# BALL MILLING PROCESS

by

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# I. ABSTRACT

Ball Milling process is a grinding process; like other grinding processes it produces loose wear particles. In theory the average size of a loose wear particle, when equillibrium is reached, will depend on the elastic energy stored in it. Continuous experiments with a Ball Milling machine brought up a few important points.

The values obtained for the average particle size in Ball Milling process compare very well with similar experiments in pure grinding. The results also compare well with the predicted value given by the theory of a loose wear particle. Load variation during the process has no effect on the final average size of the particles. Lubrication reduces the equillibrium size of a loose wear particle as predicted by the theory.

To conclude, the Ball Milling process is a grinding process and contains all the features predicted by the theory. Values for average grain size under various conditions can be siad to follow the following analytical expression:

$$d = 60{,}000 \; \frac{W}{P}$$

## II. LIST OF ILLUSTRATIONS

13) Copper ground in various jars with various size allumina balls. The graph shows the variation of average grain size with duration of the experiment.

14) Dial soap ground by itself, compared with the same soap ground with copper and then extracted from the mixture of soap and copper.

15) Glass ground by itself in the medium jar.

16) Copper ground with copper balls under two different humidity conditions.

## III. ACKNOWLEDGMENT

I would like to express my deep gratitude to Professor E. Rabinowicz, and to R. G. Foster, whose continuous help and consultation made it possible for me to complete my work.

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## IV. INTRODUCTION

The Ball Milling process is widely used in industry for the purpose of producing various metal powders. The average size of the particles constituting those powders have to meet certain specifications. Unfortunately very little is known with regard to the actual process and most metal powders produced presently are produced by trial and error.

It has been observed that a Ball Milling process is a grinding process. If the above assumption is true then the behavior of the ground particles should conform with the theory of a loose wear particle.

It is our intention to investigate the Ball Milling process and to determine its relation to other grinding processes. A further course of investigation will be to examine the theory of the loose wear particle and to see whether it can be applied to the Ball Milling process and how well do the experimental results agree with the theory.

## V. THEORY

The theory of a loose wear particle states that the equillibrium size of a wear particle is not random but is a function of the elastic energy stored in it.

E. Rabinowicz and R. G. Foster in "Effect of Surface Energy On the Wear Process" deal directly with the nature of the wear process. The analysis of the wear process led finally to a theoretical determination of the size of loose wear particle namely:

$$d = 60{,}000 \frac{W_{ab}}{P}$$

where:

d - equillibrium size of the loose wear particle.

$W_{ab}$ - Work of adhesion between two metals a and b ground together.

P - Penetration hardness of the metal.

The theory states that the above parameters are the only factors determining the size of the wear particles and all other factors which might seem relevent to the process should be discarded.

## VI. TEST EQUIPMENT

### a) Ball Milling Machine

The ball milling machine and the various sized jars are the basic equipment used to produce the loose wear particles. As seen in illustration no. (4) the machine has two levels operating concurrently. The jars lie between two long cylinders one of which is driven by the electric motor and the other rotates freely. The driven cylinder rotates at a constant speed. The jar is forced into motion as a result of the friction force between the jar and the driven cylinder. The space between the cylinders is adjustable to enable the use of various jars of different diameter. The jars used on the ball milling machine have 3 different sizes:

Small Jar

Medium Jar

Large Jar

Since the driven cylinder rotates at a constant speed and we assume no slip condition between the cylinder and the jar, the jar's wall rotates at an identical speed to that of the cylinder. The angular velocity of the different jars is different. The wall velocity of the jar has been found to be 100 ft/sec.

b) Sieves

A standard set of sieves was used to determine the average size of the particles. The set contained the following sizes:

| | |
|---|---|
| 2000 μ | 250 μ |
| 1410 μ | 177 μ |
| 1000 μ | 125 μ |
| 710 μ | 88 μ |
| 500 μ | 62 μ |
| 350 μ | 44 μ |
| | 44 μ |

Additional set was provided to determine sizes below 44 μ. The set had the following mesh sizes:

| | |
|---|---|
| 45 μ | 10 μ |
| 20 μ | 5 μ |

c) Electric Balance

The Electric Balance was used to determine the weight contained in each sieve. The weight could be read directly from the balance. The containers used for weighing the particles were small standard alluminum containers.

d) Dehumidification Equipment

One of our objectives was to provide dry air to the jars. To that end we used large plastic bags filled with "Drierite". The jars were introduced inside the plastic bags and when zero humidity condition had been established inside the bag the jars were closed and sealed. A humidity indicator provided us with information concerning the relative humidity inside the plastic bag.

## VII. MECHANISM OF THE BALL MILLING PROCESS

In order to gain more insight into the actual mechanism of the Ball Milling process and in an attempt to determine optimal operating speed, we mounted a variable speed motor on the ball milling machine and operated it with open jars. We examined three effects:

1) Operation at various speeds.
2) The motion inside the jars of various sized balls.
3) The motion inside in various sized jars.

It has been observed that the motion inside the jars was identical regardless of the possible combination of speed, ball size and jar size. The balls invariably remained near the bottom of the jar. They rotated with the same angular velocity of the jars and maintained a no slip condition with the walls of the jar as can be seen from figure 2. The balls arranged themselves in rows and at no time jumped over one another. The equillibrium position of the rotating balls remained at about 5 to 10 degrees from the bottom of the jar.

The above observation lead to some interesting conclusions. The size of the jar is irrelevant to the average size particle that is produced. It can be useful in producing bigger quantities. The angular speed with which the jars are rotated has no bearing on the average size of the particles produced. The mechanism remains the same regardless of the size of balls used.

## VIII. EXPERIMENTAL PROBLEMS

While experimenting with copper, silver and gold, it was noticed that the particles produced were black. The cause for the black color was believed to be either oxidation of the metal or adherence of some contaminant to the metal.

### a) Oxidation

The metals were assumed to have been oxidized because of the black color of the particles. The cause for the oxidation was believed to be the considerable humidity in the air. However, the argument failed to explain the black color in the case of the gold. Gold does not form any oxides. Also, when the copper was run with soap as a lubricant it maintained its original color. It is doubtful whether soap alone is sufficient to prevent the metal from oxidizing.

### b) Contaminants

Another trend of thought led to the belief that the jars' walls were previously contaminated with lubricants and during the new experiments the lubricants depleted from the walls and mixed with the particles. To test the validity of that assumption we took the following action. We heated the jars in a furnace to 500°F for a period of 2 hours. The purpose was to evaporate any possible organic contaminant. Before every experiment the jars were cleaned with acetone to prevent the collection of contaminants. Also the jars were cleaned with dilute Nitric Acid between one experiment to the other to dissolve any metallic residuals from previous experiments. None of the above measures seemed to change the results.

c) Initial Sample

The sample introduced into the jars consisted initially of thick chips cut from the desired metal. It has been found that there is some relation between the thickness of the chips and the black color. When the thickness of the chips was 10 thousands of an inch or less the original color was maintained. However, for thicker chips a gradual appearance of black color took place. If our assumption regarding oxidation proved to be valid then our solution could be explained in the form of increased surface area. The thin chips had a considerably larger total surface area than did the thick chips. Since the quantity of oxygen in the jar was fixed, the possibility of forming an oxide with the thin chips was reduced as compared to the thick chips. On the other hand if our assumption regarding contamination was valid then the phenomenon could be accounted for in the following manner. It would take a longer period of time to establish equillibrium between the walls of the jar and the metal ground inside it if the chips were thick as compared with thin chips. Therefore it would be more likely for a contaminant to deplete from the walls in the case of thick chips. A final answer has not been given yet. Further investigation in this direction seems to be worthwhile.

