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OAK RIDGE NATIONAL LABORATORY

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ORNL/TM-7276

A Multispecimen Dual-Beam Irradiation Damage Chamber

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ABSTRACT

An irradiation damage chamber that can be used to rapidly simulate fast neutron damage in fission or fusion materials has been designed and constructed. The chamber operates in conjunction with dual Van de Graaff accelerators at ORNL to simulate a wide range of irradiation conditions, including pulsed irradiation. Up to six experiments, each with up to nine 3-mm disk specimens, can be loaded into the ultrahigh vacuum chamber. Specimen holders are heated with individual electron guns, and the temperature of each specimen can be monitored during bombardment by an infrared pyrometer. Three different dose levels may be obtained during any single bombardment, and the heavy-ion flux on each of the nine specimens can be measured independently with only a brief interruption of the beam. The chamber has been in service for nearly three years, during which time approximately 250 bombardments have been successfully carried out. An appendix contains detailed procedures for operating the chamber.

INTRODUCTION

The utility of heavy-ion bombardment to simulate the neutron irradiation of materials lies not only in the enormous reduction in time required to obtain a given level of damage but also in the increased precision with which most of the irradiation parameters can be measured and controlled. The most important of these are the irradiation temperature and the particle flux; it is worthwhile to expend considerable effort to monitor these closely. A clean high-vacuum environment is also a prerequisite because the damage in the affected near-surface layer of the specimen can be too easily modified by intrusion of contaminants from a poor vacuum. And since one of the principal uses of heavy-ion irradiation experiments has been the rapid comparative screening of large numbers of proposed alloys, the capacity to accommodate many specimens per bombardment and several bombardment experiments per loading is a very desirable attribute of any simulation facility. We describe here a new irradiation damage chamber that has been constructed with the above goals in mind. In addition, this chamber is one of the few in operation that

permit the simultaneous injection of light ions like He^+ or H^+ (from a 400-kV Van de Graaff accelerator) along with the primary damage, which here is usually produced by a 4-MV beam of $^{58}Ni^{2+}$ ions from a 5.5-MV (maximum) Van de Graaff accelerator. In this report the various subsystems of the damage chamber are described followed by the Appendix, which sets forth the detailed procedures currently employed in the loading and operation of the facility.

CONSTITUENT SYSTEMS

Chamber Body and Vacuum System

The ORNL facility for heavy-ion simulation of neutron damage has been recently described.[1] An overall view of the two accelerators and their respective beam lines is given in Fig. 1. This report is concerned only with the damage chamber (item k in Fig. 1) and ancillary equipment. The chamber is shown in more detail in Fig. 2. The body is constructed from intersecting stainless steel tubes, the outer ends of which terminate in standard copper gasket flanges. Either of two interchangeable target assemblies (Fig. 3) attaches to the chamber. The target assemblies have six individual target-heater modules; a large bellows, which permits 230 mm of linear motion; and all the power and instrumentation feedthroughs. A precision machine-tool bed attached to the chamber body is used to manually align any desired target with the common intersection point of the two charged particle beams. These beams enter the chamber by separate ports with a 15° angle between them. Other ports at the same angle relative to the heavy-ion beam permit illumination and visual observation of the specimens during bombardment.

Clean ultrahigh vacuum is provided by a 1000 L/s cryopump together with two liquid-nitrogen sorption pumps that are used for initial pumpdown. The cryopump is attached to the chamber through a large-diameter gate valve and an elbow coupling to minimize radiant thermal load on it from the specimen heaters. It is employed below about 0.25 Pa (2×10^{-3} torr). If no specimens are being heated, the chamber can be pumped to a base pressure as low as 9×10^{-7} Pa (7×10^{-9} torr). Specimens are

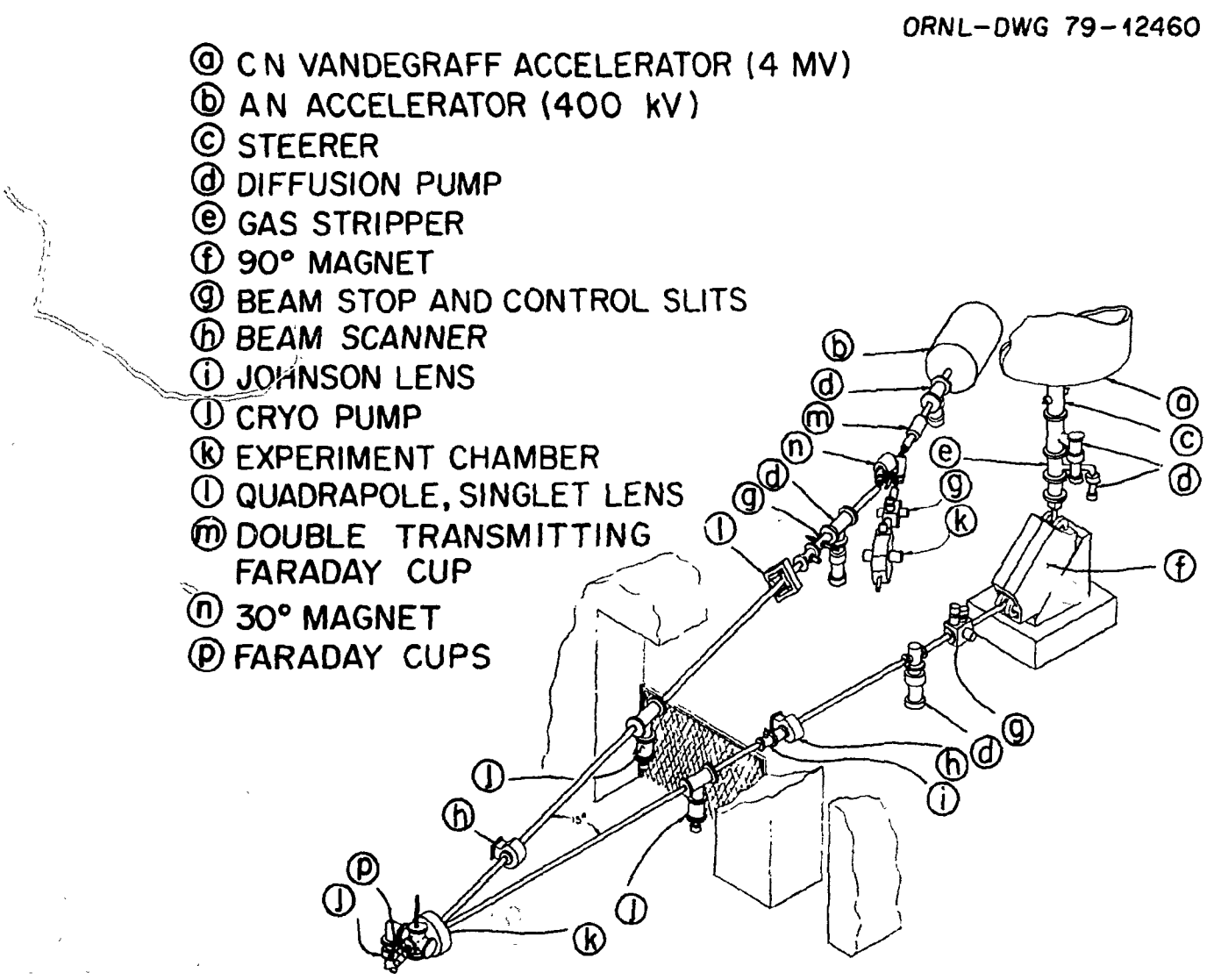

Fig. 1. Schematic of the Two Accelerators and Their Respective Beam Lines to the Radiation Damage Target Chamber (Lower Left).

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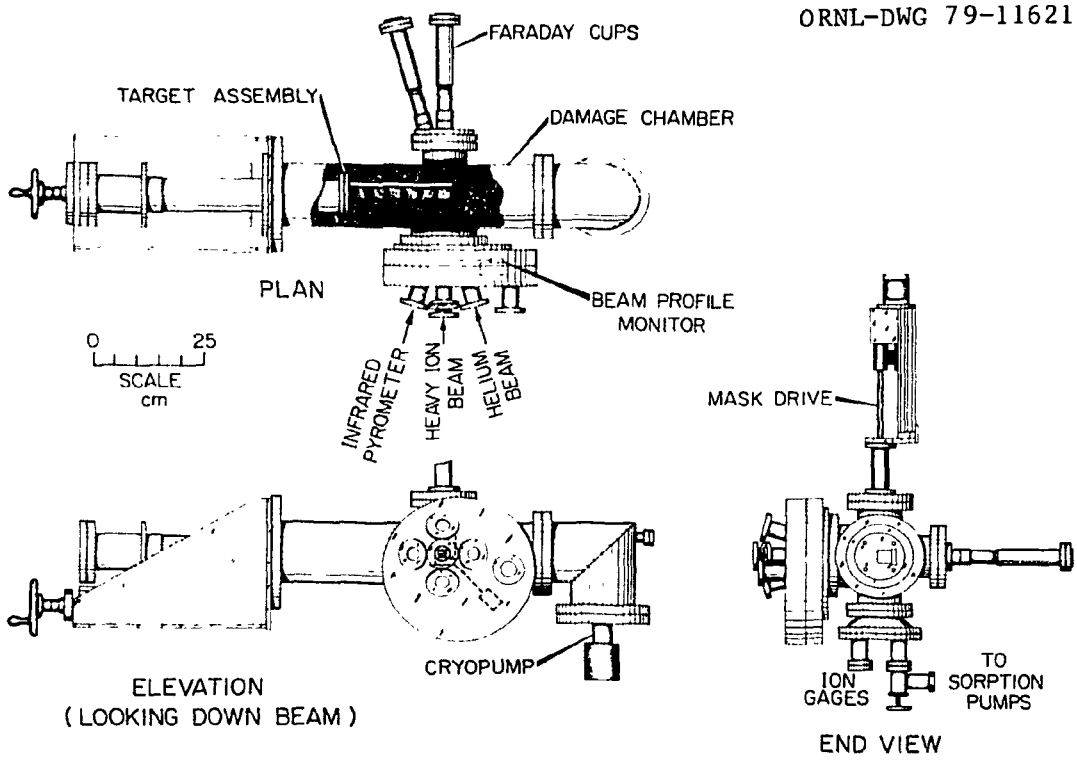

Fig. 2. Layout of the Radiation Damage Target Chamber.

