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GRAPHITE RADIATION DAMAGE AS A REACTOR OPERATIONAL PROBLEM

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GRAPHITE RADIATION DAMAGE  
AS A REACTOR OPERATIONAL PROBLEM

BY

D. H. Curtiss

Irradiation Processing Department  
Hanford Atomic Products Operation  
General Electric Company  
Richland, Washington

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By D. H. Curtiss

Irradiation Processing Department  
Hanford Atomic Products Operation  
General Electric Company  
Richland, Washington

ABSTRACT

Radiation Damage to the graphite moderator has played an important role in the history of reactor operation at Hanford. The operational problems, potential and real, which result from dimensional instability, stored energy, and gasification, as well as the solutions to these problems are discussed. The key to the graphite problems is operating temperature and its control. At present, stack expansion remains in the fringe regions of the original reactors, which coupled with the contraction of the central region gives rise to curvature problems in the top portion of the reactors. Stored energy is not a safety hazard because the release spectrum is such that a spontaneous and self sustained release cannot occur. Oxidation of the graphite moderator is controlled by maintaining operating temperature of the stack such that no significant amount of oxidation occurs.

INTRODUCTION

In a horizontal tube graphite moderated reactor, the graphite serves the dual function of moderation of the fast neutrons and structural support of the fuel elements, process tubes, coolant and parts of the reactor shields. Thus the various changes in graphite resulting from neutron bombardment which effect the structural integrity or nuclear safety of the reactor are of prime importance in reactor operation. The structural integrity of the moderator is primarily affected by graphite dimensional stability and by the resultant loss of mechanical strength from the reaction of the moderator with oxygen bearing gases, while stored energy plays an important role in nuclear safety. Thus radiation damage to graphite is involved in reactor safety, day to day operating efficiency, and overall reactor life.

It is the purpose of this paper to discuss these three areas of reactor operation as they are affected by graphite radiation damage and to present in a somewhat historical manner how the problems which evolved were met. For simplicity the discussion will be divided into three principal parts - distortion, stored energy, and oxidation with some general comments to summarize the philosophy of reactor operation with regard to the graphite problems.

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## DISCUSSION

### A. Distortion

In the early days of operation of the original Hanford reactors, it was soon apparent that the postulations of Wigner and Szillard were real and posed serious threats to continued long term operation. The graphite stack which was composed of a tightly packed criss cross matrix of graphite bars approximately  $4\text{-}\frac{3}{16}$ " x  $4\text{-}\frac{3}{16}$ " x 48" began to expand at a rapid rate. The stack deformation was such as to cause outward bowing of the far side biological shield, a lifting of the top shield, distortion of both vertical and horizontal control rod channels, and difficulty in charging fuel elements into the distorted process tubes. The general stack distortion followed the cosine neutron flux distribution within the reactor. The vertical distortion of the top centrally located process tube is illustrated by Curve A of Figure I. It is to be noted that despite a considerable distortion of the top and far side shields, little or no distortion of the charge and discharge faces was observed.

With the reactors literally bulging at the seams, extensive reactor measurements<sup>(1)</sup> were taken to learn more about the problem. One such measurement, when coupled with laboratory studies, lead to a fundamental change in stack design. This measurement consisted of measuring the relative expansion of tube blocks and filler blocks. It was shown that the tube blocks were sustaining a major portion of the expansion. The results of these measurements are shown graphically in Figure II.

From the large quantity of engineering data compiled from reactor measurements and from the comprehensive laboratory studies, it became apparent that the key to the distortion problem was the graphite operating temperature. Laboratory studies had shown that radiation damage could be thermally annealed and that radiation annealing was even more effective in reducing the physical expansion. Further laboratory studies had shown that when the sample exposure temperature was raised to 200-250°C,<sup>(2)(3)</sup> there was negligible graphite expansion and that even at the lower exposure temperatures samples expanded only perpendicular to the extrusion axis of the original bar.<sup>(4)(5)</sup> These two points seem to account for the relative expansion of tube blocks and filler blocks and for the observation that the charge and discharge faces had not been appreciably distorted.

Based on this information, the operating temperature was substantially increased by replacing the inert helium gas atmosphere with carbon dioxide.<sup>(6)</sup> The effect of this change in operation can be seen in Curves B, C, D of Figure I. Thus expansion was arrested and to a very large extent has been eliminated as a threat to continued reactor operation. Stack distortion is limited to small portions in the very top corners of the original reactors where the contracted central region coupled with the original fringe expansion results in a curvature problem.

Reactors which were subsequently constructed incorporated two design innovations. As noted previously, reactor measurements indicated that a substantial temperature gradient existed within the lattice unit. Heat transfer calculations substantiated these measurements and the temperature gradient is illustrated in Figure III. It was quite apparent from this work

that one way to live with graphite expansion was to isolate the tube block in a manner such that if it expanded the effects would be considerably reduced.<sup>(7)</sup> This isolation was termed undercutting and is shown graphically in Figure IV. Subsequently, the concept of coring<sup>(8)</sup> was evolved and was used to give a more uniform graphite temperature distribution throughout the entire graphite stack. This concept of coring is also shown in Figure IV. Thus by changes in stack design and by the use of carbon dioxide, graphite distortion in the new reactors is not an operational problem.

## B. Stored Energy

With the postulation by Szillard that neutron bombardment of graphite would result in the storage of potential energy and the early experiments which confirmed this postulation,<sup>(9)(10)</sup> the possibility of an uncontrolled release of stored energy caused considerable concern in the operation of the Hanford reactors. However, as more data became available, it became apparent that a stored energy release was improbable.<sup>(11)</sup> The pertinent data which were developed are summarized as follows:

1. At cold test hole exposure temperatures, total stored energy builds up as a function of exposure. This is shown in Curve A of Figure V.
2. The release spectrum of stored energy for cold test hole exposures changes with the extent of radiation damage. This is shown in Figure VI.
3. Total stored energy as well as the release spectrum of highly irradiated graphite can be annealed. This is shown as Curve B in Figure V and in Figure VII.
4. The buildup of stored energy is an inverse function of the exposure temperature. This is shown in Figure VIII.

It is apparent from these items that, as in the case of distortion, the temperature distribution within the graphite stack, as well as within a given lattice unit, plays a major part in the buildup of total stored energy as well as the release spectrum. From the lattice temperature gradient (cf Figure III) and utilizing the front to rear cosine distribution of temperature, the entire reactor stack can be zoned into four parts; namely, fringe filler blocks, fringe tube blocks, central filler blocks, and central tube blocks. By removing samples from the moderator at specific locations and determining the total stored energy and the release spectrum of these samples, the stored energy condition of the reactor stack can be evaluated. An evaluation<sup>(12)</sup> of the stored energy in Hanford reactors which utilized this zoning approach was first made some years ago. While the results must of necessity be somewhat generalized, it is appropriate to say that while a considerable quantity of stored energy exists in the Hanford reactors its presence does not constitute an operational or safety problem. No attempts to anneal the graphite stack by deliberately triggering the stored energy have been made at Hanford. As in the case of distortion, the control of stored energy has been through the control of graphite operating temperature.

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### C. Oxidation

While increasing the graphite temperature and the use of carbon dioxide gas atmosphere arrested the stack expansion, the potential chemical reaction of the moderator with carbon dioxide and other oxygen bearing gases posed another unknown threat to long term reactor life.<sup>(13)</sup> Little was known about the effects of reactor radiation on chemical reactions such as these. A considerable amount of work was then initiated both in the laboratory as well as in the reactor to scope this potential problem. The principal worry was that there might be sufficient gasification of the moderator to substantially reduce the structural strength of the stack and thus again limit the reactor life. Studies were initiated involving the effect of oxidation on mechanical strength, rates of reaction of graphite and  $O_2$ ,  $CO_2$ , and water vapor at various temperatures for irradiated and unirradiated samples, the effect of the presence of gamma radiation and reactor radiation on rates of oxidation, and the role of surface area in graphite oxidation. To describe these studies in detail would require several papers in itself. Let it suffice to summarize here the pertinent points which affect graphite oxidation as an operating problem.

An experiment was set up to determine the effect of oxidation on the mechanical strength of graphite.<sup>(14)</sup> The conditions chosen were determined largely from work done at Pennsylvania State University under Dr. P. L. Walker<sup>(15)</sup> and an analysis of reactor conditions. It was felt that if chemical reaction was taking place within the reactor, the rate of the reaction would be very slow and would therefore result in uniform oxidation rather than surface oxidation as shown by Walker. Thus oxidation of the test samples was to be accomplished in a manner to produce uniform oxidation. The test sample, a right circular cylinder 1" x 2" with a 3/4" axial hole, was oxidized in a dry, deoxygenated stream of carbon dioxide flowing at a rate of 500 cc/min. and at a temperature of 950°C. The rate of oxidation was varied from 0.5%/day to 6%/day. Figure IX summarizes the results of this study for unirradiated samples. Figure X shows the results for one set of irradiated samples. While it was recognized that the experimental procedure was not as elegant as might be desired, the results did provide excellent comparative data. The general conclusions reached were:

1. For non-irradiated graphite there is a rapid decrease of compressive strength during initial oxidation. The rate of decrease in compressive strength gradually lessens as the sample is further oxidized.
2. For non-irradiated graphite, the compressive strength is decreased by a factor of 2 for 10% oxidation and a factor of 6 for 25% oxidation.
3. Irradiated samples exhibited greater compressive strength than non-irradiated samples. However, the same relationship between compressive strength and density was observed.

