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WANL-TME-1008

October, 1964

Test Report On Piezoelectric Crystal Discs
Experiment No. 23/W003
Irradiated At The Ground Test Reactor
(GTR Test 13)
(Title Unclassified)

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November 17, 1964

Mr. J. L. Dooling
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Sacramento, California

Subject: WAML-TAE-1008, "Test Report on Piezoelectric Crystal Disc
Experiment No. 22/WB03 Investigated at the Ground Test Reactor
(GTR Test 13)," October 1964

Dear Mr. Dooling:

Transmitted herewith are sixty (60) copies of the subject report. This
report is transmitted for your information.

Respectfully,

H. F. Fought
Program Manager
NERVA Nuclear Subsystem

Enclosures - 69

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TEST REPORT ON PIEZOELECTRIC CRYSTAL DISCS

EXPERIMENT NO. 23/W003

IRRADIATED AT THE GROUND TEST REACTOR

(GTR Test 13)

(Title Unclassified)

Group I

Excluded From Automatic Downgrading
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1.0 INTRODUCTION

1.1 GENERAL

This report describes the Westinghouse Astronuclear Laboratory (WANL) Test 23/W003 (GTR-13)¹ on piezoelectric accelerometer crystals, conducted at the Nuclear Aerospace Research Facility (NARF) of General Dynamics/Fort Worth in June, 1964. This was the tenth in a series of irradiation tests of NERVA components using the three megawatt Ground Test Reactor.

1.2 TEST HISTORY

Irradiation Test 23/W003 was designed as a screening test to determine the relative radiation resistance of several piezoelectric materials used in this type accelerometer. The main advantages of the piezoelectric accelerometer are small physical size and high frequency response.

This test, originally scheduled for irradiation in the Plum Brook Reactor Facility, was transferred to the Ground Test Reactor because of the decision not to expose the Plum Brook primary coolant water to the silver contacts of the test samples. The silver contacts were required to measure the electrical parameters of the test samples.

2.0 PIEZOELECTRIC CRYSTAL DISCS

2.1 SUMMARY OF TEST CONDITIONS

2.1.1 Items Tested

Twelve types of piezoelectric discs from three manufacturers were tested and are identified in Table 2.1. In addition to the basic composition of the crystal discs shown by the table, all types contain small traces of other inorganic agents intended to improve certain characteristics of the crystals.

2.1.2 NRX Conditions

Piezoelectric accelerometer crystals must survive the following temperature and radiation environments to perform satisfactorily in the NERVA reactor:

Temperature, °R	90° to 800°*
n/cm ² (E<1.86 ev)	10 ¹¹ to 10 ¹⁶
n/cm ² (E>2.9 Mev)	8.5 x 10 ¹⁶ to 6 x 10 ¹⁷
Gamma Exposure, ergs/g(C)	6.0 x 10 ¹⁰ to 4.0 x 10 ¹¹

*Gamma heating will result in internal temperatures far in excess of ambient.

2.1.3 GTR Test Conditions

Irradiation time for the piezoelectric crystals was 295 Mw-hr. The GTR reactor power profile is shown in Figure 2.1. Thirty-four piezoelectric crystals were installed in a wire mesh basket in rows with a perforated spacer separating each row as shown in Figure 2.2. The basket was suspended

Table 2.1

Piezoelectric Crystal Disc Test Samples

<u>Group</u>	<u>Mfg.</u>	<u>Type</u>	<u>Qty.</u>	<u>Basic Composition</u>	<u>Curie Temp. (°F)</u>	<u>Test Code Disc Identification</u>	<u>Thickness (in.)</u>
1	Clevite	PZT-5A	4	PbZrO ₃ -PbTiO ₃	660	1, 2, 1*, 2*	0.1
2	Columbia	-	3	PbZrO ₃ -PbTiO ₃	670	3, 4, 5	0.07
3	Gulton	G115	3	PbZrO ₃ -PbTiO ₃	750	A-1, 2, 4	0.03
4	Gulton	G1195	3	PbZrO ₃ -PbTiO ₃	570	B-4, 5, 6	0.03
5	Gulton	HDT-31	3	PbZrO ₃ -PbTiO ₃	>570	C-1, 2, 4	0.03
6	Gulton	H4-13	3	PbZrO ₃ -PbTiO ₃	555	D-1, 2, 3	0.03
7	Gulton	HS-21	3	BaTiO ₃ -PbTiO ₃	240	E-2, 4, 5	0.03
8	Gulton	HD-11	3	BaTiO ₃ -PbTiO ₃	240	F-1, 2, 4	0.03
9	Gulton	G797	3	PbZrO ₃ -PbTiO ₃	550	G-1, 2, 3	0.03
10	Gulton	G639	3	PbZrO ₃ -PbTiO ₃	620	H-1, 5, 6	0.03
11	Gulton	G53	3	PbZrO ₃ -PbTiO ₃	>525	I-1, 3, 5	0.03

Large crystals, 1.5 in. dia. x 0.100 in. thick. 1, 2 and 1 were heat treated, while 2* was not; therefore, this group was considered as two types. The diameter of the small crystals was 0.5 in.

