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PROJECT 9536

R. Ohe - 11-14 - C
Chemistry Section C7X

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DATE 10-24-46

SUBJECT Pile Chemistry and ~~General~~

Week Ending October 23, ~~1946 WEEKLY~~

Section C-

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ANL-OCS-77
This document consists of 8 pages and 2 figures.

WEEKLY ABSTRACTS

October 24, 1946

Section C-II

Pile Chemistry and General Chemistry

Week Ending October 23, 1946

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INV. 0-47

HIGH TEMPERATURE PILE PROGRAM
(C. A. Boyd)

Reaction of Steam and BeO (M. G. Berkman)

Additional experiments were carried out to determine the effect of varying water vapor pressure and steam flow rate on the reaction between steam and BeO. The time for each run was 2 1/2 hours.

Run	Temp. of BeO pellet °C	Rate of Steam Condensation ml/min	Water Vapor Pressure mm Hg	Weight of BeO lost %
49	1400	0.4	60	0.40
50	1400	0.007	50-60	0.13

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JE Savely 4-20

The experimental set-up is now being modified for the purpose of making it possible to increase the steam flow rate and still keep the system at a relatively low pressure. The efficiency of the condensers (or traps) in the system must be increased in order to realize the desired end.

Graphite Impregnation (M. A. Kanter, R. W. Phillips, A. L. King)

Tests on the stability of solutions of UNH in hexone at elevated temperatures were concluded. No suitable inhibitor for the decomposition of the solution was found so that the impregnation work is now being directed toward the use of other solvents.

In an effort to increase the absorption of UNH from aqueous solutions, the use of wetting agents was investigated. Twenty-one of the commercial agents have been tested for solubility in UNH water (conc. 34.5 gms/100cc) and for wetting power toward graphite. On the basis of these tests, nine have been selected as possible agents. To date impregnations of small graphite cylinders were made in the manner described in ANL-OCS-57 from aqueous solutions of UNH containing small percentages of wetting agents. The results are as follows:

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Wetting Agent	Percent Weight Increase, 3 Samples		
None	1.80	1.67	1.85
Aerosol 1B	2.07	1.80	1.65
Tergitol 80	1.68	1.97	2.22
Naccconol NRSF	2.35	2.35	2.30
Duponol 80	1.91	1.46	2.11
Duponol ME	2.25	2.10	2.21
Duponol G	1.82	2.06	2.25

All solutions contain 34.5 gms of UNH per 100 cc of solution plus fractional percentages of the wetting agents. Of the agents considered so far, only two, Naccconol NRSF and Duponol ME, show a positive reproducible gain over the use of pure aqueous solutions. Some fifty-odd commercially available agents are yet to be investigated.

Annealing of Irradiated Samples (J. L. Weeks)

Annealing run #V was made under vacuum at 900°C for ~ 3 hours. Results of linear changes for irradiated mixed oxide samples are given in Table 2. The standard unirradiated samples showed no change. Due to the fact that the improvements on the thermal conductivity apparatus have not been completed, no such measurements were made after run #V. Any definite conclusion as to the validity of these results must await further runs with corresponding thermal conductivity determinations. However, these results do seem to indicate that complete annealing may be obtainable.

Future work will include the construction of an improved annealing apparatus which will allow the samples to be heated at the desired temperature for specified time, instead of the present method of allowing several hours for the samples to heat up to temperature. As thorough an investigation as possible will then be made on the variation of the annealing rate with time as well as the determination of the threshold temperature for annealing, if such there be.

It is planned to start annealing tests on additional prisms from the 63 day and 130 day bombardments as soon as modulus determinations have been completed.

Table 2

Sample Ident.	Original Length	After 25 day Irrad.	% Change from Orig.	After Annealing Run #*	% Linear Dimension Change Annealed Out
#26 (pellet), H.D., H-90, 2% UO_2	0.2421	0.2437	+ 0.66	0.2422	94
#27 (pellet), H.D., H-90, 2% UO_2	0.2742	0.2759	+ 0.64	0.2748	76
C-25 (prism) H.D., H-87, 2% UO_2	1.7338	1.7418	+ 0.46	1.7373	56

* See Table 2, ANL-OCS-53 for conditions and results of runs I through IV.

Irradiation Effects on BeO and BeO-UO_2 Samples (W. L. Weeks, S. R. Gaarder, D. Rich, J. R. Gilbreath)

The third Hanford bombardment (Bombardment 9-S; 133-day irradiation) has been received and all of the sample cans have been transferred from the original containers to smaller lead shields and have been transported to New Chemistry.