The reaction rates of graphite with oxygen<sup>(16)</sup> and carbon dioxide<sup>(17)</sup> were determined over a range of temperatures using both irradiated and non-irradiated samples. The experimental method consisted of placing the sample in an electric furnace, passing purified gas over the sample and monitoring the effluent gas over a finite period of time. A final check was made at the end of each run by measuring the weight loss of the sample and comparing this with the calculated accumulated weight loss from the gas monitoring. The agreement was excellent. In case of oxygen, carbon dioxide and carbon monoxide were monitored by suitable absorbents. In the case of carbon dioxide, carbon monoxide was monitored by passing effluent gas over iodine pentoxide and collecting liberated iodine followed by back titrating with sodium thiosulfate. The gas flow rate was maintained at 100 cc/min. In each case the rates of reaction for various temperatures (cf Fig. XII & Fig. XIII) were corrected to 0% oxidation. The results of a single run with oxygen showing carbon dioxide and carbon monoxide distribution are shown in Figure XI. The rates of reaction for various temperatures for oxygen are shown in Figure XII and for carbon dioxide in Figure XIII.

The activation energy for the reaction of oxygen and irradiated and non-irradiated graphite were calculated and found to be 34.6 kcal/mole and 38.6 kcal/mole respectively. The activation energies for the reaction of irradiated and non-irradiated graphite samples with carbon dioxide were found to be 51.5 kcal/mole and 61.5 kcal/mole respectively.

In general, the reaction rates increased as the oxidation proceeded. During the initial part of the oxidation runs, considerable abnormalities were encountered in the carbon dioxide studies. However, the subsequent increase in rate was observed as the oxidation proceeded and it was felt that this was the result of a preferential oxidation of the binder material in the graphite and the subsequent increase in surface area. Studies of surface area<sup>(18)(19)</sup> by the B.E.T. technique using nitrogen, tended to confirm these postulations. It was found that the surface area of the graphite samples increased markedly upon slight oxidation. Further, a comparison of the change of surface area of solid graphite samples and coke flour which had received the same graphitizing heat treatment as the solid samples showed that the surface area of the coke flour was relatively unchanged. This tends to confirm the preferential attack concept. These results are shown in Figure XIV.

A number of in-reactor studies were made to determine the effect of reactor radiation on the chemical reactions of graphite and oxidizing gases. The experimenter at Hanford is cursed or blessed, depending upon your point of view, with the fact that the irradiation facilities are in production reactors. The facilities for studying gas-graphite reactions were particularly compromised by the goal of operations; namely to produce plutonium. Nevertheless, most of the engineering data on graphite oxidation was so obtained. Three pertinent studies were made. One study<sup>(20)</sup> involved the exposure of static systems, contained in quartz ampules, in a water cooled facility. The systems studied were graphite-oxygen, graphite-carbon dioxide, graphite-water vapor, and pure carbon dioxide. The complete study is contained in an unclassified report - HW-40142.<sup>(21)</sup> Briefly, the results indicated 1) a very slow reaction of CO<sub>2</sub> and graphite with the formation of carbon monoxide which decomposes to form a suboxide of carbon which in turn polymerizes, 2) a reaction of oxygen and graphite, which appears to be

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about three times as fast as the  $\text{CO}_2$  reaction, to form carbon dioxide, 3) a reaction of water vapor and graphite to form carbon dioxide and hydrogen; the extent of the reaction is dependent upon the quantity of water vapor, and 4) pure carbon dioxide decomposes very rapidly to an extent of about one percent to form carbon monoxide.

A similar study was made in that static systems were exposed to the intense gamma radiation of the Materials Test Reactor spent fuel elements at about room temperature. The systems studied were graphite carbon dioxide, graphite oxygen, and pure carbon monoxide. The complete study is contained in a declassified report - HW-31929 REV. (22) The results are briefly summarized: 1) the reaction of oxygen and graphite to form carbon dioxide went essentially to completion; 2) the reaction of carbon dioxide and graphite was very small, and appears to be limited to surface absorption; 3) carbon monoxide decomposes to form carbon dioxide and a solid polymer of carbon suboxide with an empirical formula  $\text{C}_4\text{O}_3$ . A notable difference in the results of these studies is that in the graphite-carbon dioxide system, reactor radiation induces a substantial reaction with the formation of carbon monoxide and subsequent suboxide while the gamma radiation does not.

The third study was the exposure of graphite samples in a reactor to flowing gas with the temperature maintained in the range  $370\text{-}425^\circ\text{C}$ . (23)(24)(25) The samples contained in aluminum tubes were supplied with independent gas systems on a single pass basis. Typical results from these runs are shown in the following Table.

TABLE I  
IN-REACTOR OXIDATION OF GRAPHITE SAMPLES  
AT TEMPERATURE RANGE  $370\text{-}425^\circ\text{C}$

<u>Gas</u>	<u>Reaction Rate in %/1000 Days</u>	
	<u>Unirradiated</u>	<u>Previously Irradiated</u>
$\text{CO}_2$	-1.02%	-1.78%
$\text{O}_2$	-27.9	-58.4
$\text{H}_2\text{O}$ vapor in $\text{N}_2$	-2.1	-3.1

It is to be noted that these rates are considerably higher than those obtained in ex-reactor runs at these temperatures.

From these experiments and from measurements taken on the reactors, it was apparent that graphite oxidation and the resultant loss of mechanical strength posed a real problem to reactor operation. Again the key to the problem was reactor temperature control. Further it was evident that only in-reactor experiments or reactor operation on a test controlled basis would yield appropriate data for establishing proper limits for reactor operation. One approach to the problem of oxidation was extensive monitoring of the actual oxidation occurring. (26) This was done by placing samples in bare process channels and observing the sample weight

loss with reactor operation. While this was an "after the fact" approach, it did and still is yielding considerable practical information. The full reactor experimental approach was also used.<sup>(27)(28)</sup> This consists of operating a reactor under conditions which are somewhat in advance of the process limits in order to obtain operating information which will permit a further definition or refinement of process limits. The net effect of such an approach is to obtain more information on graphite oxidation than would be possible by running tests in single or multiple process channels so that reactor moderator temperature may be optimized for production and reactor life.

#### D. Graphite Temperature Monitoring

As a corollary to the above discussions of distortion, stored energy, and oxidation, it is essential to reactor operation that the graphite temperature be adequately monitored within the entire moderator stack.<sup>(29)</sup> Since the original reactors were equipped with only six thermocouples located within the active zone, it was necessary to devise an extensive monitoring system. This was accomplished by placing a specially constructed graphite thermocouple stringer in an empty process channel. The stringer contained eleven thermocouples appropriately positioned along the channel. The number of stringers required vary with each reactor; in general seven stringers are used in the original Hanford reactors. The stringers are located in a ring in the process channel pattern and each stringer is approximately ten lattice units from the central channel. An individual thermocouple mounting and the general location of the stringers are shown in Figure XV.

When the newer reactors were constructed, the importance of moderator temperature was recognized and adequate instrumentation was incorporated in the stack structure. However, through one cause or another, these thermocouples are failing and again the thermocouple stringer approach is to be used to maintain adequate monitoring.

#### E. General Monitoring

As has been indicated in the above discussion, reactor monitoring plays an important role in Hanford reactor operation. Distortion of the graphite stack is routinely monitored by measuring the elevation of the top process tubes relative to their respective front face locations. The measurement is made by a mercury manometer device which utilizes a leveling technique. The graphite oxidation is routinely monitored by placing weighed samples in an empty process channel (and thereby ambient reactor temperature and gas atmosphere) and observing the sample weight loss per unit time of reactor operation. Stored energy content is monitored by the periodic removal of solid core samples from various locations in the reactor. The measurement of other physical properties of these core samples acts as a secondary check on distortion and oxidation. Core samples have been found to yield much more meaningful data than scrapings or powder because property gradients can be measured.

CONCLUSIONS

From this discussion, several conclusions can be made.

- 1) Stack distortion, which at one time seemed to threaten the continued operation of Hanford reactors, is a minor problem at the original reactors and does not appear to be a problem at the newer reactors.
- 2) Stored energy content in the Hanford reactors is fairly large but offers no problem to operation or nuclear safety. A substantial release of stored energy could only be accomplished in the event of a complete coolant failure.
- 3) Graphite oxidation is controlled within established limits by adequate monitoring and by an extensive operating knowledge of the moderator temperature.
- 4) Graphite temperature instrumentation and monitoring programs are essential and provide first hand data on the reactor moderator integrity.

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- Figure XV Thermocouple Mounting and Stringer Location.