Another phenomenon associated with the initial sample was the shape of the chips. Starting chips of 10 thousands or below became flat as grinding time increased. On the other hand thicker chips tended to become round balls after a short period of time. The phenomenon might be explained by the mechanism of the ball milling process. The allumina balls when rolling tended to round objects resembling a square or a shere but would flaten objects below certain thickness.

## IX. METHOD OF EXPERIMENTATION AND MEASUREMENT

a) Jars

To be able to effectively experiment with the allumina jars it was necessary to clean the jars after every experiment. The most effective way to clean the jars was found to be in the use of acids. Dilute Nitric Acid was used in most cases where metals were ground except for gold. When gold was ground it was necessary to use Aqua Regia.

b) Maintaining equillibrium with the jars' walls

In order to get pure metal powder it was necessary to avoid depletion of the allumina from the balls or the walls. To achieve that it was necessary to establish equillibrium between the metal and the walls. To that end we used thin chips as the initial sample. The chips were ground to a powder quite rapidly and the walls were coated with a sufficient quantity of metal to prevent any depletion of the allumina. When the sample was taken out for sieving we did not scrape the balls or the walls. It was sufficient to shake it a few times. Any attempt to scrape the walls would disturb the existing equillibrium.

c) Sieving

To avoid any contamination of the powder when taken out for sieving, we found it most convenient to empty the jar directly into the sieve. The 2000 μ mesh is strong enough to absorb the impact of the falling balls. There was no loss in metal powder and that enabled us to observe changes in grain size distribution from one sieving to the other.

d) Size of Sample

The size of the required sample was found to vary greatly when using different size jars or different size balls. The size of the sample at different conditions should be as follows:

| | | |
|---|---|---|
| Medium jar | 5 big balls | 30-40 gms metal |
| Medium jar | 150 small balls | 70-80 gms metal |
| Big jar | 10 big balls | 70-80 gms metal |
| Big jar | 300 small balls | 120-130 gms metal |

## X. EXPERIMENTAL RESULTS

a) Ball Milling vs. Grinding

A great deal of information has been collected on various grinding processes. The results agree very well with the theory and the average size of the loose wear particle was found to obey the relation:

$$d = 60,000 \frac{W}{P}$$

One of our objectives was to determine whether the ball milling process, a grinding process, and how well do the results compare with other grinding processes. Values for the equillibrium particle size of copper was taken from S.M. Thesis by Robert G. Foster. For copper ground in atmospheric pressure at room temperature and dry air the equillibrium size of the loose wear particles were found to be 80μ.

We ran two types of experiments. The first consisted of allumina balls, allumina jars, and copper chips. The second type of experiment involved allumina jars, copper balls and copper chips. Graphs 12, 13 show the results. The different combination of jars and balls followed a different path as time went on. However, they all reached the same range of values toward equillibrium. Most values lie between 70 and 95 μ. It seems that the values for the average size particle are close enough to previously observed values to justify the conclusion that the ball milling process agrees well with the theory.

b) Average size vs. load

Theory predicted that the equillibrium size of a loose wear particle is a function of the work of adhesion and the penetration hardness only. To verify the above postulate we performed the following experiments:

<u>Silver</u>

Silver was ground in the medium jars for a period of 120 hours. One jar contained 5 big balls and the other contained the equivalent weight of 150 small balls. The results are shown in graph 11. The difference is average size after 120 hours was 15 μ.

<u>Copper with Allumina balls</u>

Copper was ground in the medium and big jars with various size allumina balls. The total weight of the balls was kept constant. The results are shown in graph 13. All the experiments show definite tendency to converge as equillibrium size was reached. After a period of 168 hours the range of the average particle size was 15 μ.

Copper with copper balls

Graph number 12 shows the results of grinding copper chips in allumina jars and copper balls. Each jar contained a different size ball. The graph shows a difference of 10 μ after 192 hours.

The above results show that load does not effect the average size of a loose wear particle. The experiments are consistant with one another and with the theory of a loose wear particle.

c) Lubrication

The theory predicts that an introduction of a lubricant with the ground metal will reduce the equillibrium size of the loose wear particle. Any lubricant introduced between two ground metals will reduce the work of adhesion and consequently the average size of the loose wear particles. To verify the above prediction we ground copper and soap in the big jars. The copper and soap were taken out and separated by dissolving the soap in water. The pure copper was then sieved. The results are shown in graph number 10. After a period of 120 hours the average size of the copper was 52.5 μ. That should be compared with copper that was ground under the same conditions but without any lubricant. The average size under those conditions was 80 μ. The difference is about 30 μ and agrees well with the theoretical prediction.

d) Effect of humidity on average size

To examine the effect of humidity on average size we ran two tests in the medium jar with big copper balls. The first test was run for a period of 192 hours at 40 % relative humidity. The second test was run for a period of 204 hours at 0% relative humidity. The results of those tests can be seen in figure 16. Both samples ended up at the same average size.

It seems that humidity has no effect on the average size of the particle at equillibrium. However it would be advisable to examine the results under more extreme conditions of humidity to ensure the validity of our results.

## XI. CONCLUSION

As was stated at the beginning the purpose of the thesis was to establish the relation between the ball milling process to other grinding processes. The results obtained from ball milling various metals show a very good agreement with the results obtained in other grinding process.

We also stove to establish the correlation between the theory of a loose wear particle and the ball milling process. The results conform well with the theoretical predictions. Load variations, use of lubricants and changes in humidity had the predicted effect on the average grain size at equillibrium.

## XII. RECOMMENDATIONS

Many problems and points of interest have arisen during the experiments. Few of them were resolved by us and few remained unresolved. It seems that further investigation into those problems is warranted.

### Reproducibility

It has been mentioned earlier that on many occasions the specimen taken out of the jars were black. In order to be able to get the original color of the metal we used thin chips. However the phenomenon itself has not been explained satisfactorily. It is suggested that further experimentation will be carried out in that direction to establish the cause of the phenomenon.

