery, recrystallization, and grain growth. Secondary processes included redistribution of impurities and absorption of gases. The latter was pronounced in air and helium atmospheres, less so in vacuum.

b. Kinetics of recrystallization and growth of Zircaloy-2 and Zircaloy-2 were essentially the same in helium and air, other conditions being equal. In vacuum, the rates for recrystallization were grossly decreased.

c. The limited differences in microstructure or hardness noted can be attributed to contamination from air during air or helium anneals. This was not true with electrical resistivity, where pronounced changes occurred above 500 C in air and helium, but were minimal in vacuum.

d. Vacuum anneals were suggested in thin sections where contamination may have adversely affected mechanical properties. Air or helium anneals should be satisfactory for more massive sections or where contamination is not a factor.

ACKNOWLEDGMENTS

This program was initiated as part of a comprehensive effort to study the effects of variables in heat treatment and fabrication of zirconium and zirconium alloys. The efforts of a number of persons made this program possible. Acknowledgment goes to L. F. Kendall, R. L. Hales, J. C. Tobin, and R. Todd who assisted in the rolling operations. The assistance of M. D. Jones with the annealing is gratefully acknowledged. Thanks go to R. L. Hales for making the resistivity measurements and to L. A. Hartcorn and his staff for their excellent metallographic work. The corrosion testing was done by the Bureau of Mines, Albany, Oregon. Special mention is due H. Kato and members of his group who conducted the testing there. We wish to thank J. C. Tobin for reviewing the manuscript and to acknowledge the many valuable discussions with him during the course of this work.

REFERENCES

1. Bush, S. H., R. S. Kemper and D. L. Gray. Recovery and Recrystallization of Zirconium and Its Alloys: Part I--Specimen Conditions, Cold Working, Heat Treatment, and Survey of the Literature, HW-69678. May, 1961.
2. Gray, D. L. Recovery and Recrystallization of Zirconium and Its Alloys: Part II--Annealing of Cold Worked Zirconium, HW-69679. March, 1961.
3. Bush, S. H., R. S. Kemper and D. L. Gray. Recovery and Recrystallization of Zirconium and Its Alloys: Part IV--Corrosion Behavior, HW-69681. May, 1961.
4. Richards, E. L. Metallographic Examination of Zircaloy-2 Subjected to Various Annealing Treatments, WAPD-RM-156. November 26, 1952.
5. Goodwin, J. G. and K. M. Goldman. Production Annealing of Zircaloy-2, WAPD-120. May 25, 1955.
6. Johnson, D. E. Some Effects of Cold Rolling, Annealing and Aging on the Mechanical Properties of Zircaloy-3b, HW-53621. November 15, 1957.
7. Herenguel, J., D. Whitwham and J. Boghen. "Recovery and Recrystallization of Zirconium and Its Alloys in Relation to Their Cold Work:" 1370-1372, Comptes Rendus 244, No. 10. March 4, 1957.

TABLES
OF
DATA

TABLE I

METALLOGRAPHIC EVALUATION OF CHANGES IN MICROSTRUCTURE OF COLD-WORKED ZIRCONIUM ALLOYS WITH TIME AND TEMPERATURE OF ANNEAL

Material	Per Cent Cold Work	Tempera-ture, C	Time, Minutes	Atmosphere	Recrystalli-zation	Grain Growth	Grain Size: ASTM	Grain Size: Diameter, mm	Precipitate	Remarks
Zircaloy-2	10	---	---	---	---	---	7.5-8	0.022-0.026	Limited	Limited striae, relatively smooth surface
	25	---	---	---	---	---	---	---	Limited	Similar to 10 per cent
	50	---	---	---	---	---	---	---	Limited	Pronounced grain elongation; surface roughening
Zircaloy-3A	10	---	---	---	---	---	7-8	0.022-0.030	Nominal	Relatively smooth surface
	25	---	---	---	---	---	---	---	Nominal	Similar to 10 per cent
	50	---	---	---	---	---	---	---	Nominal	Pronounced grain elongation; surface roughening
Zircaloy-3V	10	---	---	---	---	---	7-8	0.022-0.030	Limited	Relatively smooth surface
	25	---	---	---	---	---	---	---	Limited	Nominal distortion
	50	---	---	---	---	---	---	---	Limited	Grain elongation; roughening
Zircaloy-2	10	300	10	Air	---	---	---	---	Limited	Grain boundaries sharper
	10	300	100	Air	---	---	---	---	Limited	---
	10	300	1000	Air	---	---	---	---	Limited	---
Zircaloy-3A	10	300	10	Air	---	---	---	---	Limited	A few twins; light precipitate
	10	300	100	Air	---	---	---	---	Limited	---
	10	300	1000	Air	---	---	---	---	Increasing	---
Zircaloy-2	25	300	10	Helium	---	---	---	---	Some	---
	25	300	100	Helium	---	---	---	---	Increasing	---
	25	300	1000	Helium	---	---	---	---	Similar to 100 minutes	---
Zircaloy-3A	25	300	10	Helium	---	---	---	---	Limited	Grain and grain boundary precipitate
	25	300	100	Helium	Precipitate or nuclei (100 and 1000 minutes)	---	---	---	More	---
	25	300	1000	Helium		---	---	---	More	---
Zircaloy-2	50	300	10	Vacuum	---	---	---	---	Extensive	Rough surface
	50	300	100	Vacuum	---	---	---	---	Extensive	Rough surface
	50	300	1000	Vacuum	---	---	---	---	Extensive	Rough surface
Zircaloy-3A	50	300	10	Vacuum	---	---	---	---	Extensive	Globular precipitate
	50	300	100	Vacuum	---	---	---	---	Same	---
	50	300	1000	Vacuum	---	---	---	---	Same	---
Zircaloy-3V	50	300	10	Vacuum	---	---	---	---	Same	Spheroidal precipitate network at or near grain boundary
	50	300	100	Vacuum	---	---	---	---	Increasing	Similar to 10 minutes
	50	300	1000	Vacuum	---	---	---	---	More	Grain boundary precipitate almost continuous
Zircaloy-2	10	400	10	Air	---	---	---	---	Greater than 300 C	Possible subgrain growth
	10	400	100	Air	Nuclei?	---	---	---	Scattered	A few twins
	10	400	1000	Air	Possible <5 per cent	---	---	---	---	Twins

TABLE I (Contd)

Material	Per Cent Cold Work	Tempera-ture, C	Time Minutes	Atmosphere	Recrystallization	Grain Growth	Grain Size ASTM	Grain Size Diameter, mm	Precipitate	Remarks
Zircaloy-3A	10	400	10	Air	---	---	---	---	Limited	---
	10	400	100	Air	---	---	---	---	---	---
	10	400	1000	Air	---	---	---	---	---	Grain boundaries sharper
Zircaloy-2	25	400	10	Helium	---	---	---	---	More precipitate than 300 C	Some grain boundary sharpening
	25	400	100	Helium	---	---	---	---	More precipitate than 300 C	---
	25	400	1000	Helium	---	---	---	---	More precipitate than 300 C	---
Zircaloy-3A	25	400	10	Helium	---	---	---	---	Some	---
	25	400	100	Helium	Precipitate or nuclei	---	---	---	More	---
	25	400	1000	Helium	2-5 per cent	---	---	---	More	---
Zircaloy-2	50	400	10	Vacuum	---	---	---	---	Less than 300 C	Sharper
	50	400	100	Vacuum	---	---	---	---	---	Surface smoother than 300 C
	50	400	1000	Vacuum	---	---	---	---	---	---
Zircaloy-3A	50	400	10	Vacuum	---	---	---	---	Increased	---
	50	400	100	Vacuum	---	---	---	---	Same	Ledged appearance
	50	400	1000	Vacuum	---	---	---	---	General	---
Zircaloy-3V	50	400	10	Vacuum	---	---	---	---	Some	Detail sharper than 300 C
	50	400	100	Vacuum	---	---	---	---	Increasing	Clear structure
	50	400	1000	Vacuum	---	---	---	---	Increasing	Less precipitate than 300 C and 1000 minutes
Zircaloy-2	10	500	10	Air	---	---	---	---	Similar to 400 C	---
	10	500	100	Air	Nuclei in several areas	---	---	---	Similar to 400 C	---
	10	500	1000	Air	Some new grains	---	---	---	Similar to 400 C	---
Zircaloy-3A	10	500	10	Air	---	---	---	---	---	---
	10	500	100	Air	Possible nuclei	---	---	---	---	Nuclei or precipitate
	10	500	1000	Air	None visible here	---	---	---	---	---
Zircaloy-2	25	500	10	Helium	---	---	---	---	Similar to 400 C	---
	25	500	100	Helium	Possible nuclei	---	---	---	Similar to 400 C	---
	25	500	1000	Helium	Limited nucleation	---	---	---	Similar to 400 C	---
Zircaloy-3A	25	500	10	Helium	Precipitate or nuclei	---	---	---	Some	---
	25	500	100	Helium	5-10 per cent	---	---	---	Some	Grains very small
	25	500	1000	Helium	Extension	---	---	---	Some	---
Zircaloy-2	50	500	10	Vacuum	---	---	---	---	Similar to 400 C	---
	50	500	100	Vacuum	---	---	---	---	---	---
	50	500	1000	Vacuum	Possibly a few nuclei	---	---	---	---	---

TABLE I (Contd)

Material	Per Cent Cold Work	Temperature C	Time, Minutes	Atmosphere	Recrystallization	Grain Growth	Grain Size ASTM	Grain Size Diameter, mm	Precipitate	Remarks
Zircaloy-3A	50	500	10	Vacuum	None	---	---	---	Less precipitate	---
	50	500	100	Vacuum	Nuclei?	---	---	---	---	Stringered precipitate
	50	500	1000	Vacuum	Limited	---	---	---	---	Globular precipitate
Zircaloy-3V	50	500	10	Vacuum	---	---	---	---	<400 C and 10 minutes	---
	50	500	100	Vacuum	---	---	---	---	Increasing	Coarse globular precipitate; also fine precipitate
	50	500	1000	Vacuum	<1 per cent	---	---	---	Increasing	---
Zircaloy-2	10	600	10	Air	None	---	---	---	Limited	---
	10	600	100	Air	Nuclei	---	---	---	---	---
	10	600	1000	Air	Essentially recrystallized	Limited local growth	6.5-9	0.016-0.035	---	---
Zircaloy-3A	10	600	10	Air	Some	Growth of equiaxed grains	---	---	Some	---
	10	600	100	Air	Substantial	---	~8	0.022	Decreasing	---
	10	600	1000	Air	~100 per cent	Definite	5-7	0.030-0.065	---	General light precipitate
Zircaloy-2	25	600	10	Helium	None	---	---	---	Limited	---
	25	600	100	Helium	>95 per cent	---	<8	<0.022	---	---
	25	600	1000	Helium	100 per cent	Some	7-9	0.016-0.030	---	General coarsening
Zircaloy-3A	25	600	10	Helium	Some	---	---	---	Some	---
	25	600	100	Helium	100 per cent	Same	7.5-10	0.011-0.020	Some	Mixed grain size
	25	600	1000	Helium	100 per cent	General	8-10	0.011-0.022	Some	General growth of smaller particles
Zircaloy-2	50	600	10	Vacuum	None	---	---	---	---	Sharp structure
	50	600	100	Vacuum	Some	---	---	---	---	---
	50	600	1000	Vacuum	~75 per cent	---	---	---	---	Many tiny grains
Zircaloy-3A	50	600	10	Vacuum	Definite	---	---	---	General	---
	50	600	100	Vacuum	~20 per cent	---	---	---	Decreasing	---
	50	600	1000	Vacuum	100 per cent	---	10	0.011	---	---
Zircaloy-3V	50	600	10	Vacuum	Possible nuclei	---	---	---	Limited	Striae
	50	600	100	Vacuum	1-2 per cent	---	---	---	Limited	---
	50	600	1000	Vacuum	100 per cent	---	9	(0.016)	Limited	Striae in structure
Zircaloy-2	10	700	10	Air	By subgrain growth	Yes, mixed	6-9	0.016-0.045	Limited	Sharp structure, mixed grain size
	10	700	100	Air	Complete	Definite	7	0.030	Limited	---
	10	700	1000	Air	Complete	Increased	6	0.045	Limited	---
Zircaloy-3A	10	700	10	Air	Some	---	~8	0.022	Some	General light precipitate
	10	700	100	Air	100 per cent	Yes	5-7	0.030-0.065	---	---
	10	700	1000	Air	100 per cent	Similar to 100 minutes	5-7	0.030-0.065	Limited	---

TABLE I (Contd)

Material	Per Cent Cold Work	Temperature C	Time, Minutes	Atmosphere	Recrystallization	Grain Growth	Grain Size ASTM	Grain Size Diameter, mm	Precipitate	Remarks
Zircaloy-2	25	700	10	Helium	~50 per cent	---	---	---	Limited	---
	25	700	100	Helium	100 per cent	---	7-8	0.016-0.30	Limited	---
	25	700	1000	Helium	---	---	8	0.022	Limited	More uniform grain size
Zircaloy-3A	25	700	10	Helium	100 per c nt	---	8-10	0.011-0.022	Uniform	Equiaxed
	25	700	100	Helium	100 per cent	Some	8	0.022	Uniform	Equiaxed
	25	700	1000	Helium	100 per cent	---	8	0.022	Uniform	---
Zircaloy-2	50	700	10	Vacuum	40-60 per cent (very fine)	---	---	---	---	---
	50	700	100	Vacuum	100 per cent	---	8-10	0.011-0.022	---	---
	50	700	1000	Vacuum	100 per cent	Limited	7(9)10	0.030(0.016)0.011	---	---
Zircaloy-3A	50	700	10	Vacuum	~100 per cent	---	9<10	0.016<0.011	Essentially none	---
	50	700	100	Vacuum	100 per cent	Yes	8(9-10)	0.022(0.011-0.026)	Limited	Equiaxed
	50	700	1000	Vacuum	100 per cent	Yes	8-10	0.022-0.011	Limited	---
Zircaloy-3V	50	700	10	Vacuum	~100 per cent	---	9-10	0.011-0.016	Nominal	---
	50	700	100	Vacuum	100 per cent	---	---	---	---	---
	50	700	1000	Vacuum	100 per cent	Definite	9-10	0.011-0.016	Nominal	Scattered stringers of globular precipitate
Zircaloy-2	10	800	10	Air	100 per cent	Yes	8-9	0.016-0.020	Nominal	---
	10	800	100	Air	100 per cent	Pronounced	7-some 6.5	0.030(0.035)	Extensive	Random precipitate
Zircaloy-3A	10	800	10	Air	100 per cent	Yes	5.5-6.5	(0.035)-0.055	General	---
	10	800	100	Air	100 per cent	Yes	6(7)	(0.030)-0.045	Grain boundary	Increased growth
Zircaloy-2	25	800	10	Helium	100 per cent	---	6(7)	(0.030)-0.045	---	---
	25	800	100	Helium	---	---	7(8)9	0.016(0.022)0.030	---	---
	25	800	1000	Helium	---	Definite	(7)8&9	(0.016)0.022-0.030	---	---
Zircaloy-3A	25	800	10	Helium	100 per cent	---	(6)7	0.030(0.045)	---	---
	25	800	100	Helium	100 per cent	Limited	8-10	0.011-0.022	Less precipitate	---
	25	800	1000	Helium	100 per cent	Definite	7.5-8.5	0.019-0.026	---	---
Zircaloy-2	50	800	10	Vacuum	100 per cent	---	7	0.030	---	Precipitate at grain boundaries
	50	800	100	Vacuum	---	---	8(9)	(0.016)0.022	Nominal	Equiaxed grains
	50	800	1000	Vacuum	---	Coarsening	8(9)	(0.016)0.022	---	Nominal precipitate
Zircaloy-3A	50	800	10	Vacuum	100 per cent	Yes	7-8	0.022-0.030	---	---
	50	800	100	Vacuum	100 per cent	Similar to 10 minutes	8	0.022	---	Equiaxed, very uniform grains
	50	800	1000	Vacuum	100 per cent	Definite	7-8	0.022-0.030	Some	Stringent precipitate

( ) Majority of grains this size.

TABLE II

RECRYSTALLIZATION AND GROWTH OF ZIRCONIUM ALLOYS

	Zircaloy-3A			Zircaloy-2		
Cold Work:	10 per cent			10 per cent		
Atmosphere:	Air			Air		
Time, minutes / Temperature, C	10	100	1000	10	100	1000
300	N	N	N	N	N	N
400	N	N	N	N	N	N
500	N	I. R. ?	N	N	N	I. R. ?
600	I. R.	I. R.	~R & G	N ?	I. R. ?	I. R.
700	>I. R.&G	R & G	G	R & G	I. R.	~R
800	R & G	G	--	R & G	G	G
					>G	--

	Zircaloy-3A			Zircaloy-2		
Cold Work:	25 per cent			25 per cent		
Atmosphere:	Helium			Helium		
Time, minutes / Temperature, C	10	100	1000	10	100	1000
300	N	N	N	N	N	N
400	N	N	I. R. ?	N	N	N
500	N	I. R. ?	I. R.	N	N ?	I. R.
600	I. R. ?	R	R & G	N	>I. R.	R & G
700	R	G	G	>I. R. (~50 per cent)	R	R & G
800	R & G	G	>G	R	R	G

	Zircaloy-3A			Zircaloy-2		
Cold Work:	50 per cent			50 per cent		
Atmosphere:	Vacuum			Vacuum		
Time, minutes / Temperature, C	10	100	1000	.10	100	1000
300	N	N	N	N	N	N
400	N	N	N	N	N	N
500	N	N	I. R.	N	N	N
600	I. R. ?	>I. R.	R	N	I. R.	R(~75 per cent)
700	R(~100 per cent)	R & G	G	I. R. (~60 per cent)	R	G
800	R & G	G	>G	R	R	R & G

Code: N - No change
I. R. - Recrystallization initiated
R - Complete recrystallization
G - Grain growth