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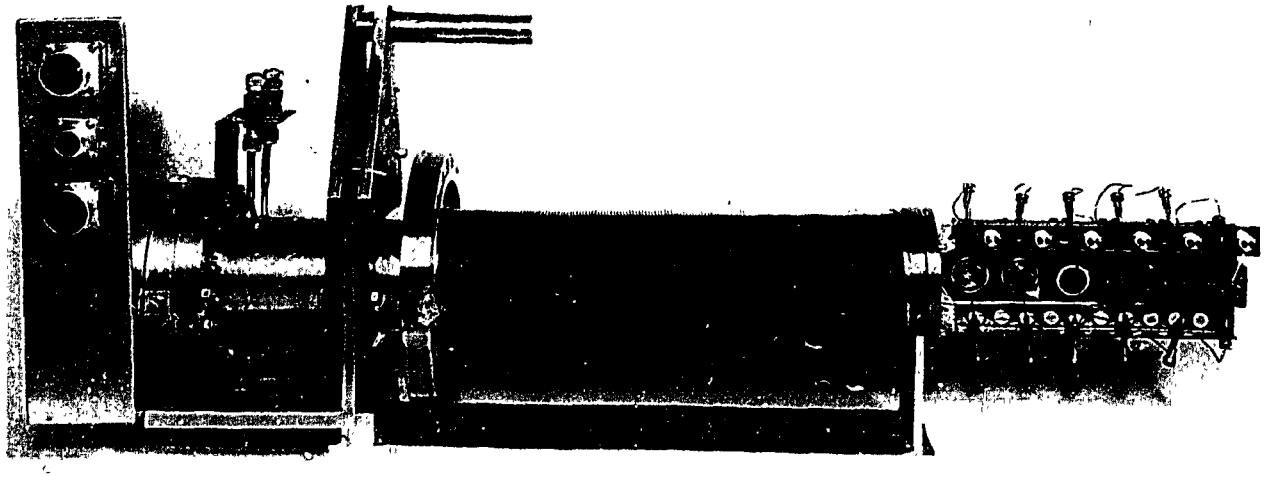

Fig. 3. One of the Target Assemblies, Removed from the Damage Chamber.

bombarded at elevated temperatures generally with a pressure in the low 10^{-5} to high 10^{-6} Pa (low 10^{-7} to high 10^{-8} torr) range. Both a nude ionization gage and an intermediate-range Varian Millitorr gage are used to monitor the pressure, while a Varian quadrupole residual gas analyzer (RGA) is available to evaluate the composition of the remaining atmosphere. A typical spectrum from the RGA is shown in Fig. 4. The primary peaks in such a spectrum are readily determined and are indicated in the figure. The water vapor pressure is high, presumably from the strong dipole moment of the H_2O molecule, which makes it resistant to pumping. The dissociation of H_2O in the analyzer gives rise to OH and H peaks. The H_2 and CO_2 peaks are assumed to be contaminants of the stainless steel in the chamber itself. The argon peak is from the argon gas stripper upstream from the chamber.

Built into each target assembly is a resistance-heating loop and a water-cooling channel. Heating to about 100°C can hasten outgassing just after pumpdown, while the water line hastens the cooling of specimens and heaters to ambient temperature, which is necessary before they can be exposed to air.

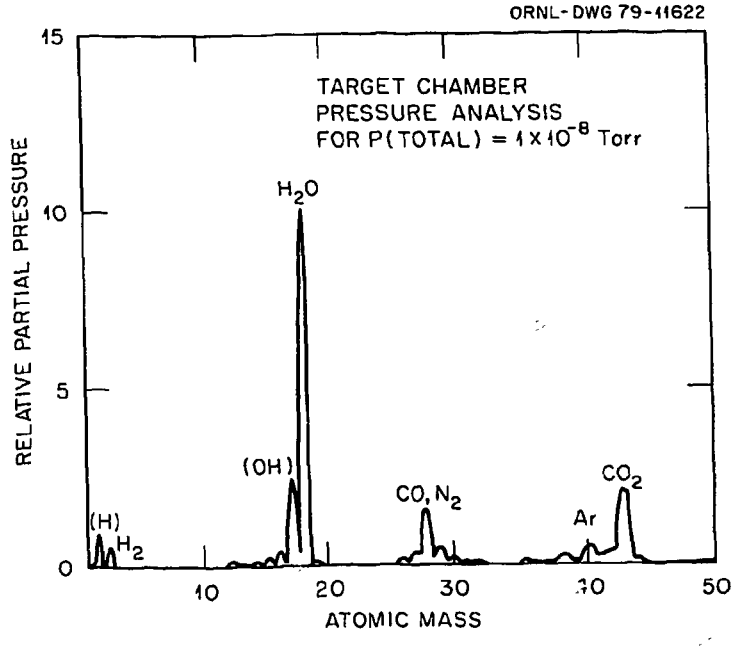

Fig. 4. Residual Gas Analyzer Spectrum of Target Chamber Atmosphere. Pressure: 1×10^{-8} torr = 1.3×10^{-6} Pa.

Electron Gun Heaters

Figure 5 gives a view of a target assembly with each of the six specimen holder-heater modules in progressive stages of assembly. The heaters are dispenser cathode triode-type Y646B electron gun assemblies.* It is necessary to mount them in plug-in sockets, as shown, because prolonged exposure to oxygen or water vapor is deleterious to the cathode emitting surface. Thus the guns are kept in a vacuum desiccator for any extended time that they are not in the damage chamber under vacuum. The support structures around the guns were machined from Macor† glass-ceramic

*Product of Eimac Division of Varian Associates, 1678 South Pioneer Road, Salt Lake City, Utah 84104.

†Product of Corning Glass Works, Corning, New York 14830.

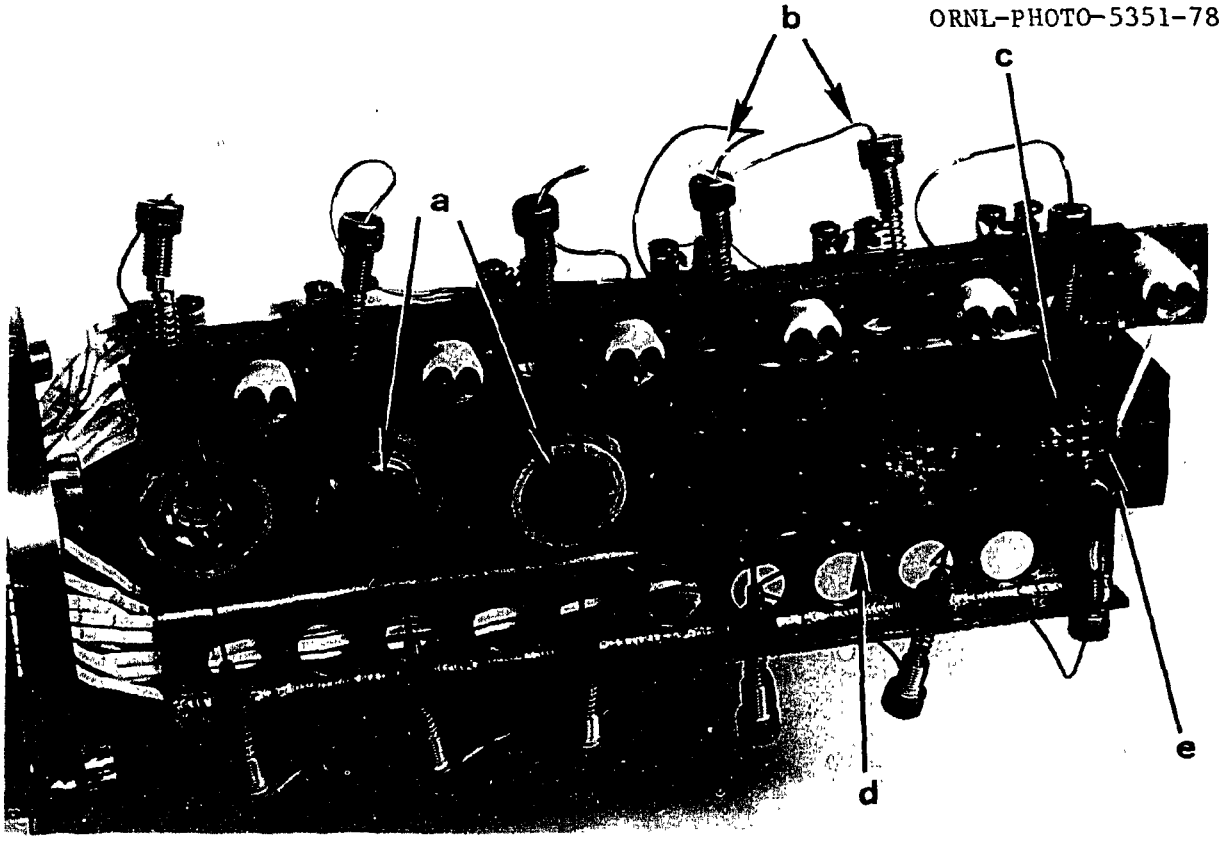

Fig. 5. Detailed View of the Target Assembly Showing the Six Heater Stations in Progressive Stages of Assembly from Left to Right, including: (a) Electron Guns, (b) Spring-Loaded Thermalizer Block Thermocouples, (c) Nine-Disk Specimen Holder, (d) Tantalum Heat Shield, and (e) Specimen Thermocouple.

