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THERMAL CYCLING EQUIPMENT

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R. M. Mayfield and S. T. Zegler

INTRODUCTION

During the past several years the Metallurgy Division at Argonne National Laboratory has been engaged in an extensive study of the effects of thermal cycling upon the dimensional stability of uranium. In the early stages of this project it became evident that a detailed study of all the variables involved in the process would be extremely time consuming, unless completely automatic cycling equipment were built that could operate reliably on a twenty-four-hour basis. To this end, it was proposed to build three cycling units at Argonne as follows: (1) an induction cycling unit, in which the specimens are enclosed in a NaK-filled bomb and the whole assembly heated by a high-frequency coil; (2) a vertical tube cycling unit (Woosher), in which the specimens are transferred between the centers of two NaK-filled tanks held at different temperatures and; (3) a low-temperature cycling unit, in which the specimens are enclosed in a NaK-filled bomb and transferred between two oil tanks at different temperatures.

The three proposed cycling units have been built and tested in regard to (1) temperature range attainable, (2) rate of cycling, (3) reliability of operation, and (4) intercomparison of results obtained. This report contains a detailed description of each unit and a brief discussion of some comparative test results in accordance with the above outline.

References are given for the detailed ANL engineering drawings for the three units.

INDUCTION CYCLING UNIT

Design and Operation

A general view of the Induction Cycling Unit is shown in Figure 1. The power source is an Ecco high-frequency spark gap converter, with a rated input of 20 KVA. A multiturn work coil 3-1/4 in. O.D. by 10 in. long having five turns of 3/16 in. diameter copper tubing per inch, has been used in connection with the converter. The work piece which this coil has suitably served to heat in cycling tests is a stainless steel container, 2-3/4 in. O.D. by 16 in. over-all length, as shown in Figure 1. Container size can, of course, be varied but must be held within a size dictated by coil design for efficient heating. A recirculating water system of fifty-gallon capacity is used to supply cooling water to the work coil and the Ecco converter.

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Specimens to be given cyclic thermal treatment are placed in the stainless steel container (bomb) as illustrated in Figure 2. In the vertical position, the rods, normally 0.364 in. in diameter by 2 in. long, are not restrained, except by virtue of their own weight, and are free to elongate. A clearance of 0.020 in. between the rods and the specimen racks permits unhindered cross-sectional changes. As many as twenty-four specimens have been cycled at one time using this arrangement. Present design of specimen racks allows for thirty-six such specimens in four inches of vertical length.

A vacuum-tight seal is drawn on the container by means of the metallic ring seal between the cover and the container flange. A Cenco-Megavac mechanical pump has been used to obtain vacuums of the order of six microns as measured with a thermocouple gauge. After evacuating, the container is filled with purified NaK alloy to a level at least two inches above the specimens. Dry argon is then introduced above the NaK alloy to a pressure of about one atmosphere. An expansion chamber is coupled to the entry of the specimen container, as shown in Figure 1, as a safety measure to compensate for increased pressure at elevated temperatures.

Temperature is measured by means of thermocouples located inside the well at the center of the container, adjacent to the specimens. The upper and lower temperatures of the thermal cycle are controlled by two preset microswitches arranged on the indicating mechanism of the recorder. The temperature indicator, in effect, acts as a start and stop switch to the converter. A schematic wiring diagram of the electrical circuits is shown in Figure 3. A Wheelco Limitrol, connected to a second thermocouple located in the same well, is used to prevent possible overheating. When the container and specimens reach the desired upper temperature of the heating cycle, the microswitch arrangement on the recorder activates a relay which opens the "converter to coil" circuit and closes the fan circuit. The container is then air cooled by the fans to the desired lower temperature. When this temperature is reached, the fan circuit is opened and the coil circuit energized.

After the desired number of cycles has been completed, the container is allowed to cool to room temperature. Specimen removal is accomplished by partially evacuating the container and introducing mineral oil to cover the NaK alloy. (This is done to reduce the safety hazard involved in the handling of the NaK.) After the cover has been removed from the container, the specimens are withdrawn and washed in butyl alcohol. The mineral oil and NaK are discarded.

The detailed ANL engineering drawing numbers for this unit are given in the appendix.

Temperature Range Obtainable

The maximum temperature obtainable in this type of heating is primarily a function of the load, frequency, and coil design. In cycling tests to date, however, the maximum temperature has been governed by the use of NaK eutectic alloy, which has a boiling point of 780°C . Tests thus far have consequently been restricted to temperatures below 700°C . The lowest temperature of the cycle is room temperature.

