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# Growth of gadolinium single crystals

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# Growth of gadolinium single crystals

## **Abstract**

Single crystals have become vitally important to many areas of research in metallurgy, engineering, physics and chemistry. With the introduction of theories about dislocations and their movement during plastic deformation of metals came a need for single crystals to test these theories. The fact that theoretical yield strengths are almost 1000 times higher than those normally observed needs to be explained to satisfy the curiosity of the metallurgist. The explanation may also have very practical applications. It is interesting to note that whiskers, metallic single crystals in tiny hair-like shape, do have strengths approaching the theoretical. Dr. Halden (13), manager of ceramics research at Stanford Research Institute, reports that techniques are being developed to use this strength in composites and self-bonded structures.

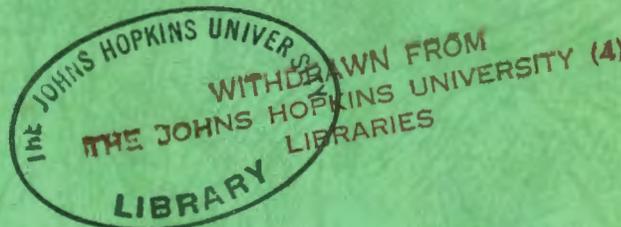
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**IOWA STATE UNIVERSITY**

GROWTH OF GADOLINIUM  
SINGLE CRYSTALS

by

Idelle Marietta Peterson and Morton Smutz

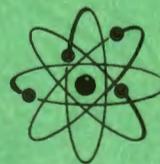
**AMES LABORATORY**

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**RESEARCH AND  
DEVELOPMENT  
REPORT**

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**U.S.A.E.C.**



IS-977<sup>✓</sup>

Engineering and Equipment (UC-38)  
TID 4500 October 1, 1964

UNITED STATES ATOMIC ENERGY COMMISSION  
Research and Development Report

GROWTH OF GADOLINIUM  
SINGLE CRYSTALS

by

Idelle Marietta Peterson and Morton Smutz

July, 1964

Ames Laboratory  
at  
Iowa State University of Science and Technology  
F. H. Spedding, Director  
Contract W-7405 eng-82

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Washington 25, D. C.

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IS-977

GROWTH OF GADOLINIUM SINGLE CRYSTALS\*

Idelle Marietta Peterson and Morton Smutz

ABSTRACT

Single crystals have become vitally important to many areas of research in metallurgy, engineering, physics and chemistry. With the introduction of theories about dislocations and their movement during plastic deformation of metals came a need for single crystals to test these theories. The fact that theoretical yield strengths are almost 1000 times higher than those normally observed needs to be explained to satisfy the curiosity of the metallurgist. The explanation may also have very practical applications. It is interesting to note that whiskers, metallic single crystals in tiny hair-like shape, do have strengths approaching the theoretical. Dr. Halden (13), manager of ceramics research at Stanford Research Institute, reports that techniques are being developed to use this strength in composites and self-bonded structures.

The first task in many solid state physics studies is to produce single crystals because this permits one to measure physical properties without taking into consideration the complexities introduced by the presence of grain boundaries and the multiplicity of crystal orientations. Due to the orderly arrangement of atoms in a single crystal and to the

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\* This report is based on an M. S. thesis submitted by Idelle Marietta Peterson to Iowa State University, Ames, Iowa, July, 1964.

fact that the atoms are different distances apart in different crystallographic directions, physical properties are anisotropic. Unique measurements of these properties can be obtained only from single crystals.

Chemists use single crystals for research into molecular structure. Single crystals of the rare earths may prove to be especially illuminating in this area due to their progressive difference of one electron in the 4f shell.

Examples of developments by physicists and chemists which were dependent on single crystals are the use of germanium and silicon crystals in semiconductors, the use of quartz crystals for frequency control in radio transmission, and lately, the use of ruby crystals for lasers and masers. Just as the single crystals were important to discovering these uses, the uses stimulated improvements in growth techniques. Undoubtedly this cycle will be repeated for many other materials.

The purpose of this study was to develop a method for growing single crystals of gadolinium which might also be applicable to the other rare earths. The development of the ion exchange process for separating the rare earths and the fluoride reduction process for preparation of pure rare earth metals at the Ames Laboratory made enough metal available for such a study. Gadolinium was chosen because it does not oxidize at ordinary temperatures and it was easy to machine. Also, it is representative of many of the rare earths in that it has a high temperature phase transformation. Gadolinium changes from a hexagonal form to a face-centered-cubic form at 1264°C. (20).

## GENERAL METHODS OF CRYSTAL GROWTH

A good review of crystal growing techniques up to 1959 is given by Honeycombe (14). These techniques fall into four general categories depending on the physical state in which the atoms or molecules are transported to the growth surface: crystallization from solution, solidification of a pure melt, condensation of a vapor, and growth in the solid state.

Growth from the liquid is most commonly used and it includes several techniques: Czochralski (8), Bridgeman (4), and zone-melting. The Czochralski method, or crystal pulling, uses a single crystal seed dipped into a crucible of molten metal and slowly withdrawn. In the Bridgeman method a crucible of molten metal is slowly cooled and solidified from one end, either by moving the crucible out of a hot furnace into a cooler region or by moving the furnace relative to a stationary crucible. These methods are not satisfactory for growing gadolinium single crystals because the transformation from the hcp to the fcc cubic structure would damage or destroy any crystal grown from the melt due to density changes accompanying the phase change.

Pfann (18) gives a very complete discussion of zone melting. A narrow hot zone slowly traverses a crucible of metal or a narrow hot zone is moved along a vertical rod. The temperature may or may not be above the melting point. The same objection as for the Czochralski and Bridgeman

methods would apply to this method using temperatures above the melting point of gadolinium.

Growth in the solid state includes the strain anneal method and the phase transformation method. The strain anneal method was first used by Carpenter and Elam (6) in 1921 to grow single crystals of aluminum. Its main advantage for gadolinium is that single crystals can be grown below the phase transformation temperature.

## PRINCIPLES OF THE STRAIN ANNEAL TECHNIQUE

The strain anneal technique for growing single crystals consists of straining and annealing just as the name implies. The stresses left in the metal from the straining process and grain boundary surface energy produce the driving force for the nucleation and growth of new crystal grains during the subsequent anneal. Hopefully, conditions can be chosen which will allow only one grain to grow at the expense of the rest.

The factors which influence the final grain size are amount of strain, penultimate grain size (grain size before straining), the uniformity of the penultimate structure, the time-temperature annealing program, degree of preferred orientation in the penultimate structure, and amount and type of impurities in the metal. The effects of these factors are complex. For instance, the grain size and uniformity, the degree of preferred orientation, and the impurities will all affect the residual stress after straining. Because of this complexity only qualitative rules have emerged from the research of many investigators. Burke and Turnbull (5) give a very good review of this research.

In metallurgy texts, i.e. Samans (19), one often finds recrystallization diagrams. These are three dimensional surfaces--final grain size is plotted against strain and temperature of anneal. Actually these diagrams are valid

for only the particular metal composition, penultimate grain size, and degree of preferred orientation of the metal used in obtaining the experimental data. To be complete, the values of each of these other variables should be recorded along with these diagrams since the surface is dependent upon them. All diagrams of this kind have the same general shape, however, with the maximum grain size observed at low strains and high annealing temperatures. In the strain anneal process for growing single crystals one therefore wants to operate at these strains and temperatures.

As indicated on these recrystallization diagrams, a critical strain is found where the crystal size is maximized, often in the range zero to five per cent. Honeycombe (14) recommends the use of a tapered tensile specimen to obtain this critical strain. However, the originator of this idea seems to be Chappell (7) who used it in his 1914 study of the recrystallization of iron. Although other investigators (10, 15) report using tapered rods to determine the critical strain and may have thought of it independently, Chappell seems to have been the first.

Using a tapered rod is an excellent idea because it permits one to find the best value of strain with an economy of material. Also, of even greater importance, it isolates the effect of strain. All of the parts of the tapered specimen, coming from one piece of metal, are much more likely

to have one common, metallurgical history than several specimens. The impurities are likely to vary much less throughout the tapered specimen than among several different specimens.

