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ANNEALING STAGES IN EXPLOSIVELY DEFORMED COPPER

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Paul Gordon, Donald Brillhart
and
Arnold Preban

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Abstract. OFHC copper, explosively deformed at pressures from 75 to 435 kilobars, has been found to anneal in several different stages--a recrystallization stage proceeded by three to four recovery stages. The first recovery stage, at temperatures under 100°C, is marked by an appreciable decrease in density rather than by a density increase as is normally expected in annealing. A sample of high purity copper deformed in tension exhibited at least three of the four recovery stages, including the first stage with its accompanying decrease in density.

Evidence has also been found that the explosively deformed samples contained high residual internal stresses and, at pressures of 250 kbars and above, a significant gradient in imperfection structure in the deformation direction. This gradient affected the rate of recrystallization but not the prerecrystallization hardness.

Annealing Stages in Explosively Deformed Copper

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I. Introduction

A series of investigations have been undertaken with the object of studying the annealing behavior of explosively deformed copper. The first phase of this program has been completed. In this phase, samples of fine-grained, OFHC copper, explosively deformed at pressures in the range 75 to 435 kilobars*, have been annealed isochronally at temperatures from room temperature up to those sufficient to give complete recrystallization. The annealing characteristics of these samples and of a sample deformed conventionally in tension were studied by means of microhardness and high precision density measurements.

The results of the study are described in this report. The second phase of the research is now being pursued; in the second phase annealing of the explosively deformed copper is being studied isothermally at temperatures selected on the basis of the isochronal results. In addition to hardness and density measurements, changes in stored energy are being investigated by means of isothermal microcalorimetry.

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*One Kilobar = 10^9 dynes per cm^2 (approximately 1000 atmospheres)

II. Experimental Details

For the explosively deformed specimens, O.F.H.C. copper with a grain size of 0.013 mm. was prepared in the form of 1/2" x 3" x 3" plates. The explosive loading was carried out at the Eastern Laboratory, Explosives Department, E.I. duPont de Nemours and Co., Inc., Gibbstown,N.J.; through the courtesy of Dr. George Dieter; the techniques have been described in the literature[1]. Pressures of 75, 145, 250, 345 and 435 kilobars were used, provision being made to quench the samples in water immediately following the loading to minimize annealing effects.

The conventionally deformed sample was cut from a polycrystalline, cylindrical bar of A.S. and R. high purity copper (reported to be 99.999% copper) which had been deformed at room temperature to 30% elongation in tension.

Hardness testing was conducted with a Gries Reflex Microhardness Tester using a 100 gram load on a Knoop diamond. The machine was checked from time to time with a well annealed, carefully polished copper standard. Each reported hardness value is the average of twenty readings taken on a single sample surface; the reproducibility of the average of a given set of readings was found to be about $\pm$ 3 KHN. For each explosively deformed sample,

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[1] Proceedings of Conference on Response of Metals to High Velocity Deformation, 1960.

measurements were made on three surfaces (see Figure 1). One of these-referred to as the transverse, or T, surface--was parallel to the direction of the explosive loading; the second--called the longitudinal L*, or front, surface--was perpendicular to, and facing, the direction from which the loading came; the third surface, referred to as the longitudinal L, or rear, surface--was opposite to surface L*. For the tensile sample, hardness measurements were made on a surface perpendicular to the tensile axis.

To facilitate accurate correlation, in the case of the explosively deformed samples the density measurements were made on the same samples used for the hardness testing. The standard method of weighing in air and in a liquid was employed, weights being determined on a Mettler H-16 semi-micro balance having a sensitivity of 0.01 milligrams. For the weighing in liquid the sample was suspended by a platinum wire and a gold carrying cage in a bath of ethyl phthalate. This liquid was chosen because it has a reasonably high density, low surface tension against metals and a low vapor pressure. The temperature of the bath was held slightly above room temperature (in an air-conditioned room) and controlled to within $\pm$ 0.001°C over periods of several hours; for longer periods the bath temperature varied somewhat more, but at each density measurement was read to $\pm$ 0.001°C with a Beckman differential thermometer kept in the bath. The reported densities were all corrected to a single temperature