The minimum temperature range of cycling has not been definitely established. With the described coil and container designs, uniform heating and cooling has been found feasible within a range of 50°C . With modified designs so as to control heating and cooling rates it appears cycling over somewhat smaller ranges is possible.

Rate of Cycling

The heating rate, with a given load, is a function of the input power to the work coil and the coil design (number of turns). A lower or higher input power can be preset on the converter, but once set there is no arrangement whereby it may be varied during operation.

Cooling rate is also relatively inflexible, since it depends largely on the upper and lower temperatures and the load, influenced, of course, by the convection of air from the fans. Water cooling, to vary the rate, is not deemed advisable because of the ever-present hazards from the NaK alloy.

With fast rates of heating it has been found that large thermal gradients occur in the vertical column of NaK. By lowering the rates, however, uniform temperatures in an eight-inch column have been realized. The temperature at the center of the specimens under such conditions is within plus or minus 5°C of the temperature recorded in the thermocouple well. Similar uniformity of temperature has been obtained on cooling.

In a typical thermal cycle, between 100°C and 550°C (Figure 8), the total heating time is six to eight minutes and the total cooling time twenty to twenty-five minutes. Generally, an average of fifty-five such cycles is obtained in a twenty-four-hour period.

Reliability of Operation

The Induction Cycler has now operated continuously for over three years without major difficulties. Two sets of spark gaps are on hand so that cycling operations are not held up while the gaps are being reconditioned. The water system is subject to cleaning and repair at yearly intervals.

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Dimensional changes, including warp, can be reliably reproduced in independent runs of duplicate material, because the specimens are not subject to severe thermal gradients, restraint, and so forth. The number of cycles is accurately and automatically counted. The Wheelco Limitrol gives a safety factor which on occasion has proved necessary to prevent overheating. Spring tension on the microswitches has been found to relax after continued use, resulting in a time lag before the relay circuits are activated. This has caused as much as a 10°C drop below the low-temperature limit. Maintenance must, therefore, be carefully performed to insure accurate results.

On heating, thermal expansion of the container reduces the $1/4$ in. air gap to the work coil, resulting occasionally in high-frequency arcing. One method to overcome this difficulty is to place quartz spacers $3/16$ in. in diameter along the full length of the coil and clamp the container securely to the cycling rack.

The use of expansion chambers of suitable volume has been found necessary to prevent the increased pressure on heating from forcing the NaK out of the container through the entry ports or damaging the rubber tubing.

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VERTICAL TUBE CYCLING UNIT (WOOSHER)

Design and Operation

A general view of the Vertical Tube Cycler, with the auxiliary automatic controls, is shown in Figure 4. The unit consists of two tanks of NaK alloy one above the other, with a NaK-filled tube passing through the center of both tanks. The schematic diagram, Figure 5, reveals the detailed arrangement, including the valve and head assembly at the upper end of the specimen transfer tube. During cycling, the test pieces are transferred within the transfer tube by the automatically controlled drive motor assembly at the top. Microswitches locate the end of travel each way, usually at the center of each tank.

The loading and unloading operation is relatively fast and simple. Specimens to be cycled are placed in a rack suspended on a stainless steel cable from the cable reel within the head. The specimen rack is then reeled up into the head, and the complete assembly is mounted on top of the valve. The head chamber is evacuated (about 6 microns)¹ and filled with argon to a pressure equal to that in the transfer tube below the closed gate valve, about 3 - 5 psi gage. The electronic control system is set for the desired holding time in each tank, and the drive motors are set for the required speed of transfer. The gate valve is then opened and the rack, containing the test specimens, is lowered to the mid-point of the hot tank. The universal drive shaft is connected between the head and the drive motor assembly, and the automatic cycling starts by closing the control switch. Upon completion of the last exposure in the hot tank, the specimen rack is reeled up into the head assembly and allowed to cool. The valve is then closed, and the head is removed. After removal, the inside of the head is usually sprayed with water to remove any NaK that may be present, and the specimens are removed. The drive motor assembly consists of two 115V DC 1/20-hp motors. The output of both motors passes through a differential, and each motor has a separate speed control box. Spur or worm gear sets are used on the output shaft of the motors for extremely low speeds. With this arrangement heating and cooling studies are greatly simplified, since combinations of transfer times² ranging from a few seconds to approximately one hour are presently possible. The detailed ANL engineering drawing numbers for this unit are given in the appendix.

¹This vacuum, as measured by a Stokes-McLeod gage, has proven to be entirely adequate and with careful operation, the NaK in the transfer tube remains clean for several months.

