The highest measurable age in a counting time of 48 h is 43 000 years. This limit is calculated from the claim of a net activity in the sample of four times the limit of error (σ). The counter has just been applied to date some Siberian mammoths, where the highest age was found to be 44 000±3500 years.[3] A counting time longer than 48 h was then applied.

It is possible to operate this counter at higher pressure and thus be able to go further back in the past. It is our opinion, however, that the operating condition for the counter is most satisfactory at a pressure not above 2 atm.

In order to measure higher age, we have to make use of our large counter (counter 1),[3] which has been rebuilt in the same way as the small one. Shielding of the counter in the iron chamber (22 cm thick) will be provided by the iron ring counter and by a 1.5-cm-thick layer of lead. The predicted background is 5 counts/min, and the standard net count of modern carbon about 70 counts/min, which gives possibilities for dating samples up to 50 000 years in a counting time of 48 h.

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Controlled Flash Heating of Metal Filaments*

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A simple inexpensive instrument for flash heating metal filaments has been designed which is being used in studying sorption phenomena at very low pressures and in measuring heat capacities and total emissivities of metals. The instrument permits selecting both absolute heating rates and their time dependence; in particular, filament temperatures increasing linearly with time can be obtained. Furthermore, the heating circuit allows the attainment of a preselected maximum filament temperature without appreciable over-shooting. This paper describes the concept and design of the device; detailed experimental results are being reported elsewhere.

I. CONCEPT OF THE DEVICE

THE basic circuit, shown in Fig. 1, consists of a bank of storage batteries as dc power supply, three potential taps E_n in series with three variable resistors R_n of high current rating, the sample filament of resistance R, and a three-position switch S. During a heating cycle the switch is moved through the three positions, shorting consecutively the three circuits through the filament. In this discussion the three heating conditions are called initial, transient, and final stages.

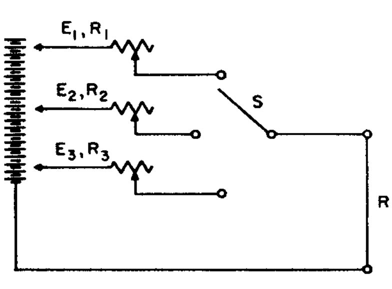

FIG. 1. Schematic of flash circuit.

The energy balance during electrical heating of the filament can be expressed by the differential equation

$$Wdt = I\cdot E\cdot dt = cdT + Hdt, \tag{1}$$

where W is the power input, I the current, E the potential, c the heat capacity, H the sum of all power losses (by emission and by thermal conductance through the gas phase and the leads), t the time, and T the temperature.

Initial and Final Stages

In this discussion we limit ourselves to steady-state conditions for these stages. They are characterized by constant average temperature T_s and resistance R_s of the filament (as well as constant filament potential E_s and current I_s). Since the power dissipation depends only on the filament temperature (and the temperature of the envelope), Eq. (1) reduces to

$$I_s \times E_s = H = W(T_s). \tag{2}$$

It further follows that if the resistance-temperature function is known also, the steady-state temperature T_s is completely defined in terms of either experimental parameter, I_s or E_s.

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Transient Stage

For this stage we assume that the energy losses H are small relative to the power input W and can be disregarded. This assumption is justified in first approximation if the filament is long and is in a high vacuum, if the heating rate is high, and if the final temperature is moderate. The following simple rate-law is now obtained from Eq. (1) (using Ohm's law):

$$dT/dt = (c^{-1})\cdot E\cdot I \equiv c^{-1}(E_2/R_2+R)^2\cdot R \equiv A\cdot 4r/(1+r)^2, \quad (3)$$

where $r=R/R_2$ is relative filament resistance, and $A=E_2^2/(4c\cdot R_2)$ is the heating rate at the instant when the filament resistance becomes equal to the series resistance ($r=1$).

Equation (3) can be integrated if one assumes (as is approximately found for metals) that the heat capacity c is a constant and that R (or r) is a linear function of T

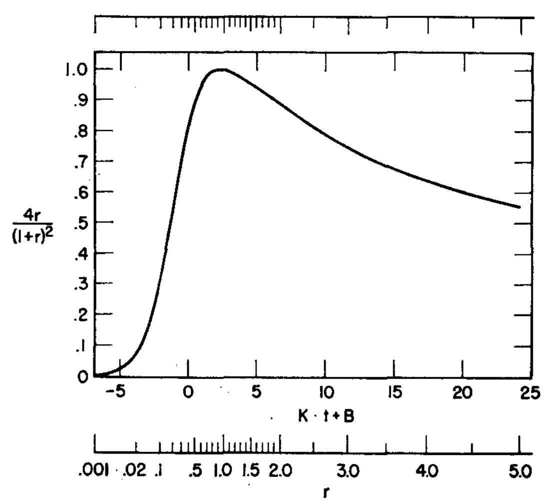

FIG. 2. Flash heating of a filament. Normalized heating rate $4r/(1+r)^2(\sim dT/dt)$ vs normalized time $Kt+B$.

