NATIONAL BUREAU OF STANDARDS REPORT

2244

AN APPARATUS FOR THE STUDY OF ADIABATIC
SELF-HEATING OF SOLIDS AND LIQUIDS

by

A. F. Robertson
and
W. Raskin

NBS

U. S. DEPARTMENT OF COMMERCE
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- Office of Basic Instrumentation
- Office of Weights and Measures.

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NBS PROJECT **NBS REPORT**

1002-10-4720 February 4, 1953 2244

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1. INTRODUCTION

An investigation of the thermal decomposition of materials has led to the study of self-heating of specimens under conditions which resemble an adiabatic process and result in only a small heat exchange between the specimen and the surroundings.

Previous study in Sweden[1] and a study undertaken at the National Bureau of Standards[2], carried out under constant ambient temperature conditions, involved a study of self-heating and self-ignition in wood fiberboards. In this process, the specimen was supported in an oven maintained at constant temperature. The temperature distribution through the specimen was monitored by means of imbedded thermocouples. Using octagonal specimens, the heights and widths of which were equal, it was found that the center of the specimen self-heated to temperatures higher than at any other point. In cases where the ambient temperature was sufficiently high, the self-heating continued to ignition of the specimen, even though the surface of the specimen was only slightly warmer than the ambient air at the time ignition occurred. This resulted from the fact that, as the specimen self-heated, heat was lost to the surroundings; however, since the center of the specimen was insulated to a greater extent, due to the greater amount of material surrounding it, the peak temperatures developed at this point. Such a system permits study of self-heating behavior of specimens of a given size and shape, at a given ambient temperature. A large number of tests is required to survey the field of specimen sizes, shapes, and ambient temperatures.

To overcome these limitations of the constant temperature method a new furnace has been devised. It was constructed for the purpose of obtaining data on the self-heating characteristics of materials in the temperature range from 30 to 500°C, in such a way that their size and shape would not appreciably affect the results obtained. This is done by first bringing the specimen and furnace air to equilibrium at some preselected temperature, and then allowing the specimen to heat throughout its volume in such a way that the temperature gradient within it is nearly zero. This is accomplished by a control device which keeps the furnace temperature very closely equal, at all times, to the temperature of the center of the specimen. In this manner, the entire volume of the specimen heats at the same rate, providing the process is not controlled by the rate of gaseous diffusion in the material, producing a time-temperature curve that may be characteristic of the material being tested.

Somewhat similar types of apparatus [3,4] have been constructed for the study of spontaneous heating of coal. In both cases, however, the furnaces had a comparatively large thermal inertia, and as a result could not follow rapid changes of specimen temperature.

2. DESCRIPTION OF APPARATUS

The furnace and its related control equipment are shown in figures 1, 2, and 3. The apparatus consists of a furnace and its components, within the chamber of which the specimen is supported on a wire attached to an electric strain gage dynamometer arranged to record continuously the weight of the sample. The associated equipment includes a pyrometer controller, a twelve-point pyrometer recorder and controller, a servo-amplifier and a servo-motor driving a variable voltage autotransformer which supplies power to the furnace heating coil, a galvanometer type relay, and two latching relays for automatic control of the operation of the furnace. A cathode ray oscillograph is useful for monitoring controller operation. A block diagram of the system is shown in figure 4.

3. METHOD OF OPERATION

In the performance of a typical test, the specimen is suspended in the furnace by means of a wire secured to a strain gage dynamometer. Thermocouples are mounted at the center and near the surface of the specimen, as well as in the air within the furnace. Air is then supplied to the furnace at any desired rate through the tube provided. During the initial warm-up period, a constant selected furnace temperature is maintained by the pyrometer controller which provides an "on-off" heating cycle. When the interior of the specimen reaches, and due to self-heating slightly exceeds, the air temperature within the furnace, a galvanometer relay is actuated and by means of a latching relay automatically disconnects the thermostatic controller. The latter relay also initiates operation of a servo-controller which supplies heat to the furnace as needed to maintain the smallest feasible temperature difference between the interior of the sample and that of the gases within the furnace. The sensing element for this controller is a differential thermocouple responsive to the temperature difference between the center of the specimen, and that of the surrounding furnace gases. Continuous records are provided by means of a twelve-point recorder of the furnace atmosphere temperature, and two or more temperatures within the specimen, as well as the specimen weight. A limit switch on the pyrometer recorder serves to disconnect power to the furnace and its related equipment when the furnace and/or specimen temperature exceeds a selected value. A circuit diagram of the electrical connections of the various parts of the instrument is shown in figure 5.

