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PRECISION CIRCUITRY FOR CONDUCTING JOULE HEATING EXPERIMENTS

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ABSTRACT

Kinetic studies of the motion and precipitation of point defects in solids require accurate knowledge of the specimen thermal history. Electrical heating by Joule's effect has been employed in a number of metallic systems. In general the desired conditions have been established manually. The present communication describes transistorized circuits designed to meet the requirements of such heating experiments. Both time and temperature are electronically regulated. Pulsing rates of the order of 10^4 degrees per second are possible with minimal overshoot. Temperatures in the range 173°K to 373°K could be controlled to within ±0.1°. Above 373°K the temperature regulation was ±0.5°. These performance figures are primarily a function of the limitations imposed by the experimental set-up. The circuitry has general application to experiments where precision electrical heating of metallic crystals is desired and where the average specimen temperature can be estimated from the known temperature dependence of its electrical resistivity.

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INTRODUCTION

Detailed studies of the motion and precipitation of vacancies and interstitial subsequent to quenching, irradiation, and cold work require accurate knowledge of the specimen thermal history. In particular when isothermal or isochronal anneals are carried out,[1] it is essential to know the annealing time and temperature; rise time and profile; regulation characteristics at temperature; and decay time and profile.

Electrical heating by Joule's effect has been employed in a number of investigations involving quenched, face-centered cubic metals (e.g., copper[2] silver,[3,4] gold,[5] and aluminum[6]). Since the change in the residual resistivity, measured at 4.2°K or 77°K, was used to detect the quenched induced damage and to study changes in this quantity due to the addition of thermal energy, Joule heating has obvious experimental advantages.

In the cases cited, the electrical conditions have been established manually. Recently Emrick[7] heated gold specimens by means of a shaped electrical current. The advantage of such a technique is a very short rise time at the expense of regulation when the desired temperature is reached.

During studies by the authors of vacancy behavior in quenched and annealed copper[8] and aluminum,[9] the desirability and need for electrical control of thermal cycles became quite apparent. The present communication describes control circuits designed to satisfy the requirements of those experiments. The circuitry has general application to experiments where precision electrical heating of metallic crystals is desired and where the average specimen temperature can be estimated from the known temperature dependence of its electrical resistivity.

GENERAL DESCRIPTION

The components of the control system are shown in block form in Fig. 1. In the present case the metallic crystal was located in an experimental chamber (a) which was embedded in liquid helium or liquid nitrogen. The chamber was evacuated and then filled with helium gas to a low pressure. The specimen was introduced as the unknown resistance in a precision, Kelvin, double bridge (b). The bridge was operated in series with an external d.c. transistorized regulator (c), the power to which was supplied by a bank of low internal resistance batteries (d). The resistance of the specimen was brought equal to the preset bridge value by controlling the current through the bridge. This was accomplished by amplifying the bridge output and using the amplified signal to drive the series regulator. The control loop employed a high grain, d.c., differential amplifier (e). The output of the bridge was also monitored by a null indicator (f), thus allowing the operator to bring the bridge to a fine balance with a zero adjust located in the series regulator circuit. Since a Kelvin double bridge must be used with a yoke and potential leads whose lengths are imposed by the experimental set-up, a potential lead compensator (g) had to be added to the bridge circuit in order to preserve its accuracy.

The annealing time was controlled by a preset digital counter (h). This unit was electronically coupled with a SCR switching circuit (i) which was placed in series with the d.c. regulator. The timer was actuated coincidental with the closure of the current loop or at some later moment by the operator. At the completion of the preset time interval, a pulse from the digital counter activated the open circuit controlling the SCR switch.

The average temperature of a test specimen was determined from published data of the temperature dependence of the resistance ratio $R(T)/R(273^\circ K)$. The resistance of a well annealed crystal was determined accurately at $273^\circ K$ prior to any experiment and the corresponding value of $R(T)$ at the desired annealing temperature, T, was calculated. The data of Meechan and Eggleston,[10] and Leeds and Northrup[11] were used as standards in the case of copper, and Balluffi and Simmons[12] in the case of aluminum.