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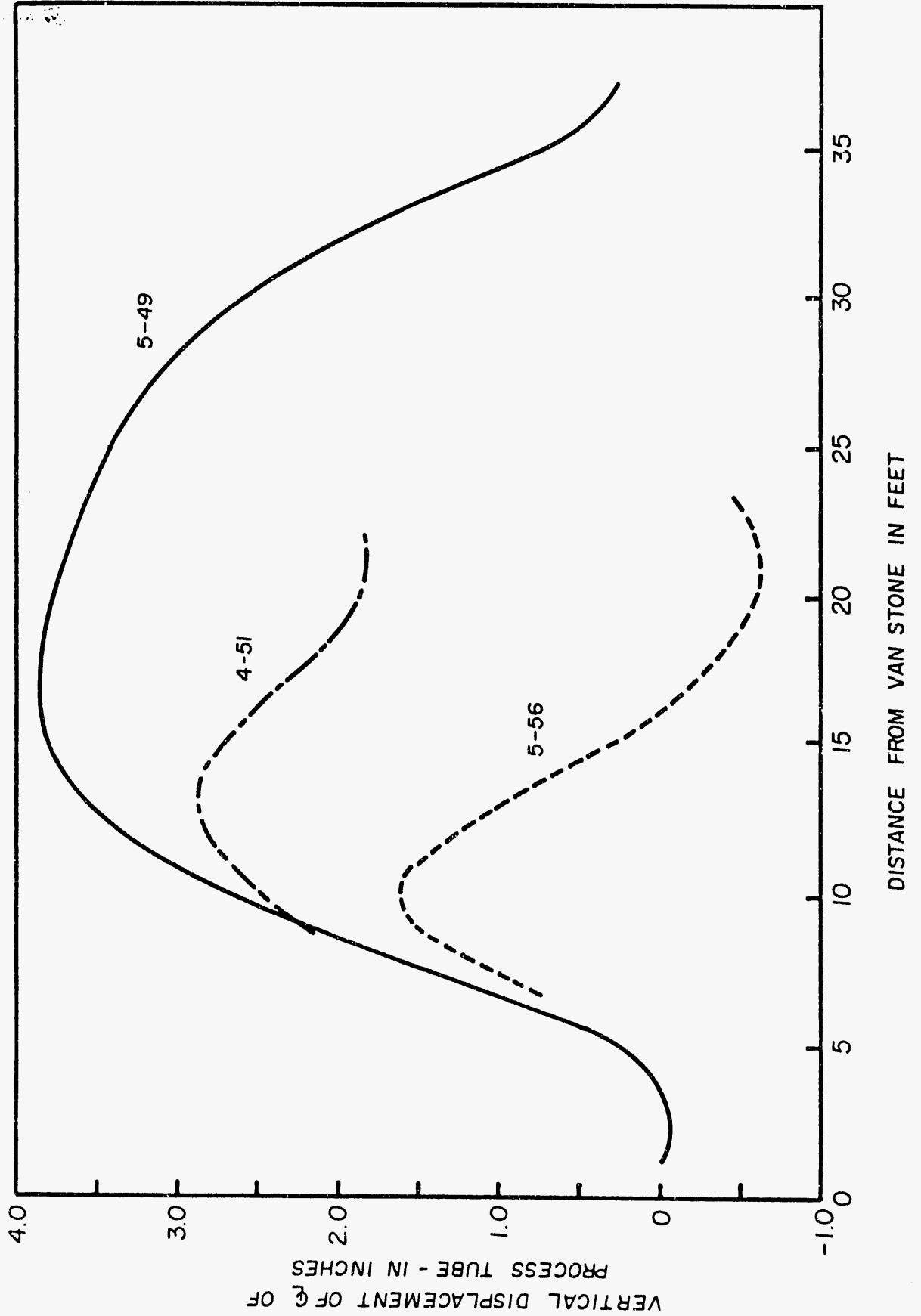
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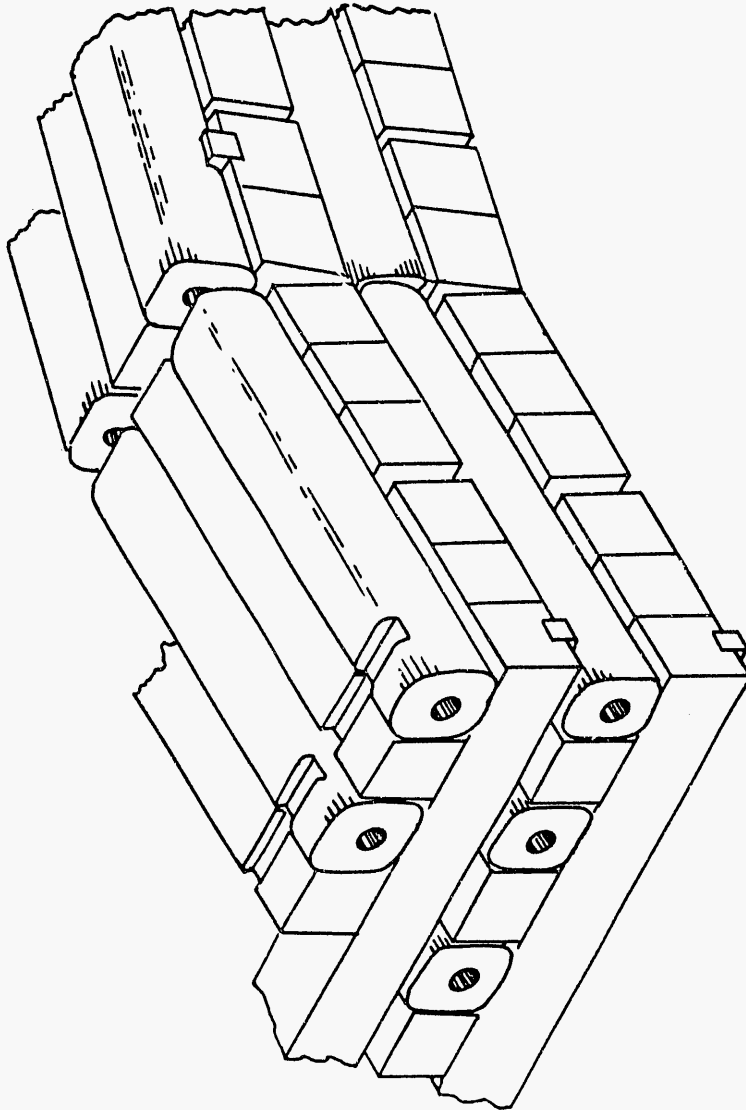
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FIGURE I - TYPICAL EXPANSION OF ORIGINAL REACTOR



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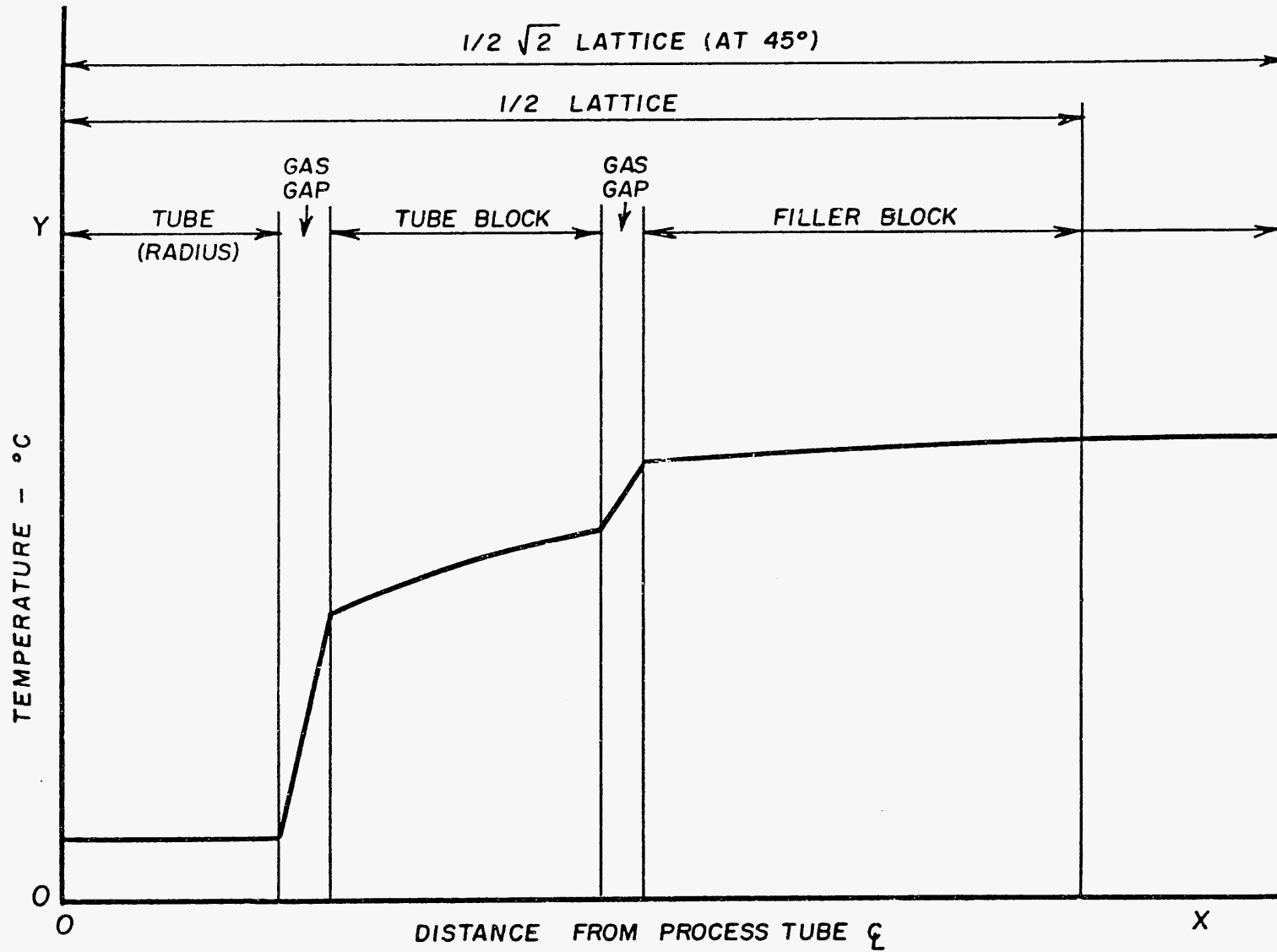
FIGURE II - LOCALIZED TUBE  
BLOCK EXPANSION