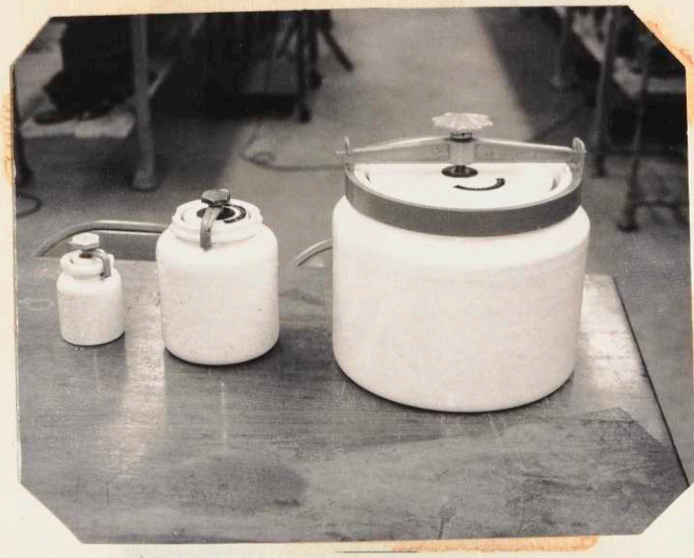
### Effect of speed on average size

While observing the internal mechanism of the ball milling process we came to the conclusion that speed would have very little or no effect on the average size of the particles. However no experiments were carried to verify our assumption. We suggest further examination of that point.

### Free Fall

During our experiments it has been suggested by Professor E. Rabinowicz to build a hollow cylinder closed at both ends.

The cylinder would contain chunks of metal. It will be mounted in its center on a shaft and will be rotated so that the chunks of metal would fall freely from one end of the cylinder to the other. The cylinder would be connected to an electro magnet that would rotate half a revolution at a time and would rest for a short period of time while the metal chunks travel from one end to the other. Due to lack of time no such device was built. We suggest further investigation into this process.



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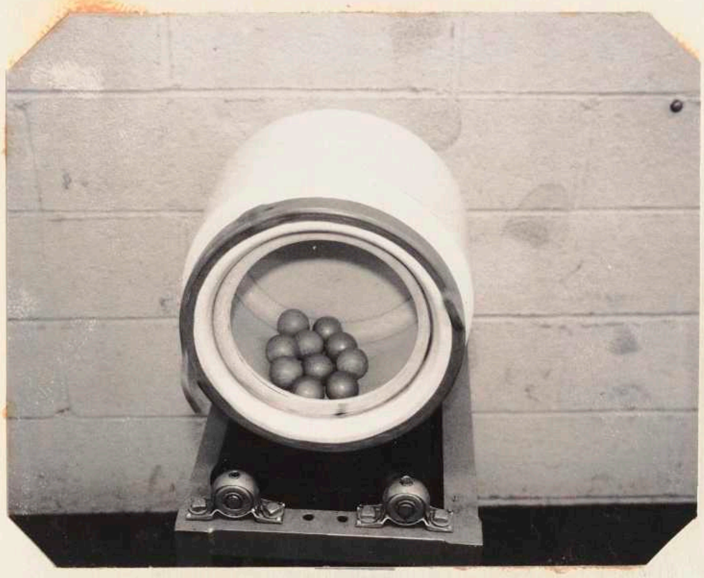

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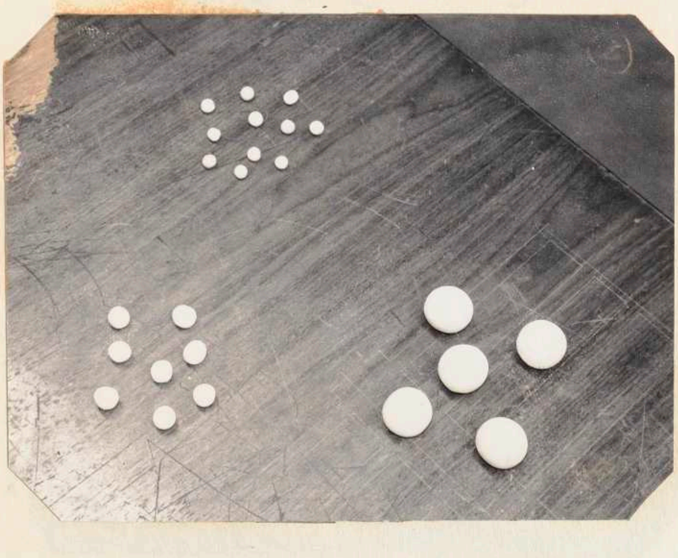

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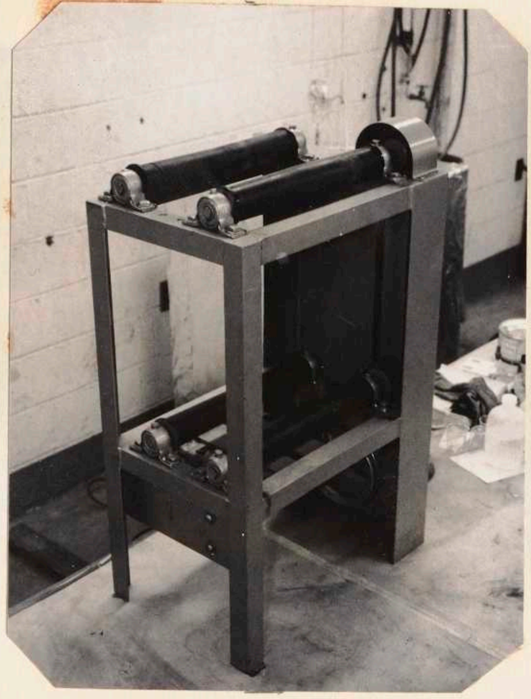

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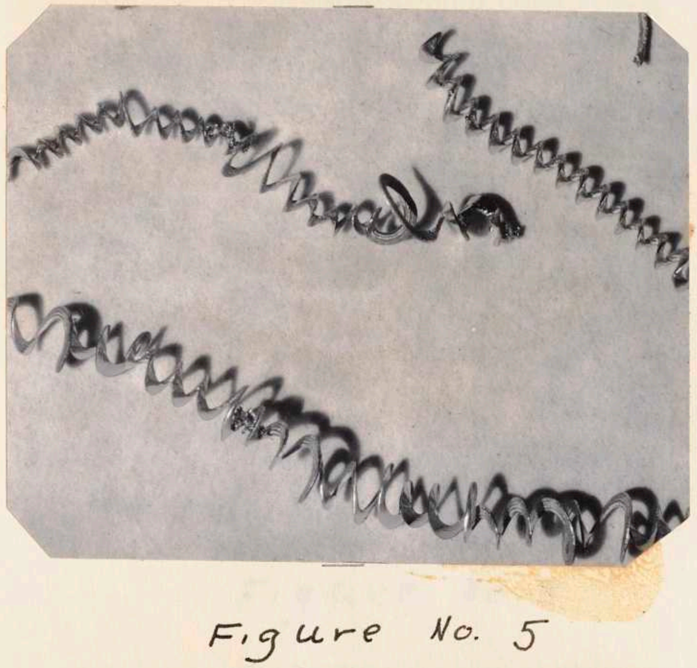