4. FURNACE CONSTRUCTION DETAILS

The furnace itself has been designed to have small heat capacity and losses and thus permits rapid changes in its temperature with moderate power input. Figures 2 and 3 show construction details of the furnace. The major portion of the furnace chamber is formed by a one-gallon Dewar flask. A plug of diatomaceous silica three inches thick forms the top closure. This plug, together with the interior furnace structure and blower drive, are mounted on an asbestos-cement sheet, held by an external supporting device. The interior of the furnace is provided with a stainless steel baffle which directs air circulation around the specimen, shields the specimen from radiant heating, and supports mica strips carrying the nichrome heater winding. This winding is designed to dissipate a maximum of 700 watts at 135 volts. The blower rotor is a standard model 3 in. in diameter and having a face two in. wide, and is driven by an external electric motor. The blower rotor is mounted on a hollow shaft through which the wire which supports the specimen passes with only slight rubbing friction. The shaft passes up through the refractory plug and is supported by sealed ball bearings. Power for driving the blower is furnished from a d-c motor through a rubber belt.

The parts of the temperature controlling and recording system and related equipment are of standard commercial construction as listed in figure 5. One exception to this is the servo-motor which has been modified to reduce its speed from the nominal 27 r.p.m. to 1/4 r.p.m. This comparatively slow speed has been used to slow the response rate of the system but nevertheless allows the furnace to follow closely the temperatures developed by the self-heating of the materials studied.

5. FURNACE OPERATION

This furnace has been in operation for more than a year and has provided reliable performance with need for only minor repair. The typical set of test data shown in figure 6 was obtained during a test of a laminated wood fiberboard specimen 3 in. in diameter and 2-5/8 in. long. The starting temperature for this test was set at a value considerably above the lowest known temperature at which self-heating would occur in order to shorten the period required for the completion of the reaction. During this test, air was supplied to the interior of the furnace at the rate of 200 ml per min; this was equivalent to 0.06 air change per minute. It is evident from figure 6 that the furnace temperature followed the specimen temperature very closely. The maximum temperature difference between the center of the specimen and the furnace atmosphere after automatic operation commenced appears from the record to have been less than 1°C. The actual temperature weight relationships shown on this curve are the result of complex processes and a discussion of the relationships is beyond the scope of the present paper. An example of this is the final temperature rise at constant weight seen near the end of the test.

Another method for evaluating the performance of this type of furnace is to conduct a test on a specimen formed of inert material. If there is actually no heat exchange between furnace gases and the specimen, the specimen temperature should remain constant and the furnace atmosphere should remain constant at very nearly the same temperature. Such a test was performed on a specimen of fire brick with the results shown in table 1. These results indicated that there was a small heat flow between the specimen and the surrounding air which is considered not serious for the studies now under way with this apparatus.

6. CONCLUSIONS

1. An apparatus has been constructed for the study of the self-heating of combustible materials under conditions such that the heat exchange to or from the exterior of the specimen is kept at a low value.

2. Tests with this device for study of the self-heating of specimens of wood fiberboard have demonstrated that the apparatus performed in the manner desired.

3. Tests with inert material indicate that the stability of the system in the temperature range from 100 to 300°C is such that the rate of temperature change of the specimen can be kept at or below 2°C per hour.

Table 1 - Temperature Drift of the System when Operated with an Inert Specimen

Furnace and Specimen Temperature	Rate of Temperature Change	Duration of Test
°C	°C/hr	hr
100	+1.0	3
200	-0.5	2
300	-2.0	2

References

1. Bjorn Holmgren, Self-Ignition of Porous Wood Fiberboard, Svensk Papporstidning, V.51, No.10, May 31, 1948.

2. Nolan D. Mitchell, New Light on Self-Ignition, Quarterly of the National Fire Protection Association, Volume 45, No. 2, Oct. 1951.

