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# An Investigation of the Hardening Properties of some Alpha Copper Indium Alloys

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AN INVESTIGATION OF THE
HARDENING PROPERTIES OF SOME
ALPHA COPPER-INDIUM ALLOYS

by .CHARLES W.McPHERSON

### A THESIS

SUBMITTED to the DEPARTMENT of METALLURGY in PARTIAL FULFILLMENT of the REQUIREMENTS for the DEGREE of BACHELOR of SCIENCE in METALLURGICAL ENGINEERING

MONTANA STATE SCHOOL of MINES
BUTTE; MONTANA
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#### INTRODUCTION

Precipitation hardening alloys has assumed a major role in modern day development of alloys. In his search for stronger, harder materials, the metallurgist has often been limited by the cost of metals which posses the needed properties. Partially as a result of this limitation and partially because of the non-existence of materials satisfying his requirements, the metallurgist has turned to heat treatment to obtain desired characteristics.

From the studies of various research men, precipitation hardening and work hardening have developed. Heat treating technique has so improved that metallurgists can now magnify many properties far beyond their ordinary values. Both metallurgists and physicists have advanced theories to explain the phenomena, and although most research men agree on fundamentals, their detailed explanations are often conflicting and misleading.

This thesis will not attempt to correlate the numerous

theories on precipitation hardening or work hardening, but will merely list the main ideas necessary for background. the main function of this paper is a rudimentary study of copper-indium alloys and the effects of time, temperature, and composition on their hardening properties in the alpha or solid solution state. In conjunction with this investigation some research has been initiated on recrystallization and grain growth of these same alloys.

## Previous Work

Little information on age hardening of these particular alloys is available, this lack of information being due to the high cost of indium and the resulting reluctance of industry to experiment in this field. Two German scientists, weibke and Eggers, have done considerable work with this system. They determined a constitutional diagram by approximate X-ray methods. Although William Hume-Rothery later modified their diagram by more precise thermal analysis, a portion of the diagram conderning the present work follows.

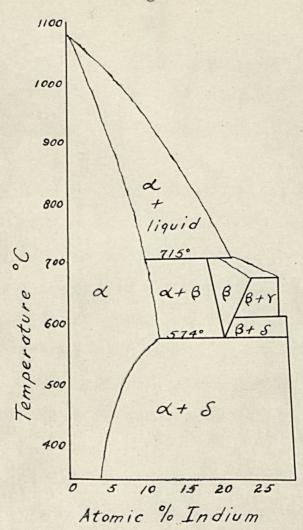


Fig. 1- Part of Copper-Indium Phase Diagram

In their determination of the copper-indium diagram Weibke and Eggers noted that the hardness of copper was only slightly increased by the addition of indium. In their work on age hardening they found that the hardening effect increased with the percentage of indium. They reheated the various homogenized alloys at 200, 300 and 400°. At 200° they noted no

hardening effect up to 400 hours; at 300° and 100 hours these investigators obtained a maximum hardness four times as great as that of the homogenized sample.

## Hardness

the investigator of hardening properties must have a knowledge of hardness and its measurement. Hardness may be defined as (1) the resistance to indentation, (2) stiffness or temper of wrought products, and (3) machinability characteristics. These are the principal methods of explaining hardness, but they do not completely satisfy all possibilities. "Colonel Martel defines hardness of metals as the resistance to displacing the molecules at the surface unit volume."

Actually hardness cannot be explained so simply, as it is dependent on many complex interatomic forces. Therefore, an actual measurement of hardness cannot be expressed in terms of the fundamental physical until of length, time, force, and mass. Hardness has been divided into two major

<sup>1.</sup> Weibke and Pleger, Precipitation Hardening in the System Copper-Indium and the Effect of Nickel and Cadmium on It, C.A. 30,7524.

<sup>2.</sup> Metals Handbook, The American Society for Metals, 1948, P. 7., Cleveland Ohio

Landau, D., Hardness, The Nitralloy Corporation, 1943, New York, P. 14.

fields, "static" hardness, measured by permanent deformation of the tested material and related to the elastic limit of the material, and "dynamic" hardness, measured in terms of elasticity.

The Rockwell Hardness Tester employed in this investigation actually measures the permanent deformation produced in the metal. A 10 kilogram load is first applied to the metal to overcome any surface imperfections; then a major load is applied, the weight being dependent upon the penetrator used. The major load is then removed and the difference in depth of the impressions of the major and minor loads is registered in terms of hardness on the appropriate scale.

Conversion of readings from one scale to another is difficult because the conditions for one method of testing vary from the others. The only possible way to translate from one scale to another is by comparing readings of the first scale with reading of the second scale, such values being obtained from identical material. Such a material may not exist for comparison of the desired scales.

