THE UNIVERSITY OF MICHIGAN
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# STUDIES IN METAL POWDERS - BEARING COMPOSITIONS

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PART II

# SYNOPSIS

With the development of powder metallurgy, iron powder finds increased applications as a raw material. A particular mention may be made of iron base bearings which are replacing their conventional counterparts in industry due to their self-lubricating properties and their superiority over other porous bearings such as the bronzes due to their higher hardness, strength and a favorable coefficient of thermal expansion which matches closely with that of the steel shafts. With this in view, a study has been made about the preparation of iron powder by electrodeposition and the utilization of this powder in the fabrication of the porous bearings.

The electrodeposition of iron powder has been carried out in a cell provided with mild steel anodes and stainless steel cathodes, employing ferrous chloride bath. The operating variables with their limits used are as follows:

| | |
|---|---|
| Concentration of the bath.... | 25 - 60 g/l |
| Temperature.................. | 22° - 45°C |
| pH........................... | 3 - 5.5 |
| Current density.............. | 40 - 150 amp./sq.ft. |
| Interval of powder removal... | 5 - 60 min. |

The effect of the following modifying agents has also been studied: β-naphthalene sulphonate, glycerine, glucose and gelatin. The powder is tested for its particle size, apparent density, flow properties and specific surface.

This powder mixed with electrolytic copper powder is used as a raw material for the study of the bearing compositions. Bearings from these powder mixtures are pressed in a specially designed die and sintered in hydrogen. The effect of the following variables on the properties of the bearings have been studied.

| | |
|---|---|
| Briquetting pressure......... | 5 - 35 tons/sq.in. |
| Sintering temperature........ | 900 - 1300°C |
| Sintering time............... | $\frac{1}{2}$ - 8 hours |
| Copper content............... | 0 - 30 percent |
| Graphite content............. | 0 - 6 percent |
| Particle size................ | 0 - 100 percent<br>(-48+100 mesh) |

The dimensional and density changes after sintering are measured and the bearings tested for their porosity and radial compressive strength.

# FOREWORD

The importance of powder metallurgy lies in the ability of this technique to produce complicated metal shapes within a close limit of tolerance[1] by a process requiring much less skill and time than machining, and much lower temperatures and fewer finishing operations than casting. Moreover the products of powder metallurgy may possess all the physical characteristics of their fused and cast counterparts as well as many that cannot be obtained thereby. Refractory metals and cemented carbides are examples of materials whose high melting points prohibit the application of fusion methods. Even the most modern methods of centrifugal casting have failed to produce products in which two or more metals insoluble in each other in solid or liquid state (such as copper and lead) are combined in a structure of uniformly distributed constituents. Yet another example is the self-lubricating bearing having a multitude of inter-connecting pores which serve as a storage for lubricating oil.

The new trend in powder metallurgy is to displace the conventional methods of production of metal parts because of the saving of time and cost. A striking example is the gear of an oil pump. By orthodox methods only skilled machinists have to be employed to prepare these articles whereas by the new technique even a layman may be trusted to operate an automatic press. Another major consideration is that in cutting a machined gear from a cast blank, about 64% of this metal is lost in chips whereas by powder metallurgy technique over 99% of the raw material is fully utilized.[2] Powder metallurgy is however no panacea and its applications are subject to certain limitations.

According to Patch[3] these limitations are based on three main factors: material, men and machines. The characteristics of the powder determine to a large extent the properties of the finished product. Balke[4] has shown the importance of purity and density for the attainment of maximum physical properties. The cost of the powder, though unimportant in small and intricate articles produced on a large scale, does form an appreciable factor when the parts are large.

The equipment used in powder metallurgy industry imposes certain other restrictions. For economical production, expensive and large presses of high capacity are essential. Another limitation is the design of the dies and punches. Strength and wear resistance of the die and the toughness and fatigue resistance of the punches have to be considered. Other limitations are imposed by the complicated and expensive equipment for the production of the powder, for conditioning them and storing without contamination by oxygen or moisture. Furnaces operated at high sintering temperatures involve many replacements.

Overcoming the above mentioned limitations calls for a knowledge which can be gained by a systematic investigation. Hence the present work was undertaken, to study the production of iron powder by electrodeposition on a semi-pilot plant scale, and to utilize the powder in the production of self-lubricating bearings and study the different factors which affect their properties.

# PART I

## STUDIES IN METAL POWDERS

## ELECTRODEPOSITION OF IRON POWDER

# I. INTRODUCTION

Iron powder forms a basic raw material in powder metallurgy. When prepared electrolytically, there are three distinctly different methods used.[5] (1) A hard brittle deposit is obtained which is subsequently ground to a powder. (2) The metal is deposited in a soft spongy condition and lightly rubbed into a powder. (3) The powder is deposited directly as such from the electrolyte. Ferrous chloride and ferrous sulphate baths have been used. A few preliminary trials showed that coherent deposits are more often obtained with ferrous sulphate bath than with ferrous chloride bath. It was therefore decided to use ferrous chloride bath and the present investigation is concerned with the direct deposition of iron powder from the chloride bath.

## Literature Survey

Several investigators have previously prepared iron powder using varied conditions of bath composition, current density and temperature. However, it is not possible to say what conditions should be employed to obtain a powder having a particular screen analysis, good flow and density properties which are the controlling factors in determining the properties of powder compacts.

Kroll[6] obtained dendritic iron of 100-300 mesh size by using molten ferrous chloride diluted with an equimolar mixture of NaCl and KCl to a concentration of about 20-30 percent ferrous chloride, a current density of 1440-2800 amp./sq.ft. and a voltage of 1-5 volts.

Stoddard[7] recommends a bath containing ferrous chloride 45-200 g/l., a temperature of 70-90°C, a current density between 50 and 300 amp./sq.ft. and a pH between 1.2 and 2.4. Addition of 0.1-20 g/l of a manganese salt serves to prevent coarsening of the grain size, and to carry out electrodeposition at a lower temperature.

Wranglen[8] used a solution containing 15 g/l of iron as ferrous chloride and 100 g/l of ammonium chloride and studied the effect of temperature from 20°C to 90°C and of pH from 2.5 to 6.5 on the nature of the deposit. He found that higher current densities tend to deposit finer powders and that the pH of the bath and hydroxide formation exert an important influence on the nature of the deposit.

Casey[9] prepared iron powder using a cell having stainless steel cathodes and iron and carbon anodes such that about 5-10% of the current passed through the carbon anodes. The bath was 30-90 g/l ferrous chloride, 10-80 g/l ammonium chloride, pH 4.0-6.3 and current density 40-200 amp/sq.ft.

Cudd and Freeman[10] produced iron powder using aqueous ferrous chloride or sulphate solution containing 250-300 g/l of the salt, a temperature of below 40°C, a current density from 25-140 amp/sq.ft. and a pH between 1.8-2.0. Modifying agents like gelatin saponins or peptones were incorporated in the bath and were found to effect a reduction in the particle size.

Primavesi[11] has described a method to produce iron powder using iron or aluminum cathodes, separated from graphite anodes by a diaphragm. The anodes are covered with iron scrap to prevent the generation of chlorine. The operating conditions are: electrolytic bath - 10-100 g/l ferrous chloride and 50-200 g/l ammonium chloride free from organic impurities, pH 3.0-6.5, temperature below 70°C and a cathode current density of 50-200 amp/sq.ft.

The following table briefly summarizes the main conditions employed by the various workers.

TABLE I

| Investigator | Extra Additions | Concentration g/l | pH | Current Density amp./sq.ft. | Temp.°C |
|---|---|---|---|---|---|
| Kroll | Na & K chlorides fused bath | 200-300 | .. | 1500-3000 | .. |
| Stoddard | Manganese salt | 40-240 | 1.2-2.6 | 25-300 | 70-90 |
| Wranglen | Ammonium chloride | 15 | 2.5-6.5 | 100-200 | 20-50 |
| Casey | " " | 30-80 | 4.0-6.3 | 40-200 | .. |
| Cudd and Freeman | Gelatin, peptone saponin | 250-300 | 1.8-2.0 | 25-140 | Below 40 |
| Primavesi | Ammonium salts | 10-100 | 3.0-6.5 | 50-200 | Below 70 |

The object of the present investigation was to study quantitatively the effect of the different controllable variables on the deposition of iron powder from ferrous chloride bath and hence obtain conditions for the deposition of iron powder suitable for use in powder metallurgy.

## Electrodeposition: Chemistry and Mechanism of the Process

### Chemistry

Electrodeposition is the most significant method for powder production of high melting point metals. The method is precisely the reversed application of the well-known electroplating process.

Iron cathodes can be used although stainless steel or monel metal serves better due to the bright surface polish and resistance to chemical attack. Mild steel is generally used for anodes though pure iron would be ideal. In some cases when chemical attack is appreciable, insoluble anodes such as graphite or lead are used together with iron.

The principal cathode reaction is the discharge of ferrous ions in the form of loose powder. However, when the supply of metallic ions from the solution becomes inadequate, hydrogen evolution invariably occurs. Due to the depletion of hydrogen ions in the cathode film, the pH is usually higher than in the bulk of the solution and hydroxide precipitation is the subsidiary reaction.

The electrodeposition in powder form occurs necessarily at less than 100% cathode efficiency, whereas the anode efficiency is generally above 100% due to anodic solution and chemical attack.

### Mechanism

Special conditions in the electrolyte cause the deposit to be in the form of loosely adherent fine crystals. The electrodeposition of the metal causes a reduction of metallic ions in the vicinity of the cathode; this is restored by mechanical movement and convection of the solution, by diffusion and by ionic migration in the electric field. The film of liquid for a distance of about 0.5 m.m. from the cathode is not readily disturbed by agitation and convection, and ionic migration is also slow (Circ. $5 \times 10^{-4}$ cm/sec/V/cm).[12] Thus, diffusion through the stationary cathode layer is the principal cause of the supply of ions. When the rate of deposition exceeds the rate of supply, a part of the current is diverted to an alternate cathode process, viz., hydrogen evolution. The metal ion concentration and the cathode potential change very rapidly across the relatively thin film of cathode solution, the result being the formation of excrescences on the cathode. These receive a higher current density, contributing to the growth of numerous tree-like, dendritic deposits which may be easily separated from the massive cathode by a slight disturbance.

Wranglen[8] discusses the factors important for the formation of powdery deposits. These are:

(1) Low metal overvoltage

(2) Depletion of the metal content of the cathode film

(3) Formation of basic material in the cathode film.

It is doubtful whether low metal overvoltage per se is responsible for powder deposition, and it may as well be claimed that low recrystallization temperature[13], low melting point or low tensile strength promote powder deposition. However, the fact remains that metals with low metal overvoltage are much more easily obtained in powder form than the high overvoltage metals. Furthermore, the other properties are found to coincide with low metal overvoltage.[14]

Under conditions favoring a high depletion of metal ions in the cathode film, the deposited metal cannot form a coherent coating, but tends to grow away from the cathode and out into the bulk of the solution where the metal ion concentration is higher. Chiefly, these conditions may be enumerated as an initial low concentration of the metal, a high current density and a low temperature.

Precipitated flocks of hydroxide present in the film of the cathode may settle down on the cathode surface and prevent it from growing, whereas the free parts of the cathode continue to grow. Precipitation of hydroxide is promoted by the use of a neutral or only slightly acidic solutions and by hydrogen evolution which causes a depletion of hydrogen ions in the vicinity of the cathode. Lower temperatures form a contributing factor due to the slower rate of diffusion.

It must be noted that only precipitated hydroxide (gel form) causes the formation of powdery or spongy deposits. Basic material in the colloidal form (sol form) in the cathode film often has a contrary effect fostering the deposition of fine-grained, smooth and hard deposits[8].

In the case of iron powder, depletion and a low cathode current efficiency are favored by the same conditions of electrolysis, viz., low temperatures, high current densities and low initial concentration of the bath. Thus, depletion and hydroxide formation are the principal causes of deposition of iron powder.

# II. EXPERIMENTAL

## Preparation and Analysis of the Solution

The electrolyte used was an aqueous solution of ferrous chloride C.P. The normal strength of the solution was 30 g/l $FeCl_2.4H_2O$. On standing, and similarly after electrolysis, the solution becomes turbid due to the slight hydrolysis of the ferrous salt. However, this does not affect the composition appreciably, and the precipitate can be easily filtered. The iron content was estimated by titration against potassium dichromate solution using potassium ferricyanide as an external indicator.

## Apparatus and Experimental Procedure

The apparatus used in these experiments was similar to that used by Joshi et al.[15] The electrolytic cell consists of a glazed porcelain rank with a bottom outlet. Stainless steel cathodes and mild steel anodes (6" x 4") spaced 2" apart are suspended from two threaded parallel rods and kept in position by nuts. The rods are mounted on brackets which are screwed to two spindles. The turning of the spindles enables the entire assembly to be raised or lowered.

The bath liquid is circulated by a pump; a heat exchanger incorporated in a circuit controls the temperature of the bath. The current is supplied by a motor generator set operating at 20 volts. A proper resistance included in the circuit enables the control of the cathode current density.

The volume of the solution used in the experiments is about six litres. The experiment is carried out for one hour, the powder being gently tapped off the cathode at intervals of ten minutes. The powder is removed from the bottom of the tank, washed free of the electrolyte and dried.

## Recovery of the Product

As it is essential to obtain a pure and unoxidized product, considerable attention has been given to the washing and drying of the powder. This is particularly important in the case of iron powder owing to its reactivity in the presence of moist air. Thus, a freshly deposited sample kept under water for a week is found to oxidize to the extent of 80%.

Among the numerous methods tried, particular mention may be made of vacuum drying, drying in a reducing atmosphere, washing the powder with antioxidants like tartrate, stearic acid, etc., incorporation of modifying agents like gelatin in the bath. Though some of these prevent oxidation to a certain extent, none of them was quite satisfactory. A method recently suggested[12]* involves washings with too many solutions and was therefore not followed. It was observed that the powder, when heated in a reducing atmosphere, loses its reactivity and can be stored without any further oxidation taking place. The electrodeposited powder is hard[12] and in order that it may be useful in powder metallurgy, it is essential to decrease its hardness by an annealing treatment in hydrogen at a temperature varying from 600-700°C, even though the powder might have been washed and dried without much oxidation taking place during drying. Hence, the procedure followed was to heat the wet powder, washed free of the electrolyte, to a temperature of about 650°C in an atmosphere of hydrogen, special care being taken to remove the final traces of oxygen from hydrogen. Thus, both drying as well as annealing was achieved in one operation.

It must be mentioned that the temperature used in drying also affects the particle size. If the temperatures are those at which caking takes place, then much coarser particles result, and powders which are apparently non-flowing may have an appreciable flow rate. At the temperature used for drying (650°C), caking of the powder was not noticeable.

## Powder Characteristics

The iron powder so prepared was in each case tested for purity, particle size distribution, apparent density, flow rate and specific surface.

### Purity

The purity of the powder was determined by estimating the percentage of metallic iron in the sample.[16] The powder is treated with mercuric chloride solution whereupon the metallic iron reacts to form ferrous

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*A procedure referred here[12] consists of the following scheme. The product is washed first with tap-water and the supernatant liquor decanted, then with 1% sulphuric acid and then again with tap-water. A small quantity of 10% citric acid solution is mixed and allowed to stand for a few minutes. The mixture is again washed with tap-water and then dilute ammonia added. The wet powder is thoroughly washed by decantation with tap-water until the supernatant liquor is colorless, and finally with distilled water after which it is rapidly vacuum filtered, rinsed with acetone and rapidly dried either on a hot plate or preferably in vacuum or dry hydrogen or coal gas. This involves too many washings and was therefore not followed.

chloride, the mercuric salt forming calomel or metallic mercury. Any other impurity including iron oxide remains unaffected. The mercurous salt or any mercury so formed must be filtered off before the solution is titrated against standard dichromate. The metallic iron content was usually found to be above 96% and the total iron content about 98.5 to 99%.

## Particle Size Distribution

Particle size distribution is important in all powder utilizations, particularly in molding practice. A mechanical sieve shaker with Tyler screens was employed. The results are represented on the basis of median particle size which is the average particle size of the sample.

## Apparent Density

Apparent density is important from the aspect of die construction and its influence on the characteristics of the finished product. It was determined according to A.S.T.M. specifications.[17]

## Flow Rate

Flow rate is the time required (sec.) for a powder sample of standard weight to flow through an orifice in a standard instrument according to a specified procedure. The apparatus used was the same as the one employed to find the apparent density. The results are based on the time required in seconds for the flow of 25 g. of the dried powder.