²Transfer time in the Woosher is defined as the time required to transfer the specimens from the center of one tank to the center of the other. It is, therefore, essentially a measure of the heating or cooling rate of the specimens.

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The schematic wiring diagram of Figure 6 shows the drive motor assembly as an integral part of the general electronic control wiring. The automatic cutoff device shown in the diagram makes it possible to automatically stop the cycling after a designated number of cycles has been completed. This device stops the cycling with the specimens in the cold tank. The "up" half of the last cycle is completed whenever the operator is ready to remove the specimens.

The desired tank temperatures are maintained by automatically controlling the current supplied to the calrods inside the tanks. For optimum temperature control a predetermined constant current is also supplied to the external calrod, or calrods, around the lower periphery of each tank.

Dowtherm is pumped through the coil in the cold tank whenever the temperature exceeds the set value. This becomes necessary when using fast transfer times and low cold-tank temperatures, owing to the frequent transfer of heat from the hot tank. The dowtherm pump is automatically controlled.

Temperature Range Obtainable

Thermal cycling tests run in the Woosher have not exceeded 600°C in the hot tank nor gone below room temperature in the cold tank. Short runs at higher temperatures are possible, but it would be inadvisable to operate for any length of time at temperatures approaching the boiling point of NaK (780°C). With a temperature difference of 50°C (hot tank 550°C, cold tank 500°C) flat temperature zones of 16 in. and over were obtained in either tank. Figure 5 shows a temperature distribution plot obtained with the hot tank at 550°C and the cold tank at 100°C. Numerous other combinations have been investigated. Low-temperature cycling is easily accomplished (50°C to 300°C).

Rate of Cycling

The rate of cycling in the Woosher is made flexible by the proper combination of transfer time and the holding time at temperature. The rate of cycling is limited only by the rate of heating and cooling of the test pieces. To obtain some idea of the order of magnitude of this limitation, thermal studies were made on a 2 in. long by 0.364 in. in diameter stainless steel rod during transfer from one tank to the other. Measurements were made over a range of temperatures from 50°C in the cold tank to 600°C in the hot tank. Typical results were as follows: With a thirty-second transfer time from a temperature of 200°C to 550°C, only four seconds were required for the specimen to reach temperature equilibrium; decreasing the transfer time to five seconds, between the same two temperatures, increased the time for temperature equalization to thirty-five seconds. A thirty-second transfer time between 50°C and 600°C required no time at all for temperature equalization since the test rod was at temperature by the time it

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reached the center of the 600°C zone. In general, over all the different temperature ranges analyzed, using a thirty-second transfer time required a maximum of about thirty seconds for equalization at either temperature, hot or cold. With a five-second transfer time, a maximum time of sixty seconds was required for equalization.

Typical heating and cooling curves between 100°C and 450°C, showing the effect of a five-second and a thirty-second transfer time, are reproduced in Figure 7. (It will be noted in Figure 7 that the transfer time down is faster than the time up. This difference is due to the fact that the rack weight influenced the transfer time down and made it difficult to control accurately. Only one drive motor was being used at the time these measurements were made.)

Characteristic time-temperature relationships of the Woosher cycle, based on some of the information obtained in the above temperature measurements are plotted in Figure 8. Hypothetical test runs between 100°C and 550°C using a "two-minute cold - two-minute hot" and "two-minute cold - twenty-minute hot" cycle with a five-second transfer time, are shown in comparison with a characteristic time-temperature cycle in the Induction Cyclor.

Reliability of Operation

The Woosher has operated continuously for nearly three years without serious difficulties. No particular hazards are involved in unattended operation, since automatic controls are available for every phase of operation. Wheelco Limitrols are used to prevent overheating of the tanks, and any interruption in power will completely shut down the unit until manually restarted.

The cycles are counted electronically by the automatic cutoff device and recorded on the Brown circular chart instrument. This visual record is made possible by a thermocouple in the transfer tube near the center of the hot tank. Each time the cold sample comes up it drops the temperature momentarily, giving a "pip" on the recorder chart. (With slow transfer times no graphic record is obtained.)

On several occasions the specimen rack has stuck in the transfer tube during thermal cycling. Proper design of the racks, combined with proper loading of the specimens, has eliminated this trouble.

LOW-TEMPERATURE CYCLING UNIT (WIG-WAG)

The Wig-Wag cycling unit consists essentially of two oil baths, one hot and one cold, with a mechanical arrangement for transferring the specimens, contained in NaK-filled containers, from one tank to the other. A schematic diagram of the equipment is shown in Figure 9. The procedure of loading the specimens into the containers and filling the containers with NaK is similar to that described under the Induction Cycler. After the specimen containers have been hung on the arms of the Wig-Wag, the electronic controller is set and cycling starts.