using the literature value of the expansion coefficient of copper and a measured value for the ethyl phthalate of 6.4 x 10^{-3} per ^{o}C. In order to insure that conditions remained essentially constant over time periods of weeks or longer, the copper hardness standard was also used as a density standard against which to check the bath. For each density measurement, a sample was thoroughly cleaned in carbon tetrachloride, distilled water and research grade acetone. It was then suspended in the ethyl phthalate and the bath and sample given two to three hours to come to temperature equilibrium before weighing. The reproducibility of the density measurements was found to be $\pm$ 10^{-4} gms/cm^3, or about 0.001% for copper. Each density was measured at least twice; individual values were accepted only if found to be reproducible within 10^{-4} gm/cm^3. The results are reported as fractional changes based on the fully annealed values, since the bath was not calibrated on an absolute basis.

In order to avoid edge effects, before cutting samples from the explosively deformed plates 1/4 inch was trimmed from the plate perimeters and discarded. Samples were then cut as indicated in Figure 1 from the remaining portions of the plates and ground to 1/4" x 1/2" x 1 1/4". All samples were then given a mechanical metallographic polish followed by electropolishing, a total of 0.030" being removed from each dimension. The cutting and grinding were carried out with the samples in contact with dry ice; at no time were

the samples allowed to become warmer than room temperature. Microhardness measurements were made on each sample in the last stages of polishing, and the electropolishing was continued until no further change in hardness was noted. The same careful procedure was used in preparing samples from the tensile bar.

III. Results and Discussion

The data obtained by hardness measurements are presented graphically in Figure 2. The hardness results divide the annealing process into two general regions--recrystallization, in which for each sample there is a large, precipitous drop in hardness, and recovery, the pre-recrystallization annealing period in which no major hardness changes take place. The scatter in the hardness data during recovery is such that little can be said from this data alone concerning the phenomenological details of the recovery region. For the explosively deformed samples, there does, however, appear to be a significant difference in the trend of the hardness for the transverse direction on the one hand and the longitudinal on the other during annealing at temperatures up to about 100°C. For each sample, the longitudinal and transverse hardnesses change in opposite directions during this early annealing, and seem to come together at about 100°C. This very probably means that the explosive deformation has left internal stresses in the samples which

are annealed out at temperatures up to 100°C. The directions of these early hardness changes are such as to indicate that at deformation pressures up to 250 kbars the residual stresses are tensile in character parallel to the deformation direction and compressive perpendicular to this direction; at the higher deformation pressures the residual stress directions are reversed. This interpretation is supported by the fact that at two of the three lower pressures--namely 145 and 250 kbars--the deformed copper plates contained some cracks, the planes of which were perpendicular to the deformation direction. At the higher pressures no cracks were found.

Examination of the hardness data in the recrystallization range reveals that at 250 kbars and over recrystallization takes place non-uniformly throughout the explosively deformed samples. In each of these cases, the longitudinal surface L*--the front surface of the sample, facing the direction from which the deformation came--recrystallized last, and the opposite longitudinal surface recrystallized earliest, with the transverse surface intermediate between the two. In all three cases, however, no significant differences in hardness were evident for the various surfaces just prior to recrystallization. It seems probable, therefore, that at these high pressures there is a considerable gradient in the nature of the imperfection structure from front to rear of the deformed plates; this gradient,

however, must be such as to have little effect on the overall hardness. At pressures below 250 kbars, the gradient in recrystallization behavior was not observed.

The changes in density during annealing of both the explosively deformed samples and the sample deformed in tension are presented in Figure 3. The latter portion of the curve for the 145 kbar sample is drawn dotted because electropolishing of this sample after the 196°C anneal opened a small crack in the sample surface. The data points from there on are self-consistent, but were all plotted in Figure 3 at values 0.75×10^{-4} units higher on the density ordinate than the actual measured values in order to obtain the most probable correlation between these and the earlier points.