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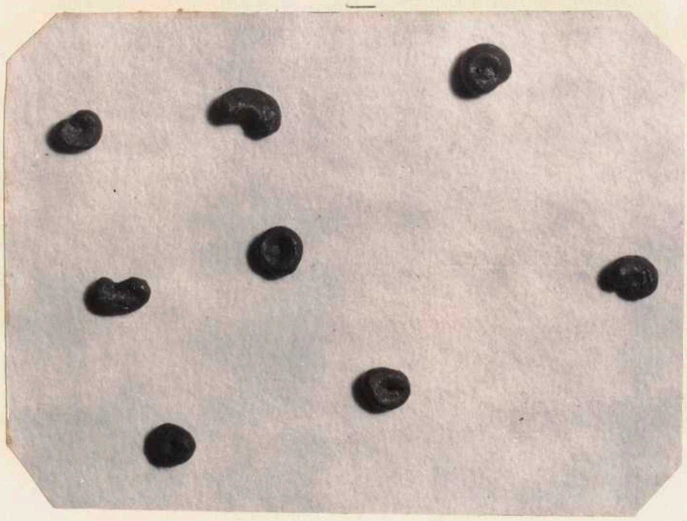

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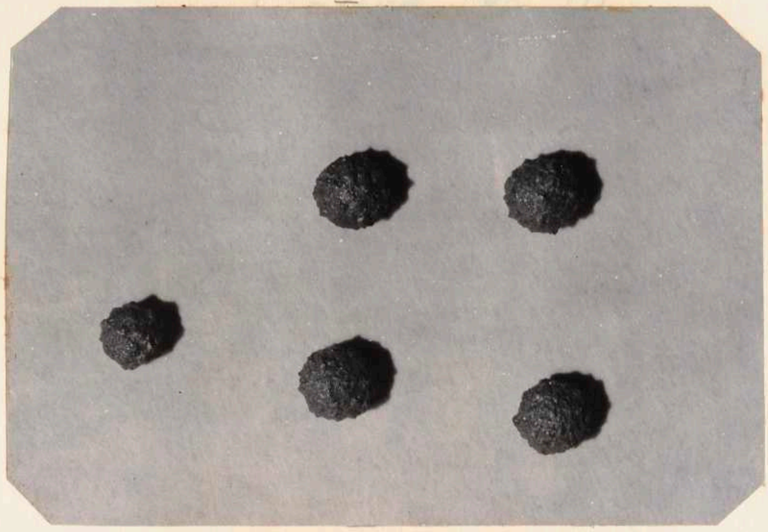

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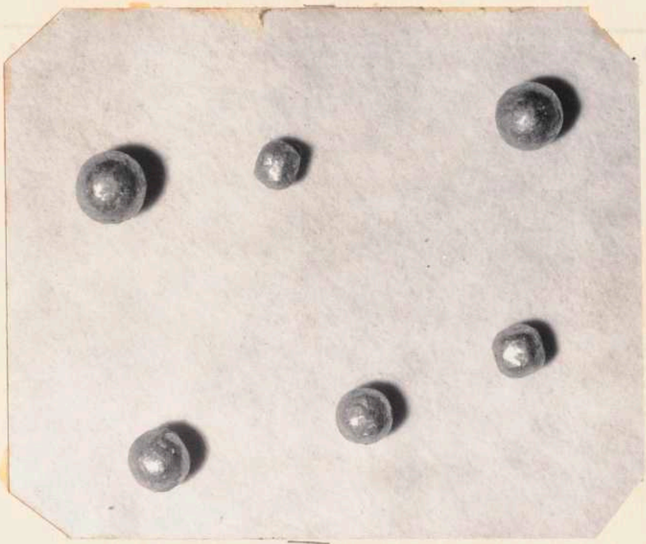

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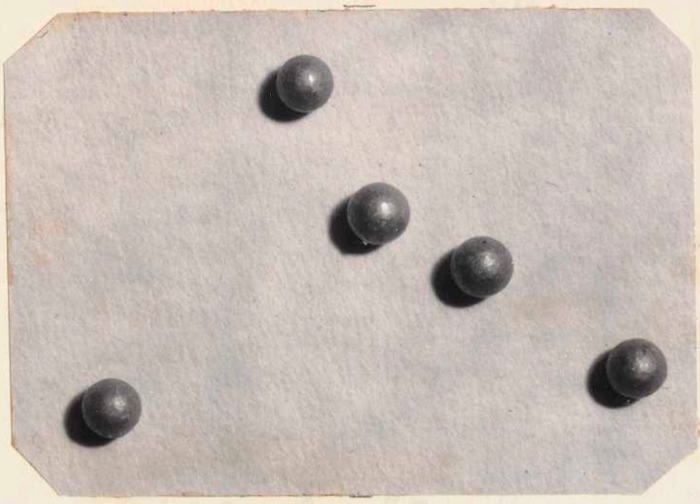
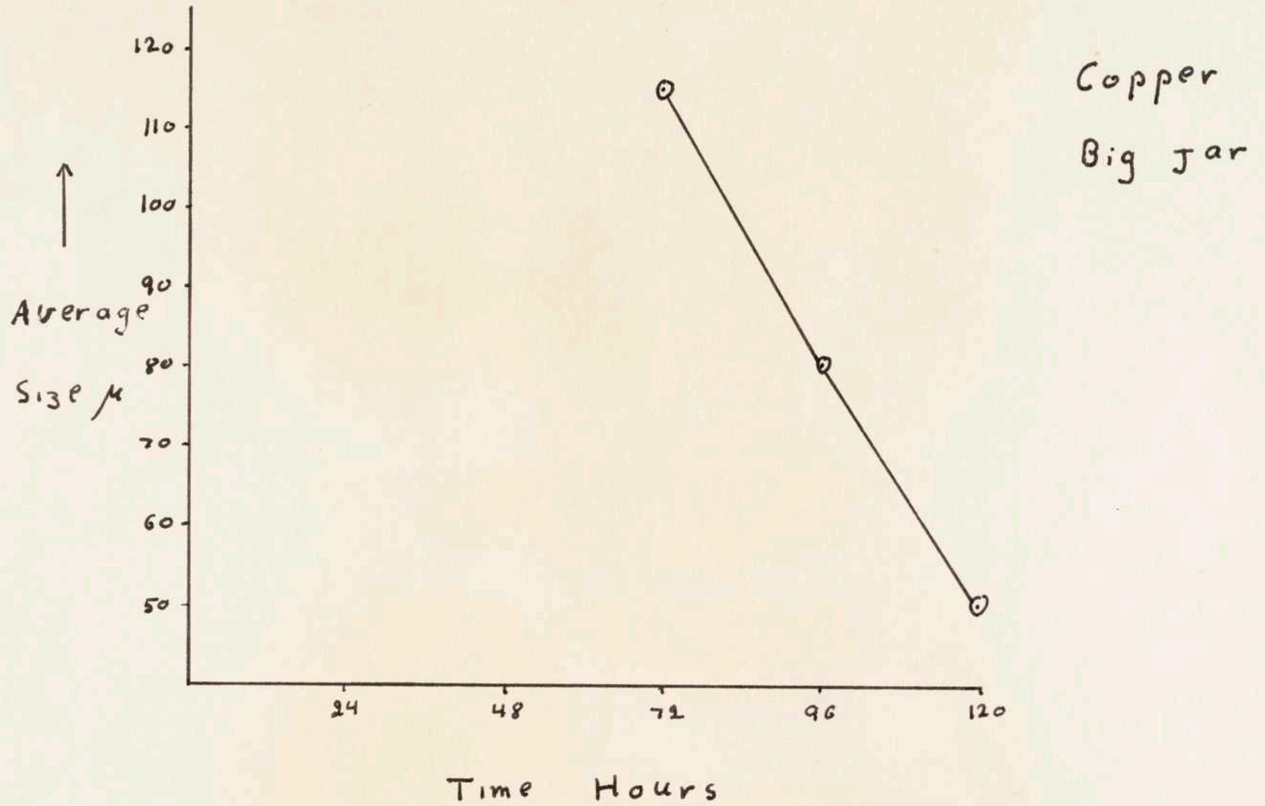