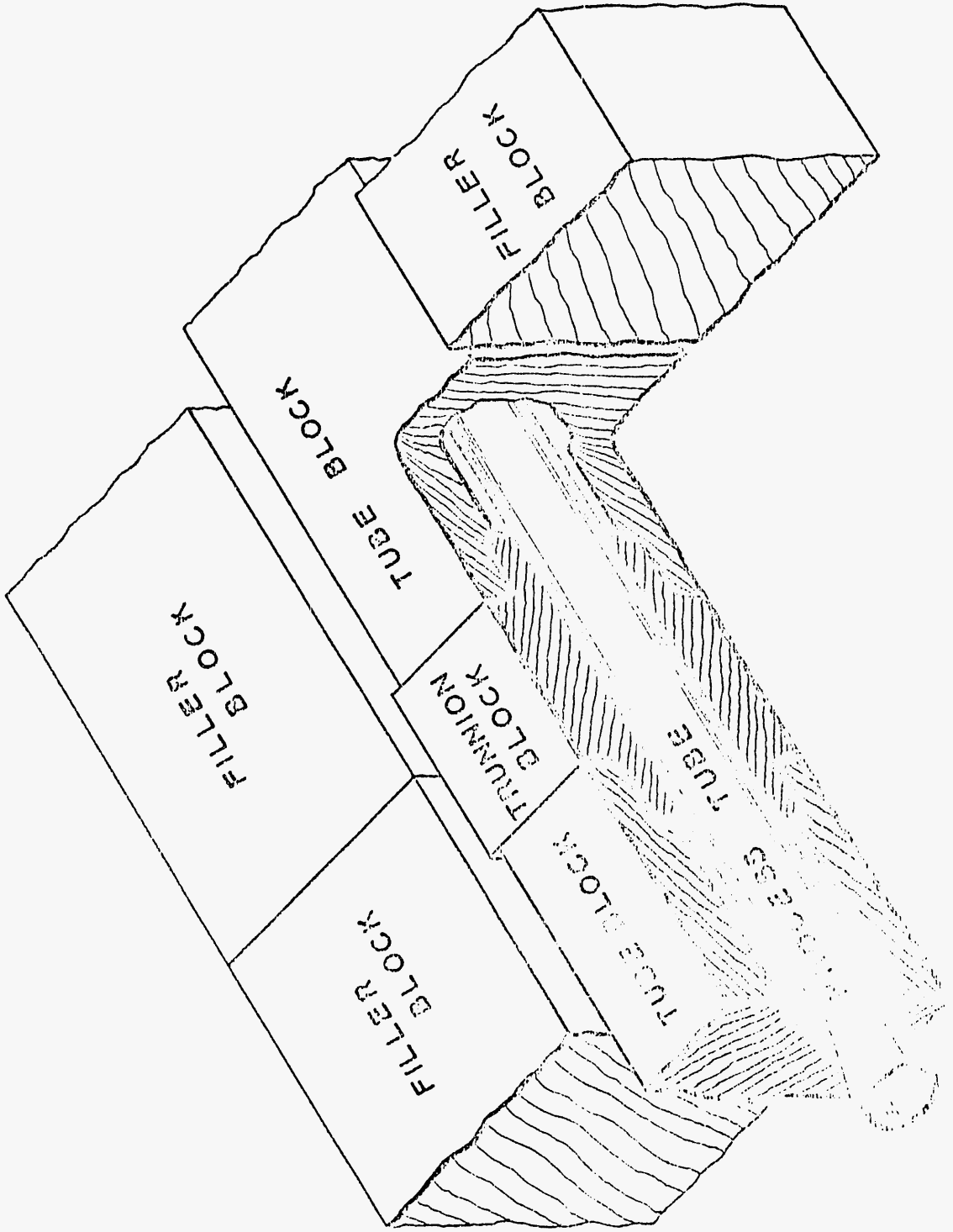
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# FIGURE III - GRAPHITE LATTICE UNIT TEMPERATURE GRADIENT