material. The electron guns operate with the cathode drawing about 10 mA at 600 to 800 V negative to ground, while the specimen assembly at ground potential serves as the anode. Very sensitive control is afforded by varying the potential on the fine mesh grid from about 0 to −2 V with respect to the cathode. The cathode is heated by an internal filament, which requires about 1.5 to 2.0 A at 10 to 12 V. Since the grid and filament are operated relative to the negative cathode potential, their power supplies must be electrically isolated from ground via an isolation transformer and physically enclosed within a Plexiglas cage. Across this potential difference, control is accomplished by a pair of voltage-frequency optical isolation couplers.

Upon first startup of each gun after air exposure, a specific "conditioning" sequence is carried out (see Appendix) to reactivate the emitter surface. The time required to condition a gun can vary from less than 5 to more than 30 min, depending largely on the brevity of the previous air exposure.

Specimen Holders

The specimen holders mount directly in front of the electron gun heaters. There is a space of about 0.8 mm between the electron gun grid and the lower portion of the specimen holder (thermalizer block). The two parts of the specimen holder, the thermalizer block and the face plate (Fig. 6), clamp the specimens between them. In the back of the face plate are recesses to locate the specimens. The thermalizer block surface is ground flat and smooth to ensure good thermal contact with the specimens. Both specimen holder parts are fabricated from Kulite-112* machineable tungsten alloy to reduce problems of bonding with most specimen materials. A crushable ring of annealed 0.1- to 0.2-mm-thick platinum wire is placed between the face plate and the specimen front surface to accommodate small variations in specimen-to-specimen thickness so that none are loose in the holder. A clamping force of about 550 N (124 lb) is applied by a modified toggle-clamp tool to partially compress the platinum gaskets. The four

*Product of Kulite Tungsten Company, 1040 Hoyt Avenue, Ridgefield, New Jersey 07657.

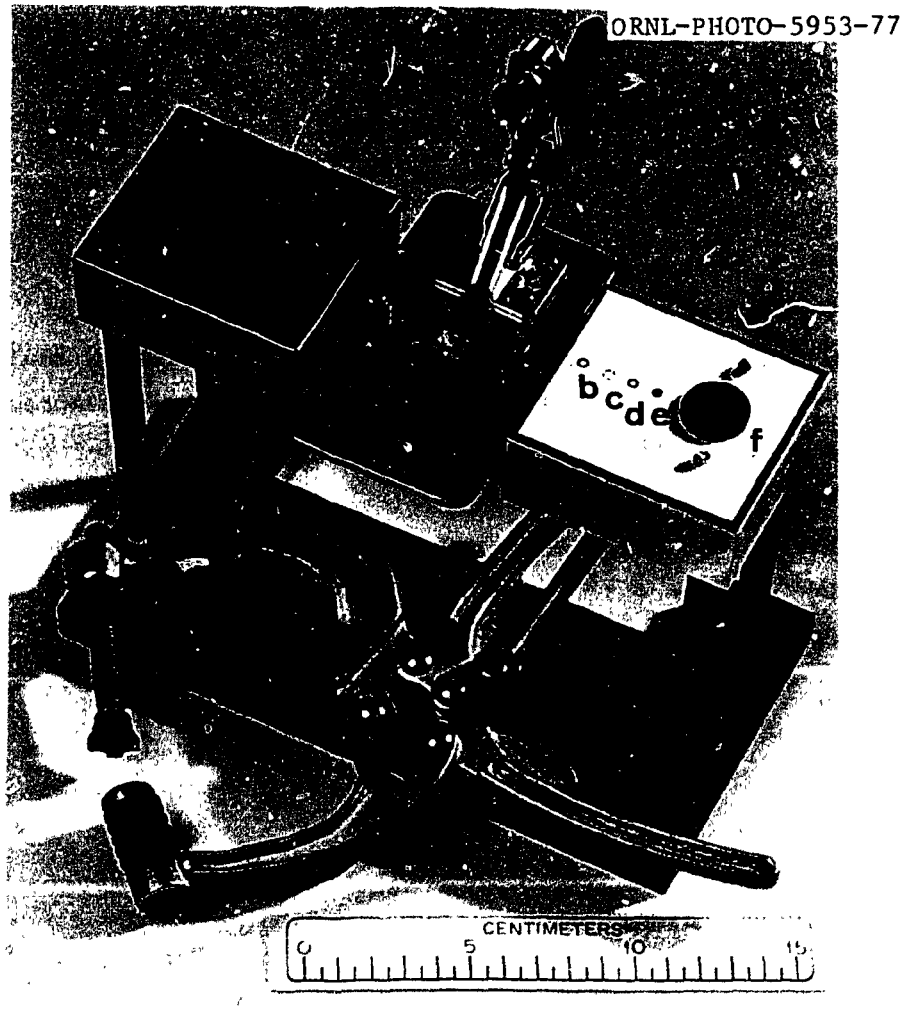

Fig. 6. Disassembled Specimen Holder in the Loading Jig. Components are: (a) face plate, (b) miniature bar mask, (c) platinum wire gasket, (d) optional 0.05-mm-thick oxidized stainless steel spacer, (e) specimen, and (f) thermalizer block.

2-56 socket-head screws that hold together the specimen holder are tightened only to maintain (rather than develop) the pressure. The components and fixture used in loading specimens are shown in Fig. 6. An alternative style of specimen holder has been made that has a 16.6-mm-diam by 3.6-mm-deep well; this can be used to mount specimens of other shapes or sizes.

Temperature Control

Temperature control begins with the signal from either of two 1-mm-diam sheathed Chromel-P-Alumel thermocouples, which are spring loaded into 6-mm-deep holes in the edge of each thermalizer block (these pairs of control thermocouples can be seen in Fig. 5). A third thermocouple output, available at each station for measuring the specimen surface temperature, uses 0.13-mm-diam Chromel-P-Alumel wires spot welded near the periphery of a dummy specimen. All these signals are led through a switching panel that permits any of the three to be used for control. Any one may also be displayed on a room-temperature-compensated digital indicator. Temperature is controlled by an L&N Speedomax H strip-chart recorder with current-adjusting-type (CAT) controller and adjustable-zero-adjustable-range (AZAR). The control signal is fed back (via optical isolation) to the electron gun grid potential. The result is control that is both rapid in response and stable. With an electron gun that has been "conditioned" since its last air exposure, a specimen holder starting at ambient temperature can be raised to and held at 700°C in less than 5 min, and any such irradiation temperature can be maintained within approximately ±1°C for hours. One specimen temperature is measured directly by the thermocouple, while up to eight others are measured relative to the first by an infrared pyrometer that can be sighted on each individual specimen.

Beam Control and Diagnostics

This apparatus is intended to facilitate the irradiation of large numbers of specimens. To do this we have chosen to cover a relatively large target area with a beam as uniform as possible. A number of techniques have been utilized to create such a beam, notably the devising of the unique split field lens by C. H. Johnson.[2] However, to be successful these techniques require thorough monitoring and analysis of the beam in the target region. Diagnostic equipment is traversed by the heavy-ion beam in the following order: a beam profile monitor, a movable assembly of nine miniature Faraday cups, a movable aperture mask, the target, and

finally a deep Faraday cup. The light-ion beam has its own beam profile monitor (located about 1 m ahead of the target plane) and its own deep Faraday cup.

Considering the heavy-ion beam line components in further detail, there is initially a pneumatically actuated gate valve followed by an 11.0- by 11.0-mm fixed square aperture, which defines the final beam that enters the damage chamber. Just after the aperture and located 150 mm ahead of the specimen plane is the beam profile monitor, a Physicon model MS-10. This device has a thin V-shaped vane, which we have shaped such that the blades sweep across the square section beam approximately parallel to the sides of the square. Oscillating at a rate of 11 Hz, the vane intercepts only about 3% of the beam flux. However, the edge-on vane geometry is such that secondary electron emission is enhanced by a factor of about 5, thereby effectively amplifying the current signals to about 15% of the true current. As shown in Fig. 7, the current signal is divided into roughly equal parts. One path is led directly to a current digitizer, whose output is recorded in scalers. The other branch is amplified to drive the signal from the scanner over long cables to oscilloscopes and a current meter in the console area. A signal averager is sometimes used to accumulate profiles during a run.