4. Symposium on the Significance of the Hardness Test of Metals in Relation to Design, American Society for Testing Materials, 1943, Philadelphia, Pa. pp. 841-42

## THE ALLOYS of An the industion Chimage

Six alloys of different composition were cast from indium donated by the Consolidated Mining and Smelting Company of Canada and Anaconda wire bar copper, the specimens ranging from 3 to 18 per cent indium. Although no chemical analyses were run on the alloys, subsequent microscopic study of each sample revealed very few oxide inclusions.

To conserve indium the first alloy east contained 18 per cent indium. A sample was then cut from the bottom of the cast alloy, a bottom section being used to avoid inclusions and insure homogeneity. Sufficient copper was then added to the remainder of the first casting to obtain a second alloy of 15 per cent indium. A second specimen was then prepared. This process was repeated until six samples had been obtained, one each of 3,6,9,12,15, and 18 per cent indium.

All alloys were melted in a small induction furnace.

No flux was required in the melt. The mold was cut from graphite and consisted of three pieces clamped together.

The dimensions of the mold were approximately 3/g in. by 2 in.

and a graphite mold was used to decrease the possibility of oxidizing the copper. All specimens were reduced 25 per cent in thickness by repeated passes through a set of hand rolls. The rolled alloys were then heated to 600° C for a period of three hours and quenched. This process promoted recrystallization, which in turn tended to homogenize the samples. Each sample was then cleaned of oxide coating and smoothed on opposite side to provide a good surface for subsequent Rockwell B hardness measurements. Metallographic Studies

A sample was cut from each of the homogenized samples for study under the microscope. After the alloys had been polished, they were etched with ammoniacal hydrogen peroxide. Microscopic study revealed a single phase in all but one alloy. The alloy containing 18 per cent indium revealed the presence of a second phase. This second phase, however, is probably beta and does not give a true picture of solid solution hardening. Small amounts of impurities, probably oxides, were revealed in the specimens.

A sample of each alloy at 40 per cent reduction was also mounted for study along with other samples which had been heated at 200° C and 500° C for various lengths of

time to study recrystallization and grain growth. The lack of a suitable etching agent on some of the higher indium alloys made the results of this investigation rather inconclusive. Some values were obtained for the lower indium alloys by using ammoniacal hydrogen peroxide or concentrated sulphuric acid as the etching reagent. The grain size of each of the specimens was then measured by a micrometer eyepiece for measuring grain size.

Effect of Increasing Indium on Rockwell B Hardness

Rockwell B hardness tests were run on all the quenched homogenized specimens. These tests showed an increasing hardness with increasing amounts of alloying elements (see page 29). The curve produced by plotting atomic per cent indium against Rockwell B hardness shows several irregularities.

Norbury explained this increased hardness as being due to residual stresses set up in the parent lattice by atoms of the solute metal. In his work he found the indentation hardness increased linearly with the atomic per cent of alloying element. Norbury also found that the hardening effect was directly proportional to the difference in atomic radii of the solute atoms and solvent atoms. The stresses set up by solute atoms oppose the dislocation of the lattice

of an alloy.

Grain size also influences hardness, which increases with decreasing grain size. Smaller grain size means a greater number of crystals. This presents a greater variety of crystal orientation and thereby increases resistance to deformation. The factor of different grain size may account for some of the variance in the hardness as atomic per cent indium curve. Because of the irregularity of the curve, comparison of the copper-indium alloys in solid solution is difficult.

## Work Hardening

After a small section of each specimen had been removed for mounting and microscopic study, the alloys were cut in half. One half was used for the study of cold-working properties; the other half of each alloy was later used in age-hardening tests.

## Theory

working. Cold working is defined as "deforming a metal plastically at such a temperature and rate that strain hardening occurs." A metal which has been deformed below

<sup>5.</sup> Metals Handbook, P. 7

its elastic limit tends to return to its original atomic location in equilibrium with regards to its original lattice structure. If a metal is strained beyond its elastic limit, however, plastic deformation takes place by block movement along grain boundaries or successively preferred slip planes of the grain. The most favorably oriented grains, those in which the gliding planes are at an angle of 45 degrees to the direction of strain, will undergo deformation first. The degree of deformation is limited by surrounding grains in polycrestalline material. After the first deformation, and increased load is necessary for further deformation. As deformation continues more and more slip planes are rotated into the planes of the applied force until a preferred orientation results. This process may be continued until the ultimate strength is reached and the specimen fractures.