DESIGN CONSIDERATIONS AND CIRCUITS

The basic requirements of the control system are briefly considered before discussing the various components in detail. In the related experimental investigations, the annealing temperatures, T, of interest could be divided into two ranges of importance, i.e., $173^\circ K \leq T \leq 373^\circ K$ and $373^\circ K \leq T \leq 773^\circ K$. In the former case regulation of $\pm 0.1^\circ$ was sought and in the latter case regulation of better than $\pm 1^\circ$ was considered acceptable. In order to appreciate fully the advantages of 0.1° temperature regulation, the specimen resistance must be measured with an accuracy of at least 0.05%. This results from the fact that the change of electrical resistivity, ρ, with temperature is, in the first approximation, given by

$$\frac{\Delta\rho}{\rho} = \alpha \, \Delta T \qquad (1)$$

where the coefficient, α, is of the order of 0.5% per degree for the quoted temperature ranges and metals under consideration.

It is of particular importance that the control system has the capability of bringing a test crystal to the desired temperature in a short period of time commensurable with minimal overshoot. For the annealing times employed, say t_{min} = 120 sec, a rise time of 0.1 to 0.2 seconds seemed desirable. For a pulse from 4.2°K to 273°K this would correspond to a linear heating rate of between about 1,000°/sec and 3,000°/sec. It is this rise time requirement which determines the large transient power the regulator circuit must be able to release and therefore the magnitude of the transient current the total system must be able to handle without experiencing damage (e.g., i_{max} = 15 amps for the case of an aluminum test specimen).

Lastly, it was important that an annealing cycle be terminated with minimal perturbations in the steady state power required to maintain any particular temperature. This criterion necessitated the need for SCR switching.

It was felt by the authors that a precision bridge offered the best possible solution to the present control problem. The resistance at temperature of test specimens was between 0.1 mΩ and 1.2Ω; holding currents of between 0.2 and 5 amps were required; and a maximum transient current of 15 amps was needed. The Honeywell, portable, Kelvin, double bridge (Model 1622) was selected. It provided the desired 0.05% accuracy; covered the resistance ranges of interest; and was capable of carrying the necessary transient currents for a few milliseconds. An important feature of the Kelvin bridge, relative to ratio sets, is the bridge's low and reasonably constant output impedence to the galvanometer (i.e., < 1kΩ).* In addition the potential lead compensation exposed below would not be practical with ratio sets because of the compensations dependence on the bridge ratio, A/B.

* Ratio sets would not meet the input requirements of the dc amplifier which was considered suitable for the present application.

For the purpose of further discussion, it is useful to establish the equations of the bridge. Referring to Fig. 2a, let X be the resistance of the specimen introduced as the unknown; R the reference resistance set on the bridge; and assume a current, I, is flowing from the voltage source. When I enters the bridge it is divided into a large fraction, i, flowing through the reference resistance and test crystal, and a small fraction, i', flowing through the arms A and B. When the bridge is unbalanced a voltage, V_o, appears at the galvanometer output. If the resistance of the yoke, d, is small, if the resistance of the external potential leads, m and p, can be neglected, and if a/b = A/B, then the Kelvin double bridge is equivalent to a Wheatstone bridge. Its equations can then be written as:

$$V = \frac{I}{\frac{1}{A+B} + \frac{1}{R+X}} \tag{2}$$

$$I = i + i' \tag{3}$$

$$V = (A + B)i' = (R + X)i \tag{4}$$

and

$$V_o = Bi' - Ri \tag{5}$$

Solving this system for V_o yields

$$V_o = \frac{BX - AR}{A + B + R + X} I \tag{6}$$

If X is written as $X = \frac{A}{B} R + \Delta X$ and taking notice of the fact that $R + X \ll A + B$, Expression (6) becomes

$$V_o = \frac{B}{A + B} \cdot \Delta X \cdot I \tag{7}$$

Thus the error signal in volts, V_o, is proportional to the error Δx in the specimen resistance and hence temperature. The sensitivity of the technique depends directly on the bridge ratio, A/B, employed and on the current, I, flowing through the circuit.