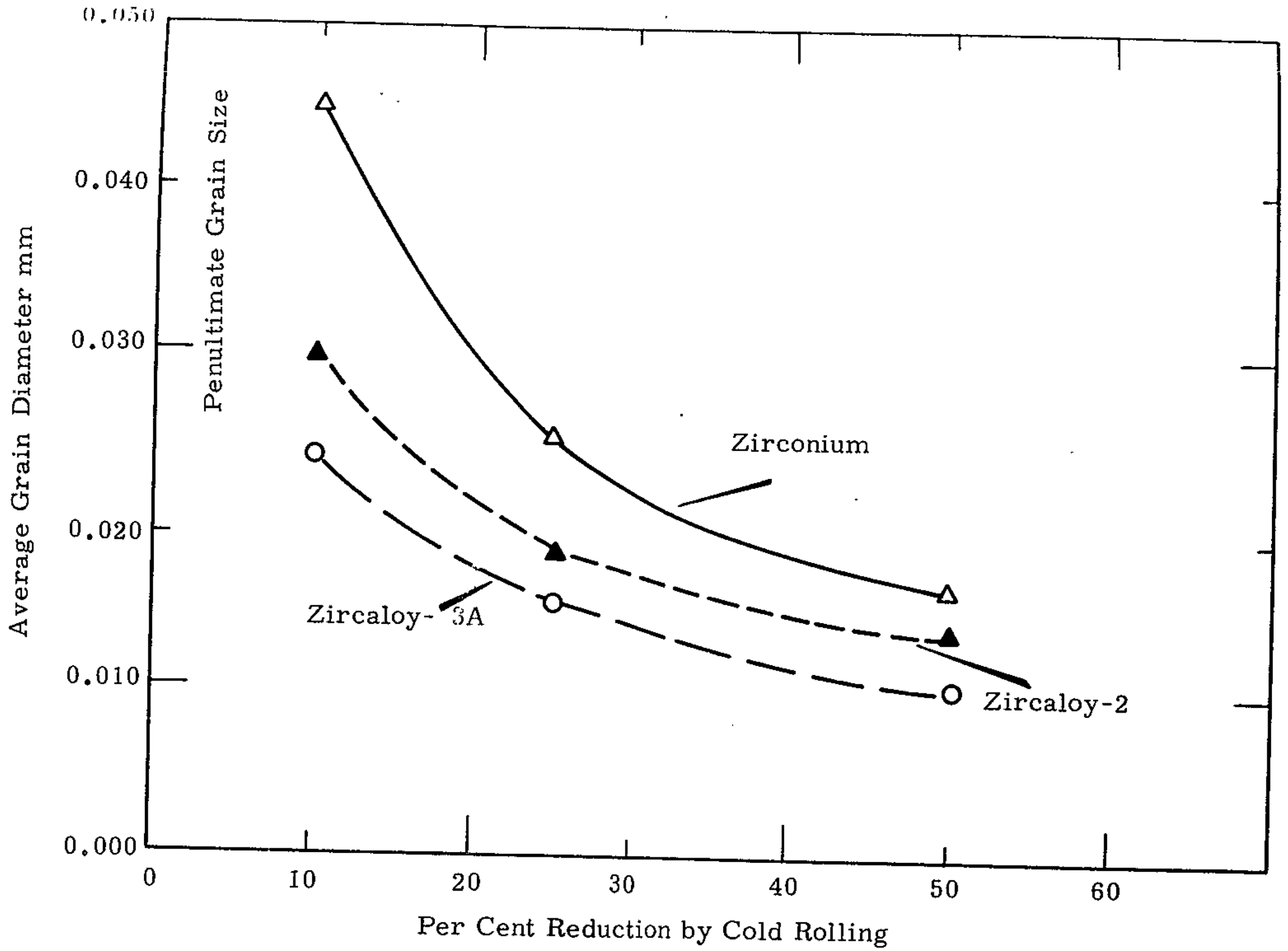
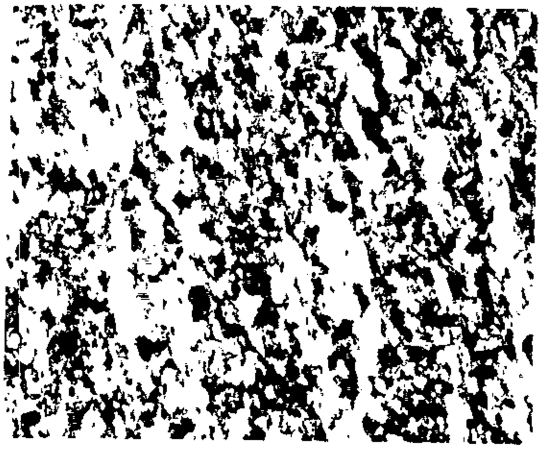
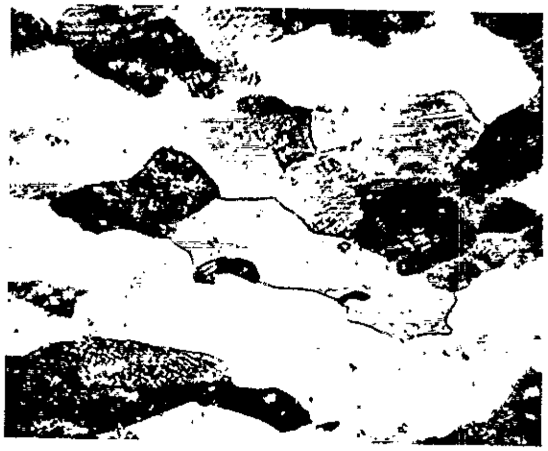


FIGURE 1

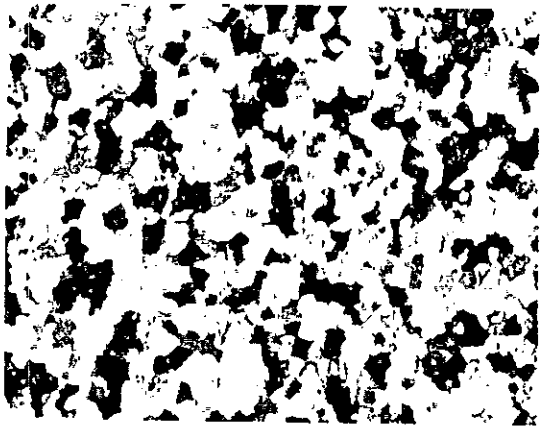
Grain Size when Recrystallization Appears Complete, as a Function of Cold Work

100 X

10-Minutes Anneal

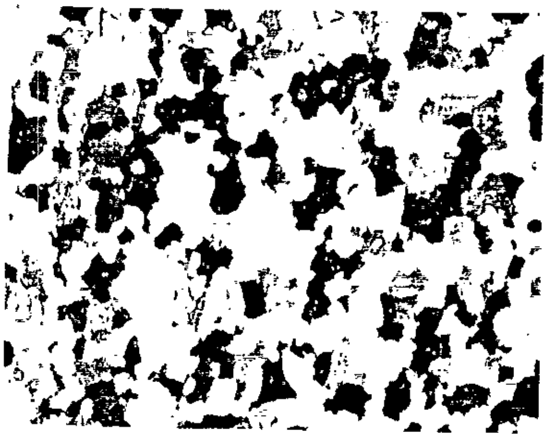
1000 X




100 X

100-Minutes Anneal

1000 X




100 X

1000-Minutes Anneal

1000 X

FIGURE 2

Microstructure of Zircaloy-2 Cold Worked 10 Per Cent, then Annealed at 700 C in Air; "B" Etch; Anodized

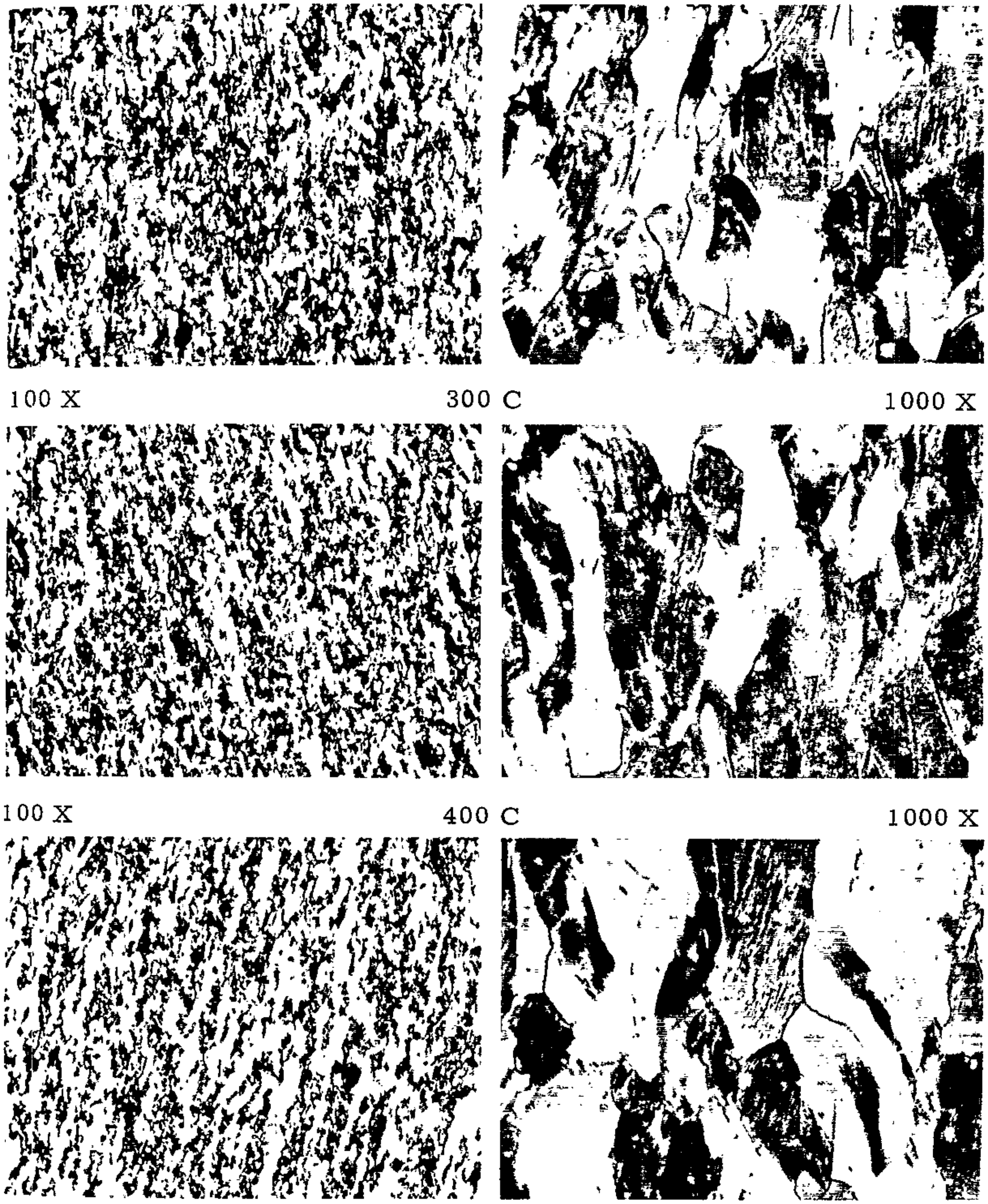
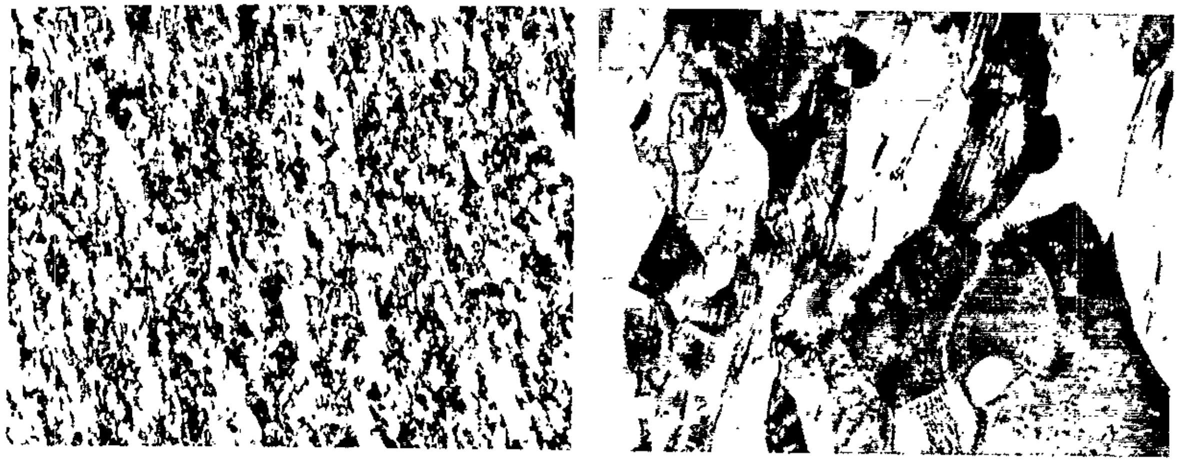
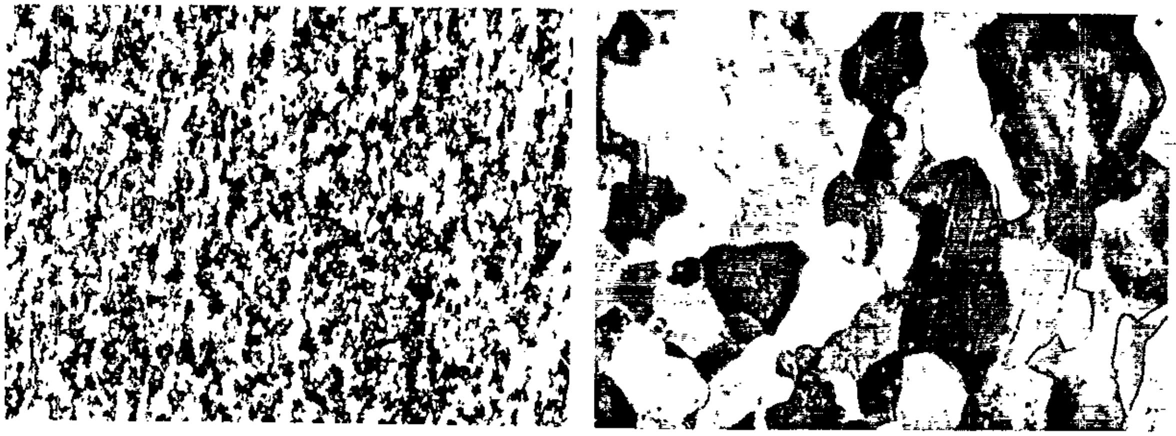


FIGURE 3

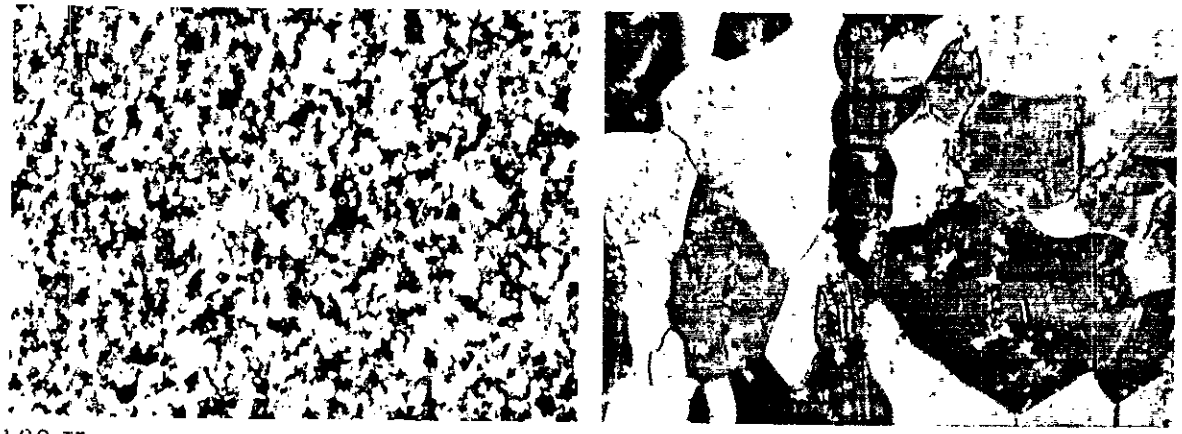
Microstructure of Zircaloy-2 Cold Worked 25 Per Cent, then Annealed 100 Minutes in Helium at Various Temperatures; "B" Etch; Anodized



100 X 10-Minutes Anneal 1000 X



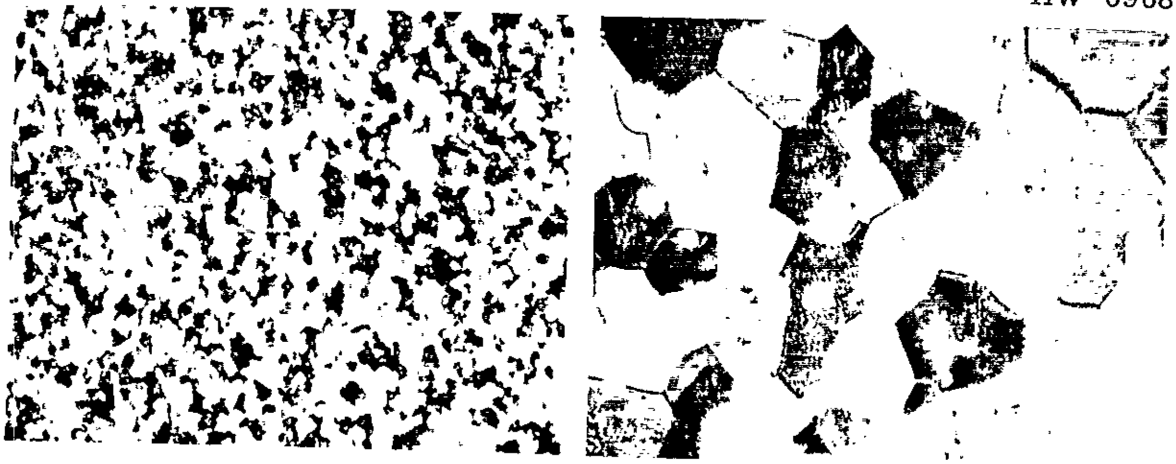
100 X 100-Minutes Anneal 1000 X



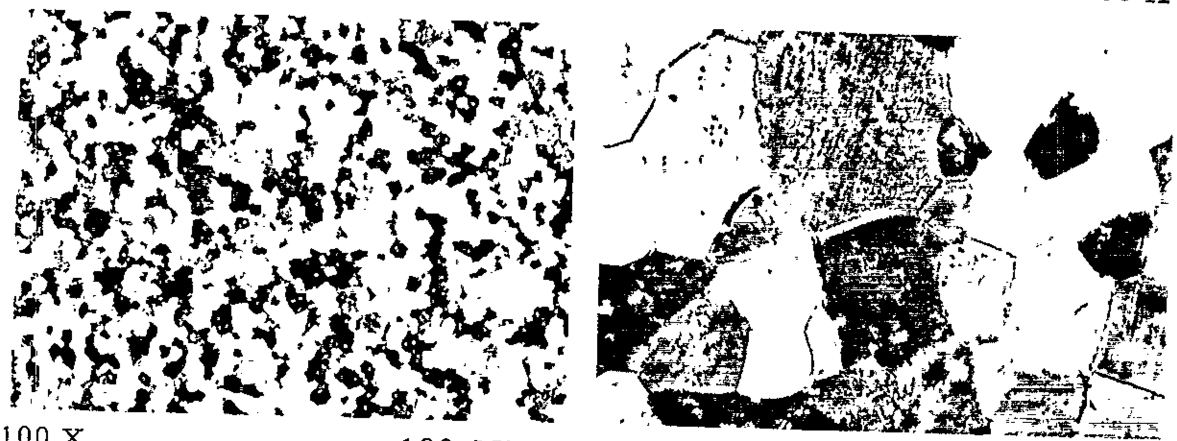
100 X 1000-Minutes Anneal 1000 X

FIGURE 4

Microstructure of Zircaloy-2 Cold Worked 25 Per Cent, then Annealed at 600 C in Helium; "B" Etch; Anodized



100 X 10-Minutes Anneal 1000 X



100 X 100-Minutes Anneal 1000 X

100 X 1000-Minutes Anneal 1000 X

FIGURE 5

Microstructure of Zircaloy-2 Cold Worked 25 Per Cent, then Annealed at 800 C in Helium; "B" Etch; Anodized