The information thus obtained is desirable because it is continuously obtained during a run. However, it must be calibrated against true measures of the beam uniformity and total intensity to be quantitatively useful. Both the current meter and the current loop to the digitizer are calibrated and checked before and after each run by withdrawing the target assembly to expose the heavy- and light-ion deep Faraday cups (DFCs). These cups have suitable dimensions of 200 mm in length by 30 mm in diameter to retain all secondary emission and thus give a true absolute measure of the beam intensity. The ratio of scaler counts from the vane current to the scaler counts of DFC current is the profilometer calibration. Typical calibration values vary only about 5% over a one-magnitude variation of incident beam current. However, it is well known that secondary electron current, such as that produced by the profilometer vanes, is a sensitive function of nonconducting films that deposit on a conducting surface. Drifts in the calibration have been attributed to this effect as

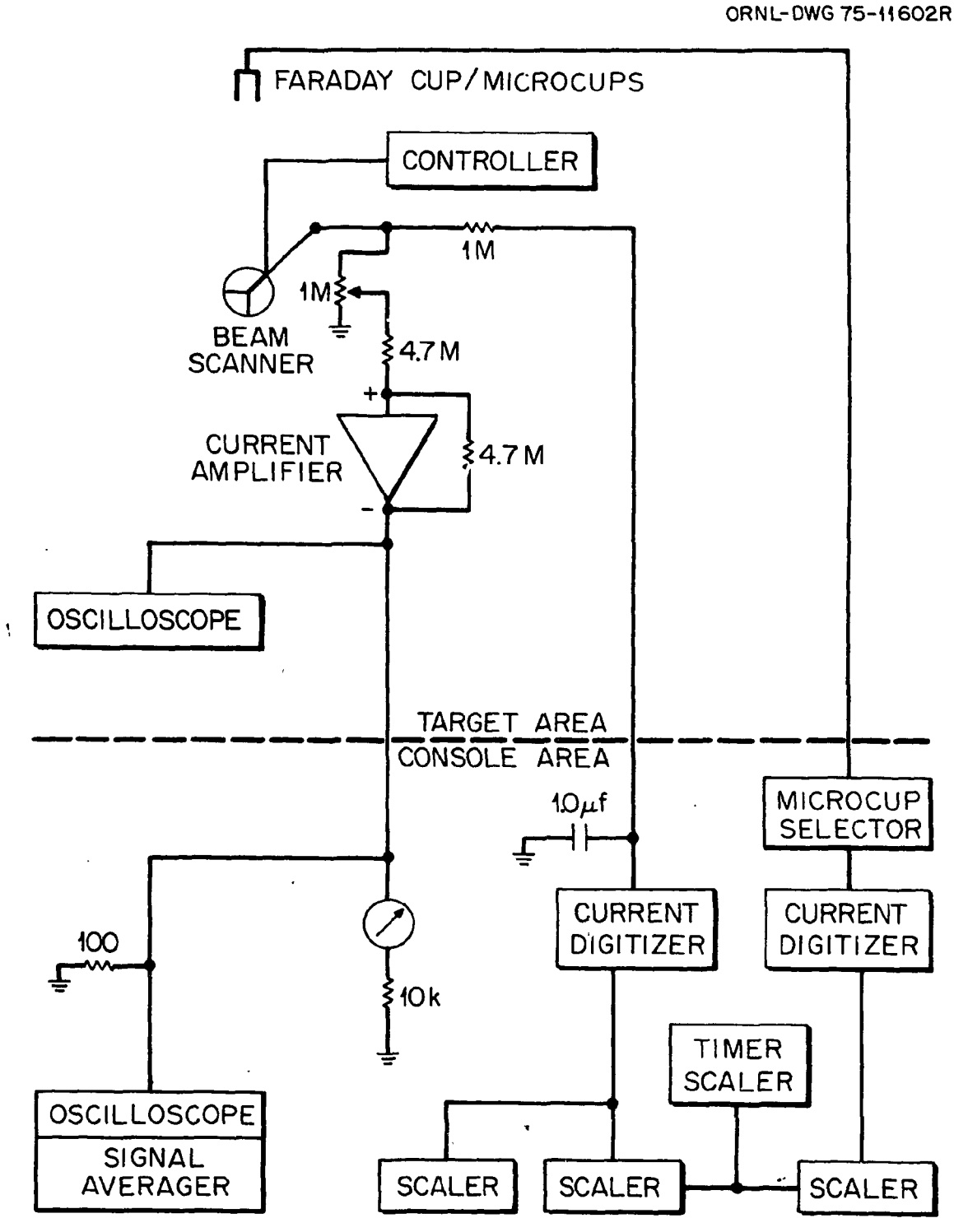

Fig. 7. Block Diagram of Electronics for the Beam Monitoring System. Signals originate at the beam scanner (continuously in service) or at the Faraday cup or microcups used at selected times. The current signals are then routed to measurement devices located at the Van de Graaff console area.

well as to temperature effects when the profilometer vanes have been isolated from beam bombardment for over an hour. With these periodic calibration checks, the value of the total ion flux incident upon the target is believed to be known within ±10%.

With regard to beam *uniformity* over the full target area, it can be shown that since the X-Y profilometer scan is a linear average of the actual beam current, a square-wave signal does not uniquely specify a uniform beam. Comparisons must be made between the shape of the profile as seen on the oscilloscope display and a microaperture analysis of the beam cross section at the target site. The latter can be accomplished by advancing a mask with a diagonal array of 1.0-mm-diam holes across the beam. When processed by a digital signal averager, the resulting signal from the DFC yields a true measure of beam uniformity against which the beam profile monitor output may be compared. However, this approach is rather time consuming and can only be carried out with the target removed from the ion beam. An array of nine miniature Faraday cups (Fig. 8) is the alternative now used to measure the beam current in the exact positions at which the specimens are located. These cups have an aperture

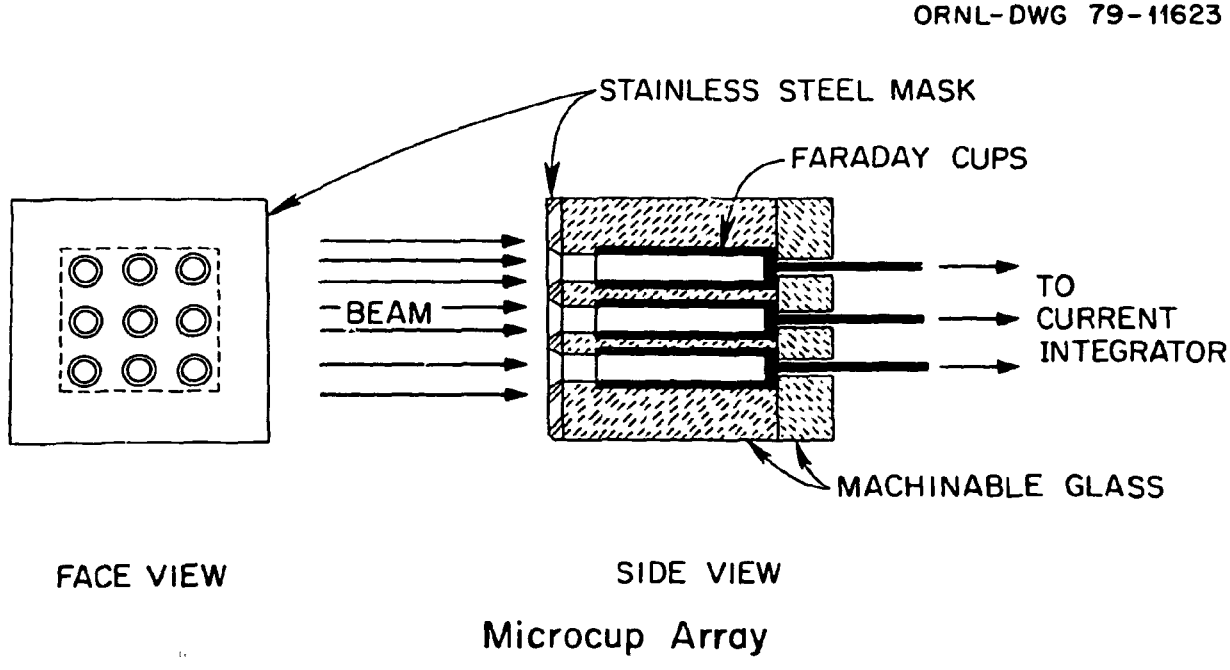

Fig. 8. Two Schematic Views of the Nine-Hole Miniature Faraday Cup Array Used to Assess the True Beam Intensity on Each Target Disk.

plate in front to collimate the beam and reduce secondary electron interference. They have a length-to-diameter ratio of 4.45 and an entrance diameter of 2.41 mm. The cups are symmetrically located in the beam on 3.43-mm centers. A pneumatically actuated bellows is used to insert the assembly in the beam directly in front of the target. The operation of this device and recording of its data shortly will be computer controlled.

The movable mask (with the diagonal array of holes) is frequently put to a different use. Its unperforated lower portion may be positioned to initially cover and then at chosen times expose progressive rows of specimens, thus yielding up to three different doses during a single bombardment of one three-by-three array of specimens.