## Specific Surface

Specific surface is the surface area of one gram of the powder usually expressed in sq. cm. Powder metallurgy technique is fundamentally based on this factor. That it is related to particle size is obvious. Finer the particle size, the larger the surface area. Apart from this, it depends on the history of the powder. The one prepared electrolytically has the largest specific surface due to its dendritic porous structure.

The apparatus used for the determination of the specific surface was a modification of the air permeability method, the one designed by Lea and Nurse.[18] The specific surface is calculated using Carmen's equation.[19]

## III. RESULTS

### General Plan of Work

The general plan of work and the experimental procedure adopted in these investigations are as follows:

A. Preliminary studies on the range of concentration and current density giving satisfactory deposits

B. Detailed study of the influence of:

  (1) Current density
  (2) Iron concentration
  (3) Temperature of the bath
  (4) pH of the bath
  (5) Interval of powder removal
  (6) Addition agents on:

    (a) Powder current efficiency
    (b) Particle size distribution
    (c) Apparent density
    (d) Flow rate
    (e) Specific surface

C. Studies of the drying conditions on the characteristics of the powder.

### Preliminary Studies

Preliminary studies showed that the deposition range for iron directly in the powder form is narrow. Powder deposition was found to be unsatisfactory at low current densities. Low iron concentrations gave a powder of a spongy nature, whereas high concentrations deposit a mixture of coherent and powdery iron.

### Detailed Studies

The following table gives the values of the conditions held constant during each series of experiments and the extreme plausible limits of the operating variables.

TABLE II

| Variable | Fixed Value | Extreme Limits of Variation |
|---|---|---|
| Current density | 90 amp./sq. ft. | 40-150 amp./sq. ft. |
| Iron conc. as $FeCl_2 \cdot 4H_2O$ | 30 g/l | 25-60 g/l |
| Temperature | 35°C | 20-45°C |
| pH | 4.5 | 3 - 5.5 |
| Interval of powder removal | 10 min. | 5-60 min. |

The results obtained by employing the different operating variables are given in Tables III to VII and the results are shown graphically in Figures 1 to 6.

Experiments have been carried out in the presence of addition agents like glucose, glycerine, sodium β-naphthalene sulphonate, and the results are given in Table VIII.

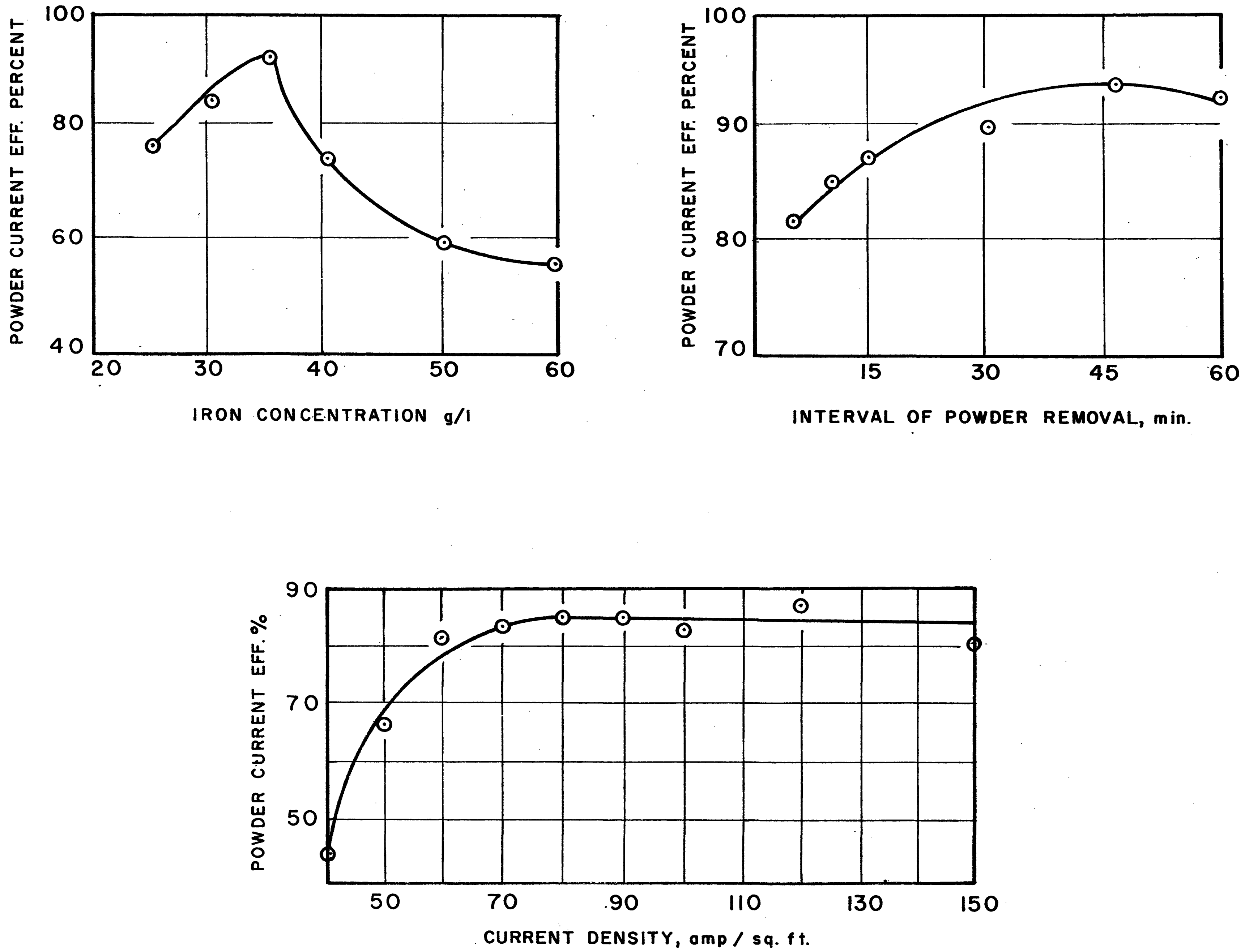

FIG I. EFFECT OF THE VARIABLES ON POWDER CURRENT EFFICIENCY

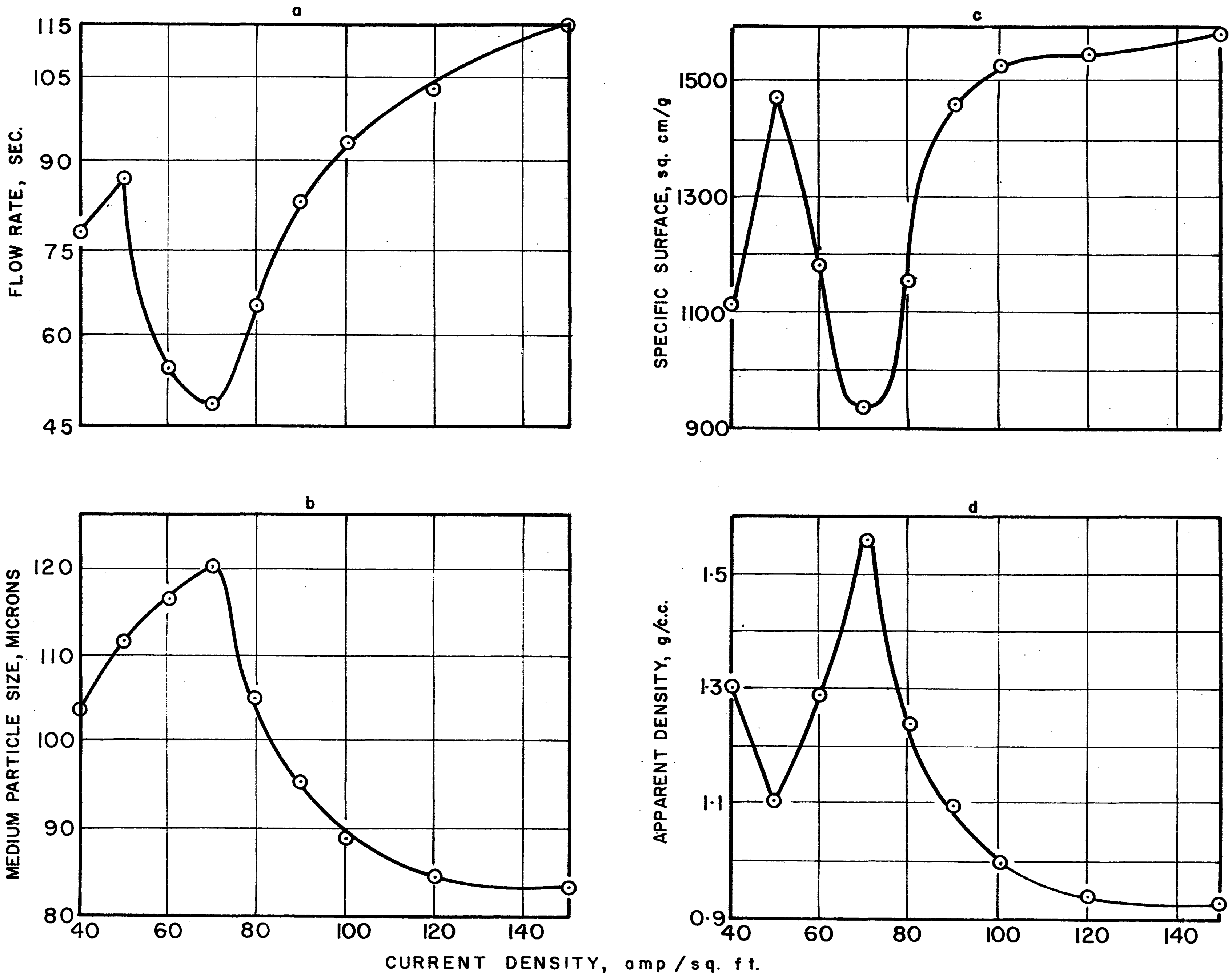

FIG 2 EFFECT OF CURRENT DENSITY ON POWDER CHARACTERISTICS.

# TABLE III

## EFFECT OF CATHODE CURRENT DENSITY

Concentration of the bath - 30 g/l $FeCl_2.4H_2O$ ; pH - 4.5 ; Temperature - 35°C

Interval of powder removal - 10 min.; Temperature of drying - 650°C under hydrogen

| Current density amp/sq.ft | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm/g. | Remarks |
|---|---|---|---|---|---|---|
| 40 | 45.0 | 103.2 | 1.30 | 7.75 | 1112 | Codeposition of coherent and loose powder |
| 50 | 67.7 | 113.2 | 1.10 | 8.7 | 1471 | Powdery |
| 60 | 81.9 | 116.4 | 1.29 | 5.4 | 1183 | " |
| 70 | 83.5 | 120.2 | 1.56 | 4.8 | 933 | " |
| 80 | 85.5 | 105.2 | 1.24 | 6.5 | 1100 | " |
| 90 | 85.1 | 95.2 | 1.10 | 8.3 | 1460 | " |
| 100 | 83.3 | 89.8 | 1.00 | 9.4 | 1530 | " |
| 120 | 87.5 | 84.8 | 0.94 | 10.3 | 1550 | Excessive evolution of hydrogen and heat |
| 150 | 80.0 | 83.2 | 0.92 | 11.8 | 1590 | |

The results are presented graphically in Fig. 1 and Fig. 2.

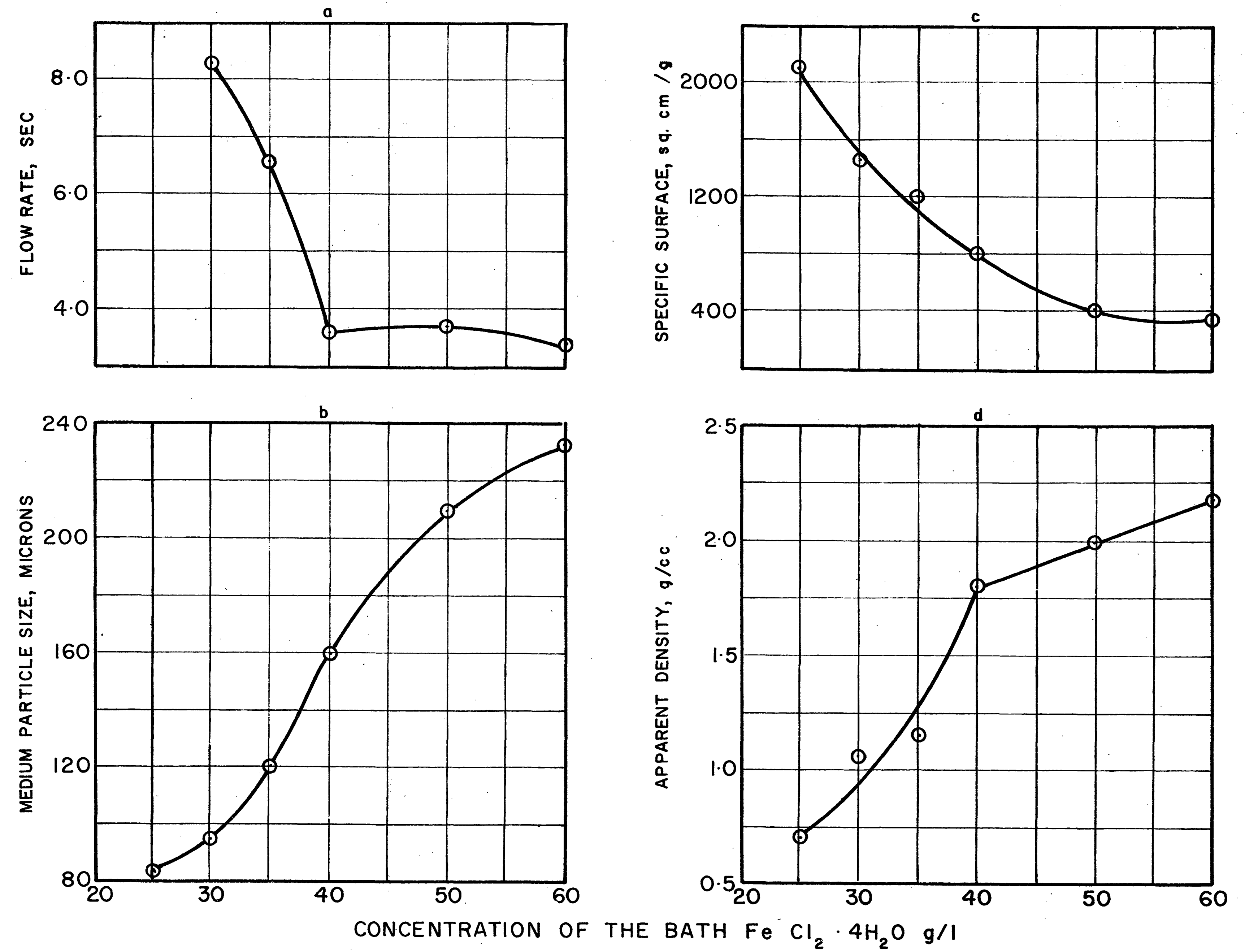

FIG 3 EFFECT OF IRON CONCENTRATION ON POWDER CHARACTERISTICS

# TABLE IV

## EFFECT OF THE CONCENTRATION OF FERROUS CHLORIDE IN THE BATH

pH - 4.5
Temperature - 35°C
Cathode current density - 90 amp./sq.ft.
Interval of powder removal - 10 min.
Temperature of drying - 650°C under hydrogen

| Conc. g/1 | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm./g | Remarks |
|---|---|---|---|---|---|---|
| 25 | 77.5 | 85.0 | 0.715 | xx | 2183 | Spongy |
| 30 | 85.1 | 95.2 | 1.10 | 8.3 | 1460 | Powdery |
| 35 | 92.5 | 121.5 | 1.15 | 6.6 | 1200 | Powdery |
| 40 | 74.5 | 161.2 | 1.8 | 3.6 | 618 | Mixture of coherent and loose powder |
| 50 | 60 | 210 | 2.0 | 3.7 | 407 | |
| 60 | 55 | 233 | 2.17 | 3.5 | 359 | |

The results are presented graphically in Fig. 1 and Fig. 3.