Impurities have been both blamed for the failure of the strain anneal technique and praised for contributing to its success. For instance, Dunn and Nonken (9) say, "It is well known that the tendency toward normal grain growth increases with approach to 100 per cent purity, or conversely, that impurities can be used to retard normal grain growth...It is almost certain, therefore, that some impurities are essential in our method of growing single crystals of silicon iron." On the other hand, Fisher (10) reports that impurities provided the sites of nucleation in his attempts to grow single crystals of alpha-uranium. The difference seems to be due to the type and amount of impurity, but so far no one has explained how to find the optimum amount and type except by trial and error.

Williamson and Smallman (22) report that the rate of growth is the greatest when the orientation of the growing crystal is rotated from 30 to 40 degrees with respect to the original orientation if there is any preferred orientation in the material. Several confirmations and no contradictions have been found to this report (2, 3, 5).

Since the grain boundary surface tension is one of the driving forces of growth, it is reasonable to expect that

a small penultimate grain size might be best.

These were the factors that were considered in choosing the conditions used in this study. However, they were only rough guidelines and no attempt was made to optimize conditions.

## PREVIOUS WORK

Only two reports of attempts to grow single crystals of gadolinium could be found in the literature. Graham (12) used a single crystal of gadolinium grown by the Czochralski method to study the magnetocrystalline anisotropy. It was grown in an argon atmosphere using a tantalum crucible. The crystal was actually grown by J. W. Rutter and T. A. Sawyer of the General Electric Research Laboratory and Graham used it for measuring its magnetic properties. Graham did not mention the size of the crystal but he did say that the gadolinium contained less than 0.3 per cent metallic impurities and a plate-like precipitate amounting to a few per cent by volume was visible metallographically. He suggested that this precipitate probably was gadolinium oxide.

Nigh (17) also needed single crystals of gadolinium for measurements of its magnetic and electrical properties. The technique developed for growing them was to melt the sample under argon in an arc furnace which had a water-cooled copper hearth and a nonconsumable tungsten electrode. The large grains ranging in size from five mm. to three cm. in diameter grew during annealing periods 60 hours in length. The annealing program was 12 hours at 1050°C., 1100°C., and 1200°C., respectively, in a 25°/cm. gradient. Finally the anneal ended with 12 hours more at 1225°C. without a temperature gradient. This method is essentially a strain

anneal method. The stresses, instead of being introduced by plastic deformation, are caused by the high thermal gradient in the metal during the arc-melting operation.

Since the main problems one encounters in trying to grow gadolinium single crystals are due to its high melting point, the change from hcp structure to fcc just below the melting point, and its reactive nature, it is interesting to note how single crystals of other metals with these characteristics are grown. Iron and uranium also undergo solid state phase transformations. In the case of both of these, the strain anneal technique has been most successful in producing single crystals. Talbot (21) grew single crystals of alpha-iron by a two hour anneal at 880°C. (after raising to temperature in four hours) preceded by a critical tensile strain of three per cent. Abowitz, Hughes, and Barton (1) have designed a furnace with a strong thermal gradient (350 °C./in.) for growing single crystals of iron after straining the specimens two per cent. Fisher (10) determined that the critical tensile strain was 1.0 to 1.2 per cent for uranium with a tapered tensile specimen and produced large grains by annealing just below the alpha-beta transformation temperature. Langeron and Lehr (16) used the phase transformation volume change to produce the strain in zirconium and then annealed for a long time between 500°C. and 800°C.

Dunn and Nonken (9) of the General Electric Co. were

very successful in growing oriented single crystals of silicon iron sheet. Their general method consisted of cold-rolling and annealing in hydrogen to produce a fine, uniform grain size. After critically straining 2.5 per cent in tension they slowly moved the specimens through a slit in a water-cooled, copper jacket into a furnace at 1750°F. at a rate of 1/4 to 1/2 inch per hour. The gradient was about 1800°F. per inch. The most interesting and novel idea in their work was the way they obtained a desired orientation. This is their description of how they did it:

- "1. One or more crystals are obtained in the end of a sheet (or strip) by placing it into the hot portion of a furnace with a high temperature gradient.
2. The sheet is removed and one crystal is selected for the seed crystal. It must be adjacent to the matrix.
3. A transmission Laue pattern of the seed crystal is obtained.
4. The orientation of the seed crystal is obtained from the Laue-gram and the axes and angles of rotation are determined with the aid of a Wulff net.
5. The unwanted crystals are cut away leaving the seed crystal connected to the strip by a narrow neck.
6. The seed crystal is clamped in the crystal reorienting apparatus and is changed to the desired orientation with respect to the sheet, by bending the neck of the specimen while at red heat.
7. The seed crystal in the new orientation is then made to grow and replace the matrix of the entire sheet by passing the sheet slowly through the hot zone of a high-temperature-gradient furnace."

## EQUIPMENT AND PROCEDURE

Material used

Vacuum distilled gadolinium of 99.9 per cent purity was obtained from Dr. A. H. Daane's group within the Ames Laboratory. After distillation, the metal was arc-melted into ingots from which cylinders were cut. Since purity and grain size of the starting material are important variables in strain anneal crystal growth, they were measured. Table I shows the results of the spectrographic analyses for metallic impurities. A fluorine analysis indicated 800 ppm. The grain size was 800 grains per square inch, rounded off to the nearest 100.

Determination of tensile properties

A tensile specimen with a two inch gage length 0.320 inches in diameter was machined (similar to the tapered specimen of Figure 1 except that it had a constant diameter). It was strained in a Model FS-10 Universal Screw Power Testing Machine (10,000 lbs. capacity) at a strain rate of 0.02 inches per minute until it fractured. The total extension was measured by an extensometer, Model DC-5 DN-10-20 made by American Machine and Metals, Inc. The load and extension were automatically recorded. The stress-strain curve obtained is shown in Figure 2.

The ultimate strength was  $19.4 \times 10^3$  psi at an elongation of 20.5 per cent. This is lower than the ultimate

Table I. Spectrographic analysis of gadolinium specimens after final anneal, ppm.  
 (accurate to within 20 per cent of correct value)

Impurity Element	Sample				
	1A	2A	1B	2B	3B
Ca	50	25	25	25	25
Al	15	15	15	15	15
Cr	20	50	50	20	20
Fe	120	120	120	120	120
Si	25	53	25	200	25
Mg	10	10	10	36	10
Ta	200	200	200	200	200
Cu	35	100	200	100	35
Ni	20	20	20	50	20