Recently added to the damage facility is a capability for producing pulsed beams. A pivoting beam stop actuated by a solenoid has been installed in each beam line about 1 m ahead of the target region. The beam stops may be activated either individually or exactly simultaneously. The dual binary programmable timer allows a continuously variable range of pulsing parameters as rapid as 0.5 s. Both beam-on and beam-off periods can be independently adjusted. Experiments are under way to explore the influence of pulsed vs continuous beams with particular attention to pulse conditions representative of those that are expected in tokamak fusion reactors.

PERFORMANCE

The damage chamber has been in service for nearly three years. To date some 250 bombardments have been carried out involving irradiation temperatures ranging from 300 to 850°C and achieving damage levels as high as 600 dpa. Many of the experiments have utilized simultaneous helium injection. Some have even involved simultaneously implanting both deuterium and helium along with the heavy-ion damage ("triple beam" bombardment[3]) as a further advance in the simulation of fusion reactor conditions.

ACKNOWLEDGMENTS

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REFERENCES

1. M. B. Lewis, N. H. Packan, G. F. Wells, and R. A. Buhl, "Improved Techniques for Heavy-Ion Simulation of Neutron Radiation Damage," *Nucl. Instr. and Methods* 167: 233 (1980).
2. C. H. Johnson, "A Ring Lens for Focusing Ion Beams to Uniform Densities," *Nucl. Instrum. Methods* 127: 163 (1975).
3. K. Farrell, M. B. Lewis, and N. H. Packan, "Simultaneous Bombardment with Helium, Hydrogen, and Heavy Ions to Simulate Microstructural Damage from Fission or Fusion Neutrons," *Scr. Metall.* 12: 1121 (1978).

APPENDIX

CURRENT OPERATIONAL PROCEDURES (JANUARY 1980)*

I. Specimen Exchange Procedure

A. Loading Specimen Holders with Transmission Electron Microscope (TEM) Disk Specimens

1. Clean face plates and thermalizer blocks with alcohol.
2. Place face plate (item a in Fig. 6) beam-side down into the loading jig (Fig. 6), locating the plate's outer notch in a consistent direction (e.g., to the lower right).
3. (Optional) If individual 0.025-mm-thick (0.001-in.) bar masks are to be used to permit postbombardment step-height measurements, load them first in each recess with the bar oriented vertically on the side opposite the outer notch.
4. Next, load the crushable platinum wire rings (normally 0.125 mm thick), gently pushing them flat, if necessary, with the end of a rod of like diameter.
5. (Optional) To reduce adherence of the platinum wires to soft specimen materials or at high temperatures, one can next insert 0.050-mm-thick stainless steel washers that have been given an oxidized surface finish (item d in Fig. 6).
6. Load the specimens face down.
7. Load the specimen to whose face a thermocouple has been welded. In lieu of the bar mask and washer of steps 3 and 5 above, one can use 0.20-mm-thick platinum gasket wire.
8. Carefully place the thermalizer block, smooth-side down, over the specimens without dislodging them (this is invariably difficult with the thermocouple specimen). The thermocouple holes in the thermalizer block must be oriented properly with respect to the face plate, that is, for face plate inverted with its outer notch at a 4-o'clock position, as in step 2 above, thermalizer block edge holes must be at 7- and 1-o'clock positions.

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*Terms (other than initialisms) in all capital letters refer to labels on equipment presented schematically in Fig. 1A at the end of the text.

9. Apply the hold-down clamp, which locks into position.
10. Removing the knurled index pin, invert the pivoting table. Two of the 2-56 by 1/8-in. socket head screws can be inserted through the face plate and lightly tightened.
11. Again invert the pivoting table, release the hold-down clamp, remove the specimen holder, and install the other two screws.
12. Place the specimen holder between the jaws of the Destaco 463-S clamping plier such that the thermocouple lead and the four socket screw heads align with the cutouts in one anvil. Clamp until the plier locks to apply a preset pressure to the holder for the purpose of deforming the platinum wire gaskets enough to retain all the specimens, even if they differ slightly in thickness. The plier develops about 400 N (90 lb) of force per 3 mm (1/8 in.) of spring compression.
13. While the holder is so clamped, tighten the four screws to retain this compression. Then the specimen holder can be released.
14. Thread the specimen thermocouple wires through a segment about 20 mm (3/4 in.) of double-hole ceramic insulator. The specimen holder is now ready to load.

B. Breaking Vacuum and Unloading a Target Assembly (TA)

1. Vent excess pressure, if any, in the sorption pumps by removing and replacing the small rubber stoppers. Then fill both styrofoam Dewars surrounding the pumps as well as the glass Dewar on the backfill cold trap. For good performance the pumps should have 1 to 2 h to prechill before being used.
2. Check the nitrogen gas cylinder (behind the instrument racks) to be sure gas is available for backfill.
3. Isolate the damage chamber by closing both beam line gate valves (switches at 16 in Fig. 1A, which is a detailed illustration of the damage chamber controls). Also, close the gate valve to the light-ion backscattering chamber situated downstream from the damage chamber. Turn off the ionization gage filament (at 5 in Fig. 1A), and check that the residual gas analyzer is off (power switch OFF).

4. Disconnect the four electrical cables to the TA. Disconnect the water lines, the supply line (fittings painted *red*) first followed by the drain line (*blue*).
5. Crank the TA all the way into the chamber.
6. Close the cryopump gate valve (item 20 in Fig. 1A). Then open the roughing valve R3.
7. Open the needle valve (green knob) to admit nitrogen backfill gas a little beyond zero on the gage. Then close needle valve and valve R3.
8. Unscrew all the TA sealing bolts so that the two semicircular tool steel "torque rings" (used in lieu of nuts) can be removed.
9. Gently pry apart the TA flange from the chamber body, using a screwdriver in the gap at the top (one is separating the copper gasket seal, which often sticks a bit).
10. Crank the TA fully out of the chamber.
11. Couple the crane trolley to the lifting handle of the TA. Then remove the expandable (cam-action) locating dowel and the four anchor bolts that secure the TA to the movable stage.
12. Lift the TA with the crane just sufficiently to clear the fixed end of the stage [about 3 mm (1/8 in.)]. To operate the lift, set the direction toggle switch to UP and activate the power switch for several brief pulses (the mechanism coasts considerably between them).
13. Roll the TA back to the crane post, being very careful not to let the handle slide out of the trolley. Holding the TA, release the spring-loaded latch and allow the TA to pivot downward 90°, bringing the specimen holders upright.
14. Remove all the specimen holder retaining screws (with ceramic standoffs). It is mandatory to wear gloves for this and the following steps to keep the chamber's internal parts clean.
15. Unscrew and withdraw the sheathed thermocouples that go into the edges of the thermalizer blocks.
16. Disconnect the specimen thermocouple wires from their terminal posts (press down the spring-loaded posts to release).

17. Lift out the specimen holders and place them in marked petri dishes.

C. Reloading the Target Assembly

1. Place the new specimen holders in position with the notch in the rim of each face plate aligning with a small index pin.
2. Reinsert fully the sheathed thermocouples, getting the knurled knobs finger tight. Check that the thermocouples "bottom out" in the thermalizer blocks by retracting them slightly against spring pressure and releasing.
3. Replace the specimen holder retaining screws (with ceramic standoffs) and tighten them just snug.
4. Hook up the specimen thermocouples. Use a small magnet to identify the ferromagnetic wire (Alumel) and connect it to the larger (and also ferromagnetic) terminal post.
5. Visual and Electrical Tests
   (a) Visually check that the specimen thermocouple wires do not touch each other or any parts of the specimen-heater assembly, including the face plate.
   (b) Pivot the TA back up to the vertical position where it latches. On the back of each station one can see three terminal screws, which we can denote from top to bottom as F_1 and F_2 (for the filament) and G (for the grid). With a VOM meter on the "ohms times 10" scale:
      (1) Check continuity of filament (measure 0 between F_1 and F_2, the top and middle terminals).
      (2) Check that F_1 or F_2 to ground (e.g., the bar) is open (practically infinite resistance).
      (3) Check that G to ground is open.
      (4) Check that G to F_1 or F_2 is open.
   (c) (Optional) One can check the correct functioning of the thermocouples by reconnecting the TA cables and heating briefly each specimen position with a heat gun, looking for a response on the specimen thermocouple readout.
6. Fit a new 171-mm-OD (6.75-in.) copper gasket onto the TA sealing flange, where sheet metal retainers will support it in place.