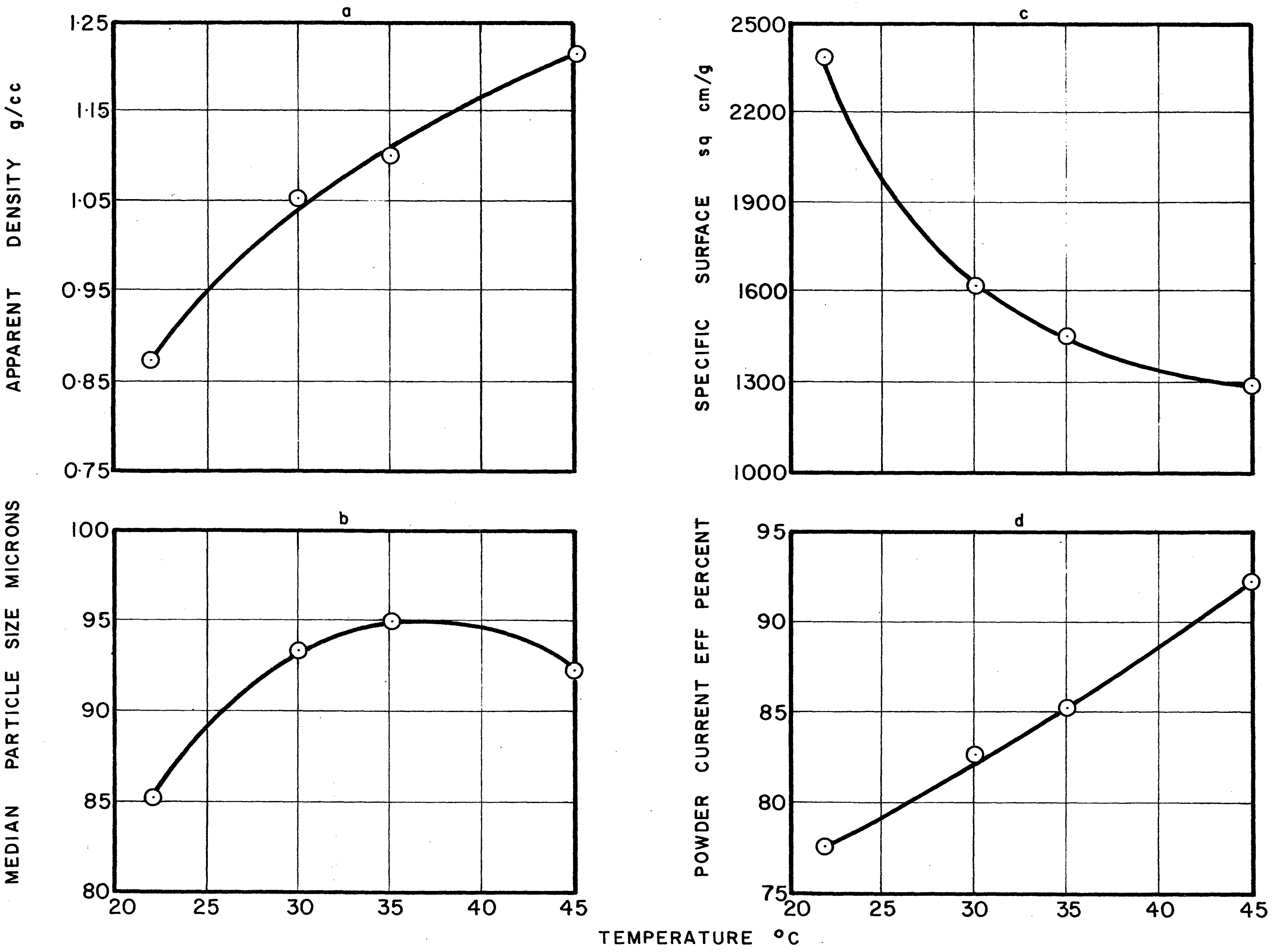

FIG 4 EFFECT OF TEMPERATURE ON POWDER CHARACTERISTICS

## TABLE V

## EFFECT OF TEMPERATURE OF THE BATH

Concentration of the bath - 30 g/l $FeCl_2 \cdot 4H_2O$

Current density - 90 amp./sq.ft.

Interval of powder removal - 10 min.

Temperature of drying - 650°C under hydrogen

pH - 4.5

| Temperature °C | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm./g | Remarks |
|---|---|---|---|---|---|---|
| 22 | 77.5 | 85.2 | 0.875 | x x | 2379 | Spongy deposit<br>73% Fe metallic |
| 30 | 82.5 | 93.3 | 1.04 | 9.3 | 1620 | Powdery |
| 35 | 85.1 | 95.2 | 1.10 | 8.3 | 1460 | " |
| 45 | 92.3 | 92.1 | 1.22 | 6.6 | 1288 | " |
| 55 | x x | x x | x x | x x | x x | Coherent |

The results are presented in Fig. 4

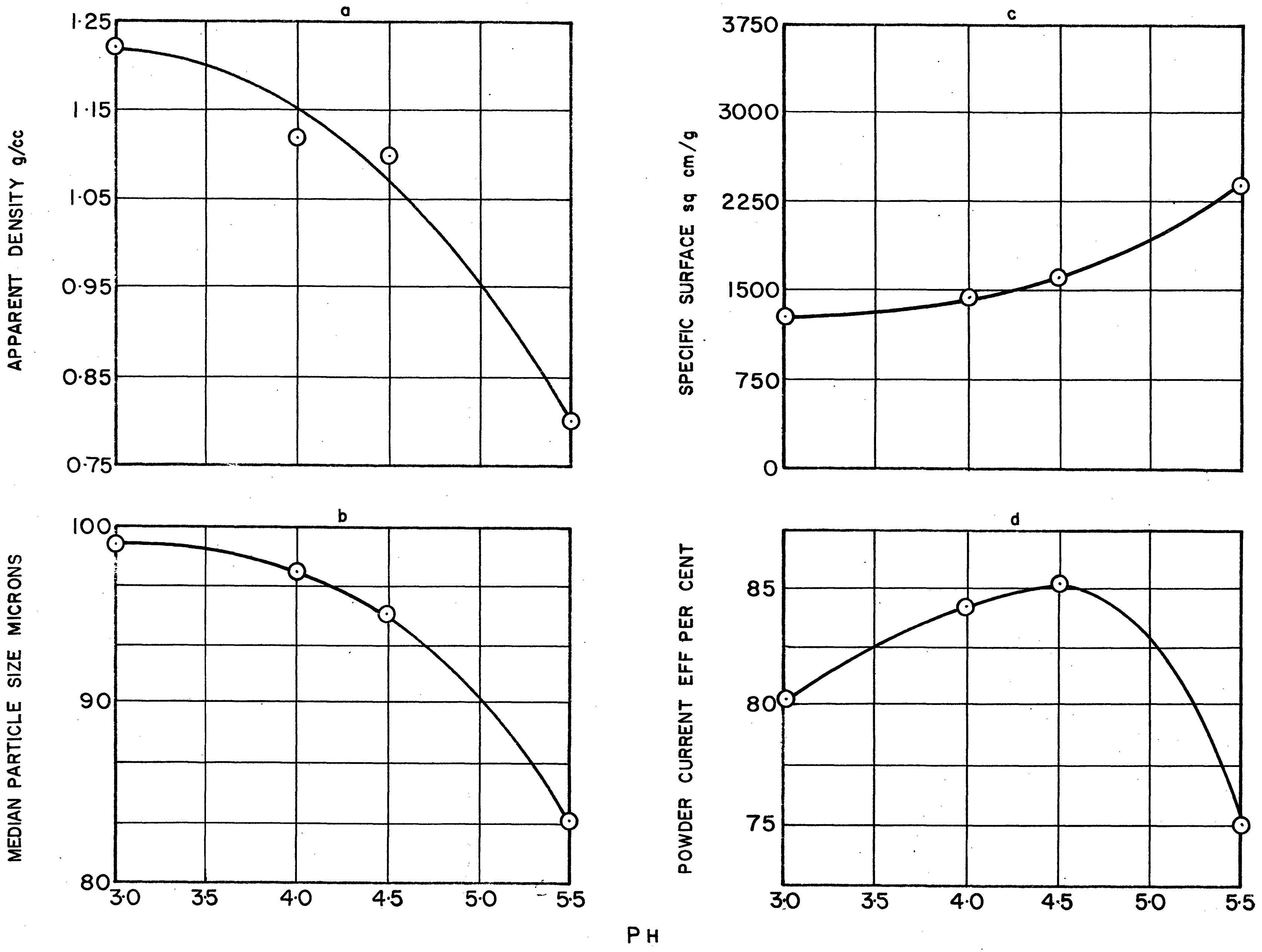

FIG 5 EFFECT OF PH OF THE BATH ON POWDER CHARACTERISTICS

## TABLE VI

## EFFECT OF THE VARIATION OF THE pH OF THE BATH

Concentration - 30 g/1 $FeCl_2.4H_2O$
Current density - 90 amp/sq.ft
Interval of powder removal - 10 min.
Temperature - 35°C
Temperature of drying - 650°C under hydrogen

| pH | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm./g | Remarks |
|---|---|---|---|---|---|---|
| 2.5 | xx | xx | xx | xx | xx | Mixture of coherent and loose powder |
| 3.0 | 80.3 | 99.0 | 1.22 | 6.1 | 891 | Powdery |
| 4.0 | 84.0 | 93.5 | 1.14 | 7.8 | 1400 | " |
| 4.5 | 85.1 | 95.2 | 1.10 | 8.3 | 1460 | Addition of acid not necessary |
| 5.5 | 75.1 | 83.5 | 0.795 | xx | 2951 | Basic material deposited 68% Fe(met.) |

The results are presented graphically in Fig. 5

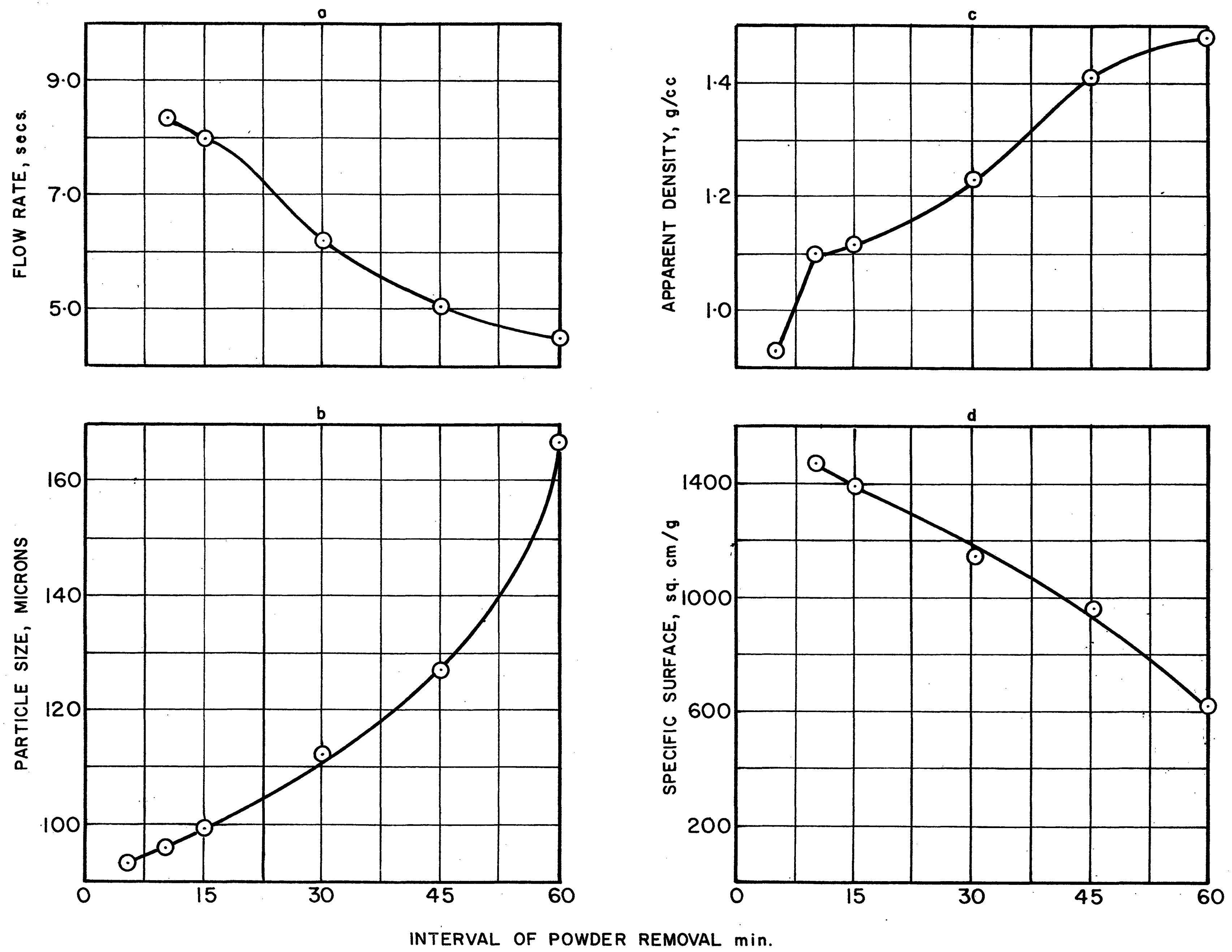

FIG 6 EFFECT OF INTERVAL OF POWDER REMOVAL ON POWDER CHARACTERISTICS

## TABLE VII

## EFFECT OF INTERVAL OF POWDER REMOVAL

Concentration of the bath - 30 g/l
Current density - 90 amp./sq.ft.
Temperature of the bath - 35°C
Temperature of drying - 650°C under hydrogen
pH - 4.5

| Interval of powder removal min. | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm/g | Remarks |
|---|---|---|---|---|---|---|
| 5 | 82 | 93.3 | 0.93 | x x | 2603 | Spongy |
| 10 | 85.1 | 95.2 | 1.10 | 8.3 | 1460 | Powdery |
| 15 | 87.5 | 99.2 | 1.11 | 8.0 | 1395 | " |
| 30 | 90.5 | 112.9 | 1.29 | 6.2 | 1123 | " |
| 45 | 94.0 | 126.5 | 1.47 | 5.0 | 964 | " |
| 60 | 93.0 | 166.6 | 1.48 | 4.5 | 620 | " |
| 120 | xx | xx | xx | xx | xx | Hard, very coarse |
| 180 | xx | xx | xx | x x | xx | Coherent |

The results are presented graphically in Fig. 1 and Fig. 6

## Addition Agents

In electroplating practices of various metals, various organic as well as inorganic additions have been found to improve the plating from a coarse to a fine-grained structure. Their effects, however, are yet to be thoroughly understood so that their use is purely empirical. The effect is attributed to their influence upon the polarization or cathode potential and structure of the metal deposit.

There are basically no fixed rules for addition agents. They may be specific in their action, i.e., an addition agent that works with one metal does not necessarily work with another. Various other factors like the pH of the bath have also some influence. Instead of a single addition agent, two or more are sometimes needed in combination to produce the desired effect.

The subject is thus a wide one and the practice is one of trial and error. In the present series of investigations, the action of glucose, glycerine and sodium naphthalene sulphonate has been studied in details and qualitative observations made on the influence of gelatin, saponin and peptone. The results are presented in Table VIII on the following page.

## Drying Conditions

It must be mentioned that the temperature used in the drying and annealing the powder also affect the particle size. When the temperatures are those at which caking takes place, much coarser powders result and powders which are apparently non-flowing may have an appreciable flow rate. An atmosphere of hydrogen is preferable because it presents a reducing atmosphere, imparts a bright appearance to the powder and on the whole improves the powder characteristics. City gas and nitrogen were tried but the appearance of the powder was dull and the flow properties poor.

## TABLE VIII

## EFFECT OF ADDITION AGENTS

Concentration of the bath - 30 g/l, $FeCl_2.4H_2O$

Current density - 90 amp./sq.ft.

Temperature - 35°C; pH - 4.5

Interval of powder removal - 10 min.

