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EVALUATION OF THE SELF-CALIBRATING THERMOCOUPLE AS A FRONT END TO A SMART TEMPERATURE MEASUREMENT SYSTEM*

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Evaluation of the self-calibrating thermocouple as a front end to a smart temperature measurement system[a]

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ABSTRACT

An evaluation of the novel self-calibrating thermocouple was performed to determine whether the sensor would be acceptable as a front end to a smart temperature measurement system. The evaluation consisted of a fast-ramp test, long-term drift tests, and physical examinations including X rays, microphotography, and energy-dispersive spectrometry. The results of the tests show that the sensor is a viable industrial-grade device worthy for use in this application. However, recommendations for improving fabrication of the assembly are made and caveats are given for conditions that may constrain the use of the sensor in certain situations.

SUBJECT INDEX calibration methods, fixed points, thermocouple diagnostics

INTRODUCTION

The objective of the work reported here was to test the commercially available self-calibrating thermocouple to see whether it is worthy as a front end to a smart temperature measurement system. The thermocouple used in this work is a special type, called a *self-calibrating thermocouple*, available from Isothermal Technology Ltd. in England. It is special because it contains an encapsulated high-purity metal near the thermojunction, as shown in Fig. 1. If the thermocouple is heated through the melting point of the metal, the temperature record will indicate a plateau during the transition from solid to liquid state. A typical temperature record through the melting point is shown in Fig. 2. After melting is complete, the temperature record will recover to the original heating slope. By noting the temperature of the plateau point and comparing it with the known melting point of the metal, a single-point calibration check can be performed.

The thermojunction is not in direct physical contact with the encapsulated metal. A ceramic insulator between the thermojunction and the encapsulated metal metallurgically isolates the two components so that they will not form an alloy with each other. An alloy could pose two detrimental threats. First, if the encapsulated metal alloyed significantly, its melting point could no longer be used as a reference because of the change in the alloy melting point from the pure-metal melting point. Second, if the thermojunction alloyed significantly, its Seebeck coefficient would no longer be known. This could be a problem if the alloying proceeded far enough into the thermoelements such that the alloyed elements were not isothermal along the affected path. The thermocouple electromotive force (emf) versus temperature curve would no longer match the original. The problem of diffusion of metals at the thermojunction is not unique to this type of thermocouple. It has even been postulated that small errors can be caused by the simple interdiffusion between thermoelements at elevated temperatures.[1]

Although the thermojunction and the encapsulated metal are not in physical contact, they are thermally coupled because of their close proximity. Therefore, the reading from the thermocouple will also indicate a plateau.

The thermocouple is intended to be used in a process in which the normal operating temperature is somewhat above the melting point of the metal. The calibration point can be checked as the process is ramping up to temperature, and if the metal does not supercool significantly, it can be checked when the process temperature is ramping down by using the freezing point of the metal.

The main feature of using the self-calibrating thermocouple with a data acquisition system that will automatically determine the calibration point is that it can determine whether the primary sensing element is operating properly. In addition, every component in series with the temperature signal is checked (i.e., thermocouple extension wire, temperature transmitter if used, signal wire, analog-to-digital converter on data acquisition system, and software residing in data acquisition system).

Previously reported work on the self-calibrating thermocouple includes the design of an algorithm to run on a microprocessor-based data acquisition unit that will automatically detect the calibration point of the self-calibrating thermocouple[2] and a thermodynamic model of the self-calibrating thermocouple.[3]

In the field of smart temperature measurement systems, a trade-off exists between simple sensors with complex signal processing and modified sensors with simple signal processing. In the first case, a good example is Johnson noise thermometry. The primary sensor is not modified at all, but complex signal processing is involved. The thermocouple investigated here is an example of the second case. The sensor has been modified to include the encapsulated metal for calibration-point reference. The signal processing algorithms are not too complex. However, the integrity of the sensor is lessened by the modification. The time constant of the thermocouple is lengthened by the encapsulated reference metal mounted close to its thermojunction, and the long-term reliability will surely be affected when the encapsulated metal is in the liquid state with far greater vapor pressure than when it is in the solid state. The encapsulated-metal vapor is bound to attack other components of the thermocouple system over its lifetime.

