COMPUTERIZED CONTROL AND DATA ACQUISITION SYSTEM
FOR QUENCHING AND ANNEALING THIN METAL WIRES*

by

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ABSTRACT

In the study of the thermal generation and the annealing of point defects, e.g., vacancies, in metal crystals, it is necessary to have accurate and reproducible control over the thermal history of the specimen. This paper describes a computer-controlled system for the rapid thermally pulsing, quenching and annealing (thermal cycling) of thin metal wires. The system features complete software control of the temperature vs time profile of the sample. Using programmable gain instrumentation amplifiers and a crystal-controlled programmable time base, both rapid and accurate ($dT/dt \leq 10^6$ °K/sec; $\Delta T \leq \pm 5^\circ\text{K}$) control of the thermal cycle is obtained.

INTRODUCTION

Quenching has long been a powerful technique for the study of point defects in crystals. Over the past decades, it has been widely used in the investigation of thermally generated vacancy defects in metals. Since high heating and quenching speeds are required, the specimens used in quenching experiments are usually thin metal wires. The quenching temperatures are usually close to the melting point. In order to minimize the exposure of the specimen to high temperature where both evaporation and shape changes due to surface tension effects can occur, a rapidly operating computerized control and data acquisition system was designed. This system has been successfully applied to vacancy quenching experiments on tungsten¹. It can also be used for the study of vacancy generation and annealing kinetics in any system requiring very rapid heating and short holding time at elevated temperatures.

PRINCIPLES OF OPERATION

In quenching experiments, a specimen is heated rapidly to the desired quench temperature (T_Q), then maintained at T_Q long enough to reach equilibrium, and finally quenched. In gas quenching² or "radiation quenching" in vacuo¹, the relatively large heating current required to hold the specimen at T_Q is rapidly switched to a much lower measuring current in order to produce the quench. The resistance vs time during the quenching cycle are recorded; thus giving an exact record of the thermal history of the specimen.

A simplified block diagram of the computerized closed-loop control system which we have constructed for this purpose is shown in Fig. 1. The heating current is supplied by a power driver controlled by a Data General Nova 1220 minicomputer through the digital-to-analog converter (DAC). The voltages developed across the specimen (V_x) and the standard resistor (V_s) are monitored by differential inputs, programmable gain instrumentation amplifiers (PGIA's). These amplifiers feed a high speed analog-to-digital converter (ADC) which closes the feedback loop with the system.

The temperature of the specimen is determined by its D.C. resistance. To eliminate geometrical factors of the specimen, a resistance ratio (RR) is used instead.

$$RR(T) = \frac{R_s}{R(273)} \cdot \frac{V_x}{V_s}$$

where R_s is the resistance of the standard resistor, and $R(273)$ is the specimen resistance at 273°K. A look-up table of temperature vs RR in appropriate temperature increments is contained in the computer's memory, allowing rapid conversion from the calculated $RR(T)$ to actual temperature by interpolation.

The computer contains a crystal-controlled programmable timer (PTM) which has a dynamic range of 100 μ sec to 54.6 min. (Its details are described in the next section.) The PTM is used to control the heating rate, holding time at T_Q and the frequency of measurements made by the ADC. The programmable gain feature (gain = 1 to 1.6×10^4) allows a very wide dynamic

range of voltages and currents to be measured while maintaining the full scale accuracy of the ADC.

The most important feature of this system is its programming flexibility. With the use of PTM and PGIA's, various experimental conditions can be achieved under software control. Since the function of the computer is primarily one of control and not calculation, assembly language programming is used exclusively. The program has been written so that at the beginning of each run, the computer requests, for quenching experiments, the following information: 1) PGIA gains, 2) quenching temperature (T_Q), 3) number of data points while holding at T_Q and during quench (N_Q), and 4) the ADC sampling frequencies. At the conclusion of a run, the complete thermal history of the specimen is plotted as a temperature vs time profile. A flow chart of the software control on the quenching cycle is shown in Fig. 2.

SYSTEM DESCRIPTIONS

The time base is generated by counting down to zero from a computer-preset number at a crystal-controlled clock frequency (also computer selectable) of either 10, 1, .1 or .01 kHz. Thus, the time base period will be N/f , where N is the number to be counted down, and f is the selected clock frequency. Fig. 3 shows the schematic logic diagram of the programmable timer. All devices are 7400 series TTL integrated circuits, with the exception of the 100 kHz Statek crystal oscillator. By issuing a "DATA OUT" command and 0 to 3 I/O (input/output) pulses, the

computer selects both N and f, respectively. Upon reaching zero, the SN74193 borrow output goes low and activates the computer's interrupt system. Thus, by setting both f and N, a time base with a range from 0.2 msec to 54.6 min is available.