Temperature of drying-650°C under hydrogen

| Addition Agent | Addition Agent concentration percent | Powder current efficiency percent | Median particle size microns | Apparent density g/c.c. | Flow rate secs. | Specific surface sq.cm./g |
|---|---|---|---|---|---|---|
| Nil | Nil | 85.1 | 95.2 | 1.10 | 8.3 | 1460 |
| Sodium naphthalene sulphonate | 0.05 | 91.5 | 122.8 | 1.56 | 5.0 | 545 |
| | 0.10 | 94.0 | 114.1 | 1.305 | 5.9 | 760 |
| | 0.25 | 81.0 | 87.2 | 1.03 | 12.0 | 890 |
| | 0.50 | 73.2 | 77.3 | 0.87 | 14.3 | 1040 |
| Glycerine | 0.05 | 91.6 | 142.4 | 1.664 | 5.2 | 575 |
| | 0.10 | 90.5 | 150.2 | 1.517 | 5.6 | 639 |
| | 0.25 | 81.0 | 110.2 | 1.142 | x x | 1390 |
| | 0.50 | 80.0 | 104.0 | 0.892 | x x | 1970 |
| Glucose | 0.05 | 92.3 | 133.0 | 1.215 | 8.8 | 1170 |
| | 0.10 | 80.7 | 116.2 | 0.950 | x x | 2020 |
| | 0.25 | 78.3 | 80.3 | 0.72 | x x | 2220 |
| | 0.50 | xx | xx | xx | x x | xx |

## IV. DISCUSSION OF RESULTS

### Powder Current Efficiency

Considering the process of electrodeposition, at low current densities, the discharge of ions occurs slowly, and so the rate of growth of nuclei exceeds the rate at which the new ones form. This gives a coarse-grained deposit. As the current density is raised, the rate of formation of nuclei will be greater and the deposit will be fine-grained. At very high current densities, the rate of discharge of ions is very fast, so that in the vicinity of the cathode, there is depletion of the ions required for discharge and, as a result, the crystals tend to grow out into the bulk of the solution. It is at this stage that the metal deposits in the form of dendritic, tree-like powder.

It may be noted that below a current density of 50 amp./sq.ft. the operation is not plausible due to the codeposition of a coherent and powdery deposit. Below this value of the current density, it appears that the rate of discharge of ions is not fast enough to cause sufficient depletion of cations in the vicinity of the cathode so that dendritic outgrowth is not very much favored.

With an increase in current density above this value, conditions favor depletion and hence facilitate powder deposition, until the powder current efficiency reaches an optimum value of 85.5 percent at a current density of 80 amp./sq.ft. In this range, hydrogen bubble evolution, by obstructing crystal growth, has the same effect as depletion, in promoting powder deposition. Thereafter, evolution of hydrogen and heat is excessive so that the powder current efficiency decreases to 80% at 150 amp./sq.ft.

The change in the concentration of the electrolytic bath has the most deciding influence. It provides a very limited range for the proper deposition of the powder. At low concentrations, viz. 25 g/l, the current-carrying capacity of the solution is low and the secondary reaction, viz. hydrogen evolution, is encouraged. This increases the pH of the cathode film solution which, with the high depletion of cations, deposits a very spongy powder with basic material and with a low current efficiency of 77.5 percent. Increasing the concentration increases the rate of diffusion and accelerates powder deposition process giving an optimum value of 92.5 percent powder current efficiency at a concentration of 35 g/l. At a concentration of 40 g/l, the deposit is a mixture of coherent and loose powder and any further increase in the concentration increases the amount of the coherent plating, and decreases the amount of loose powder (Fig. 1).

The influence of concentration on the rate of formation of new nuclei is uncertain. Certain workers hold the opinion that the presence of a large number of ions in a concentrated solution favors the formation of fresh nuclei. Certain experiments by Glazunov[21], however, indicate that the rate of formation of nuclei is actually decreased by increasing concentration, but the plating is due to an increase in the rate of crystal growth across the cathode surface, combined with a decrease in the rate of growth in a perpendicular direction.

Increase in temperature has two effects which oppose each other. In the first place, diffusion is favored so that depletion is counteracted and coherent deposits encouraged; on the other hand, hydrogen overvoltage is decreased so that gas evolution is encouraged which facilitates powder formation. The ultimate effect depends upon which factor is predominant. At moderate temperatures, it is the first factor that has the deciding influence, whereas the second factor is important only in electrolytic baths of fused salts.[8] At 22°C the powder is very spongy and the current efficiency is 77.5 percent. It increases to 92.3 percent at 45°C (Fig. 4d ) above which the deposit attains a coherent nature.

The pH of the bath has a peculiar effect on the powder current efficiency. As can be seen from Fig. 5d, an optimum value of the electrolytic bath maintains itself without the addition of any acid. At a higher pH, the deposit is spongy, basic material is precipitated in a large quantity and the efficiency falls to 75.1% at 5.5 pH. At a value of pH below 3.0, coherent powder is deposited and the operation is not plausible.

The effect of interval of powder removal on the powder current efficiency is shown in Fig. 1. It can be noted that as the powder formation continues undisturbed, the current efficiency increases. More energy seems to be spent on the formation of fresh nuclei than in the growth of the previously formed powder crystals. The efficiency increases to a peak value of 94 percent at 45 min. interval and is very nearly the same at 60 min. interval.

On the whole it is found that the powder current efficiency is low for a spongy deposit and increases as the powder becomes coarser in nature and suddenly decreases when the deposit tends to be coherent.

## Particle Size and Apparent Density

The increase in current density from 40 amp./sq.ft. to 70 amp./sq.ft. increases the particle size of the deposit. Further increase in current density causes a deposition of fine-sized powder (Fig. 2b). In most powder deposition processes, the current density is above the limiting value for the given electrolyte and hydrogen evolution is the side reaction. As the current density is increased, the hydrogen evolution is increased, and the bubbles interfere with the crystal growth, thus giving a fine-sized powder. Secondly, due to the discharge of the hydrogen ions, the cathode film solution tends to be alkaline and deposit basic material which is the second factor in the formation of fine-sized crystals. This is illustrated by the current density range above 70 amp./sq.ft. Up to this value, the hydrogen evolution is not sufficient to interfere with crystal growth and the particle size increases between 40 amp./sq.ft. and 70 amp./sq.ft.

With increase in iron concentration, the rate of formation of new nuclei is decreased and there is an increase in the rate of growth of crystals.[21] This is borne out by the experimental results by the increase in particle size with increase in concentration (Fig. 3b). With increase in temperature, the effective thickness of the diffusion layer is decreased, and this favors an increase in particle size (Fig. 4b). With increase in the pH of the bath, the cathode film solution tends to go far in the alkaline region and deposits hydroxides. This causes the deposit to be fine-grained and spongy (Fig. 5b). It may be mentioned that in all the cases, as the particle size increases, the powder becomes hard and compact and ultimately becomes coherent. This is well illustrated when the interval of powder removal is steadily increased. Up to an interval of powder removal of one hour, the powder falls freely from the cathode. At the end of two hours, it tends to cling to the electrode, while if allowed to grow undisturbed for three hours, it forms an adherent plating.

Being closely related to particle size and shape, (the dendritic or tree-like shape is peculiar to all electrolytic powders), the changes in apparent density are similar to those in particle size. However, when the powder is codeposited with an adherent plate, and similarly when the particle size is large, the particle shape becomes rounded instead of the characteristic dendritic shape. In these cases, there are apparent irregularities in the nature of the curves of particle size and apparent density. Figs. 2b and 2d show that at 40 amp./sq.ft. current density, the powder is codeposited with a coherent deposit, and though the particle size is smaller than that at 50 amp./sq.ft., the apparent density is larger. A similar effect can be observed in Figs. 4a and 4b. At 45°C, although the particle size is smaller, the apparent density is larger than at 35°C.

## Specific Surface and Flow Rate

Since flow rate and specific surface are related to apparent density, it may be noted that specific surface and flow rate curves are similar (Figs. 2c, 3c, 4c, 5c and 6d, and Figs. 2a, 3a, 4a) and the curve characteristics are just the reverse of those for apparent density.

## Effect of Addition Agents

Among the various addition agents tried, gelatin, saponin and peptone were found to have similar influences on the deposition of the powders. These effects may be briefly summed up as follows:

(a) The solution becomes viscous.

(b) A large amount of stable froth is formed at the cathodes with the powder getting entrapped in hydrogen bubbles collecting at the surface and thereby getting oxidized to a certain extent.

(c) Hydroxides separate from the solution and washing the powder is difficult and slow.

(d) The range of the powder is increased with the use of these reagents. Thus a good powdery deposit is obtained from a solution of 60 g/l iron content with the use of 0.05 percent gelatin. Similarly, a powdery deposit is obtained by using 0.25 percent gelatin in a solution containing 120 g/l of iron chloride. The particle size also becomes increasingly finer with increased additions of these reagents, if other conditions are maintained constant. The addition agents are surface active substances and are adsorbed on the crystal nuclei, thus preventing their growth; the discharged ions are consequently compelled to start new nuclei and the result is the fine-grained deposit.

Sodium naphthalene sulphonate, glycerine and glucose were found to be quite suitable and produced no froth. The effect of these addition agents on the powder characteristics is given in Table VIII. Special mention may be made of sodium naphthalene sulphonate. A concentration of 0.05 - 0.1 percent improves the flow characteristics, the average particle size and the apparent density. Within this range, the powder current efficiency is also high and increases as the percentage of the addition agent increases.

# V. CONCLUSIONS

The following conclusions may be drawn from the present investigations:

(1) The range of conditions for direct deposition of iron in powdery form from ferrous chloride solution is narrow. For the conditions employed it was found that below a current density of 50 amp./sq.ft., the operation is not plausible because of a coherent deposit. An optimum current efficiency of 85.5 percent is obtained at 80 amp./sq.ft. Above this, the current efficiency decreases due to an increase in the evolution of heat and hydrogen.

(2) The important factor is the concentration of the bath. The optimum concentration is found to be 35 g/l (though 30 g/l was also quite satisfactory). More dilute bath gives a spongy powder which presents difficulty in processing while a more concentrated bath gives a mixture of coherent and powdery deposit.

(3) Coherent deposits are favored by low cathode current density, high temperature and high acidity. Lesser depletion of ions in the cathode film solution and higher rate of diffusion or diminishing of the thickness of the diffusion layer are factors leading to coherent deposits.

(4) The maximum value of powder current efficiency is obtained at 45°C at a pH of 4.5 (the pH of the electrolytic bath without any addition of acid), and when the interval of powder removal is long enough above 30 min.

(5) The median particle size and the apparent density increases with iron concentration, temperature, interval of powder removal and with current density up to an 80 amp./sq.ft. beyond which these decrease with a further increase in current density. The flow rate and the specific surface decrease in close conformity with increase in particle size and apparent density. The powders codeposited with coherent platings show abrupt changes in all their characteristics.

(6) A low concentration such as 0.05 - 0.1 percent of sodium naphthalene sulphate, glycerine and glucose, improves the flow properties, increases the average particle size, apparent density and the powder current efficiency. Gelatin, saponin and peptone, though they give better powdery deposits and increase the range of deposition, are not suitable due to the formation of froth and due to washing difficulties on account of the higher viscosity of the solution.

# PART II

## BEARING COMPOSITIONS

## IRON-COPPER POWDER BEARINGS

# I. INTRODUCTION

The production of porous bearings is a natural application of powder metallurgy since all its important processing methods lead to materials which are porous per se. The two principal uses of pores in metals are as storage space for a liquid auxiliary substance such as oil or grease lubricants, or as separating cells for impurity-containing liquids. To the latter category belong the porous filters, diaphragms and similar products, while the porous bearings and bushings constitute the former. To fulfill the purposes, the pores must be intercommunicating in character as well as of carefully controlled shape, size and distribution. Pressing and sintering have been found to suit ideally to produce these very desirable properties. The pore volume incorporate in the bearings varies from about 15-40% by volume, but is usually in the neighborhood of 25%. Depending upon the bearing composition and the nature of oil, between 4-6% by weight of oil is needed for a pore volume of 25% to be satisfied. Although in certain cases provision has to be made to furnish lubrication from outside, in most applications the lubrication is produced from within the bearing itself.

## Mechanism of Self-Lubrication

The underlying principle of self-lubrication is briefly as follows. When the motor is started, the rotating shaft brings the oil to the surface by a direct as well as an indirect way. Firstly, the rotating motion of the shaft causes a pumping action. Secondly, as Bowden and Ridler[22] have found out, the bearing surface temperatures shoot to surprisingly high altitudes, in some cases between 600°C to 1000°C, even though the mass of the metal remains cool. Such frictional heat is the second aid to the flow of oil from pores to the surface. At the interface between the shaft and the bearing, the oil forms a protective film preventing direct metal-to-metal contact. A steady supply of oil is furnished from the pores to the entire bearing surface, resulting in a continuous and uniform oil film, even at low rotating speeds. The thickness of the film is automatically regulated by the surface temperature which in turn is governed by the load and the rotating speed. Upon completion of the running cycle, the oil is re-absorbed into the interior of the bearing by capillary forces and the cooling of the bearing and the housing. However, a thin film is retained by surface tension, thus preventing metal-to-metal contact at all times. This alternate action is repeated every time the motor starts or stops and, during running, the bearing furnishes adequate lubrication for a long time without losses from dripping or leakage.

## Bearing Compositions

Formerly, it was believed that in a bearing alloy, there should be a fine distribution of hard compounds in a relatively soft matrix. The basic theory for this was that the hard components would support the load and the softer material, wearing away slightly, would form a reservoir for oil. This has since been proved incorrect and today it is felt that any metal or alloy can function as a bearing so long as it possesses certain properties to a degree as outlined by Mougey[23], viz. mechanical strength, bonding characteristics, high melting point, fatigue resistance, antifriction properties, nonscoring characteristics, conformability, embedability, thermal conductivity and corrosion resistance. Tin and lead form the bases of two important groups of bearing alloys; other metals such as copper, cadmium and aluminum are used in other types. Among the porous bearings and bushings, copper-tin compositions were originally used exclusively and are still used most commonly. Their main applications have been in small motors of fractional horsepower capacity. However, they have an inherent weakness in their low strength so that frequent replacements are necessary on appliances of heavy loads. As a remedy to this, the iron base bearing is a more recent improvement and forms the subject of investigation in this thesis. These have distinct merits in their higher hardness and strength as compared to the bronzes and possess a favorable coefficient of thermal expansion which matches with that of the steel shaft. Langhammer[24] compares the properties of bronze and iron-copper bearing materials produced by Amplex Division, Chrysler Corporation, U.S.A., and the results show that for the same specific gravity, the iron-copper bearing material has two and a half times the ultimate strength, one and a half times the Brinell hardness, three times the resistance to permanent deformation, and much smaller coefficient of thermal expansion although the porosity was about 5% less than the bronze bearing material.

## Literature Survey

For the manufacture of bearings by the powder metallurgy method, iron and copper powders are combined in various ways. Several workers have studied the methods of combination of these powders, their compressibility and the physical and mechanical properties of the bearing metals.

Schwartzkopf[25] pressed iron skeletons from powder and sintered to 85% density. Molten copper was then infiltrated in the skeleton and thermally treated to secure hardness.

Tormyn[26] patented a process wherein iron powder is pressed to the general shape and size, sintered at 2000°F, copper powder applied to the surface and the whole repressed to compact the copper powder and density of the iron, and then resintered at 1900°F in a non-oxidizing atmosphere.

Truesdale[27] in another patent employs a method to coat individual iron powder particles with copper. Finely divided $Cu_2O$ is intimately mixed with iron powder and reduced at 150-600°C by hydrogen until the reduction is complete. Additions of about 1% CuCl was found to aid the complete envelopment.

Langhammer and Calkins[28] prepared porous metal bearings using a mixture of 10% copper and 90% iron such that the particles of iron are on the average ten times the particles of copper, and sintering the briquette in a non-oxidizing atmosphere at a temperature between the melting points of the two metals.

Bosch[29] found that the compressibility of iron-copper mixtures is improved by using copper powder such that its volumetric weight is smaller than that of iron powder.

Kelly[30] studied the influence of copper content and sintering time and temperature on the properties of iron-copper compacts and found that the tensile strength of a compact containing 10% copper sintered at 1200°C for ten minutes compared favorably over other copper contents with similar treatment, and was greater than that of pure iron sintered at any temperature for any length of time. The strength and the ductility of 10% copper compositions increase with time for a sintering temperature of 1100°C. At constant sintering conditions of 1100°C and one-half of an hour, both the strength and the ductility increase with copper content up to 30%, the values being three times at 30% Cu-Fe than at 5% Cu-Fe compositions.

Northcott and Leadbeater[31] engaged in similar investigations established a relationship between tensile strength and porosity of the compacts and found that for constant porosity, maximum strength was obtained with 10% copper content. However the best physical properties and highest densities were obtained for 25% copper-iron ratio sintered in hydrogen for one hour at 1100°C when copper was in the liquid phase.

Chadwick, *et al.*[32] working with different kinds of iron powders obtained results analogous to those of Northcott and Leadbeater. The optimum tensile strength and elongation values were obtained with 25% copper contents. It was found that iron powders of low compressibility (reduced) gave better physical properties than the readily compressible (electrolytic) iron powders.