All of the cans containing pure BeO have been opened and all of the prisms and pellets have been measured for possible changes in linear dimensions. It is interesting to note that after cutting partially through the lid on one of the cans, there was sufficient pressure inside this can to blow the lid off with moderate violence scattering several of the pellets. Fortunately, this did not occur on opening the rest of the cans, and consequently it may have been due to impurities present in this particular can. However, great care will be taken in opening the BeO-UO_2 cans as a similar accident in that case would be quite serious.

A summary of all the data which has been obtained from the three bombardments as far as linear changes in pure BeO samples are concerned is shown in Table 1. Although these data are somewhat inconsistent, it is apparent that irradiation does cause a slight expansion of hot-pressed BeO. This expansion, however, is definitely not linear with the time of bombardment. Furthermore, the actual expansion observed in all cases but one was less than 0.1%.

The thermal conductivity of the BeO prisms will be determined as soon as a new thermal conductivity apparatus is finished by the shop. The remainder of the cans containing the mixed oxide samples cannot be opened until a stack is installed on the hot laboratory. This is to be finished by next Monday, but at least another week will elapse before any measurements can be made on the new mixed oxide samples since the hot laboratory is to be used by Karp for his elastic modulus measurements on active samples.

Table 2

Average Percent Expansion of BeO Samples Under Various Hanford Bombardment Times

Norton Mat Co	24 days Bombardment	63 days Bombardment	252 days Bombardment
Pellets			
B-58	0.20%	0.04%	0.06%
B-59	0.9	0.05	0.96
B-85a2		0.02	0.00
B-85a2	0.95	0.28	
B-85a5			0.20
Prisms			
B-60		0.92 (4)	0.04
B-256		0.06 (2)	0.03 (1)
B-8		0.02 (4)	0.01 (4)
B-158			0.05 (5)

(Numbers in parentheses indicate the number of samples.)

Petrification Tests on Impregnated Graphite (J. Mason, J. Zasler)

Difficulty is being experienced in using the mercury gap converter because of overheating of the high frequency leads; heating tests will begin as soon as this difficulty is overcome.

It is planned to determine amounts of U volatilized on heating by a colorimetric method capable of detecting microgram quantities of U (the method is described in Mon-C-28). Standard curves for this procedure are being drawn up.

ANALYTICAL PROBLEMS

(R. S. Hopkins)

Wet Chemical Analysis (R. Dean, R. Hagelhoen, R. Jensen, R. Telford)

R. Services

1. Four alloys of uranium, columbium, and zirconium were analyzed for columbium and zirconium.
2. One uranium graphite ignition residue was analyzed for silicon, iron, and uranium.
3. Three chapters of the PPR were read and edited.
4. The analysis of beryllium for fluorine will continue.

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3. Two thorium samples were analyzed for carbon.
4. One uranium oxide sample was analyzed for water and loss on ignition.

B. Research:

1. Another run was made in the $BeCl_2$ volatilization apparatus. The quartz tube became plugged with $BeCl_2$ and carbon was found in the residue. The latter condition appears to be the result of using Tygon tubing. More changes are being made in the apparatus.

2. The polarographic analysis of a standard beryllium sample for lead was completed. The standard curve was prepared by adding known amounts of lead to a solution made by dissolving two grams of beryllium containing less than 5 ppm of lead in 30 ml of hydrochloric acid. Two grams of the sample were dissolved in 30 ml of hydrochloric acid for the determination.

Preliminary experiments indicate that a 20% increase in the beryllium results in a 20% decrease in wave height. The addition of 5 ml of hydrochloric acid to a control produces about the same decrease in wave height.

Spectrographic Laboratory (F. Tomkins, J. R. Brady, C. Battersea, R. Faris, M. Walsh)

A. Service Analyses:

During the past week the following analyses were completed:

1. One sample each of HCl and HNO_3 were analyzed quantitatively for all impurities.
2. Two samples of Be were analyzed semi-quantitatively for all impurities.
3. Seventeen samples of BeO were analyzed quantitatively for B and other impurities with strong lines in the ultraviolet.
4. Two samples of zinc ores qualitatively.
5. One sample of $BeCl_2$ quantitatively.
6. One sample of zinc-beryllium silicate quantitatively for impurities.

B. Research:

Microphotometric Methods. Acceptable working curves have been obtained for B, Mn, Pb, Sn over a concentration range of .1 ppm to 100 ppm. The concentration range was extended by means of a step sector.