(i.e., $dr=a\cdot dT$),

$$\ln r + r^2/2 + 2r = Kt+B, \quad (4)$$

where K is $4A\cdot a$ and B is an integration constant the value of which is determined by r_0, the relative resistance r at time zero.

On the basis of Eqs. (3) and (4) we can discuss the heating rates as function of either t or r. Figure 2 shows $4r/(1+r)^2 \equiv A^{-1}(dT/dt)$ plotted versus the linear time coordinate $Kt+B$ [evaluated numerically from Eq. (4)]; the equivalent values of r are also marked. At small r values, i.e., for constant current heating, the temperature (or resistance) and the heating rate increase exponentially with time. At the other extreme, for $r_0 \ll r \gg 1$, i.e., constant voltage heating, the temperature increases proportional to $t^{\frac{1}{2}}$, and the heating rate decreases inversely proportional to $t^{\frac{1}{2}}$. At $r=1$, the heating rate passes through a maximum,

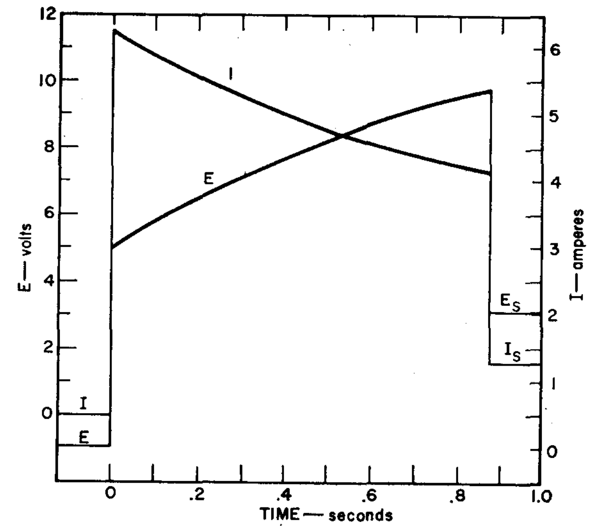

FIG. 3. Experimental record of current I and filament potential E as functions of time t.

and does not change rapidly; it drops to about 90% of the maximum value only at about $r=0.5$ and $r=2$. Thus, the temperature increases about linearly with time over quite a wide range.

For a given filament the heating curve is determined by the two parameters R_2 and E_2 which, within limits, can be preselected. R_2 fixes the temperature at maximum heating rate and thus the linear range of temperature; R_2 and E_2 set the absolute rates at any temperature.

Transitions Between the Three Stages

Transition from the initial to the transient stage is initiated manually. Transition to the final stage, however, has been designed for automatic operation. The critical switching conditions are defined in terms of the maximum filament resistance R_s (which usually will be the final steady-state resistance),

$$I_{\mathrm{crit}} = E_2/(R_2+R_s) \quad \text{and} \quad E_{\mathrm{crit}} = E_2\cdot R_s/(R_2+R_s).$$

For $R_2<R_s$ current control and for $R_2>R_s$ potential control is more sensitive. The automatic control feature thus permits even very rapid heating to any desired final temperature: it constitutes a safeguard against burnout. A detailed description of the design of the instrument is given at the end of the paper.

II. EXPERIMENTAL

The actual experimental data are obtained by recording, with a fast two-channel recorder, the current I (represented by the potential drop across standard resistors) and the filament potential E. Such a record is shown in Fig. 3. For the steady state, I_s and E_s can be measured also manually with the built-in potentiometer.

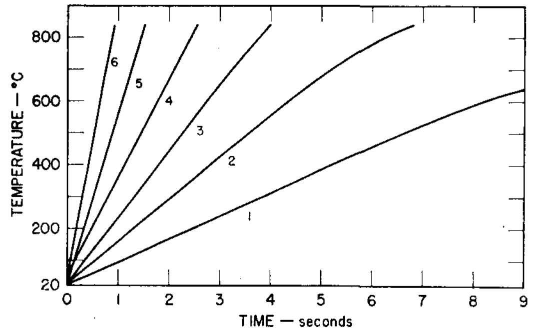

FIG. 4. Heating curves for different power input (see Table I).