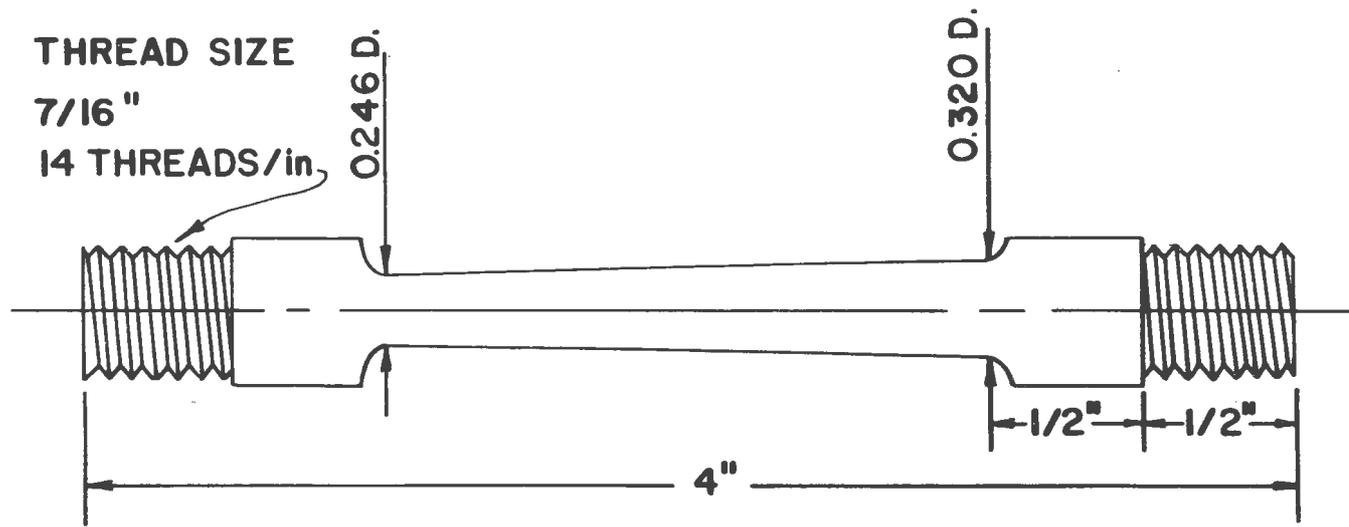


Figure 1. Tapered tensile specimen

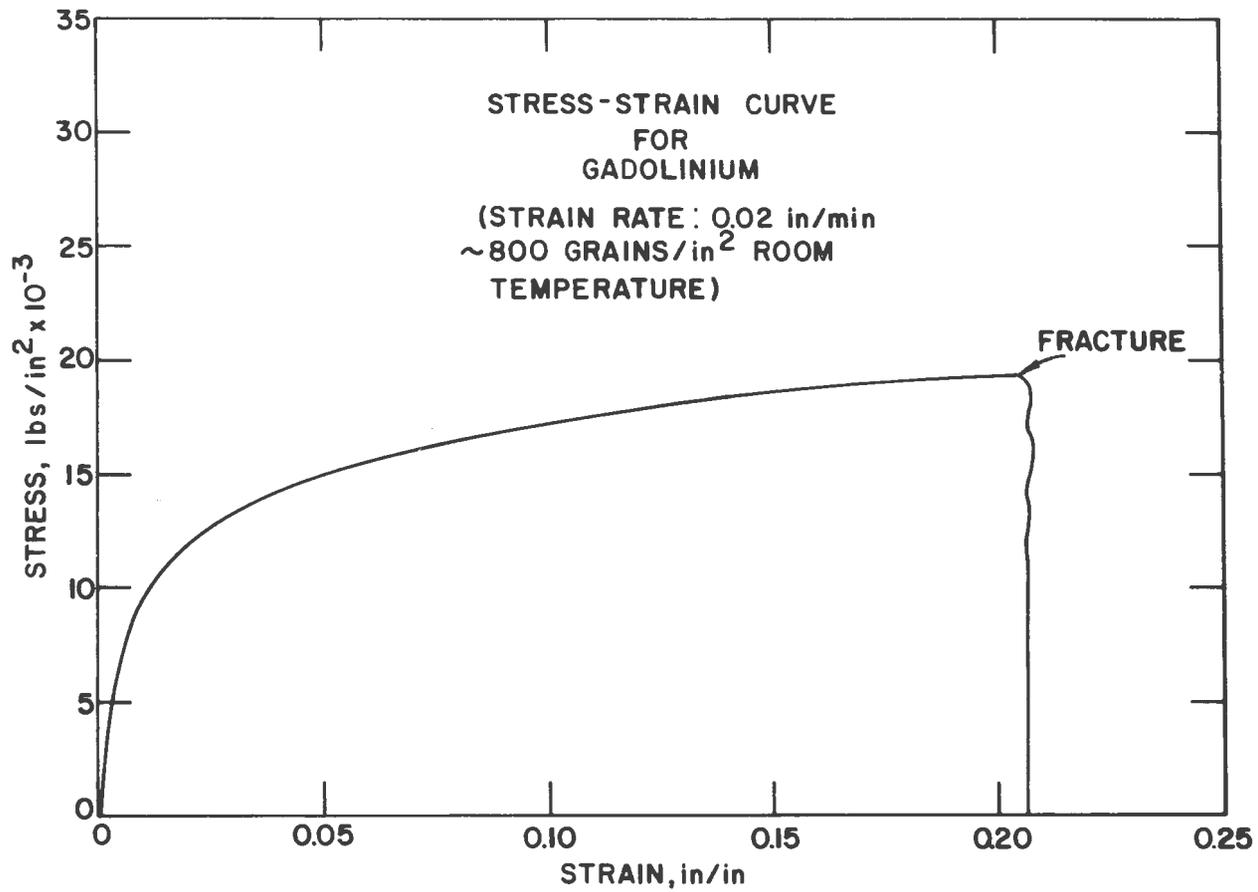


Figure 2. Stress-strain curve for gadolinium

strength of  $24 \times 10^3$  psi for extruded gadolinium reported by Spedding and Daane (20, Figure 18.2, p. 437). Also the elongation at fracture is higher than their value, 16 per cent. This may indicate that the material used in this study is of higher purity than their extruded gadolinium, but the values are reasonably close.

#### Determination of critical strain with tapered tensile specimen

One tapered tensile specimen was machined as in Figure 1. The taper was calculated to give a final elongation of 1.0 to 8.0 per cent with a 800 lb. load. It was encapsulated in a  $3/4$  inch diameter tantalum tube with ends arc-welded in place in an atmosphere of 300 mm. Hg helium. This capsule was then sealed in an evacuated 22 mm. I.D. quartz capsule. Figure 3 illustrates the arrangement. Both capsules are needed because the tantalum protects the gadolinium from reaction with the quartz and the quartz protects the tantalum from oxidation during the annealing periods. At first it was thought that the gadolinium need only be prevented from touching the quartz. However, when gadolinium which was sitting in an open tantalum cup within the quartz capsule was heated, it began to melt at  $1200^\circ\text{C}$ . The reaction was not identified, but it may have been that silicon and oxygen were gaseously transported to the gadolinium.

The tapered tensile specimen was annealed for four hours at  $1150^\circ\text{C}$ . in a furnace made of firebrick and heated

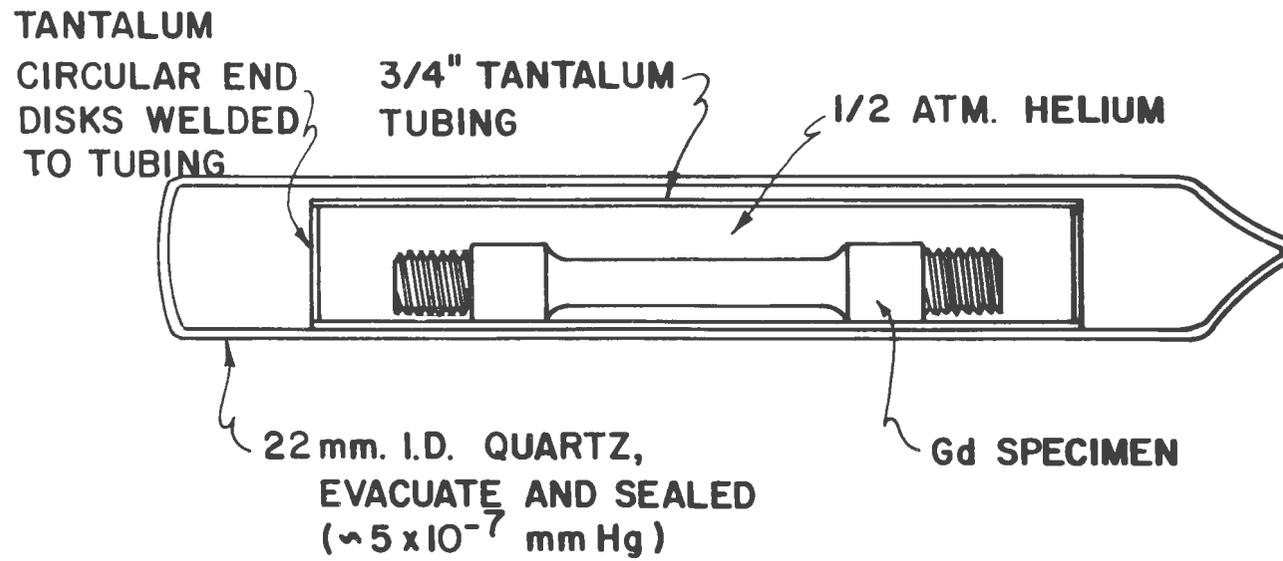


Figure 3. Tensile specimen sealed in tantalum and quartz capsules

with cylindrical silicon carbide heating elements. The outside dimensions of the furnace were approximately 13 inches on each side. The internal heated cavity had dimensions of four inches by four inches by eight inches. The four heating elements were placed longitudinally along the edges of this cavity. Firebrick plugs filled the circular entrance ports at each end of the cavity. A firebrick support held the quartz capsule as close as possible to the center of the furnace. Temperature was both monitored and controlled with a Pt-PtRd thermocouple close to, but not touching the quartz capsule midway along its length. This thermocouple was connected to a saturable-core reactor control.