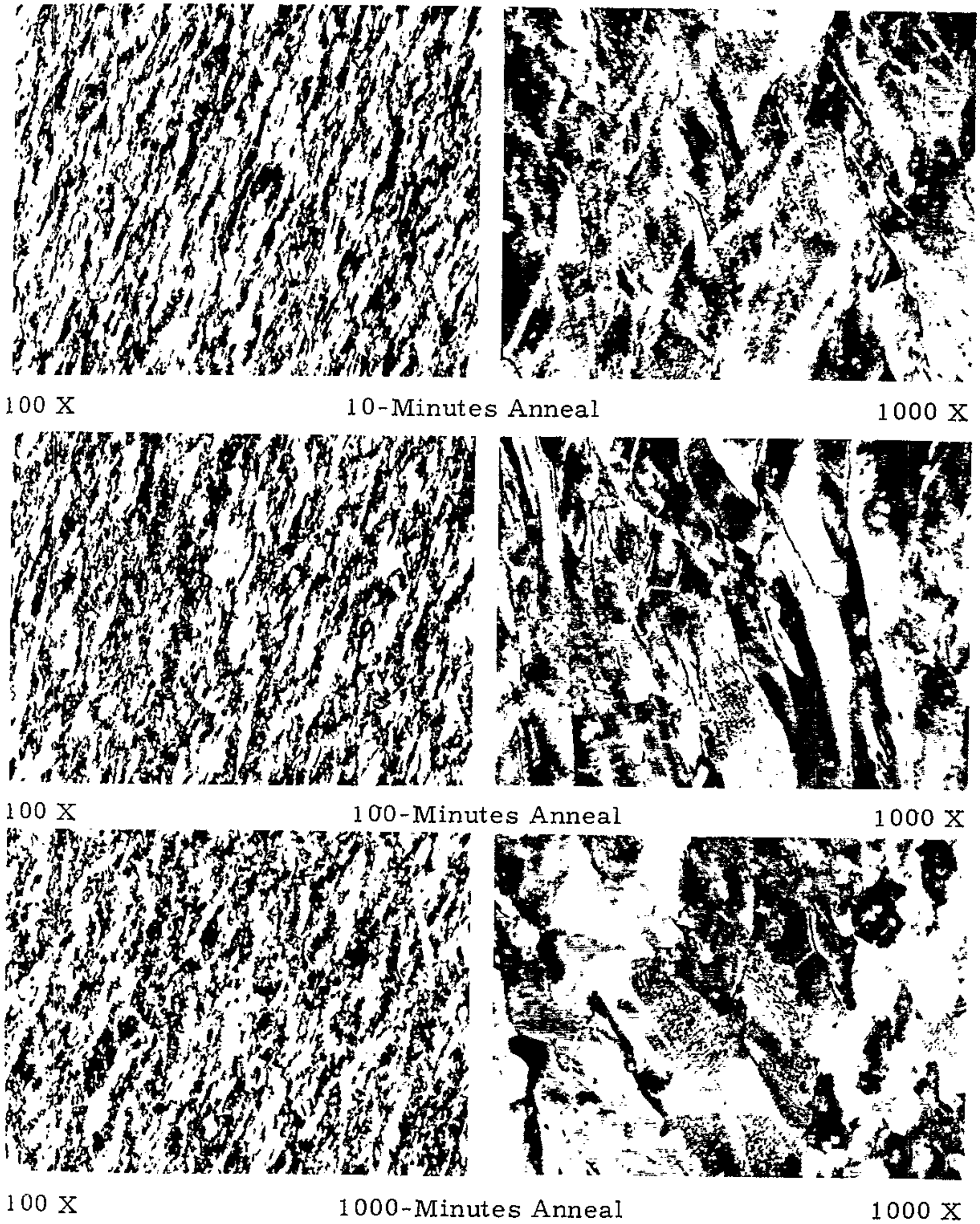


FIGURE 6

Microstructure of Zircaloy-2 Cold Worked 50 Per Cent, then Annealed at 600 C in Vacuum; "B" Etch; Anodized

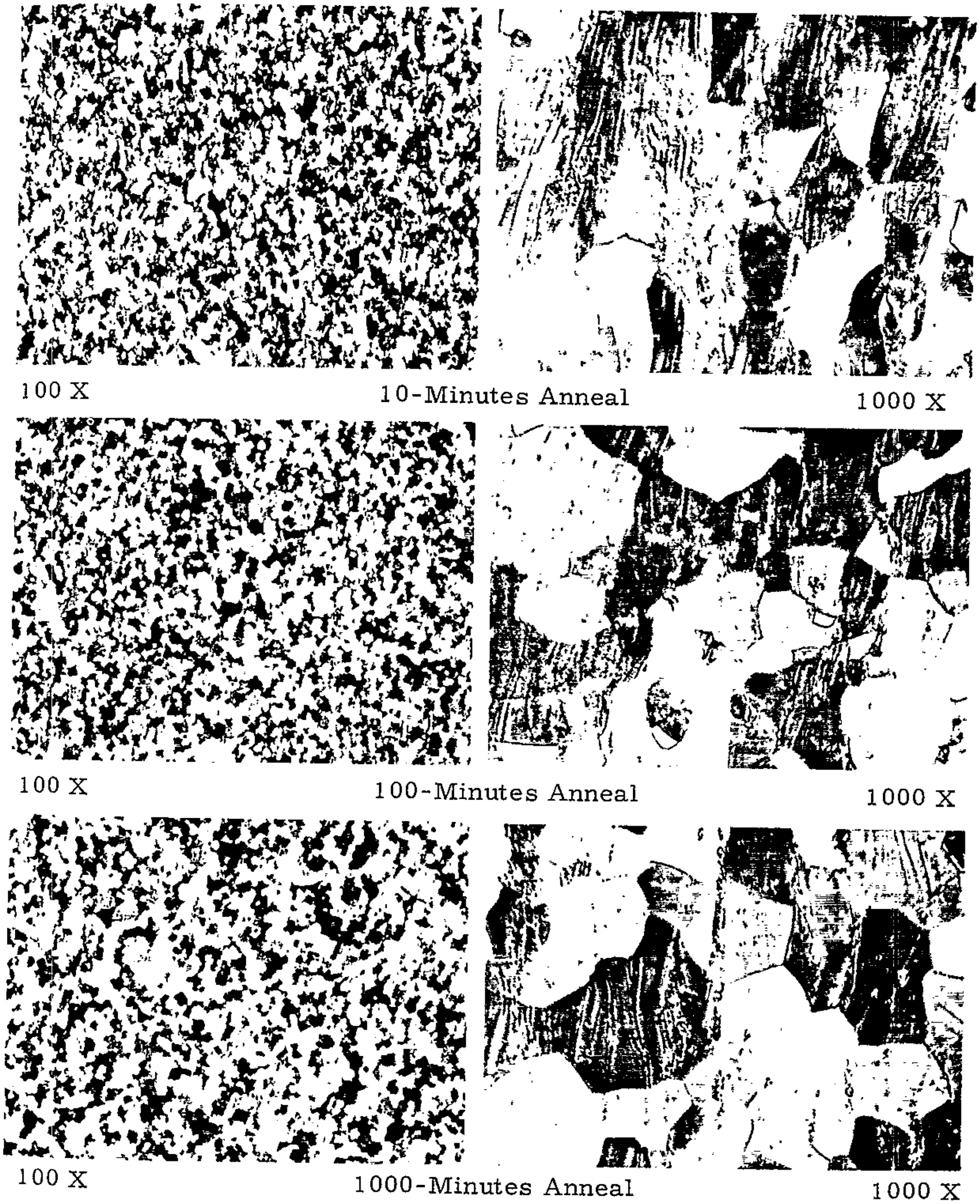
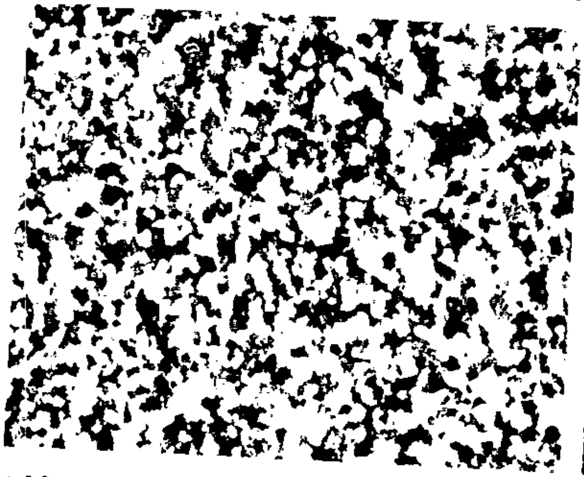


FIGURE 7

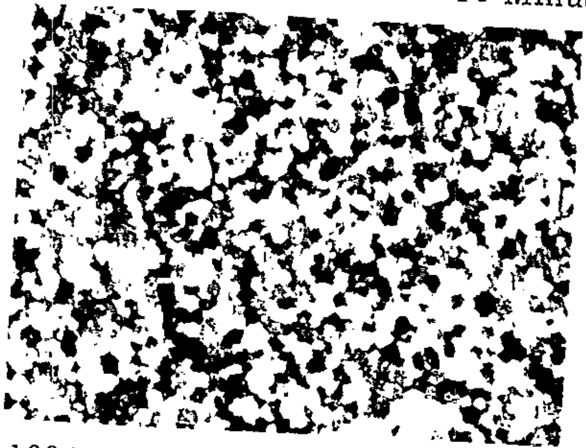
Microstructure of Zircaloy-3A Cold Worked 25 Per Cent, then Annealed at 600 C in Helium; "B" Etch; Anodized




100 X

10-Minutes Anneal

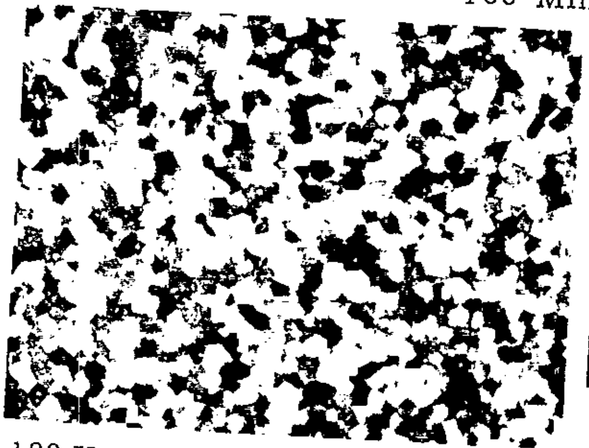
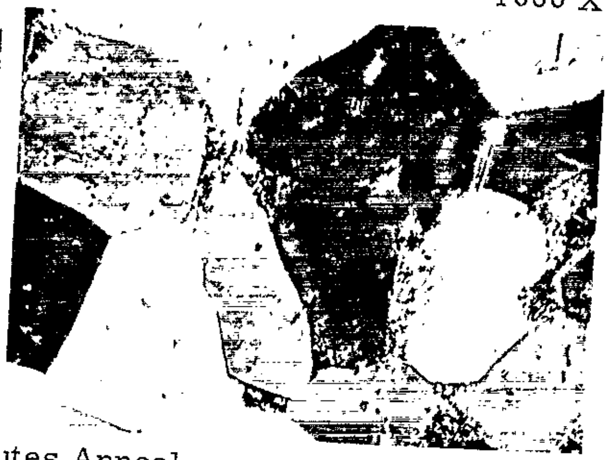
1000 X




100 X

100-Minutes Anneal

1000 X

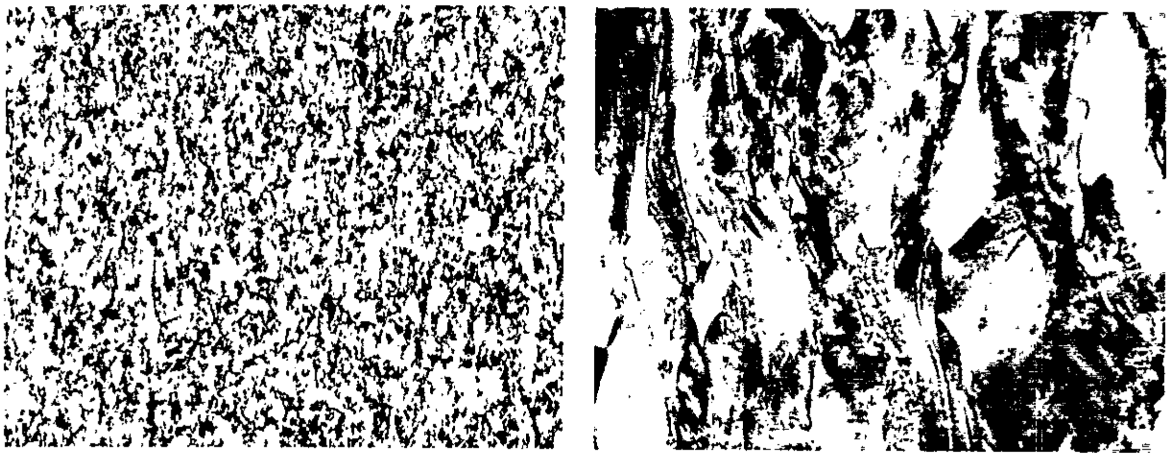
100 X

1000-Minutes Anneal

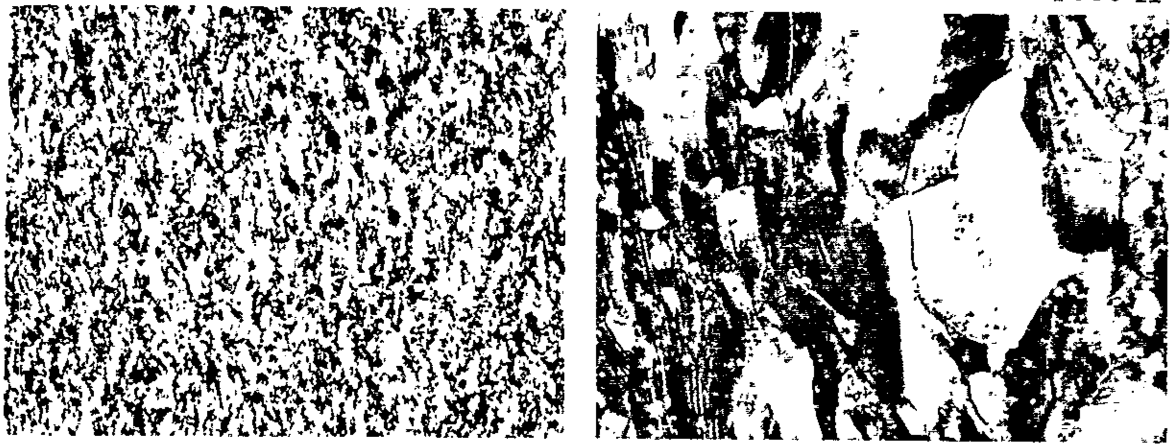
1000 X

FIGURE 8

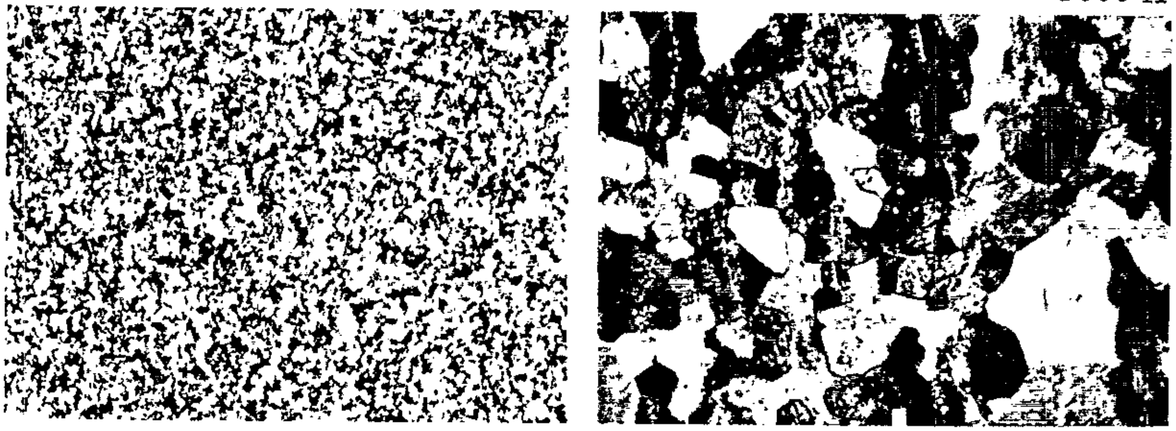
Microstructure of Zircaloy-3A Cold Worked 25 Per Cent, then Annealed at 800 C in Helium; "B" Etch; Anodized



100 X 10-Minutes Anneal 1000 X



100 X 100-Minutes Anneal 1000 X



100 X 1000-Minutes Anneal 1000 X

FIGURE 9

Microstructure of Zircaloy-3A Cold Worked 50 Per Cent, then Annealed at 600 C in Vacuum; "B" Etch; Anodized

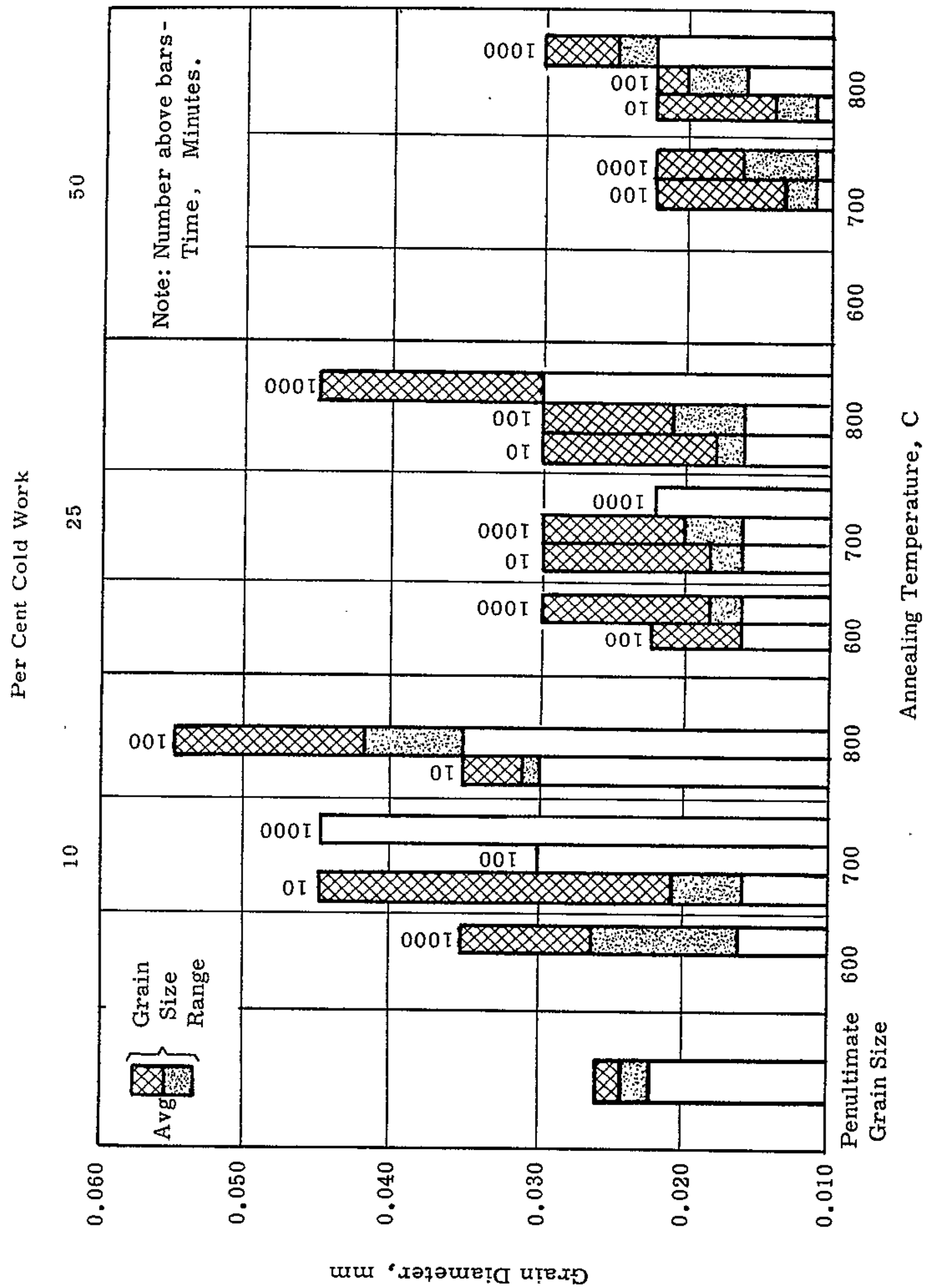


FIGURE 10

Grain Growth of Zircaloy-2 for Various Annealing Times and Temperatures as a Function of Cold Work