Tests were run with a platinum filament, which had a diameter of 0.025 cm, a length of 31 cm, and resistance of 0.625 Ω at 0°C. The resistance function $R/R_0=1+3.96\times10^{-3}T-5.84\times10^{-7}T^2$ was taken from the literature.[1] In Fig. 4, the temperature-time functions for a typical series is plotted. In this series no potential was applied to the filament in the initial state; in the transient state R_2 was 1.9 Ω (equivalent to $T=570°C$ for $r=1$), and E_2 was the variable; finally, the steady-state resistance R_s was 2.45 Ω (840°C). Straight lines are obtained over a wide temperature range, as predicted by the simple theoretical treatment. Only for the slower heating rates are pronounced deviations observed at high temperatures; they are primarily due to radiation losses. The high temperature cutoff for all runs occurs within about 50° of the preset value. This is quite satisfactory in view of the low precision in E_2 and the difficulty in measuring precisely the endpoint on the records.

Table I summarizes the source potentials, slopes dT/dt at $R=1.9$ Ω, and the specific heats calculated from the expression $C_{sp}=1/m\ (E_2^2/4R_2)/(dT/dt)$ (m is the mass of the filament). Although the heating rates cover more than a factor of ten, a fairly constant value of C_{sp} is obtained; the average value, 0.19 J/deg, is quite close to 0.15 J/deg, the literature value of 500°C, considering that heat losses have been disregarded.

TABLE I. Heating conditions and heating ranges for a series of flash heating experiments.

$R_0=0.625$ Ω, $R_s=2.45$ Ω, $R_2=1.9$ Ω
Temperature Range: 25–840°C

Run No.	E_2 (V)	dT/dt (°C/sec)	C_{sp} (J/deg)
1	6	73	0.197
2	8	132	0.194
3	10	209	0.192
4	12	309	0.187
5	16	532	0.192
6	20	890	0.180

[1] F. O. Werner and A. C. Frazer, Rev. Sci. Instr. **23**, 163 (1952).

III. DETAILED INSTRUMENT DESIGN

The detailed wiring diagram of the instrument is shown in Fig. 5. For the purpose of discussion the instrument is considered to consist of three parts: the power handling circuits including the switches, the measuring circuit, and the control circuit for automatic switching.

The power handling circuits (see also Fig. 1) are based on lead storage batteries as source of the potentials E_n which can be varied in increments of 2 V to the maximum value (at present 80 V). Each resistor R_n consists of four variable rheostats of high rating. Shorting switches permit removing any of them from the circuit; in this way the power-handling capacity is not limited to that of the rheostat with the lowest rating, and constant resistance resolution is obtained over the whole range. Transfer between stages is achieved by two switches, a manual one for the transfer from initial to transient stage, and a fast mercury relay Y1 for automatic switching from transient to final stage. The "normal-reverse" selector switch interchanges the operational functions of initial and final power circuits. This is convenient when a heating sequence is immediately followed by a cooling sequence.

The measuring circuit is a simple potentiometer consisting of mercury cells, a ten-turn, one hundred-division precision pot, and a microammeter. The voltage signal to be measured is derived either from the calibrated current shunt in the power handling circuit or from the voltage divider across the load terminals. These two signals, representing current I and filament potential E, can also be recorded through two pairs of external jacks.

In the normal operation the same current shunt is operative throughout the complete cycle; if for any reason different sensitivity in current reading is desired for the initial state, the "standard-special" switch permits using any of the available shunts for the initial state in combination with a 1-A shunt for the two other stages.

The control circuit for automatic switching is based on an operational amplifier system activated by the signal from the potentiometer as follows: The automatic relay Y1 must switch the instrument from the transient state to the final state at the instant when the filament current (represented by a voltage across the shunt) or the load voltage passes through a preselected value. To this purpose this transient voltage is compared with a potential from the reference supply which is set equal to the preselected value. (A function selector switch determines whether current or voltage is to be used as the criterion for switching from the transient state to the final state. In addition, this switch provides a reversal of polarity of the difference voltage in the case of a cooling sequence; this is necessary since specific polarity relations are required for the operation of the control amplifier.) The difference voltage is supplied as an input to the operational ampli-