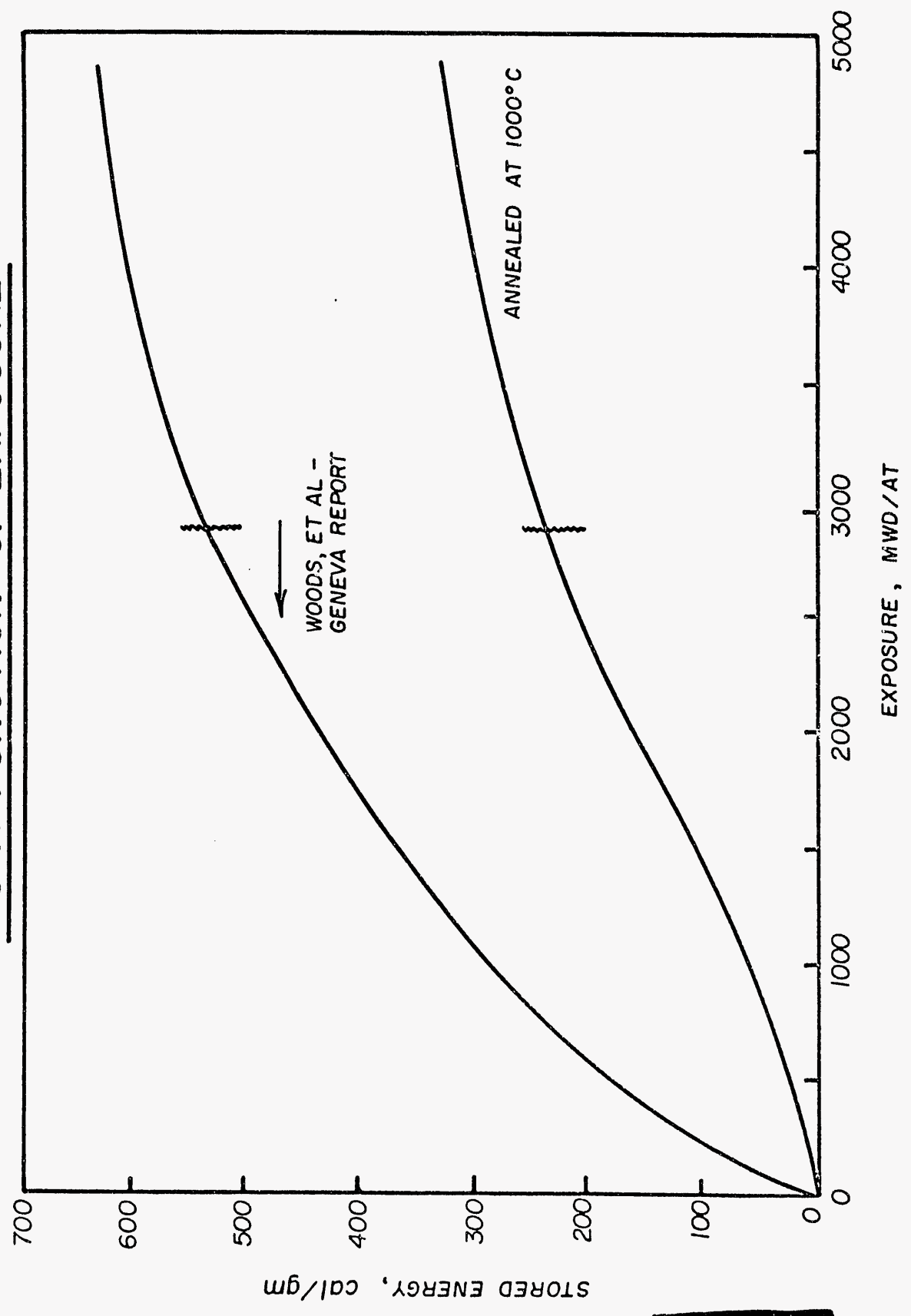


SECTION 1: TUNNION BLOCK AND  
GOING PATTERN



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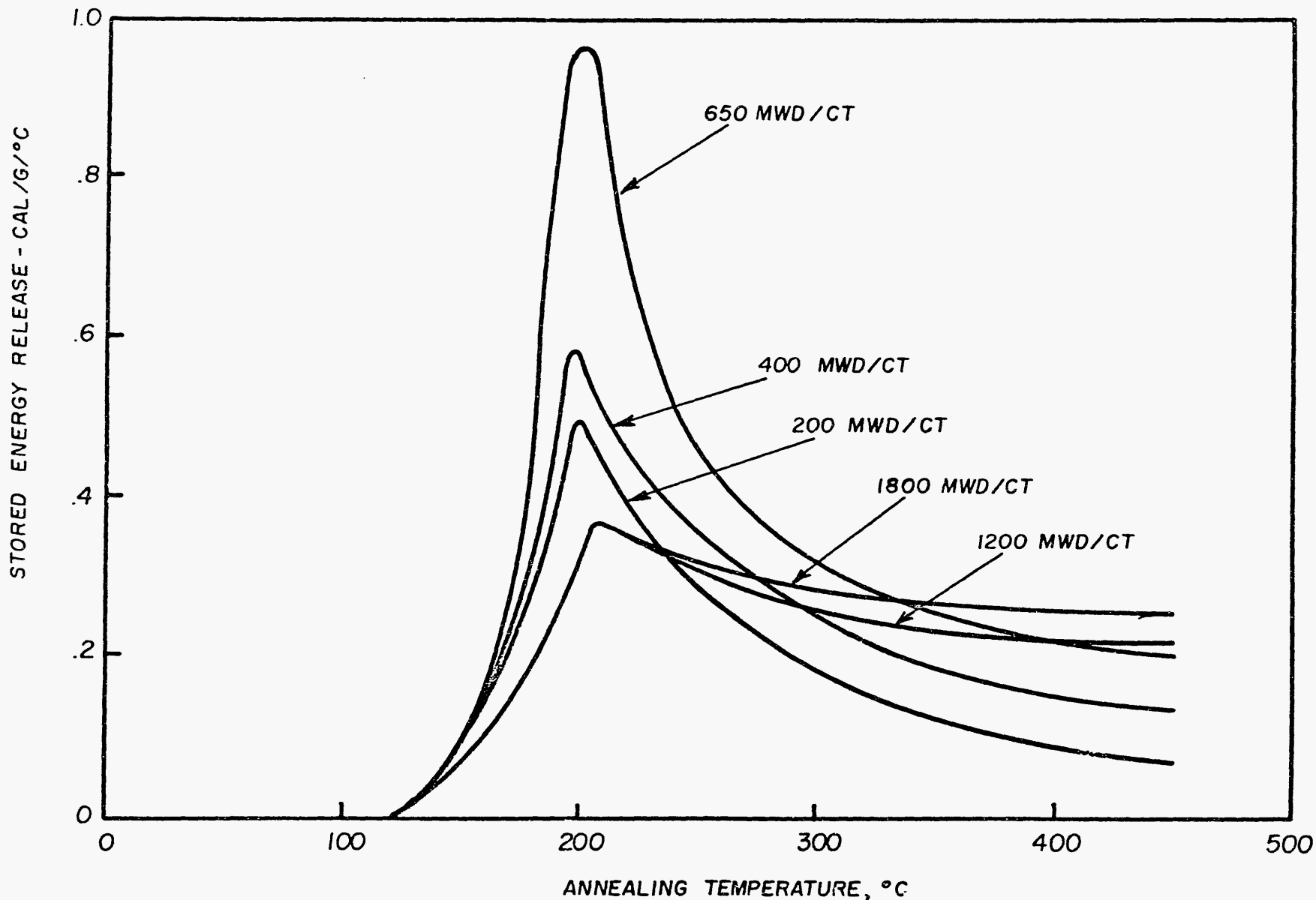
FIGURE VI - TOTAL STORED ENERGY BUILDUP  
AS A FUNCTION OF EXPOSURE



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FIGURE VI - RELEASE SPECTRUM OF STORED ENERGY FOR COLD TEST HOLE EXPOSURE



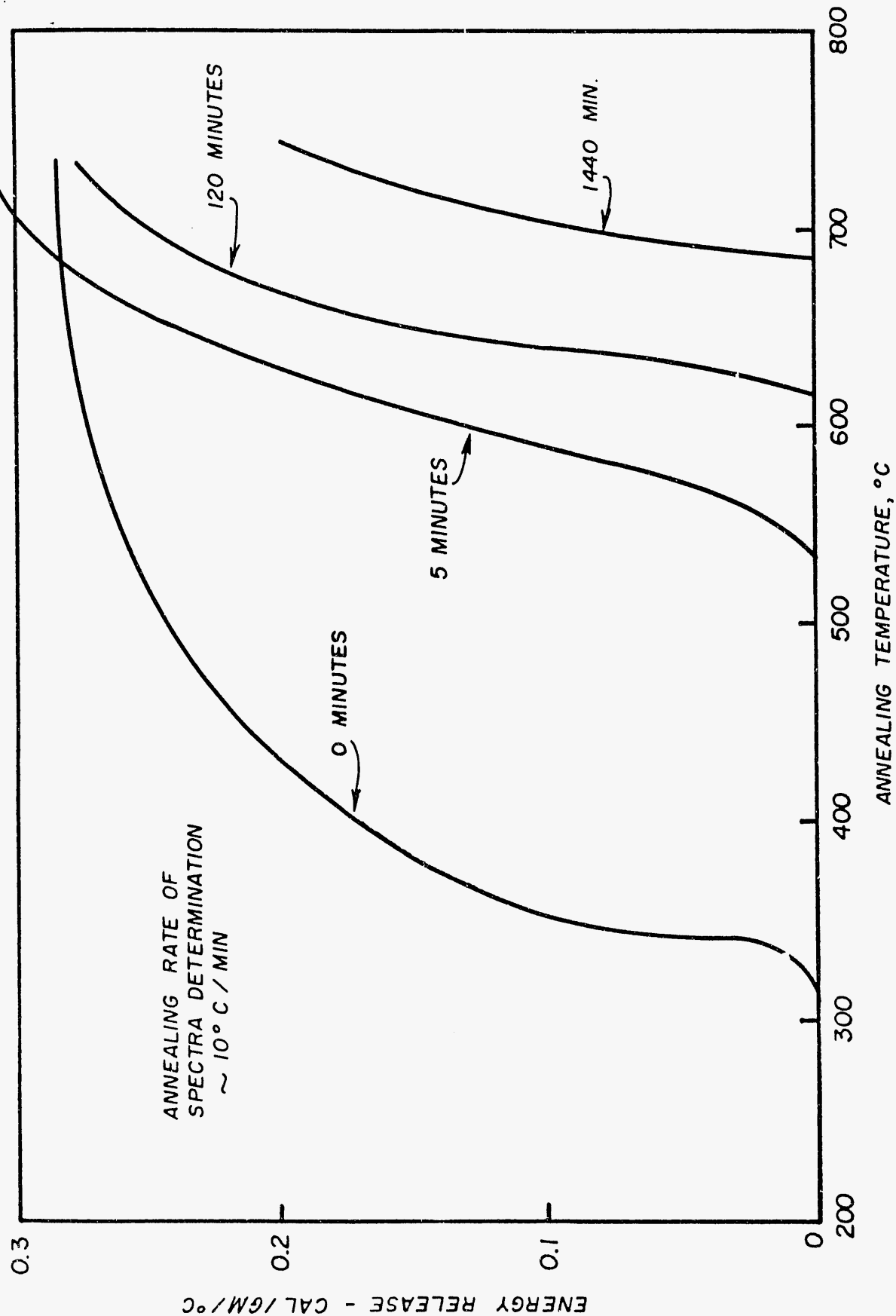
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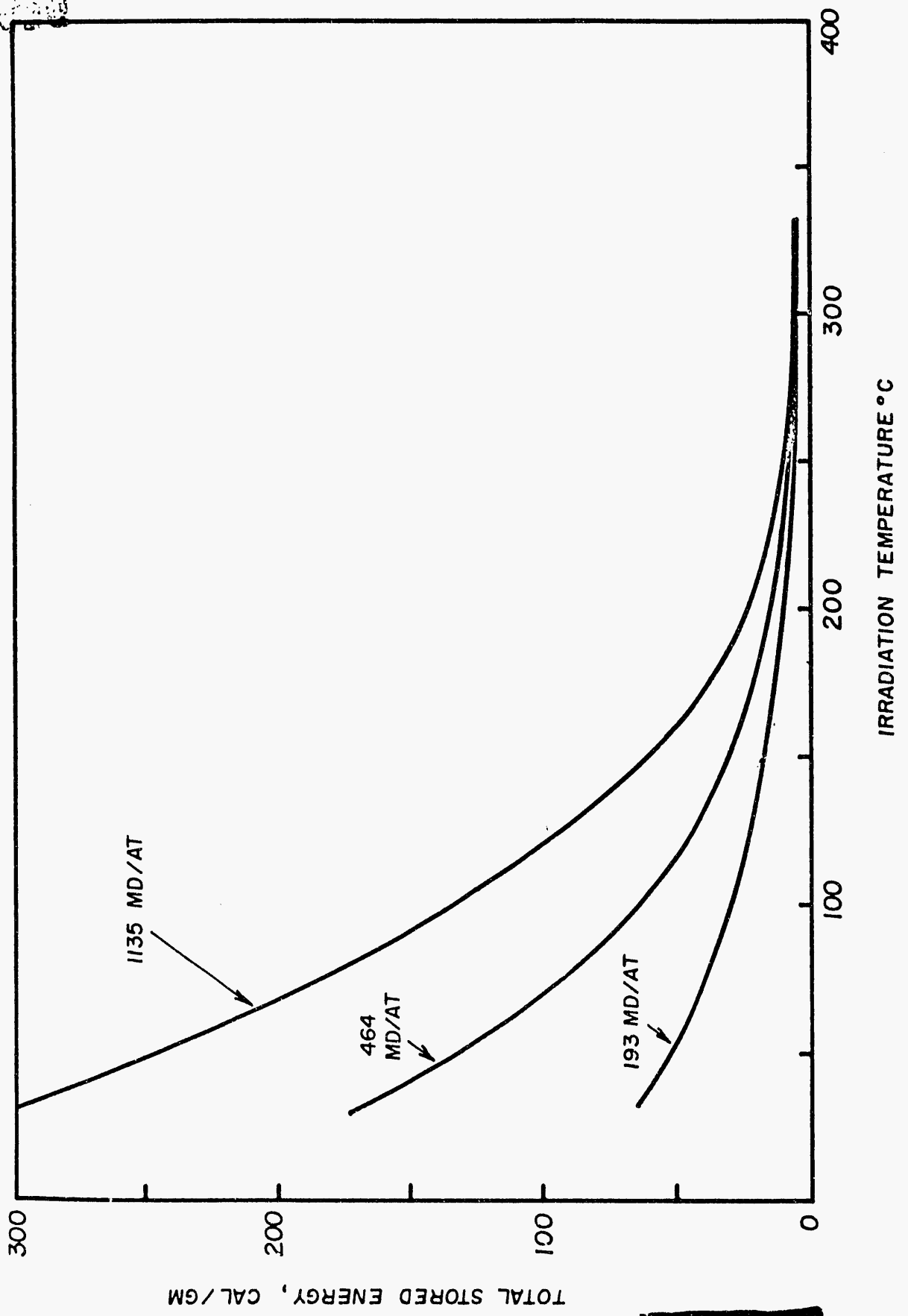
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FIGURE VII - RELEASE SPECTRUM OF IRRADIATED GRAPHITE UPON ANNEALING