From Figure 3 it may be seen that density is a much more sensitive measure of the recovery stage annealing changes than is hardness. The most striking feature of the density curves is the large <u>decrease</u> in density shown at the lowest annealing temperatures for all samples. Since it is generally expected that deformation decreases the density of a well annealed metal*, it is also to be expected that annealing increases the density, and this has invariably been found to be so in the past. In the present work, however, a well-defined, and quite appreciable, decrease in

*Exclusive of changes due to such things as sealing cracks and holes.

density is obtained on early annealing before the ultimate density increase sets in later in the annealing. To the writers knowledge this is the first reported case of such a density change on annealing. In this connection, it is worth emphasizing that not only the explosively deformed samples, but also the sample deformed in tension shows the density decrease. Since the latter sample is of quite high purity it is unlikely the density decrease can be ascribed to a precipitation process. Thus, the phenomenon may well be a general one; if so, the fact that it has not been previously noted may be due to the small absolute magnitude of the change and the low temperatures at which it occurs.

The hardness and density changes during annealing are shown together in Figure 4. In this figure, the hardness data for the three surfaces of each sample have been averaged (except in the stress-relief region below 100°C). In addition, the curves for both density and hardness have been drawn through the data points to indicate in each case what is considered to be the most probable trend based on a correlation of the two kinds of data. A study of these correlations reveals that the annealing process is made up of a recrystallization stage and from three to four recovery stages. Figure 5 summarizes graphically the temperature ranges and property changes associated with each stage

for the range of deformation studied.

The first recovery stage, marked by a decrease in density, occurs in all samples, both explosively and conventionally deformed. The temperature range of its occurrence--room temperature to about 75°C--is essentially unaffected by the amount of deformation. In this latter respect, the 345 kbar sample appears to be somewhat anomalous--the reason for this anomaly is not known, but may, for example, be associated with an ineffective water quench of the sample just after deformation. The direction of hardness change during this first stage is not certain, but is indicated in Figure 5 as a decrease on the basis of the fact that the hardness of the tensile sample decreased during this annealing period.

The second recovery stage is associated with an increase in density, which again occurs in all samples and within about the same temperature range in each--75° to 140°C (with the exception of the anomalous 345 kbar sample). The hardness change in this stage also is uncertain, but is indicated as an increase in Figure 5, again largely on the basis of the change shown by the tensile sample.

In the third temperature range of pre-recrystallization annealing little or no changes in either density or hardness take place. With the exception of the 345 kbar sample, the temperature range of this annealing plateau--about 140° to 200°C--also appears to be roughly unaffected

by the amount of deformation.

Just prior to the beginning of recrystallization a fourth recovery stage is present in the samples deformed at 75, 145 and 250 kbars. This stage is evidenced by a slow decrease in hardness and a corresponding rise in density starting at about 200°C. The onset of recrystallization appears to interrupt the progress of this fourth stage. Since the recrystallization start temperature decreases continuously with increasing deformation, the temperature range of the fourth stage also decreases in extent; for the 435 kbar sample--and probably also for the 345 kbar sample--the recrystallization start temperature is low enough so that the fourth recovery stage is absent.

The isochronal annealing data described thus reveal that the annealing process in both explosively and conventionally deformed samples is quite complex. It consists of a recrystallization stage and three or four earlier recovery stages. From previous work there seems little doubt that recrystallization involves primarily the sweeping out of excess dislocations by the movement of high-angle grain boundaries. The details of recovery phenomena in terms of imperfection movements are not nearly so clear. In the present research the data is as yet insufficient to warrant serious speculation as to these details. It is hoped that the continuation of the research by isothermal techniques, including energy measurements, will enable some clarification

of the recovery range phenomena. At present it may only be surmised that in view of the sequence of the stages, their general features and the temperature range involved, it seems probable the first and second recovery stages may be associated largely with point defects and the fourth with some form of dislocation movement.