Because of the nature of the experimental chamber, the specimen could not be connected to the bridge as recommended. The resistance of the yoke, d, and potential leads, m and p, (see Fig. 2a) will not be negligible with respect to the values of the arms A and B. The resistance reading R on the bridge and the actual resistance of the specimen X will then be related by

$$X = \frac{A + m}{B} R \qquad (8)$$

Since the value of m will certainly exceed the resistance of the potential lead provided by the manufacturer, it becomes necessary to alter the bridge by inserting a fixed resistor of value n in branch B, and q in branch b with $n \simeq q$ (see Fig. 2b). The potential lead compensation (denoted g in Fig. 1) is then simply a set of wire-wound variable resistors, i.e., m' and p'. If n and q are taken such that $n > m$ and $q > p$ the sums $(m + m')$ and $(p + p')$ can be adjusted so as to verify that

$$\frac{m + m'}{n} = \frac{p + p'}{q} = \frac{A}{B} = \frac{a}{b} \;. \qquad (9)$$

R and X are now related by

$$X = \frac{A + (m + m')}{B + n} R = \frac{A}{B} R \qquad (10)$$

and the bridge reading is correct and its accuracy preserved. Note that n and q should be chosen as small as possible in order to minimize the compensation with m' and n' for the cases $\frac{A}{B} = 1$ and 10.

The circuit diagram of the series regulator is shown in Fig. 3. A 12V, low internal resistance (<0.1Ω), dc power supply is connected to the input of the regulator. The bridge error signal is amplified and fed to the regulator at a level determined by a 'zero adjust' circuit which employs a 2N1305 transistor whose base voltage is determined by a ten turn, 10Ω potentiometer. While the resistance of the test specimen is still below the preset value, a large error signal appears at the bridge. The amplified bridge output will reach +10 to +30V above C, thus bringing V_{BE} of the 2N1305 to large positive values. The 1N3392 Zener diode prevents V_{BE} from exceeding the breakdown value for that transistor. An adjustable dummy load, placed in series with the bridge allows proper adjustment of the power brought to the specimen while the gain adjustment on the dc differential amplifier is set so as to obtain the best possible temperature control with minimal overshoot and with no oscillation when going from temperature rise to temperature regulation. The circuit was designed for two ranges of power, i.e., 0.3 to 6 watts and 2 to 30 watts. For the latter output condition a 0.1 μF capacitor is introduced into the feedback loop of the 2N1305 and the dummy load is adjusted in the range 0.1 to 1Ω.

The differential, dc amplifier should meet the following specifications: a) high sensitivity and low noise (≈ 1μV); b) high common mode rejection (≈ 300V); c) gain to 100 to 1000 at up to 10 kc; and d) fast recovery time after large input overload (< 1 msec). The last two conditions insure a fast heating rate without overshoot. The Dynamics, differential, dc amplifier (Model 6450) was employed.

For the purpose of monitoring the bridge output it was found necessary to use an electronic null indicator since overload protection and fast recovery were needed. Its sensitivity should be in the μV/cm range; its input impedence in the MΩ range; and it should have adequate isolation from the line voltage in order to avoid distrubances on the input side of the high gain, dc amplifier. The Brown, electronic, null indicator (Model 104W1-6) was used.

The timer was a Beckman, Instruments Inc., preset, digital counter (Model 5423) fed with the 60 cps line signal. Its second decade was modified so as to trigger the third decade after 59 pulses instead of 99. The annealing time could then be preset directly in seconds rather than in line cycles. Controlled anneals of 999.9 seconds were possible with this arrangement.

The switching circuit is presented in Fig. 4. It is essentially composed of a power SCR transistor (type C 45) triggered manually at the start of a thermal cycle. It is turned off af the end of the cycle by discharging an RC circuit with reverse polarity across the power transistor. This discharge is triggered by applying to the gate of a 2N2327 SCR transistor the 15 volts pulse coming from the preset digital counter. In the present circuit the time constant of the RC circuit and the charging voltage of the capacitor can be chosen so as to be adequate to turn off the power transistor without bringing any additional power to the test specimen. This is a particularly important feature of the switching circuit when considered in conjunction with its present application.

All potential leads and low signal leads were carefully shielded. The circuit was grounded at one point only, in order to avoid ground loops. It seemed most appropriate to ground a current lead as close as possible to the galvanometer output of the bridge.