Figure No. 9.

Figure No. 10



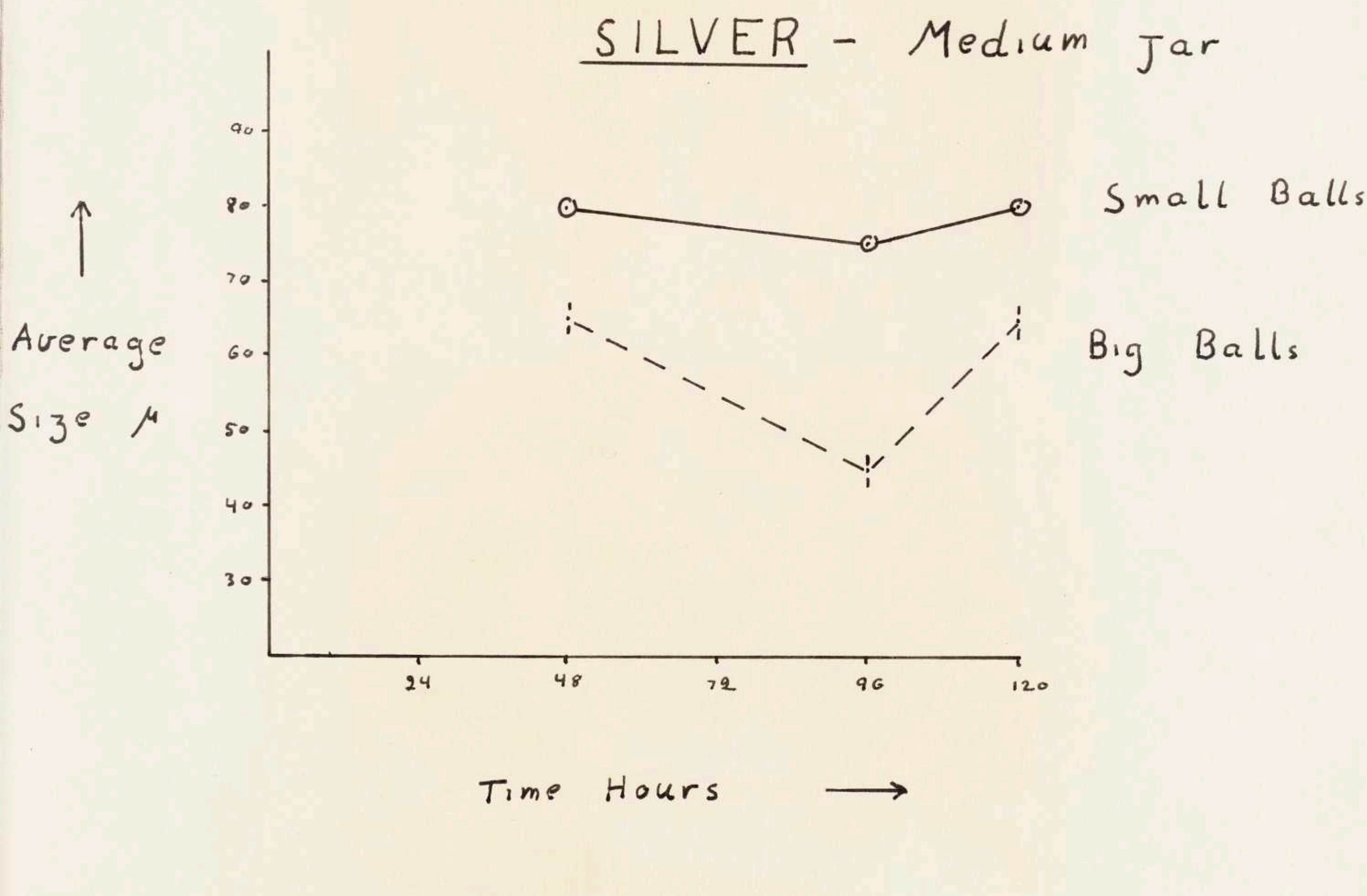
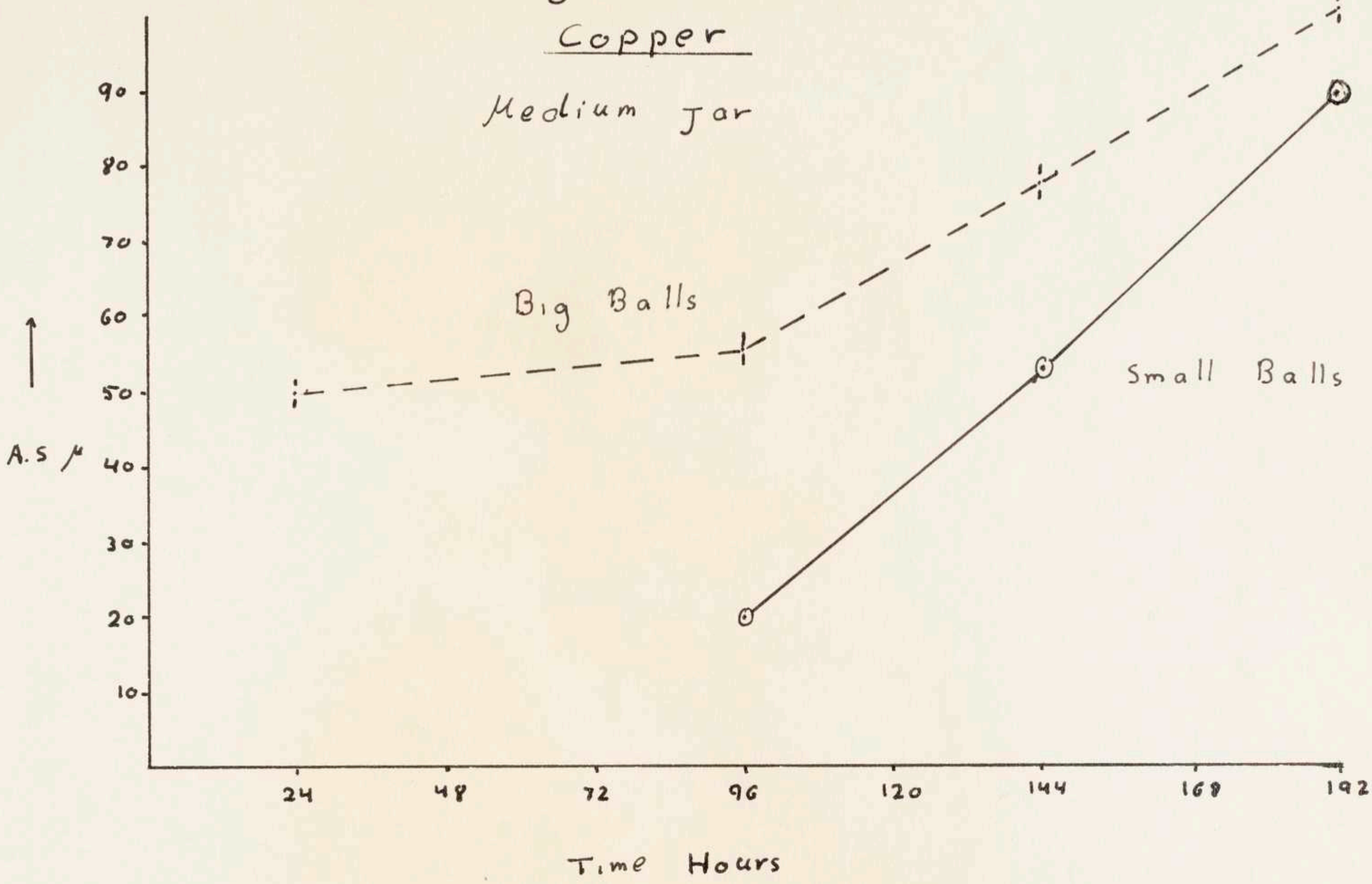