EXPERIMENTAL PROCEDURES AND RESULTS

A series of experimental tests and physical evaluations was performed on the self-calibrating thermocouple to determine whether it is an acceptable front end for this application. Three types of self-calibrating thermocouples were bought directly from Isothermal

Technology for testing: (1) type K with a tin reference cell, (2) type K with a zinc reference cell, and (3) type S with a gold reference cell. In addition, a type K thermocouple with a zinc reference cell was obtained from researchers at the University of Tennessee, where it had undergone informal, undocumented testing. At the time it was obtained, this thermocouple was disassembled, revealing an encapsulated metal-to-thermojunction interaction. This thermocouple will be referred to as the *defective thermocouple.*

FAST-RAMP TEST

The automatic determination of the phase change of a metal depends on the duration of the phase-change plateau. A suitable duration must be ensured so that the automatic algorithm will work properly. This requirement is especially applicable to a digitally sampled system. If the heating rate were too high and the sampling interval were too long, it would be possible for the phase change to occur between samples and not be noticed by the sampling system. Therefore, a test was performed to determine the phase-change duration for increasing heating rates.

A self-calibrating thermocouple with a tin reference cell was inserted into an Inconel tube furnace (see Fig. 3). The thermocouple was connected to a recorder with built-in cold junction compensation. A variac autotransformer was connected to a primary transformer coil, as shown in the figure. A secondary coil wrapped around the primary coil was connected to the Inconel tube. Current from the secondary coil was passed through the Inconel tube. By varying the number of turns on the secondary coil, the voltage to the Inconel tube could be increased or decreased. Thus, the heating rate of the tube could be varied by increasing the number of secondary coil turns. The power to the coils was applied instantaneously; no attempt was made to ramp the power. Several different heating ramp rate tests were done. Temperature records were made of each test to log the phase-change plateau duration. At a maximum ramp rate of 3.1°C/s, the duration of the phase-change plateau was ~15 s. This duration was considered to be suitable for a majority of industrial applications.

DRIFT TESTS

The defective thermocouple showed severe signs of an encapsulated metal-to-thermojunction interaction. After disassembly, electrical conductivity was measured between the thermojunction and the encapsulated metal. Apparently, the ceramic wall between the components had broken down or was somehow damaged. This interaction cannot be tolerated in an industrial-grade device that would have to perform reliably for thousands of hours. Therefore, it was decided that drift tests would be done on three of the new thermocouples obtained directly from Isothermal Technology to characterize the interaction between the encapsulated metal and the thermojunction. The tests were designed to report two characteristics of the thermocouples: (1) thermojunction emf drift and (2) calibration-point drift.

The test thermocouple and an end-closed ceramic tube were placed end to end, as in Fig. 4. A strip of platinum foil, ~5 cm wide and 12 cm long, was wrapped around the test thermocouple and ceramic tube to help keep the test section as isothermal as possible. The entire assembly was slid into a 40-cm-long tubular furnace with the leads from the test thermocouple extending out one side and the open end of the ceramic tube extending out the other end. The purpose of the ceramic tube was to provide a receptacle for a standard type S thermocouple with calibration traceable to the National Institute of Standards and Technology. The standard thermocouple was slid into the ceramic tube only when readings were to be taken, thus preventing the prolonged high temperature of the furnace from aggravating a detrimental oxidation of the thermojunction.

The leads from all thermocouples were spliced with copper wires. The thermocouple-to-copper junction of about half the wires was placed in a Kaye electronic ice-point reference cell. The other half was placed in an Omega ice-point reference cell. The free ends of the copper wires were terminated with thermocouple plugs that mated with corresponding receptacles on a data acquisition system interface box.

THERMOJUNCTION ELECTROMOTIVE FORCE DRIFT TESTS

The set point of each furnace temperature controller was set at ~30°C above the melting point of the encapsulated metal, thus ensuring an aggressive environment for decalibration caused by the higher vapor pressure of the encapsulated liquid metal as compared to the solid metal. Daily recordings were made of the test thermocouple temperatures and loop resistances. About once a week, the standard thermocouple was slid into the ceramic tube. Its temperature was allowed to equilibrate with the test assembly. Then, temperature readings were taken of the test thermocouple and the standard thermocouple to record the thermocouple emf drift.