The ADC is an Analogic AN2813M unit made up of an eight channel differential analog multiplexer, sample and hold, and a 13 bit A/D converter with a conversion time of 6 μ sec/bit. The throughput rate is 11.5 kHz. In this system, the conversion is synchronized to 10 kHz clock pulse from the PTM. The programmable instrumentation amplifier is made up of Burr-Brown 3061/25, 3602J and 3604J analog amplifier models. The 3061/25 is an instrumentation amplifier with gain set at one, and differential input impedance 50M Ω . The 3604J and 3602J are programmable gain buffer amplifiers with gains of 1-16 and 1, 10, 100, 1000, respectively.

The power driver is mainly a x2 voltage amplifier followed by current slave transistors in a Darlington configuration. The x2 gain is needed since the 0-10v range of the Burr-Brown Model DAC50 DAC is not sufficient for very high power applications.³

A point plotter is used to give the hard-copy output of the T vs t curve. For each run, a Hewlett-Packard Model 7004B x-y recorder with a point plotter plug-in is easily interfaced to the system. The maximum plotting rate is 50 plots sec⁻¹. In operation, the x-axis is calibrated as the time scale, whereas the y-axis as the temperature scale.

The programmable timer was assembled on a Data General general purpose interface board. The data registers for digital inputs to the DAC's for the plotter were built on another general purpose interface board. The DAC's and the PGIA's were built on five 10 x 16 cm P.C. boards which fit into Vero card frames.

APPLICATIONS

A. Up-quenching Experiment

In order to study the kinetics of thermal vacancy generation in metals, one would like to have the specimen heated very rapidly and accurately to a selected elevated temperature, held there for a short time, and then rapidly down-quenched^{4,5}. In this case the lattice is subjected to a large vacancy subsaturation as a result of the rapid heating, and a large driving force for the generation of vacancies exists. The heating rate and the time period held at high temperature are only limited by the analog-to-digital conversion speed and the computer execution time for the necessary temperature-sensing instructions. The holding time at high temperature can range from 0.2 msec to several minutes.

B. Down-quenching Experiment

This system was purposely designed for down-quenching experiments on tungsten¹, but has recently been successfully applied to similar experiments using fine platinum wires. Fig. 4

shows the typical thermal history of a down-quenched specimen. By rapid quenching, the high temperature equilibrium vacancies are frozen into the lattice. However, since the quenching rate is finite, some fraction of the vacancies initially present at equilibrium at T_Q are lost to vacancy sinks during the quenching period. In order to correct for this loss of vacancies, a given specimen is therefore quenched at different rates. From the known quenching rates, the quenched-in vacancy concentration corresponding to an infinite quenching rate (zero vacancy loss) can be obtained by extrapolation.

Several techniques may be used to modify the quenching rate. These include varying the quenching medium, capacitor discharge and software control. To date, only capacitor discharge has been employed, but work is progressing on direct software control of the quenching T vs t profile. This is potentially an extremely powerful tool for the study of the vacancy kinetics, since this has traditionally been the least controlled aspect of all thermal vacancy studies. One could easily envision a programmed cooling cycle designed to discern mobilities of single vacancies and the more mobile divacancies⁶.

Furthermore, this system can be programmed so that the exposure of the specimen to high temperature is just long enough to generate the equilibrium vacancy concentration at T_Q . Consequently, the corresponding shape changes due to surface tension effects and evaporation at elevated temperatures are minimized.

C. Quenching and Annealing Experiment

The present system is especially convenient to study the kinetics of the quenched-in defects at relatively lower temperatures during isothermal and isochronal annealing experiments. Fig. 5 shows the thermal history of a 75 μm diameter tungsten wire radiation quenched from 3000°K, then annealed at 2000°K for 2 sec, and finally quenched.

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FIGURE CAPTIONS

- Fig. 1 Block diagram of computer-controlled quenching and annealing system. The arrows indicate the direction of data transmission of the closed-loop control of the thermal cycling of the specimen. The teletype and cassette permit real time interaction with the experiment.
- Fig. 2 Flow chart of software control on the quenching cycle.
- Fig. 3 Schematic logic diagram of the programmable timer. BC - Binary Counter, DS - Data Selector.
- Fig. 4 Computer generated plot of thermal history of specimen during heating and quenching. Plot was obtained for 75 μ m diameter tungsten wire radiation quenched in 5×10^{-6} torr vacuum from 3400°K. Rise time during heating was ~.6 sec. Holding time at 3400°K was 1 sec. The sampling frequency during the whole thermal cycle was one in every 2 msec.
- Fig. 5 Computer generated plot of thermal history of specimen during heating, quenching and annealing. Plot was obtained for 75 μ m diameter tungsten wire radiation quenched in 5×10^{-6} torr vacuum from 3000°K, then annealed at 2000°K for 2 sec, and finally quenched down to room temperature. The sampling frequency during the whole thermal cycle was one in every 2 msec.

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.