APPLICATION

Annealing experiments have been performed with this circuitry employing both copper[8] and aluminum[9] test samples. Sepcimens were pulsed to a desired temperature directly from the liquid helium or liquid nitrogen state. In the case of copper, wires 0.005 cm dia. and 10 cm long with a 5 cm gauge length, were mounted inside a 5 cm dia. pyrex glass chamber. The chamber was filled with helium gas to a pressure of 500μ Hg and it was embedded in liquid helium. A typical oscillographic trace of the bridge error signal during a pulse from 4.2°K to 273°K is shown in Fig. 5. The rise time was about 23 msec and the corresponding average heating rate was approximately 12,000 degrees per second. For the voltage sensitivity employed no overshoot was observed. The amplifier gain setting was 100; the dummy load was 2Ω; and the series regulator was operated in the 'low' output power condition (i.e., 0.3 to 6W).

Aluminum crystals, 0.16 cm by 0.008 cm in cross section and 25 cm long with a 10 cm gauge length, were mounted in a 8 cm dia. stainless steel chamber. For these experiments the chamber was filled with helium gas to 5μ Hg pressure and it was embedded in either liquid helium or liquid nitrogen. The oscillographic trace of the bridge imbalance for pulses to 273°K was similar to that shown for copper, but the heating rate was of the order of 700 degrees per second. This slower rate was due to the larger specimen cross section combined with a larger surface area to volume ratio, thereby increasing the heat losses during a pulse. Typical operating conditions were: amplifier gain 100; dummy load, 0.5Ω; and series regulator in the 'high' output power condition (i.e., 2 to 30 W).

From measured variations in the bridge error signal, V_o, and employing Eqs. (1) and (7), it was concluded that the average temperature of a test specimen could be controlled to within ±0.1° in the temperature range 173°K to 373°K. The temperature regulation was estimated to be ±0.5° above 373°K. These error limits are applicable to both the copper and aluminum experiments.

The principal weakness of the self-heating technique arises from temperature inhomogeneities along the specimen gauge length due to end losses and/or local variations in the cross sectional area. In order to evaluate the importance of a non-uniform temperature distribution, a series of aluminum specimens (melting point 933°K) were pulsed to temperatures in the range 823° to 928°K. If the cross section of a crystal was not sufficiently uniform, local melting would take place. When the cross section was reasonably uniform, no specimen damage was observed after such pulses and melting between potential leads was induced after brief pulses to 931°K. These results were interpreted as indicating that, for ideal specimen geometry, minimal overshoot was taking place and acceptable local and average temperature control was still possible for annealing temperatures approaching the melting point of the test sample.

It is worth pointing out that the present technique has several advantages when compared with the classical method of employing stirred liquid baths. Among the important advantages are: 1) specimen handling is avoided; thin or delicate crystals may be mechanically deformed while being introduced or removed from a liquid bath or by the stirring action of the bath itself: 2) since annealing takes place within an experimental chamber whose environment can be carefully controlled, contamination of highly reactive metals can be minimized; and 3) the test sample alone is brought up to the desired temperature while the supporting elements and chamber wall remain close to the temperature of the cryogenic liquid employed, thus low temperature conditions are re-established quickly after each thermal pulse and subsequent resistance measurements can be made promptly.

In conclusion it is felt that these results, combined with reproducible data obtained from isochronal and isothermal anneals of quenched copper[8] and aluminum[9] samples, illustrate the reliability and value of the reported technique and associated circuitry.

