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FIRST QUARTERLY PROGRESS REPORT

ML-419 THERMISTOR P.E.M.

FOR PERIOD JANUARY, 1963 TO APRIL 1, 1963

CONTRACT NO. DA-36-039-AMC-01465(E)

PLACED BY INDUSTRIAL PREPAREDNESS, USASSA,

PHILADELPHIA, PENNA.

CONTRACTOR - THE BENDIX CORPORATION

FRIEZ INSTRUMENT DIVISION

BALTIMORE 4, MD.

SECRET

*Bendix-Friez*

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P.E.M. REPORT FOR ML-419 THERMISTOR

CONTRACT NO. DA-36-039-AMC-01465(E)

Report is first quarterly for period starting January, 1963,  
through April 1, 1963.

The object of this report is to summarize activities on the  
improvement of the quality and processing facilities of the Signal  
Corps ML-419 Thermistor Sensor for Radiosonde use.

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First Quarter work placed the emphasis on the following:

- A. To make a fine, pure suspension of basic ingredients.
- B. To process these ingredients to fashion rods that are free of air and foreign matter.
- C. To fashion rods to be as precise as possible, dimensionally.
- D. To vitrify these rods under repeatable, precisely controlled firing conditions.
- E. To apply lead wires to the copper electrodes, automatically.
- F. To reduce labor requirements in various stages of the process.
- G. To remove the manual operations in white reflectivity coating procedure whenever practical.
- H. To improve the reliability of reflectivity test apparatus.
- J. To improve the strength of the thermistor.

ITEM A. To produce a fine, pure suspension of basic clays.

Three ingredients have been acquired in sufficient quantity for this phase.

1. Bentonite, B C Grade, 1500 mesh as manufactured by The American Colloid Company of Skokie, Illinois. Former grade was designated as SPV of general industrial use intention. Sieve analysis 85 to 90% through 200 mesh.

The purpose of this clay is to bind and make the suspension adaptable to extrusion and to hold the mix in suspension.

2. Ferric oxide, EX 1265 as purchased from C. K. Williams & Company of Easton, Pennsylvania. A high purity ferric oxide developed for use in the ferrite industry. Former ferric oxide used was R 2199 from the same company. The purpose of this clay is to provide the conductivity for the sintered rod and when reacted with zirconium oxide and titanium dioxide to provide the required negative temperature coefficient characteristics.
3. Zirconium oxide (ZR O<sub>2</sub>) as purchased from the Titanium Alloy Manufacturing Division of National Lead Company of New York, New York. Used for chemical reactions where high purity is required.

Inquiries were made on possible better grades of ball clay from The Kentucky-Tennessee Clay Company of Mayfield, Kentucky. We were informed that we have the best grade obtainable designated as #7 ball clay, airfloated.

The titanium dioxide Ti O<sub>2</sub> is, and has been, a chemically pure grade as purchased from The National Lead Company of New York.

ITEM B. To process these clays to fashion rods that are fine in nature and free of air and foreign materials.

The following operations are involved:

1. Ball milling.
2. Filtering.
3. Pugging.
4. Extruding.

1. Ball Milling.

Wet ball milling has been used previously and found to be quite satisfactory. However, additional weight requirements of the ML-419



thermistor and better uniformity of electrical characteristic so necessary for controlled processing brought about the setting up of a program to obtain the optimum from this procedure.

Four mixes of the basic formulations were made.

- a. In two-gallon porcelain crocks with porcelain 1" dia. balls, present formulation to be used as a control batch.
- b. Present formulation ground with 3/8" high carbon chrome alloy balls as purchased from The Fisher Scientific Company.
- c. Present formulation but with the three better grades of clays as outlined previously with the 1" balls used in batch "a".
- d. Present formulation with materials used as in batch "a" with 1" balls but with an additive in the form of sodium tri-poly-phosphate  $Na_5 (F_3O_{10})$  to be used as a wetting agent.

The program for evaluation is set up as follows:

Step I. a. Ball mill four batches for one day intervals.

- b. Remove small quantities of each for particle size evaluation each day of milling.
- c. Compare visual sedimentation, etc. each day of milling.
- d. Evaluate the results up to five days of ball milling.

Step II. Prepare larger quantities of mix of the four batches to make thermistors.

- a. Evaluate results with batch #1 (the control batch) in respect to physical size, strength, and electrical characteristics.
- b. Evaluate results of (a) and make repeat runs of the best mix.
- c. Correct size discrepancies and adjust mix to temperature vs. resistance requirements.

Work on the above has progressed to the point where some results may be given on Step I in the next monthly report due May 10, 1963.

ITEM C. To fashion rods that are to be free of air and foreign materials.

