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ORNL-2372
Metallurgy and Ceramics

THE ANNEALING BEHAVIOR OF COLD-ROLLED NIOBIUM

J. P. Page

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METALLURGY DIVISION

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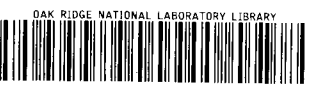
Submitted as a Thesis to the Graduate Council of the University of Tennessee in partial fulfillment of the requirements for the degree of Master of Science



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CHAPTER I

SUMMARY

Niobium has a combination of properties which makes it appear to be a very promising structural material for certain high-temperature applications. The annealing behavior of niobium is of interest for three reasons: 1) grain-size control, 2) knowledge of recovery characteristics, and 3) insight into high-temperature atomic mobility.

The annealing behavior of niobium has been outlined in a comprehensive manner. The initiation and termination of recrystallization in arc-cast and powder-metallurgy niobium have been determined by x-ray analysis as functions of annealing time, annealing temperature, and prior deformation. Annealing times of thirty-six seconds through one hundred hours, annealing temperatures of 900 through 1350°C, and prior deformations of 20 through 97.3 per cent reduction by rolling were investigated. The results indicate that niobium recrystallizes in agreement with classical theory in that recrystallization is promoted by increasing annealing temperature, annealing time, and prior deformation. No significant differences between arc-cast and powder-metallurgy niobium were noted.

The effects of one-hour anneals at room temperature (no anneal), 800, 900, 1000, 1100, and 1200°C on the room temperature tensile properties of powder-metallurgy and arc-cast niobium cold-rolled 20, 50, 80, and 90 per cent have been determined. The results indicate that the mechanical properties begin to recover at approximately 800°C.

These properties are essentially completely recovered at 1000°C in the 80 and 90 per cent rolled material, but not in the 20 and 50 per cent rolled materials, which are still in the relatively early stages of recrystallization. No significant differences in mechanical properties, or of the rates of change of these properties with annealing temperature were noted between powder-metallurgy and arc-cast material.

Although otherwise inconclusive, hardness data from isothermally-annealed specimens indicate that contamination has a marked effect on the hardness of niobium, and that microhardness measurements are, in general, an unsatisfactory method of following the course of recrystallization of niobium.

Photomicrographs of 80 per cent cold-rolled powder-metallurgy and arc-cast niobium after various annealing treatments verify, qualitatively, the results of the x-ray investigation. The recrystallized powder-metallurgy niobium appears to have a much finer grain size than the recrystallized arc-cast niobium after identical treatment.

CHAPTER II

INTRODUCTION

Niobium has an attractive combination of physical, chemical, mechanical, and nuclear properties exhibited by no other metal. Among these properties are: 1) high melting point (2468°C), 2) high strength at elevated temperature, 3) high room-temperature ductility, 4) weldability by inert-atmosphere technique, 5) resistance to corrosion by a large number of liquid media, and 6) low thermal-neutron absorption cross section (1.1 barns/atom). Nevertheless, extensive use of niobium has been discouraged by: 1) its extremely high cost (approximately $300/lb.), and 2) its susceptibility to embrittlement and oxidation by nitrogen and oxygen, respectively, at temperatures greater than 300°C. While these drawbacks have, as yet, not been overcome, their importance has been minimized by the demand for higher-temperature (and therefore higher-efficiency) power plants. The use of niobium in the Dounreay Fast Breeder Reactor,[1] and the many recent publications on niobium (see Bibliography) illustrate the growing importance of this metal.

The classic method of production of wrought niobium, as described by L. F. Yntema[2], is by the following sequence of steps: a) reaction of a stoichiometric mixture of niobium-carbide and niobium-pentoxide powders, b) comminution of the resultant metal to very fine powder, c) cold-compaction of this powder and pre-sintering of the compact in vacuum, d) high-temperature self-resistance vacuum sintering

of the pre-sintered bar, e) cold-working of the sintered mass, for densification, and f) re-sintering and cold-working to final wrought form.

Material produced by this process is generally fine-grained because the minute inclusions of un-reacted carbide and oxide tend to restrain the movement of grain boundaries. This method of production is also attractive in that, theoretically, the purity of the product is limited only by the purity of the starting material and the degree of vacuum attained in the sintering operations. Unfortunately, the powder-metallurgy technique suffers two real disadvantages: 1) it is an extremely costly, time-consuming process with a relatively low "yield", because the ends of the bar must be cropped after each self-resistance sintering operation, and 2) the size of the product is limited by the power requirements of the self-resistance sintering step in the fabrication sequence (the electric current required to heat an object to a given temperature is proportional to the cross-sectional area of the object).

The interest in reactive metals (titanium, vanadium, zirconium, niobium, tantalum, and molybdenum) has induced the development of non-contaminating (vacuum and inert-atmosphere) melting and casting techniques to the point where very large, pure ingots of these metals have been cast successfully. These techniques overcome the inherent disadvantages of the powder-metallurgy method. A casting, however, is not necessarily fine-grained. In fact, large castings of these reactive

metals have, almost invariably, a large, columnar grain structure. Whether or not this large grain size will be objectionable will depend on the service conditions imposed on the metal; for instance, large grain size has often been found to have a beneficial effect on creep properties, but may, on the other hand, have a disastrous effect on fabrication (such as extrusion, tube-reduction, and deep-drawing) properties. All mechanical properties of a metal are partially a function of its grain size; in general, a fine-grained structure is preferred to a coarse-grained structure. The refinement and control of a metal's grain size is generally accomplished through polymorphic transformation or recrystallization.

Niobium exhibits no polymorphic transformation; therefore, the only means of grain-size control of cast metal is by recrystallization. Recrystallization can generally be accomplished by either hot- or cold-working, but the susceptibility of niobium to oxidation and embrittlement at elevated temperature tends to discourage the use of hot-working as a method of grain-size control. Cold-working, followed by a recrystallization-anneal in vacuum, appears, therefore, to be the most promising method of grain-size control.

The mechanical properties of a metal will be altered significantly by cold-working; hardness and tensile strength will generally increase, while ductility will generally decrease. The return of these properties to levels exhibited by the non-deformed metal is accomplished by annealing. Annealing is ordinarily associated with complete recrystallization, but in this report it will be defined as any high-

temperature heat-treatment. Recrystallization will be defined as the growth of new, strain-free grains at the expense of the cold-worked matrix. Some metals exhibit appreciable "softening" before there is any evidence of recrystallization. This effect will be defined as "recovery" in this report.

Comprehensive reviews on the subject of recrystallization have been presented by R. F. Mehl[3], C. S. Barrett[4], and more recently by P. A. Beck[5]. At the risk of over-simplification, it may be said that the recrystallization behavior of a metal is primarily dictated by: 1) its composition, 2) the degree of cold work to which it is subjected, 3) the annealing time, 4) the annealing temperature, and 5) the metal's prior grain size. Increasing the annealing temperature and/or the annealing time will increase the degree of recrystallization of cold-worked metal. Recrystallization is promoted by the energy which is retained by the distorted crystalline lattice;[5] in other words, increasing the degree of cold work tends to increase the degree of recrystallization of an annealed metal, other factors being held constant.

The annealing behavior of niobium, in particular, is important for several reasons:

1. Grain-size control. It has been mentioned that a metal's grain size will affect its mechanical properties; therefore, a comprehensive mechanical-properties testing program on arc-cast niobium will require close control of grain size if the resultant data are to be meaningful. Metallurgists and metallurgical engineers will be faced

with the problem of choosing annealing times and temperatures and "prior deformations" which will yield a desirable microstructure in the recrystallized product. In order to minimize annealing time, a relatively high temperature must be used, but, unfortunately, the effectiveness of a vacuum system falls off rapidly with increasing temperature, thereby leading to the danger of contamination of the niobium. Also, economic factors such as furnace tie-up time and power cost favor a minimization of both annealing time and temperature.

2. Recovery characteristics. It has been reported[6] that certain refractory metals exhibit considerable recovery prior to recrystallization. If this is true for niobium, the recovery characteristics may have as much significance, for certain applications, as the recrystallization characteristics. For instance, a fabricator of deep-drawn niobium cups may find it necessary to resort to an intermediate anneal at some stage of the fabrication process; it is the recovery of ductility, not true recrystallization, which will interest him.

3. Insight into high-temperature behavior. The potential of niobium as a high-temperature structural material will be realized only after a great deal more is known about its high-temperature behavior than that known at present. The recovery and recrystallization behavior of a metal at any temperature is, in a sense, a measure of the atomic mobility of the metal at that temperature. The creep strength of a metal at the same temperature is partially a function of this atomic mobility. In fact, creep properties of certain metals have been related

to recovery[7] and recrystallization[8]. A creep-testing program is both costly and time-consuming; this is especially true for the reactive metals, for elaborate precautions must be taken to insure non-contamination of the specimens. Recrystallization behavior, on the other hand, is relatively simple to determine. If an attempt is made to raise the creep strength of niobium through alloy-development, it may be possible that a considerable saving of time and money could be realized by "screening" niobium-base alloys by examining their annealing behavior. The success of such a screening program would depend on accurate, base-line knowledge of the annealing behavior of the pure metal.

CHAPTER III

OBJECTIVES

The general objective of this investigation was to outline, in a comprehensive manner, the annealing behavior of niobium.

The specific objectives were:

1. To determine the initiation and completion of the recrystallization process in powder-metallurgy and arc-cast niobium as a function of: a) prior deformation, b) annealing temperature, and c) annealing time. The composition and prior grain structure of the material under investigation was to be known and held constant, as much as possible.

2. To determine whether or not recrystallization in arc-cast and powder-metallurgy niobium is preceded by significant recovery of mechanical properties.

3. To determine significant differences, if any, in the annealing behavior of arc-cast and powder-metallurgy niobium.

CHAPTER IV

PRIOR WORK

The only recrystallization study, *per se*, reported in the literature is that of R. T. Begley and co-workers at the Westinghouse Research Laboratories.[9] Commercially-pure powder-metallurgy niobium which had been cold-rolled to 20, 50, and 85 per cent reduction in thickness was annealed for one hour at: room temperature (no anneal), 1000, 1100, 1200, 1300, 1400, and 1500°C. The annealed specimens were analyzed by the Laue back-reflection technique and by hardness measurements. The results indicated that the 20 per cent reduced material began to recover at 1000°C, began to recrystallize at 1300°C, and was completely recrystallized at 1500°C. The 50 per cent reduced niobium had not begun to recover at 1000°C, but was completely recrystallized at 1100°C. The 85 per cent reduced material began to recover at 1100°C and was completely recrystallized at 1200°C.

E. S. Tankins and R. Maddin,[10] in an investigation of the strain-rate sensitivity of niobium, annealed swaged niobium wire at times of from one-half hour to one hour at temperatures of 1000, 1200, and 2000°C, and thereby achieved very close grain-size control. Their work, however, is not directly applicable to metallurgical practice, for they preceded their recrystallization treatment with a 2000°C purification-anneal.

H. Inouye[11] annealed small tensile specimens of 88½ per cent cold-rolled niobium for one-half hour at: room temperature (no anneal),

300, 600, 900, 1000, 1100, and 1200°C. Essentially complete recovery of mechanical properties was observed in specimens annealed at 1100 and 1200°C.

CHAPTER V

EXPERIMENTAL PROCEDURE

At the request of the Metallurgy Division of the Oak Ridge National Laboratory, four ingots of pure niobium were cast and cold-forged by members of the Battelle Memorial Institute, at that facility. These ingots were cast by the consumable-electrode process in a titanium-gettered helium atmosphere. The electrodes consisted of high-purity sintered bars purchased from the Fansteel Metallurgical Corporation. The castings were made in the form of one and one-half inch diameter ingots and were subsequently cold-forged to one-half inch thick plates. Figure 1 shows these plates as received at ORNL.

Ultrasonic and radiographic inspection of these plates was performed by the ORNL Nondestructive Testing Group. Except for a crack in plate No. 10, the plates were sound, with only occasional defects detected.

A section of one of the plates was polished and etched for macroexamination using metallographic procedures and etchants described in the Appendix. Figures 2, 3, and 4 are photomacrographs of the three major planes of the plate. These figures show the large-grained structure typical of cast niobium. The distortion of the grains is a result of the cold-forging operation.

The powder-metallurgy niobium was purchased from the Fansteel Metallurgical Corporation in the form of three-sixteenths-inch by one

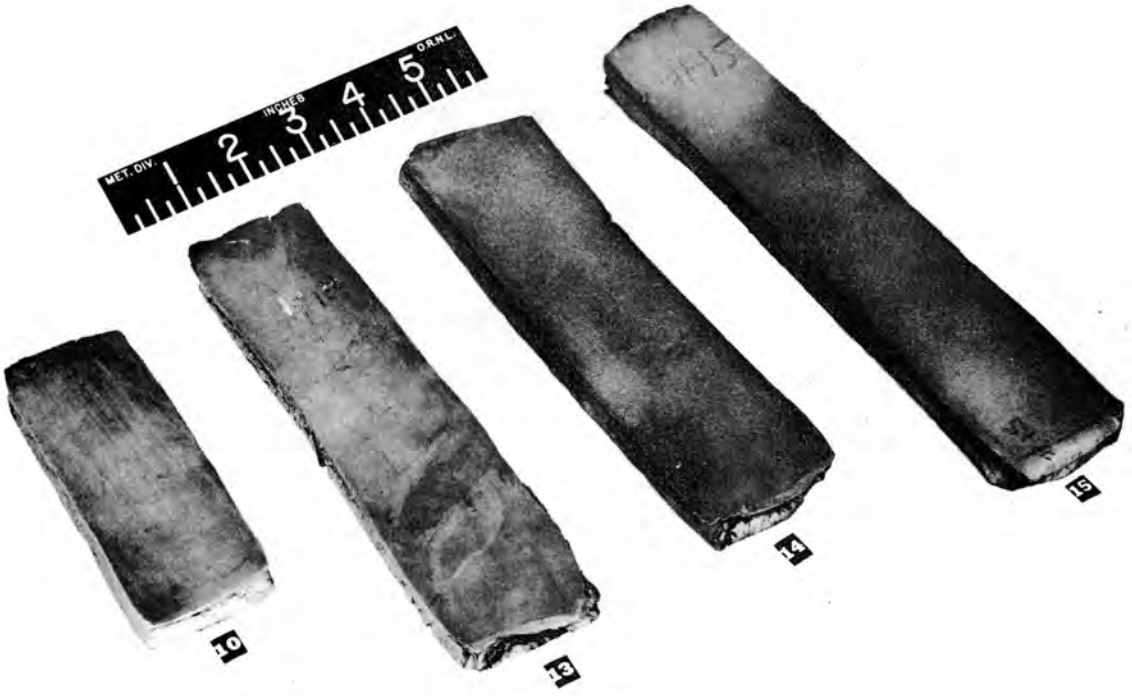

Figure 1 (Y-18332). As-received arc-cast and cold-forged niobium plates.

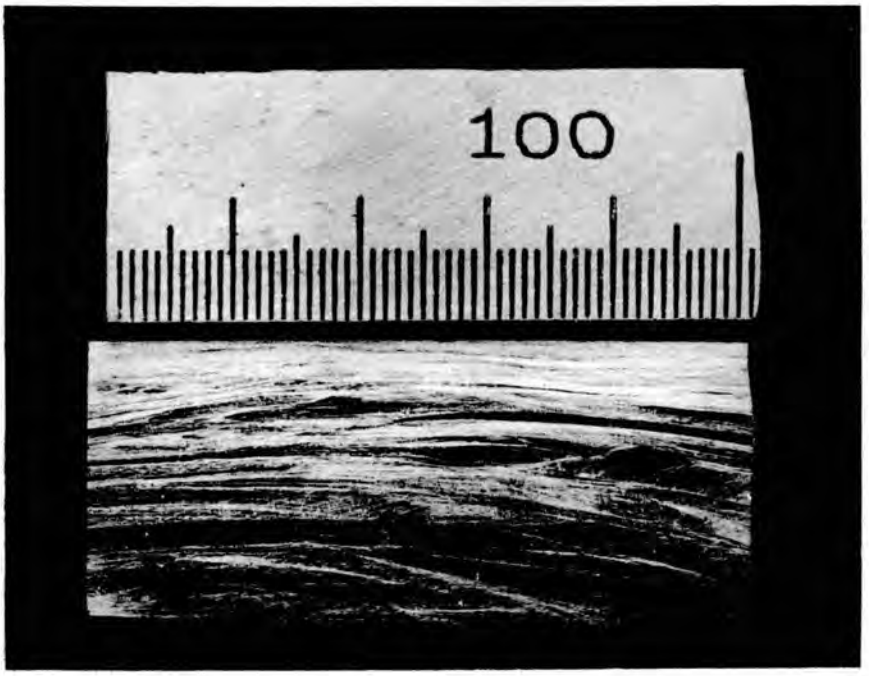

Figure 2 (Y-19119). Photomacrograph of as-received arc-cast plate; view of plane parallel to "edge" of plate. Etchant - 35 HF, 50 H_2O, 10 HNO_3, 10 H_2SO_4. 3.25X.

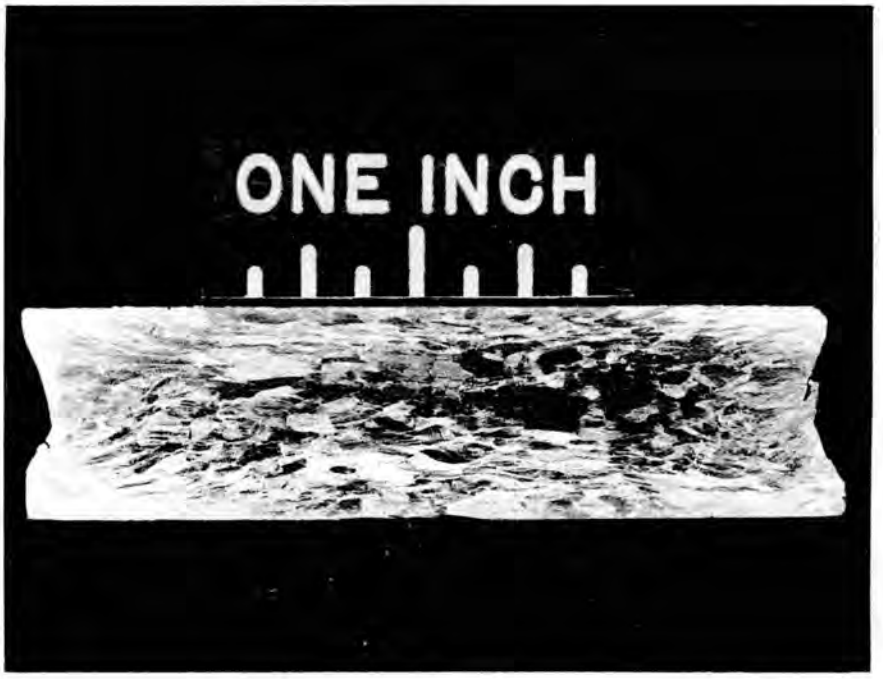

Figure 3 (Y-19118). Photomacrograph of as-received arc-cast plate; view of plane parallel to "end" of plate. Etchant - 35 HF, 50 H_2O, 10 HNO_3, 10 H_2SO_4. 2.25X.

Figure 4 (Y-19117). Photomacrograph of as-received arc-cast plate; view of plane parallel to forging plane. Etchant - 35 HF, 50 H_2O, 10 HNO_3, 10 H_2SO_4. 2.5X.

and one-half inch by two and one-fourth inch plates. A typical microstructure of the as-received plates is shown in Figure 5.

Drillings from each of the arc-cast plates and from three powder-metallurgy plates were submitted to the ORNL Analytical Chemistry Division for carbon, oxygen, nitrogen, and hydrogen analyses.* The results of these analyses are presented in Table I. Neither the precision nor the accuracy of the analytical techniques has been established. In terms of these analyses, however, there appears to be no "significant" difference in composition of the two materials.

Other than the grain structure, the most marked difference between arc-cast and powder-metallurgy niobium is in the relative amounts of non-metallic inclusions in the two materials. An as-polished microstructure of the arc-cast material is shown in Figure 6. This figure should be compared to Figure 7, an as-polished microstructure of powder-metallurgy niobium, at the same magnification. The large number of inclusions in the latter photomicrograph is quite evident. A later section of this report will show, metallographically, the effects of these inclusions, presumably oxides and/or carbides, on the recrystallization behavior of powder-metallurgy niobium.

Preliminary attempts to recrystallize the arc-cast and cold-forged material were unsuccessful. The upper temperature limit of the vacuum-annealing furnace, which is described in the Appendix, is

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*Metallic impurities were assumed similar, as both the powder-metallurgy and arc-cast plates were produced from essentially the same raw material.