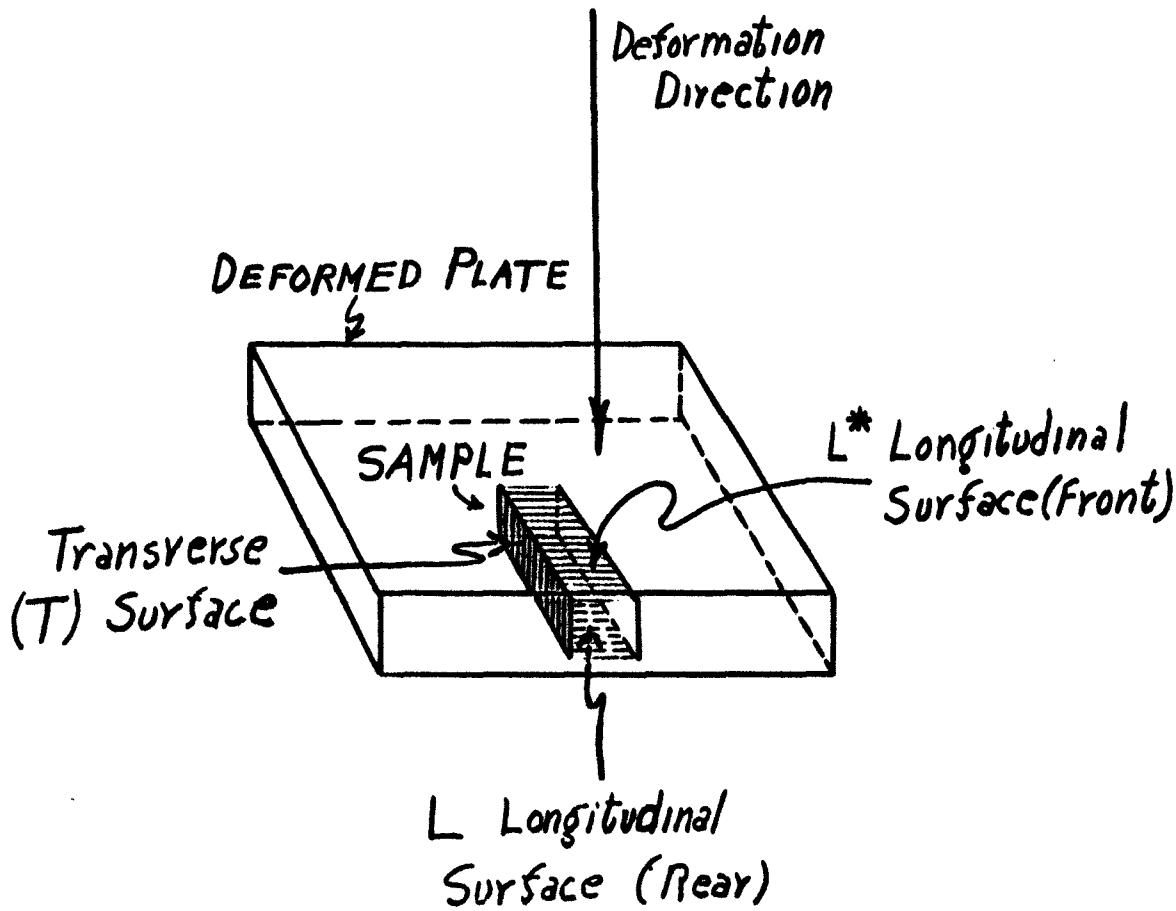

Figure 1. Typical location of sample cut from explosively deformed plate. Plate 1/2" x 3" x 3". Sample 1/4" x 1/2" x 1 1/4".