A few samples which had been run a short time previously were repeated using the microphotometric procedure. The results are shown in Table I.

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Table I
Comparison of Visual and Microphotometric Methods

Sample	12671		12672		12673		12674		12675		12676	
Method	Visual Comp.	Micro-Photom.	Visual Comp.	Micro-Photom.	Visual Comp.	Micro-Photom.	Visual Comp.	Micro-Photom.	Visual Comp.	Micro-Photom.	Visual Comp.	Mic... Photom.
B	1	0.4	1.5	2.25	1.5	1.1	2	2.2	1.5	2.2	2.0	1.3
MN	3	2.5	5	10	3	3.2	3	2.9	3.0	3.2	3.0	1.4

A set of "unknown" samples have been made by Patterson and will be run as samples by others in the laboratory. The results will be compared with the known B, Pb, Mn, Sn, content of the standard "unknowns". The first determinations have been nearly completed. The reproducibility on a given sample will also be determined.

SPECIAL PROBLEMS

(H. C. Simpson)

Quantitative Determination of Hydrogen in Na-K Alloy, (F. L. Belletire, M. Rebenak H. C. Andrews)

Full time was put in this week assembling the equipment for the Na-K analysis and for the purification of oxygen and argon used in this analysis. Fire resistant suits, masks, etc. are being furnished by the safety department to be used when we are actually handling the Na-K.

Remote Control Development (F. L. Belletire, R. Miller, R. W. Holmes, H. C. Andrews)

The hydraulic tongs have been completely redesigned and the machine shop has begun building them.

A short meeting was held this morning in regard to the progress in renovating hot lab B-2. Mr. G. B. Thorgney of the construction division, C. A. Boyd and R. Gilbreath of the high temperature pile group, and H. C. Andrews of the special problems group, were present. The need for the completion of B-2 was stressed and Mr. Thorgney felt that it can be done in two months or less. The present bottleneck is the fabrication of the heavy steel and lead doors for the wall. The factory which is making them promises delivery in about two weeks from now. After the doors have been delivered, construction work should move rapidly to completion.

It is planned to make a complete report of the hot lab and remote control development this coming week.

Thermal Conductivity by Heat Wave Method (M. B. Rebenak, F. L. Belletire, H. C. Andrews)

The thermal conductivity of pure BeO prisms of the 120 day bombardment is being determined and measurements should be completed in a few days.

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GRAPHITE PROGRAM

(T. O. Wentorf)

Sykes Investigations (R. B. Sosman)

In the measurement of stored energy accumulation in bombarded graphite by the Sykes method, it was thought advisable to attempt to anneal samples at various temperatures in the same manner in which the Sykes furnace itself behaves and to then quench them rapidly to forego in all remaining stored energy.

The Sykes method is essentially a dynamic method of studying the bombardment disturbances of graphite, since the accumulated disturbances are healed at the same time measurements are taken. It was considered possible that changes in the specific heat of the samples at elevated temperatures not due to the bombardment disturbances might be distorting the relationships obtained so far. The annealing process and subsequent study of annealed samples in the usual manner may protect the measurements from such extraneous variations.

An auxiliary multiple-unit furnace was used, the temperature being controlled by manually varying the power supply with a Variac. The same pieces machined in the usual manner for the Sykes furnace were enclosed in a quartz tube sealed at one end. The tube was then held upright in the furnace. An iron-constantan thermocouple several millimeters from the quartz tube was used to measure the temperature. The tube was evacuated and filled with hydrogen to about one atmosphere pressure.

By frequent observation of the block temperature and slight adjustment of the Variac, the heating rate of the sample could be made to approximate closely that which it would have in the Sykes furnace. When the proper temperature had been reached, the tube was quickly withdrawn from the furnace and plunged into a bath of water.

One sample has been annealed at 200°C, another at 500°C. The accompanying graph shows the Sykes method determination of the ratio of the specific heat of the former sample to that of an inert control as a function of the temperature of the sample. On the same scale are shown a second determination after the stored energy had been heated below 500°C during the first run, and also a previous determination of a similar but unannealed sample from the same bar ("T"-bar, "E"-end). A similar plot of the 500°C annealed sample is also provided.

The data for the 500°C annealed sample are being calculated and the other samples are being quenched at other temperatures for further study.