The history of the test thermocouples as compared with the type S standard thermocouple is shown in Fig. 5. Results of the first 40 d are not shown, because a problem was found with the Omega electronic ice bath, yielding a cyclic ±2°C error in the data during that period. For the type K thermocouple with the tin reference cell, the difference between the test thermocouple reading and the standard reference thermocouple reading varied considerably throughout the test. Evidently, significant thermal resistance existed between the test thermojunction and the standard reference thermojunction. The type K thermocouple with the zinc reference cell held the closest of the three tested—no more than 2°C variation throughout the test. The type S thermocouple with the gold reference cell varied more (~4°C). Because the test and reference thermocouples were not ideally thermally coupled, the results of the thermojunction emf drift tests are inconclusive. The following section discusses the correlation between the thermojunction emf drift tests and the calibration-point drift tests.

CALIBRATION-POINT DRIFT TESTS

Also at one-week intervals, the temperature of the furnace was dropped below the freezing point of the encapsulated metal. A freezing-point calibration test was performed on the thermocouple with the zinc reference cell. Because of excessive supercooling, the freezing point of the thermocouples with the tin and gold reference cells could not be taken. The thermocouple with the tin reference cell supercooled as much as 25° C, and the thermocouple with the gold reference cell supercooled more than 100° C at times (even then, the thermocouple had to be vibrated before the gold would freeze). When the thermocouple temperature had stabilized below the fusion temperature, the furnace temperature was raised above the fusion temperature again. A melting-point calibration was performed on all three test thermocouples.

Figures 6 through 8 show the history of the test thermocouple calibration-point drift tests. The temperature and calibration-point drifts, as computed by a least-squares-type fit through the experimental data, are given in Table I.

The melting-point drift of the thermocouple with the tin reference cell is negligible. The thermocouple with the zinc reference cell shows two different calibration point drift trends based on whether the melting- or freezing-point trend is used. The freezing-point trend appears to correlate somewhat with the thermojunction emf drift test for the thermocouple with the zinc reference cell. In general, the freezing point is a more accurate determination than the melting point. The melting-point trend of the thermocouple with the gold reference cell shows only a slight downward drift. The melting points varied within roughly ±0.5° C but appeared to have a reverse correlation with the thermojunction emf drift tests.

PHYSICAL EVALUATIONS

Both nondestructive and destructive physical evaluations were performed on the test thermocouples. An X-ray photograph of six test thermocouples, taken before any other tests were performed, is shown in Fig. 9. Starting with thermocouple 1 at the left and going to the right, thermocouples 1 and 2 are type K with zinc reference cells, thermocouples 3 and 4 are type K with tin reference cells, and thermocouples 5 and 6 are type S with gold reference cells. Unfortunately, it is not possible to determine how far the thermoelements extend into the surroundings of the encapsulated metal. However, on thermocouples 3, 4, and 5, it is possible to note that the thermojunction is not hidden by the metal, therefore indicating that it is not ideally thermally coupled to the metal. It would be better if the metal totally enclosed the thermojunction.

After the drift tests were complete, the junction ends of the test thermocouple assemblies were broken apart from the remainder of the assembly. As mentioned, the defective thermocouple showed visual signs of a thermojunction-to-metal reaction. The thermoelement leads were embrittled. Also, electrical conductivity was measured between the thermoelements and the metal. A visual examination of the thermocouples obtained directly from Isothermal Technology did not indicate embrittled thermoelements with these specimens, as did the defective specimen. It also appeared that the encapsulated metal was well insulated from the thermoelements by a ceramic shroud. In fact, the zinc metal fell away from the ceramic shroud and thermoelements when it was disassembled. The test specimens were axially ground to the thermojunction point, polished, mounted, and micrographed to study the crystalline nature of the thermojunction-to-metal reaction, if any. The new type K thermocouple with the zinc reference cell was not analyzed in the following tests, because as described above, the reference cell fell away from the thermojunction when it was disassembled. However, the defective type K thermocouple with the zinc reference cell did undergo the following tests. A micrograph of the initial defective thermocouple is shown in Fig. 10. The ceramic insulator is cracked, and the surrounding zinc metal has infiltrated the thermoelements. Unfortunately, this thermocouple was not tested in a furnace to see whether the reported melting point had changed much. Micrographs of the type K thermocouple with the tin reference cell and the type S thermocouple with the gold reference cell are shown in Fig. 11 and Fig. 12 respectively. These micrographs do not indicate a metal-to-thermoelement interaction.