After this anneal which was necessary to erase the previous internal stresses, the specimen was removed from the capsules and strained in tension. The loading curve is shown in Figure 4. Gage marks every 1/4 inch along the entire two inch gage length indicated a distribution of strain from one to eight per cent as had been calculated. The strain as a function of position along the specimen is shown in Figure 5. The distance between gage mark scratches was measured with dividers and a micrometer.

The specimen was sealed in tantalum and quartz as before and reannealed at 1200°C. for three days.

After removing the specimen from the capsules, one could easily see the grains due to evaporation from the grain

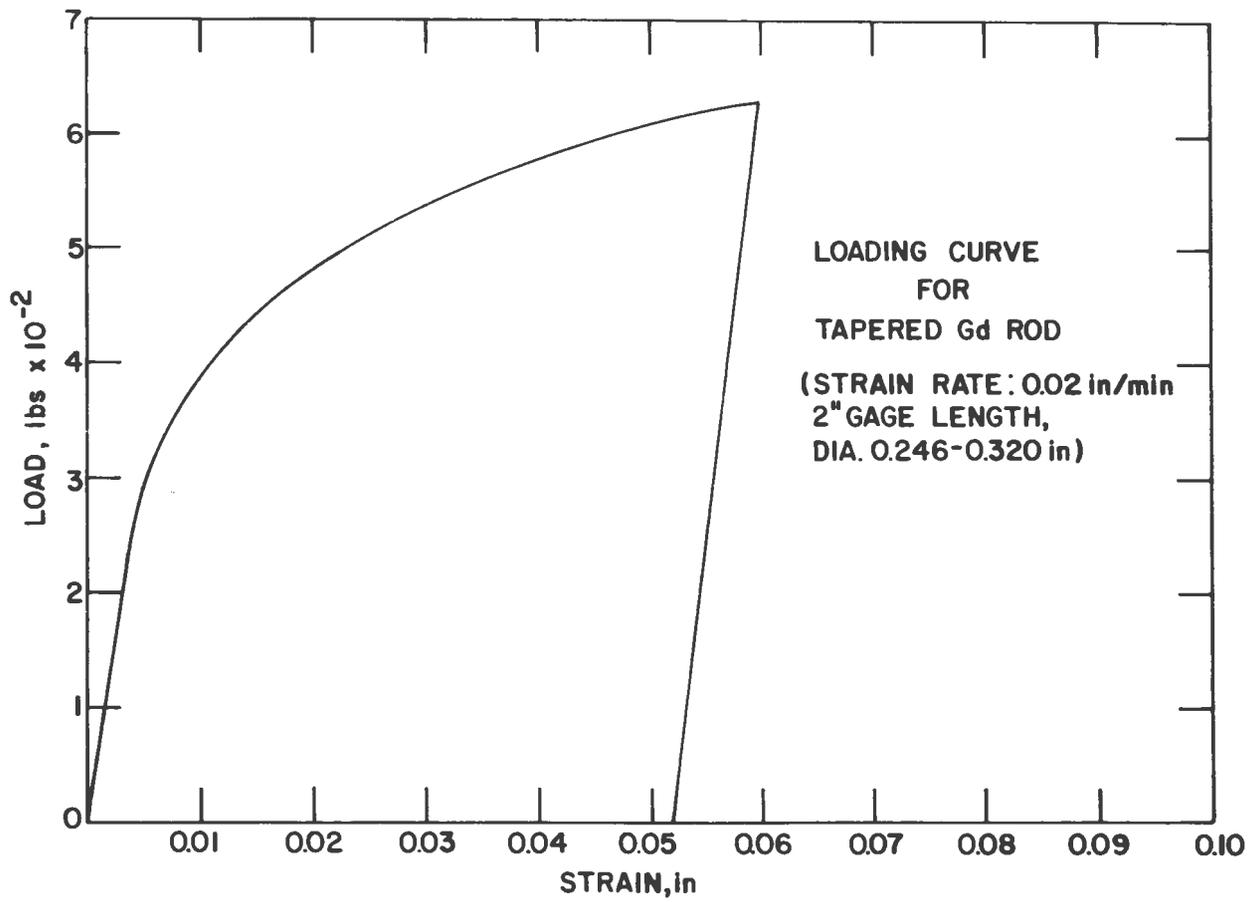


Figure 4. Loading curve for tapered Gd rod

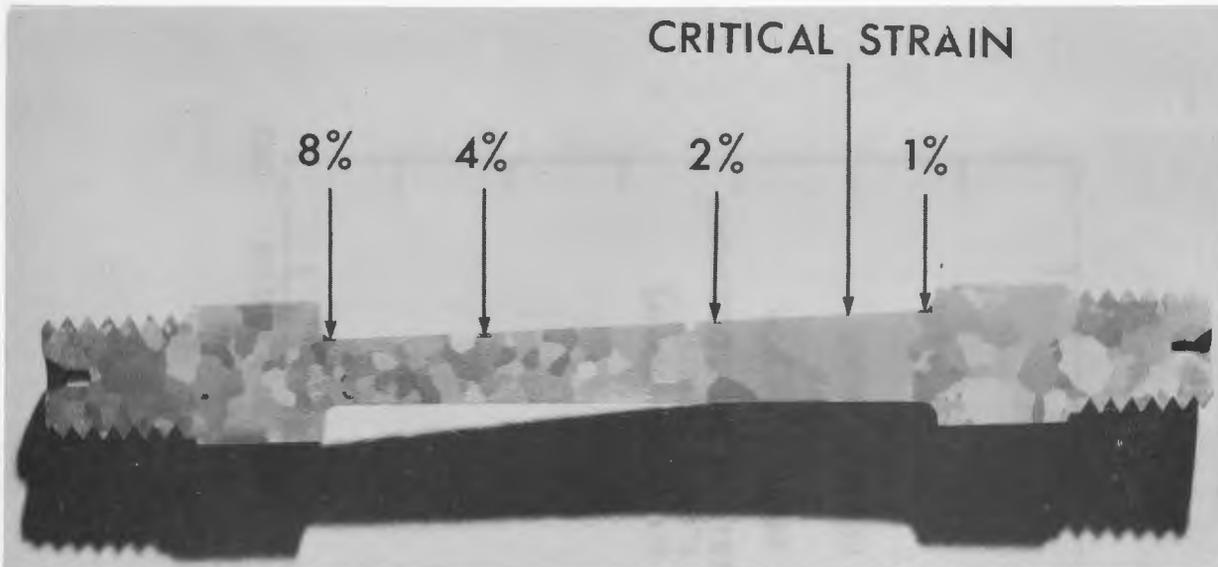


Figure 5. Tapered tensile specimen which has been sectioned longitudinally showing critical strain