Langhammer[24] compared the physical properties of bronze and iron-copper bearing metals and found that for the same specific gravity, the iron-copper bearing material has two and one-half times the ultimate strength, thrice the resistance to permanent deformation, and one and one-half times the Brinell hardness and a much lower coefficient of thermal expansion, although the porosity was 5% lower than that of the bronze bearing material.

Arata[33] found that iron-copper compacts compare very favorably in all physical properties such as ultimate strength, Brinell hardness, PV factor etc. over both pure iron and bronze compacts.

# II. EXPERIMENTAL

## The Raw Material

The iron powder used in these investigations was prepared electrolytically as described in Part I of this thesis. The copper powder was also prepared electrolytically using acid sulphate bath and electrolytic copper electrodes. Except in the series of experiments where the particle size distribution is the variable, the iron powder mix had the following size distribution:

| | | |
|---|---|---|
| -48 + 65 | mesh | 11.1% |
| -65 + 100 | " | 27.7% |
| -100 + 140 | " | 27.7% |
| -140 + 200 | " | 11.1% |
| -200 + 325 | " | 11.2% |
| -325 | " | 11.2% |

In general, the powder mix had 10% of copper and 2% graphite except in the series of experiments where these were the variables. These extra additions to iron powder were restricted to -325 mesh size. The size distribution of iron powder was chosen arbitrarily on the basis of the powder prepared electrolytically, as described in the previous experiments.

## The Die

The die is fabricated from alloy steel containing:

| | |
|---|---|
| C | 0.32% |
| Ni | 4.10% |
| Cr | 1.30% |
| Mo | 0.20% . |

After machining to a slight oversize, the die was hardened by the following treatment. The die was introduced in a furnace at 820°C and maintained at that temperature for 45 minutes. On removal from the furnace, it was rapidly cooled in an air blast. It was then tempered at 250°C and air cooled. This treatment gave the die material a tensile strength of about 100 tons/sq.in. The die surface was then ground to a mirror-like finish to a close limit of tolerance. The figure shows the setup consisting of five parts. The upper punch A is the movable part, which transmits the pressure and forms the upper end of the briquette and ejects it from the die after compression. The stationary parts are the die B which forms the outer diameter of the bearing, the core rod C which forms the inside diameter, and the base D which in conjunction with the ring E constitutes the lower punch and forms the lower end of the bearing.

### Briquetting

The core rod C is screwed to the base D, the ring E slid over the core rod onto the base and the die B placed on it. The powder is filled in the cavity so formed and the punch A brought down to compress the briquette. The guide pins insure that the punch is in alignment with the assembly. After compression, the base is unscrewed and the punch brought down further to eject the bearing compact. The green bearing had an inside diameter of 1.256" and an outside diameter of 1.763" compared to the diameter of the core rod of 1.253" and the die diameter of 1.760". Depending on the conditions of the experiment, the length of the bearing varied between 0.53" to 0.75" for a weight of about 60 g.

### Sintering

Sintering of the green compact was carried in an atmosphere of hydrogen stripped of all its oxygen content by passing over copper turnings at 600°C.

## Bearing Characteristics

### Green Density

Green density or the pressed density is the ratio of grams to cubic centimeters of a briquette compacted to specified dimensions by a designated pressure.

### Sintered Density

It is the density of the briquette after the sintering treatment.

Oil Impregnated Density

It is the density of the briquette fully impregnated with a lubricant such as oil. The oil impregnation may be carried out with the help of either heat or pressure. As specified by A.S.T.M.[34], the following procedures may be employed:

(1) The specimen shall be immersed for at least four hours in oil (viscosity approximately 200 secs. Saybolt Universal at 100°F) held at a temperature of 180 ± 10°F and then cooled to room temperature by immersion in oil at room temperature.

(2) The pressure over the specimen after immersion in oil at room temperature shall be reduced from atmospheric pressure to not more than 2" Hg after which the pressure shall be allowed to increase to atmospheric in 10 minutes, the specimen remaining immersed in oil for the whole period.

The bearing can be fully impregnated with oil by any of the procedures. However, the second method was employed because of its convenience. The oil impregnated density is calculated as follows:[34]

$$D = \frac{B}{B-C}$$

D = Density g/c.c.

B = Weight of the oil impregnated sample

C = Weight of the sample suspended in water.

A.S.T.M. standards specify that the density calculated as above should lie between 5.8 and 6.2 g/c.c. for the iron-copper bearings.[34]

## Porosity

The quality resulting from a multiplicity of pores distributed in a compact and measured as the percent voids per unit volume is called porosity.

Porosity is the basic property in powder metal bearings. The pores, being intercommunicating, serve as a reservoir for the lubricant. Obviously, the physical properties of the compact are impaired considerably by an inherent porosity. The effect is not proportional to decrease in cross-sectional area, but is largely increased by the multitude of pores, each acting as an individual source for stress concentrations and notch effects. Hence, for each application a balance must be struck between the physical properties necessary and the maximum porosity possible. Thus, for example, a bearing which is to be subjected to heavy loads at high temperatures requires a high porosity of about 35% and a specific gravity of about 5.6. Here a coarse powder has to be employed with the resultant large average pore size. For a bearing to be used for high speeds, light loads and low temperatures, a high porosity and large pore size would result in excessive losses of lubricant. In such a case, a low porosity and comparatively a smaller pore size is the correct choice and the powder will have to be of a finer size. No doubt, the lubricant plays an important part. A heavy oil with a high viscosity will be the most appropriate in the former and a light oil with a low viscosity will suit the latter applications.

The porosity is calculated as follows:[34]

$$P = \frac{B-A}{(B-C) \times s} \times 100$$

P = Porosity percent

A = Weight of the sample

B = Weight of the oil impregnated sample

C = Weight of oil impregnated sample suspended in water

s = Specific gravity of the lubricant

A.S.T.M. Standards specify that the porosity calculated as above must not be below 18% by volume.

Radial Crushing Strength

For satisfactory working, a bearing must possess a certain compressive strength. A measure of this is the radial crushing strength which is determined by compressing the test specimen between two flat surfaces, the direction of the load being normal to the longitudinal axis of the specimen. The point at which the load drops due to the first crack is the radial crushing strength.[34] According to A.S.T.M. specifications[34] the radial crushing strength determined as above shall not be less than the value calculated as follows:

$$P = \frac{KLT^2}{D-T}$$

where

P = Radial crushing strength in pounds

D = Outside diameter of the bearing in inches

T = Wall thickness of bearing in inches

L = Bearing length in inches

K = Strength constant.

For iron-copper bearings, the value of K is 40,000.

In the present series of investigations, the length of the bearing varied from 0.55" to 0.75", depending upon the operating conditions like the briquetting pressure and the composition of the powder mix. The radial crushing strength of a bearing varies directly with the length. Thus, comparison of the strength of the bearings is possible only if the bearing length remains the same in all cases. Hence, in the representation of results, the value of the strength is corrected to that corresponding to a bearing length of 0.57". This value of the length was chosen because it gives a projected area (outside diameter times length) of one square inch.

## III. RESULTS

### General Plan of Work

A detailed study was made of the influence of

(1) Briquetting pressure
(2) Sintering temperature
(3) Sintering time
(4) Copper content
(5) Graphite content
(6) Particle size on

  (a) Density
  (b) Porosity
  (c) Strength
  (d) Changes after sintering in density, length and diameter.

The following table gives the values of the variables kept constant and their extreme limits of variation.

| VARIABLE | FIXED VALUE | LIMITS OF VARIATION |
|---|---|---|
| Briquetting pressure | 20 tons/sq.in. | 5-35 tons/sq.in. |
| Sintering temperature | 1100 °C | 900-1300 °C |
| Sintering time | 1 hour | $\frac{1}{2}$-8 hours |
| Copper content | 10% by wt. | 0-30% |
| Graphite content | 2% by wt. | 0-6% |
| Particle size | 35%(-48+100 mesh) | 0-100% |

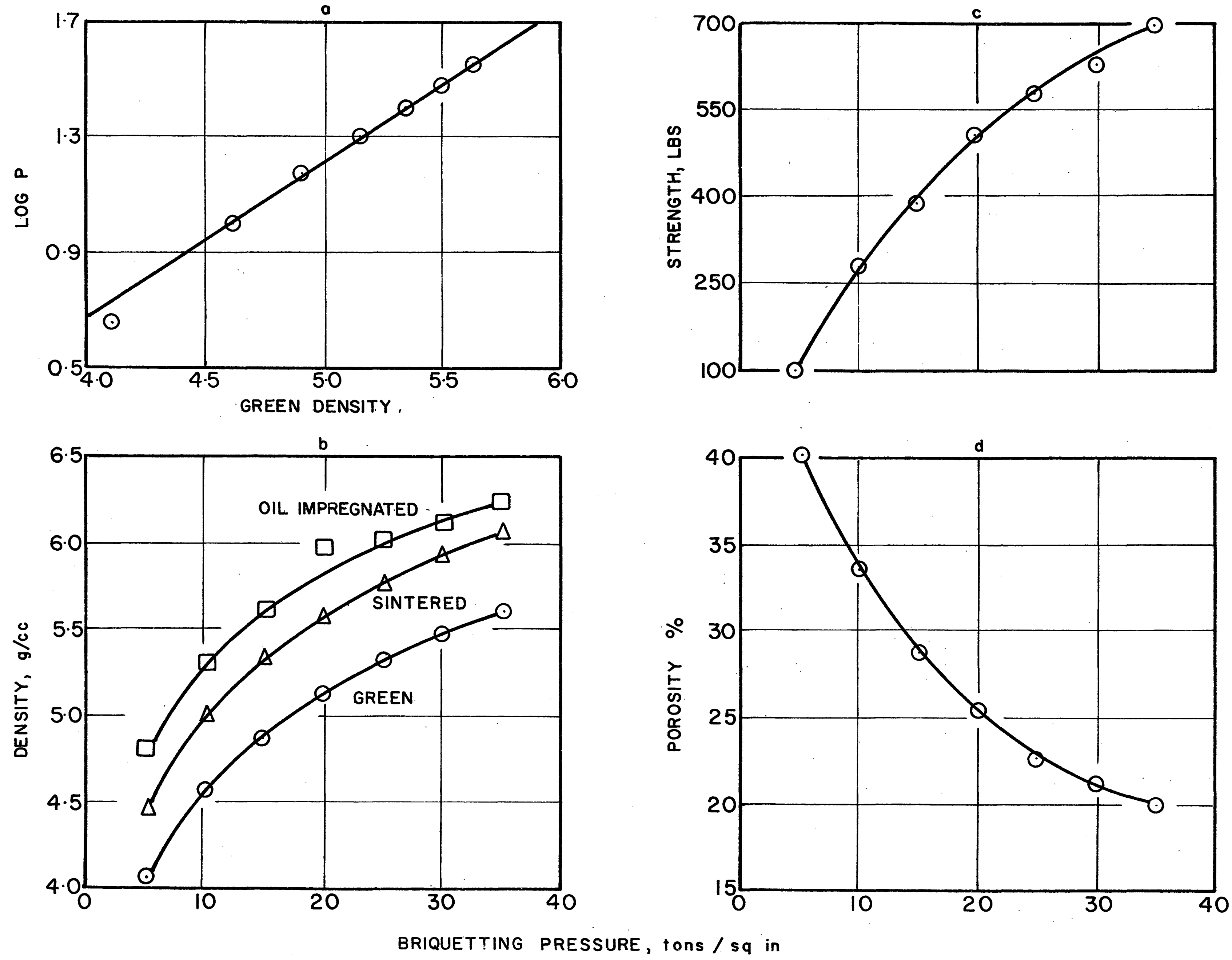

FIG I EFFECT OF PRESSURE ON BEARING CHARACTERISTICS

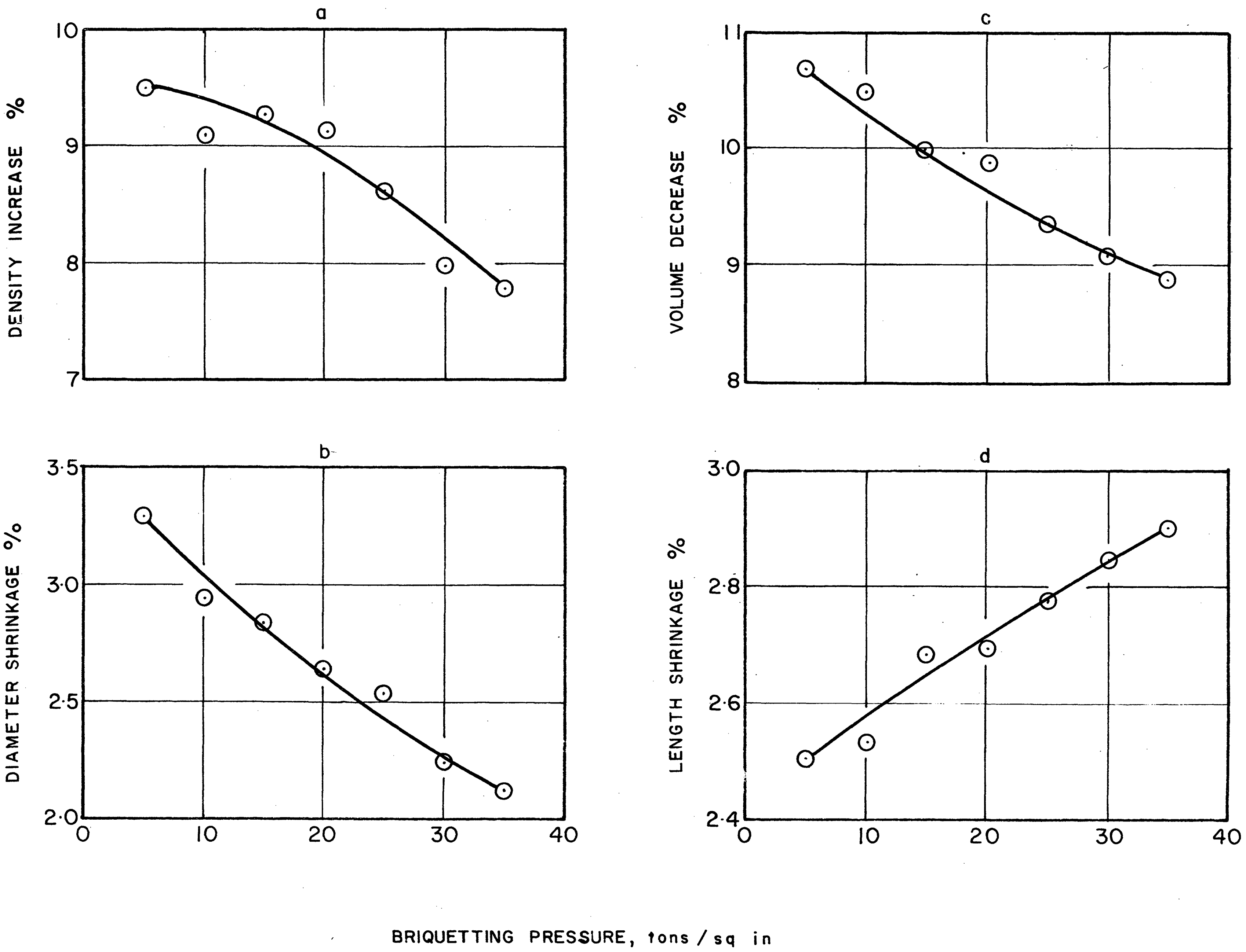

FIG 2 EFFECT OF PRESSURE ON CHANGES AFTER SINTERING

# TABLE I

## EFFECT OF BRIQUETTING PRESSURE

Copper 10 percent
Sintering time - 1 hour

Graphite 2 percent
Sintering temp. - 1100°C
under hydrogen

| Briquetting pressure tons/sq.in. | Density g/c.c. | | | Porosity percent | Radial crushing strength lbs. | Changes after sintering percent | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Green | Sintered | Oil Impregnated | | | Density increase | Length shrinkage | Diameter shrinkage | Volume shrinkage |
| 5 | 4.10 | 4.49 | 4.83 | 40.36 | 103 | 9.90 | 2.51 | 3.31 | 10.7 |
| 10 | 4.61 | 5.03 | 5.33 | 33.5 | 230 | 9.11 | 2.56 | 2.95 | 10.5 |
| 15 | 4.90 | 5.35 | 5.63 | 28.30 | 385 | 9.20 | 2.69 | 2.85 | 10.0 |
| 20 | 5.14 | 5.61 | 5.98 | 25.36 | 510 | 9.16 | 2.70 | 2.65 | 9.91 |
| 25 | 5.35 | 5.81 | 6.04 | 22.84 | 580 | 8.68 | 2.78 | 2.94 | 9.37 |
| 30 | 5.50 | 5.94 | 6.14 | 21.06 | 630 | 8.00 | 2.85 | 2.25 | 9.10 |
| 35 | 5.62 | 6.06 | 6.26 | 20.1 | 700 | 7.83 | 2.91 | 2.15 | 8.90 |