7. Roll the TA into the chamber, keeping it pointed slightly rearward (away from beam) to avoid tangling with the mask drive components. Lower the TA with the crane until it is solidly in contact with the movable stage block. Insert but leave loose the hold-down bolts. Insert the expandable dowel pin on the downstream (east) side and align the TA base plate with a scribe mark close to the edge of the movable stage block. Tighten the four hold-down bolts.
8. Place shims to center the TA flange around the TA central tube (without these the flange will be low and will make an off-center impression in the copper gasket, possibly resulting in a faulty seal). Crank the assembly into the chamber until the sealing flanges nearly touch. Push through the bolts and screw them into the semicircular torque rings. Tighten the bolts thoroughly, going around several times.
9. Pumpdown
   (a) Open valve R1 partially, not fully. Then open valve R3 similarly. Regulate valve R1 to get a steady evacuation down to about 70 Pa (500 μm), as indicated on the thermocouple gage nearby. This throttled evacuation keeps a steady stream of air, including its natural helium component, flowing into the sorption pump and minimizes any counterflow of helium out of the pump. After about 70 Pa (500 μm), close off pump 1 and open valve R2 to pump 2.
   (b) One can monitor the evacuation with the Varian dual-range ion gage control (5 in Fig. 1A) set to the Millitor gage (yellow numbers). When the vacuum is about 0.4 Pa (3 x 10^{-3} torr), close off the roughing system (valve R3) and immediately open the cryopump gate valve. The vacuum should fall at once into the 10^{-3} Pa (10^{-5} torr) range and continue dropping with time.
10. Connect all cables (if not done already) to the chamber, being sure to match any colors on plug and socket. With respect to the cooling water, connect <u>first</u> the <u>drain</u> hose (blue fittings) and delay connecting the water supply ("red") line until after the next step, if elected.

11. (Optional) Bakeout. Plug in the Electromax III Bakeout Controller (plug and power are found in the rear of its rack). Turn on the Bakeout Power Supply (33 in Fig. 1A), located at the bottom of the same rack. The system is presently preset to maintain a target assembly bar temperature about 100°C. The bar temperature may be read out on the Pyrotest 9B potentiometer, while the temperature of any specimen can be found from the digital thermocouple display (29 in Fig. 1A).

II. Operating Procedure for Electron Gun Heaters

Vacuum in target chamber must be below 7×10^{-4} Pa (5×10^{-6} torr) before heating filaments.

A. Preparations

1. Turn on the dual-range ionization gage control (5 in Fig. 1A). Vacuum must be below 7×10^{-4} Pa (5×10^{-6} torr) or damage to the electron guns may result. (Refer to the instruction manual for dual ionization control for any questions as to its operation.)
2. Turn on cooling fans (4 in Fig. 1A).
3. Turn on high-voltage translation box (10 in Fig. 9).
4. Turn on bombard controller-recorder switch (3 in Fig. 9).
5. The "preheat" system is normally used to condition a second electron gun to save time, while the "bombard" system is also engaged in gun conditioning. If the preheat section is to be used, turn on preheat controller-recorder switch (2 in Fig. 1A).
6. Press red ∇ button on bombard and/or preheat current-adjusting-type (CAT) controllers and monitor their deviation meters (31 in Fig. 1A). When deviation meters read about 0, go on to next step.
7. Make sure the filament power supply (bombard and preheat) voltage control knobs are turned fully counterclockwise to the "off" position (11 and 27 in Fig. 1A).
8. Make sure the high-voltage and thermocouple cables are attached to the damage chamber. The high-voltage cables are interlocked so that the high voltage cannot be turned on unless the cables are attached.

9. Turn on the high-voltage control circuit breaker (24 in Fig. 1A) and press reset button (23 in Fig. 1A).
10. Turn on Kepco high-voltage power supply ac switch (8 in Fig. 1A).
11. Select desired bombard station by setting the rotary six-position switch on switching panel (6 in Fig. 1A). (The BOMBARDMENT and PREHEAT CONTROL switches should never be set to the same station. This will parallel the thermocouples and high-voltage control circuits. Crossing stations should only take place with the filament voltages at zero and no plate voltage.) The three-position thermocouple switch (7 in Fig. 1A) directly below the station-selector switch specifies the thermocouple to be used for direct control of the electron gun: A and B are the thermocouples inserted into the thermalizer block, while C is the specimen front-surface thermocouple.

B. Operation

1. Increase filament voltage (11 in Fig. 1A) from 0 to 10 V very slowly (about 1 min). Keep the filament current below 2 A, preferably below 1.75 A, and do not let the pressure exceed 7×10^{-4} Pa (5×10^{-6} torr) and preferably less than 3×10^{-4} Pa (2×10^{-6} torr). Allow 20 to 30 s for filaments to reach maximum temperature.
2. Select the 0 to 200-V-dc range for the plate voltage on Kepco high-voltage power supply (25 in Fig. 1A), and turn on the dc power switch (9 in Fig. 1A).
3. Drop grid voltage to "0" by pressing yellow Δ button on the CAT unit (32 in Fig. 1A), monitoring the plate current on the high-voltage supply's dc milliammeter (13 in Fig. 1A for grid voltage and 12 in Fig. 1A for plate current). [The Kepco high-voltage power supply has a current limiter control (26 in Fig. 1A) that regulates the maximum current from the power supply.]

   If the guns are not poisoned they will emit, and a plate current will be indicated on the milliammeter. Such initial

functioning is the exception rather than the rule, but if so go on to step 5.

4. Guns failing to start in step 3 have been poisoned and will have to be "conditioned." This process reactivates the cathode by exposing fresh barium at the surface.* The cathode surface should be heated to 1240°C with about 200 V dc plate voltage and no voltage on the grid. Unfortunately, the cathode temperature can only be estimated by observing the power drawn from the filament power supply. Usually 12 V at 1.8 to 2.0 A (about 20 W) will suffice. Operating the filaments to produce 1240°C shortens the lifetime of the filament, and one should therefore try to minimize the time that they are operated at this temperature.
5. As soon as emission takes place (over 10 mA plate current for about 2 to 5 min), reduce gradually the filament voltage. However, if emission drops below 10 mA plate current, increase the filament voltage and repeat the process until emission remains above 10 mA plate current at no more than 10 V filament voltage. The longer the guns operate the better the emission should get. The filament voltage should be gradually reduced to the lowest value that will hold the temperature that one has set on the controller (next step), usually between 7 and 10 V. At the same time, increase the plate voltage selector (25 in Fig. 1A) to a higher range, eventually 400 to 600 or 600 to 800. Take it only one range at a time, looking out for a sharp drop in plate current (if you get this, fall back to the last plate voltage setting). One must avoid operating the filaments for long periods at more than 10 V.
6. Bring the temperature to that desired by manually controlling the bombard CAT controller (depress the yellow Δ button), which regulates the grid voltage, while monitoring the plate

*See report by A. Sandor[1] for a very good description of the process of reactivation.

current to keep it below 50 mA. (The current-limit control circuit in the Kepco high-voltage supply will reduce the voltage if a preset current limit of about 55 mA is exceeded. It is to gain more leeway from this current limit that operation of the plate voltage selector at a higher range — step 5 above — is desirable.)

7. In some cases the grid may contact the cathode in the electron gun structure. If it does the grid voltage will go to zero, and the gun will conduct without control. This condition is diagnosed by observing the grid voltage indicator (13 in Fig. 1A). If the meter has a constant value a little above 0 V, even while the temperature control is raised or lowered, the grid is touching the cathode. It is caused by the thermal expansion of the cathode support structure and will usually go away if you lower the filament voltage. This is possible if the gun has already been reactivated, but if not cathode conditioning should continue with the short. At the end lower the filament voltage to do away with it. If slumping takes place (emission drops off drastically), raise the filament voltage and operate with the cathode-to-grid short for a brief time and then try lowering the filament voltage again.
8. When the temperature has reached the desired level on the bombard controller-recorder, position the orange set point (30 in Fig. 1A) over the red temperature indicator, and press the AUTO button. The temperature should hold at the set point. If it keeps rising, the cathode could be shorted to the grid (see above). If it falls off, the filament voltage should be raised to improve emission. Once the set-point temperature is stable, minimize the filament voltage as recommended in step 5.

III. Procedure for Conducting a Bombardment

A. Prebombardment Steps

1. Specimens are assumed to be holding at the desired irradiation temperature (reached per Sect. II above) and at a

vacuum better than about 7×10^{-5} Pa (5×10^{-7} torr). A page in the logbook has been ruled into columns to accept data. The temperature is indicated on the digital thermometer (29 in Fig. 1A). Selection of the station to be read is determined by the six-position TEMPERATURE READOUT switch (21 in Fig. 1A). The choice of thermocouple in that station is no longer made by the "thermocouple" switch below the station selector but rather by the small rotary switch denoted AUTO/SCAN in the upper right corner of the digital thermometer. Positions 0 and 1 select thermalizer block thermocouples A and B, respectively, and position 2 designates the specimen thermocouple.

2. The TA is still fully withdrawn from the chamber so that the control room can correlate the outputs of the beam profile monitor and the nine-hole miniature Faraday cup array with that of the deep faraday cup (DFC).
3. When the control room's beam diagnostics are completed, block the beam(s) by closing both pneumatic gate valves (switches labeled VALVE SELECT IN MANUAL MODE, 16 in Fig. 1A).
4. Advance the TA into the chamber such that the desired specimen assembly is in bombardment position.
5. Calibration of the infrared pyrometer
   (a) Take an initial temperature reading from each specimen using the infrared pyrometer, including the dummy specimen with thermocouple if possible. Note that the pyrometer optics show a left-right and up-down inversion of the specimen locations. A label attached to the pyrometer pedestal helps interpret the pyrometer view. A Unidex digital X-Y positioner (28 in Fig. 1A) is used to move the pyrometer head to each discrete specimen position. Movement to a given location is accomplished by selecting first the direction (X/Y button) and the sense (+/—) of motion and then carrying out the operation by pushing the EXECUTE button.