The plastic deformation of the metal is accompanied by an increase in the hardness of the metal. Various explanations offered as reasons for this phenomenon have proved inadequate. Following is a list explanations offered for the mechanism of work hardening.

1. The shattering of the lattice forms a large number of small crystalline blocks whose slip planes are out of line and therefore prevent slip.

- 2. The preferred orientation of the grains has been listed as a possibility for work hardening, but this explanation fails to explain the hardening up to the 30 to 50 per cent deformation required for preferred orientation.
- The bending and roughening of slip planes by elastic deformation cause frictional resistance to slip.
- 4. Localized slip regions have curved ends that hinder further slip.
- 5. The disregistry of slip planes in normal grains due to their "mosaic" structure is increased with continued slip.
- 6. The calculated strength of atomic bonds would indicate that metals are much stronger than measured values. Since imperfections of mosaic structure result in weak planes, slip occurs first in the weakest planes and as these planes work harden, deformation takes place along stronger or more nearly perfect planes. The limiting strength of the work hardening is therefore that of the theoretical lattice.
- 7. The amorphous cement theory proposes a noncrystalline metal along grain boundaries and slip planes formed by movement of planes and grains. This amorphous material is much harder than crystalline metal and increases resistance to further derformation. This theory has met with insurmountable objections.

## Results

The first step in cold-working was the reduction of all 6 samples 5 per cent in cross section by rolling. The hardness of each specimen was then checked with the Rockwell

<sup>6.</sup> Doan & Mahla, The Principles of Physical Metallurgy, McGraw-Hill, 1941, New York.

Hardness Lester. The samples were reduced another

5 per cent and the hardness again checked. This procedure was repeated several times until a total reduction of about 40 per cent was obtained on all alloys.

The per cent reduction was then plotted against Rockwell
B hardness.

All curves thus obtained were similarly shaped although several irregularities were moted in some. Most differences from regular curves may be attributed to errors in making hardness tests. Because hand-rolls were used for rolling the alloys, some sections of each sample may have been strained to a greater extent than the rest of the sample. This differential cold working may have influenced the hardness readings to a slightdegree.

Values obtained range from 81 Rockwell B in a 2 per cent indium alloy at 40 per cent reduction to 23 Rockwell C in an 18 per cent indium alloy at 40 per cent reduction. With other methods a greater reduction might have been obtained and subsequent higher values for the hardness.

## Age Hardening

The procedure for the age hardening or precipitation hardening was relatively simple. Each of the homogenized

samples was placed into an electric furnace for a period of time ranging from 10 minutes to 32 hours, removed, and quenched. The operation necessitated a large number of samples because the process was repeated at 200°, 300°, 400° and 500°. Small samples used may have contributed to errors in hardness measurements.

A German scientist, Dr. Alfred Wilm discovered age hardening about forty years ago while he was working with the alloy "duralumin". Since that time, numerous theories have been advanced to explain this phenomenon. In 1919, Merica, Waltenberg and Scott offered the first explanation for age hardening. These investigators explained the increased resistance to slip of age hardened alloys as being due to the formation of a second phase along the grain boundaries and slip planes of the alloy.

Subsequent investigators have since offered many theories clarifying and changing the orginal explanation of Merica and his associates. Most of these discussions are conflicting and add little in material value to the earliest theory. A great number of the modern proposals

<sup>7.</sup> Age Hardening of Metals, The American Society for Metals, 1940, Cleveland, Ohio P.1

depend on the formation of one or more transition phases between the solid solution and final discrete particles of the precipitate. Conclusive evidence supporting any single theory is lacking, however.

A necessary requirement for age hardening is a decreasing solid solubility with decreasing temperature. This statement means that at a definite temperature an alloy of a certain composition of one phase only. With suitable heat treatment a second phase precipitates from the solid solution. This precipitate may form either preferentically at the grain boundaries and the slip planes or generally throughout the matrix. Despersion of the precipitate depends on the type of heat treatment. A system with an equilibrium diagram of the type shown in Fig. 2 is required for age hardening. To prepare an age-hardened alloy, one mout first homogenize a sample of the necessary composition at an elevated temperature where only one phase exists, a process called solution heat treatment. The alloy is drastically quenched from the elevated temperature to preserve the homogeneous structure. At the quenched temperature the alloy is supersaturated with respect to the second phase. To age harden

<sup>8.</sup> Age Hardening of Metals p. 343

the alloy, one must heat the sample to a temperature where both phases exist at equilibrium and hold at this temperature for the requered time. After this treatment, the alloy is again cooled to room temperature. Both time and temperature of the heat treatment are determined by the composition of the alloy and the properties required.