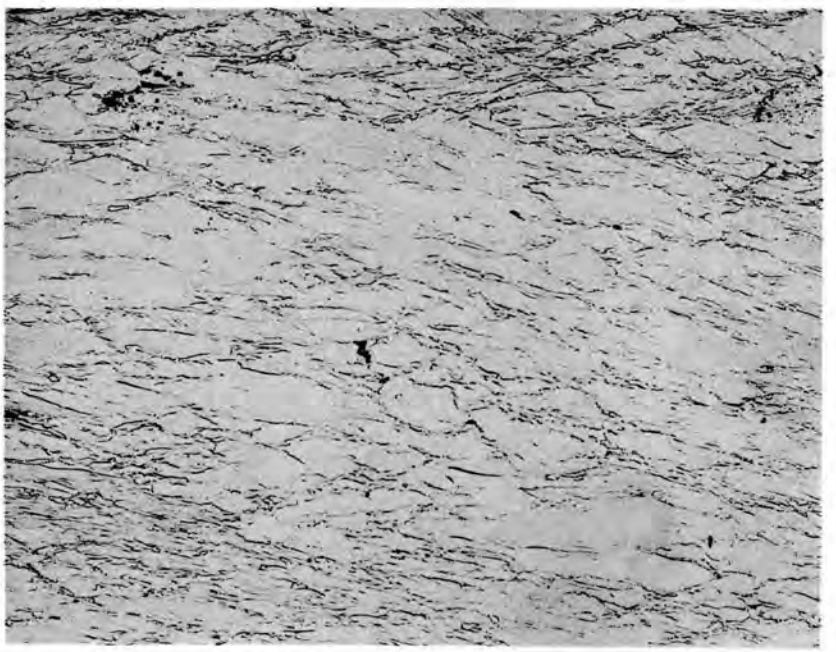

Figure 5 (Y-23152). Photomicrograph, typical microstructure, as-received powder-metallurgy niobium. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

TABLE I

CHEMICAL ANALYSES OF ARC-CAST AND POWDER-METALLURGY NIOBIUM

	C wt %	O_2 ppm	N_2 ppm	H_2 ppm
Arc-Cast Niobium				
Plate No. 10				
Near Top	0.04	900	360	13
Near Center	0.02	520	360	4
Near Center	0.01	640	350	5
Near Bottom	0.06	500	360	11
Plate No. 13				
Near Top	0.01	390	250	< 1
Near Top	0.01	490	280	< 1
Near Center	0.01	540	270	2
Near Bottom	0.01	480	280	4
Plate No. 14				
Near Top	0.01	470	260	4
Near Center	0.01	360	250	1
Near Bottom	0.05	610	290	< 1
Near Bottom	0.05	360	320	1
Plate No. 15				
Near Top	0.03	520	250	6
Near Center	0.01	380	270	4
Near Bottom	< 0.01	520	270	14
Powder-Metallurgy Niobium				
Plate "A"	< 0.01	220	380	44
Plate "B"	0.01	1900	120	15
Plate "C"	0.03	760	450	6

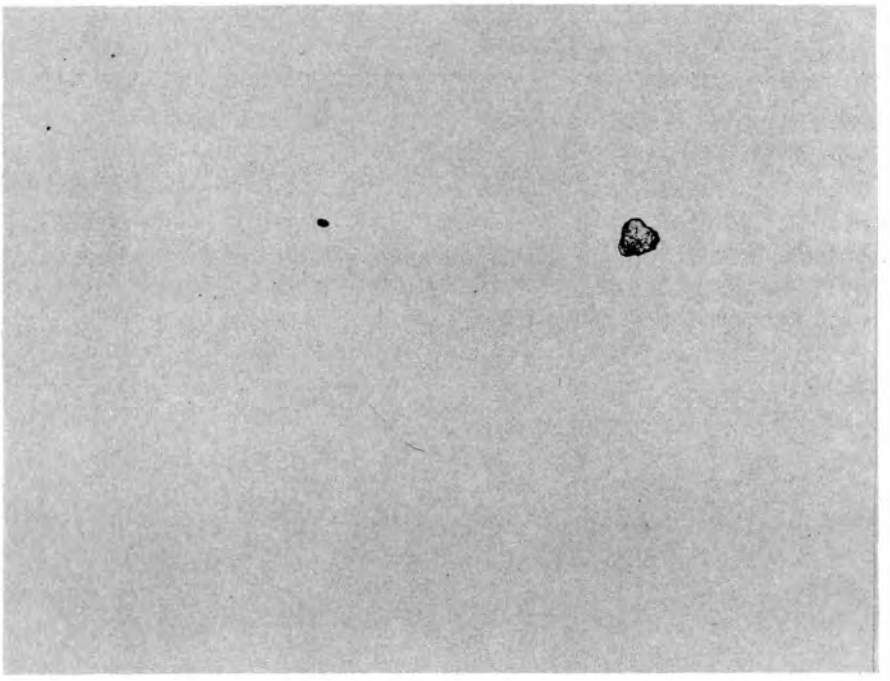

Figure 6 (Y-21327). Photomicrograph, as-polished section, arc-cast niobium. 100X.

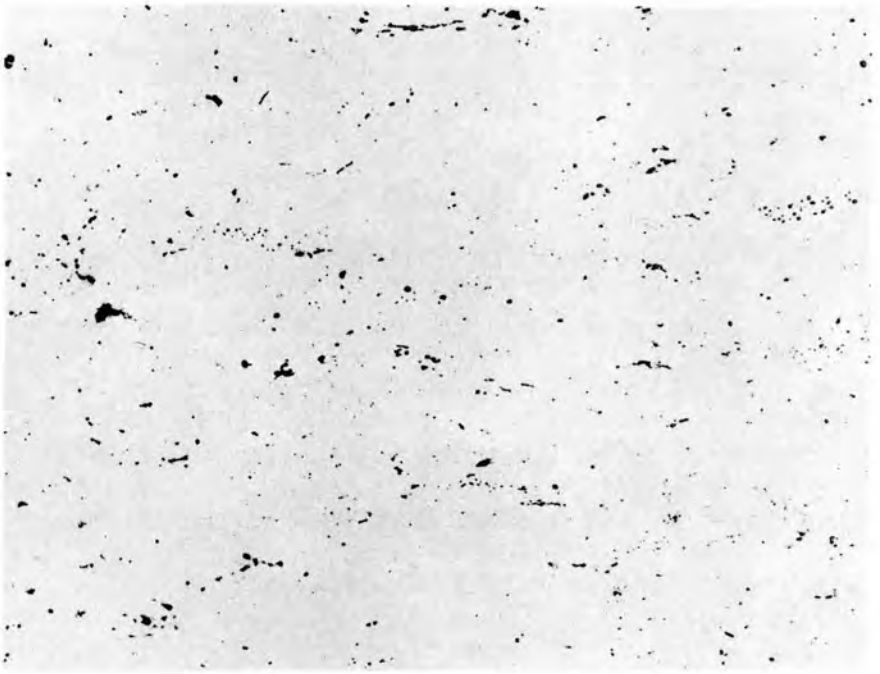

Figure 7 (Y-21320). Photomicrograph, as-polished section, powder-metallurgy niobium. 100X.

approximately 1350°C. Even long times (24 hours) at this temperature did not produce significant recrystallization, although the material softened appreciably (hardness drop of from 150 DPN to 120 DPN). To induce recrystallization and obtain a tentative "picture" of the recrystallization behavior, sections of the arc-cast plates were cold-rolled to various deformations and annealed at various temperatures. It was found that a 24-hour anneal at 1250°C on 85 to 90 per cent reduced material would produce complete recrystallization and yield a satisfactory, but not ideal, microstructure. A significant observation during these "preliminary studies" was that recrystallization of the large-grained material occurred in localized regions, generally in "bands", or planes parallel to the rolling plane. Since this condition caused estimates of "per cent recrystallization" to vary in nominally-identical samples, it was decided that the recrystallization study proper of arc-cast niobium should be performed on material which had undergone a pre-recrystallization treatment. Furthermore, this material was to have a structure as similar as possible to that of the recrystallized powder-metallurgy niobium.

Whereas the problem in the arc-cast material had been one of grain-refinement, the problem of the "matching" of this structure with the powder-metallurgy material was associated with grain-coarsening. In order to conserve both material and time, the preliminary studies on the arc-cast material had been terminated with the pre-recrystallization treatment described in the preceding paragraph. A series of treatments were then undertaken to coarsen the grain structure of the

powder-metallurgy niobium. The treatment that yielded the most satisfactory results was again a 24-hour anneal at 1250°C, but after only 50 per cent reduction in thickness.

Figures 8 and 9 show microstructures of the rolled and recrystallized arc-cast material. Figure 8 is a view of a plane normal to the rolling plane and parallel to the direction of rolling; views of this plane will henceforth be termed "parallel". Figure 9 is also a view of a plane normal to the rolling plane, but this view shows the transverse direction; similar views will henceforth be termed "transverse". These microstructures are considered typical of arc-cast and cold-forged material which had been rolled 85 to 90 per cent and annealed for 24 hours at 1250°C. Typical microstructures of pre-recrystallized (50 per cent reduction in thickness, followed by a 24-hour anneal at 1250°C) powder-metallurgy niobium are presented in Figures 10 and 11, which show parallel and transverse views, respectively. It is evident that in both materials the recrystallized grains are "long" and "flat" in the parallel and normal directions, respectively. Comparison of Figure 8 to Figure 10 and Figure 9 to Figure 11 indicates that the grain structures are quite well matched. Although a few large grains are evident, the "mean grain diameter" was estimated to be approximately 0.035 mm. by comparison to a standard chart.

Material of this type was fabricated into specimens for the x-ray analysis of the recrystallization behavior of niobium; the fabrication schedules are presented in Figures 12 and 13. In order to affect an 85 per cent reduction in thickness of the as-received

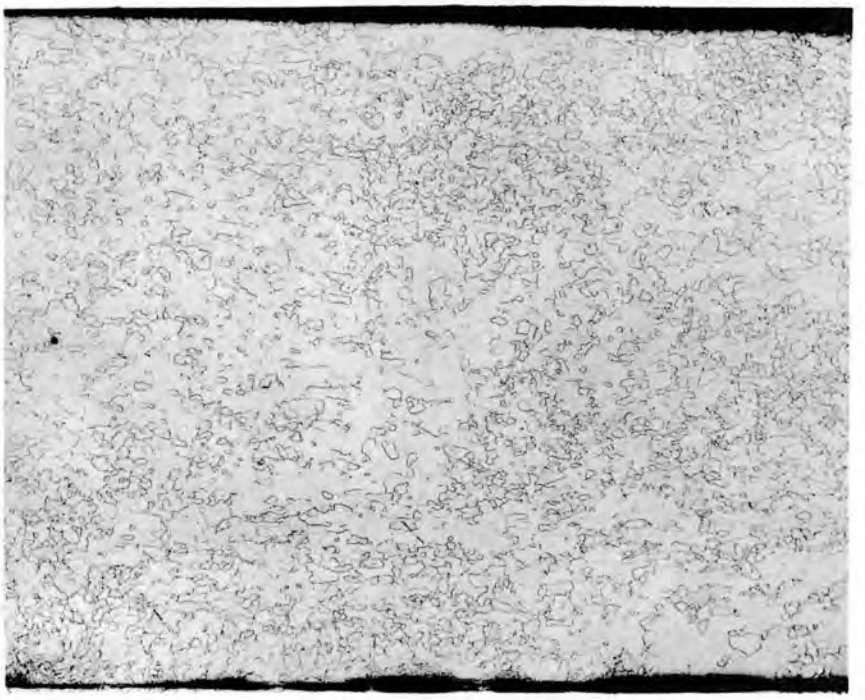

Figure 8 (Y-22632). Typical microstructure of "pre-recrystallized" arc-cast niobium, parallel view. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 75X.

Figure 9 (Y-22630). Typical microstructure of "pre-recrystallized" arc-cast niobium, transverse view. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 75X.

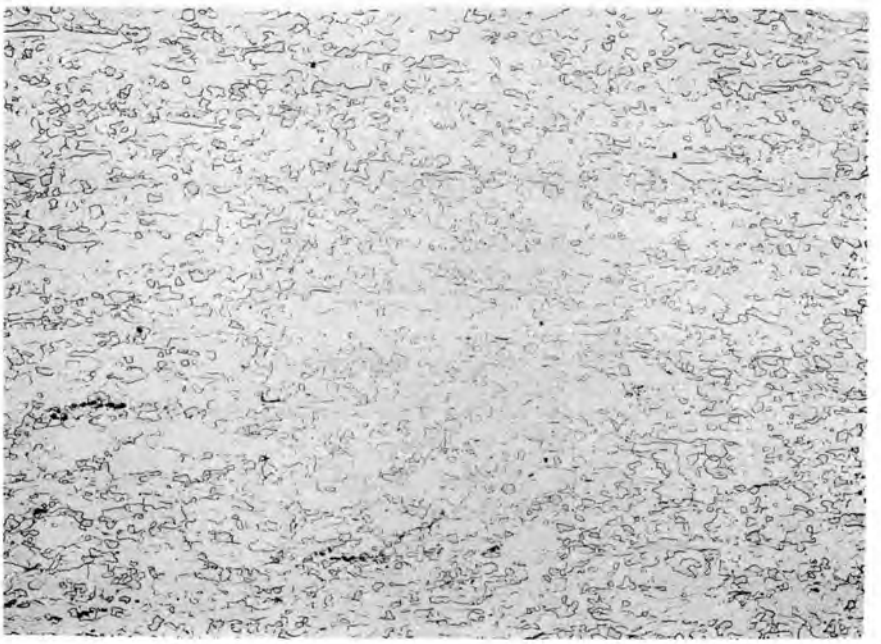

Figure 10 (Y-23136). Typical microstructure of powder-metallurgy niobium, parallel direction. Etchant - 25 HF, 5 H_2O, 5 HNO_3, 10 H_2SO_4. 75X.

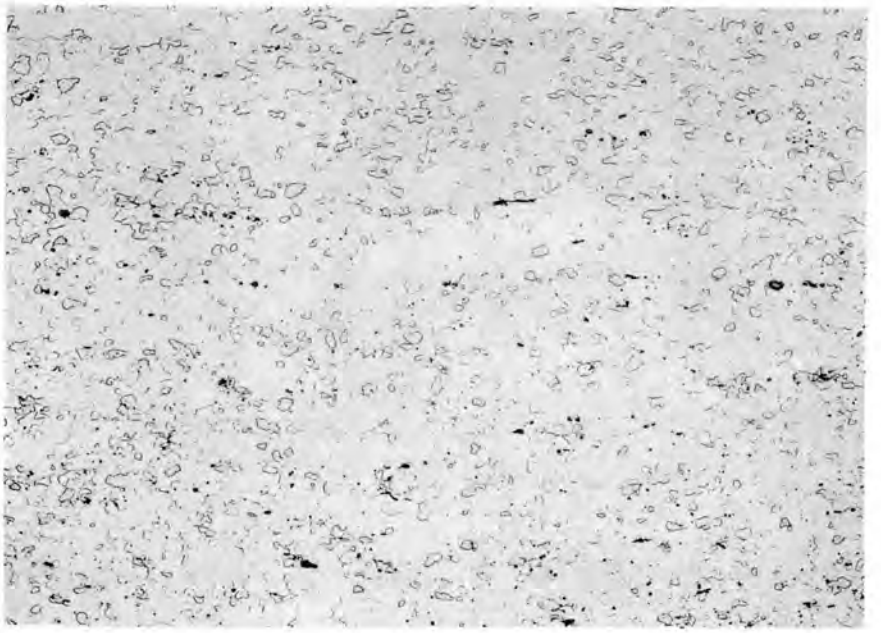

Figure 11 (Y-23135). Typical microstructure of powder-metallurgy niobium, transverse direction. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 75X.

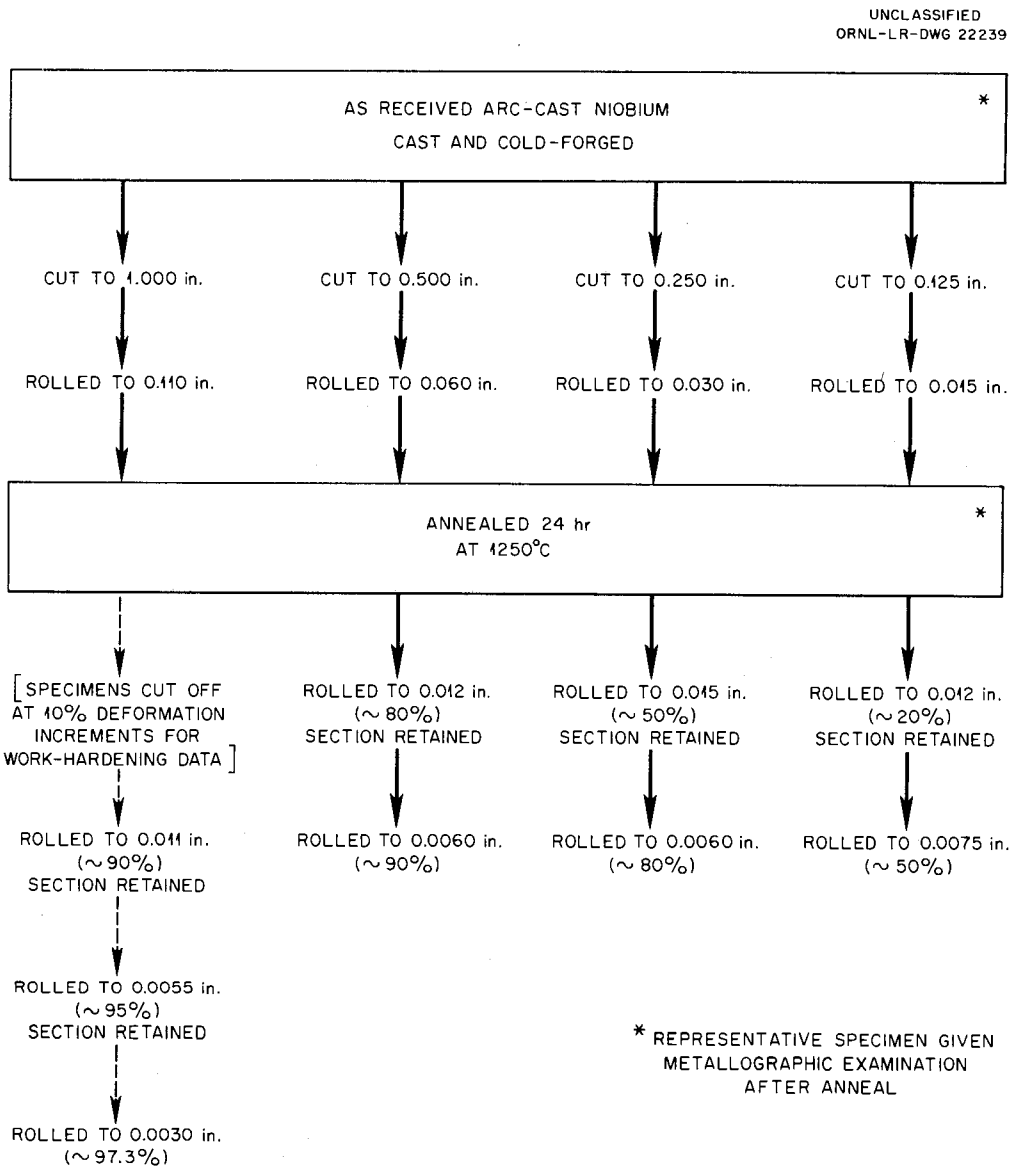

Figure 12 (Y-23164). Fabrication schedule for x-ray investigation of cold-rolled and annealed arc-cast niobium.

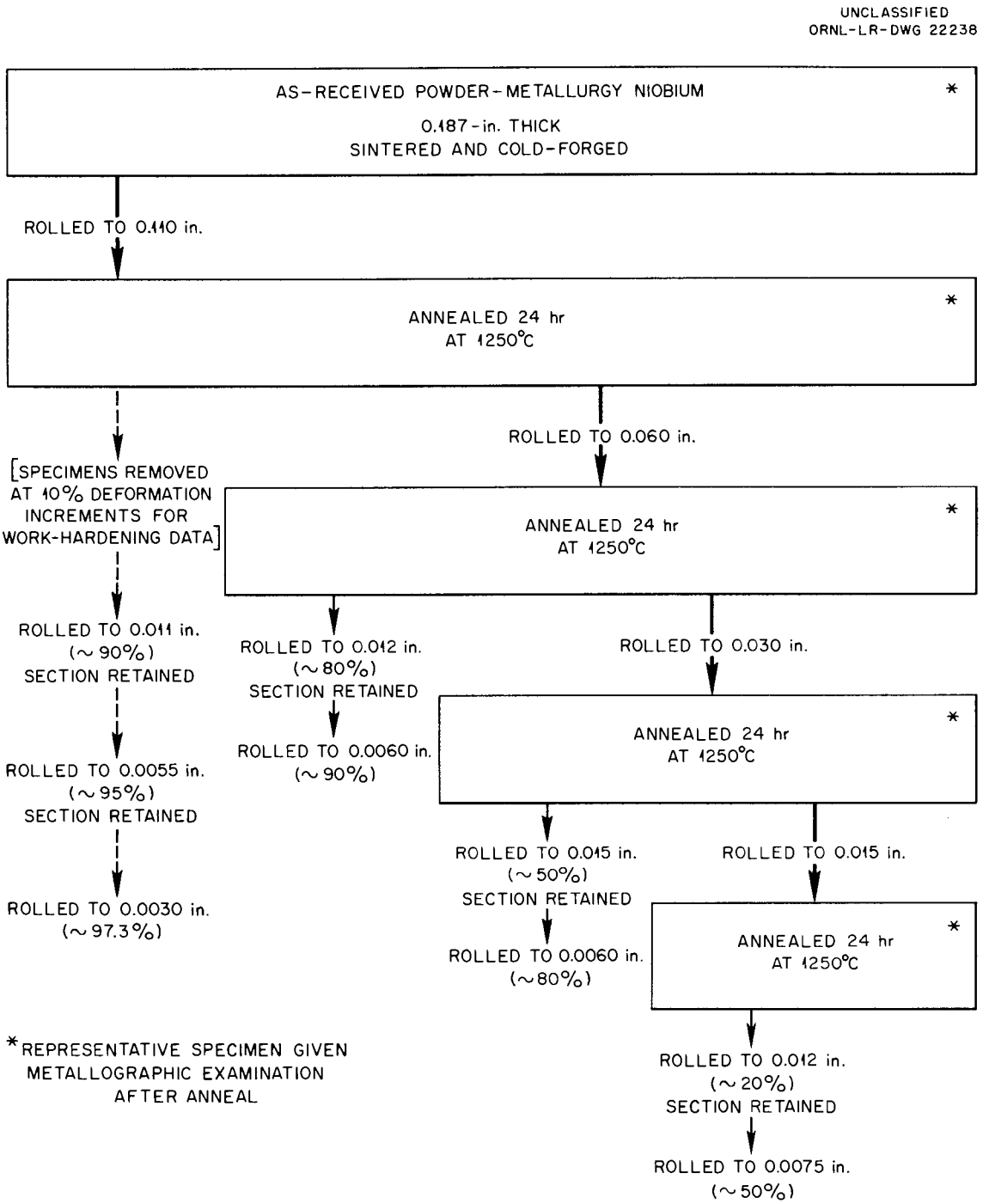

Figure 13 (Y-23169). Fabrication schedule for x-ray investigation of cold-rolled and annealed powder-metallurgy niobium.

material for the pre-recrystallization treatment, and still have a material thick enough to allow another 97 per cent reduction, it was necessary to "edge-roll" a one inch by one-half inch by two inch section of the arc-cast niobium. This was accomplished by enclosing the niobium in a one-inch thick frame of annealed copper.