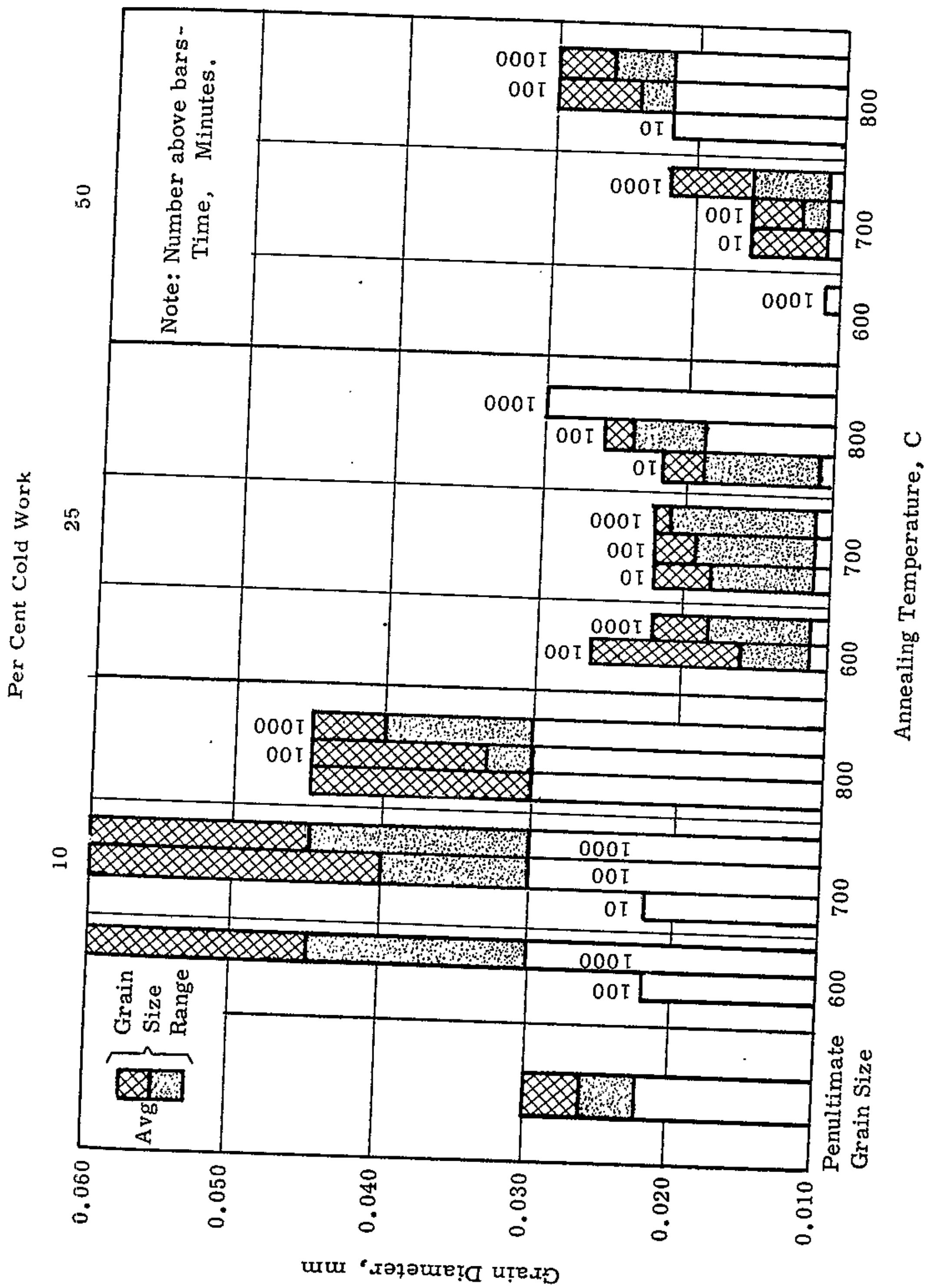


FIGURE 11

Grain Growth of Zircaloy-3A for Various Annealing Times and Temperatures as a Function of Cold Work

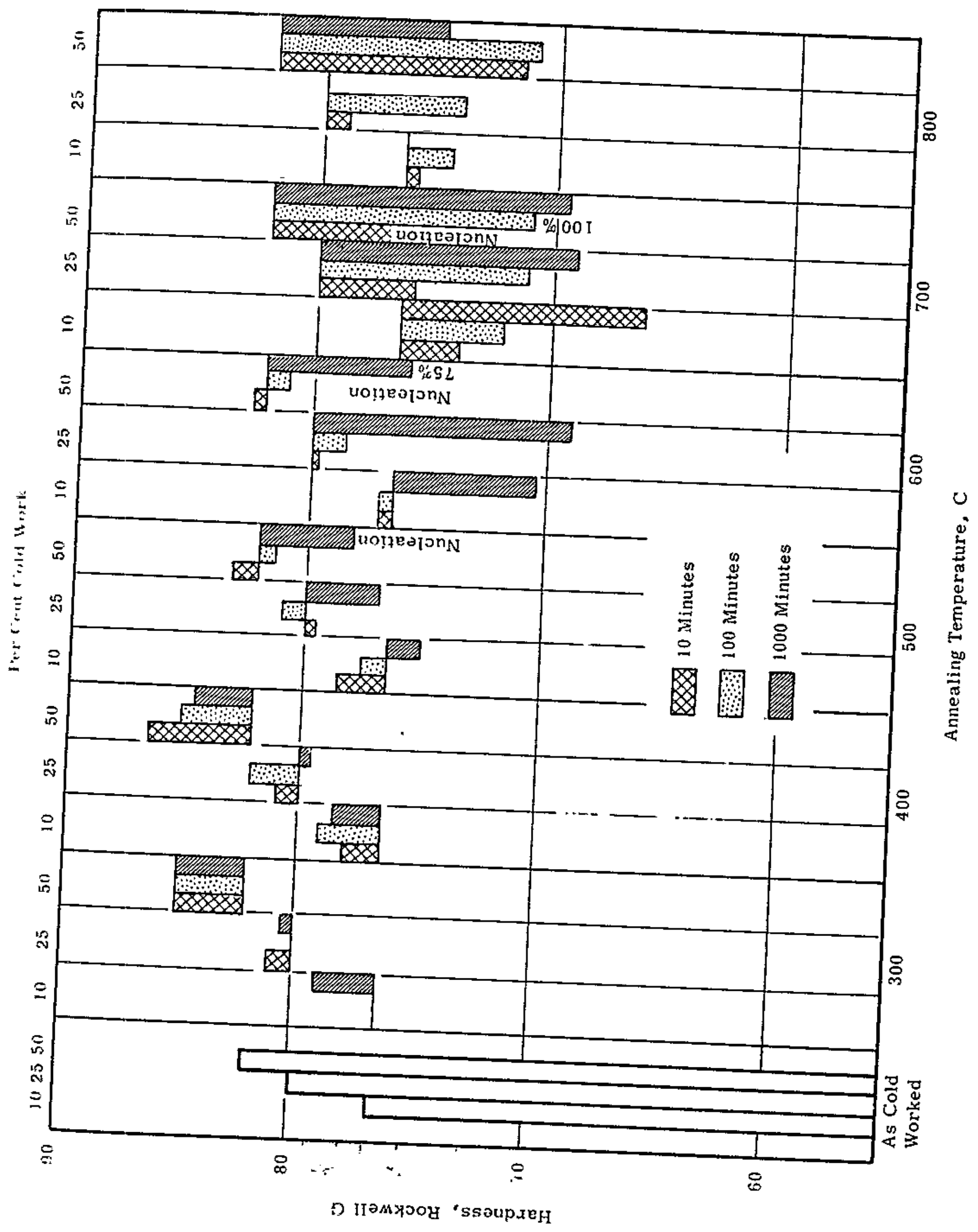


FIGURE 12

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-2; Vacuum Atmosphere

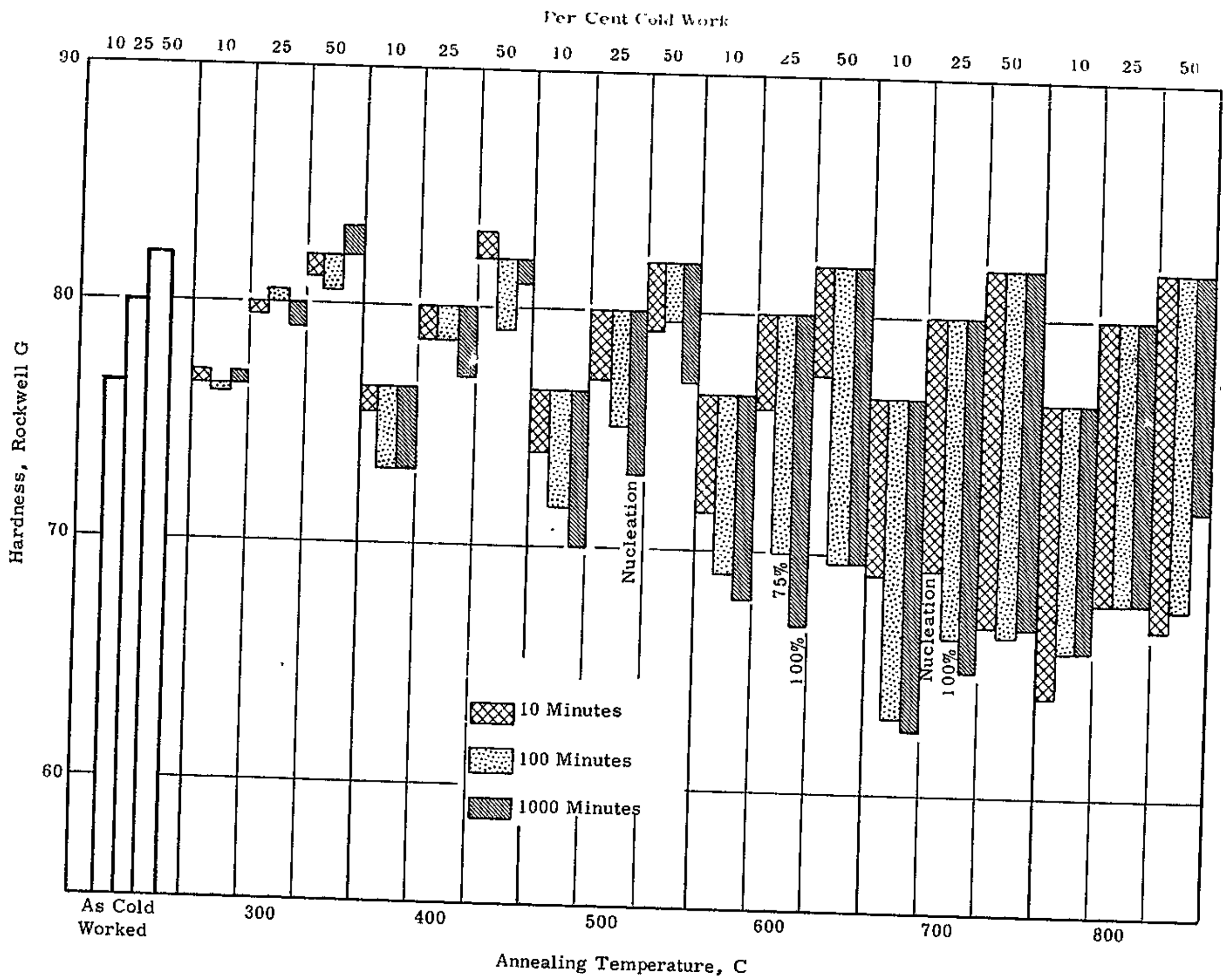


FIGURE 13

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-2; Helium Atmosphere

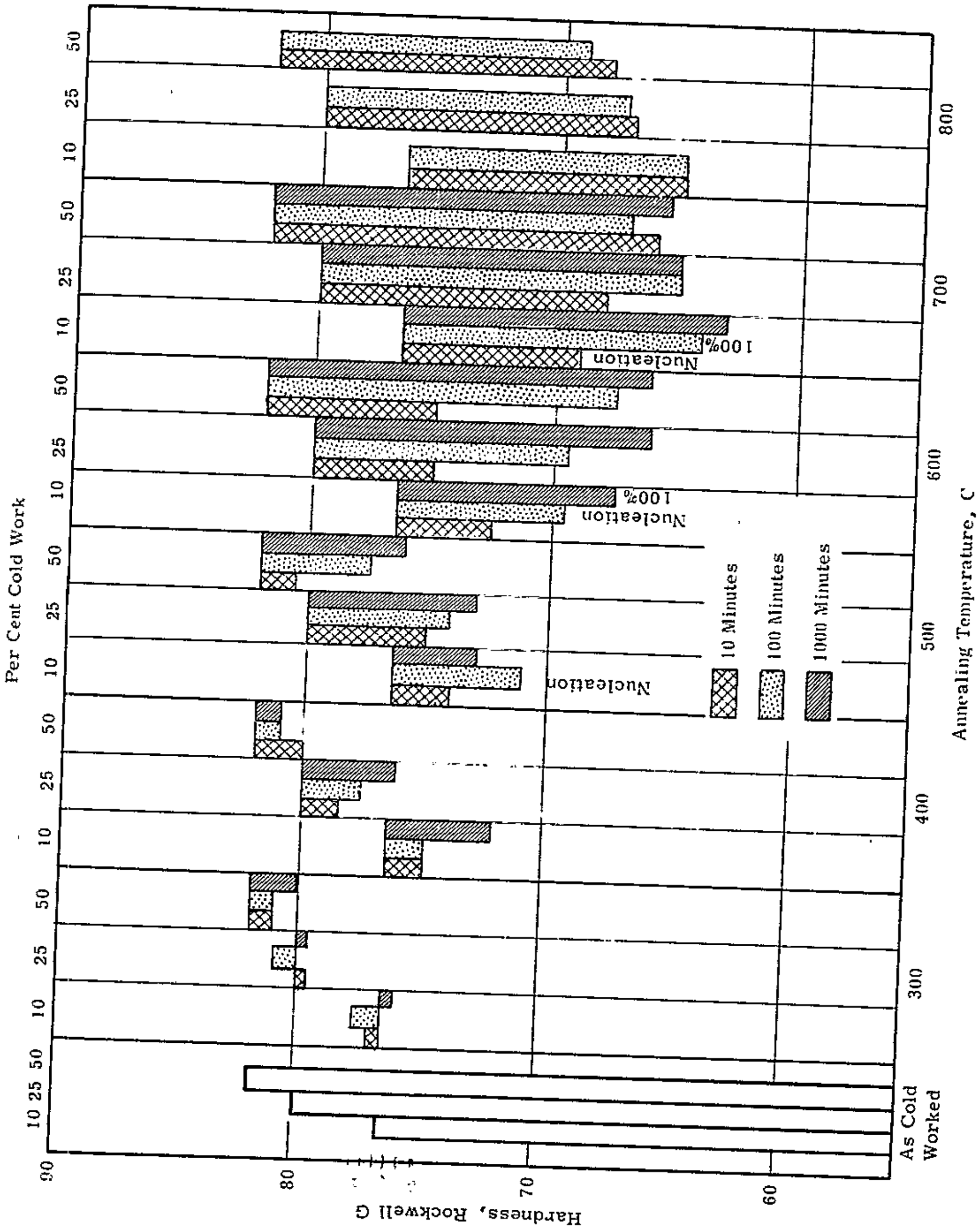


FIGURE 14

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-2; Air Atmosphere

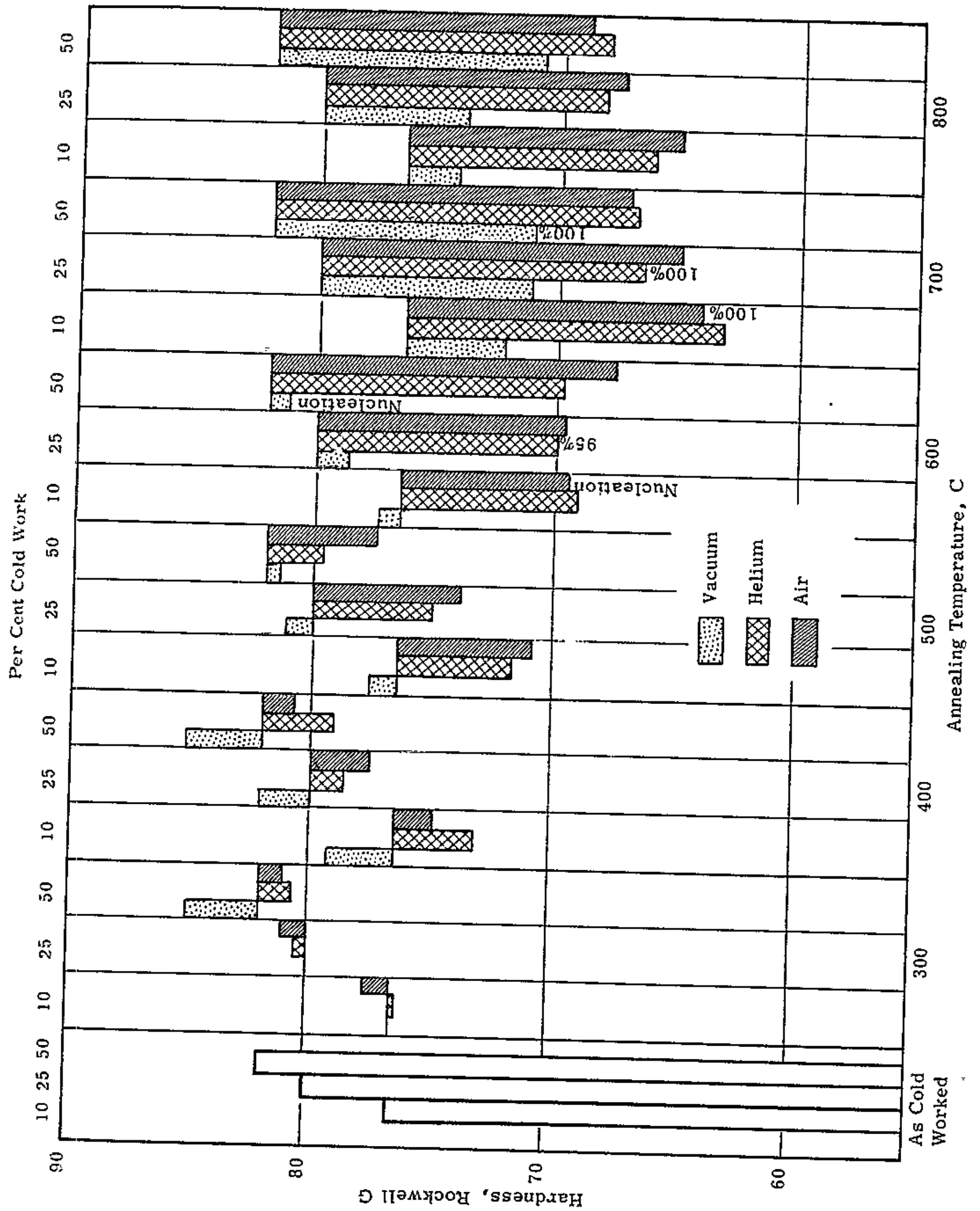


FIGURE 15

Effect of Annealing Atmosphere on the Hardness of Cold-Worked Zircaloy-2; 100 Minutes