1. Filtering.
2. Pugging.
3. Extruding.

1. Improvements are contemplated on present filtering equipment. The equipment consists of a tank of chrome lined steel; pipes to connect the Sperry Filter laboratory type press and the press itself. The improvements, if the present process is used, will be of a nature to prevent metallic particles from entering into the clay. A process that may substitute for this procedure will be discussed at a later date. The writer will be better informed on the subject, it is hoped, with a visit scheduled with instructors at Rutgers University in Brunswick, New Jersey.
2. The pugging procedure is a de-airing process. Present equipment is in need of repair and replacement of vital parts. The parts will be ordered and are available from the International Clay Company of Delaware.
3. The extrusion of clay rods is presently accomplished with the use of a hydraulic ram onto cylinders of clay mix sent onto a conveyor belt. The conveyor belt is run under infra-red lamps and dried. At the extreme of the conveyor a cutting device is employed and the green thermistors collected in a vibrated tray. The belt speed is controlled by hand for smooth extrusion.

A sensing unit composed of two photoelectric cells and light sources and amplifiers has been ordered from Leo T. McCourt & Sons of Baltimore, Md. This unit will be employed to eliminate this hand controlled operation and permit finer electronic control of extruded rods.

ITEM D. To vitrify these rods under repeatable, precisely controlled firing conditions.

Antiquated control equipment for kiln operation has been removed and in its place is a set of automatic controls that will, and is enabling us to control rise rate, soaking periods and falling rates of the ceramic kiln. The equipment consists of:

- a. A Minneapolis-Honeywell Electronik Strip Chart Electrovolt Proportioning Program Controller.
- b. A General Electric Magnetic Amplifier which is used for operation of G. E. saturable core reactors. This to assure smooth proportioning operation.
- c. A Radimatic unit for sighting the work in the kiln.

Tests of this equipment will determine the course of a possible purchase of a larger, more adaptable atmospherically controlled kiln. The tests will consist of comparison of fired lots of thermistors for uniformity and repeatability. Early indications seem to point to the need of better kilns. Temperature gradients seem to be evident in test results. Later reports will be forthcoming on this phase.

ITEM E. Automatic application of lead wires to the thermistor rods.

The United Shoe Machinery Corporation of Beverly, Massachusetts, has conducted a program for the design of such a piece of machinery. Samples of rods and lead wires were sent to Mr. E. A. Petrausha, Marketing Engineer. In a recent letter of April 15 to us by Mr. Petrausha, we are led to believe that such a machine is feasible.

The machine would consist of:

1. A wire feeding mechanism.
2. A wire guiding device.
3. A wire cut-off to required lead length mechanism.
4. A device for wrapping the wire.
5. Mechanism for holding, locating and possibly rotating the thermistor.
6. A hopper or feeding arrangement.
7. Auxiliary guides, chutes, etc.

The soldering operation would be a separate study. A meeting is being arranged through phone conversations to consider the various phases of the program.

ITEM F.

Refer to report on Items E and C. Other sketches for ease of processing are not in submission stages.

ITEM G. White reflectivity coating machinery.

It has long been recognized that manual operation of applying paint is inconsistent. A machine consisting of a rack holder (holds 35 thermistors), a motor with gear drive, and a tray for holding the white paint has been made.

This machine will be developed in three stages:

1. To coat with paint. Adjust speeds, etc. The rack moves the thermistors in an arc through the paint then back through the paint.

2. To incorporate stirring while thermistors are out of the paint. This to assure consistent agitation of the white paint.
3. To program the machinery. This will conserve labor as other operations pertinent with coating procedures such as screening, may be accomplished as the machinery is being operated.

Test results are not yet available.

ITEM H. To improve the reliability of reflectivity checks.

A machine is undergoing checks by our test equipment department. This equipment is designed to improve present facilities. The required equipment, meant to measure solar reflectivity, consists of a means of mounting a black standard ML-419 and comparing the change in resistance of a white coated production thermistor with that of the black. To do this requires also a light source, a lens, simple Wheatstone bridge circuit with a temperature compensation handled by another white standard ML-419 thermistor in the bridge circuit. The new equipment is designed to eliminate various possible errors in test, as:

1. Positioning of thermistor under test.
2. Adverse temperature conditions in the test areas.

The new equipment should, when placed in use, be capable of measurements within .5% error in the range of weighted mean reflectivity of 92% (the minimum figure allowable). Later reports will be made on this item about to be placed into use.

ITEM J. To improve the strength of the thermistor.

Following is a group of experiments on exterior coatings for strength:

Test #1.

- A. Twenty-five units were dipped in a diluted solution of LOGO plating lacquer ET-179, manufactured by the Bee Chemical Company. Thinner SC-100, air dry 1/2 hour; then oven bake at 160°C for 1/2 hour.
- B. Strength evaluations were then made on the above elements.