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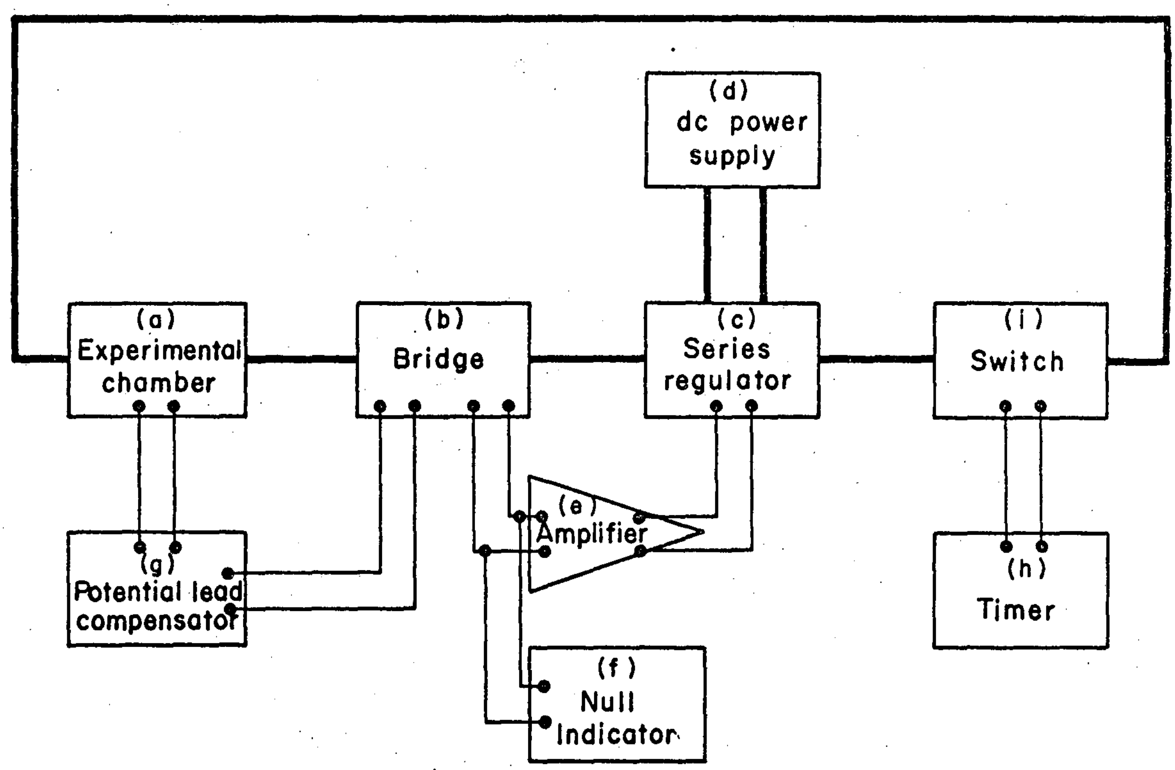

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Fig. 1 Block diagram of the components used in carrying out an annealing cycle.