The specimens chosen for study had reductions in thickness of 20, 50, 80, 90, 95, and 97.3 per cent. These reductions represent approximately equal increments in the extension of a metal during rolling. Specimens were annealed in vacuum for 36 seconds (10^{-2} hours), 6 minutes (10^{-1} hours), one, ten, and one hundred hours at 900, 1050, 1200, and 1350°C. The annealing furnaces which were used are described in the Appendix.

After being annealed, the specimens were analyzed for recrystallization in the manner described by K. Detert and K. Lücke.[12] This method, a variation of the transmission-Laue technique, consists of passing a beam of unfiltered molybdenum radiation through a very thin sample. Because the distance from the beam aperture to the specimen is identical to the distance from the specimen to the film, diffraction from a set of non-distorted planes causes the formation of a "spot" on the film. Diffraction from distorted (worked) metal causes "smears" to be formed on the film. A schematic diagram of the x-ray technique is presented in Figure 14.

Figures 15 through 19 illustrate the interpretation of a series of x-ray patterns. The high degree of preferred orientation achieved

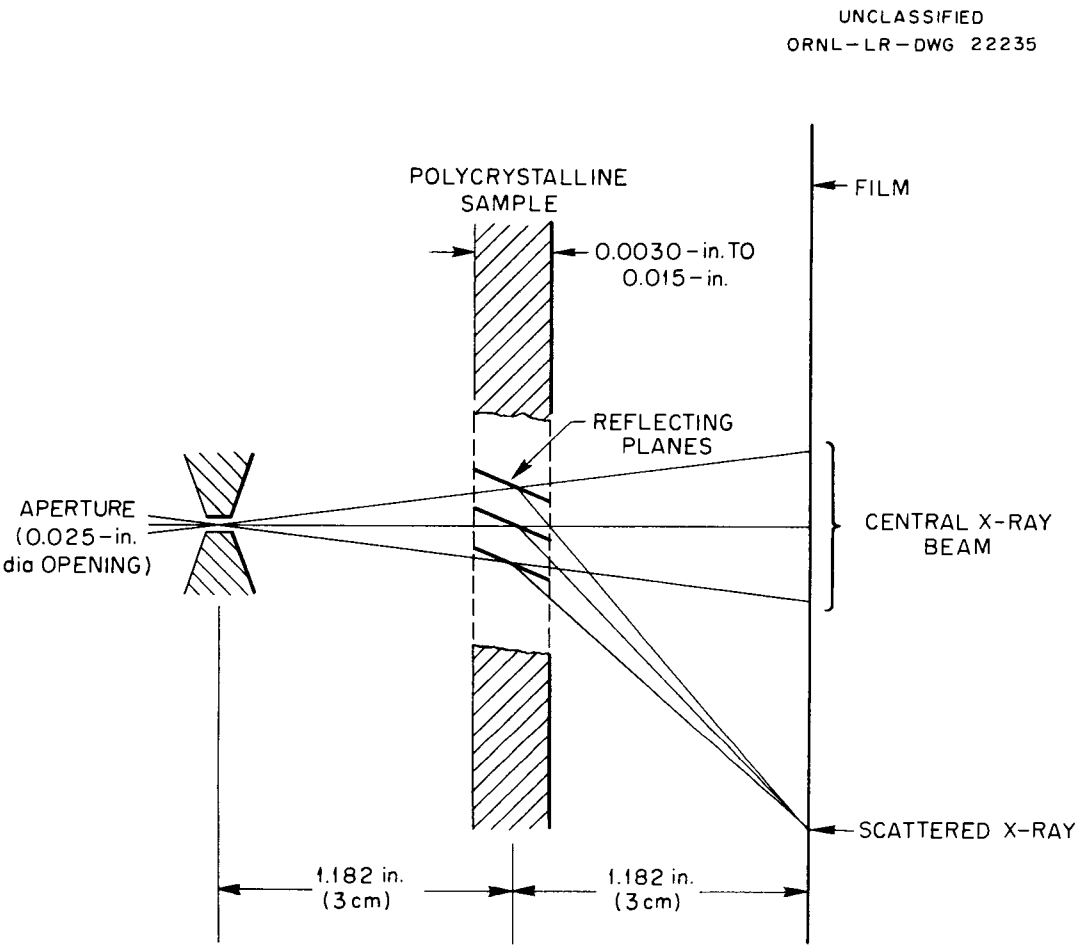

Figure 14 (Y-23160). Schematic diagram of the x-ray technique.

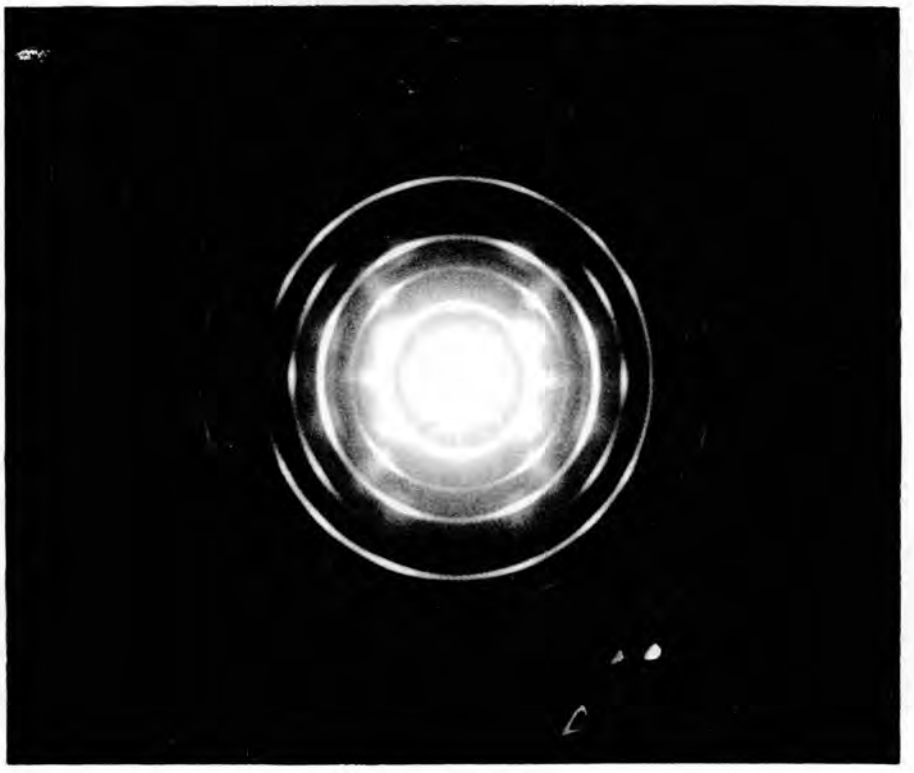

Figure 15. X-ray pattern from arc-cast niobium specimen cold-rolled 97.3%, as rolled.

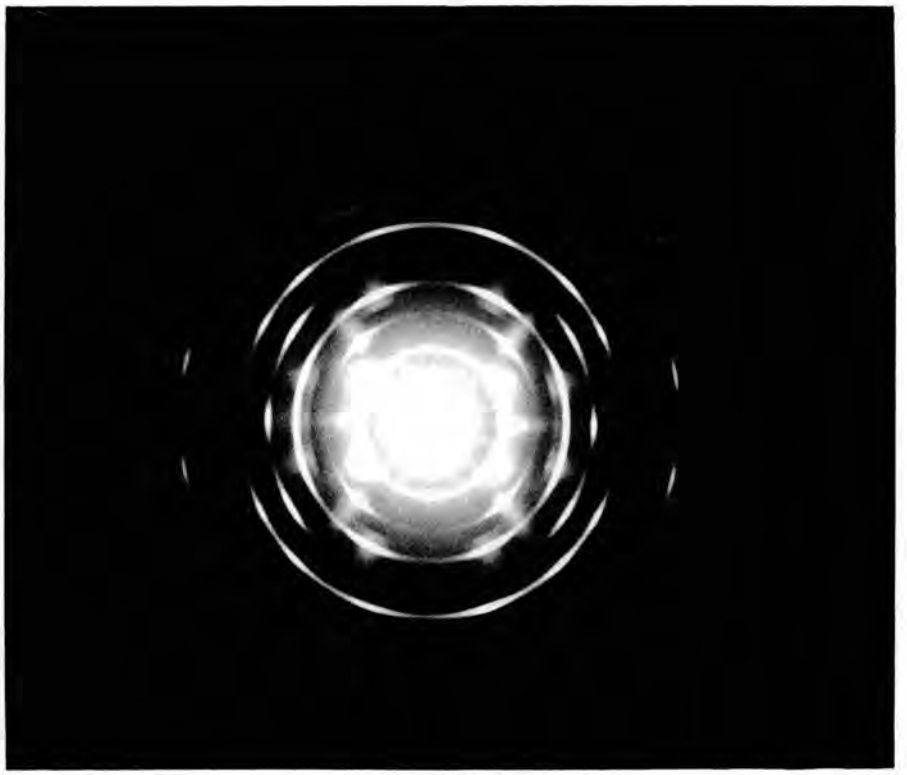

Figure 16. X-ray pattern from arc-cast niobium specimen cold-rolled 97.3% and annealed 6 minutes at 900°C.

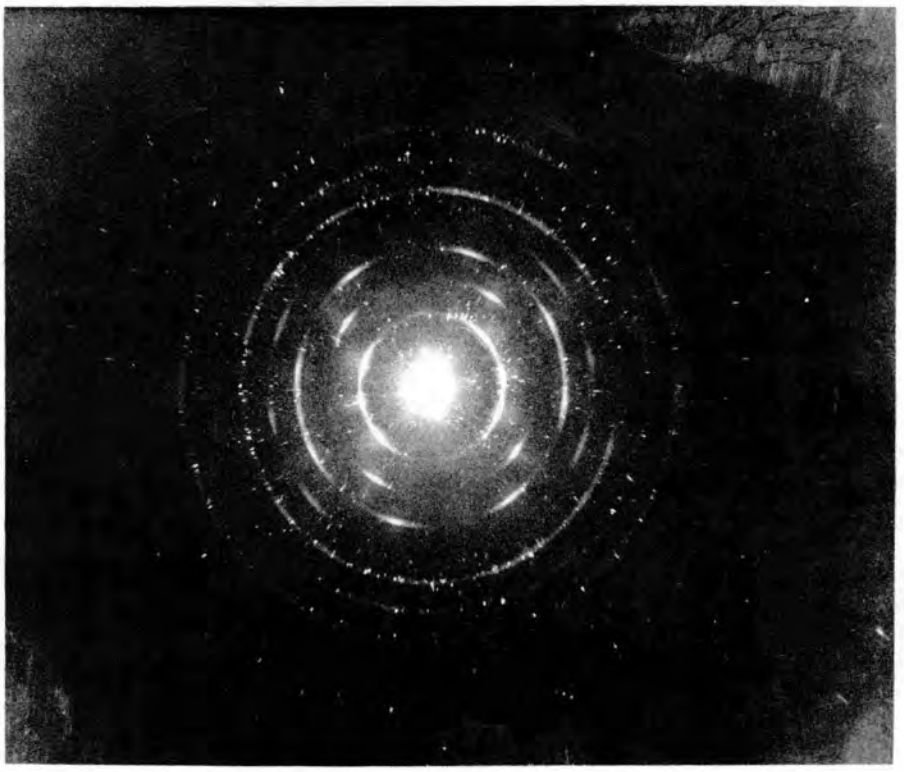

Figure 17. X-ray pattern from arc-cast niobium specimen cold-rolled 97.3% and annealed 6 minutes at 1050°C.

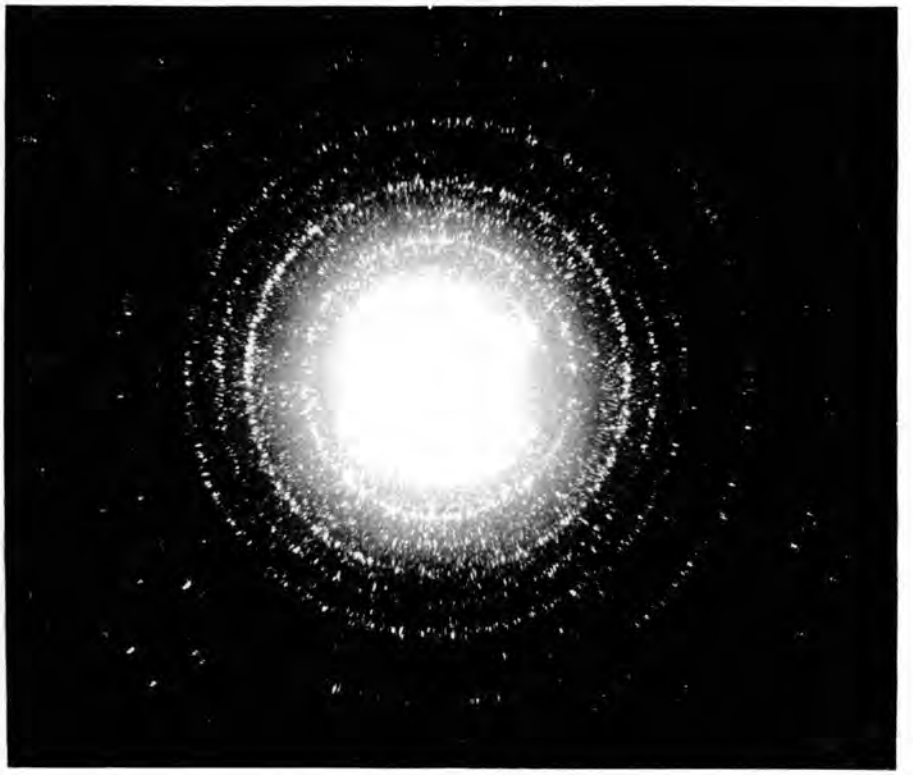

Figure 18. X-ray pattern from arc-cast niobium specimen cold-rolled 97.3% and annealed 6 minutes at 1200°C.

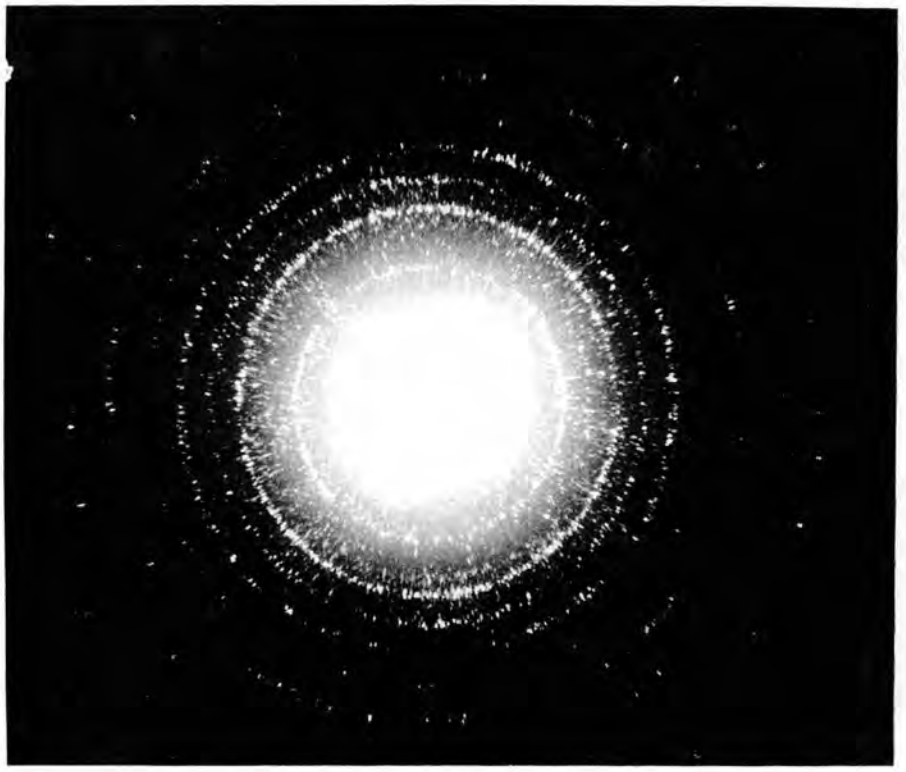

Figure 19. X-ray pattern from arc-cast niobium specimen cold-rolled 97.3% and annealed 6 minutes at 1350°C.

after 97.3 per cent reduction is obvious in Figures 15, 16, and 17.* The initiation of recrystallization is very sharply defined by the appearance of spots, such as those in Figure 17. The termination of recrystallization, however, requires a more subjective interpretation. Although it is not obvious in the photographic reproduction, the pattern from the 1200°C specimen showed a very faint trace of a smear and was therefore considered not completely recrystallized.

In order to determine the reproducibility of this technique, "control" specimens had been prepared at deformation levels of 20, 50, 80, and 90 per cent reduction in thickness. These control specimens were slightly thicker than the primary specimens (see Figures 12 and 13). After being annealed concurrently with the thinner specimens, the control specimens were etched to approximately half their original thickness and analyzed by the transmission-Laue technique. No differences were noted, indicating that: 1) the effects of segregation of impurities in the arc-cast material were negligible, 2) section size, within the range of investigation, had no effect, and 3) there were no "skin" effects.

The results of the x-ray investigation are presented in Chapter VI.

The final phase of the over-all study was concerned with the effect of annealing on the mechanical properties of arc-cast and powder-metallurgy niobium. Fabrication schedules were established which would

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*It is interesting to note that the (110) and (200) intensities correspond, qualitatively at least, to pole figures published for two other body-centered cubic metals: iron[13] and vanadium[14].

allow comparison of the mechanical-properties changes to the results of the x-ray investigation. These schedules are presented in Figures 20 and 21.

The tensile specimens were simply "punched" from the cold-rolled sheet with a blanking die designed by H. Inouye of ORNL. Careful filing of the gage section constituted the only "machining" necessary for the fabrication of these specimens. The nominal dimensions of the specimens, as punched, were: length, five inches; shoulder width, five-eights of an inch; gage length, two inches; gage width, one-fourth of an inch. Most of the specimens were 0.040-inches thick, except the 90 per cent rolled specimens, which were 0.020-inches thick.

After the prescribed annealing treatments, the specimens were pulled at room temperature, using equipment described in the Appendix. The results are described in the next chapter.

These specimens, and four isothermally annealed series of "scraps" (from the cold-rolled sheet from which the tensile specimens had been punched) afforded material for metallographic verification of the x-ray analysis, and a qualitative "picture" of the dependancy of reaction rate on temperature. Sections of the shoulders of the annealed tensile specimens and the annealed scraps were mounted in Bakelite, and then given hardness and metallographic examination; the results are presented and discussed in the next chapter. The hardness-testing equipment and procedure are described in the Appendix.