The results are presented graphically in Fig. 1 and Fig. 2

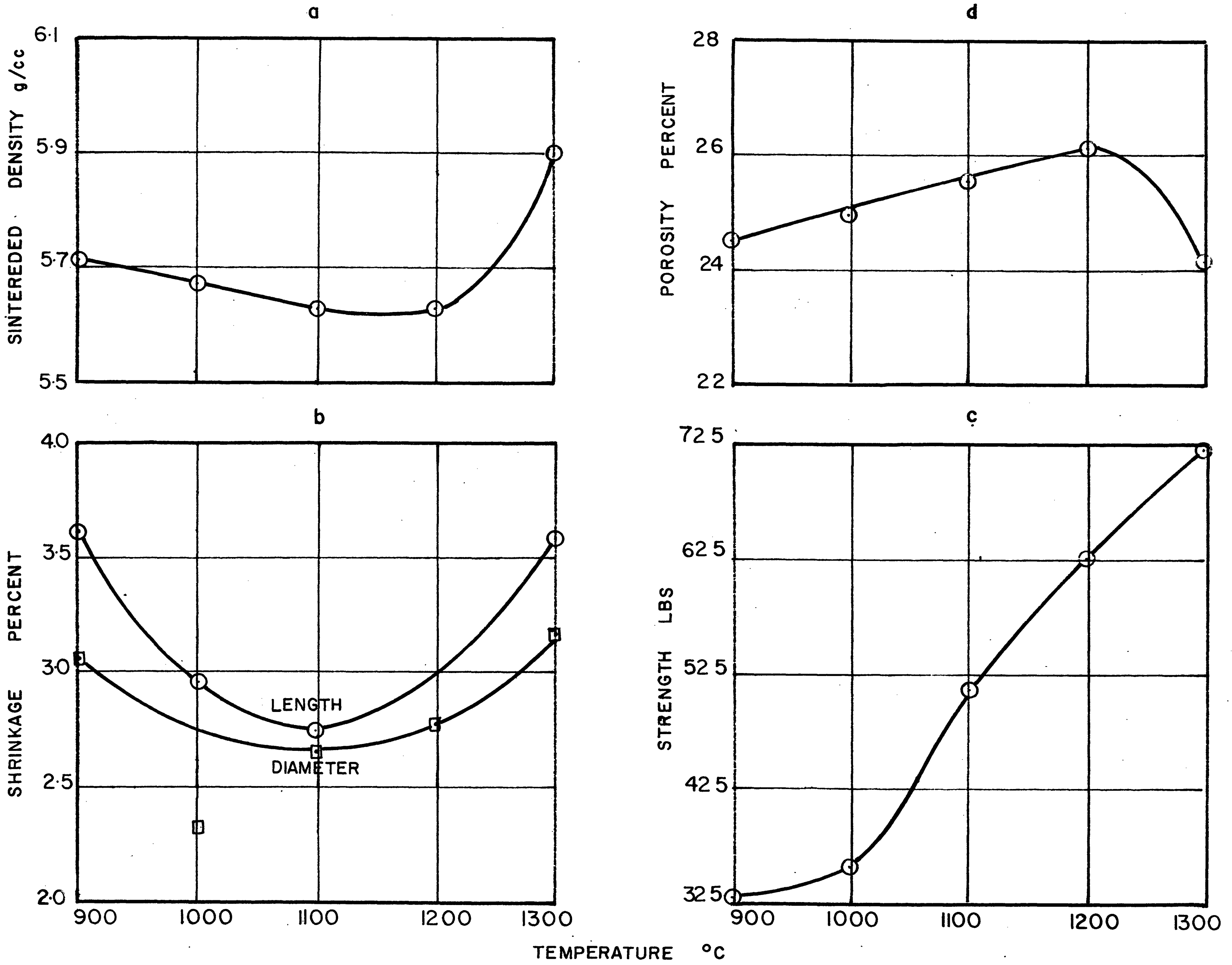

FIG 3 EFFECT OF TEMPERATURE OF SINTERING ON BEARING CHARACTERISTICS.

## TABLE II

## EFFECT OF TEMPERATURE OF SINTERING

Briquetting pressure, 20 tons/sq.in. Copper, 10 percent
Sintering time, 1 hour in hydrogen Graphite, 2 percent

| Temperature of sintering °C | Density g/c.c. | | Porosity percent | Radial crushing strength lbs. | Changes after sintering percent | | |
|---|---|---|---|---|---|---|---|
| | Green | Sintered | | | Density increase | Shrinkage in length | Shrinkage in diameter |
| 900 | 5.14 | 5.71 | 24.5 | 330 | 10.9 | 3.63 | 3.04 |
| 1000 | 5.14 | 5.68 | 25.02 | 355 | 10.05 | 2.94 | 2.32 |
| 1100 | 5.14 | 5.61 | 25.36 | 510 | 9.45 | 2.76 | 2.65 |
| 1200 | 5.14 | 5.63 | 26.16 | 625 | 9.5 | 2.98 | 2.78 |
| 1300 | 5.14 | 5.91 | 24.2 | 715 | 14.83 | 3.60 | 3.18 |

The results are presented graphically in Fig. 3

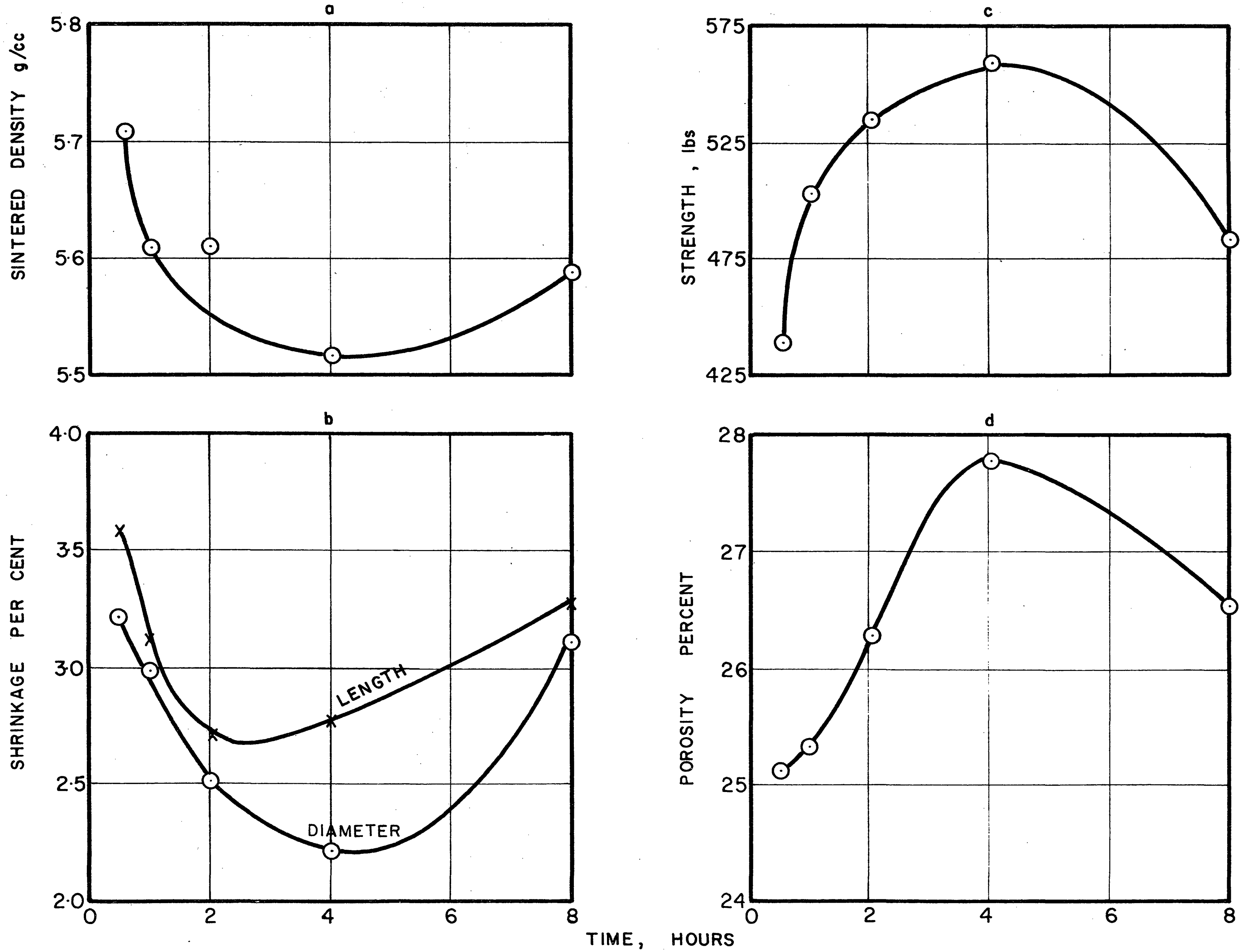

FIG 4. EFFECT OF TIME OF SINTERING ON BEARING CHARACTERISTICS

## TABLE III

## EFFECT OF TIME SINTERING

Briquetting pressure, 20 tons/sq.in.  Copper, 10 percent

Sintering temperature, 1100°C under hydrogen  Graphite, 2 percent

| Sintering time hours | Density g/c.c. | | Porosity percent | Radial crushing strength lbs | Changes after sintering percent | | |
|---|---|---|---|---|---|---|---|
| | Green | Sintered | | | Density increase | Diameter shrinkage | Length shrinkage |
| ½ | 5.14 | 5.71 | 25.12 | 440 | 10.9 | 3.25 | 3.61 |
| 1 | 5.14 | 5.61 | 25.36 | 510 | 9.15 | 2.98 | 2.65 |
| 2 | 5.14 | 5.61 | 26.3 | 535 | 9.02 | 2.52 | 2.70 |
| 4 | 5.14 | 5.52 | 27.8 | 560 | 7.4 | 2.22 | 2.80 |
| 8 | 5.14 | 5.59 | 26.5 | 480 | 8.75 | 3.12 | 3.3 |