Activation Energy Spectrum by Weak Resistance Measurement of Irradiated Graphite (C. Smith)

Experiments have continued as described in ANL-OCS-25 (8/1/46), and Guide 51 (8/29/46), using a sample from E-bar graphite irradiated for

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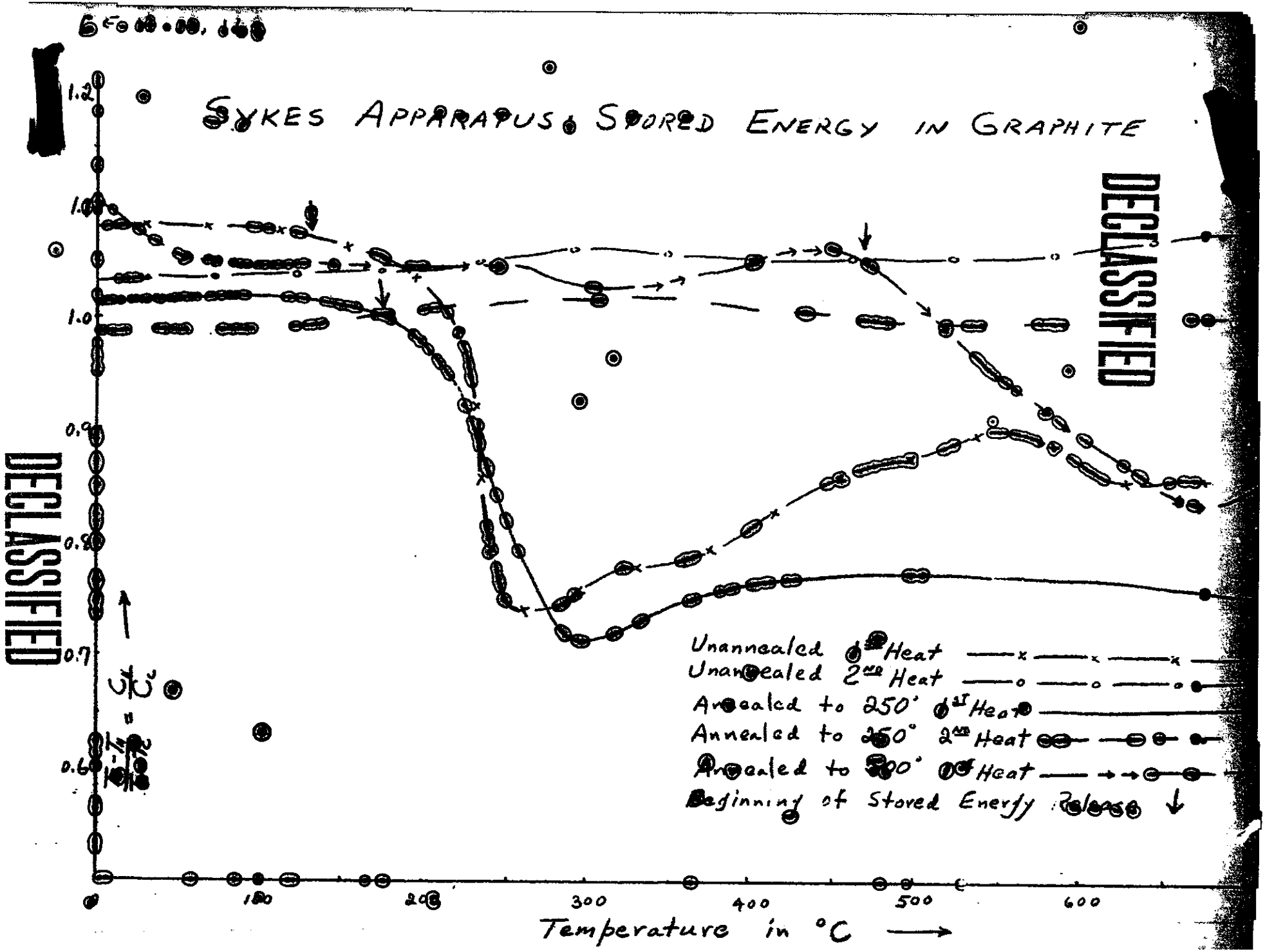
SYKES APPARATUS, STORED ENERGY IN GRAPHITE
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1.2
1.0
1.0
0.9
0.8
0.7
0.6
0
100
200
300
400
500
600
Temperature in °C
Unannealed 1st Heat
Unannealed 2nd Heat
Annealed to 250° 1st Heat
Annealed to 250° 2nd Heat
Annealed to 300° 1st Heat
Beginning of Stored Energy Release

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[illegible] made at Clinton in [illegible] on an unirradiated Whiting graphite sample for a control. The results have not been completely calculated yet, but a curve of resistivity vs temperature (see below) has been plotted from the data. The lower curve is for the Whiting blank, and the upper one shows the resistance change of the irradiated sample, as it heals. Readings were continued during the cooling but a breakdown of the Brown recording potentiometer at ~240°C made it impossible to get a complete return run. The cooling curve exactly matches the heating curve for the blank sample. On the Bubay sample it matches above ~540°C, showing that little healing takes place between this temperature and the upper limit reached in this run.