Figure 2.1 Reactor Power Profile: Reactor Core

Figure 2.2 Piezoelectric Ceramic Disc Mounting Basket
End View

in a 21-foot long watertight container which replaced a reactor fuel element in the core of the GTR reactor. Gaseous nitrogen was passed through this container to cool the crystals.

The wiring diagram for pre- and post-testing the crystals is shown in Figure 2.3. Both pre-and post-irradiation data consisted of:

1. Resonant Frequency
2. Anti-Resonant Frequency
3. Disc Voltage for the Preceding Conditions
4. Capacity
5. Insulation Resistance
6. Dissipation Factor

The test fixture used during pre- and post-irradiation examination is shown in Figure 2.4. The only data recorded during irradiation of the discs were the temperature of the two inside walls of the container, the test discs and the gas inside the container, monitored by four copper-constantan thermocouples and recorded on a Brown multipoint recorder.

2.2 DOSIMETRY

2.2.1 Neutron and Gamma

The radiation environment for the piezoelectric crystal discs shown in Table 2.2 was based on an earlier flux mapping experiment² conducted in the dummy fuel element inserted into the reactor core (NW corner) and irradiated for one hour at a power level of 1.3 Mw.

Figure 2.3 Wiring Diagram for Testing Ceramic Discs in Hot Cell

Figure 2.4 Piezoelectric Ceramic Disc Test Fixture

Table 2.2

Radiation Exposure of the Piezoelectric Crystal Discs

Neutrons/cm ² ($E < 0.48$ ev)	3.3×10^{18}
Neutrons/cm ² ($E > 2.9$ ev)	6.5×10^{17}
Gamma Exposure, ergs/g(C)	1.8×10^{12}

In the flux mapping experiment, the neutron flux and gamma doses were measured using dosimeter packets each containing: one sulfur pellet, one N₂O dosimeter, and two gold foils (one bare and one cadmium covered). Integrated neutron fluxes and gamma doses listed in Table 2.2 were obtained by extrapolation to 17,700 Mw-min, the irradiation period for the piezoelectric crystal discs.

2.3 TEST SCHEDULE

A chronological summary of events for Test 23/W003 (GTR-13) is given in Table 2.3. The irradiation commenced on June 8 and was completed on June 12.

2.4 TEST RESULTS³

The maximum allowable gaseous nitrogen coolant gas flow through the 21-foot long watertight container was not sufficient to maintain a piezoelectric disc temperature of 200°F. This maximum temperature was estimated before the test based on a similar, previous experiment². The disc temperature increased to and stabilized at 635°F. The test was run at this temperature level. The discs were removed from the reactor approximately one hour after reactor shutdown and were removed from the basket approximately three hours after shutdown. The discs and basket indicated 500 R per hour which neces-

Table 2.3 Chronological Summary of Test Activities

<u>Date</u>	<u>Time</u>	<u>Chronological Summary of Events for Test 23/W003</u>
6/8/64	0800	Test articles positioned in place
	1050	Reactor start-up and operation at 10 Kw
	1126	Start of operation at 100 Kw
	1151	Start of operation at 1 Mw
	1216	Start of operation at 3 Mw
6/9/64	1325	Reactor shutdown to take data cycle on thermocouple portions of GTR-13 test
	1350	Reactor controlled to 1 Mw
	1355	Reactor controlled to 3 Mw
	1820	Malfunction in reactor controls. Reactor power decreasing
	1915	Reactor power reached 100 Kw, repairs complete
	1917	Reactor controlled to 3 Mw
	2042	Malfunction of LN_2 flow and control system. Reactor retracted
	2220	Repairs complete
6/10/64	0137	Reactor scrammed
	0208	Reactor controlled to 3 Mw
6/12/64	1558	Reactor shutdown, run complete
	1715	Crystals removed from core
	2000	First post data cycles completed on crystals
6/13/64	1300	Second post data cycles completed on crystals
6/15/64	1200	Third post data cycles completed on crystals

sitated personnel to work rapidly and for a short period of time to record post-irradiation data. Because of this handling several discs were broken. Also several discs were not identifiable because of discoloration. Fourteen unbroken, identifiable discs were checked for electrical characteristics approximately 4, 17, 85, 160, 350 and 600 hours after terminating the radiation. These tests indicated that the discs were severely degraded electrically.