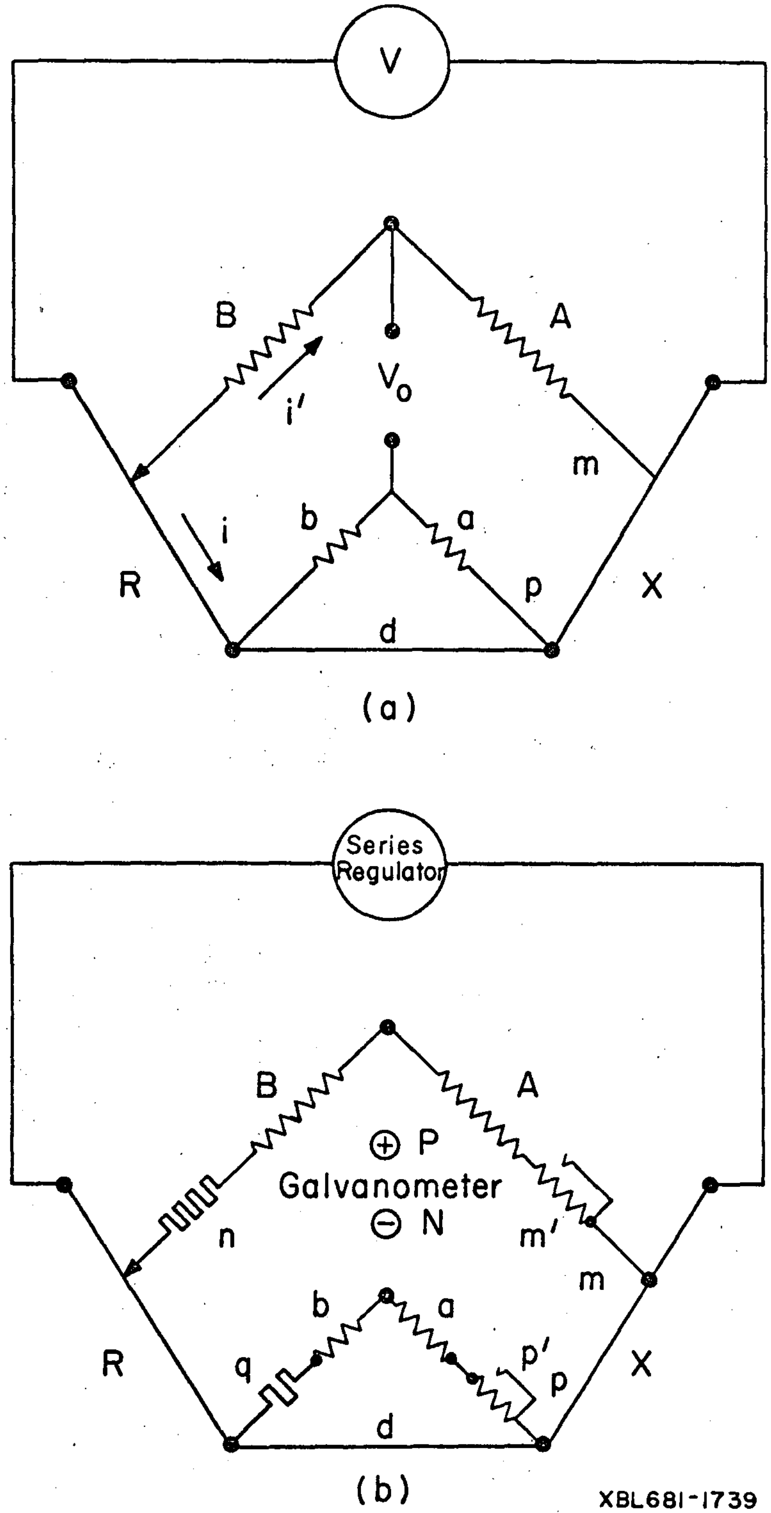


Fig. 2 Bridge Circuits: a. Kelvin double bridge.
b. modified Kelvin double bridge with variable potential lead compensation represented by m' and p'.