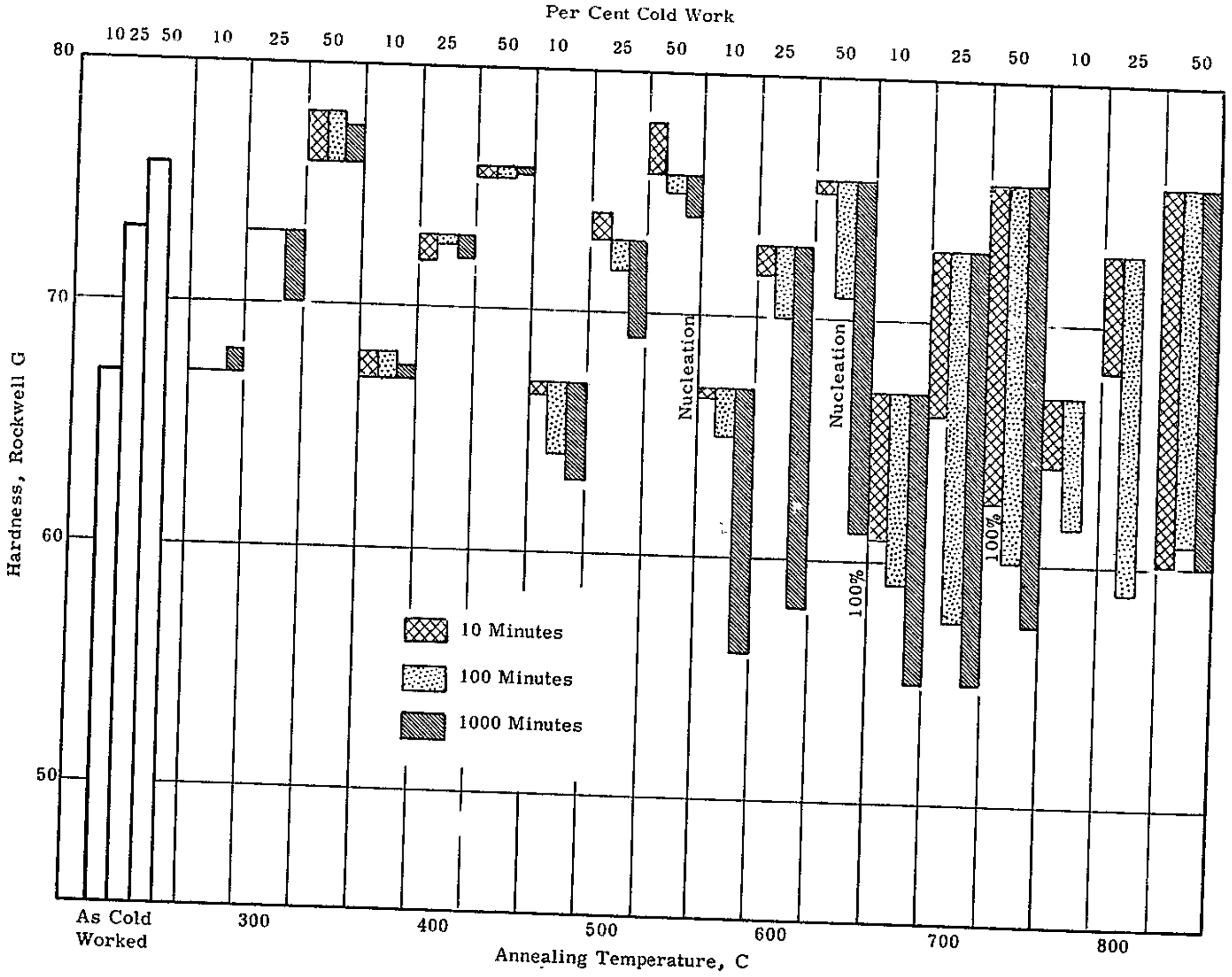


FIGURE 16

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3A; Vacuum Atmosphere

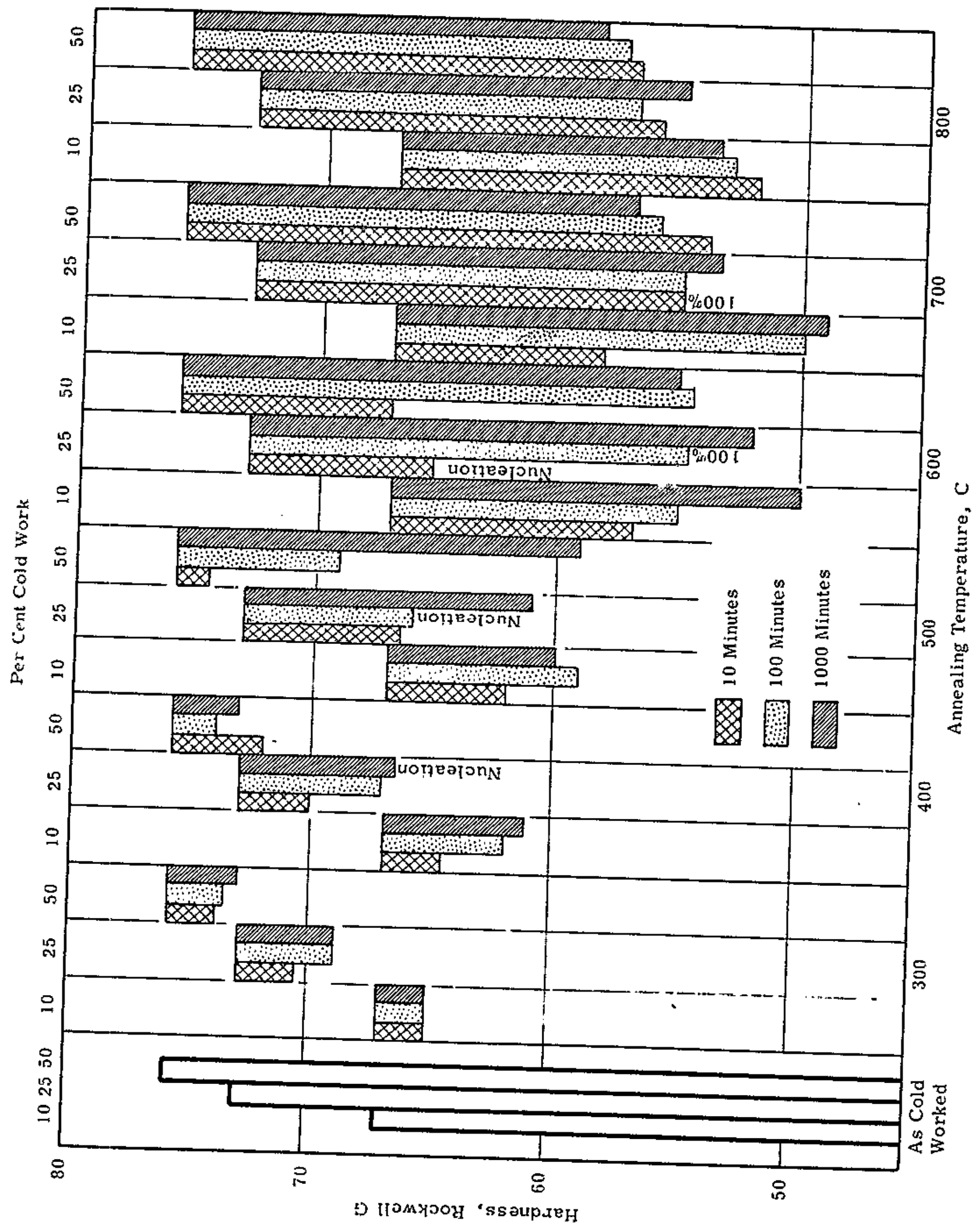


FIGURE 17

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3A; Helium Atmosphere

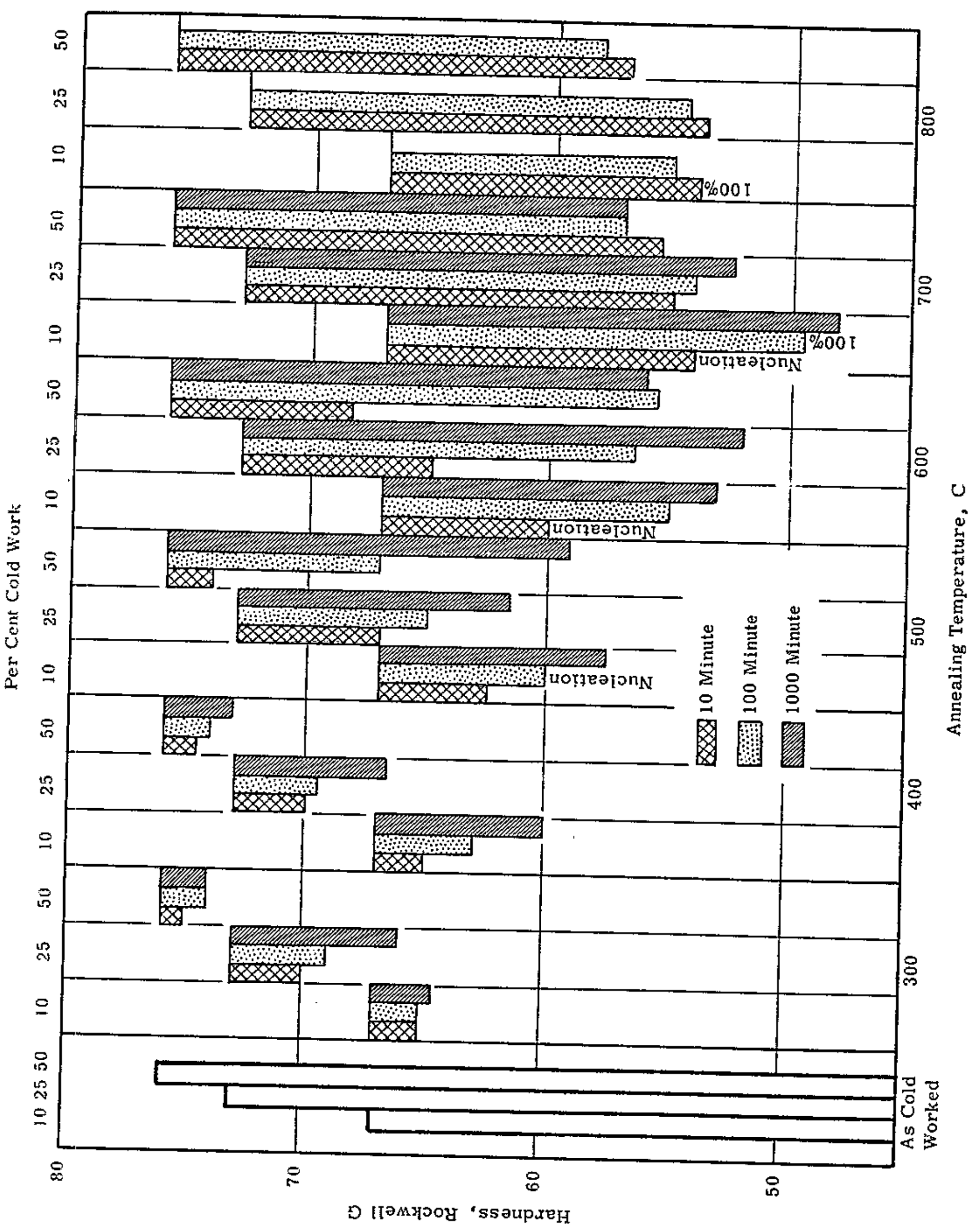


FIGURE 18

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3A; Air Atmosphere

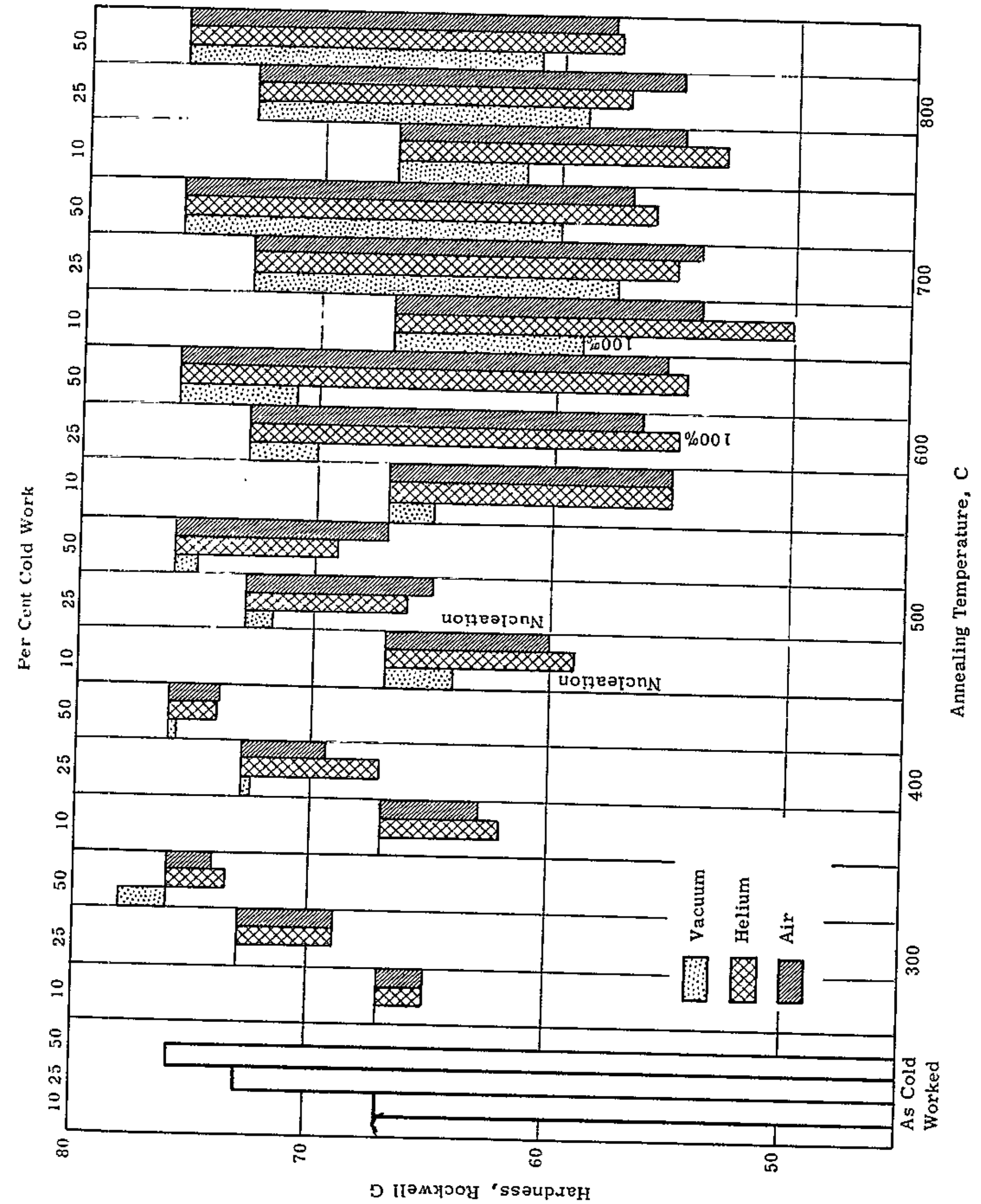


FIGURE 19

Effect of Annealing Atmosphere on the Hardness of Cold-Worked Zircaloy-3A; 100 Minutes