The specimens were also evaluated by using an energy-dispersive spectrometer on a scanning electron microscope to study the qualitative elemental composition of the metal-to-thermoelement interface. A spectrum of the initial defective thermocouple is shown in Fig. 13. The spectrum was taken of one of the thermoelements, but the elemental composition is largely zinc, indicating a severe interaction between the encapsulated zinc metal and the thermoelements. Figures 14 and 15 show similar scans of the tested type K thermocouple with the tin reference cell. Figure 14 shows the Chromel wire scan, and Fig. 15 shows the Alumel wire scan. The composition of Chromel is 90% nickel and 10% chromium; the composition of Alumel is 94% nickel, 3% manganese, 2% aluminum, and 1% silicon.[4] The scan is absent of the surrounding tin metal.

Figures 16 and 17 show similar scans of the tested type S thermocouple with the gold reference cell. Figure 16 shows the Pt-10% Rh wire scan, and Fig. 17 shows the platinum wire scan. These scans are absent of the surrounding gold metal. It is expected, although not confirmed, that similar noninteraction would have been reported of the tested type K thermocouple with the zinc reference cell because the ceramic insulator freely fell away from the metal.

CONCLUSIONS

On the bases of the drift tests and physical evaluations of the self-calibrating thermocouple, it appears that the thermocouple is a viable industrial-grade assembly and therefore a good front end for a smart temperature measurement system. However, the results are based on the testing of only three self-calibrating thermocouples, which is not a statistically large sample. Further testing should be performed before embarking on a critical project using self-calibrating thermocouples. As a result of this work, it is recommended that the self-calibrating thermocouple manufacturer take steps to ensure that the thermojunction is completely enclosed by the encapsulated metal to provide a flatter plateau.

The smart temperature measurement system is designed to provide a single calibration point. Although a system may be functioning with good agreement with a single calibration, there is no guarantee that the system is not out of calibration at other points in the system. Furthermore, no method can adjust the emf/temperature relation based on a single calibration point. However, if the calibration temperature is close to the operating temperature, then the calibration error can probably be used for the operating temperature error.

The results of this work show that unless confirmed beforehand, the freezing point of the encapsulated metal cannot be used as a calibration point, because of excessive metal supercooling.

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a) Work performed at Oak Ridge National Laboratory, managed by Martin Marietta Energy Systems, Inc., for the U.S. Department of Energy under contract DE-AC05-84OR21400.

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Table I. Results of thermocouple drift tests.

Thermocouple type	Reference cell	Thermojunction emf drift (°C/h)	Calibration point drift (°C/h)
K	Tin	0.0027	0.000081 (melt)
K	Zinc	0.00069	–0.00005 (melt) 0.0002 (freeze)
S	Gold	0.0012	–0.00028 (melt)