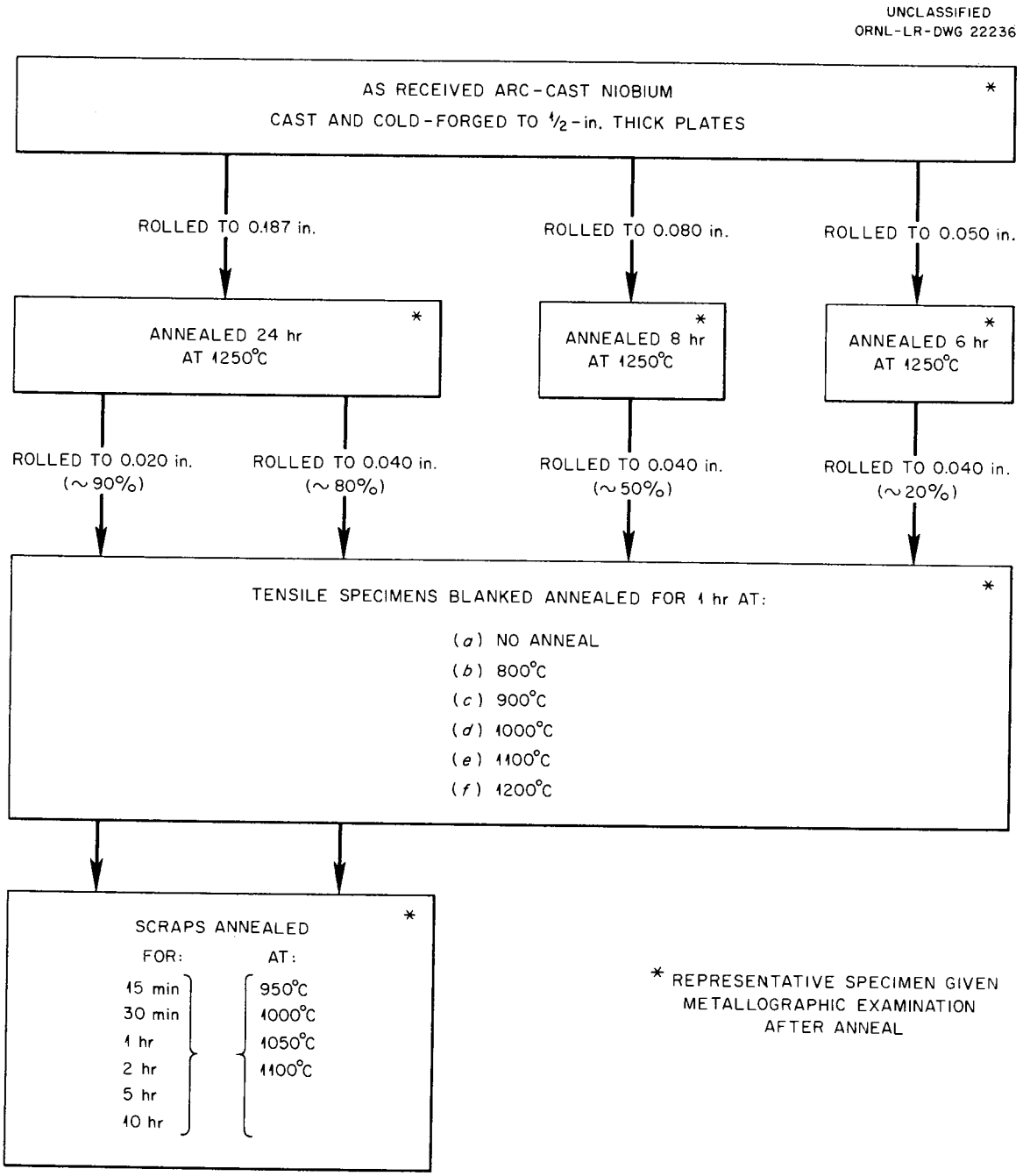

Figure 20 (Y-23168). Fabrication schedule for mechanical properties investigation of cold-rolled and annealed arc-cast niobium.

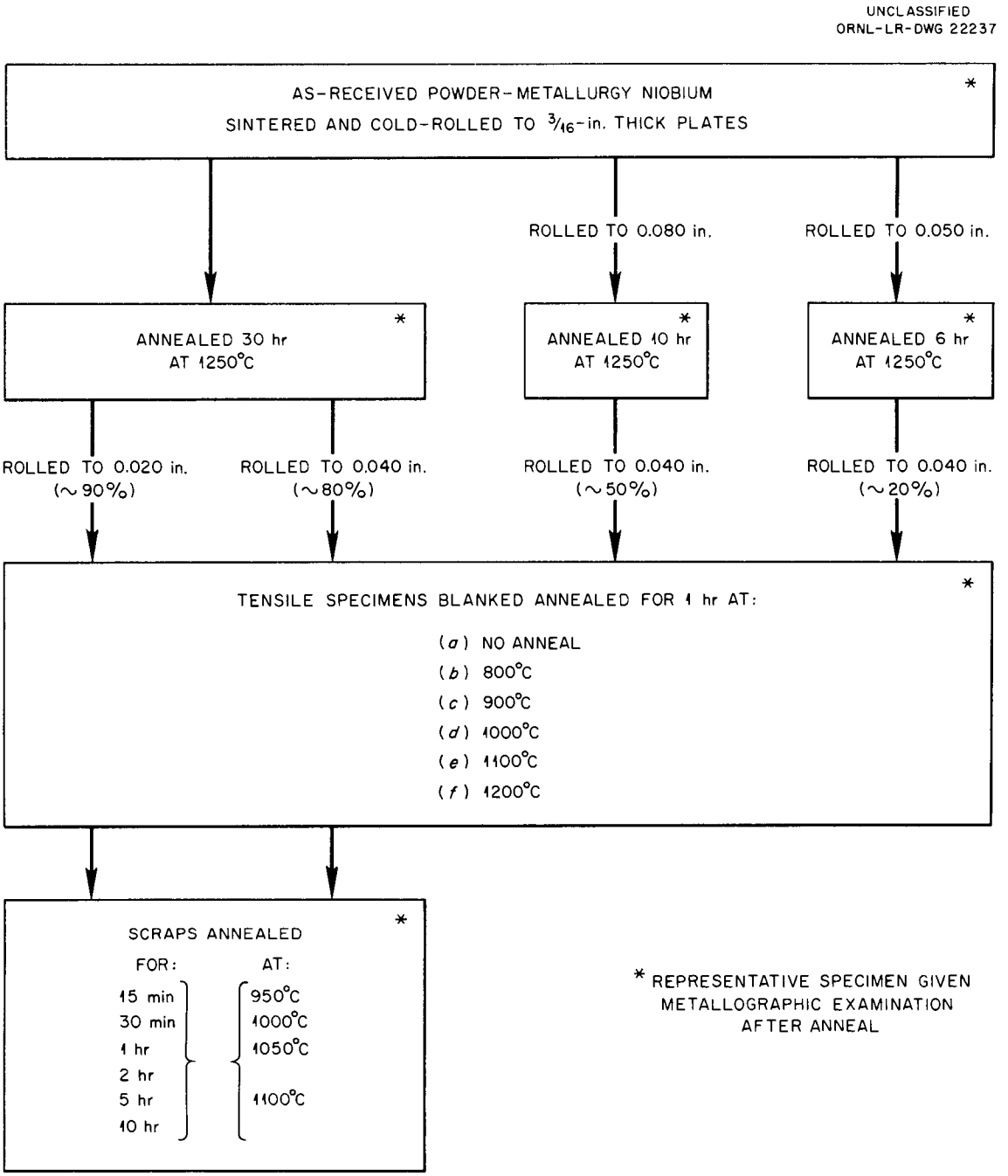

Figure 21 (Y-23170). Fabrication schedule for mechanical properties investigation of cold-rolled and annealed powder-metallurgy niobium.

CHAPTER VI

RESULTS AND DISCUSSION

A. Results Of X-Ray Investigation

The data resulting from the x-ray investigation of the recrystallization behavior of arc-cast and powder-metallurgy niobium are presented in Table II. Within the sensitivity of the experimental design and technique of this investigation, there were no significant differences between the two materials. The methods and criteria by which the data were obtained and analyzed, respectively, are described in the chapter on experimental procedure.

Interpretation of this data yielded "surfaces" representing initiation and termination of recrystallization as a function of prior deformation, annealing time, and annealing temperature. A drawing of these three-dimensional surfaces is presented in Figure 22, while drawings showing the initiation and termination, separately, are presented in Figure 23.

It must be recognized that these drawings are merely the results of an interpretation of datum points in a three-dimensional grid system. No attempt was made to "pin-point" either the initiation or termination of recrystallization. For this reason, the surfaces and lines, in Figures 22 and 23, respectively, represent zones of uncertainty rather than clean-cut separations.

TABLE II

DATA FROM X-RAY PATTERNS

Deformation	Annealing Temperature °C	Annealing Time 36 Sec.	6 Min.	1 Hr.	10 Hr.	100 Hr.
20% Reduction in Thickness	900	Smears				Smears
	1050	Smears				Smears
	1200	Smears				Smears
	1350	Smears	Smears*	Both*	Both	Both*
50% Reduction in Thickness	900	Smears				Smears
	1050	Smears*	Both*			Both*
	1200	Both*		Both		Both*
	1350	Spots*				
80% Reduction in Thickness	900	Smears				Smears
	1050	Smears*	Both*			Both
	1200	Both			Both*	Spots*
	1350	Spots				
90% Reduction in Thickness	900	Smears				Smears
	1050	Smears*	Both*			Both
	1200	Both	Both*	Spots*		
	1350	Spots				
95% Reduction in Thickness	900	Smears				Smears
	1050	Smears				Both
	1200	Both	Both	Spots*		
	1350	Spots				
97 3% Reduction in Thickness	900	Smears				Smears
	1050	Smears	Both			Both*
	1200	Both*	Spots*			
	1350	Spots				

NOTE: These data apply to arc-cast niobium only except where marked (*); data so marked are from both arc-cast and powder-metallurgy niobium.

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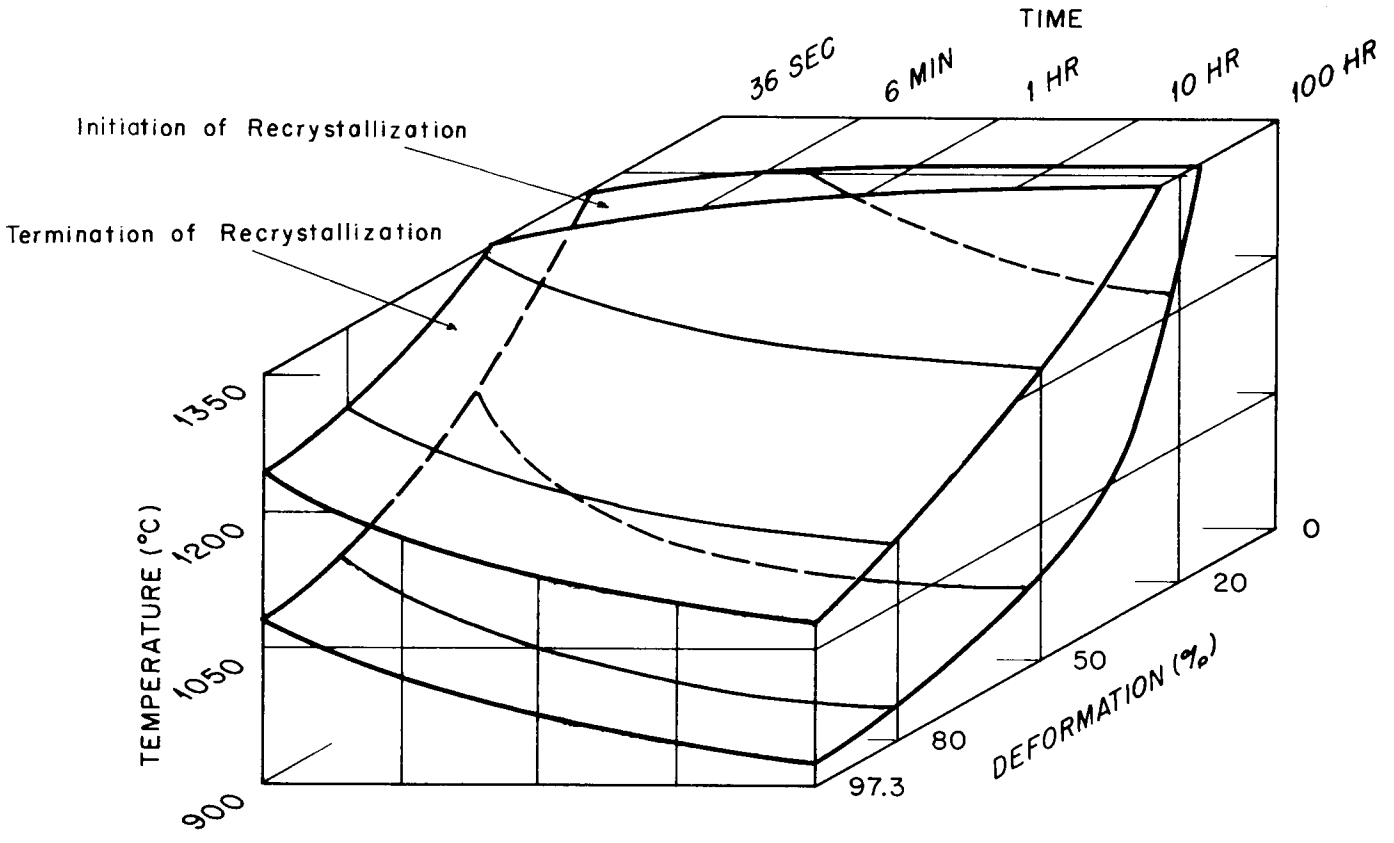

Figure 22 (Y-23165). Recrystallization in niobium as a function of annealing time, annealing temperature, and deformation.

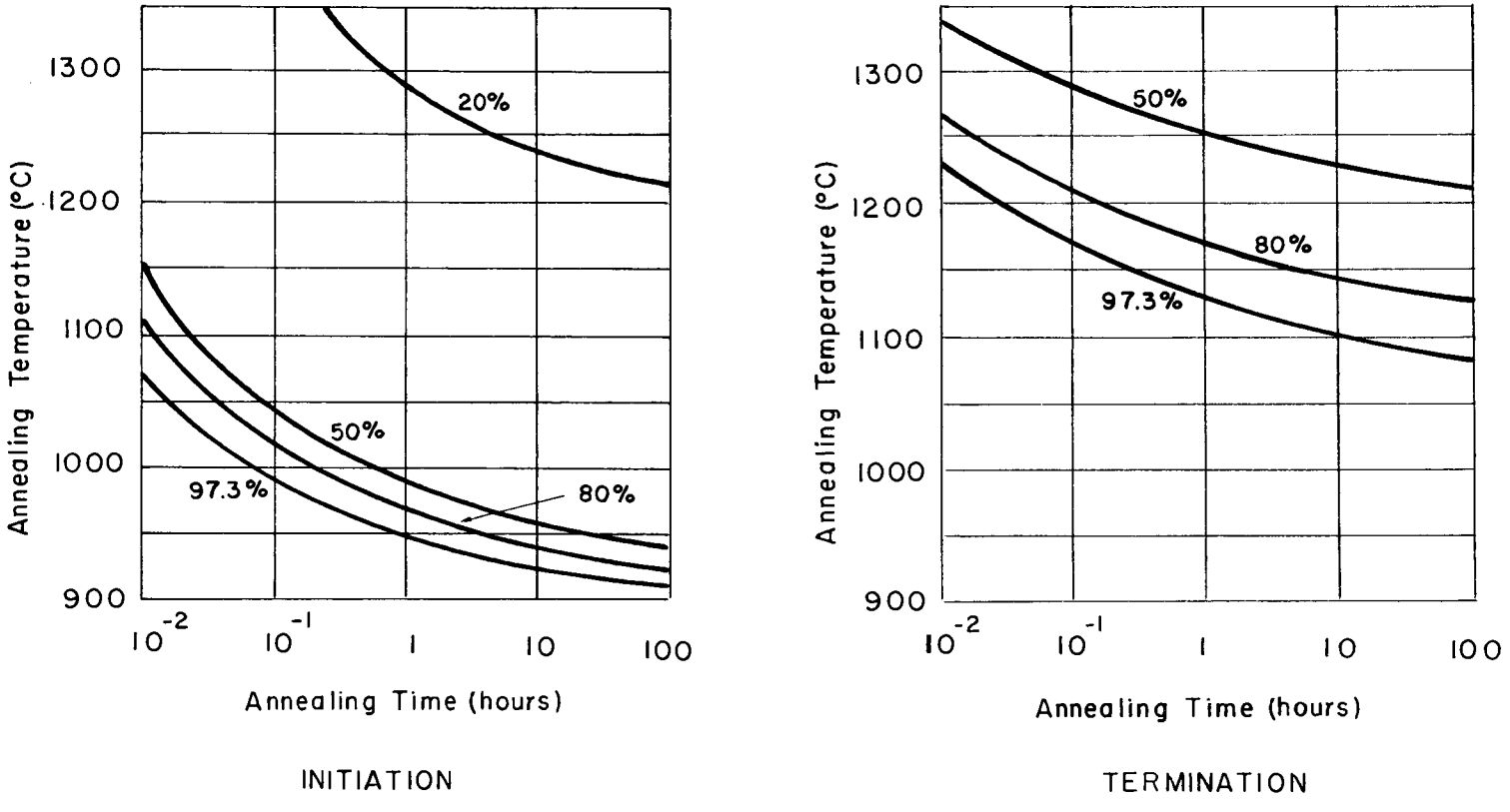

Figure 23 (Y–23171). Approximate initiation and termination of recrystallization in niobium.

The six deformation levels (20, 50, 80, 90, 95, and 97.3 per cent reduction in thickness) which were chosen for examination represented approximately equal increments of strain. The data, however, indicated that the results could be represented more satisfactorily by plotting deformation on a linear basis. Therefore, only four deformation levels, 20, 50, 80, and 97.3 per cent, are shown in Figures 22 and 23.

Attempts to correlate the x-ray results with hardness and metallographic examination were not entirely satisfactory. The specimen sizes had been kept extremely small in order to conserve material. These specimens did not, therefore, lend themselves to application of lineal analysis techniques, although in no case was there a direct conflict between x-ray and metallographic data. Hardness determinations were complicated by two factors: a) the small size of the specimen, which necessitated the use of very light loads, thereby making individual measurements sensitive to very small variations in a single specimen, and b) the effect of even minor amounts of contamination (by oxygen and nitrogen, during the vacuum-anneal) in raising hardness appreciably. Unfortunately, it was impossible to determine the effect of contamination on the recrystallization behavior. This effect, however, was thought to be negligible in comparison to the uncertainties in the experimental design.

Figures 22 and 23 are presented as general guides for recrystallization of niobium. A metallurgist, using these figures, should realize that they are only directly applicable to niobium of the

specifically-described composition and grain size. The fact that both the arc-cast and powder-metallurgy niobium exhibited similar behavior suggests, however, that some "generality" may be assumed.

B. Work-Hardening Of Niobium

Work-hardening curves for powder-metallurgy and arc-cast niobium are presented in Figure 24. Except for an initial sharp increase in hardness, the hardness of niobium is a linear function of the extension of the cold-rolled sheet.* The rates of work-hardening vary from specimen to specimen but are always very low. There is no significant difference in the work-hardening rates of the two materials; it appears that either can be rolled almost indefinitely without intermediate anneals.

C. Results Of One-Hour Annealing Treatments

As shown on the fabrication schedules, Figures 20 and 21, tensile specimens were annealed for one hour at room temperature (no anneal), 800, 900, 1000, 1100, and 1200°C. These were tensile-tested at room temperature; the results of these tests are presented in Figures 25 through 28. The limitation in the amount of available

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*In a study of lattice distortion on cold-worked Permalloy, Haworth[16] plots the broadening of the Fe Kα doublet diffracted from the (311) planes as a function of "log per cent of original thickness". This curve is identical in shape to those of Figure 24.

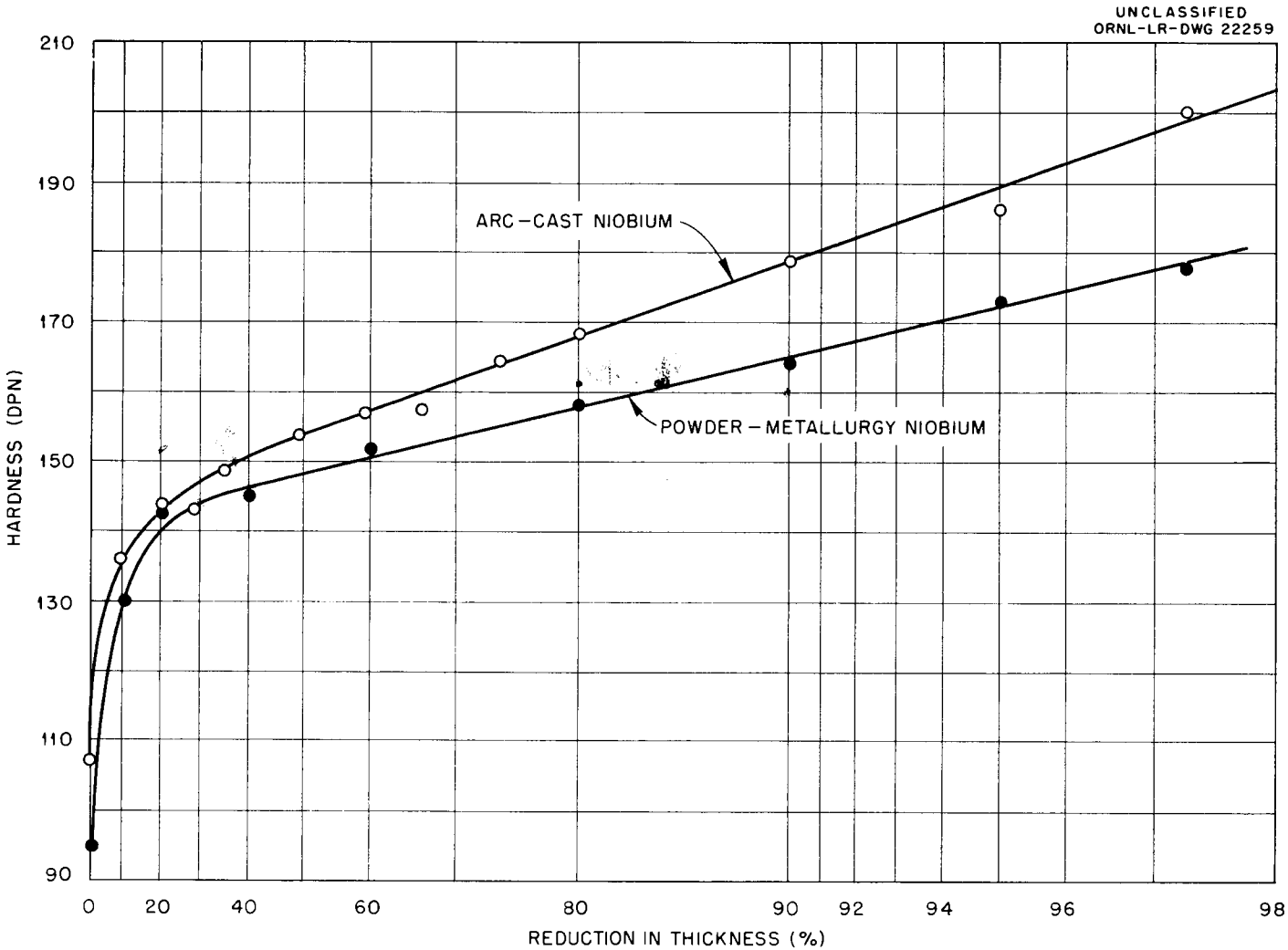

Figure 24 (Y-23162). Work-hardening of cold-rolled arc-cast and powder-metallurgy niobium.