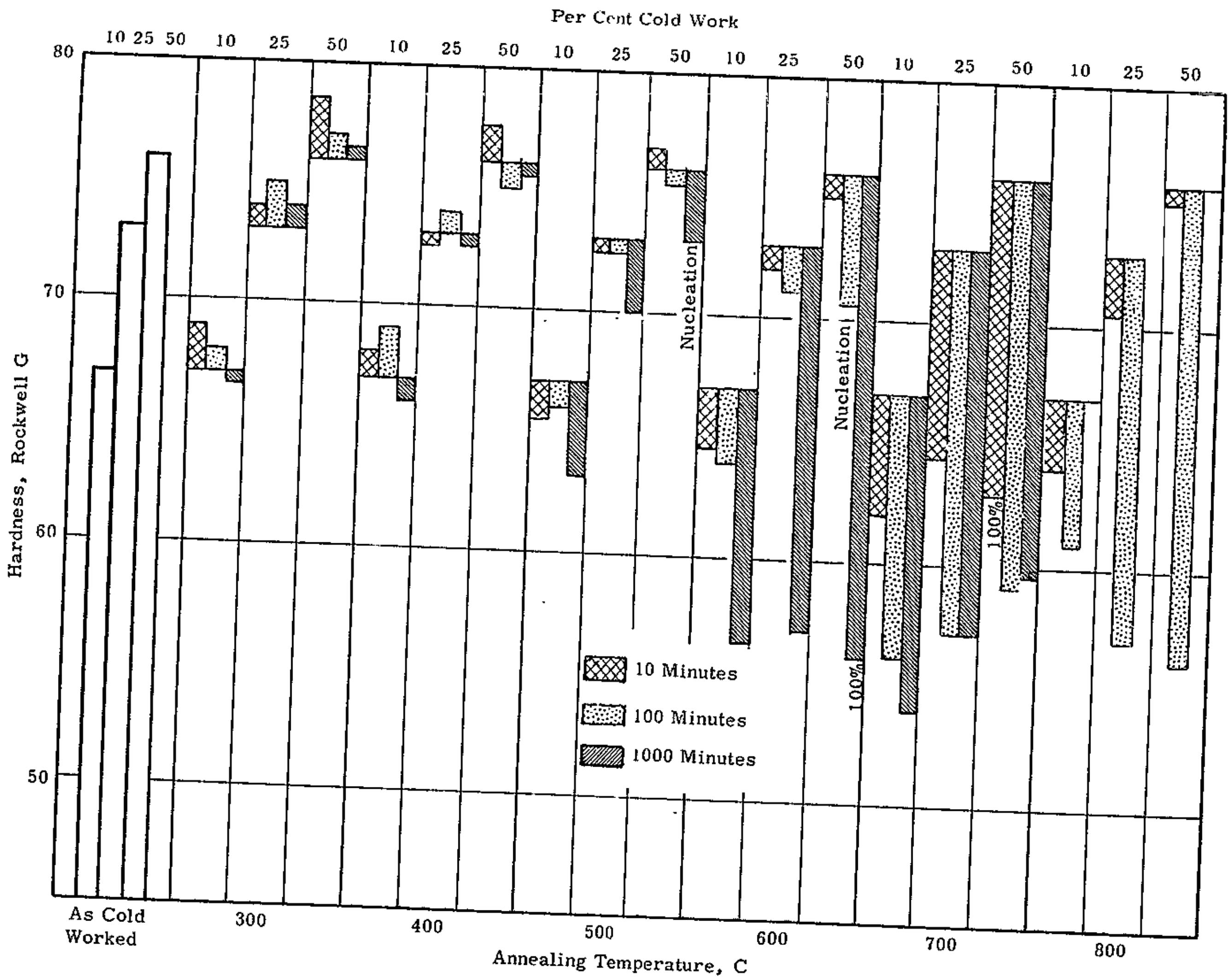


FIGURE 20

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3V; Vacuum Atmosphere

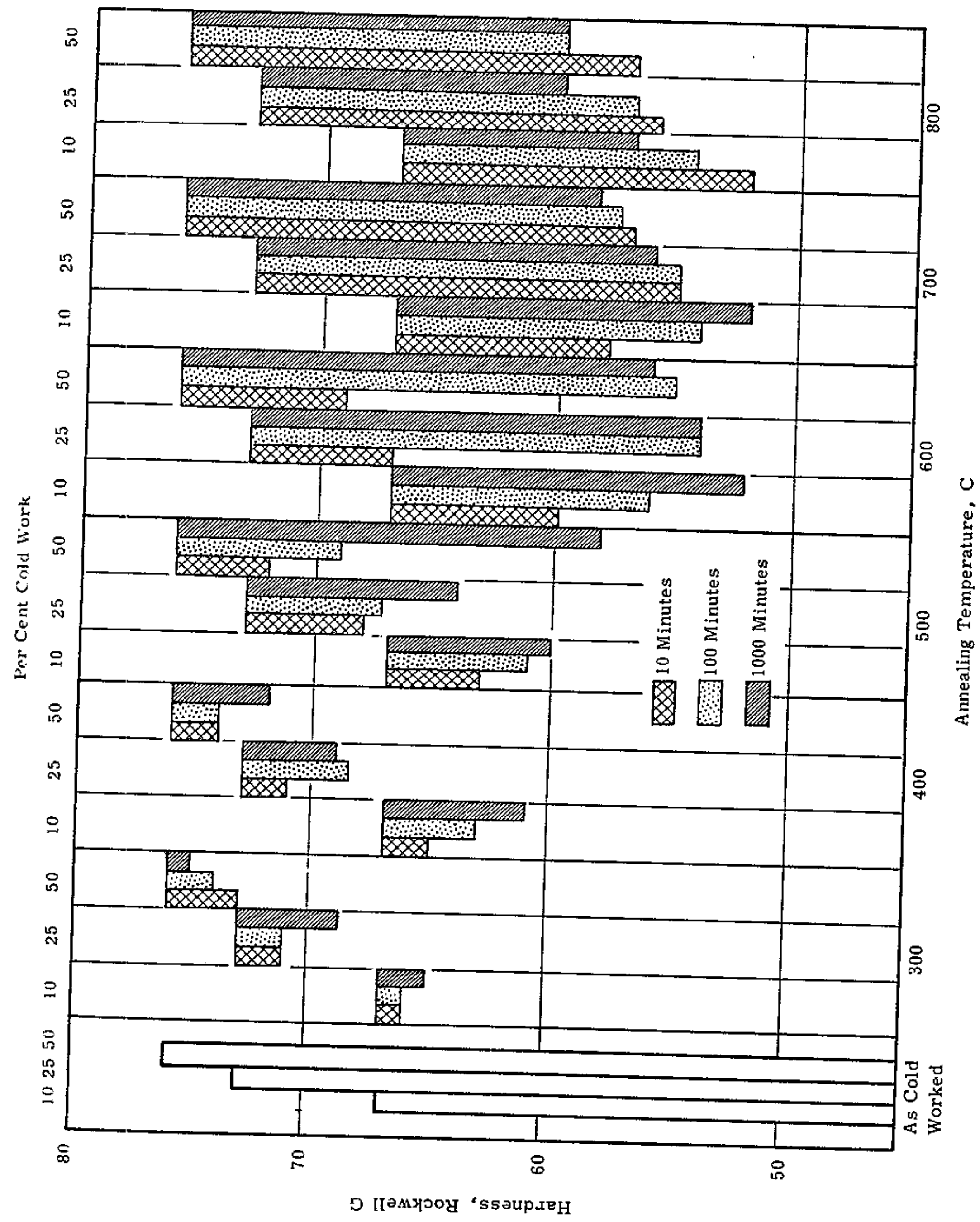


FIGURE 21

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3V; Helium Atmosphere

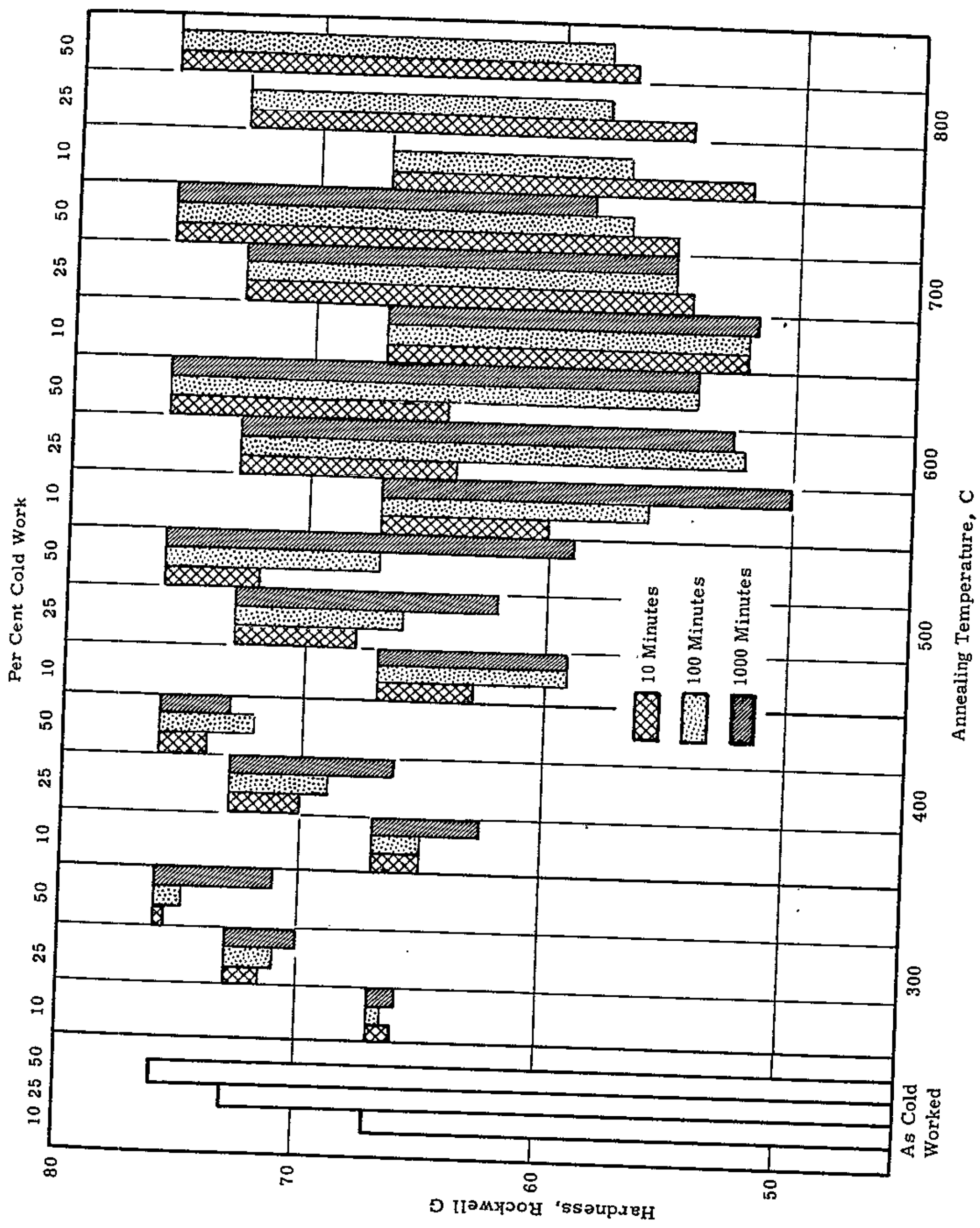


FIGURE 22

Effect of Annealing Temperature and Time on the Hardness of Cold-Worked Zircaloy-3V; Air Atmosphere

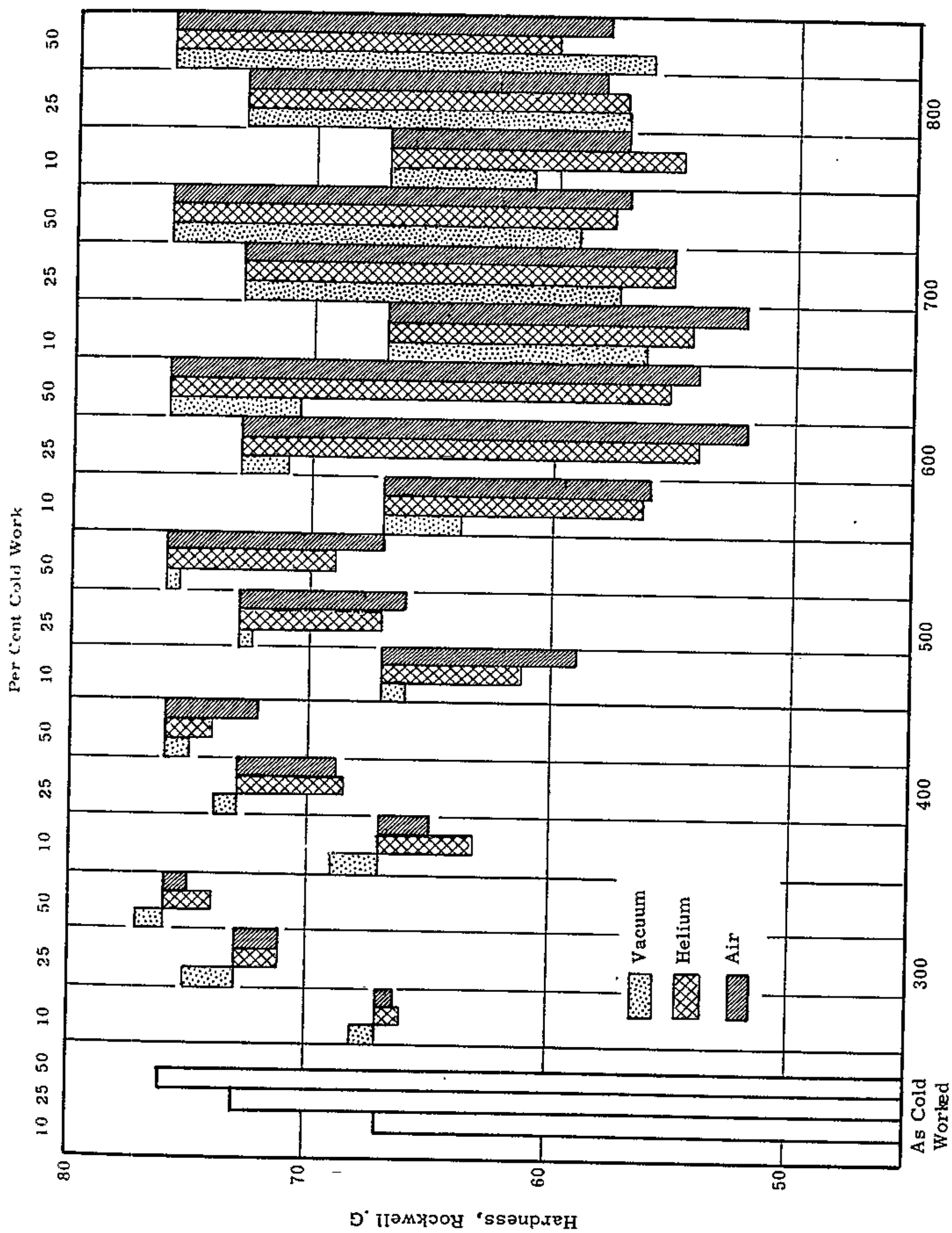


FIGURE 23

Effect of Annealing Atmosphere on the Hardness of Cold-Worked Zircaloy-3V; 100 Minutes

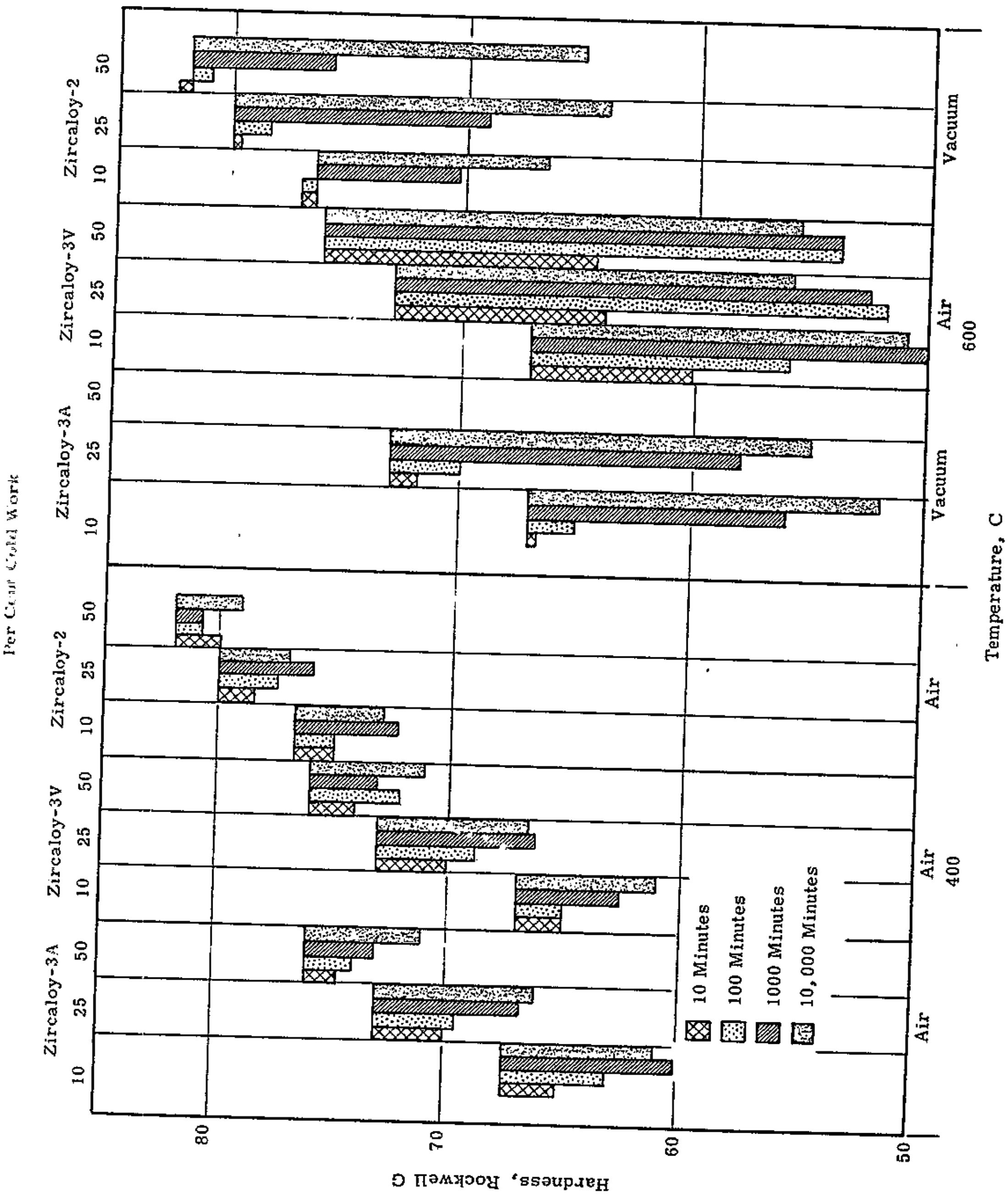


FIGURE 24

Effect of Long-Time Annealing on Hardness

AEC-GE RICHLAND, WASH.