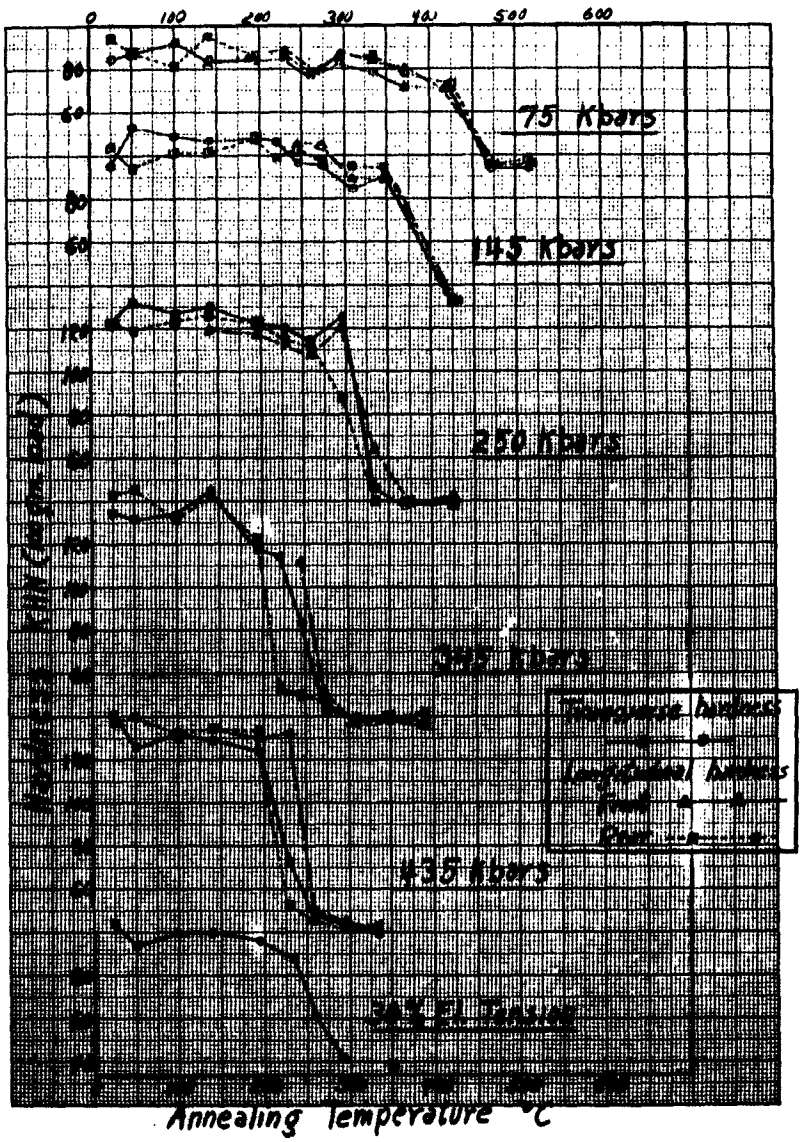

Figure 2. Hardness changes during annealing of explosively deformed samples and sample deformed in tension.

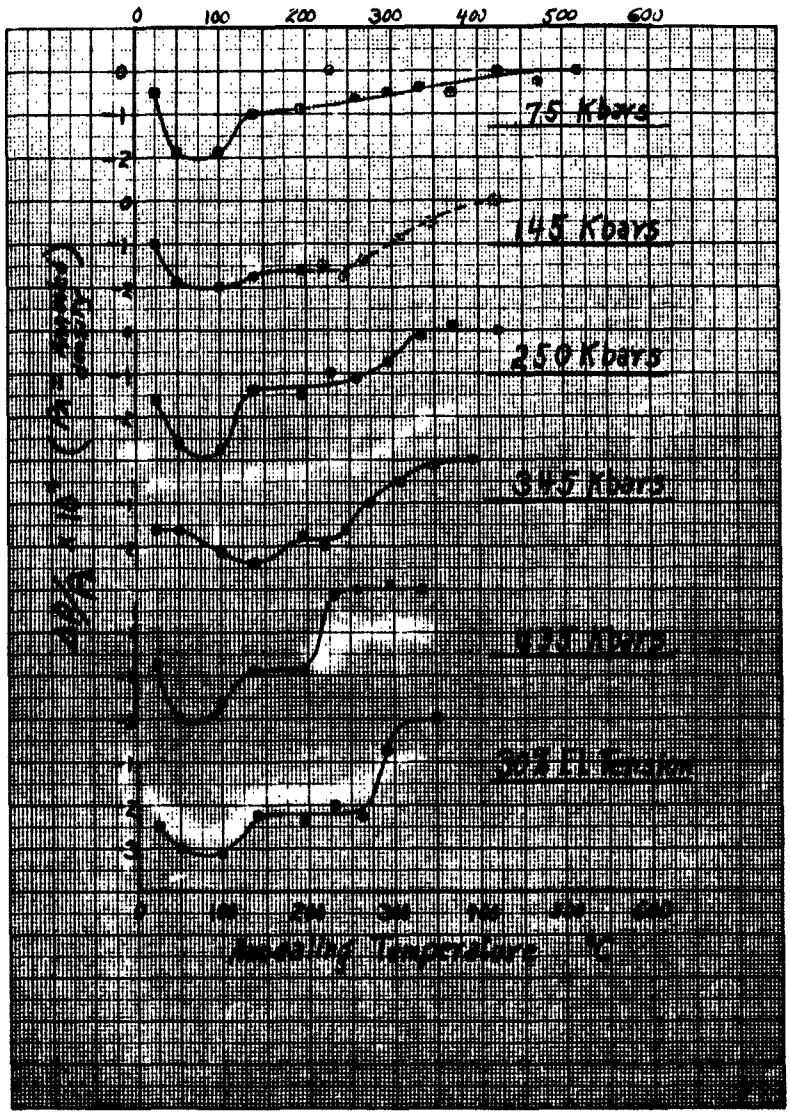

Figure 3. Density changes during annealing of explosively deformed samples and sample deformed in tension.