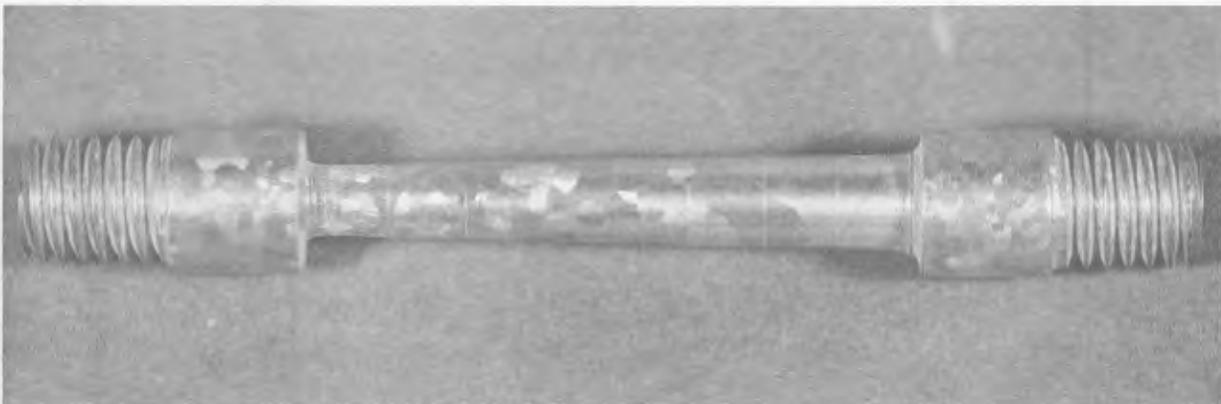


Figure 6. Tapered rod after final anneal at 1200°C.

boundaries. A gradation of grain size is observable in Figure 6. The largest grains appear in the 1.2 to 1.3 per cent strain area. The specimen was split longitudinally with a spark cutter and etched with a two per cent nitol solution. As in Figure 5, one can see that the interior grain size is the same as on the surface.

#### Crystal growing runs

Five specimens were machined according to Figure 1 except that the gage diameter was constant at 0.320 inches. They were encapsulated similarly to the tapered specimen and given similar annealing treatments--four hours at 1150°C. Samples 1A and 1B were placed in the cold furnace and then brought up to temperature in about four hours. This time was not counted as part of the annealing time. The other samples were placed directly into the preheated furnace.

After this initial anneal the samples were removed from their capsules and strained. The stress-strain curves for each are given in Figures 7-11.

Then they were sealed in tantalum and quartz again and annealed at 1200°C. for three days. Again samples 1A and 1B were placed into the cold furnace and heated to 1200°C. in approximately four hours, while the others were inserted into the hot furnace.