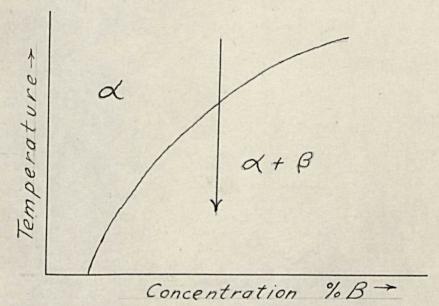


Fig. 2 - Diagram Showing Phase Relations for Precipitation

Alloys of this type may be homogeneous at room temperature but thermodynamically unstable. Therefore, when the alloy is heated to a higher temperature, stabilization of the alloy tends to take place. If the

free energy of the second phase is less than that of the initial solid solution, second phase growth is encouraged. The first phase of growth found in the copperatuminum system is Guinier-Preston zones, named in honor of their discoverers. These consist of very thin plates of second phase aggregates. The size of these platelets depends on the age hardening temperature and their diameters range in copper-aluminum alloys from less than 50A° at 25° C to 800 A° at 200° C.

On further treatment according to Wassermann and Weerts, a tetragonal lattice appears in the copper-aluminum alloys, the lattice differing from the original structure. With continued aging this transition lattice is finally transformed to the lattice of the second phase. The sequence of the precipitation may therefore be written:

Matrix - Guinier-Preston zone transition lattice -- second phase.10

This theory has been offered in explanation for low temerature hardening. Accordingly initial hardening is caused by Guinier-Preston zones, while further hardening

<sup>9.</sup> Seitz, The Physics of Metals, McGraw-Hill, 1942, New York, P. 131

<sup>10.</sup> Age Hardening of Metals, P. 403

is due to the formation of the transition lattice and the second phase lattice. These phases may overlap, but the fact that they are formed has been substantiated by X-ray analysis.

Several assumptions have been made to explain the mechanism by which the precipitated particles strengthen the matrix. Jeffries and Archer's slip interference theory states that the particles act as mechanical keys. Il A second theory is based on the deformation of the crystal lattice in the area surrounding the precipitated particle. This distortion increases the resistance to slip.

Results

The expected precipitation hardening in the alpha phase of the copper-indium system definitely appears in the graphs (pp. 31-36). The decreasing solubility of the indium in copper with decreasing temperature predicted this precipitation hardening. Because the homogenizing temperature was in excess of the maximum solubility temperature, precipitation in the high indium alloys occurred from the alpha plus beta phase rather than from homogenized alpha alone. The effect of this treatment

Metallurgy, D. W. Naostrand Co., Inc., New York, 1939, P. 71.

upon the results is not known, no attempt being made to treat other specimens for comparison. Probably the difference would not be appreciable since the amount of beta was small and may possibly have been transformed to alpha before precipitation of delta began.

A definite age hardening was evident in the alloys containing from 9-18 per cent indium with maximum hardening resulting at 200° C within the time limits employed. in the experiment. Precipitation hardening occurred sconer at the 400° and 500° temperatures, but the maximum hardness in all alloys which precipitation hardened was found at 200° C. At 200° C no appreciable hardening was noticeable, although the possibility of such hardening at longer annealing time cannot be ruled out. The age hardening curves would lead one to believe that the greatest hardness of all such alloys at 400° and 500° took place within 32 hours, the maximum time employed in the annealing. At 300° C the maximum hardness was not definitely obtained for any alloy but 6, which contains all alpha even at room temperatures and therefore should undergo no precipitation hardening.

Although many fluctuation of the precipitation hardening curves may be attributed to errors in hardness

testing, the 200° curves for alloys 2, 3, 4, and possibly 5 show a preliminary hardening. This early hardening may be a result of transition phases prior to final precipitation of delta. Other curves do not show this stage but the action may be so rapid in these cases that the results do not appear on the graphs.

The curves for alloy 6 ( see p36 ) show no precipitation hardening, but a definite hardening does occur at a short time interval, this hardening being greater at lower temperatures. One possible explanation is that weakening stresses are removed by the low temperature annealing, the process hardening rather than softening the metal. Rapid grain growth at the higher temperatures is shown by the rapid decrease in hardness.

One may conclude that within the limits of the experiment the critical size and distribution of the precipitated particles for maximum hardness occurs at 500° C.

The maximum hardness reached was in alloy 1 which had a hardness of 28.5 Rc at 16 hours.

## Recrystallization and Grain Growth

made on the cold worked samples reduced 40 per cent in cross sectional area by repeated passage through a set of

hand rolls. These deformed alloys were cut into a sufficient number of piecestto enable specimens of all to be treated at 300° and 500° for times ranging up to 2 hours in an electric furnace. On removal from the furnace, they were mounted, polished and etched for microscopic study and grain size determination.