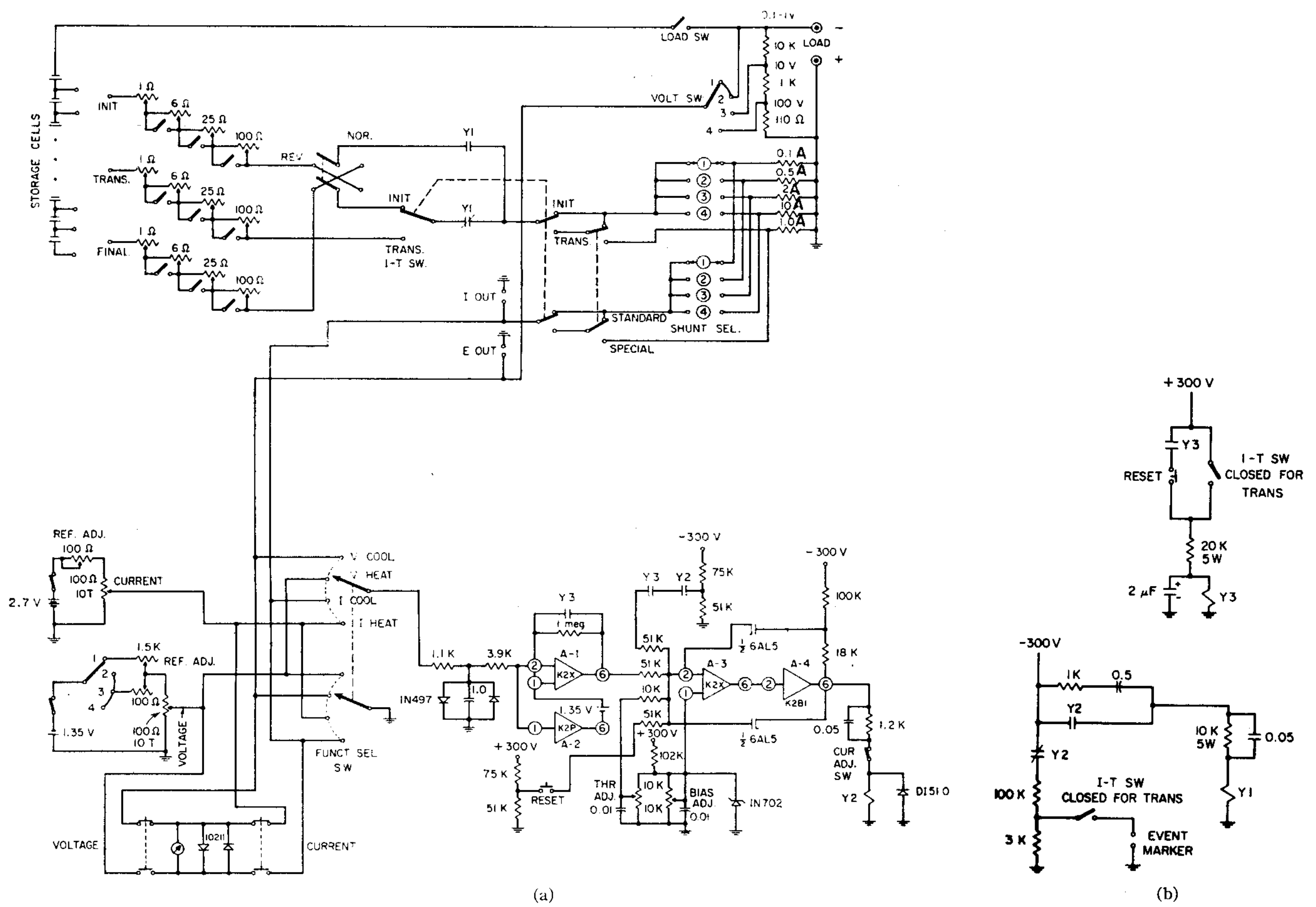

FIG. 5. (a) Circuit diagram of entire instrument. (b) Relay interconnections.

fiers A-1 and A-2. This pair provides a very high gain, and has also chopper stabilization to minimize the effects of internal noise and zero drift. Two crystal diodes are connected between the input of these amplifiers and ground, thus preventing the input voltage to the amplifiers ever to exceed the threshold voltage of the diodes. Consequently the chopper-stabilized amplifier is never driven into saturation. The output of amplifier A-1 is supplied to the amplifier combination A-3, A-4. The biased diode-feedback network around these two amplifiers causes them to operate in a two-state mode. When the voltage input to amplifier A-3 is positive, the output voltage remains at zero and relay Y2 remains open. When the input voltage to amplifier A-3 goes negative through zero, the output voltage immediately increases to 38 V causing relay Y2 to close. One of the contacts of relay Y2 is used to close the mercury relay Y1, thus switching the power handling circuit from the transient to the final stage; and a second contact is used to maintain the input of amplifier A-2 at a negative value, thus locking the system once it has reached the final stage.

A short dead time following the manual switching of the system from the initial to the transient stage is provided by relay Y3; it prevents spurious electrical transients from initiating the automatic switching operation which would transfer the system immediately to the final stage. The system is returned to the initial stage by pressing a manual reset button which overrides this feature. Since the reference supply provides a full-scale voltage of two volts (one volt in the case of control by load potential) and the operational amplifiers are designed to switch the relay on a difference voltage of five millivolts, an accuracy is provided on the order of one-quarter (one-half for load potential control) of one percent of the full scale of the range being used.

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