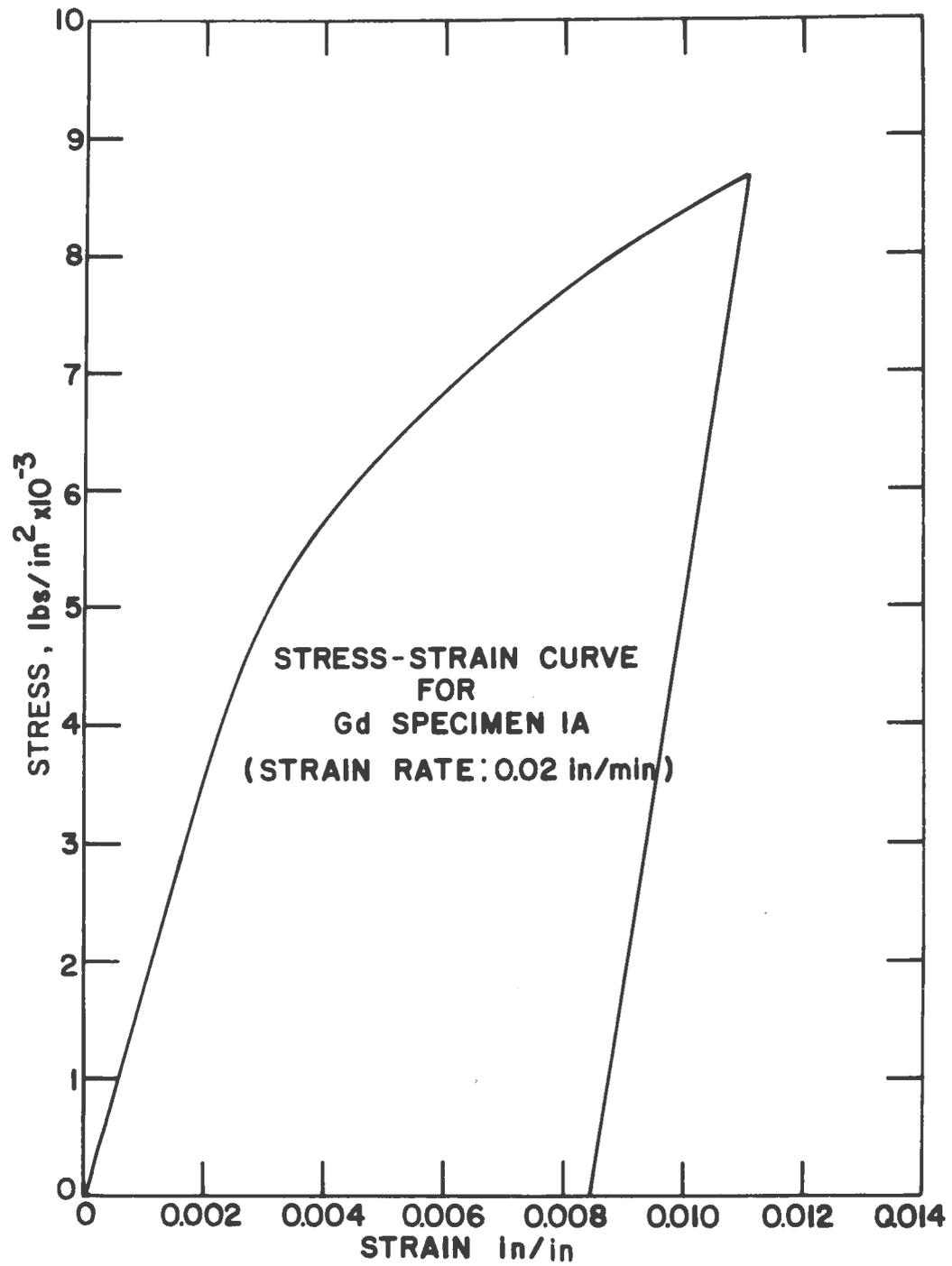


Figure 7. Stress-strain curve for Gd specimen 1A

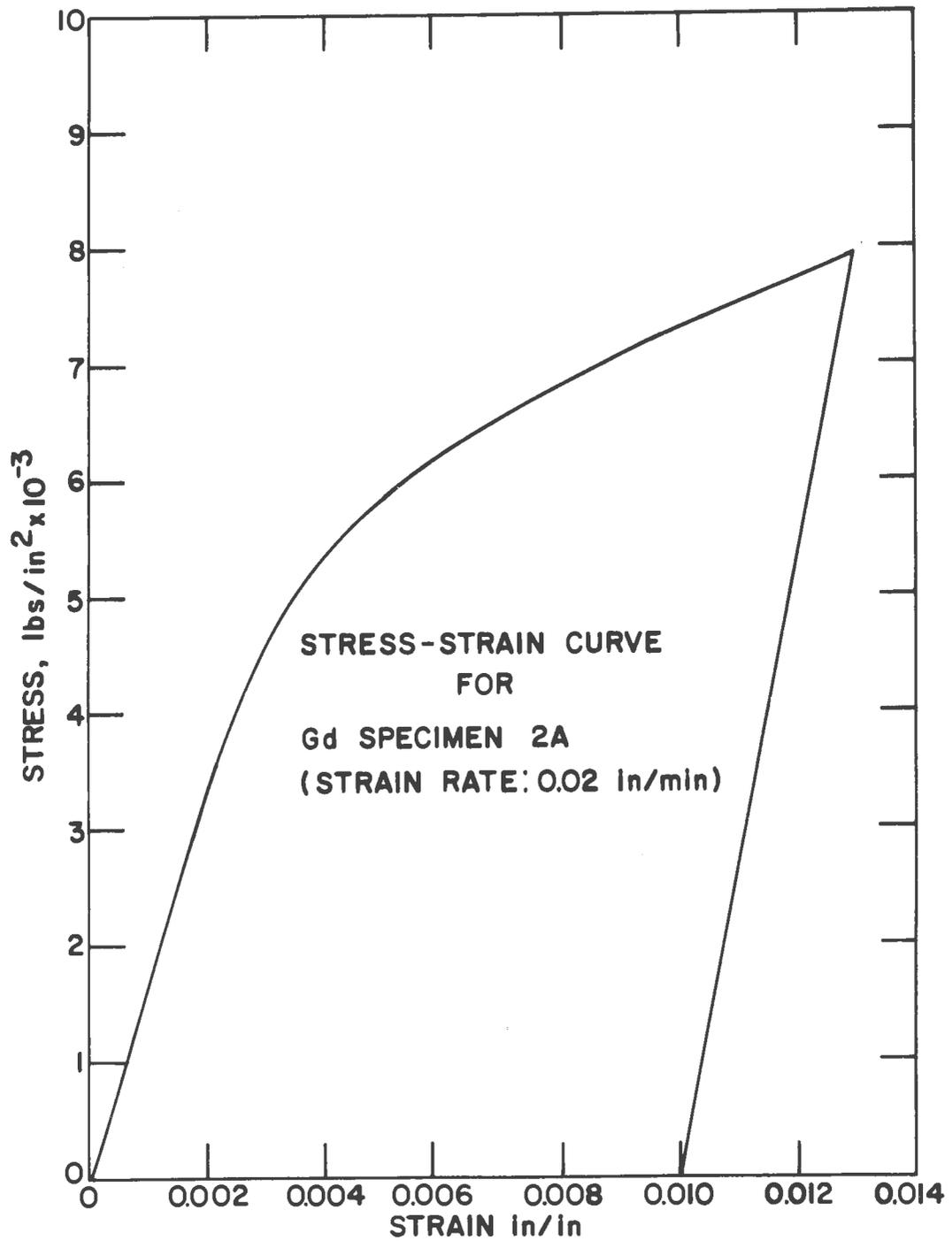


Figure 8. Stress-strain curve for Gd specimen 2A

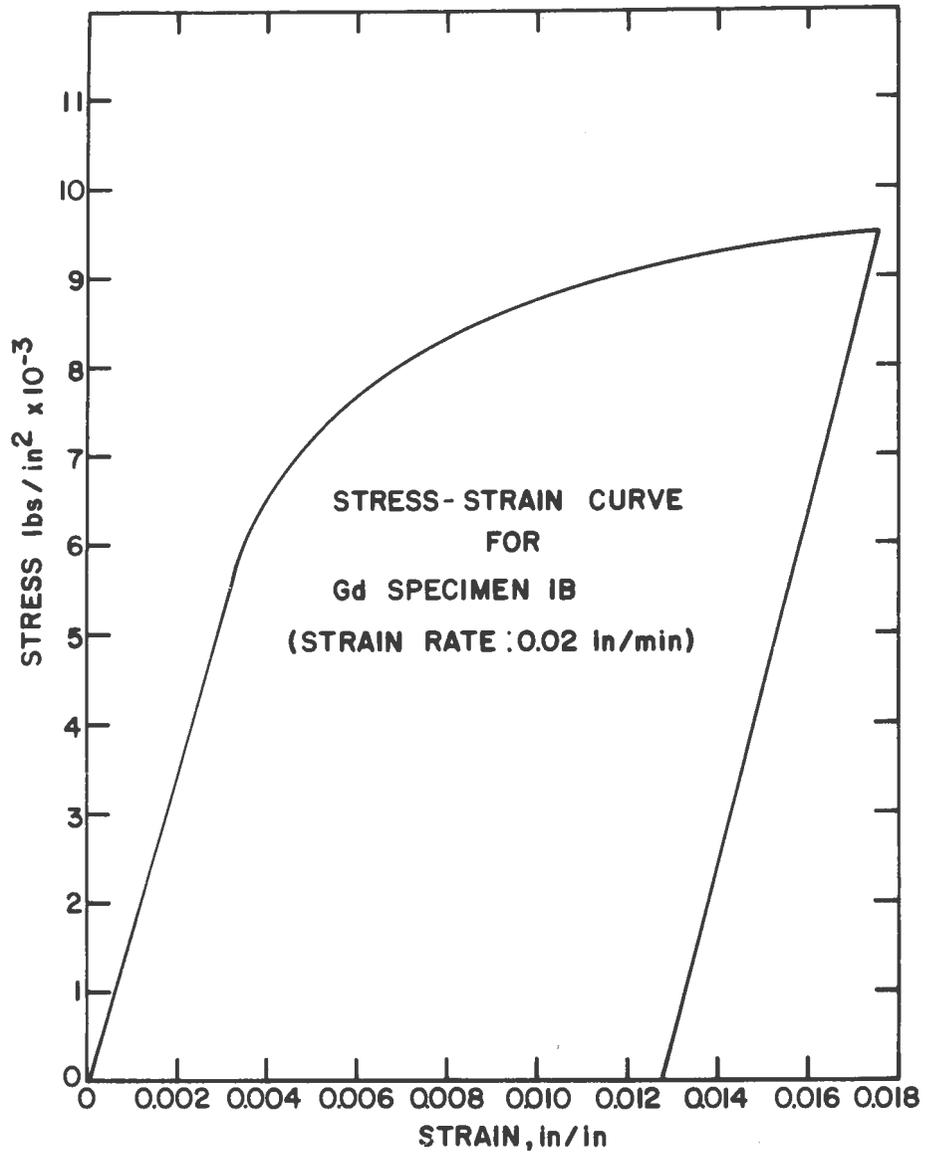


Figure 9. Stress-strain curve for Gd specimen 1B

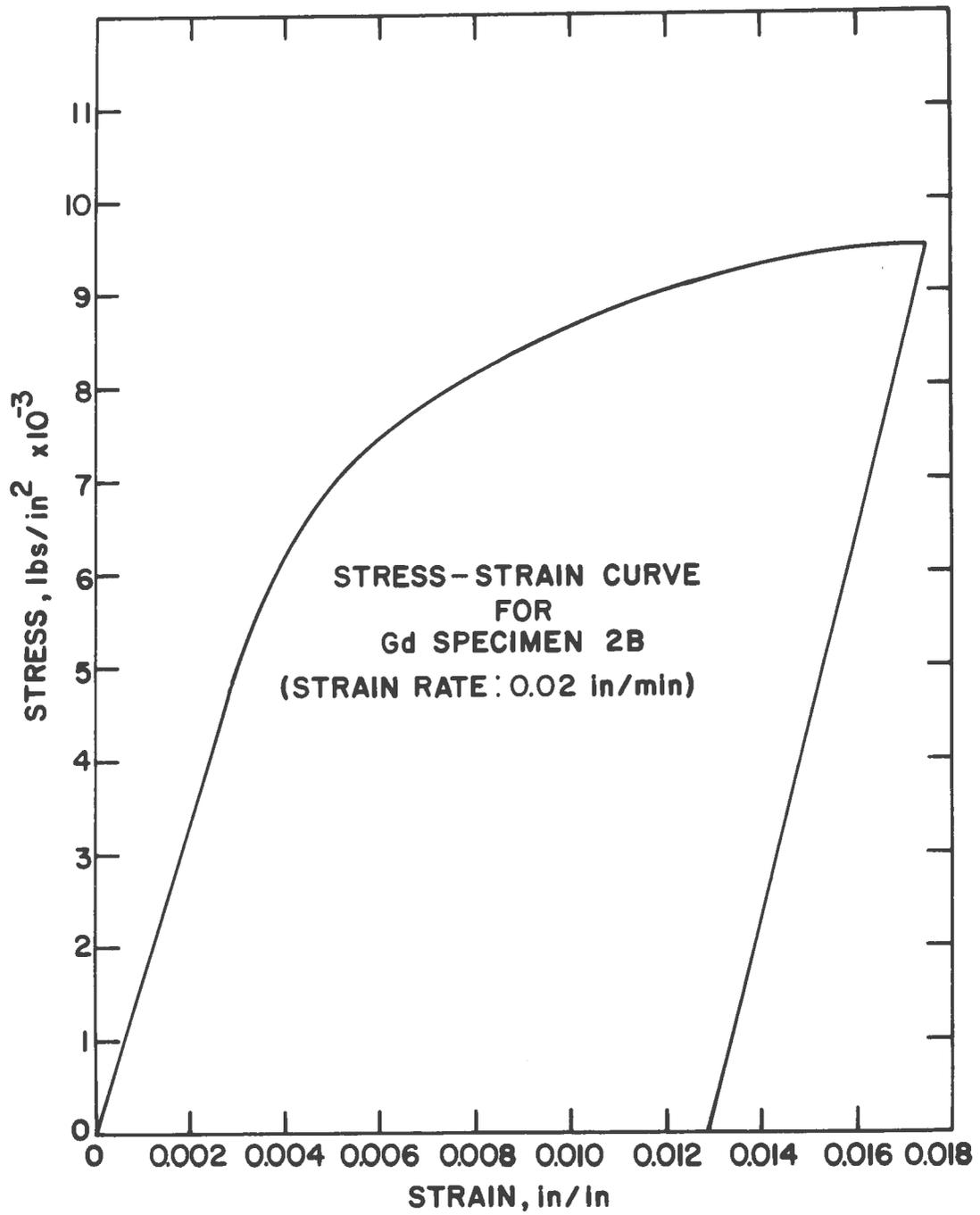


Figure 10. Stress-strain curve for Gd specimen 2B

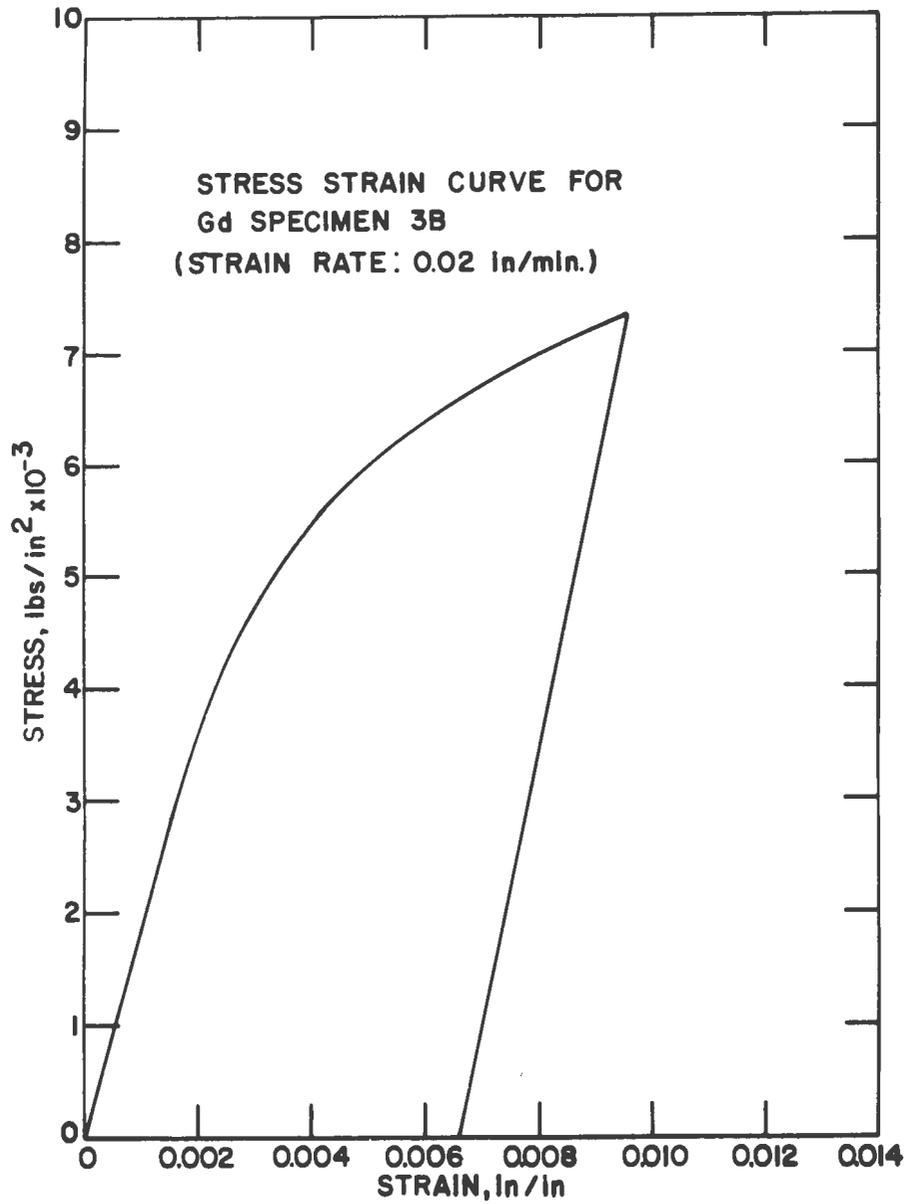


Figure 11. Stress-strain curve for Gd specimen 3B

## RESULTS AND CONCLUSIONS

The photographs in Figure 12 show the final grain sizes obtained in the crystal growth attempts. Samples 2A and 2B seem to be the most successful, yielding  $3/8$  inch and one inch single grains continuing throughout their cross-section. Laue X-ray photographs in Figures 13 and 14 show that these two large grains are relatively strain free. Also, the fact that the photographs taken after rotating the crystal 180 degrees display an inverted, mirror image confirms that the single crystal grain