Theory

A work hardened metal, although it has great hardness and strength, is in a state of unstable equilibrium. Distortion of the crystal lattice by cold working produces areas in which the symmetry of the atom arrangement has been disturbed. These disturbed zones have an excess of energy over the normal crystal structure. The greater the distortion in an area, the greater the excess energy.

Recovery - Three distinct processes occur on annealing a metal, the first of these operations being recovery. As the temperature of the metal is raised, the residual stresses in the deformed areas are gradually relieved. This recovery of the metal may be separable from subsequent recrystallization, or the processes may overlap. The relief of these residual stresses may be accompanied by an increase in hardness and an improvement of magnetic,

electrical, and miscellaneous properties. Recovery is not the complete relief of stress but a partial relief. The explanation offered for recovery is the straightening of bent planes and mending of microscopic cracks and regions of high stress. Although physical properties may vary, there is no change in microstructure during the recovery operation.

Recrystallization - After a cold-worked metal has been held at a sufficiently high temperature for a period of time, the energy of the distorted areas increases until it is high enough to break down the distorted lattice and form nuclei for stress-free crystals.

Softening of the metal may be sharply defined at the point of recrystallization if the material has not partially recovered previously. Grain growth may extend the change of strength and hardness properties. The rate of recrystallization is determined by the rate of formation of nuclei and the rate of grain growth. These two processes continue simultaneously until the entire metal has a new crystal structure.

Impurities which do not form solid solutions seem to

<sup>12.</sup> Metals Handbook p. 261.

have a variable influence upon recrystallization. One generalization of somewhat doubtful nature states that impurities have little effect on recrystallization temperatures. However, in alluminum alloys, such impurities, especially in a fine state of subdivision, exert a powerful influence in raising the recrystallization temperature. Soluble impurities or alloying elements definitely raise the recrystallization temperature, the effect of successive additions being less as the solution approaches saturation.

Four other factors determine the grain size after recrystallization: (1) the amount of deformation, (2) the annealing temperature, (5) the time at the annealing temperature, and (4) the original grain size. Rate of heating to recrystallization temperature and the temperature at which the original deformation is made are variables having a lesser influence on recrystallization.

Each factor may be evaluated from the viewpoint of the amount of energy which it contributes to the crystal lattice. When the energy content of the deformed lattice exceeds a certain minimum value, recrystallization takes place. Both deformation and temperature contribute to the minimum energy required for recrystallization. If a few small areas only have sufficient energy at a given temperature, few nuclei will be formed and large grains will result.

Grain Growth - On heating beyond the recrystallization point the new grains tend to grow. This fact is based on fundamental physical theory which states that at a given temperature a body tends to approach its lowest energy level, a condition characterized by a large grain size. Under ideal conditions, therefore, a metal would exist as a single crystal. The opposing force, the rigidity of the lattice is in equilibrium with the tendency to form a single crystal when the maximum grain size for a certain temperature has been attained. Undissolved impurities cause a small grain size at low temperatures and a narrow temperature range of extreme grain growth.

Generally grain size and hardness are effected by annealing temperature as shown in the following diagram.

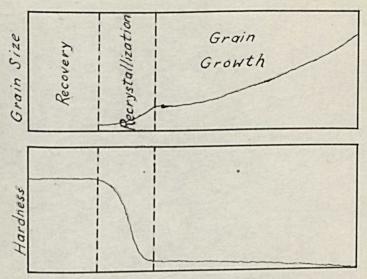


Fig. 3 Effects of Recrystallization on Hardness and Grain Size. 13

## Results

Results of the recrystallization and grain growth studies are neither consistent nor easily interpreted. Difficulties in etching alloys of higher indium content made grain size measurements impossible in some instances. At first this failure to find a suitable etching reagent was credited to the corrosion resistance of the alloy, but further study established the probability that widespread precipitation of delta from the cold-rolled alloy made the surface appear uniformly dark.

The two temperatures used for recrystallization studies were 200° C and 500° C. Since recrystallization and grain growth are usually accompanied by a softening

<sup>13.</sup> The Principles of Physical Metallurgy, P. 125

of a metal, the hardening of alloy 1 at 500° C does not conform with expected behavior. If recrystallization does not take place, there should be no substantial decrease in hardness. A possible explanation is precipitation hardening or recovery. A major decrease in hardness of alloy 1 at 500° indicates recrystallization at less than 7½ minutes. Further decrease in hardness indicates grain growth.