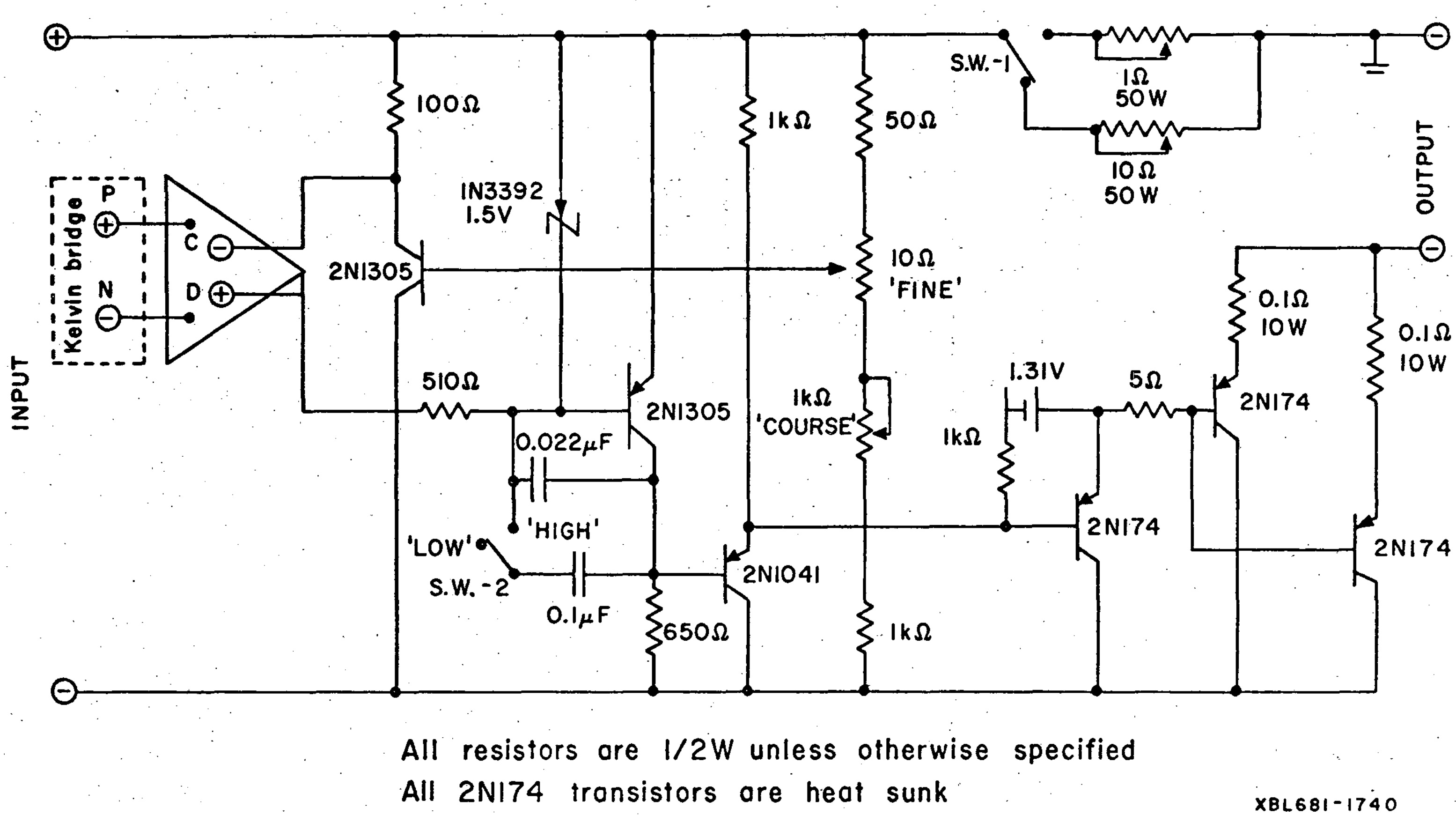
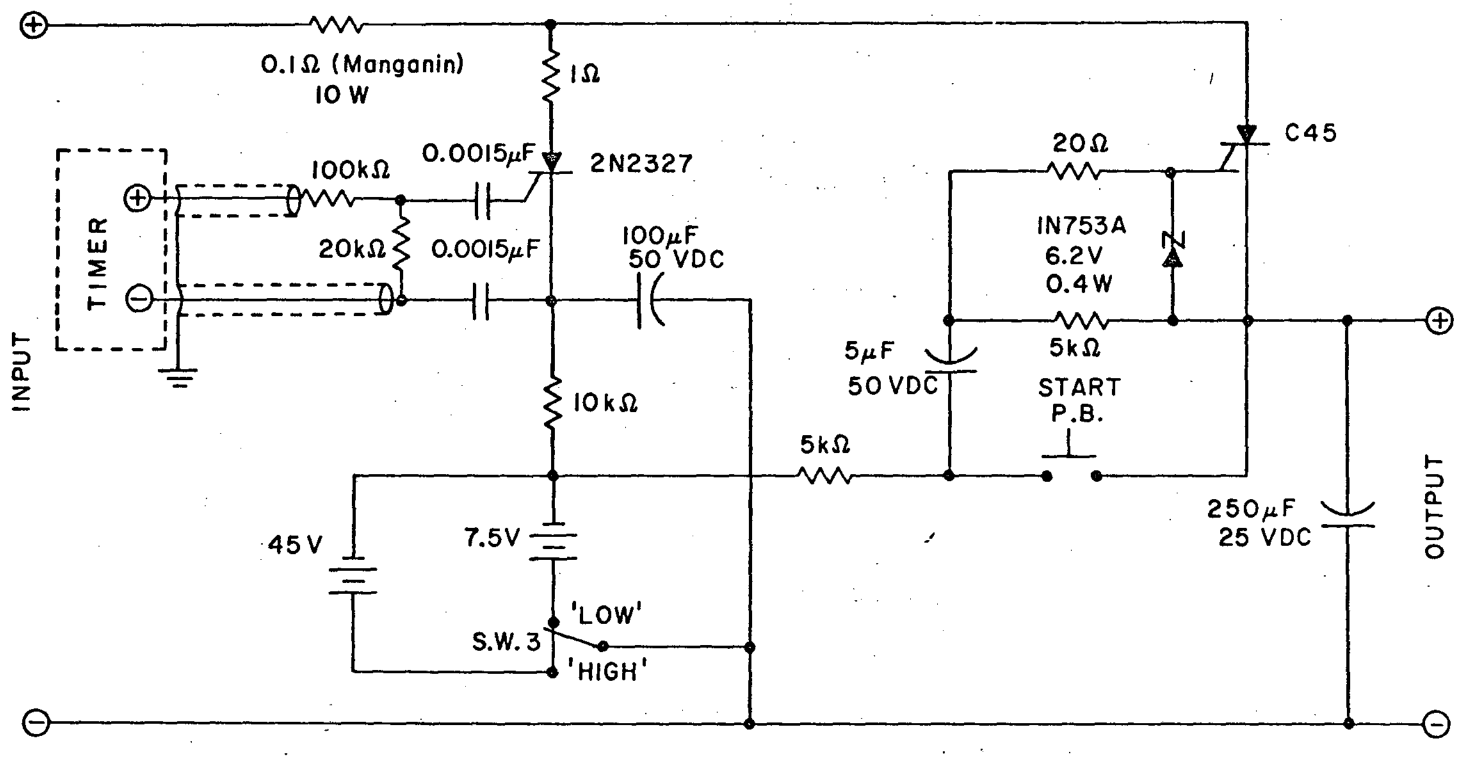