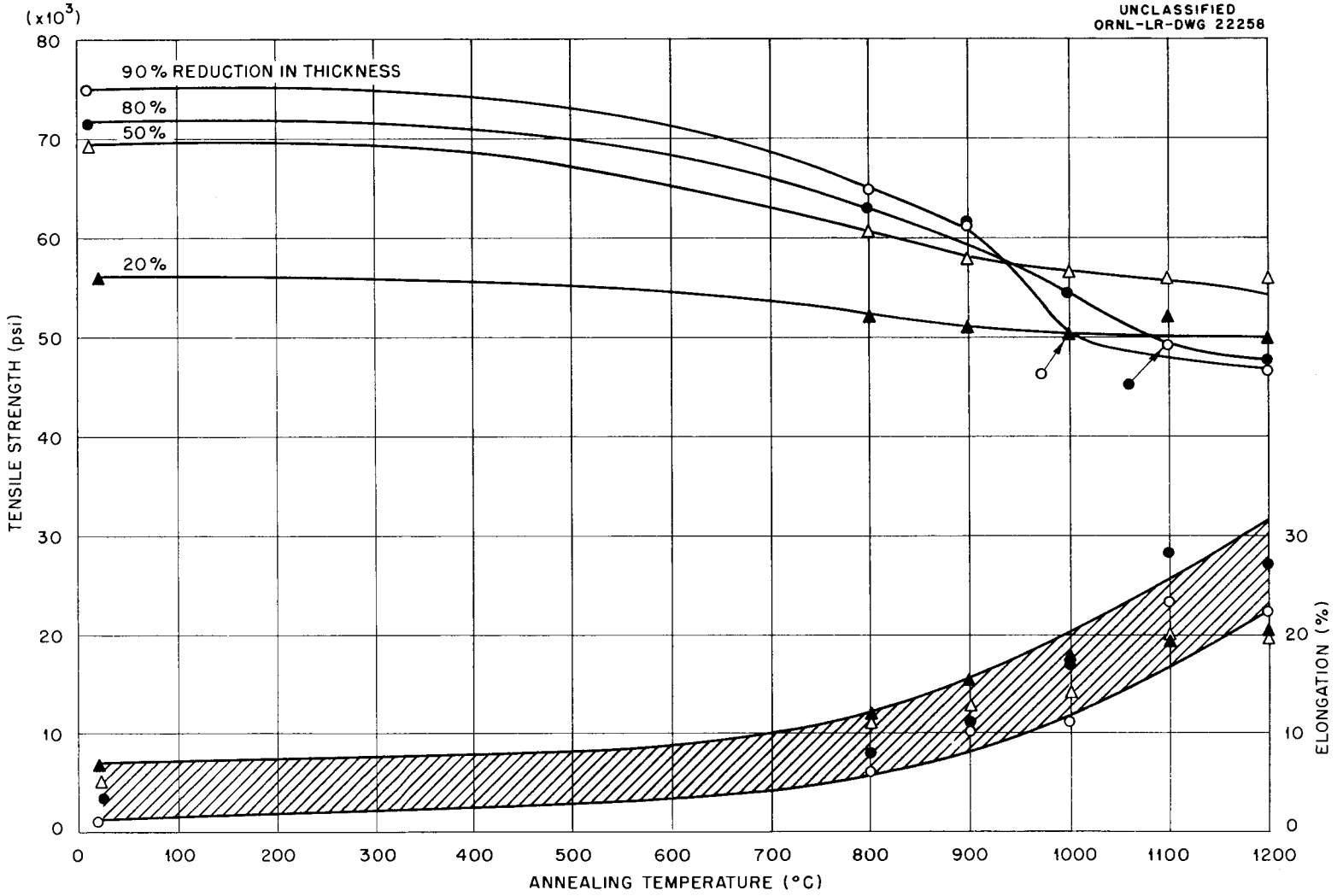

Figure 25 (Y-23161). Room-temperature tensile strength and elongation (2-in. gage length) of arc-cast niobium deformed by rolling and annealed 1 hr at indicated temperature.

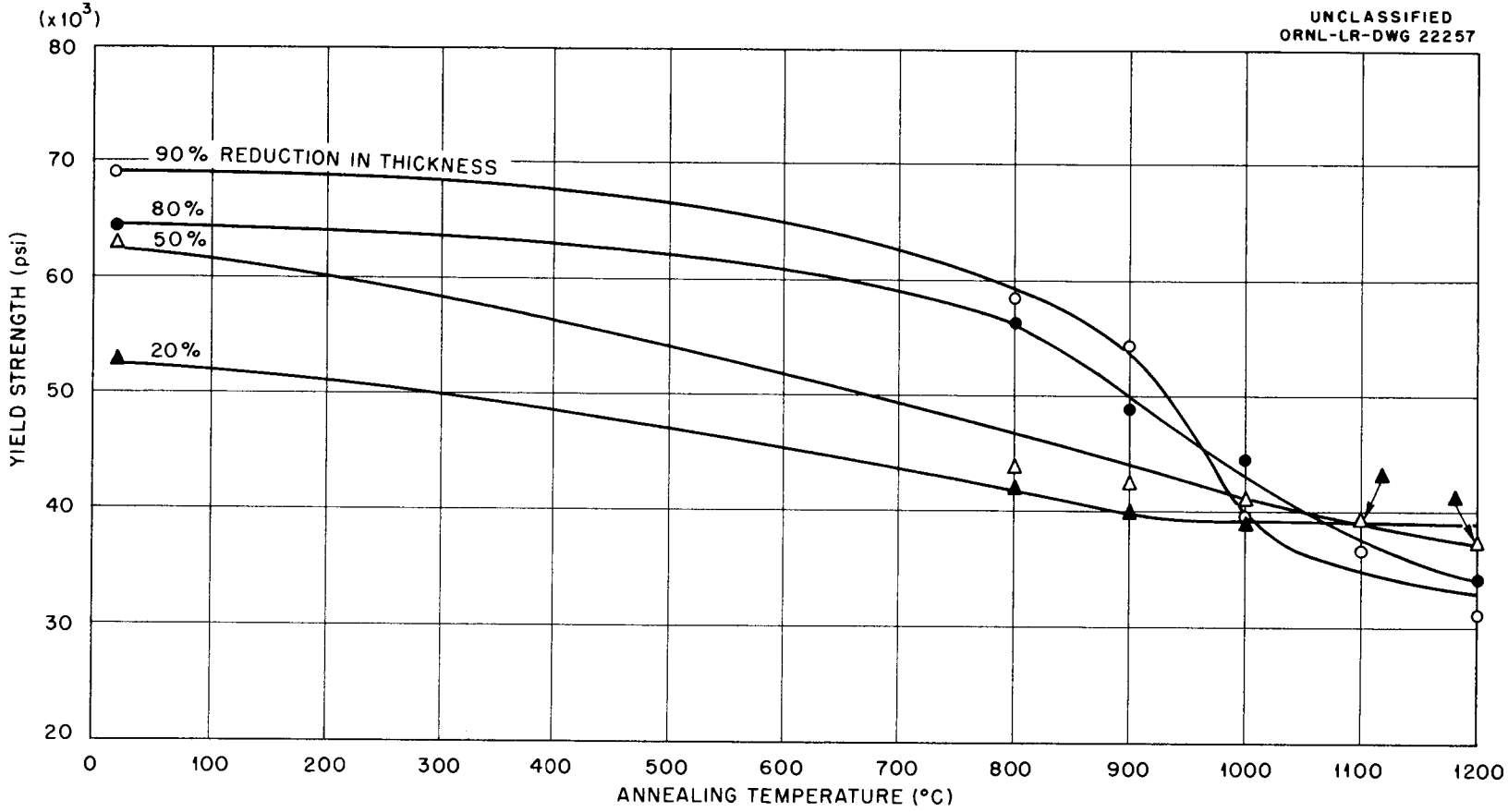

Figure 26(Y-23166). 0.2% Offset yield strength at room temperature: arc-cast niobium deformed by rolling and annealed 1 hr at indicated temperature.

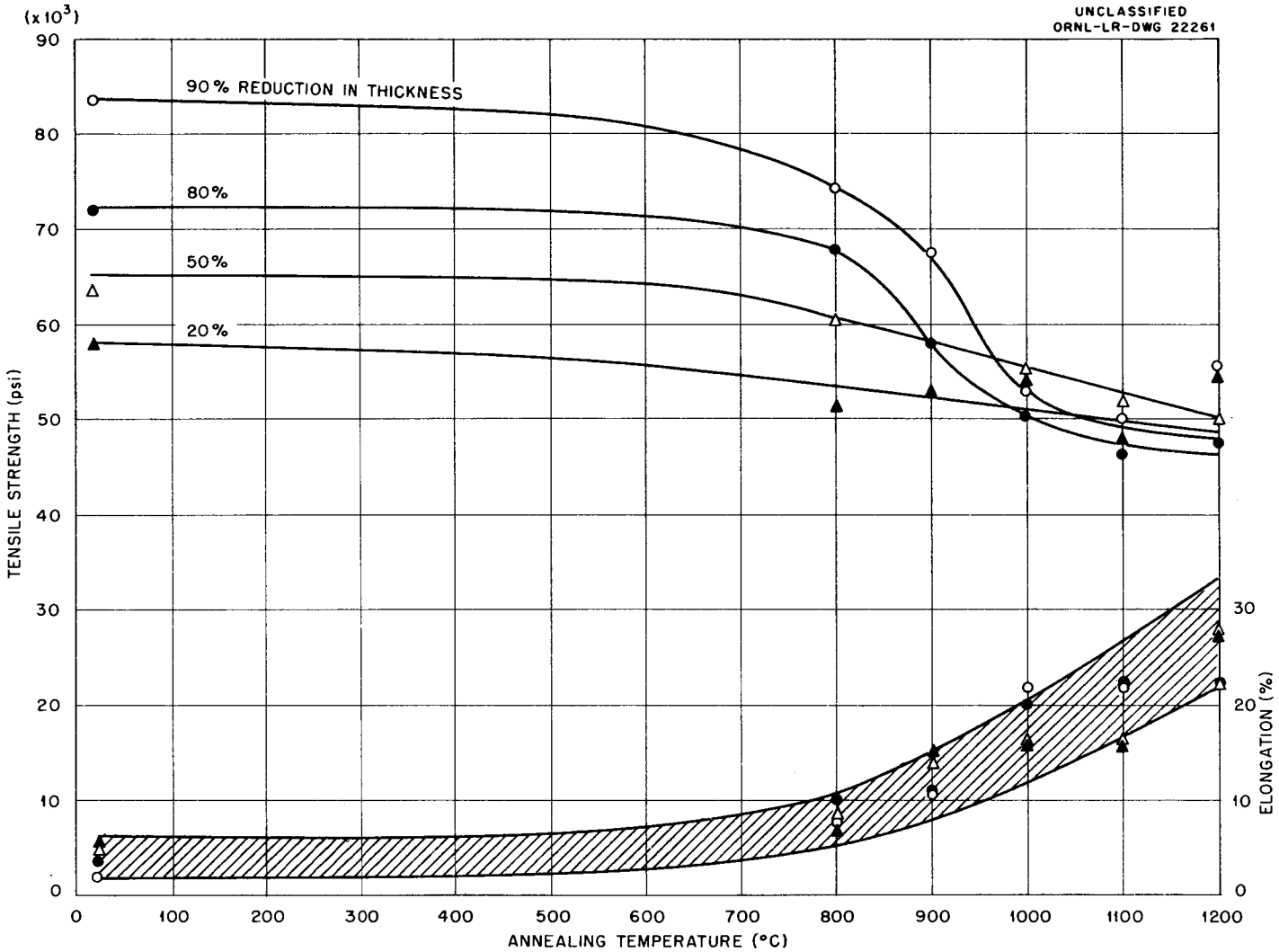

Figure 27 (Y-23163). Room-temperature tensile strength and elongation (2-in. gage length) of powder-metallurgy niobium deformed by rolling and annealed 1 hr at indicated temperature.

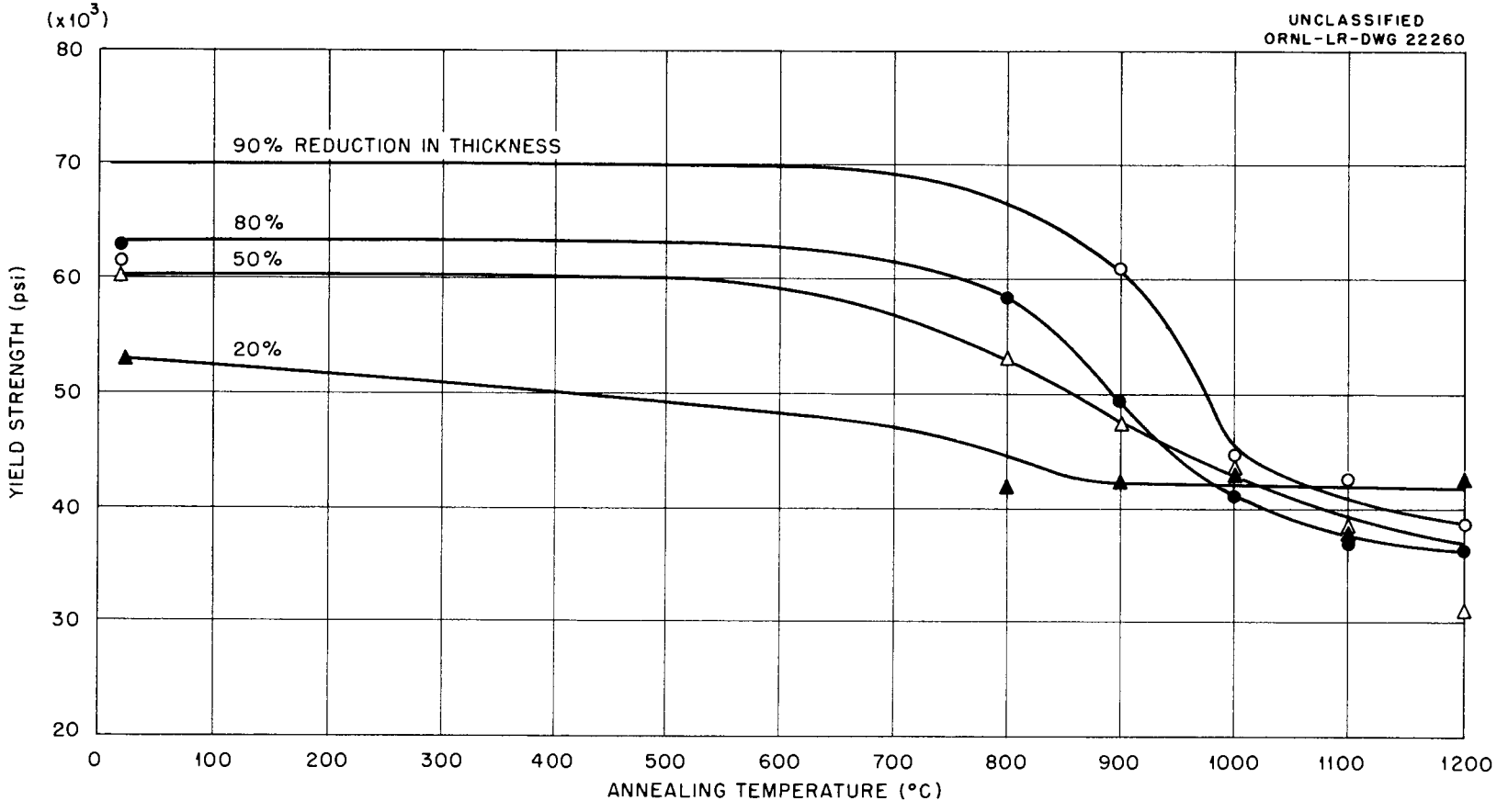

Figure 28 (Y-23167). 0.2% Offset yield strength at room temperature: powder-metallurgy niobium deformed by rolling and annealed 1 hr at indicated temperature.

material and the interest in trends rather than absolute values necessitated the preparation and testing of only one specimen of each condition. For this reason, there is a considerable amount of "scatter" in the data, and curves are drawn rather freely with respect to the datum points.

Ductility, an intrinsically inaccurate measurement, is represented as a band. It is interesting to note that, although the strength properties begin to level off at approximately 1000°C, ductility continues to increase with increasing annealing temperature.

According to Figure 23, neither the 20 nor the 50 per cent reduced material should completely recrystallize at the times and temperatures of these annealing treatments. This is partially confirmed by the fact that none of these specimens show a leveling-off in properties, as is exhibited by the 80 per cent and 90 per cent reduced specimens. Such a leveling-off in properties, of course, would be harder to distinguish for the lower reductions, for they "start off" at a lower level. Nevertheless, it is interesting to note that the 50 per cent reduced material is almost invariably stronger after a 1200°C anneal than material worked either less or more than 50 per cent. This observation leads to the interesting consideration of "maximization" of creep properties. Perhaps it would be possible to cold work niobium to a point of "optimum" work-hardening. This niobium might retain much of its strength under given service conditions, whereas niobium having an initially higher strength (by virtue of work-hardening) might recrystallize or recover to strength levels below that of the former.

The 80 per cent deformation series was examined metallographically. Photomicrographs of the shoulders of the test specimens are presented in Figures 29 through 38. Recrystallization is not evident at 900°C (Figures 31 and 32), but reference to the mechanical-properties curves shows that the strength has dropped off appreciably. It may be said, therefore, that partial recovery of strength quite definitely precedes complete recrystallization. The data do not necessarily indicate that this is true for ductility.

Comparison of the photomicrographs of powder-metallurgy and arc-cast niobium shows a significant difference in the grain size of the recrystallized structure. Neither the mechanical properties nor their rates of change with annealing temperature appear significantly different for the two materials, however.