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FIGURE VIII - TOTAL STORED ENERGY BUILDUP  
AS A FUNCTION OF EXPOSURE TEMPERATURE



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FIGURE IX - UNIRRADIATED GRAPHITE COMPRESSIVE STRENGTH AS A FUNCTION OF UNIFORM OXIDATION

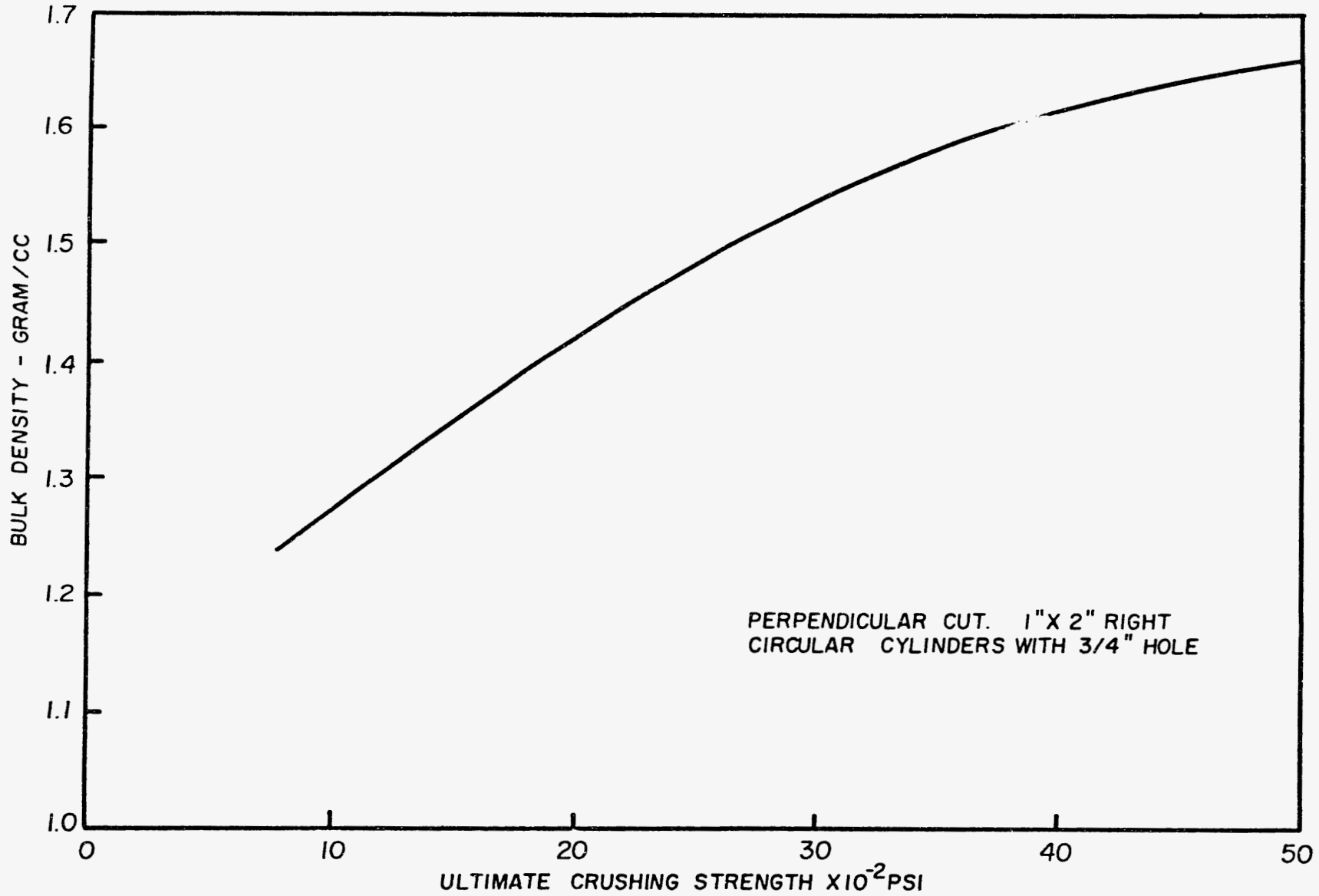
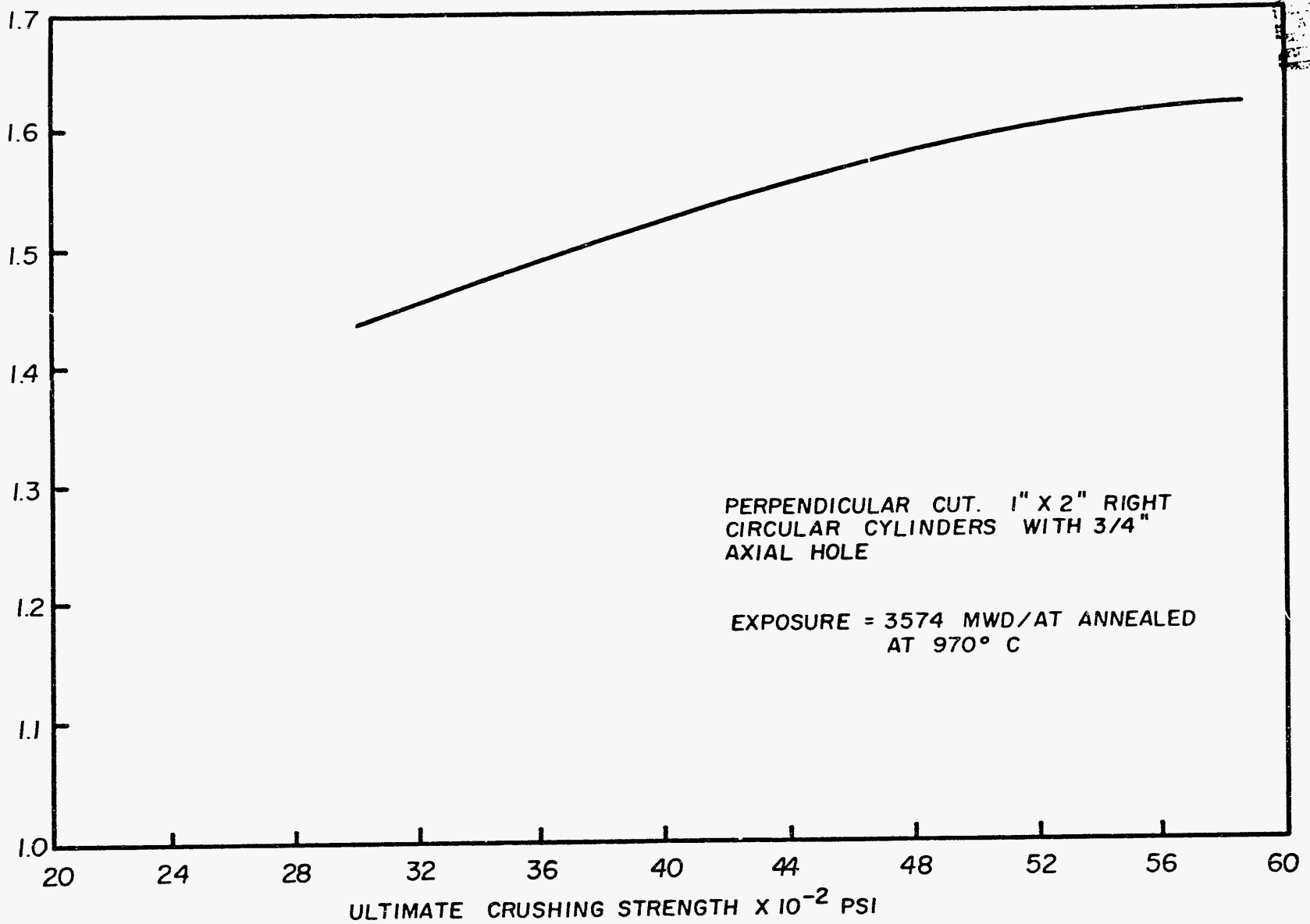