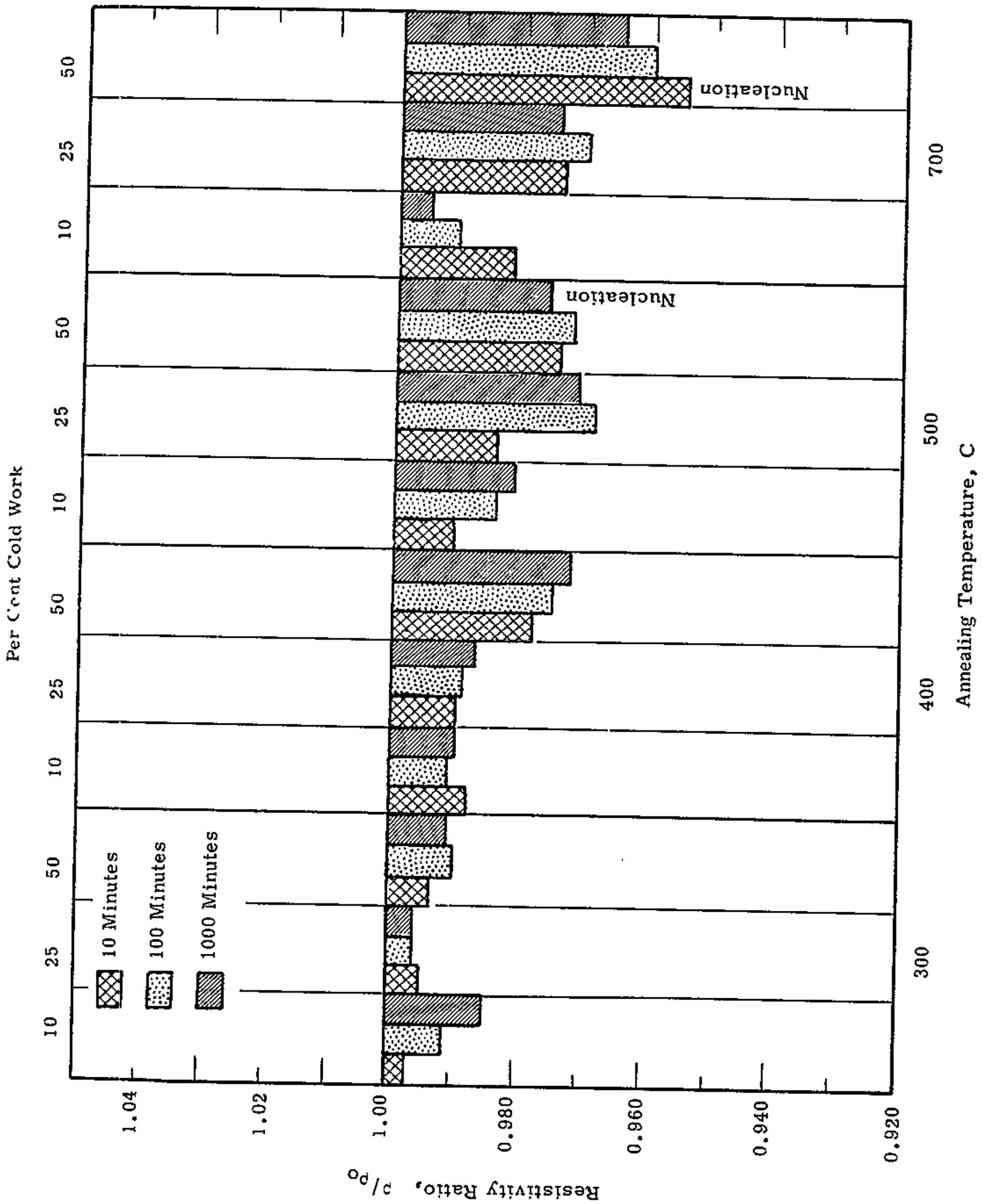


FIGURE 25

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-2; Vacuum Atmosphere

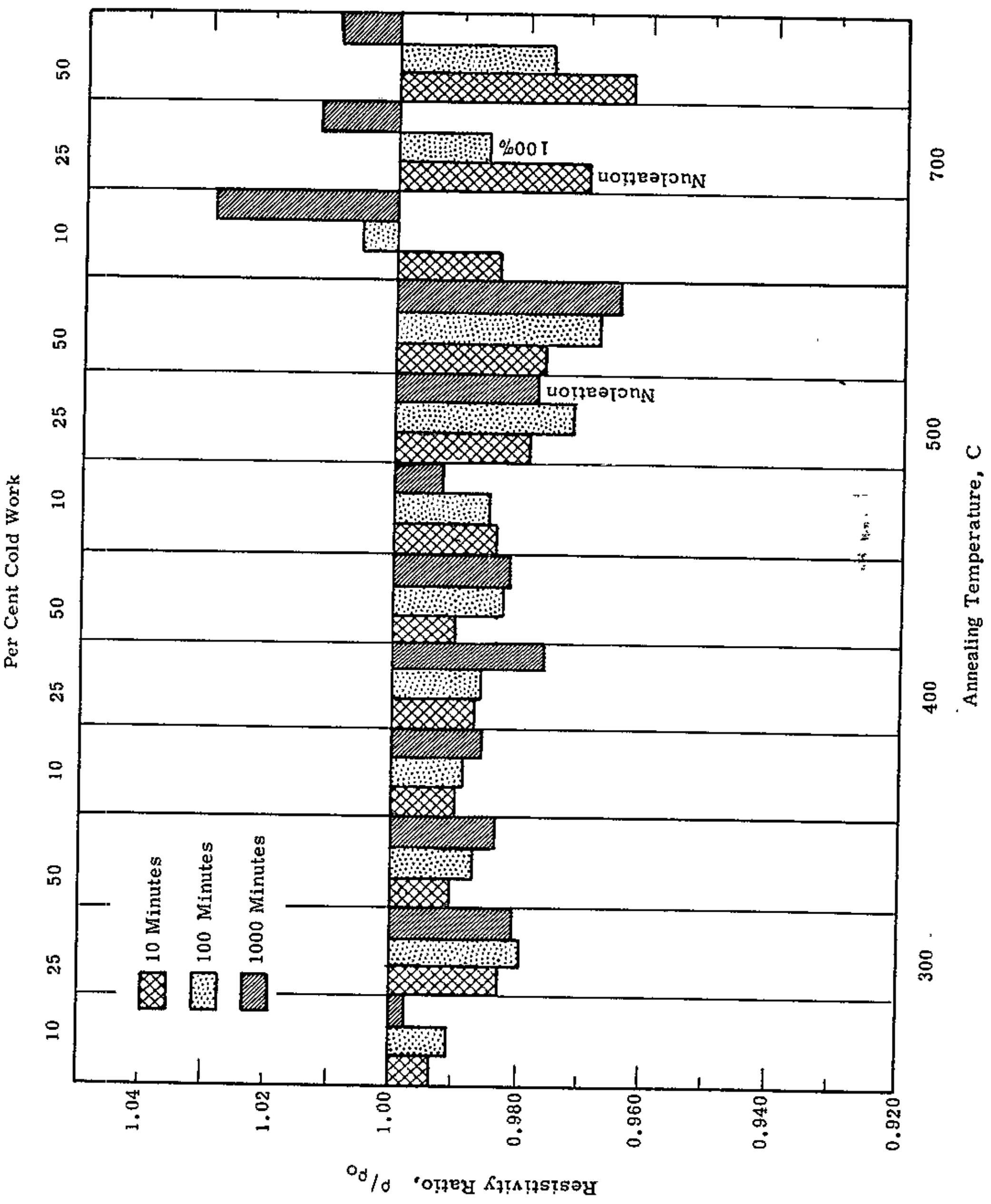


FIGURE 26

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-2; Helium Atmosphere

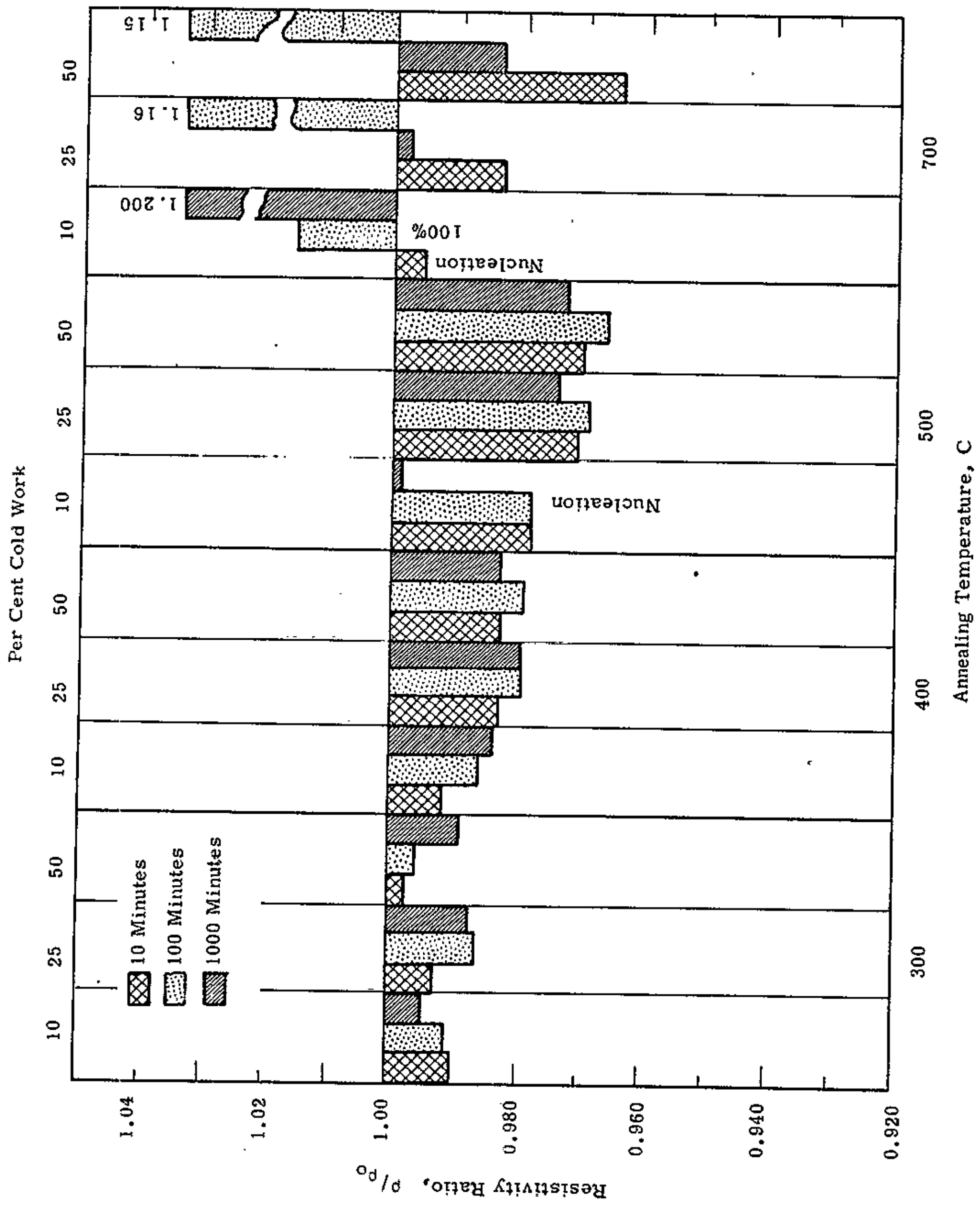


FIGURE 27

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-2; Air Atmosphere

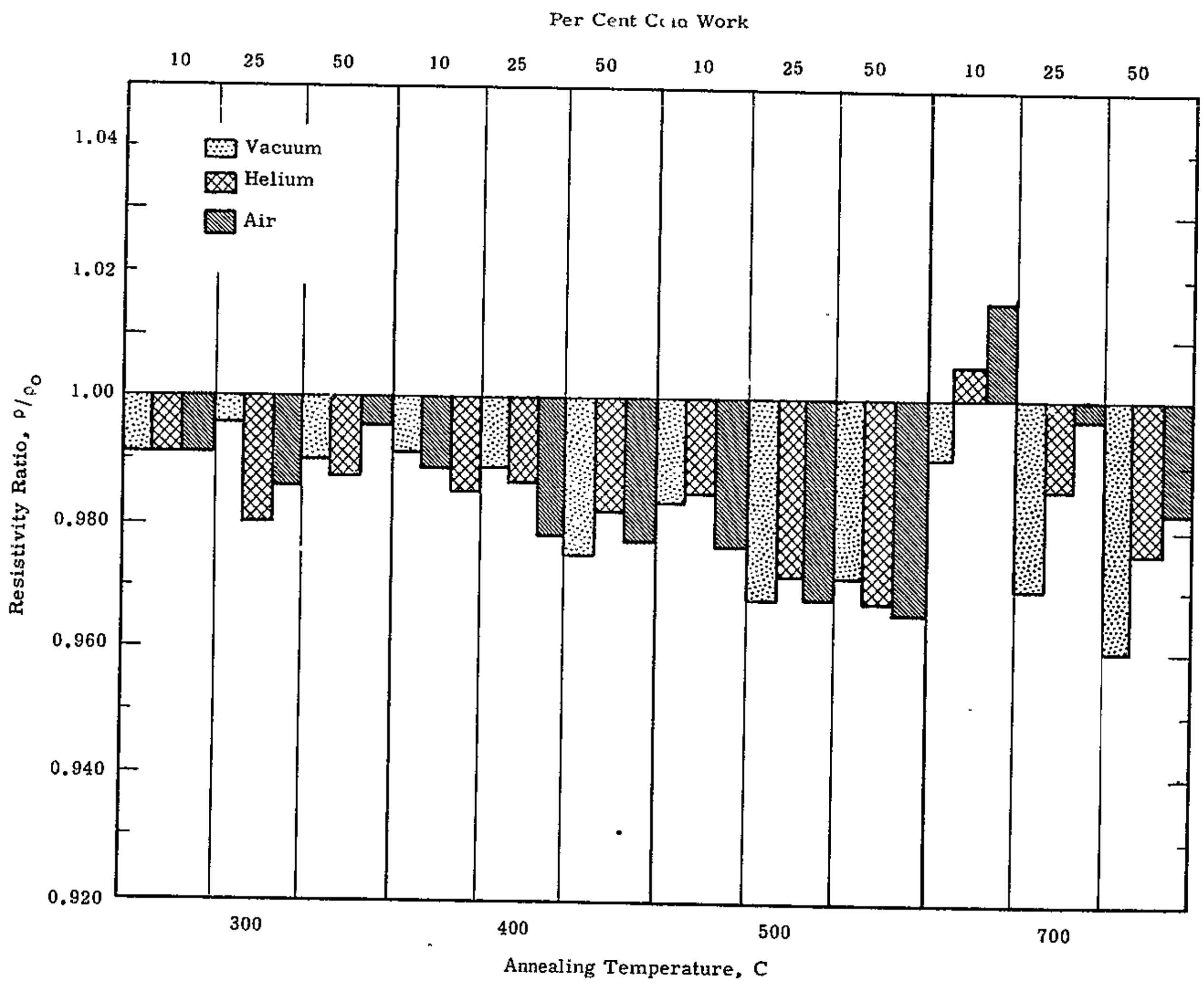


FIGURE 28

Effect of Annealing Atmosphere on the Electrical Resistivity of Cold-Worked Zircaloy-2; 100 Minutes