D. Results Of Isothermal Annealing Treatments

The fabrication schedules, Figures 20 and 21, indicate that "scraps" of 80 and 90 per cent reduced material were annealed for fifteen minutes, and one-half, one, two, five, and ten hours at 950, 1000, 1050, and 1100°C. After the annealing treatment, each specimen's hardness was determined with equipment described in the Appendix; each was then examined metallographically. The hardness data, which were disappointing, are presented in Table III. These data do not warrant extensive statistical treatment. In general it appears that niobium softens with time at temperatures of 950 and 1000°C, but that

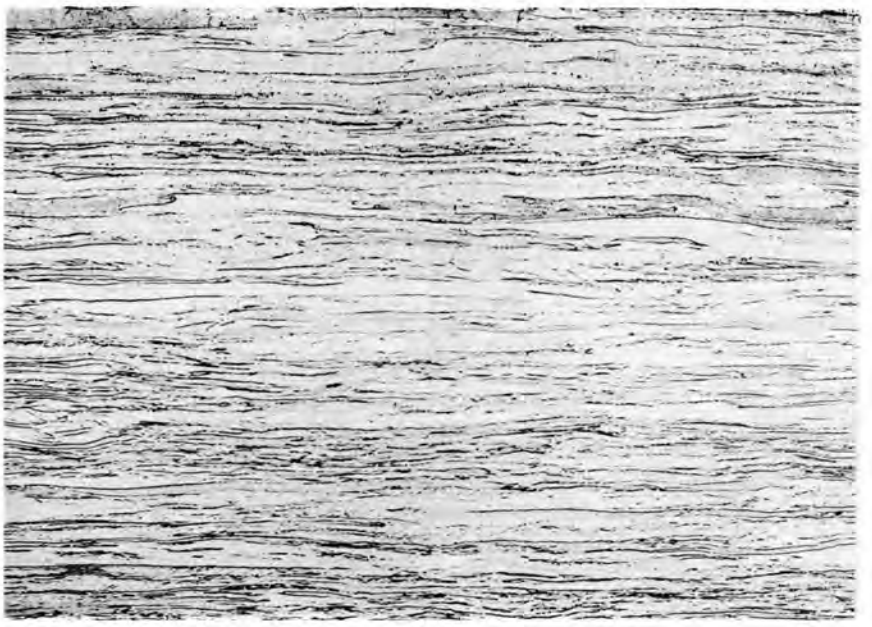

Figure 29 (Y-23124). Microstructure of 80% reduced arc-cast niobium, as rolled. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

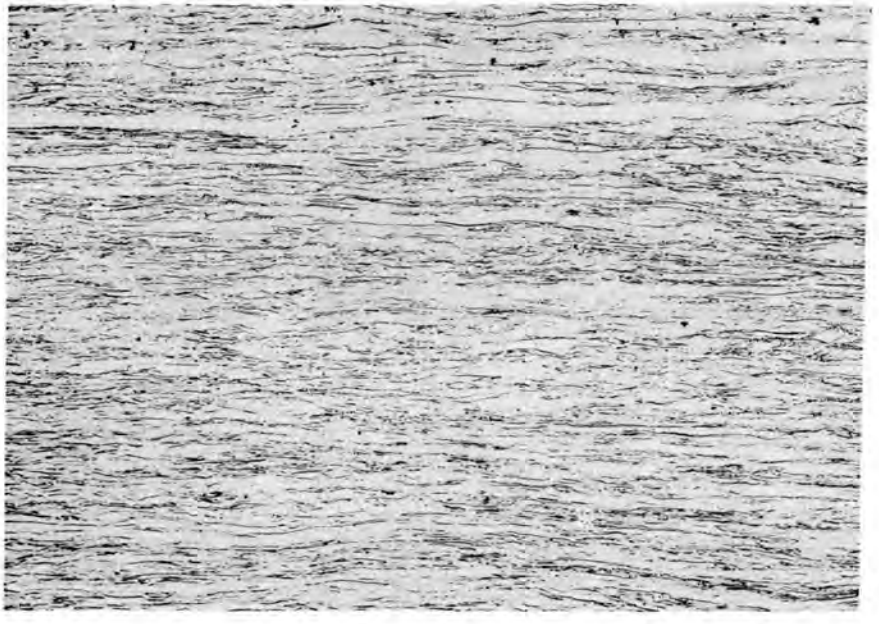

Figure 30 (Y-23134). Microstructure of 80% reduced powder-metallurgy niobium, as rolled. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

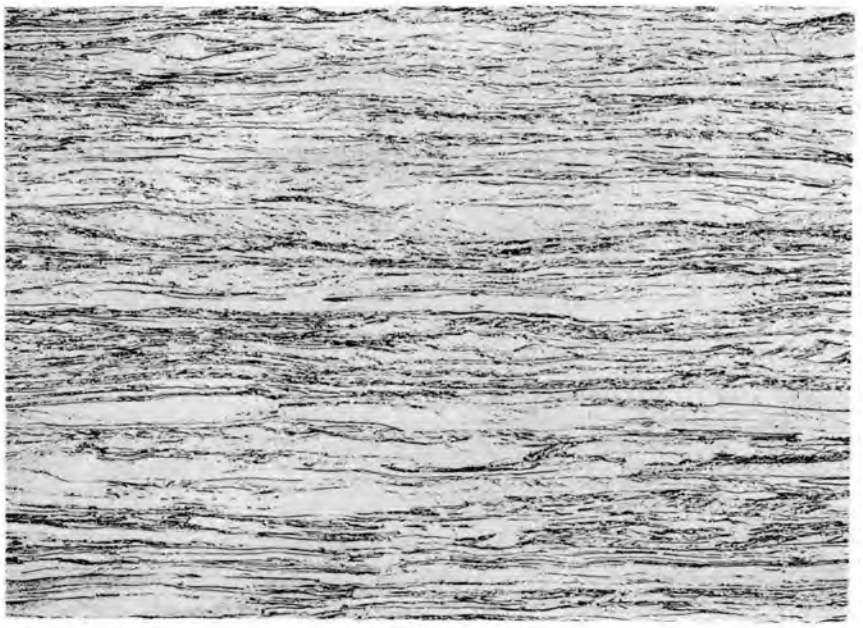

Figure 31 (Y-23129). Microstructure of 80% reduced arc-cast niobium, annealed one hour at 900°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

Figure 32 (Y-23133). Microstructure of 80% reduced powder-metallurgy niobium, annealed one hour at 900°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

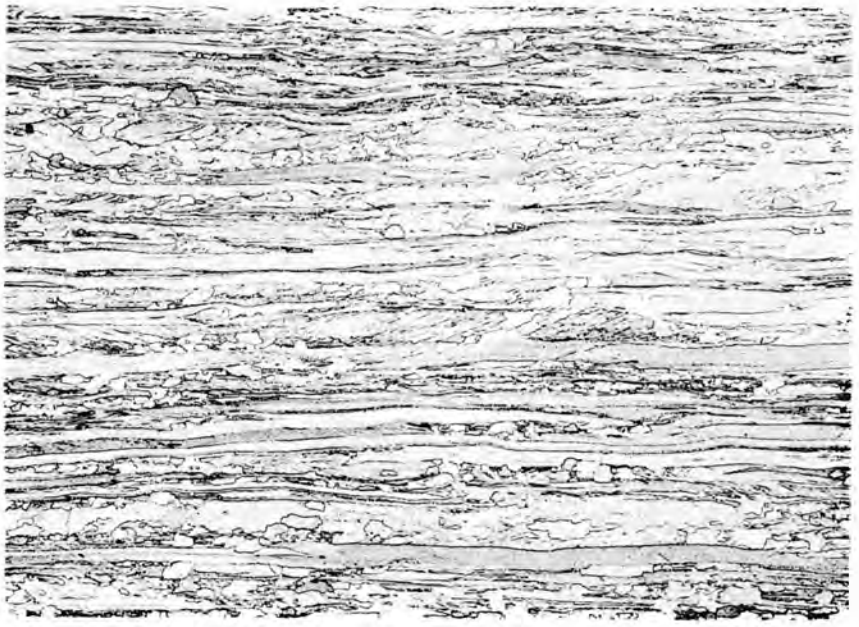

Figure 33 (Y-23128). Microstructure of 80% reduced arc-cast niobium, annealed one hour at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

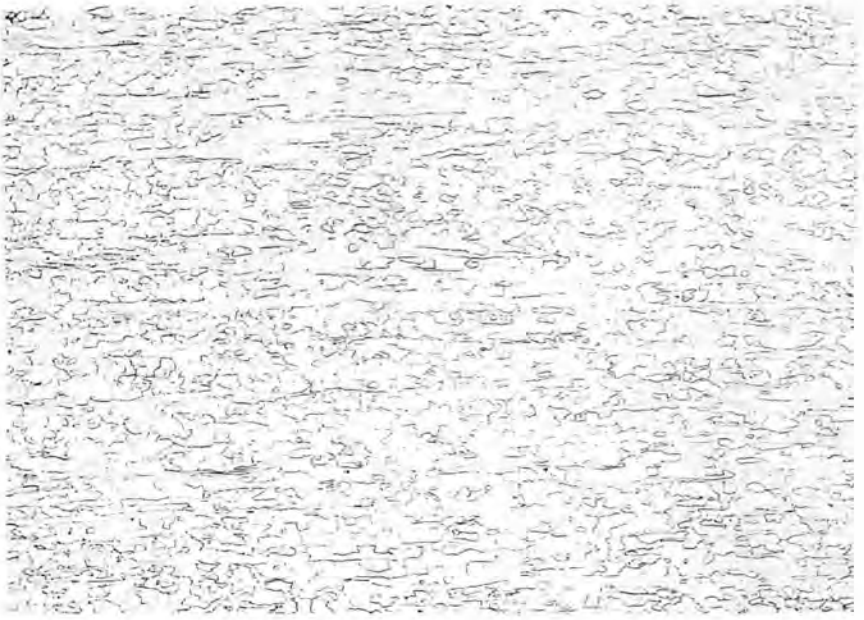

Figure 34 (Y-23132). Microstructure of 80% reduced powder-metallurgy niobium, annealed one hour at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

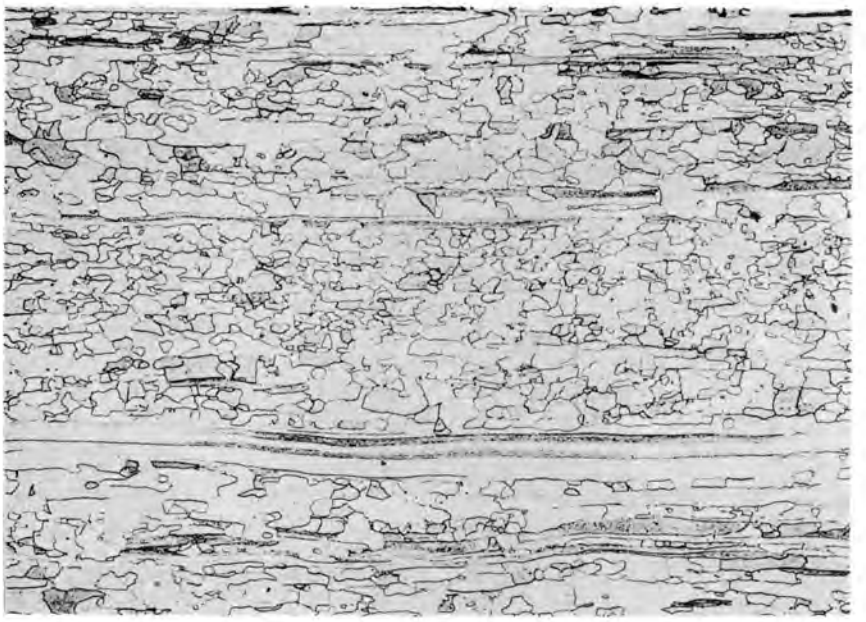

Figure 35 (Y-23127). Microstructure of 80% reduced arc-cast niobium, annealed one hour at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

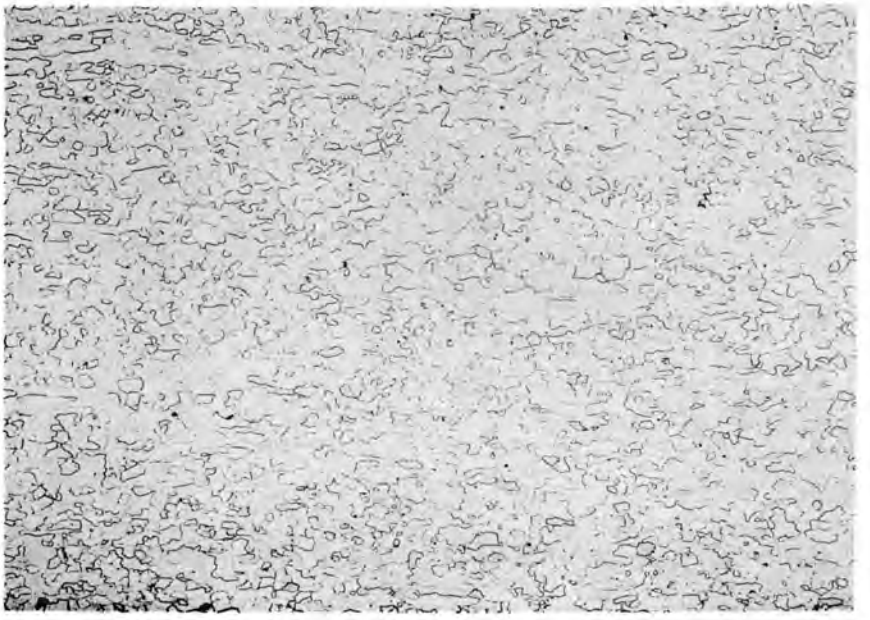

Figure 36 (Y-23131). Microstructure of 80% reduced powder-metallurgy niobium, annealed one hour at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

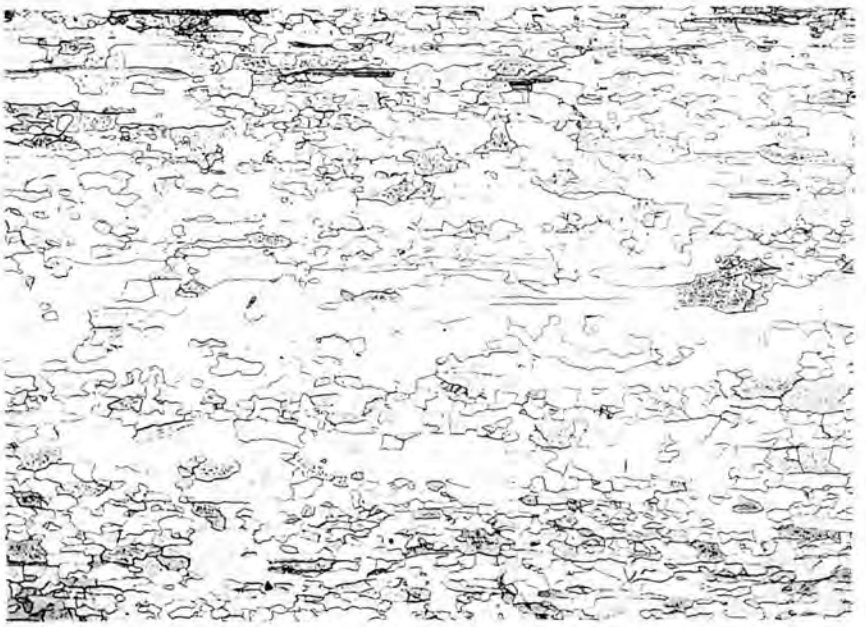

Figure 37 (Y-23126). Microstructure of 80% reduced arc-cast niobium, annealed one hour at 1200°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

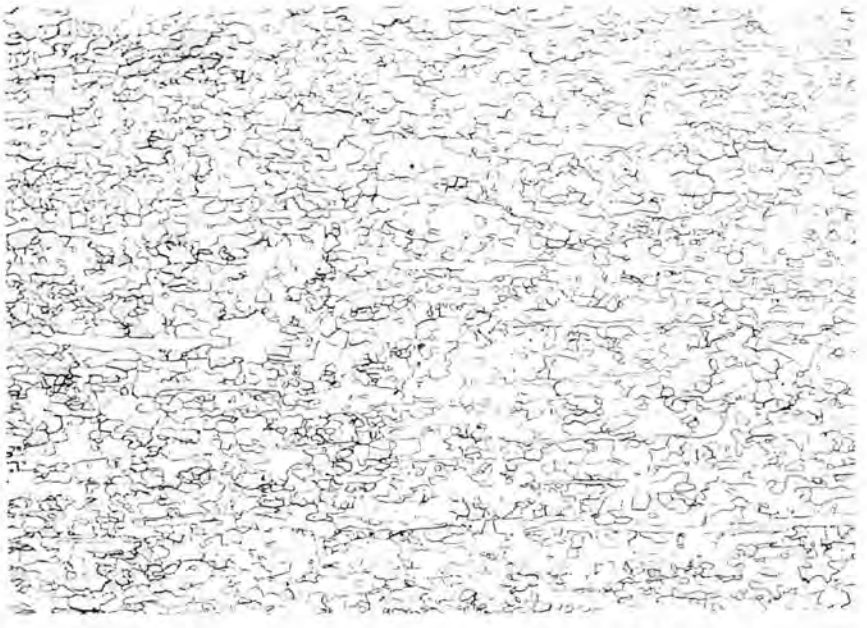

Figure 38 (Y-23130). Microstructure of 80% reduced powder-metallurgy niobium, annealed one hour at 1200°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

TABLE III

HARDNESSES (DPN) OF ISOTHERMALLY-ANNEALED SPECIMENS FOR SEVERAL ANNEALING TIMES

Annealing Temp. °C	Material and % Deformation	As Rolled	15 Min.	20 Min.	1 Hr.	2 Hr.	5 Hr.	10 Hr.
950	A-C Nb, 80	168	135	122	125	118	115	120
	A-C Nb, 90	178	140	128	125	125	114	110
	PM Nb, 80	157	146	134	125	118	117	116
	PM Nb, 90	165	144	128	128	110	114	110
1000	A-C Nb, 80	168	126	129	129	121	113	104
	A-C Nb, 90	178	132	132	127	134	107	107
	PM Nb, 80	157	143	141	121	113	115	99
	PM Nb, 90	165	134	130	116	122	124	112
1050	A-C Nb, 80	168	120	113	117	121	117	110
	A-C Nb, 90	178	124	127	119	119	112	114
	PM Nb, 80	157	124	118	103	117	116	110
	PM Nb, 90	165	119	121	117	126	121	118
1100	A-C Nb, 80	168	112	112	110	117	115	120
	A-C Nb, 90	178	125	119	115	123	121	122
	PM Nb, 80	157	125	110	109	123	109	130
	PM Nb, 90	165	128	123	119	127	129	129

softening is essentially complete after fifteen minutes at 1050 and 1100°C; additional annealing at the latter temperatures may actually lead to hardening by contamination.

Despite the over-all "scatter" of these data, the hardnesses of individual specimens of each series were found to be highly reproducible, both within a specimen itself, and between separate, nominally-identical specimens annealed concurrently. However, similar specimens from separate, nominally-identical annealing treatments yielded hardness values that varied by as much as twenty DPN hardness units. This indicates that at least part of the variation in the data in Table III is attributable to the contamination introduced during the vacuum-annealing treatment.

Figures 39 through 50 are photomicrographs of the 80 per cent cold-rolled materials after fifteen-minute, two-hour, and ten-hour anneals at 1000 and 1100°C. The two 1000°C series show the relatively early stages of the recrystallization process, while the two 1100°C series show later stages of recrystallization. Relation of these photomicrographs to the hardness data of Table III supports the hypothesis that the anomalies in hardness are due to solid-solution hardening by oxygen and/or nitrogen, for there are no anomalies in the metallographically-observed recrystallization processes at the two temperatures.

Unless very elaborate precautions are taken to insure non-contamination of specimens, it may be said that hardness measurements are an unsatisfactory method of following the course of recrystallization of niobium.

Figure 39 (Y-23142). Microstructure of 80% reduced arc-cast niobium, annealed 1/4 hour at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

Figure 40 (Y-23141). Microstructure of 80% reduced powder-metallurgy niobium, annealed 1/4 hour at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

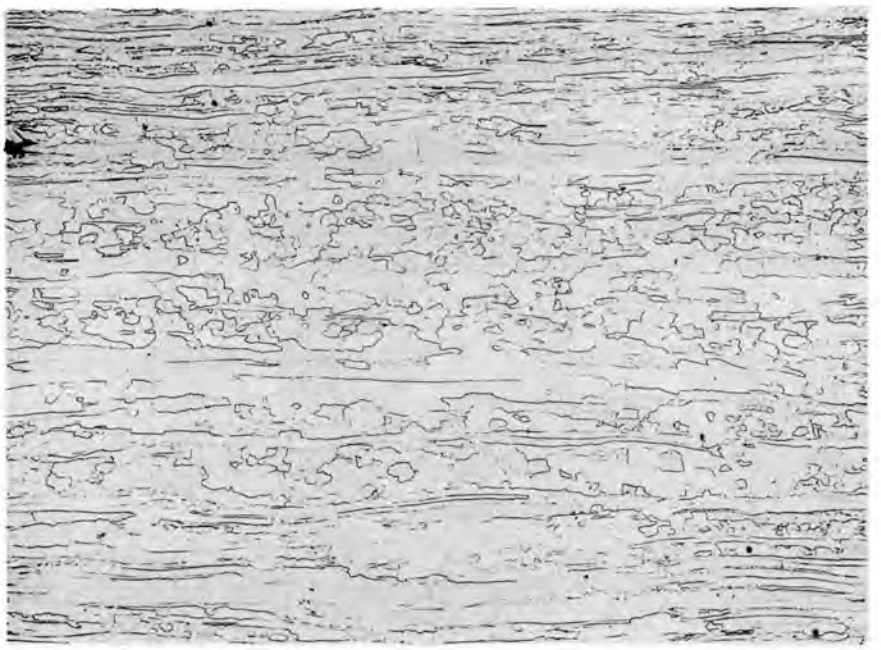

Figure 41 (Y-23138). Microstructure of 80% reduced arc-cast niobium, annealed two hours at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

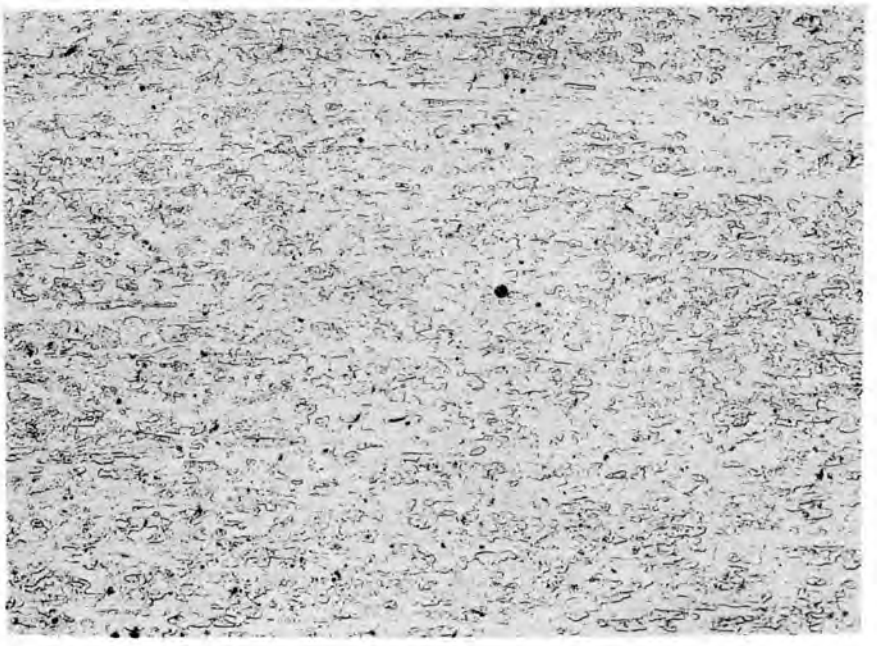

Figure 42 (Y-23137). Microstructure of 80% reduced powder-metallurgy niobium, annealed two hours at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