It was known that the Curie temperature of some, but not all, of the test samples had been exceeded. Therefore, a control experiment was run in which additional units were held at reactor temperature in an oven. The control units were baked at 620°-625°F for 62 hours. The control units provided means for the separation of radiation and temperature induced effects, thereby salvaging significant test data. An explanation of how this significant test data was salvaged follows. The fundamental piezoelectric material constant is the coupling factor which is a measure of piezoelectric strength. This figure of merit (K) relates electrical and mechanical energies as follows:

$$K^2 = \frac{\text{Mechanical energy converted to charge}}{\text{Mechanical input energy}}$$

This (K) parameter was selected for assessment of radiation induced damage.

For thin discs, such as the test samples, in radial vibration, the planar coupling coefficient (K_p) can be determined graphically by use of measured resonant frequency data. Determination of the crystal resonant frequencies is a simple measurement accomplished by use of the test circuit depicted by

Figure 2.3 incorporating the test fixture shown in Figure 2.4. In such a circuit, crystal resonant and antiresonant frequencies are indicated by crystal voltage minima and maxima as the test frequency is varied. Radiation induced crystal damage is inferred by Table 2.5 in which the irradiated samples are compared to the control samples which were baked in the oven. Table 2.4 shows that on the control units whose Curie temperatures were not exceeded the coupling factor (piezoelectric strength) remained the same for disc A5, and was reduced by only 34 per cent for disc 6. The Curie temperature was exceeded by approximately 5°F for disc H2 and the reduction in coupling factor was 61 per cent. However, for the irradiated A2 and A4 which correspond to control A5, the reduction in piezoelectric activities was 100 per cent and 85 per cent respectively. The temperature of these sample discs was well below their Curie temperature during irradiation. The coupling coefficient of the fourteen identifiable discs was determined at intervals after the irradiation. This parameter increased considerably during the first twenty hours after irradiation and then decreased again, finally becoming unmeasurable.

After the irradiation, crystal resistances and capacities which were also measured were very unstable and in most cases outside the range of the instrumentation. The dissipation factor was in excess of 60 per cent for all samples. No significant recovery occurred during the observation period.

Several of the irradiated units which showed no piezoelectric activity at post four hours were weakly piezoelectric at post twenty hours. Units

Table 2.4

Radiation Effects on Planar Coupling Factor (Kp)

<u>Test Code Disc Identification</u>	<u>Curie Temp. (°F)</u>	<u>Irradiated Units</u>		<u>Control Units</u>	
		<u>Pre</u>	<u>Post (4 hr)</u>	<u>Pre</u>	<u>Post (4 hr)</u>
1	660	0.56	0.00		
2	660	0.36	0.00		
6	660			0.62	0.41
3	670	0.47	0.10		
4	670	0.47	0.05		
5	670	0.47	0.00		
A ₁	750	0.22	0.00		
A ₂	750	0.21	0.00		
A ₄	750	0.20	0.03		
A ₅	750			0.20	0.21
D ₁	555	0.59	0.00		
D ₆	555			0.58	0.00
E ₂	240	0.31	0.00		
E ₄	240	0.34	0.00		
E ₆	240			0.35	0.00
F ₂	240	0.17	0.00		
F ₅	240			0.16	0.00
H ₁	620	0.46	0.05		
H ₂	620			0.41	0.16
15	550	0.51	0.00		
16	550			0.40	0.00

1, 2 and 5 are examples. This effect was not present in any control unit which was de-polarized by elevation of temperature above the Curie temperature.

The piezoelectric activity of the control units increased with time after they were removed from the high temperature of the oven. Stabilization was virtually complete at 100 hours, but no control (except A5 which was not damaged) recovered to its initial activity level.

2.5 TEST CONCLUSIONS

Several important conclusions result from this test and are summarized below:

1. The crystals saw an environment similar to that which would be seen by the crystal of an accelerometer in an NRX pressure vessel. This would be due to gamma heating in NRX and the low thermal conductivity inherent to the piezoelectric accelerometer structure.
2. Piezoelectric crystals tend to be depolarized and rendered useless by radiation.
3. Some recovery of activity does occur for a time after irradiation. This effect is a shortcoming with respect to the present application since it will confuse post-irradiation calibrations.
4. None of the crystal types tested could be made into an accelerometer that would be recommended for use inside an NRX pressure vessel.

3.0 REFERENCES

1. FZK-184-3, "NERVA Components Irradiation Program, Volume 3: GTR Test 13", General Dynamics/Fort Worth, September 13, 1964.
2. FZK-170-4, "NERVA Components Irradiation Program, Volume 4: GTR Test 7A", General Dynamics/Fort Worth, November 20, 1964.
3. WANL-TME-901, "Summary of NERVA Instrumentation Quarterly Design Review at WANL", Westinghouse Astronuclear Laboratory, July 29-30, 1964.