3. J. Davis and J.F. Byrne, Journal, American Ceramic Society 7, 809, 1924.

4. J.L. Elder, L.D. Schmidt, W.A. Steiner and J.D. Davis, U.S. Bureau of Mines Technical Paper 681, 1945.

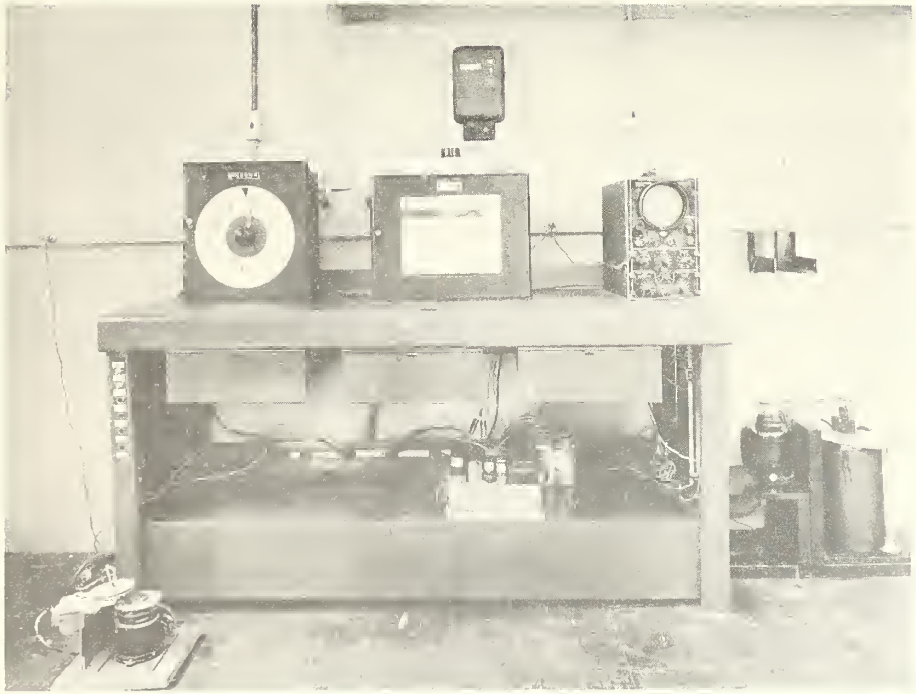


Fig. 1 Complete Assembly of Adiabatic Furnace

Fig. 2 View of Furnace Details

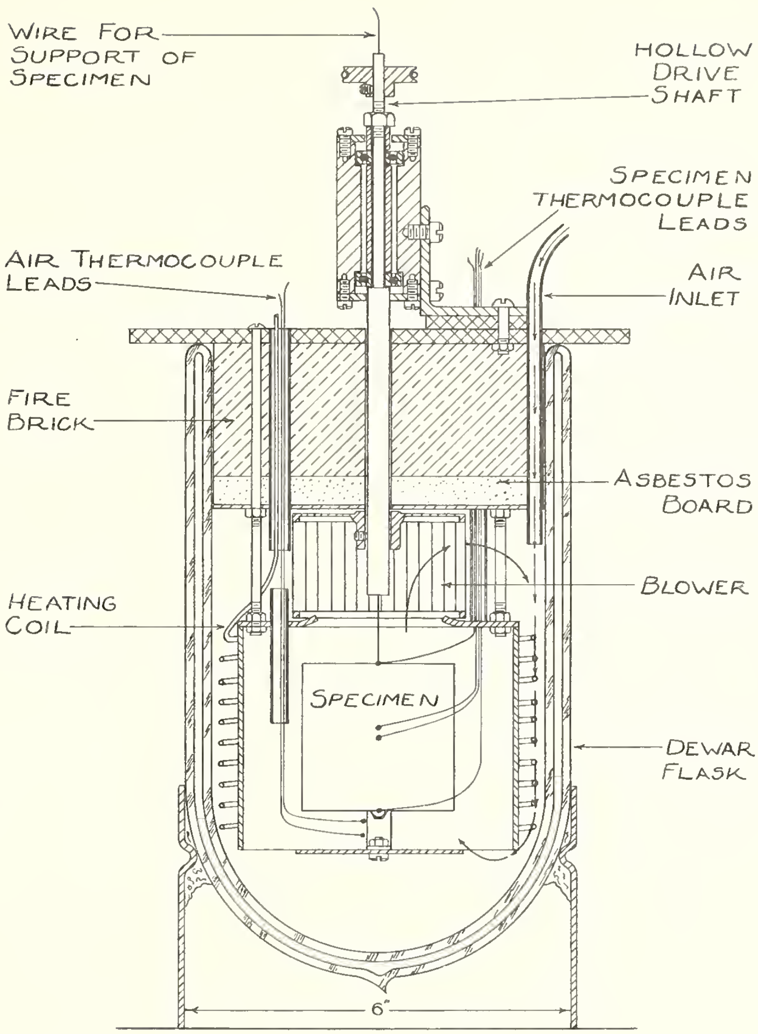


Fig. 3 Cross Section of Furnace

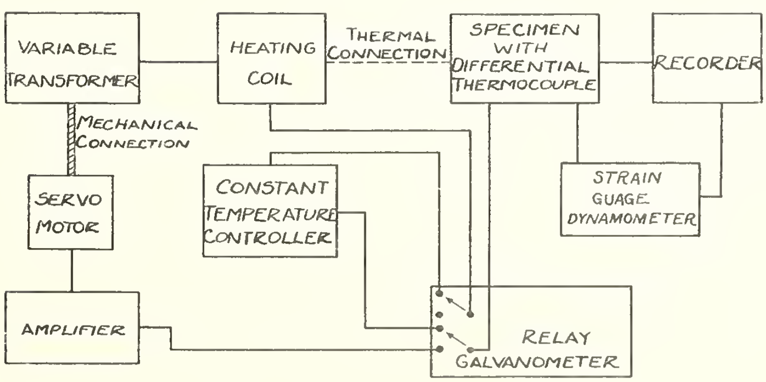
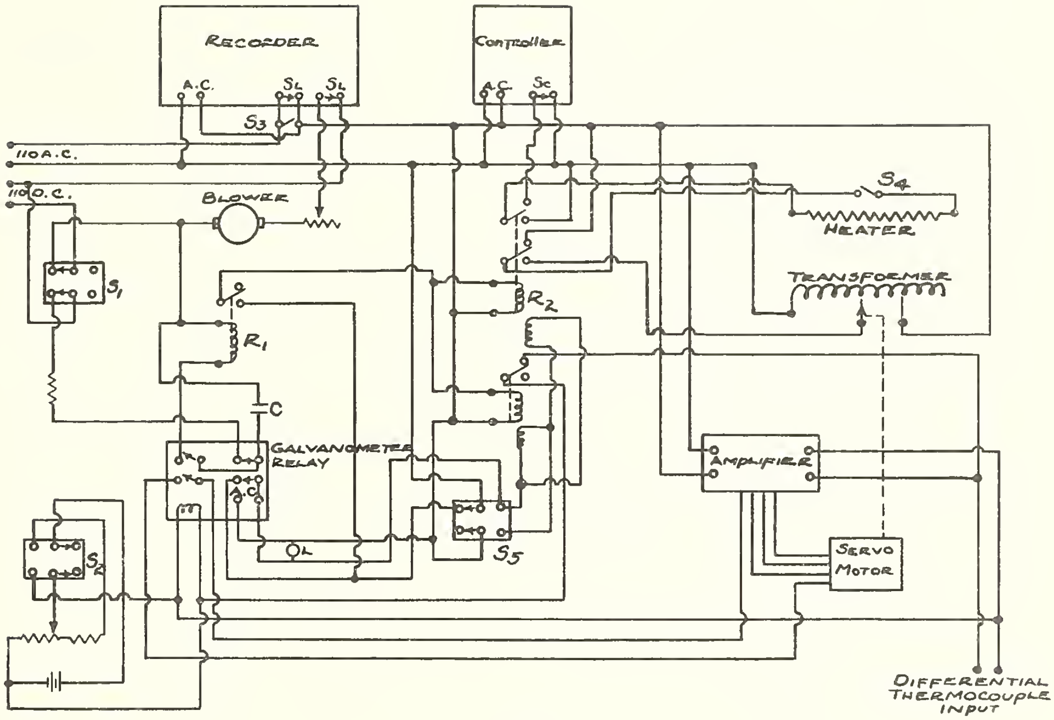


Fig. 4 Block Diagram of Control System



Legend

S_L - limit switch
S_C - controller relay switch
C - 40 uf. 250v
S_1, S_2, S_5 - DPDT toggle switch
S_3, S_4 - SPST toggle switch
R_1 - SPDT plate circuit relay 5000 ohms
R_2 - DPDT A.C. latching relay
L - Neon indicator lamp

Recorder - Minneapolis Honeywell 12-point Electronik Recorder Controller
Controller - Minneapolis Honeywell Electronik Indicator Controller
Galvanometer Relay - G-M Laboratories Inc. Galvanometer Relay
Servo Motor - Minneapolis Honeywell Servo Motor
Amplifier - Minneapolis Honeywell Servo Motor Amplifier
Transformer - Powerstat 7.5 ampere autotransformer