FIGURE X - IRRADIATED GRAPHITE COMPRESSIVE  
STRENGTH VS UNIFORM OXIDATION

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BULK DENSITY - GRAM / CC



PERPENDICULAR CUT. 1" X 2" RIGHT  
CIRCULAR CYLINDERS WITH 3/4"  
AXIAL HOLE

EXPOSURE = 3574 MWD/AT ANNEALED  
AT 970° C



**FIGURE XI - REACTION OF GRAPHITE AND OXYGEN  
AS A FUNCTION OF PER CENT OXIDATION**

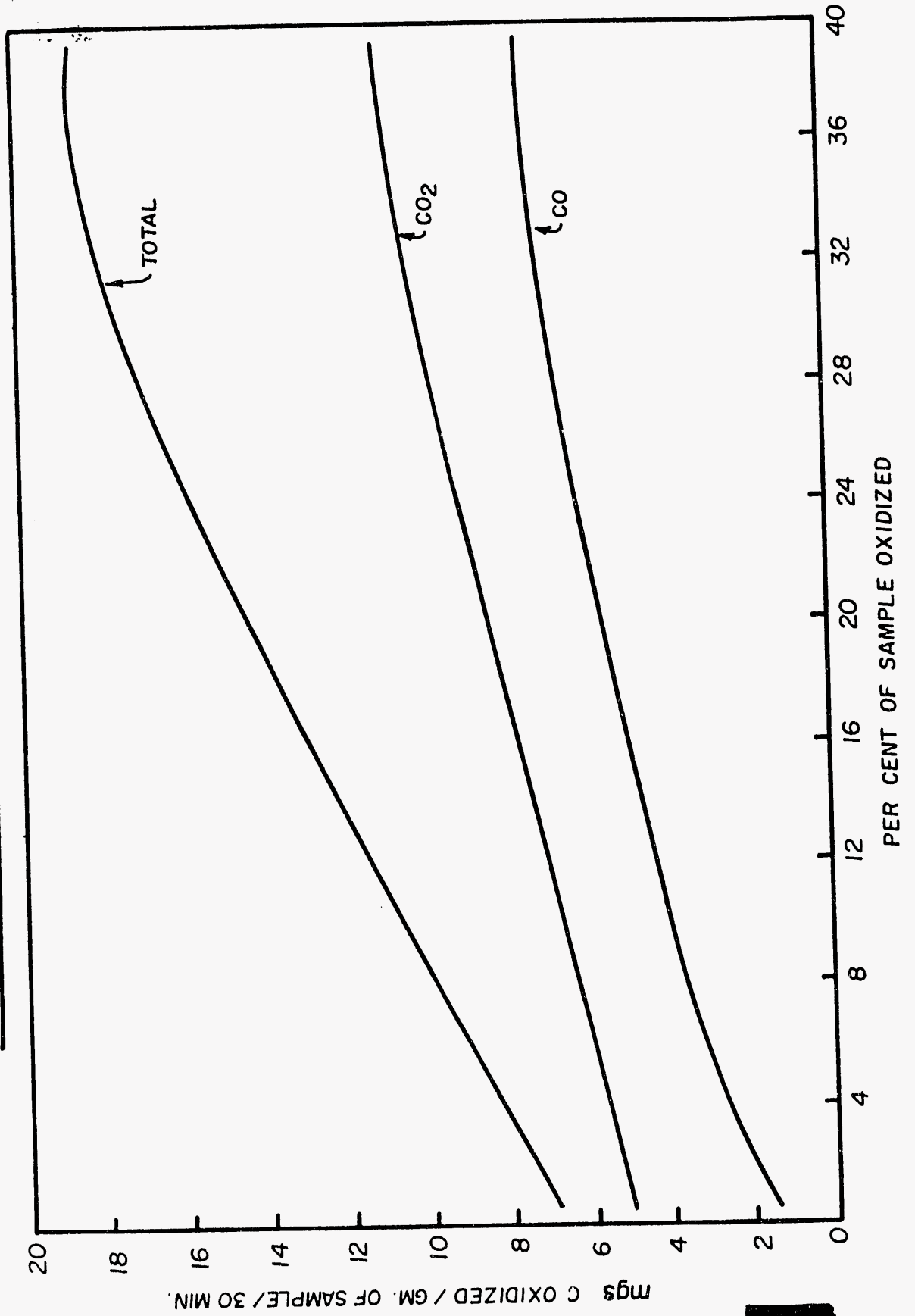


FIGURE XII - REACTION RATE OF GRAPHITE AND OXYGEN AT VARIOUS TEMPERATURES