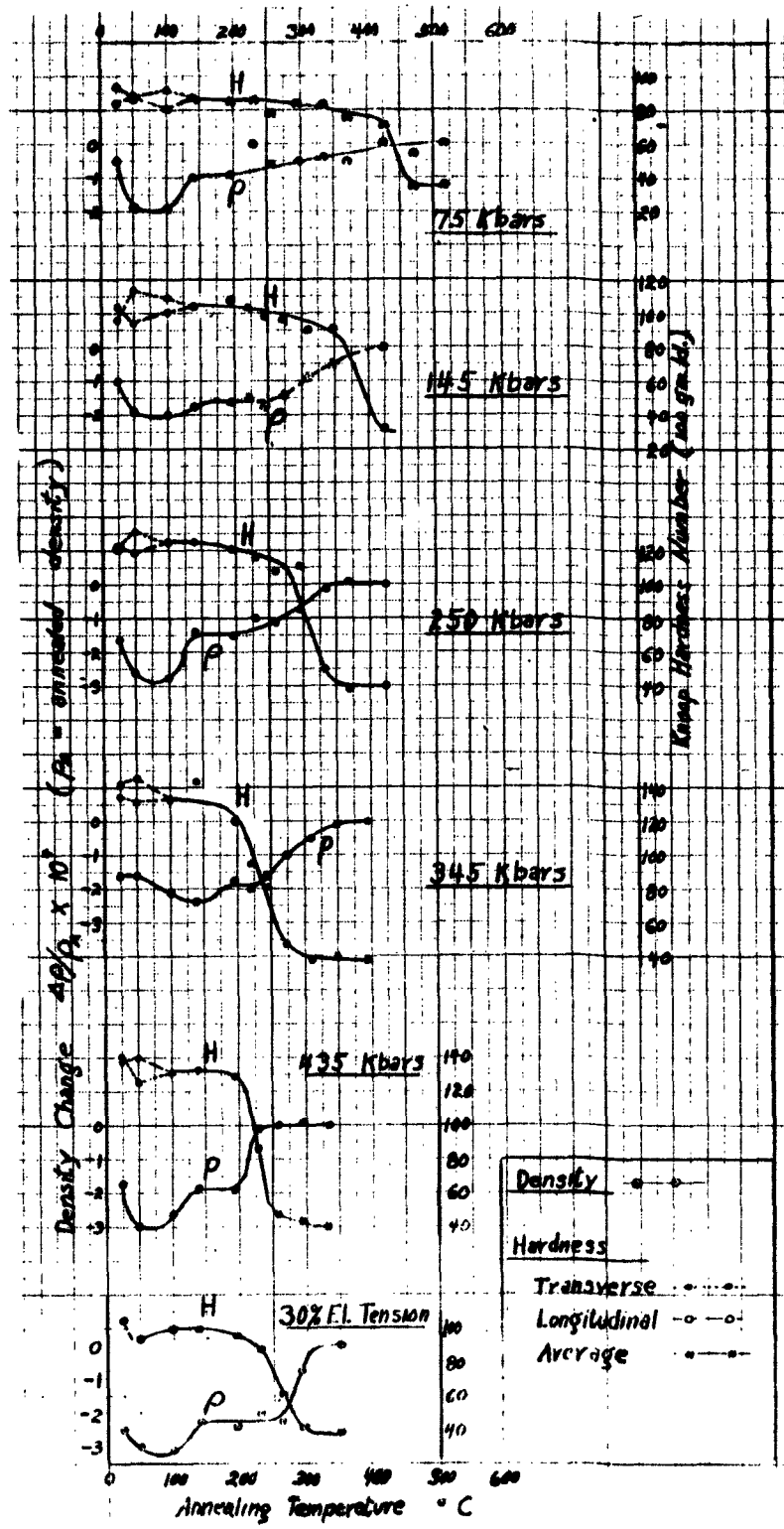

Figure 4. Correlation of hardness and density changes.