Fig. 5 Wiring Diagram of Control System (strain gage circuit not included)

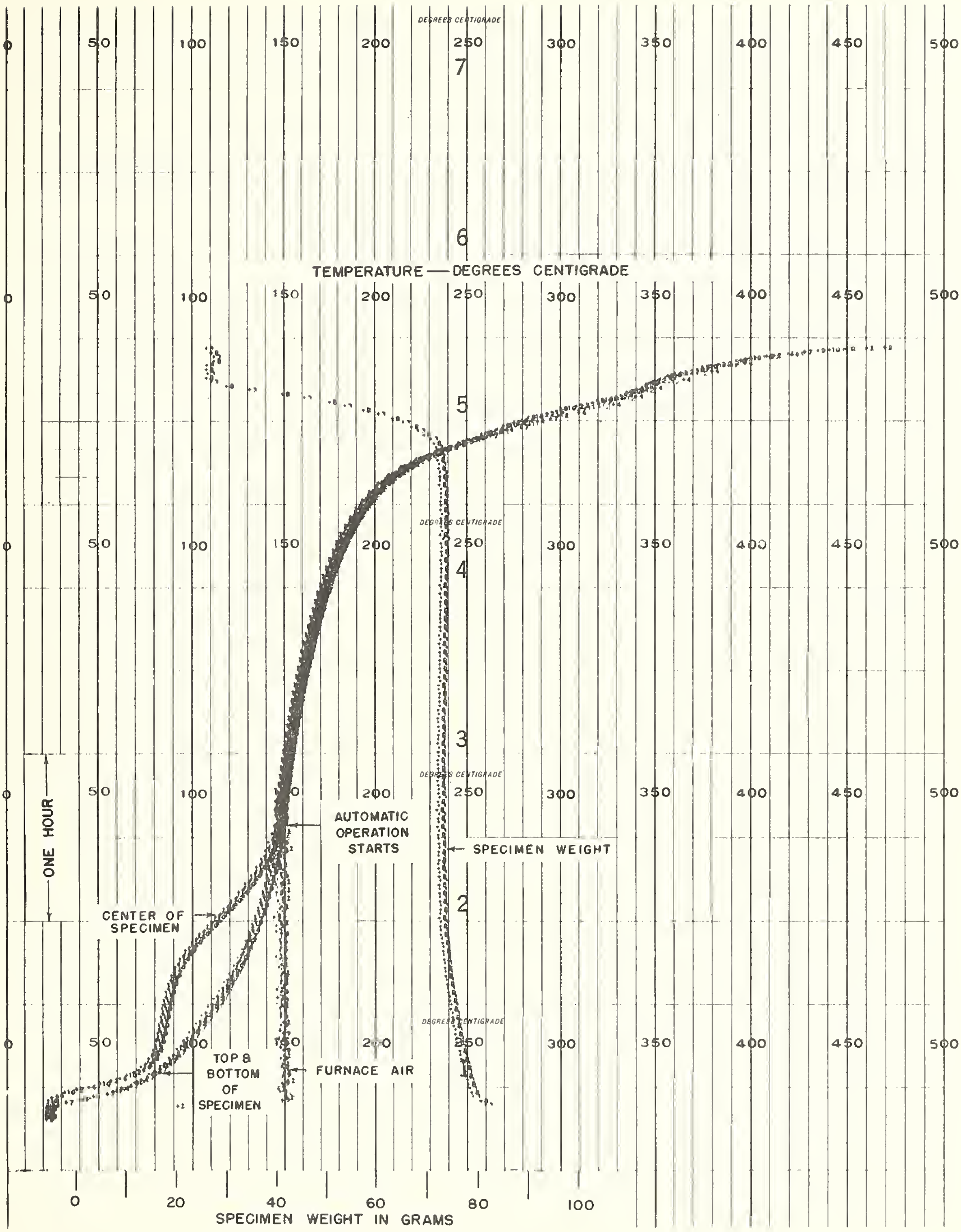


Fig. 6 Typical Time-Temperature Curve of Wood Fiberboard

THE NATIONAL BUREAU OF STANDARDS

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The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to Government Agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services and various consultation and information services. A major portion of the Bureau's work is performed for other Government Agencies, particularly the Department of Defense and the Atomic Energy Commission. The scope of activities is suggested by the listing of divisions and sections on the inside of the front cover.

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