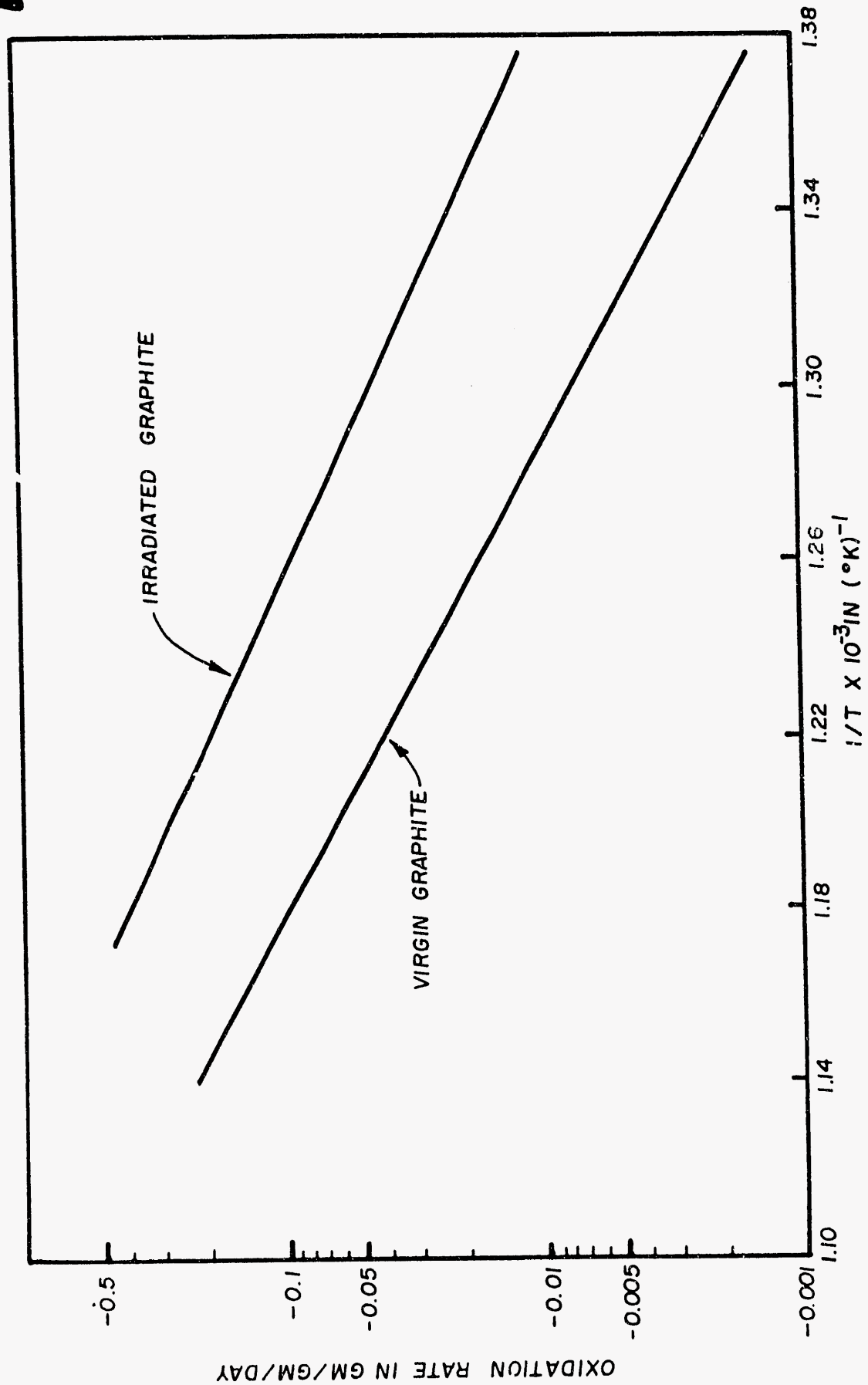


FIGURE XIII - REACTION RATE OF GRAPHITE AND  
CARBON DIOXIDE AT VARIOUS TEMPERATURES

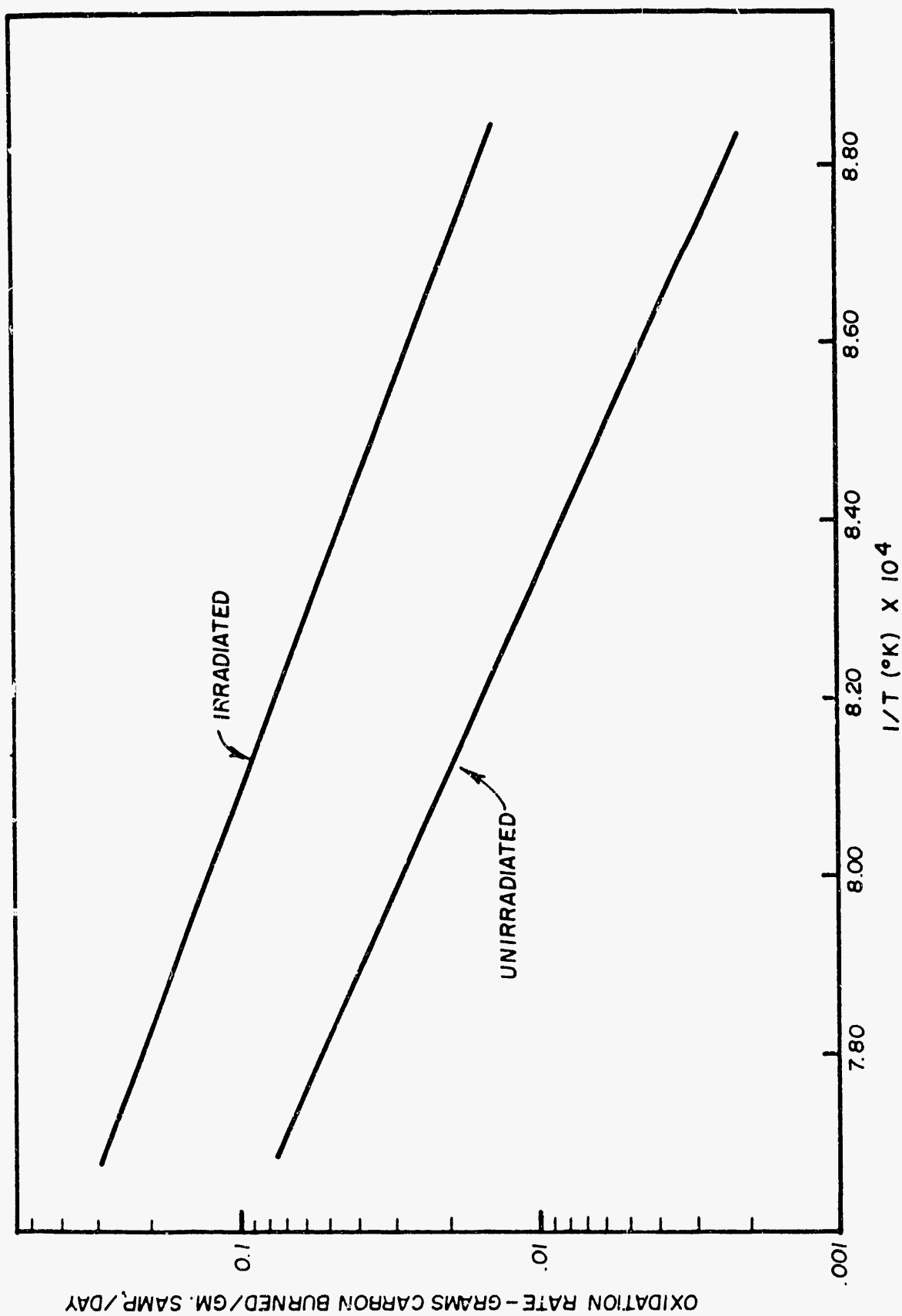


FIGURE XIV - THE CHANGE OF SURFACE AREA OF GRAPHITE WITH OXIDATION

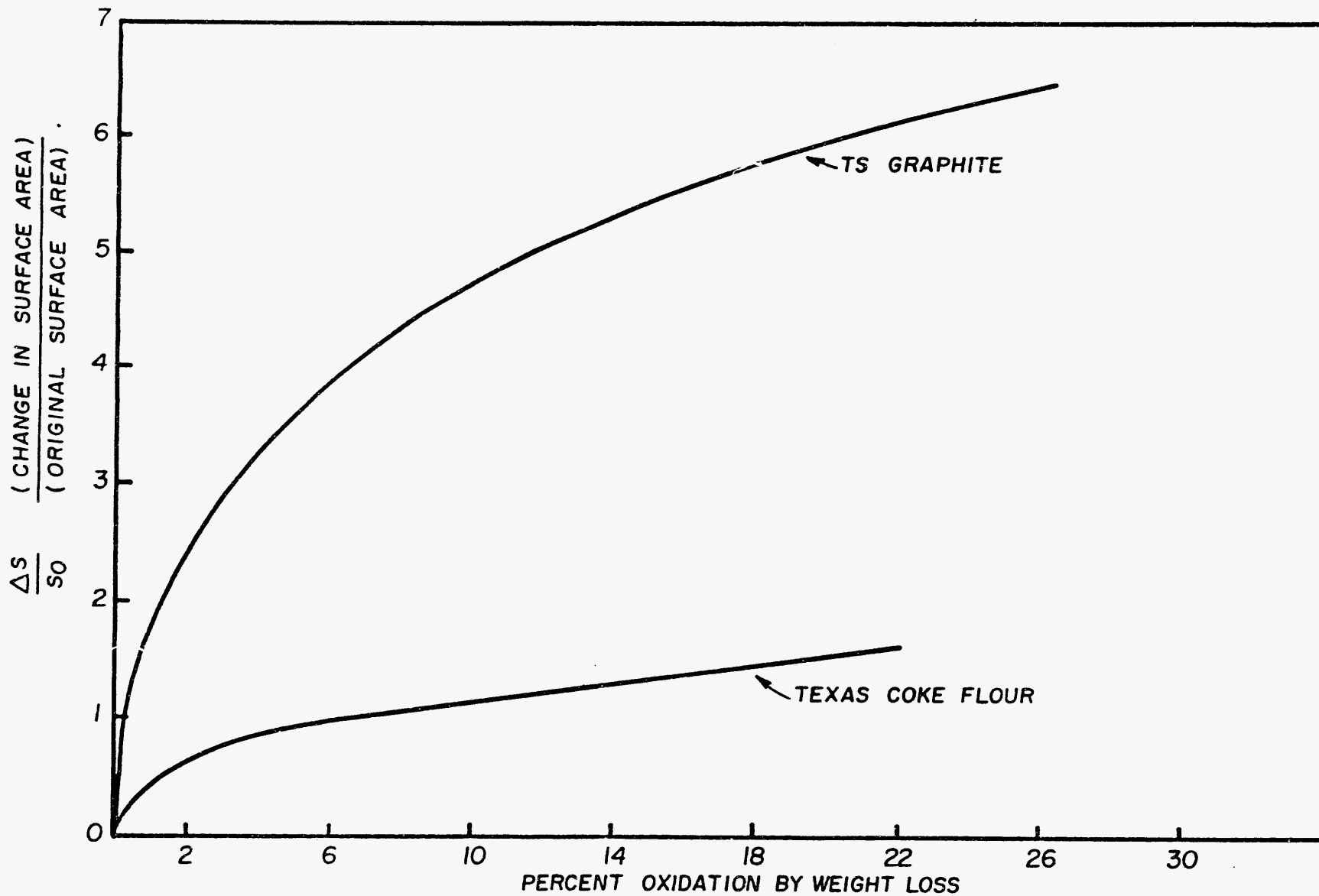
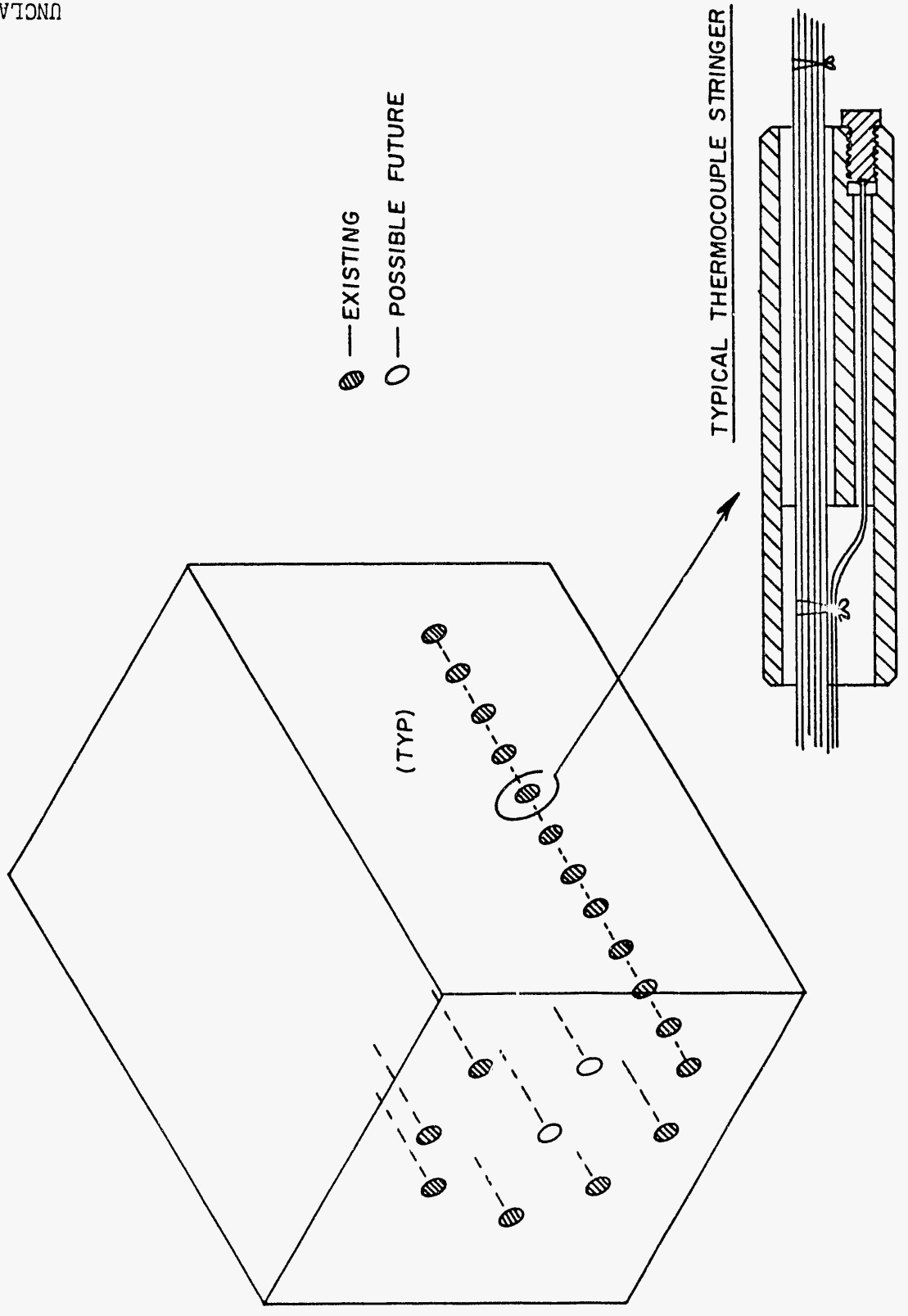


FIGURE XV - THERMOCOUPLE MOUNTING  
AND STRINGER LOCATION



UNCLASSIFIED

**END**

**DATE  
FILMED**

7 / 8 / 93