Temperature Range Obtainable

The Wig-Wag was designed primarily for low-temperature thermal cycling (up to 300°C). An oil was used with a 350°C flash point; however, no test runs were made above 300°C since the oil fumed quite badly and caused rather serious ventilation problems. The cold tank was maintained at or near room temperature for all tests.

Rate of Cycling

The duration of a complete cycle in the Wig-Wag may vary from thirty seconds up to several hours as determined by the setting on the electronic controller. The maximum number of cycles per unit of time depends on the heating and cooling rate of the container and contents. The large bomb, Figure 9, containing NaK and four stainless steel slugs (approximately 4.8 in. x 1.4 in.) required about twenty minutes to come up to the temperature of the hot tank (300°C). After transfer to the cold tank the container cooled in less than twenty minutes, providing that the cold oil was circulated. By using a smaller container, the inside diameter of which was large enough to hold six test rods, 0.360 in. diameter x 2 in. long, the time for one complete cycle between room temperature and 300°C could be reduced to ten minutes. The time required to move the containers from one tank to the other is fixed at about four to five seconds.

Reliability of Operation

The low-temperature cycler (Wig-Wag) has been operated for several thousand cycles with no trouble in the electrical or mechanical apparatus of the unit; however, certain aspects of the operation are not all that might be desired. Testing programs involving temperatures between 275°C and 300°C, for a prolonged period of time, ultimately result in the condensation of oil vapors within the ventilation system, which must be kept in continuous operation. Oil additions must be made from time to time to maintain a proper level in the hot tank. Despite the fact that the operating temperature is usually well below the flash point of the oil, a fire hazard exists, and unattended operation is precarious.

In view of these drawbacks, and since low-temperature cycling can also be performed satisfactorily in the Woosher, the Wig-Wag unit has been abandoned.

COMPARATIVE TEST RESULTS

Rolled Rods

Thermal cycling tests on uranium rolled at 300°C have been conducted in each of the three cycling units, and the results are summarized in Table I and are shown in Figure 10. All the test rods were cut from the same 300°C rolled stock.

The elongation coefficient K for the Induction cycled rods is 318 microinches per inch per cycle (standard Induction cycle, Figure 8), compared with a coefficient of about 600 for the twenty-minute hot - two-minute cold Woosher cycle and 490 for the two-minute hot - two-minute cold Woosher cycle, using a five-second transfer in each case. Cycling in the Wig-Wag between room temperature and 300°C (five minutes to heat - five minutes to cool) resulted in a K value of only 2.7 microinches per inch per cycle.

The Woosher cycled rods elongate approximately twice as much as the Induction cycled rods in the same number of cycles. Surface distortion, warping, and so forth of the Woosher cycled rods have progressed to such an extent in 700 cycles that accurate measurements are difficult. For a better comparison of K values in the two units, a smaller number of cycles should be used, 300 Induction cycles to 150 Woosher cycles, for example.

It is apparent from the above results that temperature range and time at temperature, as well as the rate of heating and/or cooling, must have a marked influence upon the rate of elongation on cycling.

Beta Annealed and Beta Quenched Rods

The rods heated into the beta phase (i.e., to 725°C) and slowly cooled grew very little compared with the rolled rods but showed severe surface roughening as can be seen in Figure 10. The rods heated into the beta phase and water quenched again grew very little as compared to the rolled rods and did not roughen nearly as badly as the slowly cooled rods. These beta quenched rods showed the best over-all stability as regards both length changes and surface roughening. Again the Woosher cycle appeared to be a more severe test of dimensional stability than the Induction Cycle.

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CONCLUSIONS

The Induction, Woosher, and Wig-Wag thermal cycling units have been built and tested, and each cyclor has been described somewhat in detail, including the temperature range attainable, rate of cycling, and reliability of operation. The first two units have proved to be completely reliable so far as unattended automatic operation is concerned. The Wig-Wag unit has been abandoned because of operational hazards and the fact that low-temperature tests can be more conveniently conducted in the Woosher.

The Woosher provides a means of greatly accelerating thermal cycling tests, resulting in a considerable saving of time. Certain cycling variables such as temperature range, time at temperature, and rate of heating and/or cooling can be easily varied. The comparative test results indicate that these variables must have a marked influence upon the growth rate and upon surface roughening.

ACKNOWLEDGEMENTS

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The initial basic design of the Woosher was conceived by L. R. Kelman, who was assisted by J. L. Armstrong in the construction details. D. C. Woodard and staff performed all the electronic control work. The Remote Control Division also assisted in this phase of the program.