During the coming week it is planned to [illegible] activation [illegible] the temperature [illegible] as outlined by [illegible]

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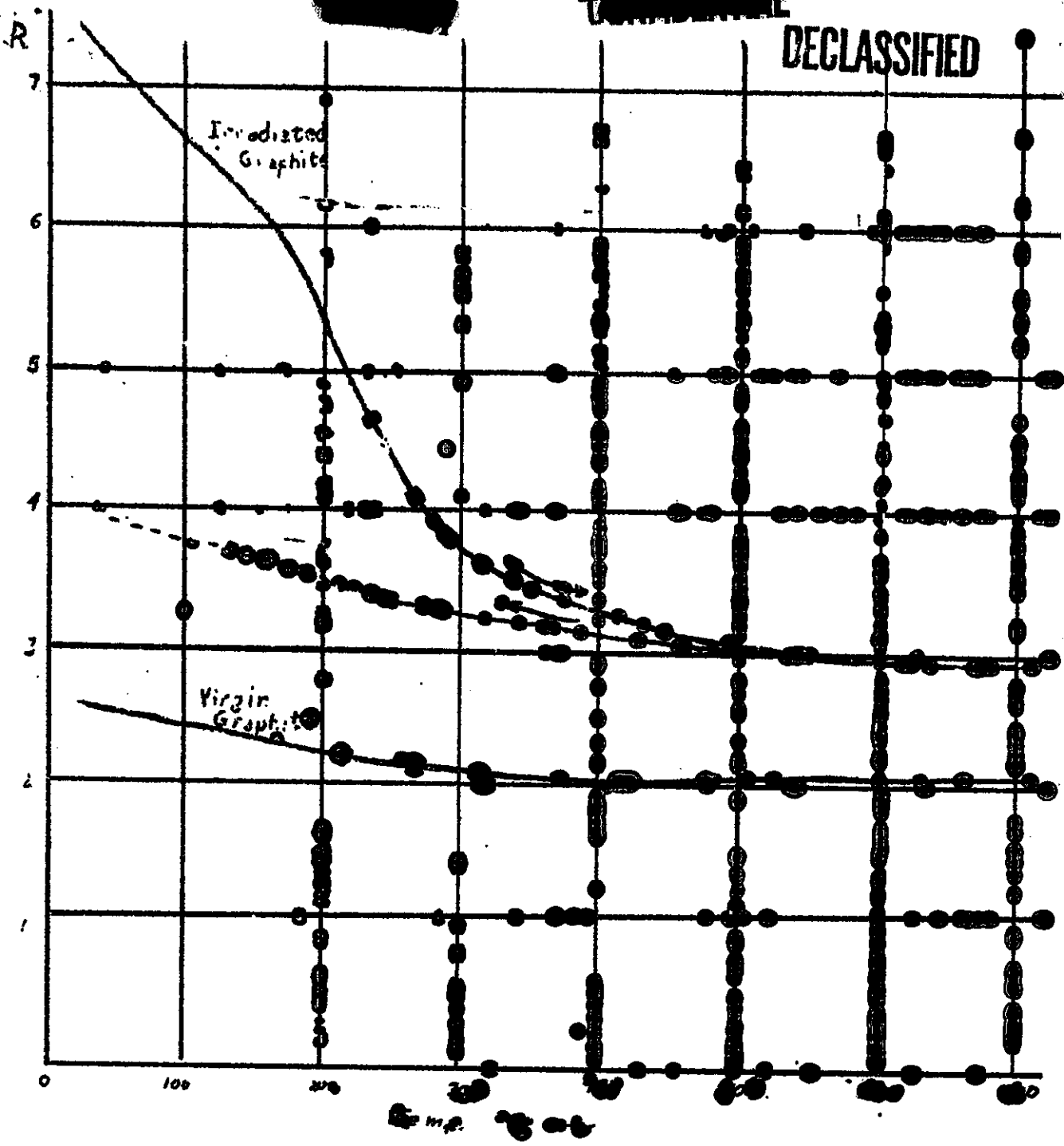

Resistance Vs Temperature

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