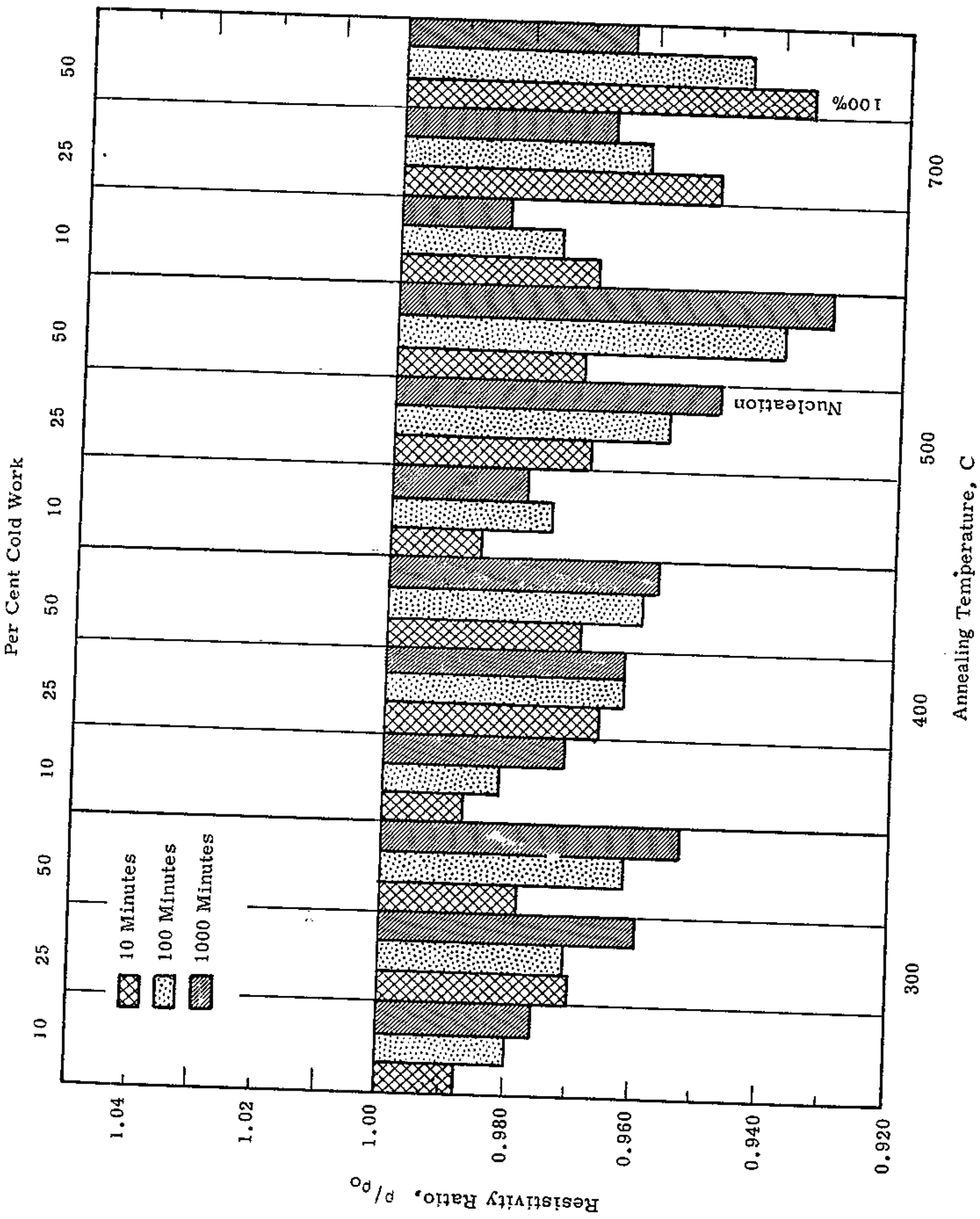


FIGURE 29

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3A; Vacuum Atmosphere

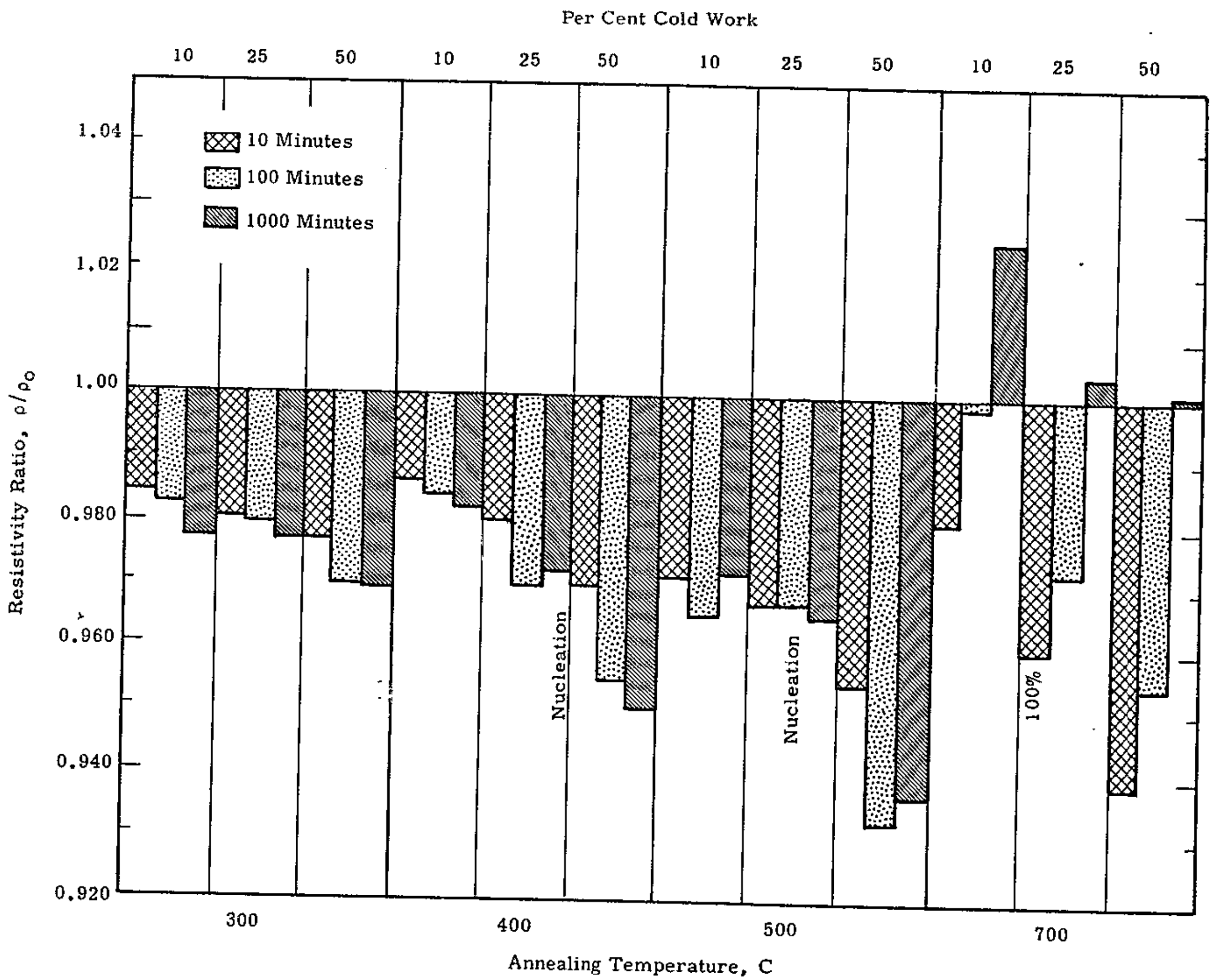


FIGURE 30

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3A; Helium Atmosphere

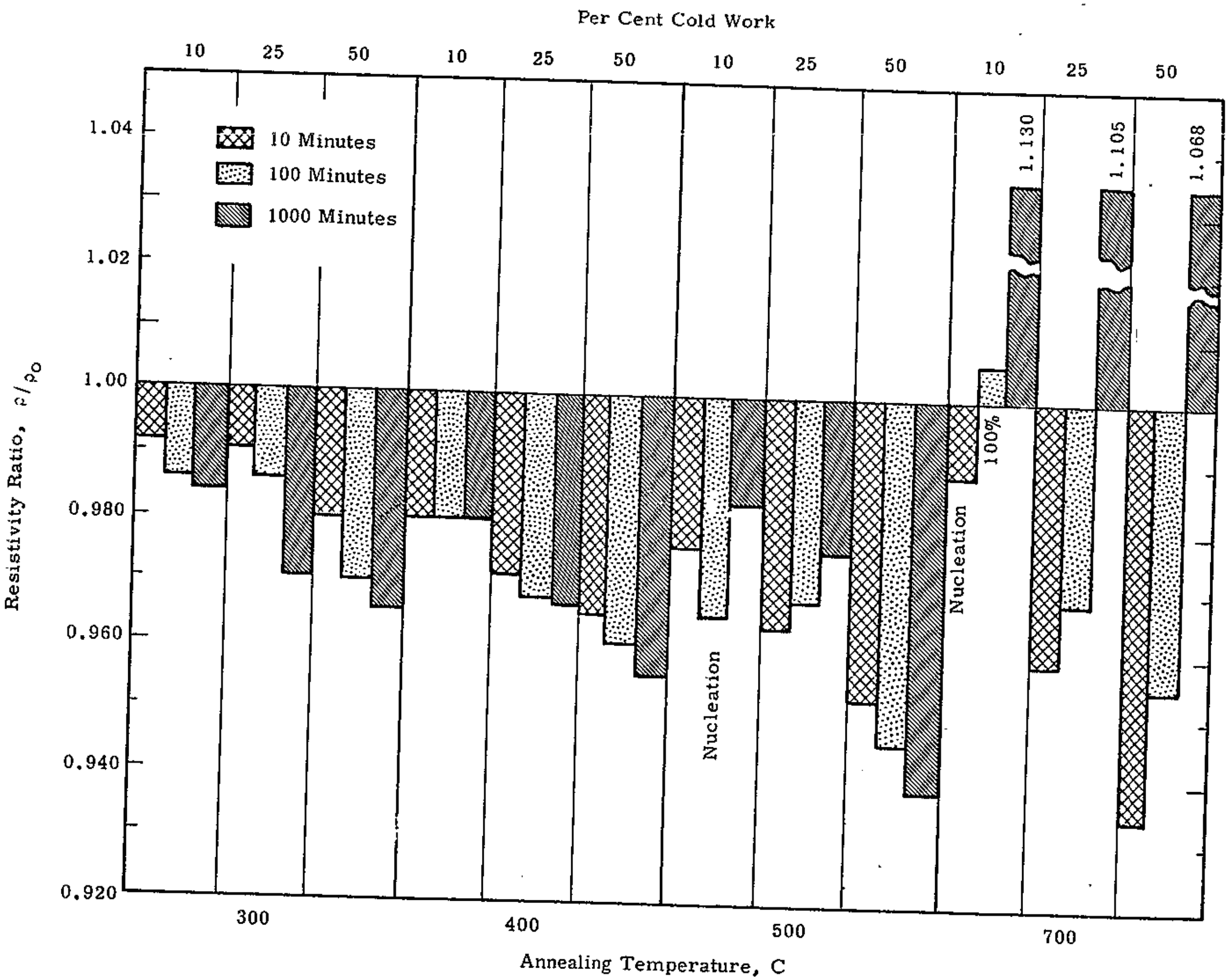


FIGURE 31

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3A; Air Atmosphere

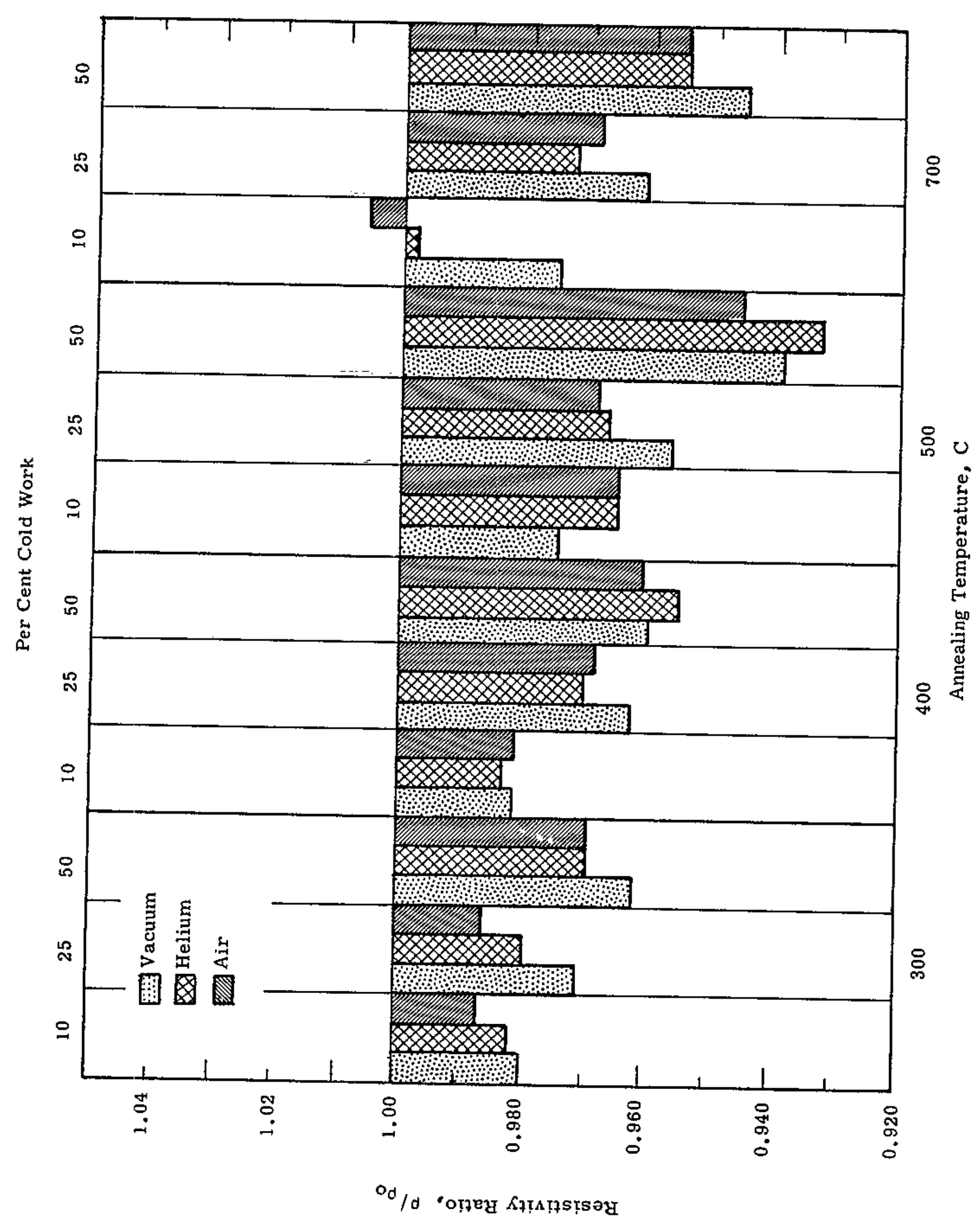


FIGURE 32

Effect of Annealing Atmosphere on the Electrical Resistivity of Cold-Worked Zircaloy-3A; 100 Minutes

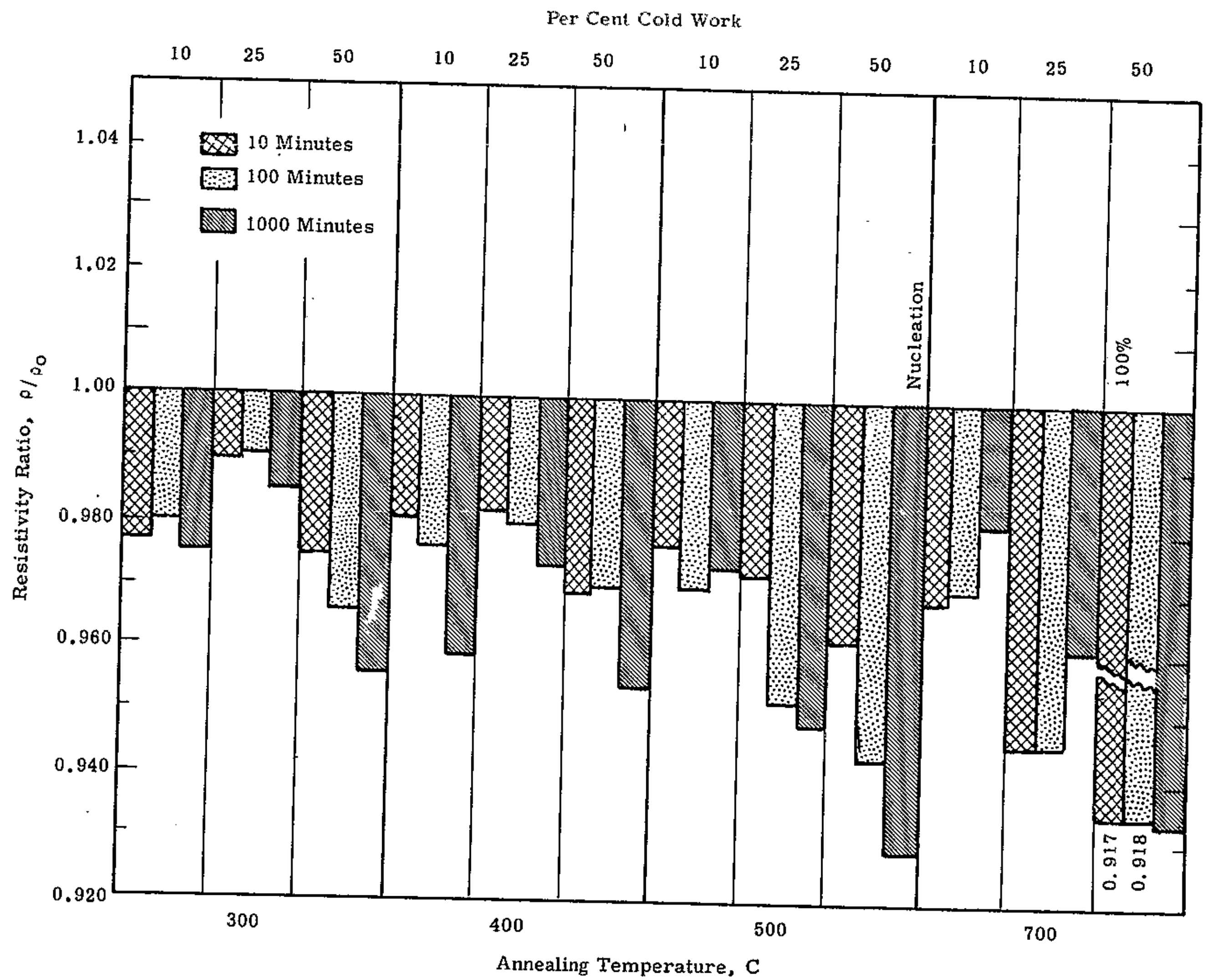


FIGURE 33

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3V; Vacuum Atmosphere

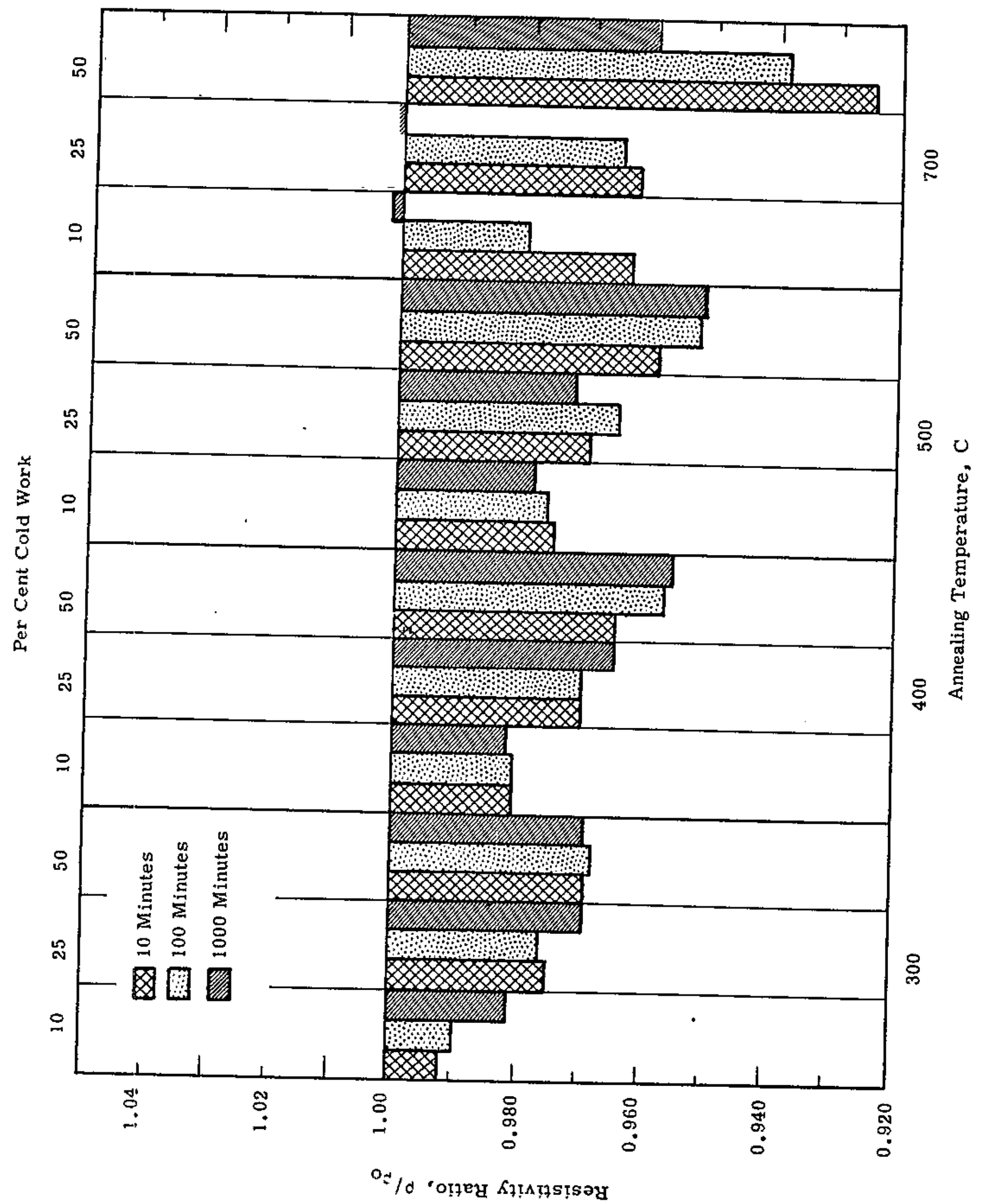


FIGURE 34

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3V; Helium Atmosphere

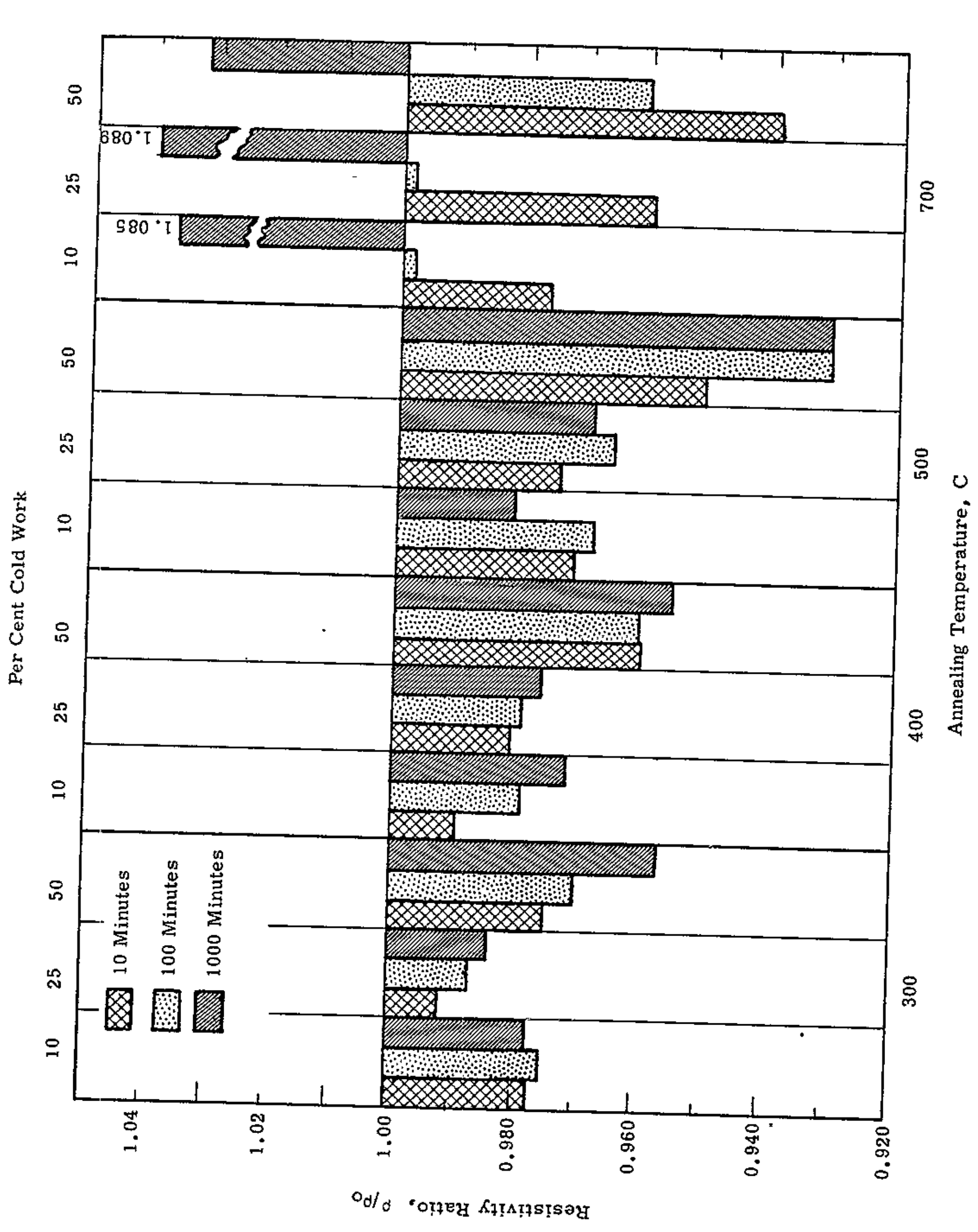


FIGURE 35

Effect of Annealing Temperature and Time on the Electrical Resistivity of Cold-Worked Zircaloy-3V; Air Atmosphere

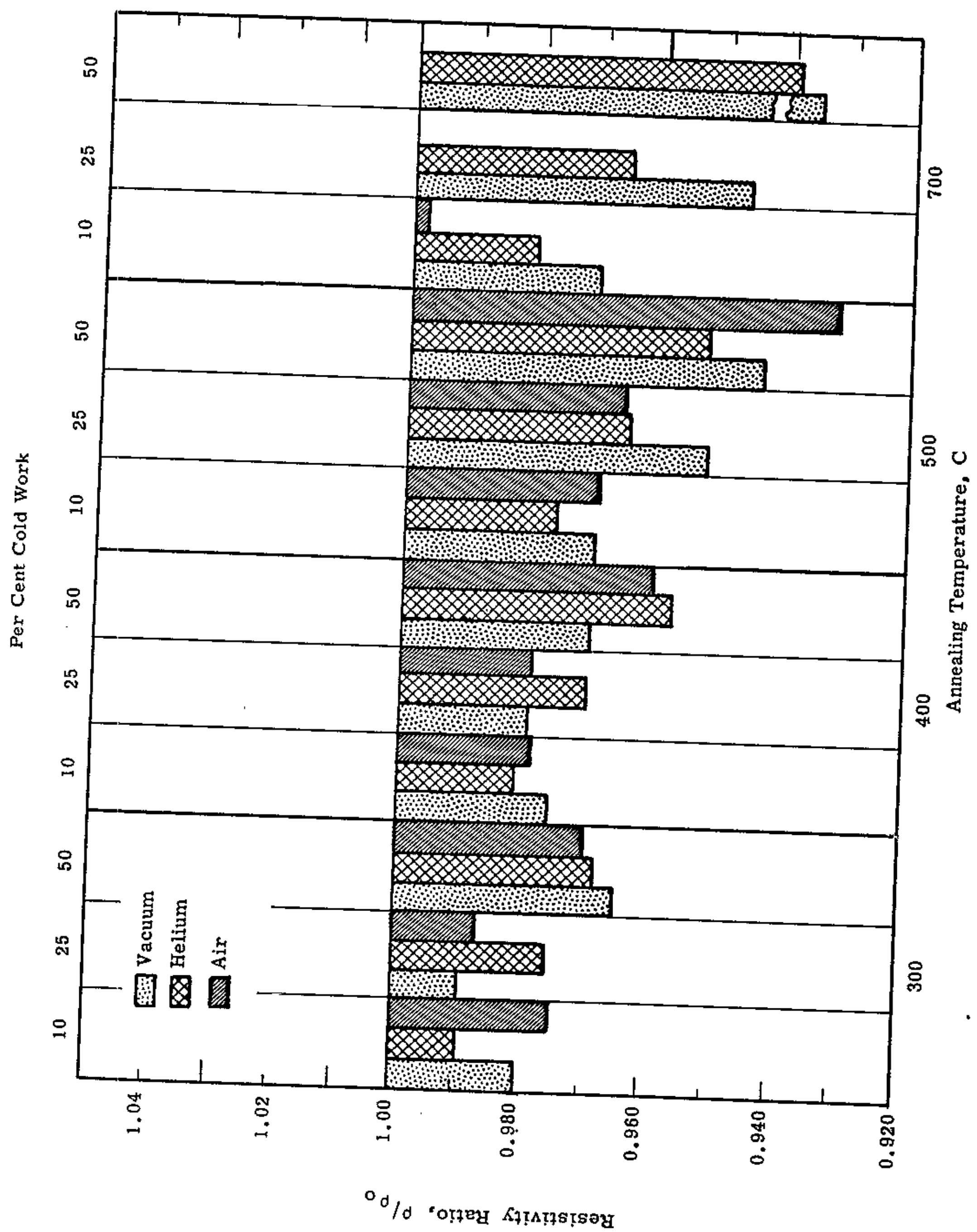


FIGURE 36

Effect of Annealing Atmosphere on the Electrical Resistivity of Cold-Worked Zircaloy-3V; 100 Minutes

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