Fig. 3 Circuit diagram of the series regulator; switches s.w.1 and s.w.2 are shown in the 'low' output power position.



All resistors are 1/2W unless otherwise specified

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Fig. 4 Circuit diagram of the SCR switching unit: switch s.w.3 is shown in the 'low' charging position.

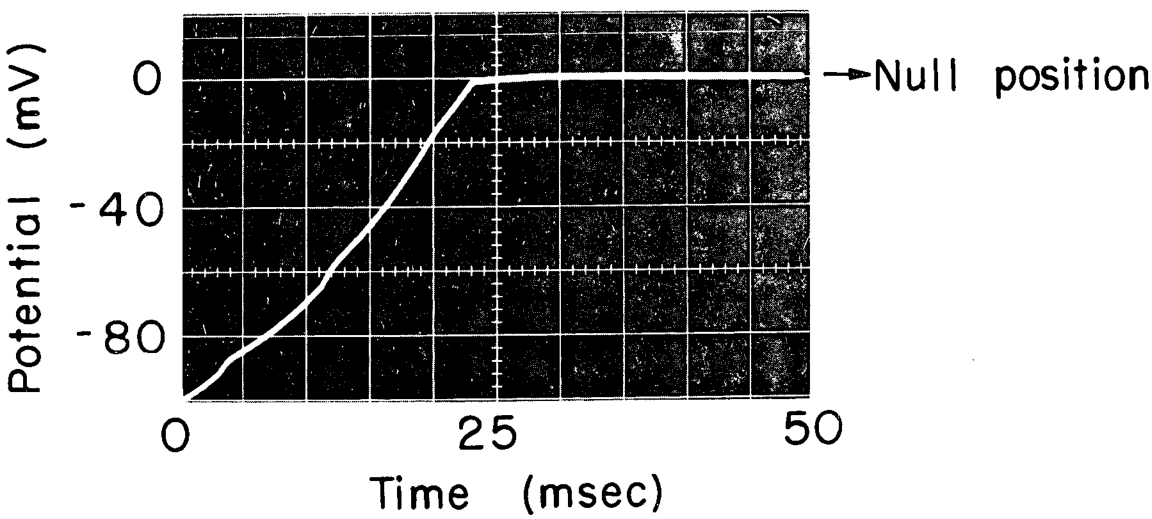

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Fig. 5 Oscillographic trace of the bridge error signal for a pulse from 4.2° to 273°K.