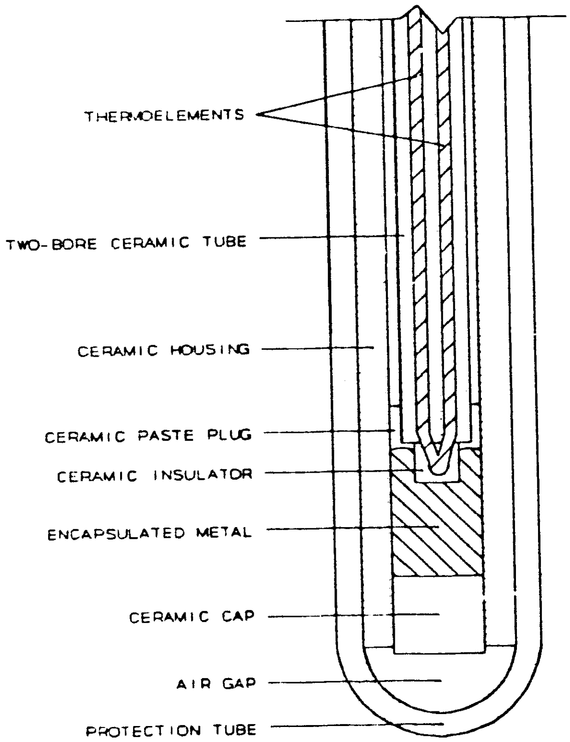

Fig. 1. Cross-sectional view of self-calibrating thermocouple.

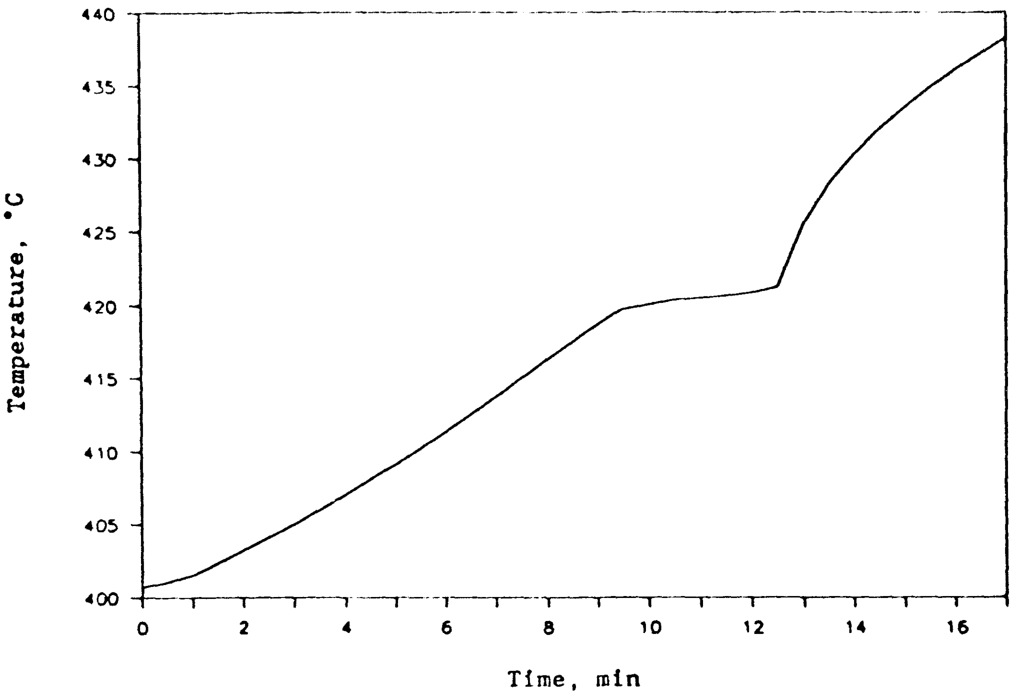

Fig. 2. Typical heating curve of self-calibrating thermocouple through melting point of reference Cell.

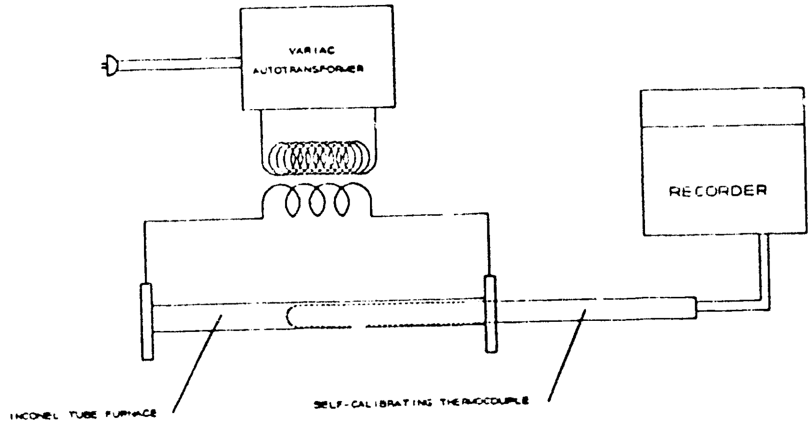

Fig. 3. Fast-ramp test equipment arrangement.