Alley 2 shows imcreasing grain size at 300° C between 30 minutes and 60 minutes but hardness is not materially affected. These contradictory results are probably due to errors in estimating grain size. Grain size could not be measured at 500° C, but hardness measurements show a definite softening before 75 minutes and further softening at longer times. Alloy 3 shows a decreasing grain size at 300° C, a probable error in measuring grain size, since there is no material fluctuation in hardness over the interval.

Alloys 4, 5, and 6 behave similarly to the others at 500°C; that is, they recrystallize at short annealing time with subsequent grain growth. From the graphs one can see that no recrystallization is apparent in alloys 4 and 6 at 300°C, and although recrystallization seems

indicated by grain size in alloy 5, hardness measurements show no apparent change.

In summary, the graphs show definite recrystallization at 500°C with short annealing times. At 500°C recrystallization is doubtful up to a period of 2 hours. Despite indications of recrystallization in some alloys, the fact that low indiam specimens showed no apparent grain growth at 300° contradicts any indication of recrystallization in alloys of higher indiam content. This follows from the generalization that increasing the alloying component tends to raise the temperature of recrystallization.

## Conclusions

Although no precise quantitative results were obtained in this investigation, several general facts can be stated as regards the hardening properties of the alpha phase of the copper-indium system

The solid solutions showed a definite increase in hardness with increasing indium content. The hardness increased rapidly at low indium content, but the rate of hardening was less at higher indium contents. The hardness of the specimen containing 18 per cent indium is not a true alpha phase measurement because of improper homogenizing temperatures and the resulting presence of a small amount

of beta. The measured hardness of each of these alloys may be obtained from the graph.

The cold working of these homogenized specimens to a reduction of area of 40 per cent produced alloys ranging from 80 to 100 Rockwell B. Following is a table of hardnesses of other cold-rolled copper alloys for comparison. 14

	Allo	y %	% Reduction	Hardness (Rockwell B)
70	Cu 3	O Ni	50	87
80	Cu 2	0 N1	50	83
		.66 AL	40	90
		0.02 Al	10	87
		•50 Sn	50	95
80	Cu 2	0 Zn	44	87

Recrystallization studies of this cold-worked metal proved unsatisfactory. From the data obtained, however, one can assume that recrystallization occurs for the 40 per cent deformed alloys between 300° and 500° C. No study of the effect of prior grain size on the recrystallization was attempted.

The major study involving are hardening produced insufficient results for any sweeping conclusions. The best temperature for manimum precipitation hardening occurs in the neighborhood of 500°C. Results indicte that

<sup>14.</sup> Wilkins and Bunn, Copper and Copper-base alloys, McGraw-Hill, 1943, N.Y.

maximum hardness lies far beyond the aging time of 32 hours employed in the test.

The preceding paragragraphs summarize the results of the investigation conerning alpha phase hardening and heat treating properties of copper-indium alloys. Better controlled operations and temperatures might have produced more valuable results, although present data indicates trends and may marrow further investigation. Increased time of precipitation hardening at 500° is necessary to find the maximum obtainable hardness. Studies in combined age hardening and cold working might instrument the discovery of harder copper-indium alloys, as shown by alloy I in the recrystallization data.

The color of alpha phase copper-indium alloys ranged from that of copper to a light bronze. The corrosion resistance of copper is increased by the addition of indium also. This property was evident from the difficulty found in etching some of the higher indium alloys.

The alloys are of no practical value at present, their limitations resulting mainly from the high cost and scarcity of indium and the fact that other alloys of copper having similar properties are cheaper.

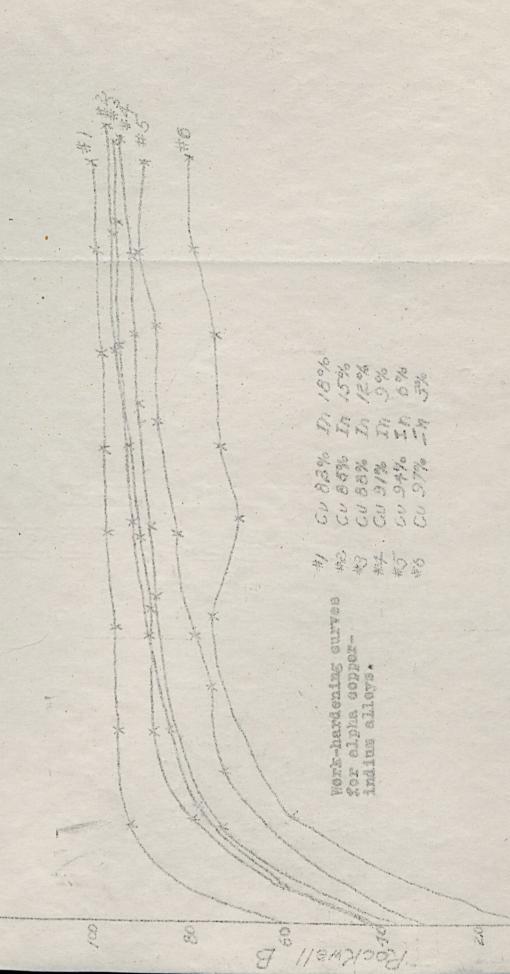
Curve showing effect of increasing indium content on hardness of alpha copper-indium alloys.