extends through the cross-section. Sample 3B appears to have never recrystallized. It was strained less than the others--only 0.66 per cent. Evidentially this is below the minimum strain needed to produce recrystallization at  $1200^{\circ}\text{C}$ .

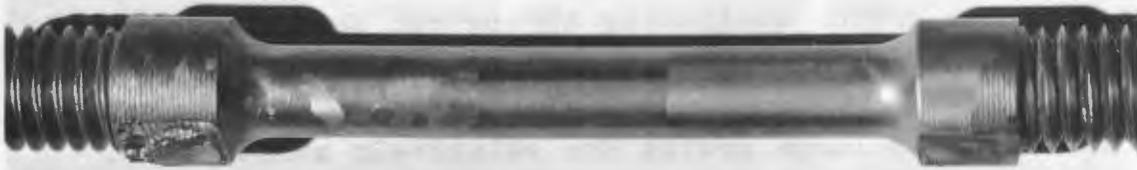
A significant result of this work is that single crystals of gadolinium can be grown by the stain anneal method. In this research there was no attempt to optimize the growth conditions.

It appears that the use of the tapered tensile specimen to find the critical strain is a very efficient method. Its use may greatly reduce the experimentation and quantity of metal required to investigate the variation of critical strain with changes in other variables. In this work, the tapered tensile specimen indicated a critical strain of 1.2 to 1.3 per cent for gadolinium metal with 800 grains per

2B



1A



2A



1B



2B



3B

Figure 12. Gadolinium tensile specimens after final anneal at 1200°C.