The results are represented graphically in Fig. 4

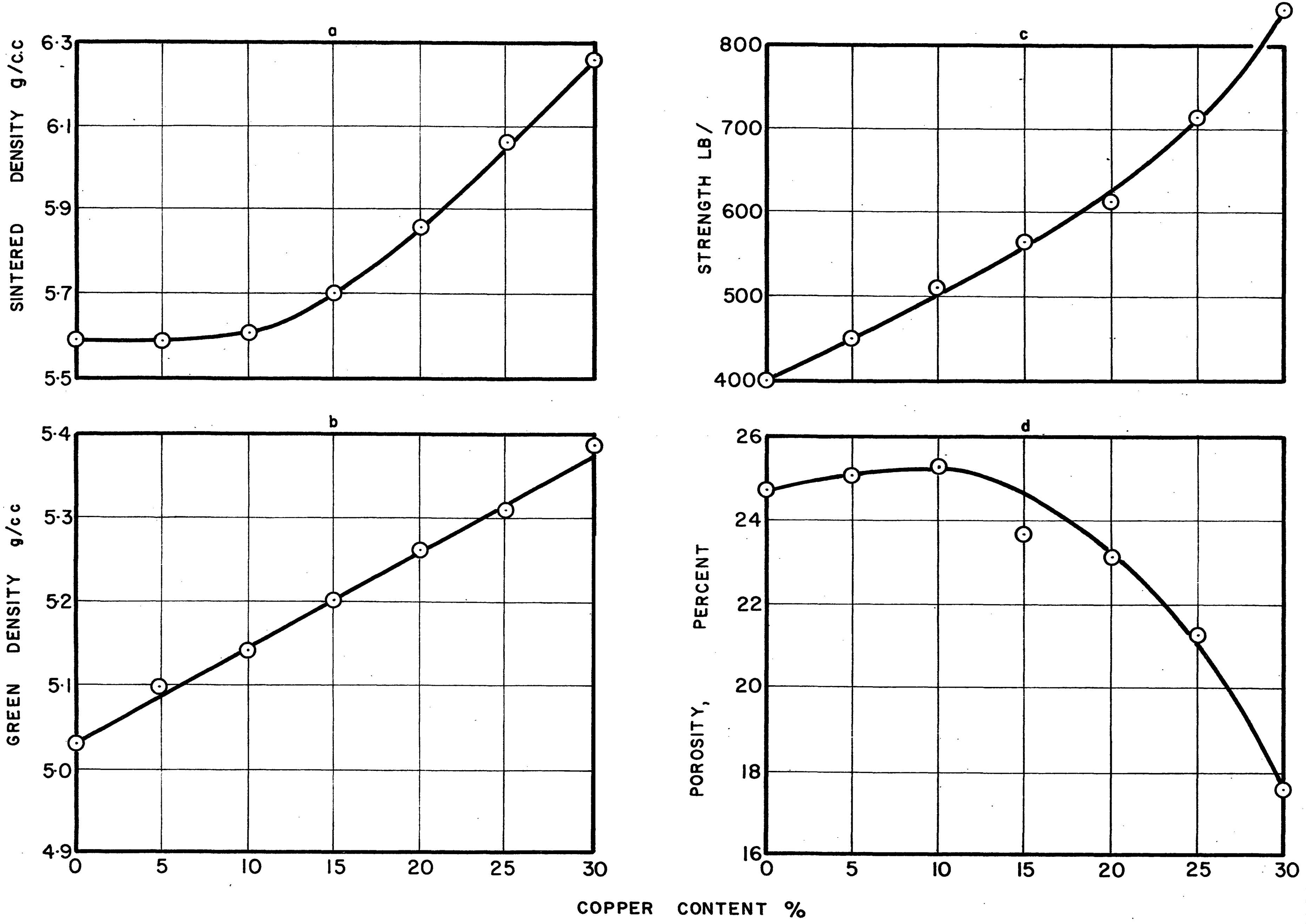

FIG 5 EFFECT OF COPPER CONTENT ON BEARING CHARACTERISTICS

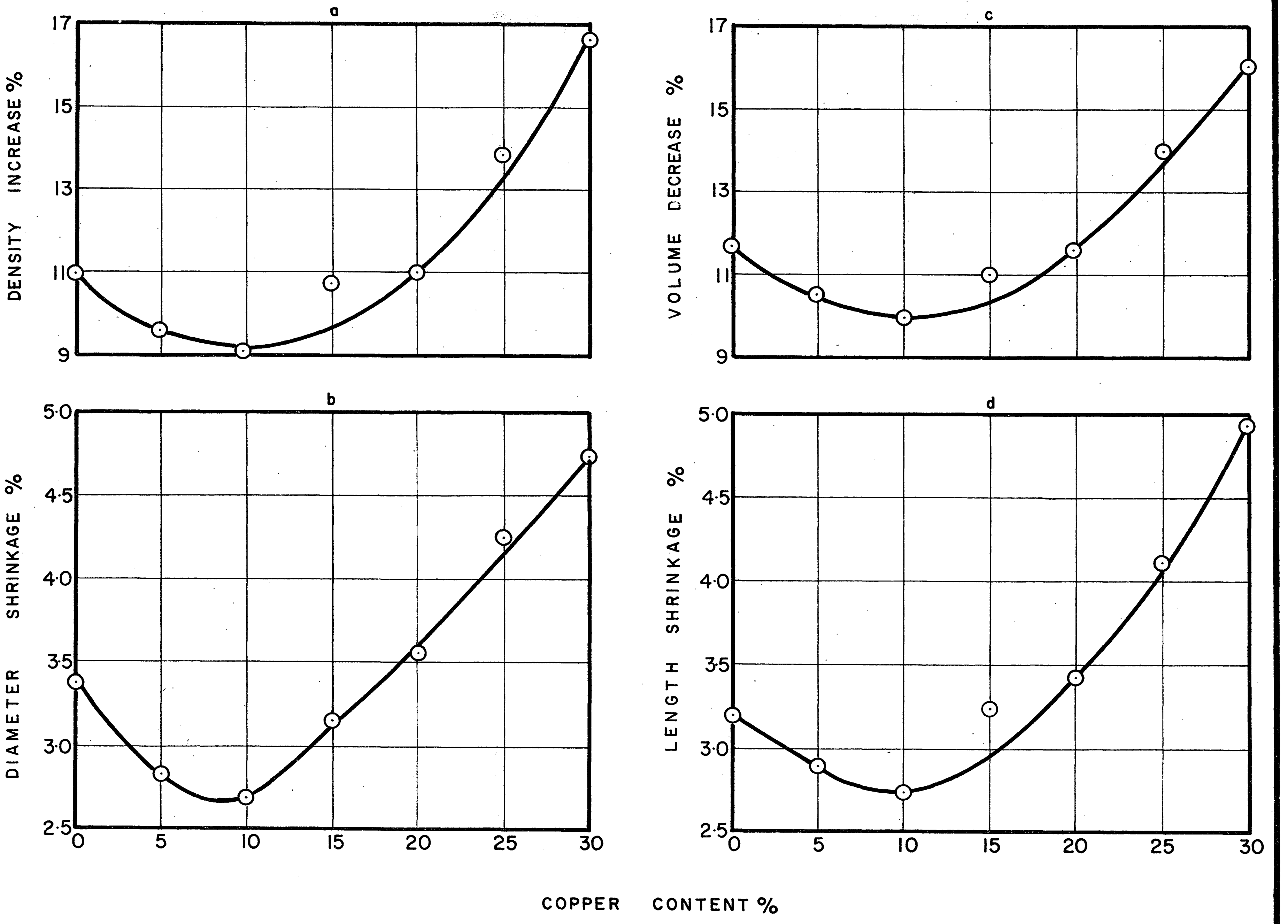

FIG 6 EFFECT OF COPPER CONTENT ON CHANGES AFTER SINTERING

## TABLE IV

## EFFECT OF COPPER CONTENT

Briquetting pressure, 20 tons/sq.in.  Graphite, 2 percent

Sintering time, 1 hour  Sintering temp., 1100$^{o}$C under hydrogen

| Copper content percent | Density g/c.c. | | Oil Impregnated | Porosity percent | Radial crushing strength lbs. | Changes after sintering percent | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Green | Sintered | | | | Density increase | Length shrinkage | Diameter shrinkage | Volume shrinkage |
| 0 | 5.03 | 5.603 | 5.82 | 24.8 | 400 | 11.2 | 3.57 | 3.4 | 12.7 |
| 5 | 5.11 | 5.60 | 5.87 | 25.1 | 450 | 9.7 | 2.80 | 2.85 | 10.5 |
| 10 | 5.14 | 5.61 | 5.98 | 25.36 | 510 | 9.16 | 2.70 | 2.65 | 9.91 |
| 15 | 5.20 | 5.77 | 6.01 | 23.7 | 565 | 10.9 | 3.2 | 3.15 | 11.0 |
| 20 | 5.27 | 5.85 | 6.04 | 23.2 | 615 | 11.1 | 3.40 | 3.57 | 11.6 |
| 25 | 5.31 | 6.05 | 6.23 | 21.3 | 715 | 13.8 | 4.10 | 4.10 | 13.9 |
| 30 | 5.38 | 6.28 | 6.44 | 17.6 | 840 | 16.6 | 4.85 | 4.76 | 15.9 |

The results are plotted in Fig. 5 and Fig. 6

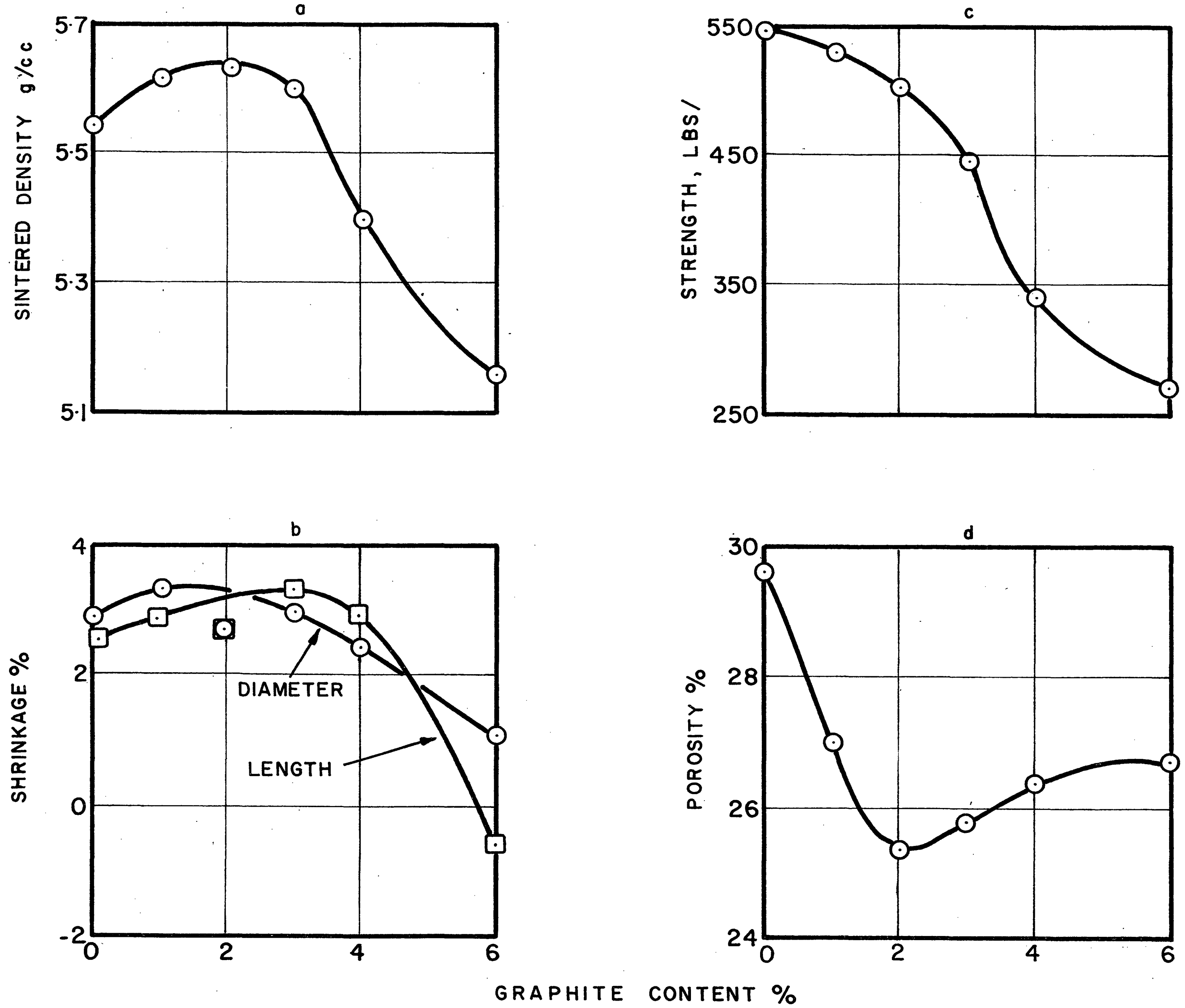

FIG 7 EFFECT OF GRAPHITE CONTENT ON BEARING CHARACTERISTICS

## TABLE V

## EFFECT OF GRAPHITE CONTENT

Briquetting pressure, 20 tons/sq.in.
Sintering temperature, 1100°C

Copper, 10 percent
Sintering time, 1 hour
under hydrogen

| Graphite content percent | Density g./c.c. | | Porosity percent | Radial crush-ing strength lbs. | Changes after sintering percent | | |
|---|---|---|---|---|---|---|---|
| | Green | Sintered | | | Density increase | Diameter shrinkage | Length shrinkage |
| 0 | 5.14 | 5.54 | 29.6 | 545 | 7.7 | 2.53 | 2.9 |
| 1 | 5.14 | 5.60 | 27.0 | 525 | 8.9 | 2.84 | 3.35 |
| 2 | 5.14 | 5.61 | 25.36 | 510 | 9.15 | 2.70 | 2.65 |
| 3 | 5.14 | 5.59 | 25.8 | 440 | 8.7 | 3.30 | 3.00 |
| 4 | 5.14 | 5.43 | 26.4 | 340 | 5.6 | 2.94 | 2.40 |
| 6 | 5.14 | 5.24 | 26.7 | 270 | 1.9 | -0.6 | 2.05 |

The results are presented graphically in Fig. 7

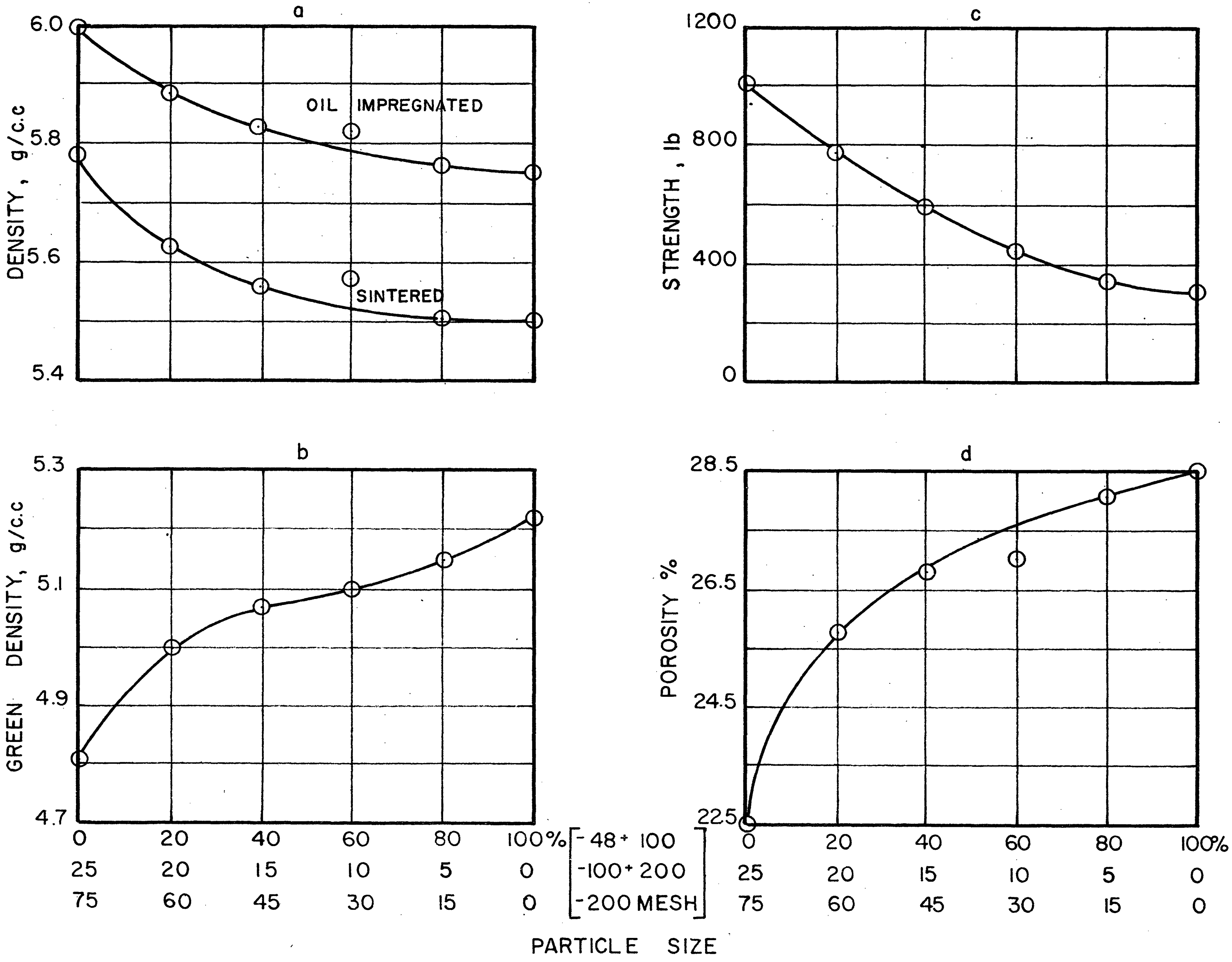

FIG 8. EFFECT OF PARTICLE SIZE ON BEARING CHARCTERISTICS.

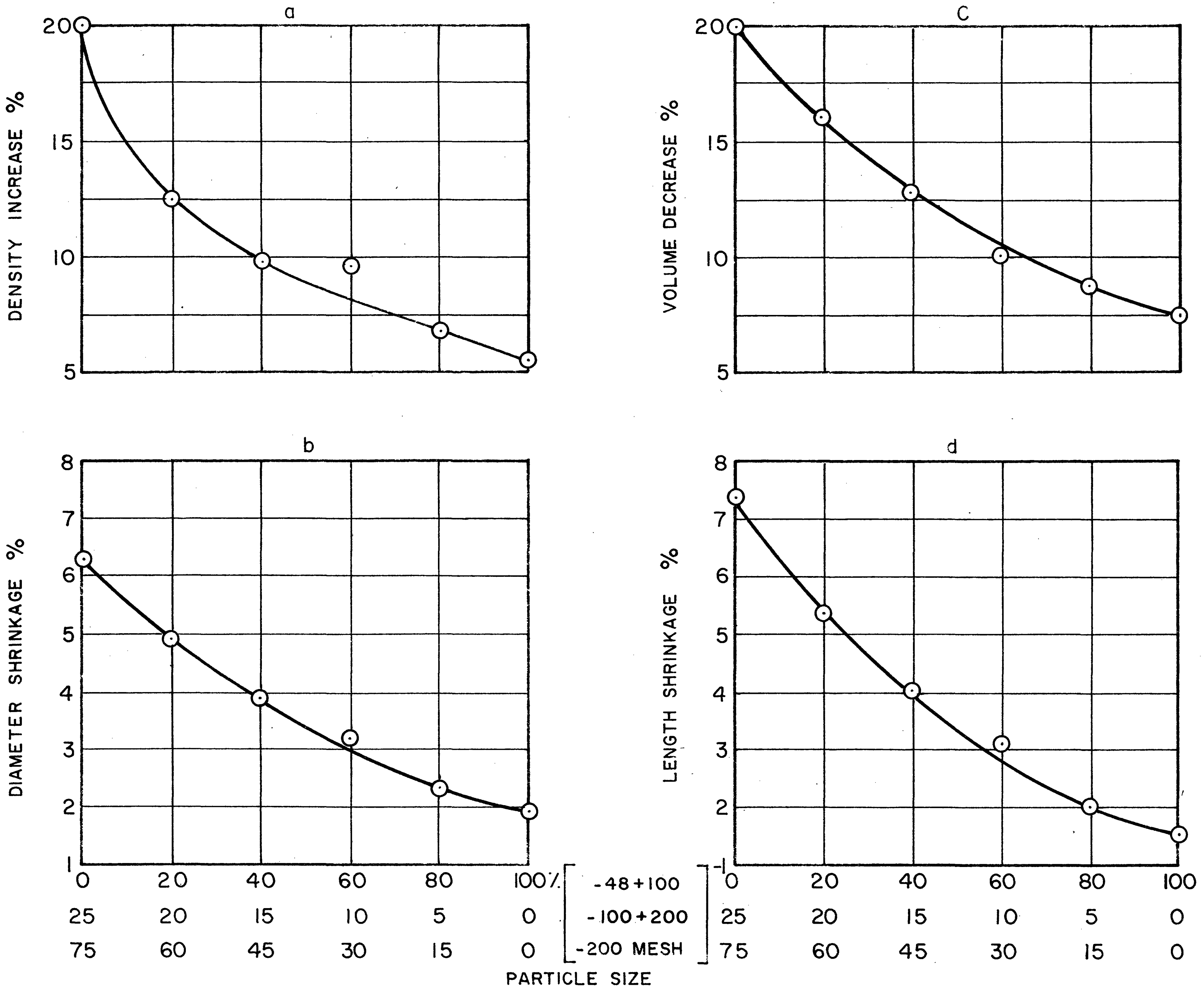

FIG 9. EFFECT OF PARTICLE SIZE ON CHANGES AFTER SINTERING

# TABLE VI

## EFFECT OF VARIATION IN PARTICLE SIZE

Briquetting pressure, 20 tons/sq.in.