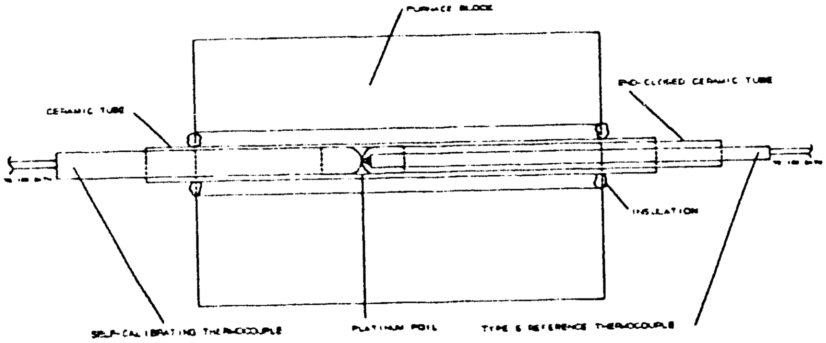

Fig. 4. Drift tests equipment arrangement.

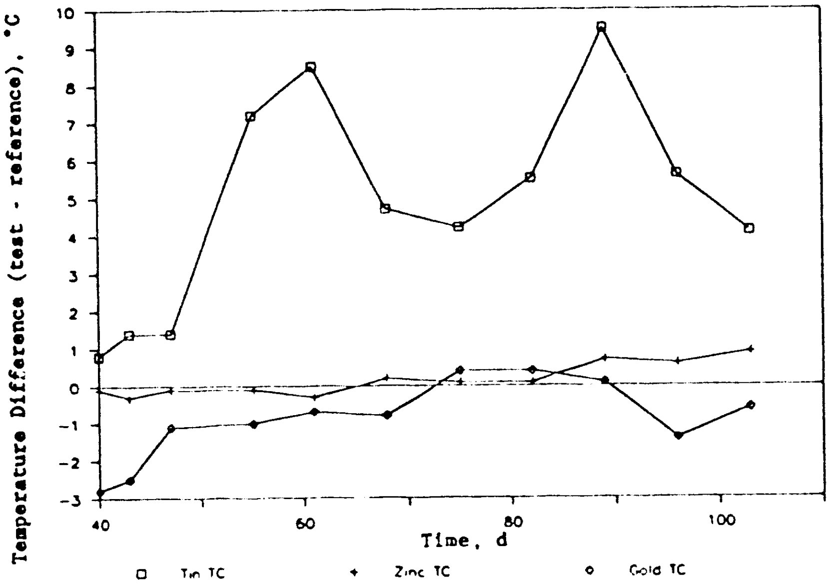

Fig. 5. Thermojunction electromotive force drift history.