F. G. Foote, H. H. Chiswik, and L. R. Kelman assisted in the direction of the program and the interpretation of results.

Table I

COMPARISON OF THERMAL CYCLING TEST RESULTS IN THE STANDARD INDUCTION,
WOOSHER, AND WIG-WAG UNITS

Fabrication and Heat-Treatment	Induction Cycling 100°C-550°C, 700 cycles			Woosher Cycling 2 min. cold-20 min. hot 100°C-550°C, 700 cycles			Woosher Cycling 2 min. cold-2 min. hot 100°C-550°C, 700 cycles			Wig-Wag Cycling 5 min. cold-5 min. hot 25°C-300°C, 2000 cycles		
	Rod No.	Elongation (%)	K*	Rod No.	Elongation (%)	K*	Rod No.	Elongation (%)	K*	Rod No.	Elongation (%)	K*
575°C anneal	7B	25.0	318	5B 6B	52.8 ⁽¹⁾ 52.5 ⁽¹⁾	607 602	6C 5C	40.8 ⁽¹⁾ 41.0 ⁽¹⁾	488 490	2A 5A 6A	0.543 0.495 0.493	2.7 2.4 2.4
T-3												
Slow-beta treatment, 725°C anneal followed by 575°C anneal	7A 9B 9C	1.08 1.23 3.34	15 18 47	4B 3B	(2) (2)		13B 3C	(2) (2)		4A 3A	0.024 0.020	0.1 0.1
T-2												
Fast-beta treatment, W.Q. after 2 min. at 725°C followed by 575°C anneal	7C 8B 8C	1.19 0.97 0.69	17 14 10	2B 1B	(2) (2)		2C 1C	(2) (2)		1A	0.000	0.0
T-1												

*Elongation Coefficient, $K = \frac{1}{L} \cdot \frac{dL}{dN} \cdot 10^6$

The table values are therefore in microinches/inch/cycle.
The value shown is based on the maximum number of cycles indicated.

(1) Approximate values.

(2) Rods too distorted to be measured accurately.

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Figure 1. General Arrangement of Induction Thermal Cycling Unit.

Figure 2. Schematic Diagram of Bomb Used in Induction Thermal Cycling.

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SCHEMATIC WIRING DIAGRAM OF INDUCTION CYCLING EQUIPMENT

Figure 3.

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Figure 4. General Arrangement of Vertical Tube Thermal Cycling Unit (Woosher).

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TEMPERATURE DISTRIBUTION IN WOOSHER

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Figure 5. Schematic Diagram of Woosher and Typical Temperature Distribution Between 100°C and 550°C.

SCHEMATIC WIRING DIAGRAM OF WOOSHER CYCLING UNIT

Figure 6.

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(a) 5-Second Transfer Rate.

(b) 30-Second Transfer Rate.

Figure 7. Typical Woosher Temperature Calibration Curves Between 100°C and 450°C.

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Figure 8. Characteristic Time-Temperature Relationships of Thermal Cycling Units.

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Figure 9. Schematic Diagram of Low-Temperature Cycling Unit. (Wig-Wag).

Figure 10. Effect of Cycling Upon Uranium Test Rods.

10a. Two-Inch Uranium Rods After 700 Cycles in Induction Cyclor.

Macro #M-4417

1X

7B

7A

9B

9C

7C

8B
T-1

8C

T-3

T-2

As Rolled

Beta Annealed

Beta Quenched

10b. Two-Inch Uranium Test Bars After 700 Cycles in Woosher Unit, 20-Minute Hot - 2-Minute Cold Cycle.

Macro #M-4418

1X

6B

5B

4B

3B

2B

1B

T-3

T-2

T-1

As Rolled

Beta Annealed

Beta Quenched

APPENDIX

Drawing Numbers of Cycling Equipment

Induction Cyclor

<u>Detail</u>	<u>Drawing Number</u>
Large Bomb	MY-1078-D
Small Bomb	MY-1045-D
Wiring Diagram*	Figure 3

Woosher

General Assembly (Tanks)	MY-915-DD
General Assembly (Head)	MY-977-D
Drive Mechanism Assembly	MY-1014-DD
Specimen Rack (Rounds)	MY-957-B
Specimen Rack (Plate)	MY-1080-B
Specimen Rack (Plate)	MY-1035-D
Cable Coupler	MY-1011-A
Wiring Diagram*	MY-1145-D

Wig-Wag

General Assembly (Including large bomb)	MY-887-DD
Small Bomb	MY-924-C
Wiring Diagram*	MY-908-C

*Includes description of electronic and control equipment.