(b) Average the readings and compare with the value being indicated by the specimen thermocouple. (It may be necessary to be selective in arriving at an average reading, ignoring a specimen that differs widely from its neighbors, which could result from insecure mounting.)

(c) Aim the pyrometer on the specimen whose initial temperature reading most closely matched the average for all specimens.

(d) Unlock the pyrometer emissivity adjustment knob and readjust it such that the indicated temperature becomes equal to the thermocouple reading.*

(e) Alternatively, for diverse specimen materials in the same bombardment, one may have to perform the above emissivity adjustment solely on the dummy specimen. Then one must assume that the pyrometer is viewing a sufficient area of representative surface unperturbed by the thermocouple bead or wires.

6. Final adjustments to the temperature controller can now be made, taking into account expected beam heating. The control room can furnish a beam current value. The magnitude of the beam heating effect depends on specimen temperature as well as beam current. It is mainly estimated from prior experience and in any case the controller can be readjusted after the run begins.

7. If any rows of specimens are to be progressively uncovered during the run to obtain several dose levels, cover the appropriate rows now by moving in the mask, using the infrared pyrometer optics for final alignment. The mask is positioned by first setting the mode switch (1 in Fig. 1A) to MANUAL BYPASS, then using the hand-held control box to drive in the mask. Resetting the mode switch to AUTO will

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*The number indicated on the "emissivity" control is not strictly an emissivity because the original control was replaced by a ten-turn potentiometer for much greater precision in setting. A calibration curve that relates the present resistance value to the original factory-calibrated emissivity function is given in Fig. 2A.

then permit automatic withdrawal of the mask at preselected dose levels (see below).

8. Reset the three Ortec 771 timer-counters (14 in Fig. 1A), which will continually monitor the accumulated dose based upon the beam profile monitor signal.
9. After a countdown over the intercom to the control room, unblock the beam(s) to commence the run (i.e., simultaneously throw both VALVE SELECT IN MANUAL MODE switches to OPEN).

B. During the Run

1. Values are normally recorded every half hour for:
   (a) temperature of each specimen, measured by pyrometer;
   (b) temperature of the dummy specimen, measured by its thermocouple;
   (c) number of counts accumulated on the counters (Ortec 771); and
   (d) vacuum level in the chamber.
2. Shortly after the run begins, the control room will advise the number of counts on the counter corresponding to the desired dose level(s). The calculation results from a correlation between the DFC and beam profile monitor signals measured just before the run. Set the final end-of-run count projection into the right hand Ortec 771 timer-counter, the count for the first mask withdrawal (to expose the top row of specimens) into the middle timer-counter, and the count for the second mask movement into the leftmost timer-counter.
3. To now establish automatic mask movement and shutdown,
   (a) set both VALVE SELECT IN AUTO MODE switches (17 in Fig. 1A) to YES,
   (b) set top MODE switch (15 in Fig. 1A) to AUTO,
   (c) set the PRESET ENABLE/DISABLE switch on each Ortec 771 timer-counter to ENABLE, and
   (d) set the ELECTRON GUN SHUTOFF switch (18 in Fig. 1A) to AUTO, if desired. This option is used if it is possible that the damage chamber may be unattended at the end of the run.

C. After the Run

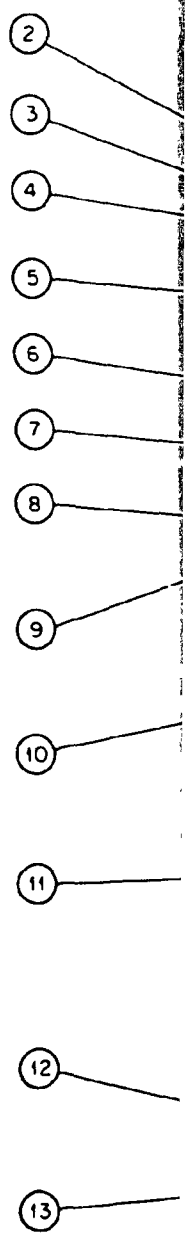
1. Upon reaching the intended dose (counts) the beam is automatically blocked by the pneumatically activated gate valve(s). The specimen heater should be cut off by depressing the red ∇ button on the recorder-controller and turning the electron gun's filament voltage knob fully counterclockwise (11 in Fig. 1A). The automatic shutdown mode (18 in Fig. 1A) may instead have been selected, in which case power to the electron gun was terminated when the counter reached a preset value. However, as soon as practicable thereafter, the above steps of depressing the recorder's red ∇ button and turning the filament voltage knob fully off should still be done now.
2. If removal of specimens from the chamber is now desired, the specimen assembly should be allowed to cool down to at least 50°C or lower, as indicated by the specimen surface thermocouple. The use of the cooling water loop, which ordinarily was already functioning during the run, hastens the cooldown markedly, as is evident in the example shown in Fig. 3A.

D. Overnight Shutdown Procedure

1. Be sure the bombard filament voltage knob (11 in Fig. 1A) is fully off.
2. Turn off BOMBARD CONTROLLER switch (3 in Fig. 1A).
3. Turn off high-voltage translation box (10 in Fig. 1A).
4. Turn off Kepco high-voltage power supply ac and dc switches (8 and 9 in Fig. 1A).
5. Push STOP button (22 in Fig. 1A).
6. Turn off the rack cooling FAN (4 in Fig. 1A).
7. Turn off the illuminator that shines into the chamber through bottom viewing port.

Fig. 1A. Controls for the Electron Gun Heaters and Vacuum System (Left Panel) and for the Temperature Readout and Control (Right Panel).

1. Mask Drive Mode: Auto/Manual
2. Preheat Controller-Recorder Power Switch
3. Bombard Controller-Recorder Power Switch
4. Cooling Fans for Rack Components
5. Ionization Gage Control
6. Station Selector, Bombard Heating System
7. Thermocouple Selector to Control Bombard Heating
8. Electron Gun Power Supply, ac Switch
9. Electron Gun Power Supply, dc Switch
10. High-Voltage Translation Box On/Off Switch
11. Filament Voltage Control (Bombard)
12. Plate Current Meter (Bombard)
13. Grid Voltage Meter (Bombard)
14. Timer-Counters to Monitor Accumulated Dose
15. Gate Valve and Mask Withdrawal Operation: Auto/Manual
16. Gate Valve Selectors in Manual Mode: Open/Close
17. Gate Valve Selectors in Auto Mode: Yes/No
18. Electron Gun Shutoff at End of Run: Auto/Manual
19. Nine-Hole Faraday Cup: In/Out
20. Valve Control, Cryopump on Damage Chamber
21. Station Selector, Temperature Readout System
22. STOP Button
23. High-Voltage Reset Button
24. High-Voltage Circuit Breaker-Switch
25. Plate Voltage Range Selector
26. Electron Gun Current Limiter Control
27. Filament Voltage Control (Preheat)
28. Infrared Pyrometer Digital Positioner Control
29. Temperature Readout
30. Controller-Recorder (Bombard)
31. Controller Deviation Meter
32. Controller Temperature-Increase Button
33. Bakeout Power Supply



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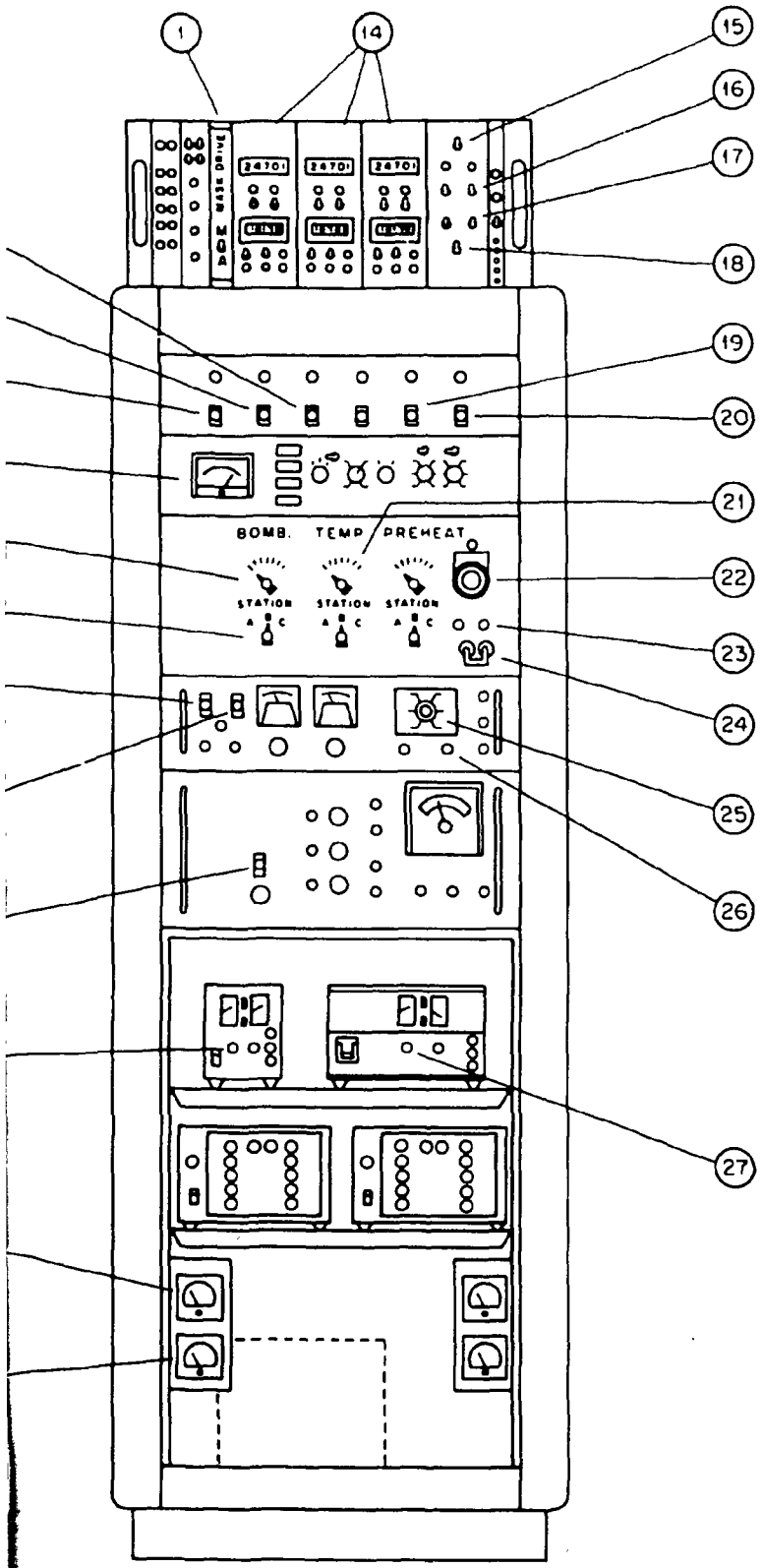