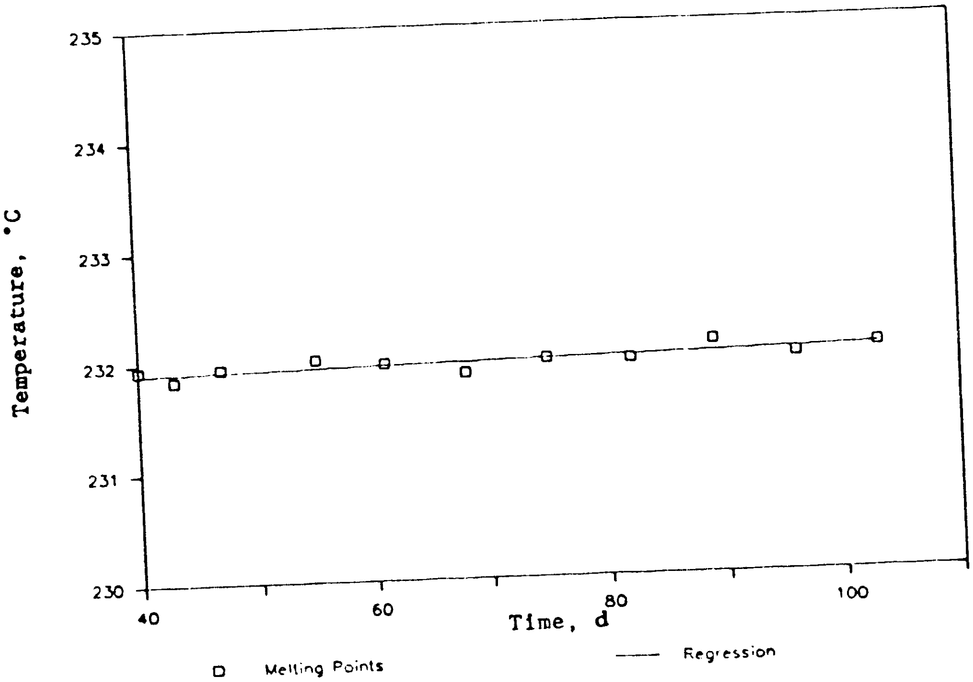

Fig. 6. Melting-point drift history of type K thermocouple with tin reference cell.

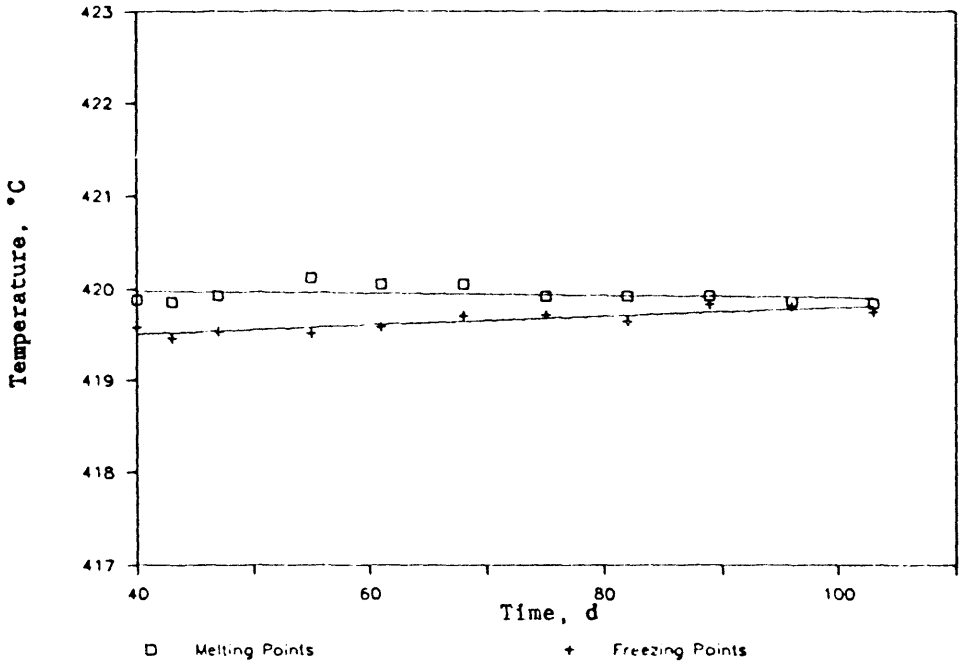

Fig. 7. Melting- and freezing-point drift history of type K thermocouple with zinc reference cell.