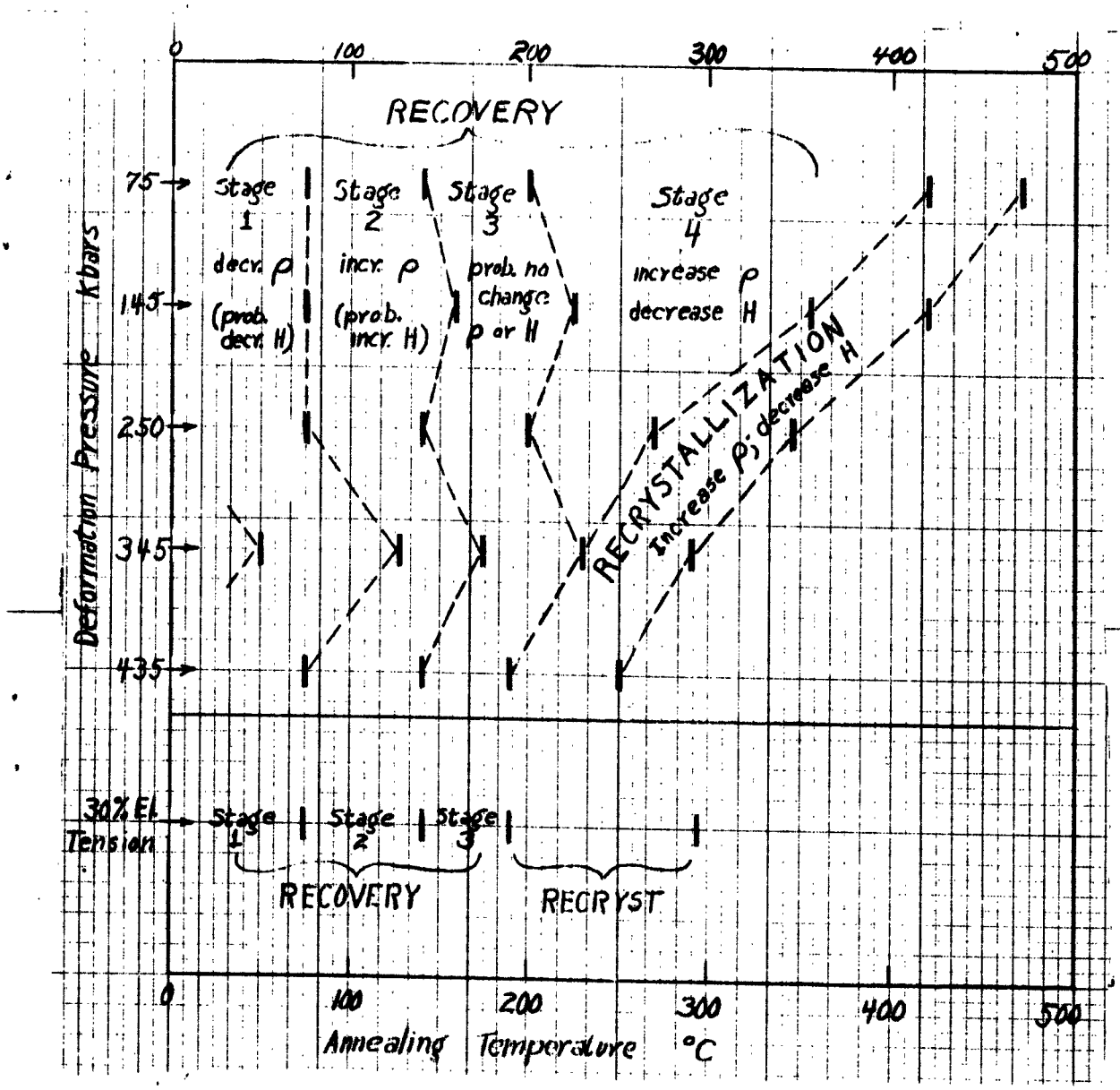

Figure 5. Summary of probable annealing stages.