Figure No. 11

30

# Figure No. 12

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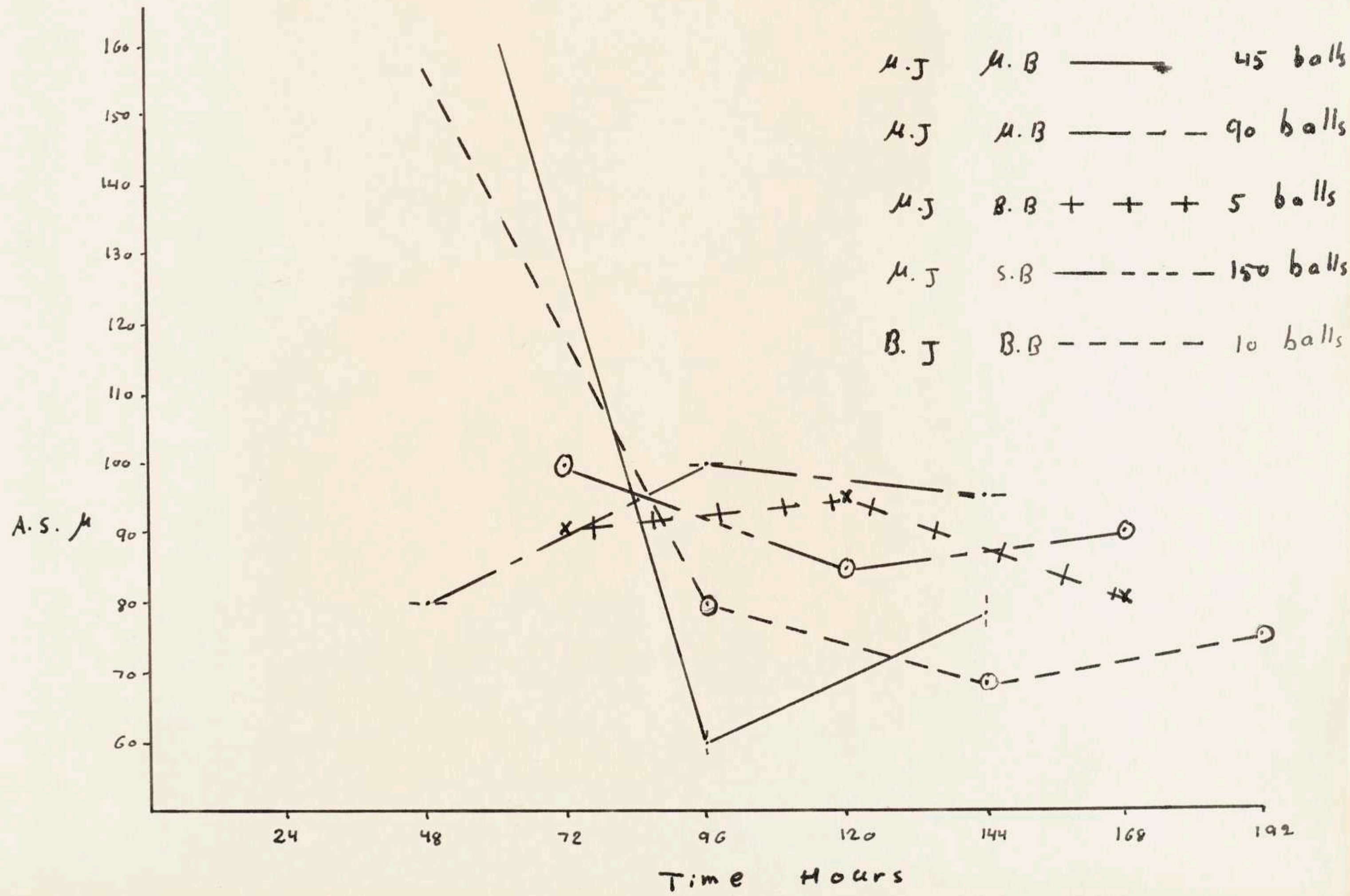