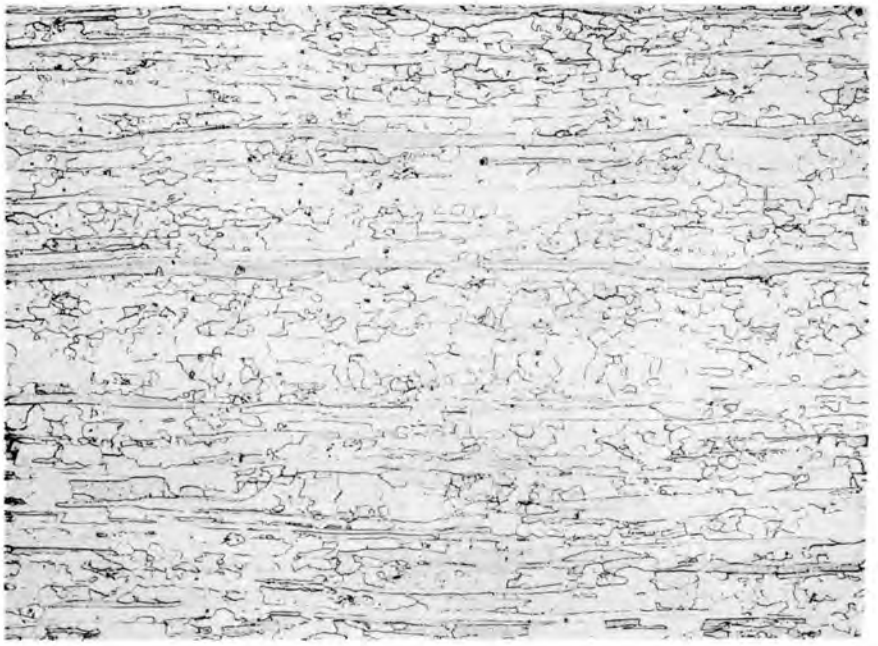

Figure 43 (Y-23140). Microstructure of 80% reduced arc-cast niobium, annealed ten hours at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

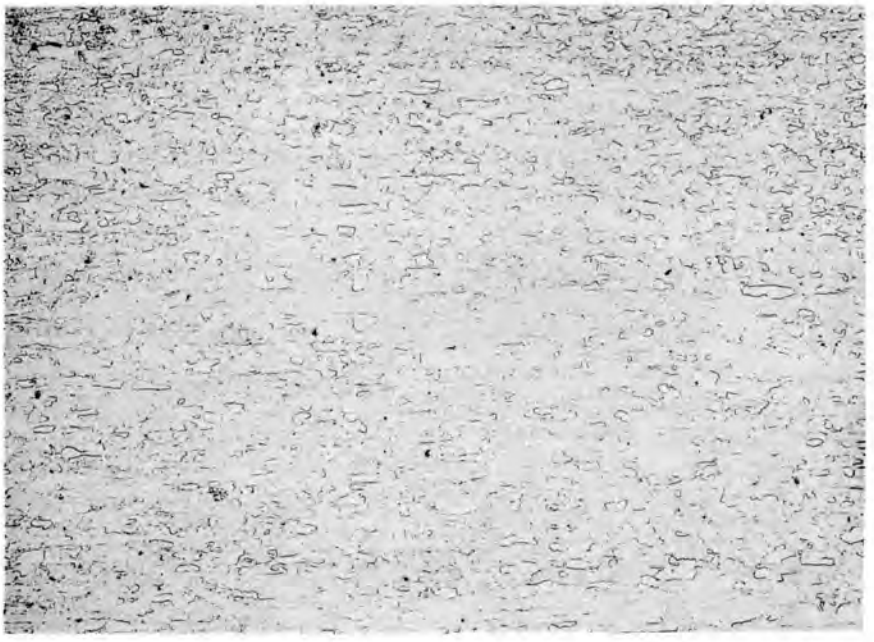

Figure 44 (Y-23139). Microstructure of 80% reduced powder-metallurgy niobium, annealed ten hours at 1000°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

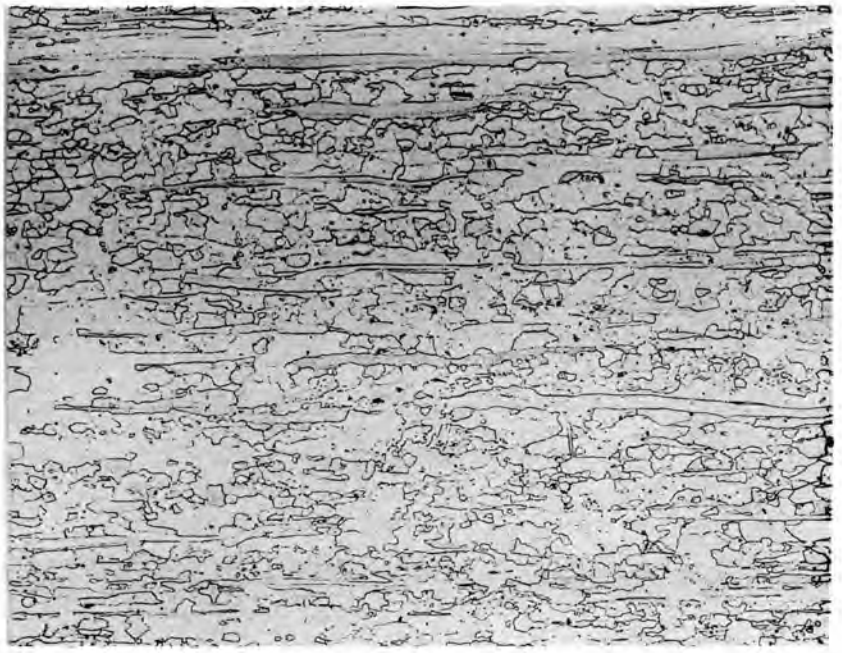

Figure 45 (Y-23159). Microstructure of 80% reduced arc-cast niobium, annealed 1/4 hour at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

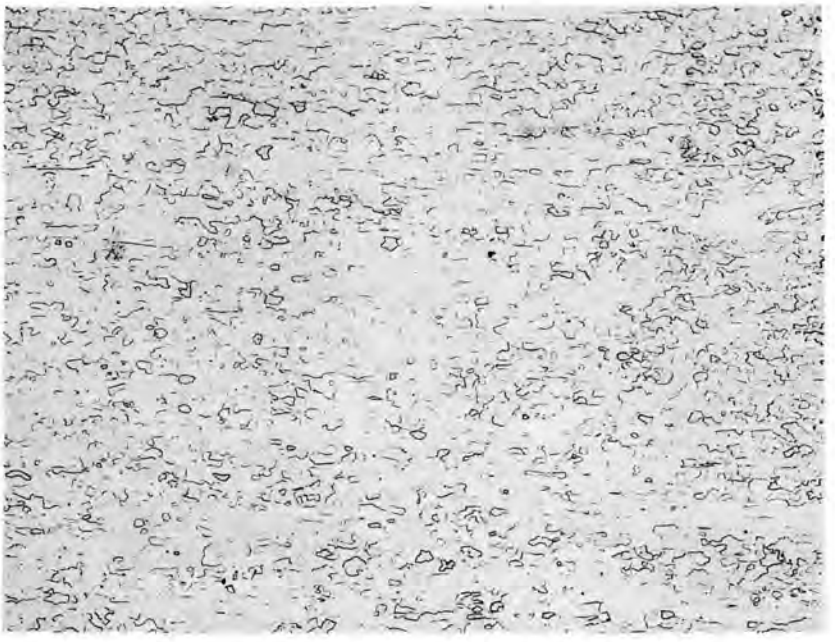

Figure 46 (Y-23157). Microstructure of 80% reduced powder-metallurgy niobium, annealed 1/4 hour at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

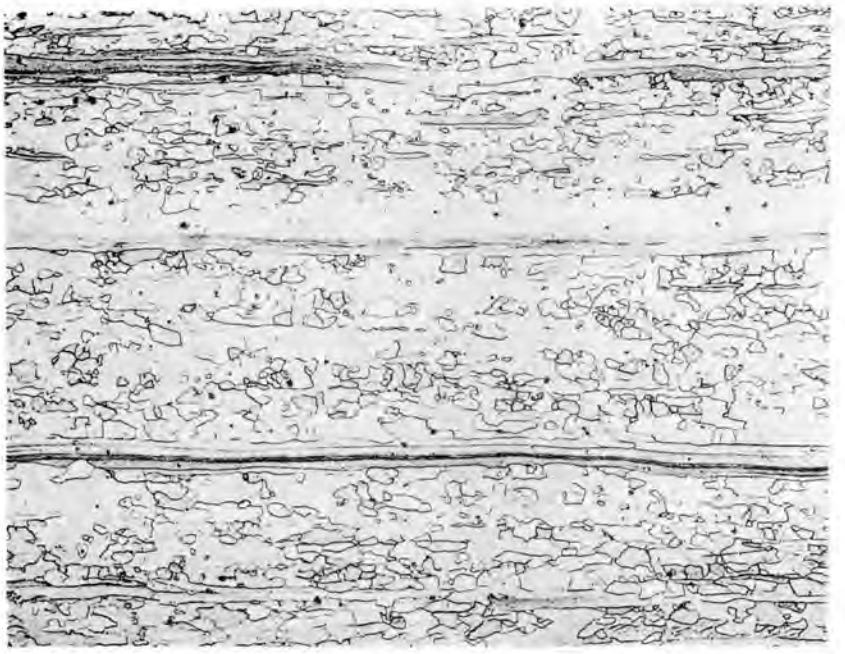

Figure 47 (Y-23158). Microstructure of 80% reduced arc-cast niobium, annealed two hours at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

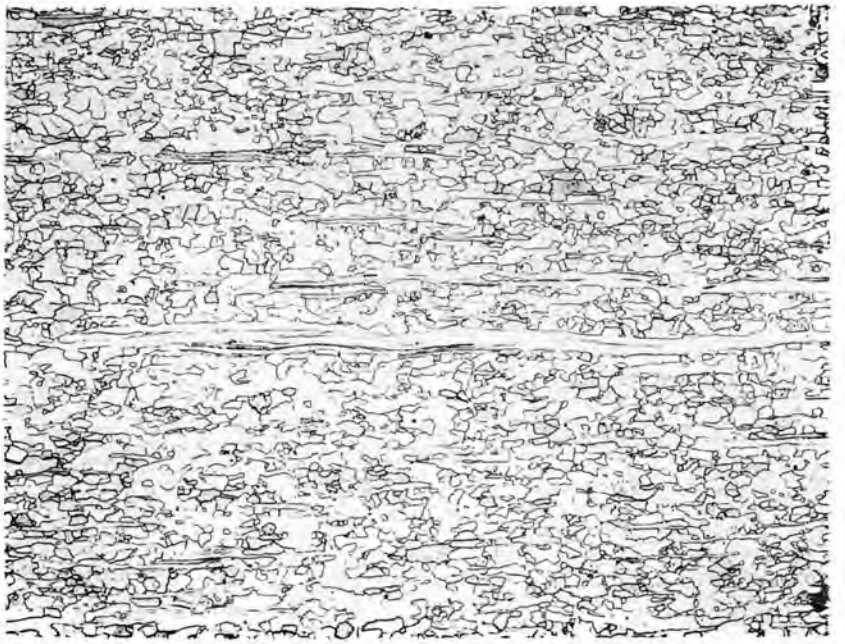

Figure 48 (Y-23156). Microstructure of 80% reduced powder-metallurgy niobium, annealed two hours at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

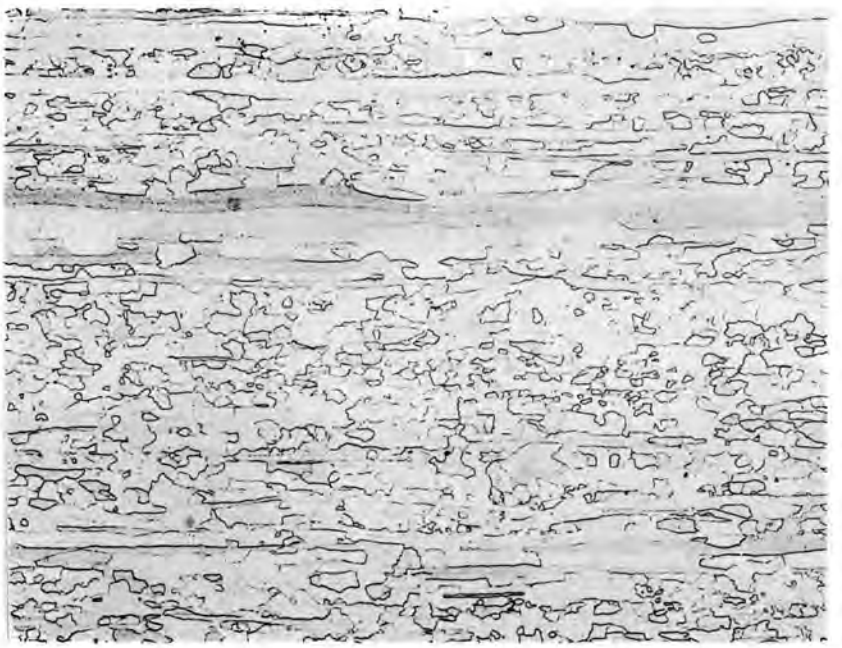

Figure 49 (Y-23155). Microstructure of 80% reduced arc-cast niobium, annealed ten hours at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

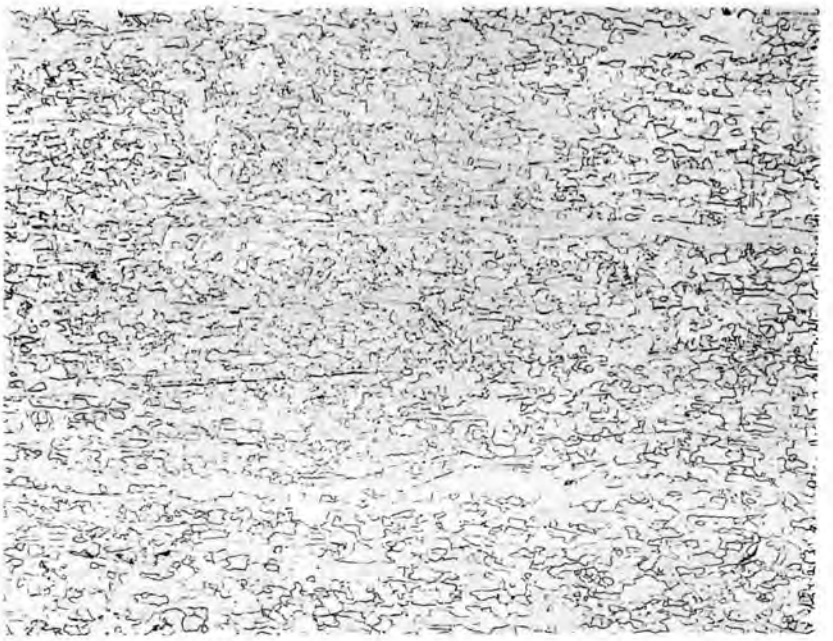

Figure 50 (Y-23154). Microstructure of 80% reduced powder-metallurgy niobium, annealed ten hours at 1100°C. Etchant - 25 HF, 50 H_2O, 5 HNO_3, 10 H_2SO_4. 100X.

CHAPTER VII

CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

1. The recrystallization behavior of niobium is in agreement with classical recrystallization theory in that recrystallization is promoted by: a) increasing annealing temperature, b) increasing annealing time, and c) increasing "prior deformation."

2. Drawings are presented which show, semi-quantitatively, the relative effects of annealing time, annealing temperature, and prior deformation on the initiation and termination of recrystallization.

3. Graphs showing the effects of one-hour anneals on the room temperature tensile properties of cold-rolled niobium are presented. These indicate that mechanical properties begin to recover at approximately 800°C. These properties are essentially completely recovered at 1000°C in the 80 and 90 per cent rolled materials, but not in the 20 and 50 per cent rolled materials, which are still in the early stages of recrystallization.

4. Although otherwise inconclusive, hardness data are presented which indicate that minor degrees of contamination have a marked effect on the hardness of niobium and that hardness is, therefore, a poor criterion for determining recrystallization in niobium.

5. Photomicrographs showing 80 per cent cold-rolled powder-metallurgy and arc-cast niobium after one-hour anneals at room tempera-

ture (no anneal), 900, 1000, 1100, and 1200°C, and after fifteen-minute, two-hour, and ten-hour anneals at 1000 and 1100°C are presented. These show the recrystallizing powder-metallurgy niobium to have a finer grain size than the recrystallizing arc-cast niobium after identical treatment.

6. Other than the recrystallized grain size, no significant differences were noted in the annealing behavior of arc-cast and powder-metallurgy niobium. The room temperature tensile properties of the two materials also exhibit no significant differences.

B. Recommendations

1. It is recommended that the temperatures of initiation and termination of recrystallization of cold-rolled niobium be determined more accurately at annealing times of one, two, and five hours. The results of this investigation give a general picture of the recrystallization of niobium; however, it would be advantageous to investigate the behavior more thoroughly for practical annealing times of one to five hours, using somewhat thicker specimens to enable a better analysis of grain size.

2. It is recommended that the effect of contamination by oxygen and nitrogen on the annealing behavior of cold-rolled niobium be investigated. It is well known that these gases have a marked effect on the hardness and ductility of niobium; their effects on the annealing behavior should also be determined. Samples containing controlled amounts of oxygen or nitrogen could be prepared by heating them in a partial vacuum of one of the two gases.

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APPENDIX

APPENDIX

EQUIPMENT

A. Rolling Equipment

Two relatively small rolling mills were used for the cold-rolling operations. The larger of the two was a 6-inch diameter by 10-inch two-high mill; this was used where heavy reductions were desired. The smaller rolling mill was a 3-inch diameter by 5-inch two-high mill; this was used for final passes in order that thickness might be controlled more accurately.

B. Annealing Equipment

1. High-temperature vacuum-annealing furnace. The furnace shown in Figure 51 was used for almost all annealing treatments. Except for the hot-zone, it is an all-glass system. A McDanel high-temperature combustion tube (mullite) passes through the hot-zone of the Burrell Glo-bar furnace. A pyrex-to-mullite seal was affected at each end of this tube. A GF-25W, glass, fractionating diffusion pump, made by Distillation Products Industries, was used to evacuate the system. Figure 52 presents a close-up view of the diffusion pump. A Cenco-Hypervac-25 mechanical vacuum pump was used as the forepump and as the roughing pump for the system during initial evacuation.

In order that the specimens might be maintained under vacuum at all times and also annealed for exactly the prescribed time, an

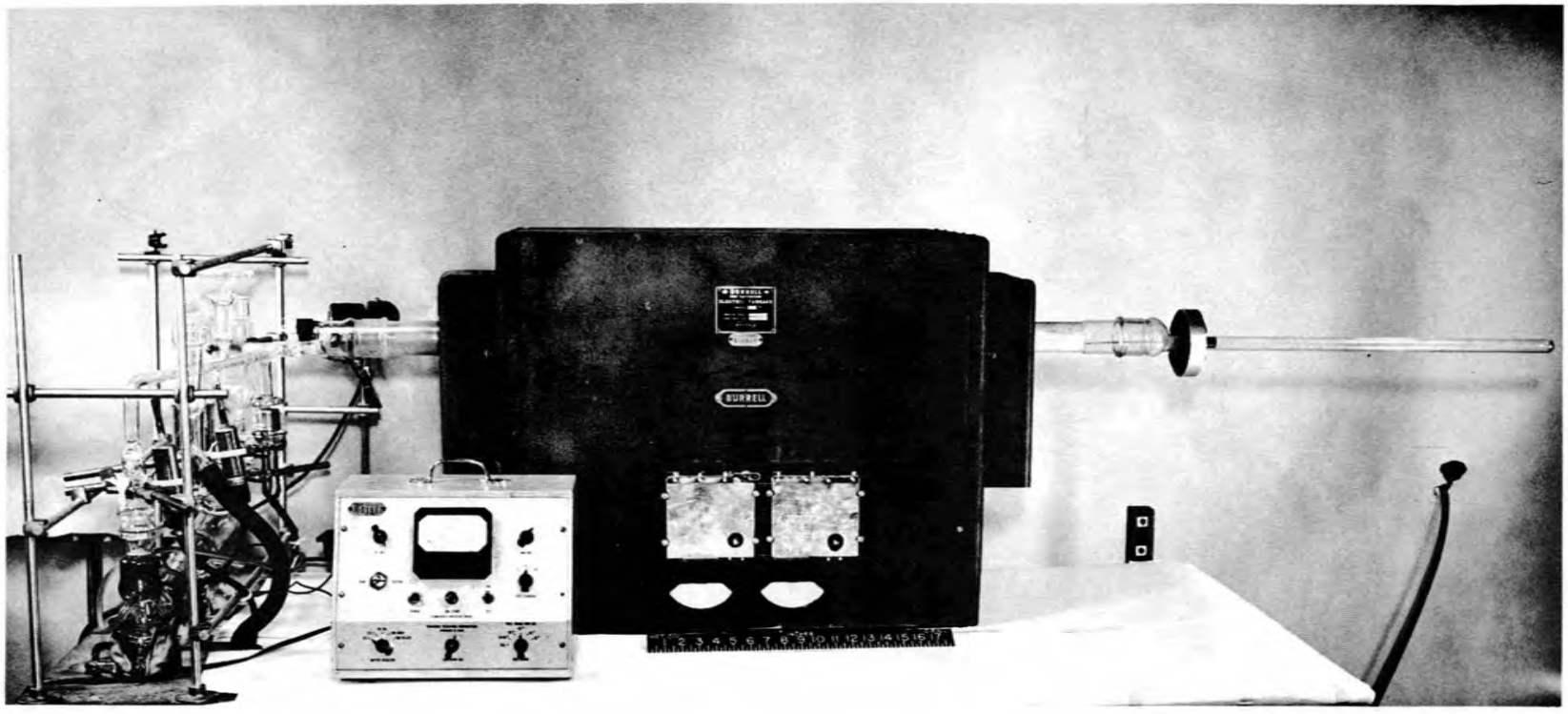

Figure 51 (Y-23050). Over-all view of vacuum heat-treating furnace.