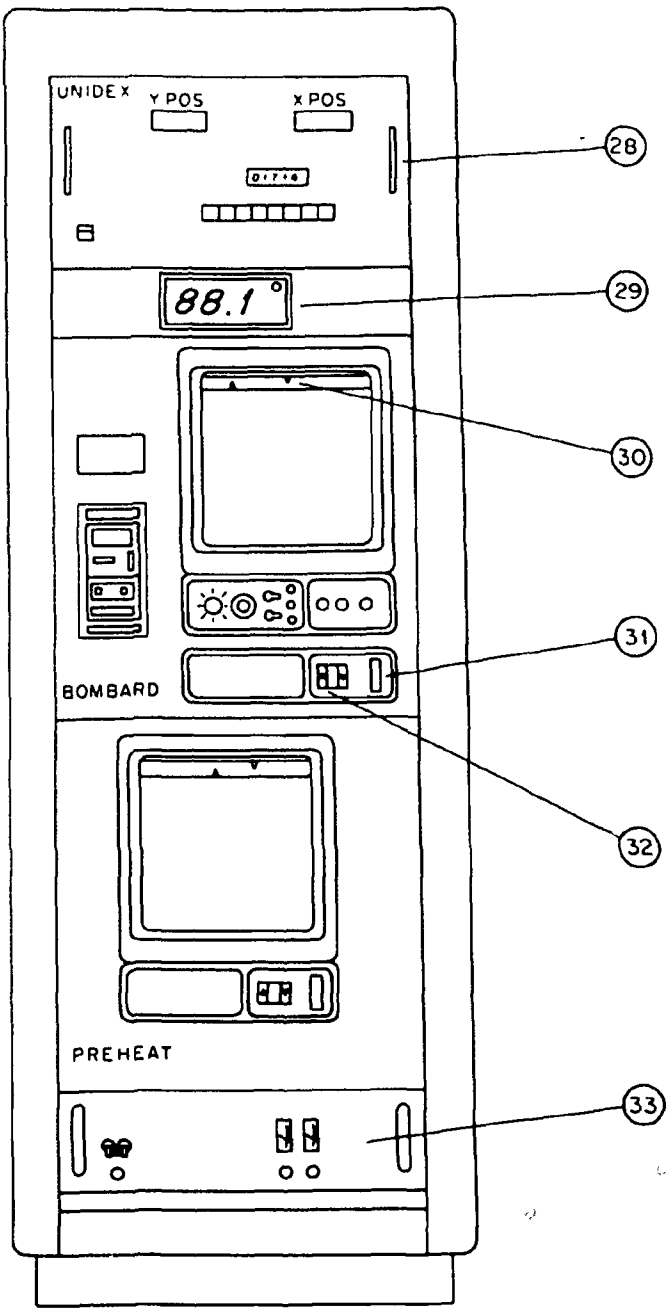

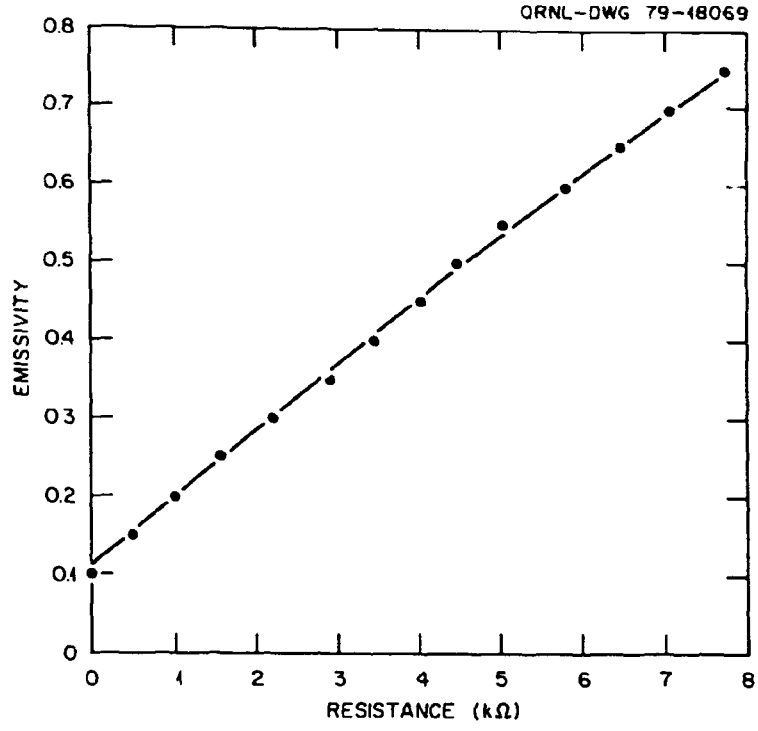

Fig. 2A. Calibration Curve for the Raytek SL 300 SC Infrared Pyrometer, Relating Resistance Value of the ORNL-Installed Ten-Turn Potentiometer to the Original Factory-Calibrated Emissivity Function.

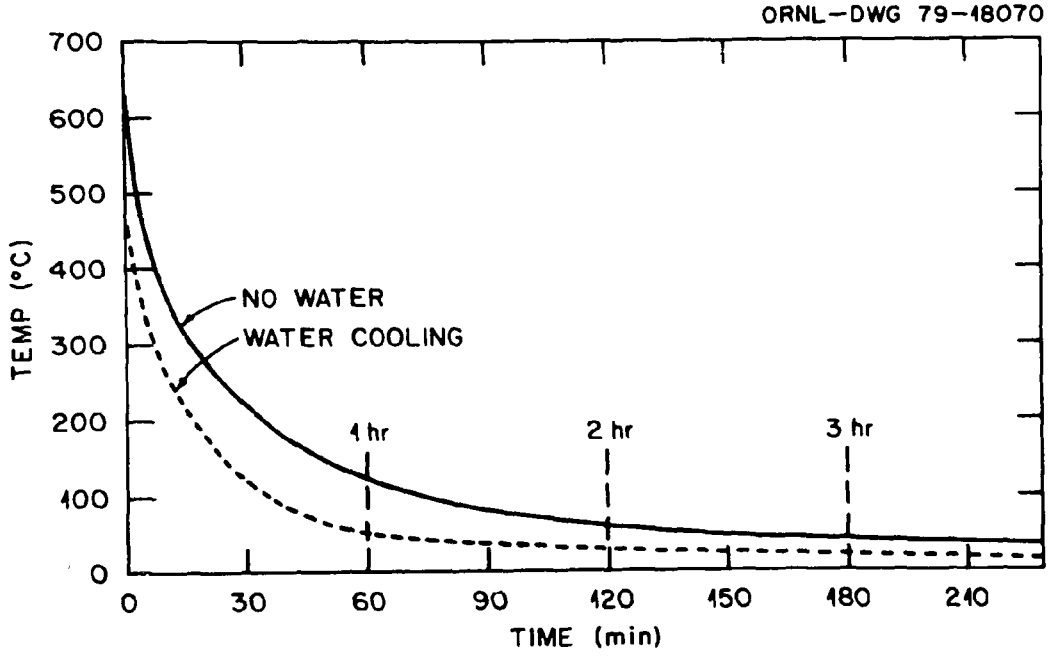

Fig. 3A. Time Required for a Specimen in Station 1 at the End of the Target Assembly to Cool from 700°C Either With or Without the Use of Cooling Water. Holding time at 700°C before cooldown was 75 min.

REFERENCE

1. A. Sandor, "Activation Process of Impregnated Dispenser Cathode Viewed in the Large-Screen Emission Microscope," *J. Electron. Control* 13: 401 (1962).