10

o 2 4 8 Atomic Percent

50

30



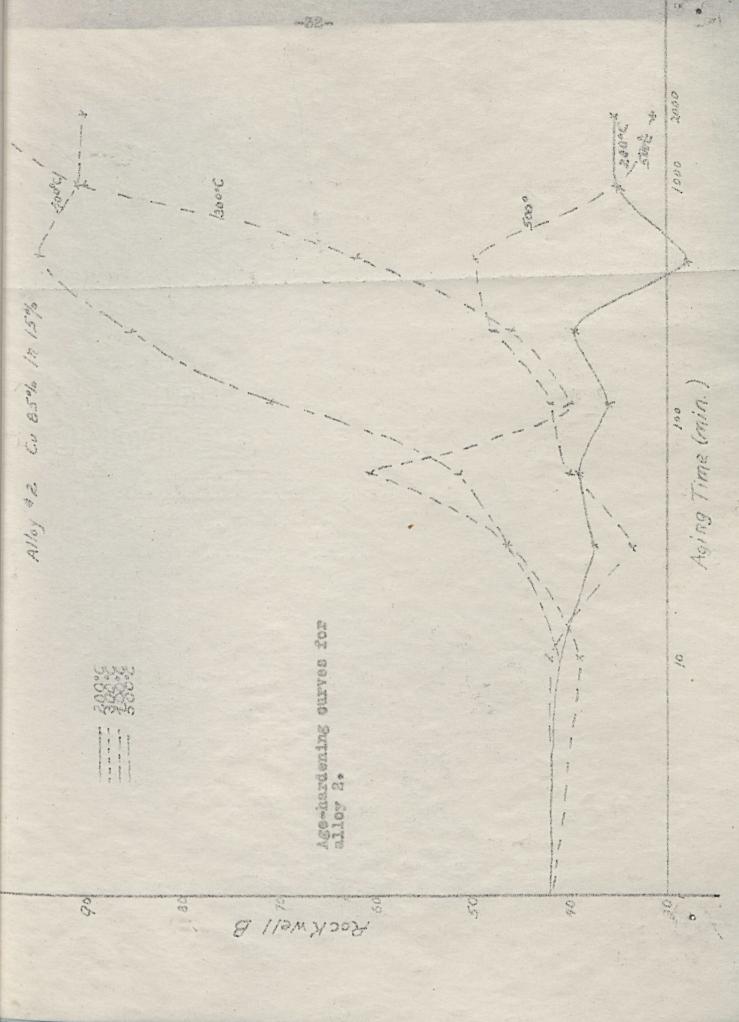
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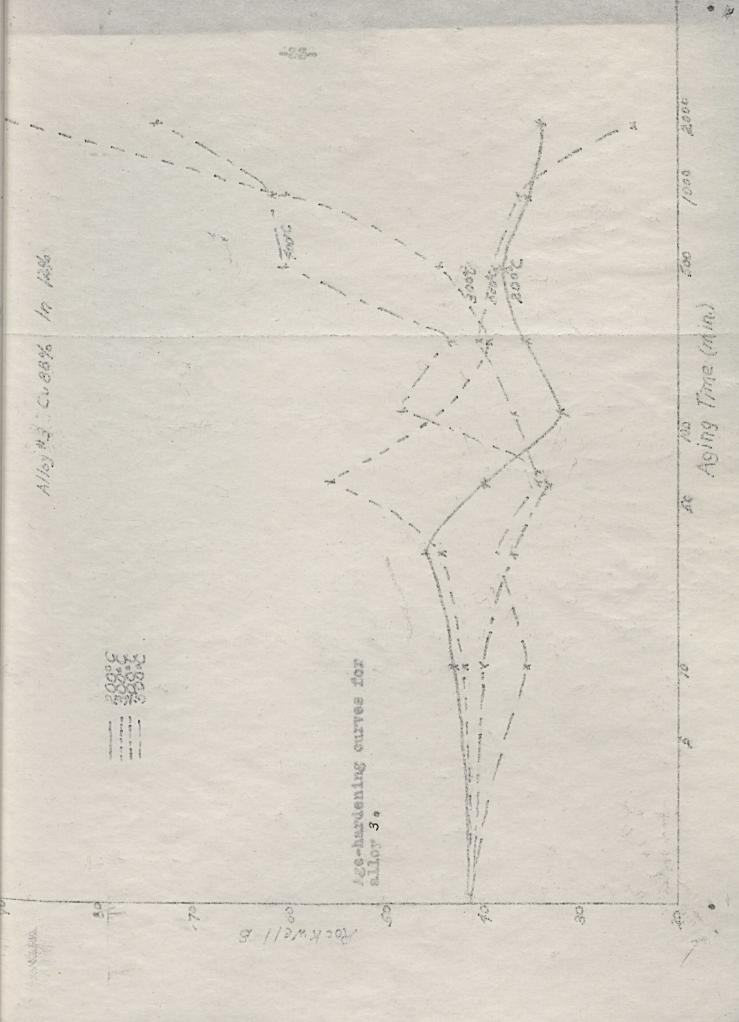
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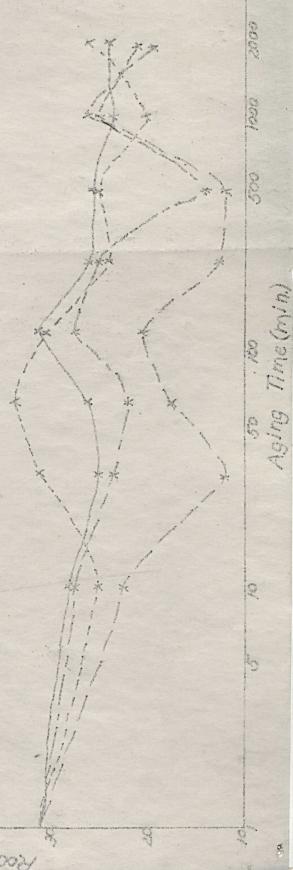