Copper, 10 percent　　Graphite, 2 percent

Sintering time, 1 hour　　Sintering temp., 1100°C under hydrogen

| Percentage -48+100 mesh iron powder | Density g/c.c. | | | Porosity percent | Radial crushing strength lbs. | Changes after sintering percent | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Green | Sintered | Oil Impregnated | | | Density increase | Diameter shrinkage | Length shrinkage | Volume shrinkage |
| 0 | 4.81 | 5.78 | 6.00 | 22.5 | 1000 | 20.1 | 6.27 | 7.27 | 19.95 |
| 20 | 5.00 | 5.62 | 5.86 | 25.8 | 760 | 12.4 | 4.90 | 5.35 | 16.05 |
| 40 | 5.07 | 5.57 | 5.83 | 26.8 | 600 | 9.85 | 3.95 | 4.0 | 12.73 |
| 60 | 5.18 | 5.57 | 5.82 | 27.0 | 440 | 9.65 | 3.24 | 3.1 | 10.0 |
| 80 | 5.15 | 5.50 | 5.76 | 28.05 | 330 | 6.8 | 2.32 | 2.02 | 8.9 |
| 100 | 5.22 | 5.50 | 5.75 | 28.55 | 310 | 5.36 | 1.92 | 1.51 | 7.46 |

The results are presented graphically in Fig. 8 and Fig. 9.

# IV. DISCUSSION OF THE RESULTS

## 1. Green Density

The effect of the different variables on the densities of the compacts is shown in Figures 1b, 5b and 8b. From Fig. 1b, it may be seen that the density steadily increases with increase in briquetting pressure. This is natural since the closeness and the area of contact of the powder particles are increased by the applied pressure and this is an index of the density of the compact. It may also be noted that as the briquetting pressure increases, it tends to exert a lesser and lesser influence on the green density. Thus, for a 10 tons/sq. in. rise in pressure from 5-15 tons/sq. in. the density increase is 0.8 g/c.c. from 4.1 to 4.9 g/c.c. Between 15-25 tons/sq. in. increase in pressure, the density increases by 0.45 g/c.c. from 4.9 to 5.35 g/c.c. and correspondingly between 25-35 tons/sq. in. The increase in density is only 0.27 g/c.c. from 5.35 to 5.62 g/c.c. This happens because at low pressures, densification occurs more by redistribution of the particles and reorientation into strata, while the frictional resistance is comparatively low. At high pressures, the free space in the compact decreases, and densification takes place by a smoothing of the crevices and filling of the voids through plastic deformation. In this phase, the die wall friction becomes an appreciable factor, and the energy expended is comparatively very large and increases beyond proportions as the density of the compact approaches the theoretical value of the metal.

From the relationship between briquetting pressure and the corresponding decrease in the volume of the compact, Balshin[35] arrives at the conclusion that if the height and the relative volume of a compact decrease in arithmetical progression, the pressure must increase geometrically. In other words, expressed as an equation:

$$\log p = Ld + C$$

where d is the density and L and C are constants depending upon the nature of the powder and conditions of compacting. Hence, if log p be plotted against d, the nature of the curve should be a straight line. Fig. 1a illustrates the linear relationship and endorses Balshin's equation.

Fig. 5b illustrates the increase in density with increase in copper content of the compact at constant briquetting pressure. The density increases from 5.03 g/c.c. at 0% to 5.38 g/c.c. at 30% copper content. Copper being a metal with a face-centered cubical lattice is easily deformable and is not subject to work-hardening at room temperatures. Iron, on the other hand, being a metal with a body-centered lattice has a greater resistance to deformation and has a great work-hardening tendency. Accordingly, lower pressures are required for coherent compacts of copper than for those of iron, and correspondingly for the same applied pressure, copper compacts are denser than those of iron. Thus, greater the copper content, greater is the density of the iron-copper compacts.

The effect of the particle size on the density is plotted in Fig. 8b. It is seen that the density decreases as the particle size decreases. This is because the pressure is absorbed primarily by the shifting of particles and deformation is only secondary. Thus, the finer sizes will give a lower density than the coarse sizes for the same pressure. However, although the density is low in the fine-sized compacts, the number of contact points is larger as can be seen from their higher strength.

### 2. Sintered Density and the Changes After Sintering

On sintering there is an increase in the density of a powder compact which is brought about by a reduction in the pore volume as well as the over-all volume. With the application of heat, sintering starts at the metal-to-metal contacts and the surface tension forces operate in a direction to minimize the surface areas. The intensity of the surface energy being less at the smooth surfaces than at the crevices, transfer of the metal takes place, thus decreasing the over-all volume and increasing the density.

Fig. 2 shows the effect of the compacting pressure on the changes after sintering. With increasing pressures, the rate of increase in density and the percent of shrinkage continually decrease. With increasing compacting pressures, the microscopic, and perhaps molecular, irregularities are lessened, and consequently during sintering, the surface energy differences being lower, the dimensional changes are also smaller.

With increase in copper content, the sintered density increases the same way as green density (Fig. 5a). However, the percent increase in density after sintering is 11.2% for 0% copper content, decreases to 9.16% for a 10% copper content and then continuously increases to 16.6% increase for a copper content of 30%. The same trend is seen in all the other changes after sintering, viz., that there is a minimum shrinkage in compacts having a 10% copper content. The increase in shrinkage with increase in copper content may be explained by the fact that the liquid phase largely removes the surface films and other irregularities which would otherwise obstruct sintering and crystillization. The rounded grains of the solid metal form closer bonds with the liquid phase because of the increased mobility. The surface tension forces of the liquid also contribute to the formation of a closely knit structure.

The effect of particle size on the sintered density and on the changes after sintering is interesting; whereas, with increasing particle size, the green density increases, the sintered density increases with decreasing particle size. This is illustrated in Figs. 8a and 8b. The change in density is particularly sharp between 0% to 20% of 100 mesh size content. The shrinkage in the diameter and the length also show the same trend as may be seen from Fig. 9. The increased shrinkage with increasing fineness of the powder mix is obtained, because, in the case of the fin sizes, during pressing, densification takes place more by redistribution of the particles than by deformation, so that during sintering there is a greater allowance for dimensional changes. With coarse powders there is a large amount of deformation and cold working during deformation itself. Secondly, with the greater surface area of the finer particles, the surface tension forces are more active. The third, though perhaps a minor factor, is that finer powders adsorb larger amounts of gases than the coarse grades. During sintering, as the gases force their way out, many new "clean" surfaces are produced, which again takes place on a larger scale in fine-sized compacts.

## 3. Porosity and Strength

Porosity and strength are inter-related properties in a porous powder compact. Increase in porosity causes a decrease in strength. With increase in compacting pressure, the porosity decreases uniformly from 40.36% at 5 tons/sq.in. to 20.1% at 35 tons/sq.in. pressure. Correspondingly, the radial crushing strength increases from 103 lbs. at 5 tons/sq.in. to 700 lbs. at 35 tons/sq.in. pressure. The pressure affects the strength by increasing the number of contact points so that the sintering progress is accelerated and recrystallization and grain growth proceeds faster (Fig. 1 c,d).

Increase in copper content of the powder mix decreases the porosity. Copper being more soft and deformable, it facilitates particle redistribution and during sintering, the liquid phase produces a well-knit structure. Strength increases with increased copper content. This is due to the higher degree of densification of the structure by close approach to individual crystallites and due to the increased grain growth, because of the removal of surface films by the liquid phase (Fig. 5c,d.).

With graphite additions, there is a decrease in the porosity from 29.6% at 0% to 25.36% at 2% graphite content, and again there is an increase to 26.4% at 6% graphite content. However, the strength decreases steadily from 545 lbs. at 0% to 270 lbs. at 6% graphite content (Fig. 7c,d.). Graphite additions improve the wear characteristics of the bearing and act as a lubricant during compaction of the powder.

With increased fineness of the powder mix, the porosity decreases and the strength increases. The increase in strength may be attributed to the larger surface area of the fine powders and the greater metal-to-metal contact (Fig. 8 c,d).

## 4. Effect of the Temperature and the Time of Sintering on Bearing Characteristics

Figs. 3 and 4 illustrate graphically the effects of the temperature and the time of sintering respectively on sintered density, the shrinkages in length and diameter, the strength and the porosity. It is known that neither the temperature nor the time affects markedly the "sintering forces" so that their main function is to minimize the effects of the factors obstructive to sintering. Jones[5] points out that the most important effect of temperature in aiding sintering is the increase in the plasticity of the metal. In the same way, the influence of time on completeness of sintering is a matter of plastic flow. More plastic the metal, the more it will flow in a given time, and in the same way, more of the metal of any given plasticity will flow for a greater length of time. Accordingly, for monometallic compacts, it is usually found that with increasing temperature and time of sintering, there is increased shrinkage and a reduction of porosity in a powder compact. In the present investigation, however, it is found that shrinkage decreases with increase in temperature between 900°C to 1100°C, and increases with further increase in temperature. Porosity increases with temperature from 24.5% at 900°C to 26.16% at 1200°C, and decreases to 24.5% as the sintering temperature is further raised to 1300°C. It is interesting to note that from 1100°C to 1200°C, there is an increase in shrinkage as well as a small increase in porosity. In the same way, porosity which is 25.1% for ½ hr. sintering time increases to 27.8% for 4 hrs. and again decreases to 26.6% for an 8-hour period.

Schecht, *et al.*[36] studied the density changes of loose iron powders of various origins and found that there is a marked drop in density starting near the $\alpha$-$\gamma$ transformation point and reaching a minimum at 1000°C. Libsch, *et al.*[37] did similar work with iron powder compacts and obtained analogous results. The second factor which influences the density changes and the porosity and shrinkage is the influence of gases and vapors. In a powder compact, the potential sources of gas are: (1) adsorbed films; (2) gases evolved by chemical action on heating; (3) mechanically entrapped air. It is unlikely that the third source plays any decisive part in the changes in porosity after sintering. Ruer and Kuschmann[38] have shown that the amount of gas adsorbed by metal powders even at room temperature is considerable. Working with copper powder prepared from cupric oxide by reduction with hydrogen at 750°C followed by evacuation at 440°C, they found that it gained 5.44 mg/100 g. within one hour. Similarly, reduced iron powder gained 20.43 mg/100 g. in the same time. The second source of gas, viz. that produced by chemical reaction or decomposition(Ex: by reduction of oxide films by hydrogen)may be the major factor at higher temperatures. If these gases escape before sintering and consolidation take place, it will be harmless, whereas, if they are evolved after consolidation, the gas pressure being considerable, an increase in the number and the size of the pores will be the result.

This affords a satisfactory explanation for the observations in the present investigations. At 900°C and onwards, sintering has progressed sufficiently and the shrinkage decrease takes place due to either $\alpha$-$\gamma$ transformation or the gas evolution or both in conjunction. Above 1000°C when the decrease in shrinkage and increase in porosity still continue, the only affecting factor is the gas evolution. By 1200°C the gas evolution is complete so that, due to the removal of oxide films, and due to the increased plasticity, there is a decrease in porosity and an increase in density at 1300°C. An additional factor is the presence of copper in the liquid phase which densifies the structure.

The steady increase in the radial crushing strength of the bearing compact, in spite of the irregular changes in porosity and density, focuses ones attention on the liquid cement theory of sintering. Theoretically, an increase in porosity should cause a decrease in strength, and this is observed true for monometallic compacts. However, in the present case, the increased plasticity of copper due to temperature more than counterbalances the negative effects of increased porosity.

The explanation for the apparent irregularities in shrinkage with increasing temperature applies equally well to explain the irregularities in shrinkage due to increased time of sintering. It appears that four hours is the optimum time, a point when the gas evolution is complete, the shrinkage minimum and the strength maximum. If the time of sintering be increased to eight hours, there is a decrease in porosity, accompanied by a decrease in strength, which is more probably due to grain growth taking place when the specimens are heated for a long time at a high temperature as 1100°C.

## 5. Operating Conditions to Obtain Bearings Satisfying A.S.T.M. Specifications

The density limits specified for iron-copper bearings fully impregnated with oil or lubricant are 5.8 and 6.2 g/c.c. All the bearing compositions tried have densities lying within this range except those obtained by using a briquetting pressure below 20 tons/sq.in., copper content of more than 25%, graphite content above 3% and having more than 60% of -48 + 100 mesh size.

The porosity need be above 18% by volume. All the bearing compositions tried satisfy this condition, except those having more than 25% copper contents.

The radial crushing strength should not be below the value calculated as follows:

$$P = \frac{KLT^2}{D-T}$$

P = radial crushing strength in lbs.

L = bearing length in inches

T = wall thickness of bearing in inches

D = outside diameter of bearing in inches

K = constant the value being 40,000 for Fe-Cu bearings.

For the standard length of 0.57" of the bearing, the radial crushing strength calculated from the above formula is 950 lbs.

The highest value of 1000 lbs. for the radial crushing strength is obtained when the bearing composition has 0% -48 + 100 mesh particle size content. In this case, the density is 6.00 g/c.c. and the porosity 22.5%. With the particle size content of 20% -48 + 100 mesh, the strength is 760 lbs. with a density of 5.86 g/c.c. and a porosity of 25.8%. In these cases, the briquetting pressure was 20 tons/sq.in. Using a higher briquetting pressure (30-35 tons/sq.in.), the bearing strength can be increased sufficiently to obtain a value much higher than the minimum necessary, meanwhile maintaining the density and porosity within proper limits. It has also been found that higher sintering temperature increases the strength without decreasing the porosity (or increasing the density) to an appreciable extent. Thus, increasing the sintering temperature from 1100°C to 1300°C increases the strength by 40%, while the density changes only by about 6% and porosity decreases by about 1%. Similarly, increase in the time of sintering up to 4 hours increases the strength accompanied by a small increase in porosity. Increase in the copper content also increases the strength. Thus, from 10% copper to 25% copper, the strength increases by about 40%, density by about 4%, while the porosity decreases by about 4%. Thus, a combination of these operating variables can be utilized favorably to obtain bearings satisfying A.S.T.M. specifications. It may then be said that to obtain bearings satisfying A.S.T.M. specifications, the following conditions may favorably be employed.

Briquetting pressure : 20-35 tons/sq.in.

Sintering temperature : 1100° - 1200° C

Sintering time : 1 - 4 hours

Copper content : 10 - 25%

Graphite content : 0 - 2%

Particle size distribution: Fine sized with as little +100 mesh particles as would be possible.

# V. CONCLUSIONS

The following conclusions may be drawn from the present investigations:

## 1. Pressure

Increase in pressure causes an increase in green and sintered density of a powder compact. With regard to green density, Balshin's relation between pressure and relative volume of a compact that $\log p = LV + C$ is found to be true within limits of experimental error for the pressure range studied in these investigations. The shrinkage after sintering decreases with increasing pressures. Porosity decreases and strength increases with increasing pressures so that a compromise must be effected between the strength necessary and the porosity possible for any given compacting pressure.

## 2. Sintering Temperature

The strength increases with increased temperatures of sintering. From 900°C to 1200°C there is a decrease in the density of the bearing and an increase in porosity. The minimum shrinkage is obtained in the temperature range of 1100°C to 1200°C. The increase in porosity with increasing temperature is most likely due to the gas evolution as the temperature increases. At 1300°C, there is a marked increase in shrinkage and a decrease in porosity.

## 3. Sintering Time

The time and the temperature of sintering act in the same direction. The optimum time seems to be 4 hours, when there is a maximum porosity and maximum strength.

## 4. Copper Content

Increase in copper content causes an increase in density, a decrease in porosity and an increase in strength. The shrinkage, however, shows a minimum for a copper content of 10% and is larger on either side. Copper in the liquid phase removes the surface films and promotes sintering which effects in the higher strength and lower porosity.

## 5. Graphite Content

Addition of graphite rapidly decreases the strength. The porosity decreases up to 2% additions and increases with further additions. The shrinkage characteristics also show a maximum between 2 to 3% graphite contents.

## 6. Particle Size

The finer the particle size, the smaller the green density. This is because the pressure is accommodated more by redistribution in the fine powders than in the coarse powders where deformation is a large factor. The same thing causes a much larger shrinkage for fine-sized compacts. A compact having 0% + 100 mesh powder has a 20% increase in density and the same decrease in volume, whereas a compact containing totally + 100 mesh powder shrinks only by 7.46% in volume and increases by 5.36% in density. This is of great concern in the matter of die design. The porosity decreases with increased fineness of the powder. The finer the particle size, the greater the strength of the compact. This is particularly noticeable between 40% and 0% (+ 100 mesh) contents. Hence, it is most desirable that a compact should have as little of + 100 mesh powder and as much of the finer sizes as would be possible.

To obtain iron base bearings satisfying A.S.T.M. specifications[34], the following conditions may be favorably employed.

Briquetting pressure : 20-35 tons/sq.in.

Sintering temperature : 1100-1200° C

Sintering time : 1 - 4 hours

Copper content : 10-25%

Graphite content : 0 - 2%

Particle size distribution : Preferably fine sized with as little + 100 mesh as would be possible.

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