Comparison tests were made on uncoated elements, and then on LOGO coated elements (25 each).

Uncoated Elements  
Breaking Point in Grams

Average of 25 units were tested and evaluated.

70  
87  
85  
84  
92  
79  
89  
86  
71  
85  
67  
81  
75  
66  
90  
84  
84  
76  
74  
89  
69  
90  
66  
79  
88  

---

2,010

Average 80.4 grams

Coated Elements  
Breaking Point in Grams

Average of 25 units were tested and evaluated.

104  
93  
97  
86  
98  
87  
88  
92  
96  
90  
89  
84  
84  
89  
92  
98  
84  
86  
97  
94  
89  
81  
104  
87  
72  

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2,271

Average 90.8 grams

Comparison: Uncoated element 80.4 grams  
Coated element 90.8 grams

Ten more elements were coated full strength with LOGO lacquer - and cured as before. Strength test as below:

97  
92  
96  
95  
88  
96  
96  
99  
92  

---

106

95.7 average of ten coated elements.

Test #2.

Five units (element ML-419) were coated with "AMBROID", full strength, allowed to air dry, overnight. Result of test as below.

104  
103  
103  
90  
90  
490

Average load strength - 98 grams.

Test #3.

Five units were coated with full strength "Glyptal" - air dry 1 hour. Oven bake at 95°C 2-1/2 hours (vendor recommendation). Results below.

108  
90  
90  
94  
96  
478

Average - 95 grams.

Test #4.

Epo Lux #100 Bee Chemical  
Epo Lux thinner  
Air dry 48 hours

Load 5 units -- Average 115 grams.

Test #5

We received the following samples from Bee Chemical Company, 2700 E. 170th Street, Lansing, Illinois.

1 pt. LOGO - topcoat clear S2R-30479  
EV-5989  
ET-437  
EV-6174  
Catalyst ET-438  
Thinner R-226

Cleaned ten ML-419, ultrasonic cleaner -- dried thoroughly, dipped in S2R-30479 - air dry 1/2 hour - then bake 1 hour 140°F.

Results of Test #5 -- All units in excess of 90 grams.  
Average 95 grams.

Test #6.

Ten units were dipped in EV-5989 - air dry 1/2 hour. Bake 1 hour at 140°F. All units excess of 90 grams.

Test #7.

Ten units were tested in EV-6174 - air dry 1/2 hour. Bake 1 hour at 265°F. 9 units average 94 grams, 1 unit broke at 90 grams.

Test #8.

Ten units dipped in mixture of equal quantities of ET-437 and ET-438. Air dry 1/2 hour - bake 1 hour at 180°F. 9 units average 92.0 grams, 1 unit broke at 90 grams.

Test #9.

One unit was sprayed with white Krylon mixture. After air drying unit broke at less than 90 grams.

Test #10.

Dipped seven units in sodium silicate solution - air dry 1/2 hour - oven baked 1 hour at 160°C. Of 5 tested units -

- a) Three broke less than 90 grams
- b) One unit at 101 grams
- c) One unit at 96 grams.

Note: Solution was not new, and not homogeneous. Suggested further evaluation with new composition.

Test #11.

Dipped four units in white porcelain enamel lacquer - air dry 15 min., oven bake 1 hour at 160°C. Breakage loads:

101 grams	
103 grams	
95 grams	
<u>95 grams</u>	
394	Average - 98 grams.

Test #12.

Five units were dipped in white porcelain enamel.

- a) Air dry 1/2 hour
- b) Oven bake 170°C  
Repeated two times - allowed 3 days before breakage testing.
- c) All broke less than 90 grams.

Results indicate not useable.

Test #13.

Repeated above black porcelain enamel - air dry 1/2 hour - oven bake 170°C. Repeated twice.

Results: all units failed 90

In addition, outside consultation was sought with the following Corporations:

- a. Hartwood Glass and Metals, Inc.  
Martinsburg, West virginia.
- b. U. S. Stoneware  
Philadelphia, Penna. and Tallmadge, Ohio.
- c. Corning Glass Works  
Corning, New York.
- d. Arcal Chemicals, Inc.  
Seat Pleasant, Md.
- e. Flood and Conklin Manufacturing Co.  
136 Chestnut St., Newark 5, N. J.
- f. Bee Chemical Company  
LOGO Division  
Lansing, Illinois
- g. The Glidden Company  
Baltimore, Md.

Conclusion: We have set into motion a plan which is basically directed to improve the strength, uniformity of both physical and electrical nature, and to eliminate the manual operations in thermistor fabrication to as large a degree as is practical.

The monthly reports to follow will be accompanied by test results and summations on the various steps of processing.