Alloy#5 60 94% D16%

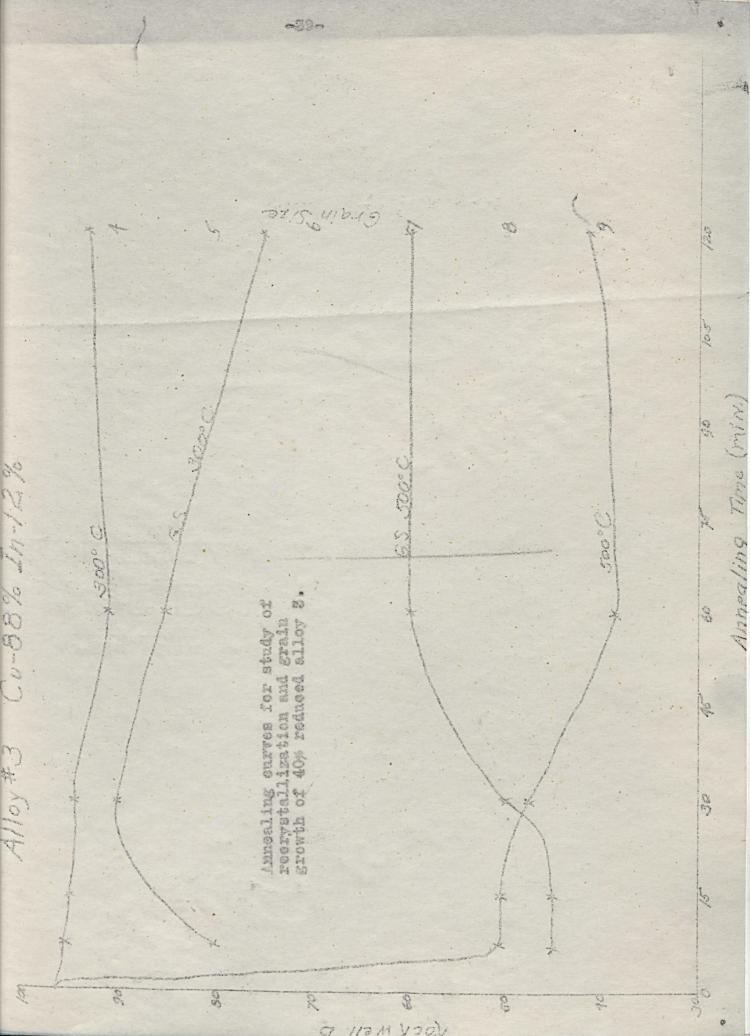
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ROCKWEII B



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