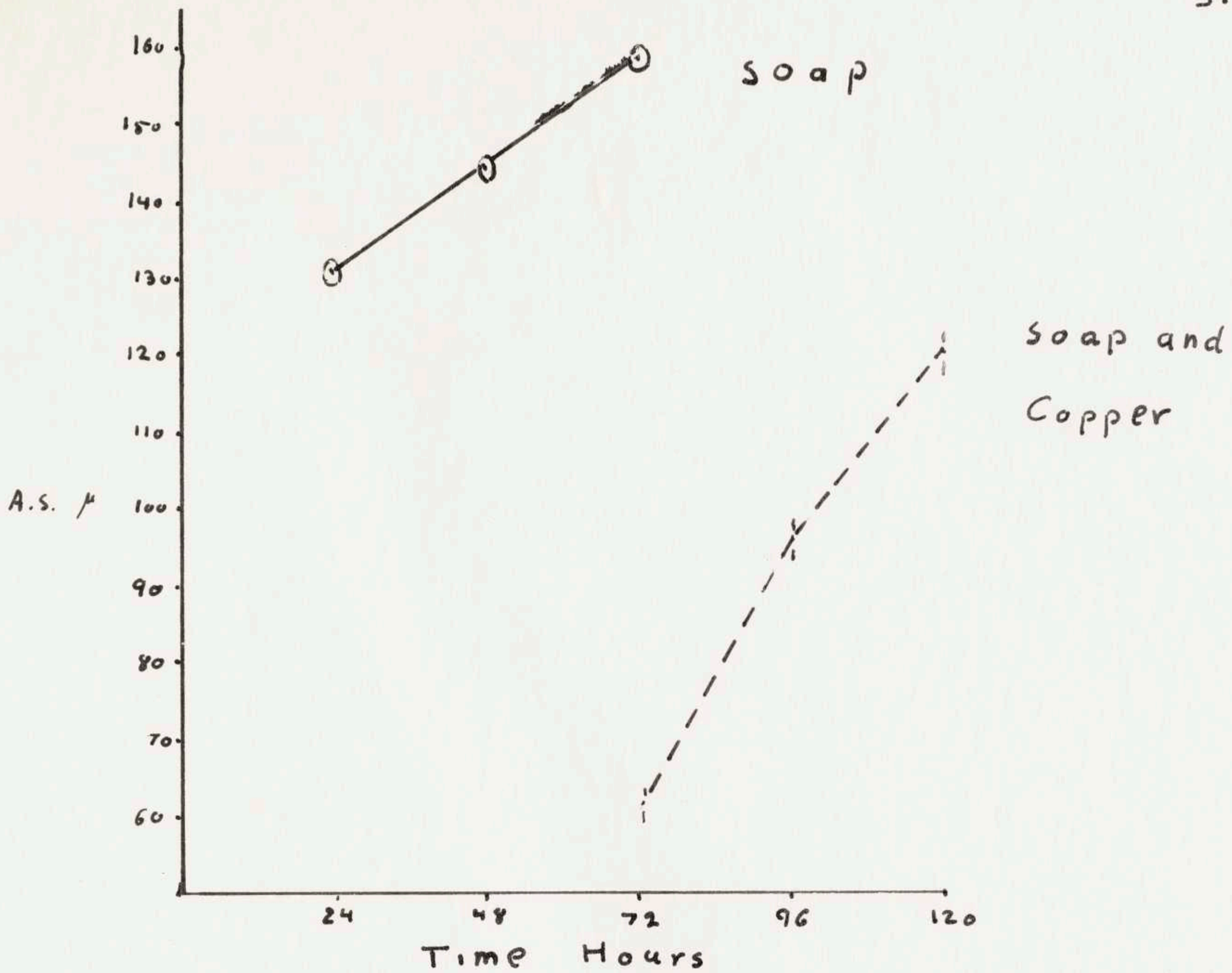
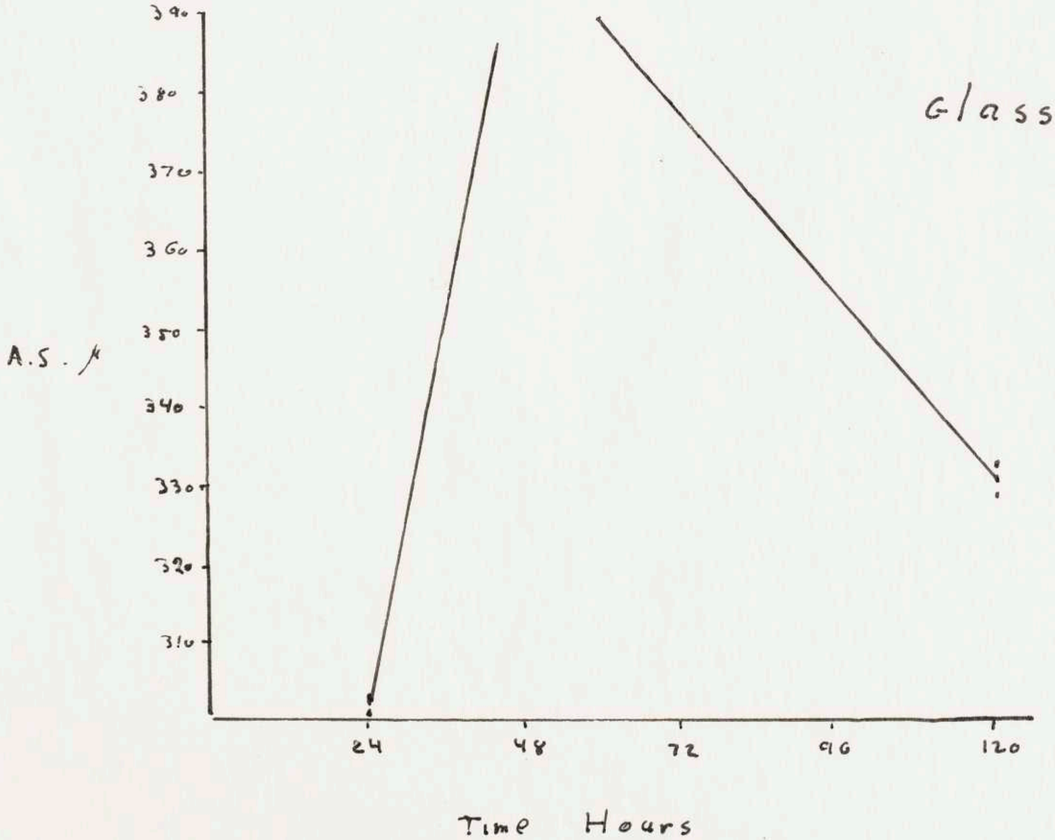