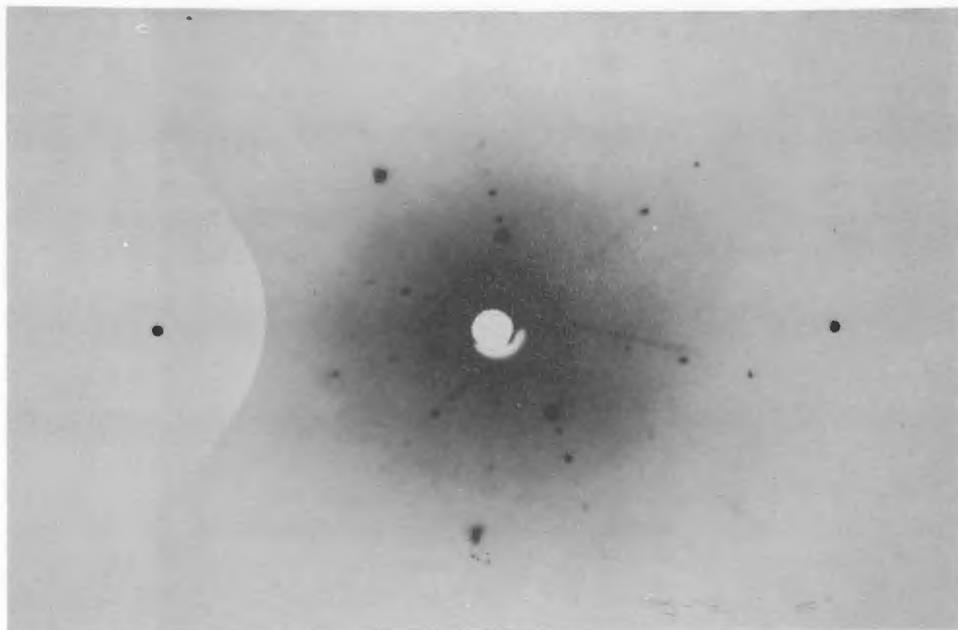
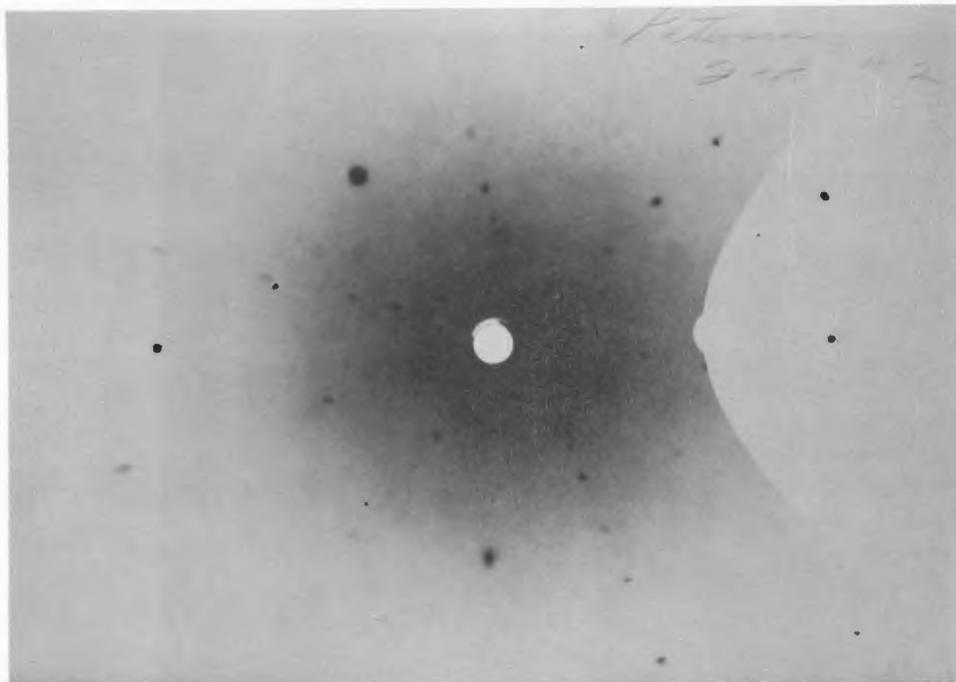


Figure 13. Laue X-ray photographs of  $3/8$  inch grain in specimen 2A; bottom photograph taken after rotating the specimen  $180^\circ$

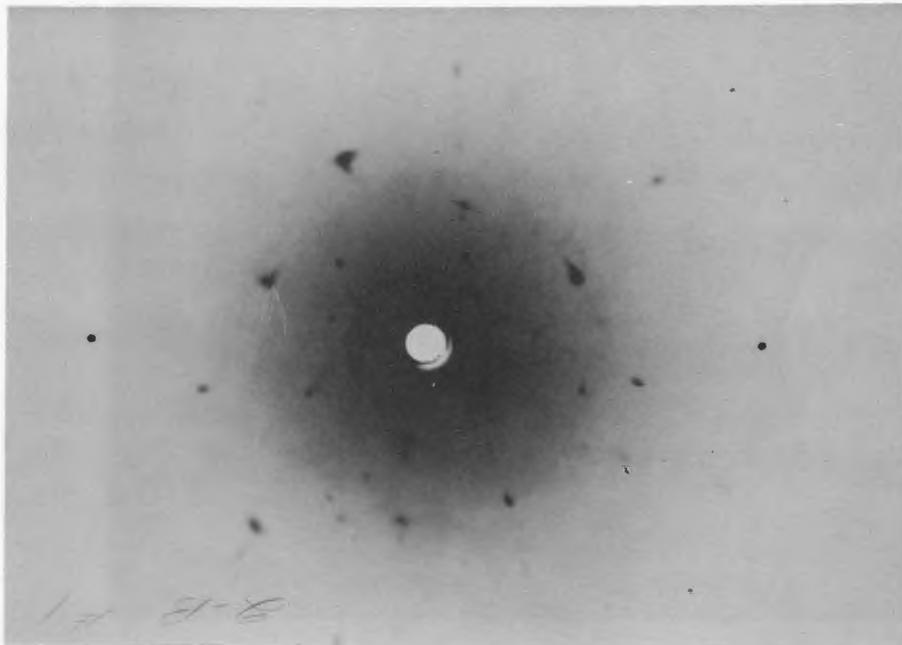
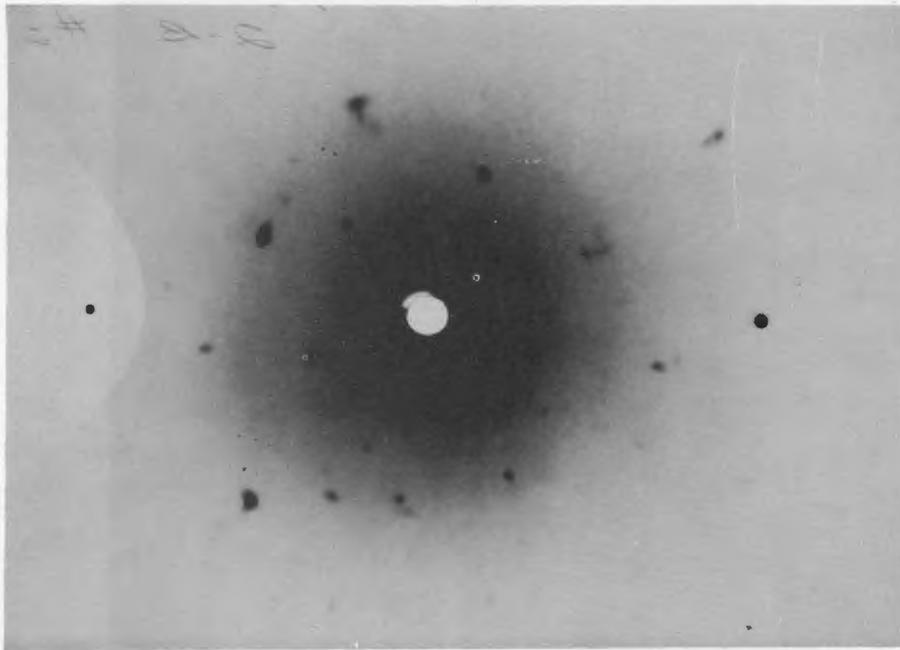


Figure 14. Laue X-ray photographs of one inch grain in specimen 2B; bottom photograph taken after rotating the specimen  $180^\circ$

square inch and a preferred orientation produced by the arc-melting with the c-axis perpendicular to the specimen axis and a 1200°C. annealing temperature.

Optimization of the strain anneal technique for growing gadolinium single crystals might be accomplished with further use of tapered tensile specimens. The critical strain could be determined for various combinations of annealing programs and penultimate grain sizes and orientations. From these critical strains, that which gave the largest final grain size would identify the optimum growth conditions.

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