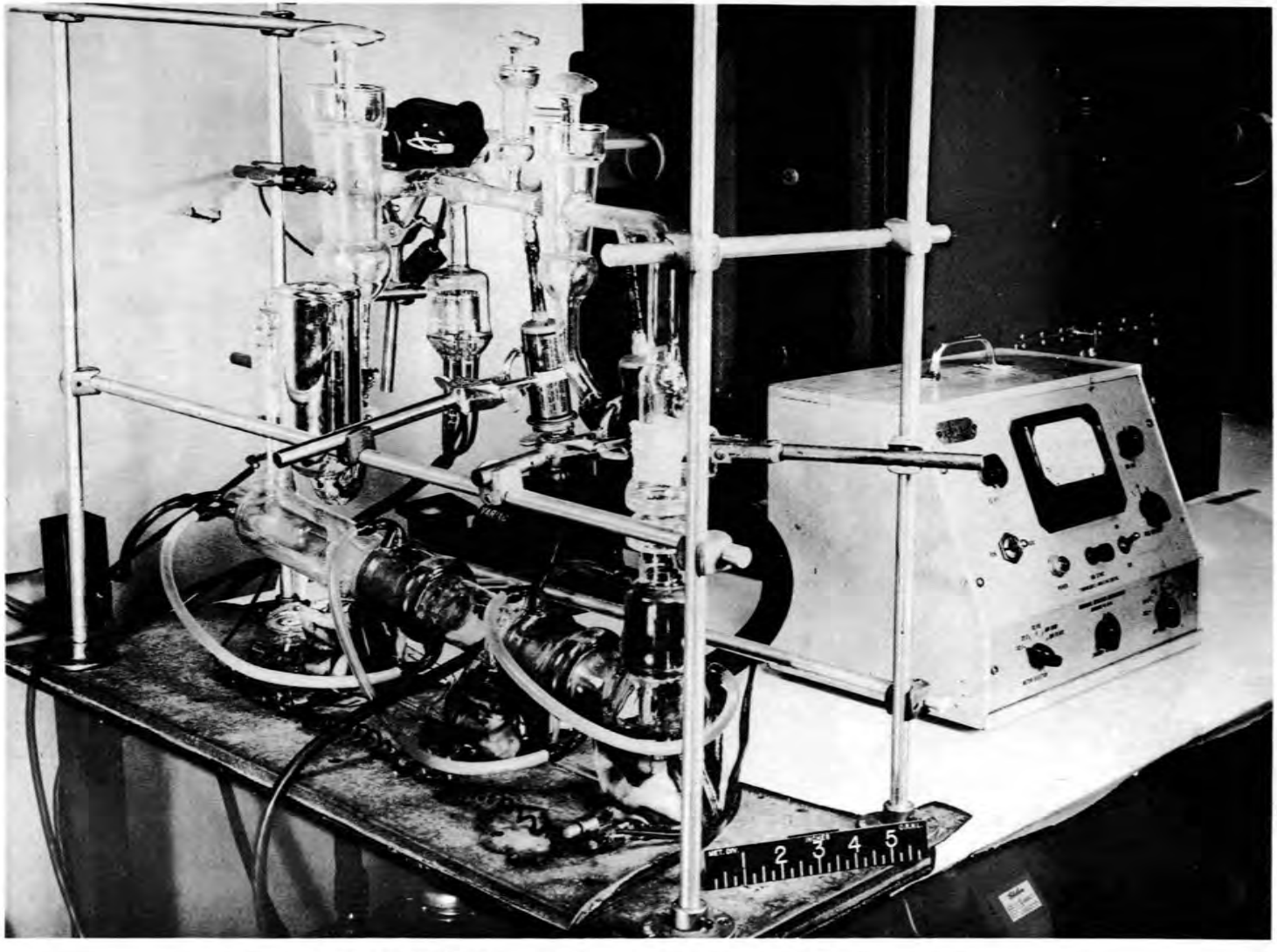

Figure 52(Y-23051). Close-up view of vacuum diffusion pump.

unusual method of loading the furnace was necessary. The furnace portion of the system was first opened to the atmosphere, then the specimens were laid in a molybdenum boat to which an iron "slug" was attached through a rigid molybdenum wire. This wire was long enough to reach from a cold-zone in the system to the hot-zone. The boat was then placed in the cold-zone of the furnace, and the furnace tube sealed by a glass cap which provided a tubular space for the molybdenum wire and iron slug. This cap can be seen at the far right on Figure 51. A vacuum was pulled on the system, and when equilibrium conditions were attained (approximately three-fourths of an hour) the specimens were inserted into the hot-zone by magnetic manipulation of the iron slug.

As with all vacuum systems, the degree of vacuum attainable was a function of the temperature of the hot-zone, the vacuum becoming poorer with increasing temperature. Figure 53 shows the operating characteristics of this system. This curve is not "absolute" in as much as the vacuum attained during various anneals at any one temperature varied considerably (by as much as half an order of magnitude). This variation was caused by variables such as surface area of the load and annealing time.

The pressure in the system was read by a National Research Corporation thermocouple and ionization gauge using an NRC Type 507 ionization tube. This tube was attached immediately adjacent to the hot-zone of the furnace.

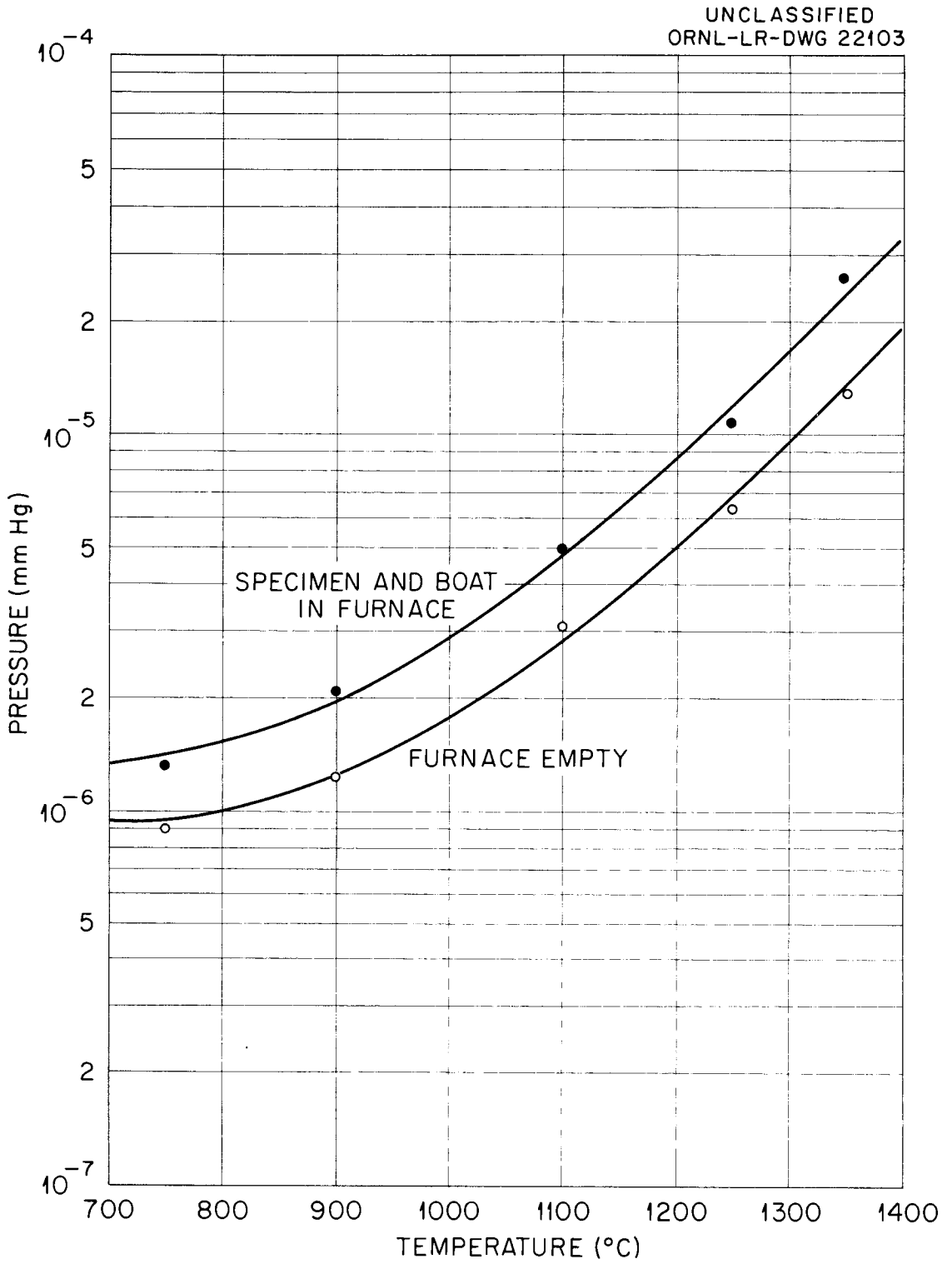

Figure 53. Temperature-pressure relationship in vacuum annealing furnace.

As can be seen in Figure 52, a liquid-nitrogen cold trap was included in the system. It was found that its effect on the vacuum was not significant, but it was, nevertheless, used during all anneals to prevent back-diffusion of the Dow-Corning Type 703 silicone diffusion pump oil.

Temperature control of this furnace was maintained by a Leeds and Northrup two-action D.A.T. (Duration Adjustment Type) Speedomax Controller. A Pt/Pt-10% Rh thermocouple was used as the control thermocouple. The accuracy of control was checked periodically with a calibrated Pt/Pt-10% Rh thermocouple which was led to a Rubicon Model 2732 portable potentiometer for measurement. Total temperature variation, due to cycling and thermocouple inaccuracy, was less than ± 5°C at 1000°C, and less than ± 12°C at 1350°C, through the six-inch long hot-zone of the furnace.

2. Short-time annealing furnace. In order to extend the range of annealing time covered by this investigation, a short-time annealing furnace was designed and built. A schematic diagram of this furnace is presented in Figure 54. A series of small specimens were "lined up" in the entry tube, the system closed to the atmosphere and opened to a vacuum system identical to that described in the previous section. After equilibrium vacuum was established, the first specimen was "pushed" off the lip of the entry tube. This fell onto a funnel-shaped hearth of boron nitride which was in the center of the hot-zone of the furnace. The specimen was "trapped" there until the mullite "plug" was raised; it then fell through the opening in the hearth, past a liquid

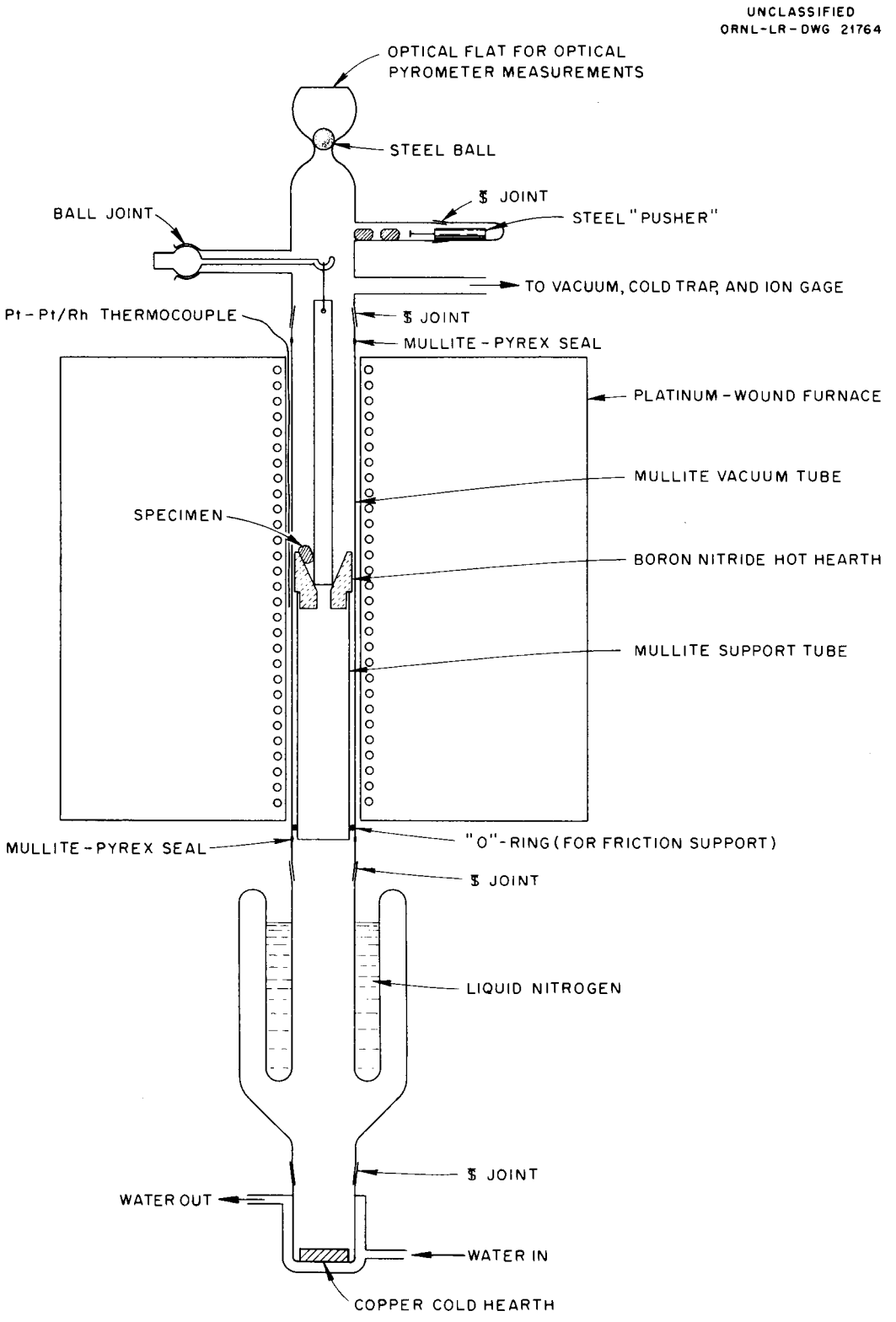

Figure 54. Schematic diagram of rapid-heat, rapid-quench apparatus.

nitrogen cold trap and onto a water-cooled copper disc.

The annealing temperatures were controlled by a Leeds and Northrup Micromax controller, using a Pt/Pt-10% Rh thermocouple. The absolute value at which the controller was set was determined by optical pyrometer measurement of the temperature of the hot hearth. There was approximately a 50°C temperature drop from the thermocouple to the hot hearth. The actual annealing temperatures were estimated to be ± 15°C of the desired temperature.

The annealing times were measured with a stop-watch. The stop-watch was started when the specimen became essentially invisible (attained "black-body" condition); this was within two to four seconds after contact with the hot hearth. The specimen was released approximately two seconds before the prescribed annealing time had elapsed to allow for a slight delay in cooling.

The only difficulty encountered in this system was associated with the unavoidable pressure rise resulting from the rapid outgassing of the rapidly heated specimens. Typical time-pressure curves for various temperatures are presented in Figure 55.

Generally, however, this was considered a satisfactory apparatus. It was felt that even shorter annealing times than thirty-six seconds could have been obtained with reasonable accuracy, had they been desired.

C. X-Ray Equipment

The x-ray equipment consisted of a precession x-ray camera adapted for

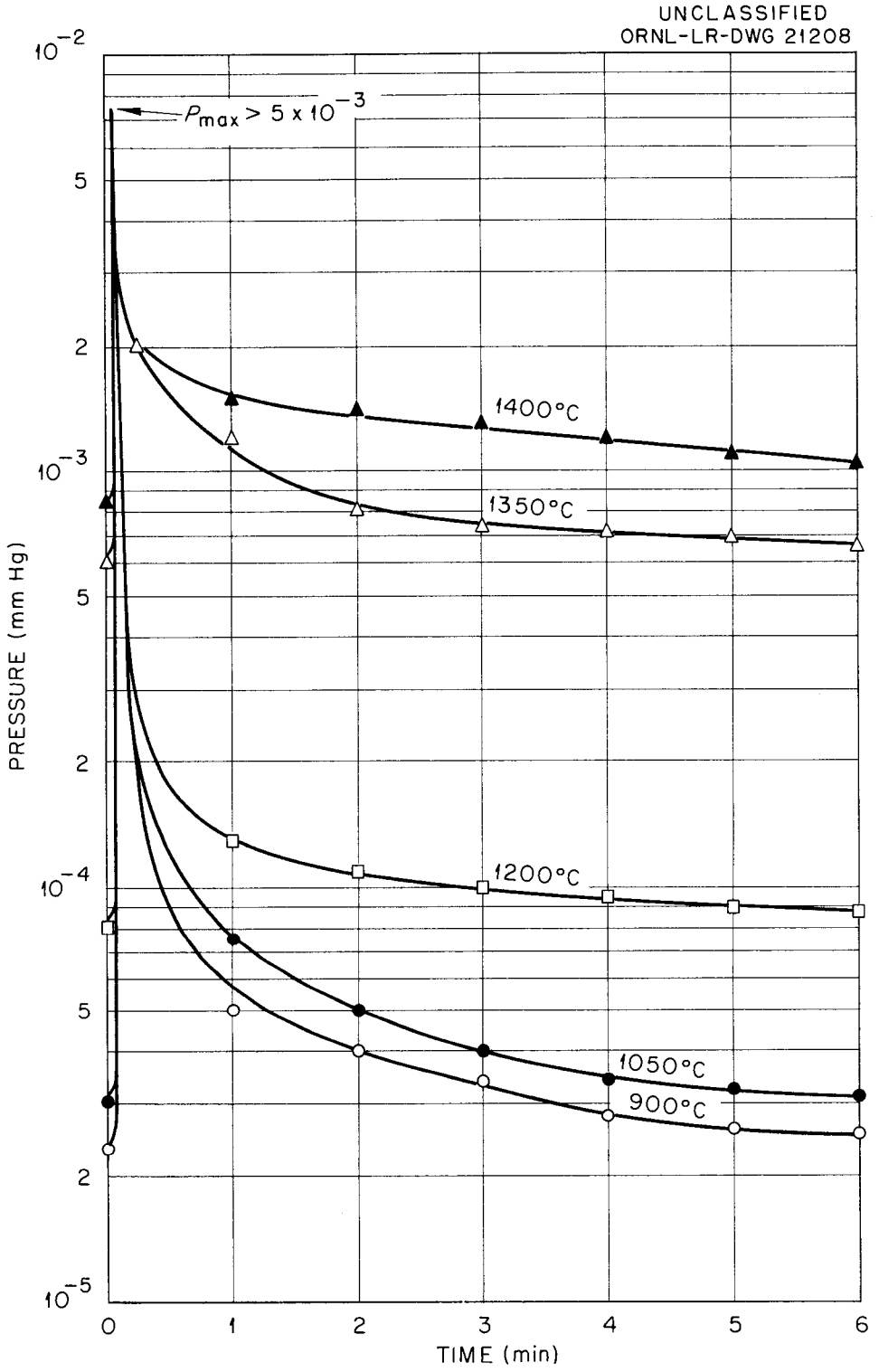

Figure 55 (Y-22801). Typical time-pressure curves for 6-minute anneals at various temperatures.

the Laue transmission technique. Molybdenum-K radiation was used, without filtering, for all specimens. The tube was operated at 55 kilovolts and 15 milliamperes. A detailed description of the x-ray technique is given in the chapter on experimental procedure.

D. Metallographic Equipment and Procedure

The specimens were mounted in red Bakelite. They were then given preliminary surfacing on 320, 400, 600, 2/0, and 4/0 papers, in that order. The next step consisted of a short polish on a silk-covered wheel, using 0.3 micron aluminum oxide (Linde A). From there, the specimens were moved to a nap-covered Syntron Vibro-Polisher where 0.3 micron aluminum oxide was again used as abrasive. The final polishing was performed on another Syntron Vibro-Polisher, with 0.1 micron aluminum oxide (Linde B) as the abrasive. The specimens were etched with a solution of 50 parts H_2O, 25 parts HF, 10 parts H_2SO_4, and 5 parts HNO_3. It was found that an excellent macro-etch could be obtained by increasing the HF content to 35 parts and the HNO_3 content to approximately 10 parts.

All photomicrographs were made with a Bausch and Lomb Research Metallograph using bright field illumination.

E. Mechanical Testing Equipment

Hardness data were taken with a Wilson Tukon Micro-Hardness Tester using a 10.25 objective and a 500-gram load. The indenter was

a 136° diamond pyramid cone. At least five impressions were made on each specimen; these were averaged and the hardness reported in DPN (Diamond Pyramid Number) hardness units.

Two Baldwin Hydraulic Tensile Testing Machines (12,000 lb., and 120,000 lb. maximum load) were used to obtain the room temperature tensile data. The five-inch long specimens were held by Templin flat-jaw grips. A type B-3M extensometer having a one-inch gage length was used to determine the 0.2% offset yield point. The strain rate (head rate) of the tests was 0.05 inches per minute.

VITA

The author, John Patrick Page, was born on July 12, 1932, in Belgrade, Yugoslavia. In 1941, he and his parents evacuated to the United States, where they lived in Washington, D. C., from 1941 to 1943, and then in Salt Lake City, Utah, from 1943 until 1955. He graduated from Granite High School (Salt Lake City) in 1950. He then attended Cornell University, Ithaca, New York, graduating in 1955 with a degree of Bachelor of Metallurgical Engineering. He was employed at the Oak Ridge National Laboratory from 1955 until 1957. He is about to enter the United States Air Force as a Second Lieutenant. In July, 1956, he married Mrs. Kathryn Lowry McHargue.

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