Figure No. 14

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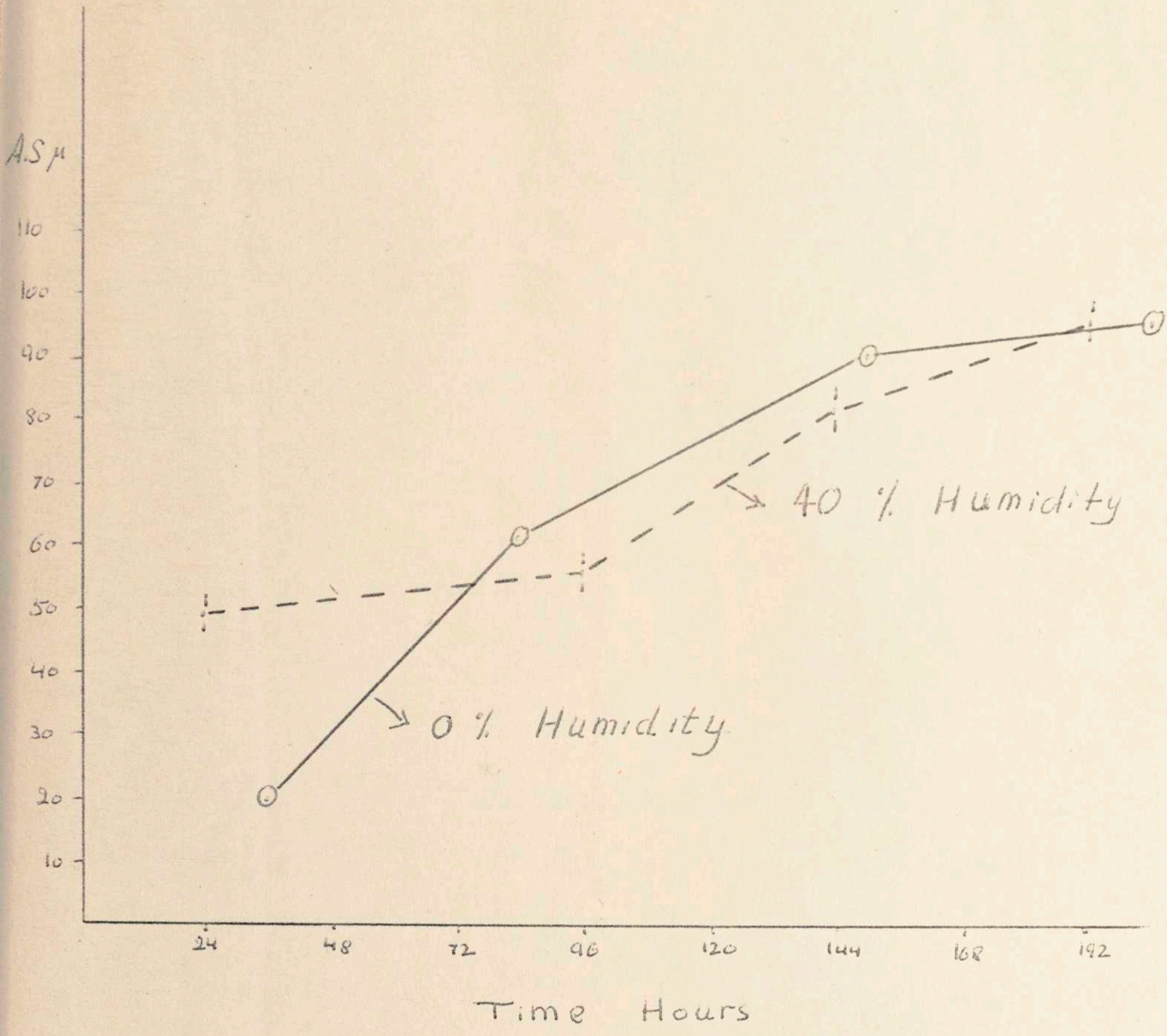
A.S μ
110
100
90
80
70
60
50
40
30
20
10
40 % Humidity
0 % Humidity
24
48
72
96
120
144
168
192
Time Hours

Figure No. 16

## APPENDIX A

Data for graph number 10

Copper extracted from the ground mixture of copper and soap.

| Material | Size of Jar | Duration Hours | Average Size μ |
|---|---|---|---|
| Copper | Big | 72 | 115 |
| Copper | Big | 96 | 79.5 |
| Copper | Big | 120 | 52.5 |

Data for graph number 11

| Material | Ball Size | Jar Size | Duration Hours | Average Size μ |
|---|---|---|---|---|
| Silver | Small | Medium | 48 | 81 |
| Silver | Small | Medium | 96 | 74.5 |
| Silver | Small | Medium | 120 | 79 |
| Silver | Big | Medium | 48 | 64 |
| Silver | Big | Medium | 96 | 44 |
| Silver | Big | Medium | 120 | 64 |

Data for graph number 12

Utilizing copper balls instead of allumina balls

| Material | Ball Size | Jar Size | Duration Hours | Average Size μ |
|---|---|---|---|---|
| Copper | 7 Big | Medium | 24 | 30 |
| Copper | 7 Big | Medium | 96 | 20 |
| Copper | 7 Big | Medium | 144 | 52 |
| Copper | 7 Big | Medium | 192 | 83 |
| Copper | 50 Small | Medium | 24 | 48.5 |
| Copper | 50 Small | Medium | 96 | 56 |
| Copper | 50 Small | Medium | 144 | 82 |
| Copper | 50 Small | Medium | 192 | 95 |

Data for graph number 13
Using allumina balls

| Material | Ball Size | Jar Size | Duration Hours | Average Size μ |
|---|---|---|---|---|
| Copper | 150 Small | Medium | 72 | 100 |
| Copper | 150 Small | Medium | 120 | 85 |
| Copper | 150 Small | Medium | 168 | 90 |
| Copper | 5 Big | Medium | 72 | 89.5 |
| Copper | 5 Big | Medium | 120 | 96 |
| Copper | 5 Big | Medium | 168 | 80.5 |
| Copper | 45 Medium | Medium | 48 | 182 |
| Copper | 45 Medium | Medium | 96 | 58 |
| Copper | 45 Medium | Medium | 144 | 72 |
| Copper | 45 Medium | Medium | 192 | 59 |
| Copper | 90 Medium | Medium | 48 | 80 |
| Copper | 90 Medium | Medium | 96 | 101.5 |
| Copper | 90 Medium | Medium | 144 | 96 |
| Copper | 90 Medium | Medium | 192 | 97 |
| Copper | 10 Big | Big | 24 | 197 |
| Copper | 10 Big | Big | 96 | 84 |
| Copper | 10 Big | Big | 144 | 68 |
| Copper | 10 Big | Big | 192 | 176 |

Data for figure number 14

Soap extracted from soap and copper mixture compared with soap ground by itself.

| Material | Ball Size | Jar Size | Duration Hours | Average Size μ |
|---|---|---|---|---|
| Soap ground with Copper | Big | Big | 72 | 62 |
| | Big | Big | 96 | 96.5 |
| | Big | Big | 120 | 120.5 |
| Soap | Big | Big | 24 | 131 |
| Soap | Big | Big | 48 | 149 |
| Soap | Big | Big | 72 | 163 |

Data for graph number 15

| Material | Ball Size | Jar Size | Duration Hours | Average Size μ |
|---|---|---|---|---|
| Glass | Big | Medium | 24 | 298 |
| Glass | Big | Medium | 48 | 402 |
| Glass | Big | Medium | 120 | 331 |

Data for graph number 16

| Material | Ball Size | Jar Size | Relative Humidity | Duration Hours | Average Size μ |
|---|---|---|---|---|---|
| Copper | Big | Medium | 0% | 24 | 48.5 |
| Copper | Big | Medium | 0% | 96 | 56 |
| Copper | Big | Medium | 0% | 144 | 82 |
| Copper | Big | Medium | 0% | 192 | 95 |
| Copper | Big | Medium | 40% | 36 | 20 |
| Copper | Big | Medium | 40% | 84 | 62 |
| Copper | Big | Medium | 40% | 150 | 90 |
| Copper | Big | Medium | 40% | 204 | 95 |

APPENDIX B

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