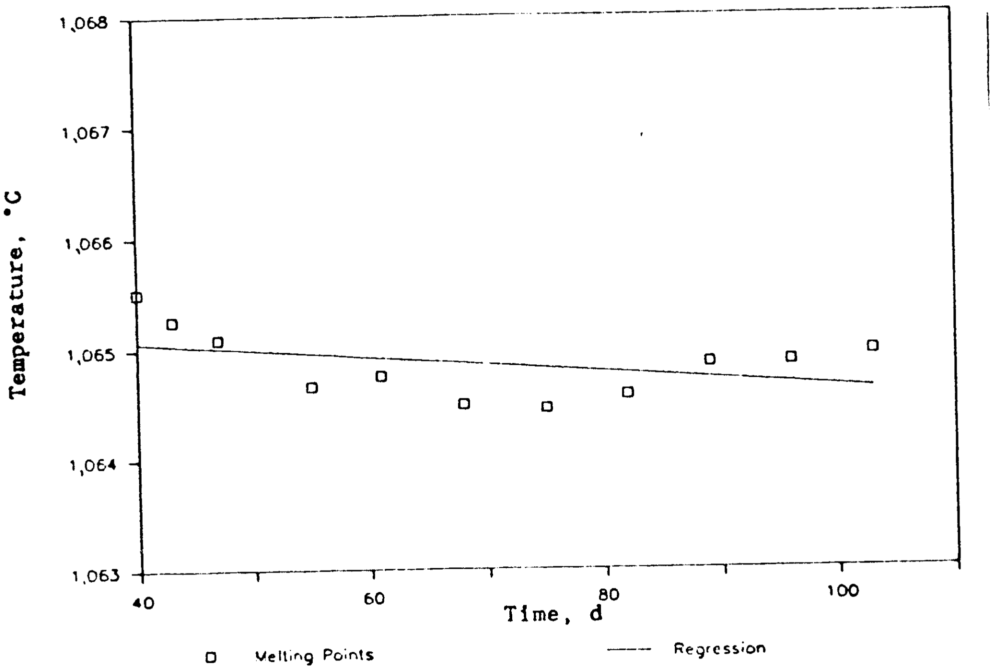

Fig. 8. Melting-point drift history of type S thermocouple with gold reference cell.

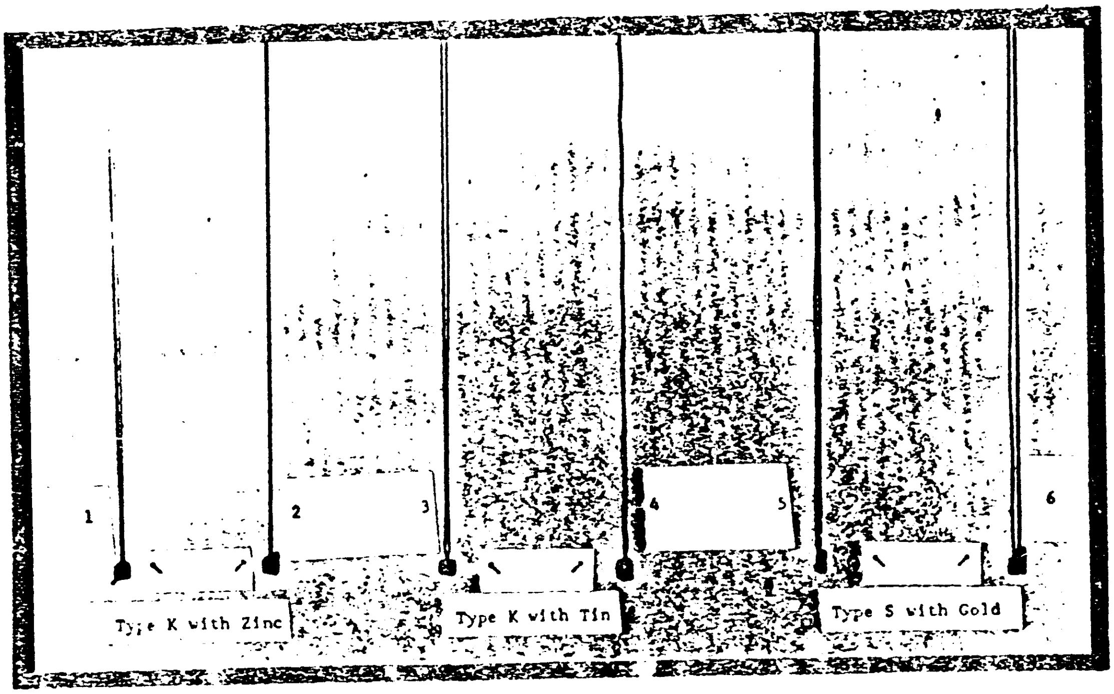

Fig. 9. X ray photograph of self-calibrating thermocouples.

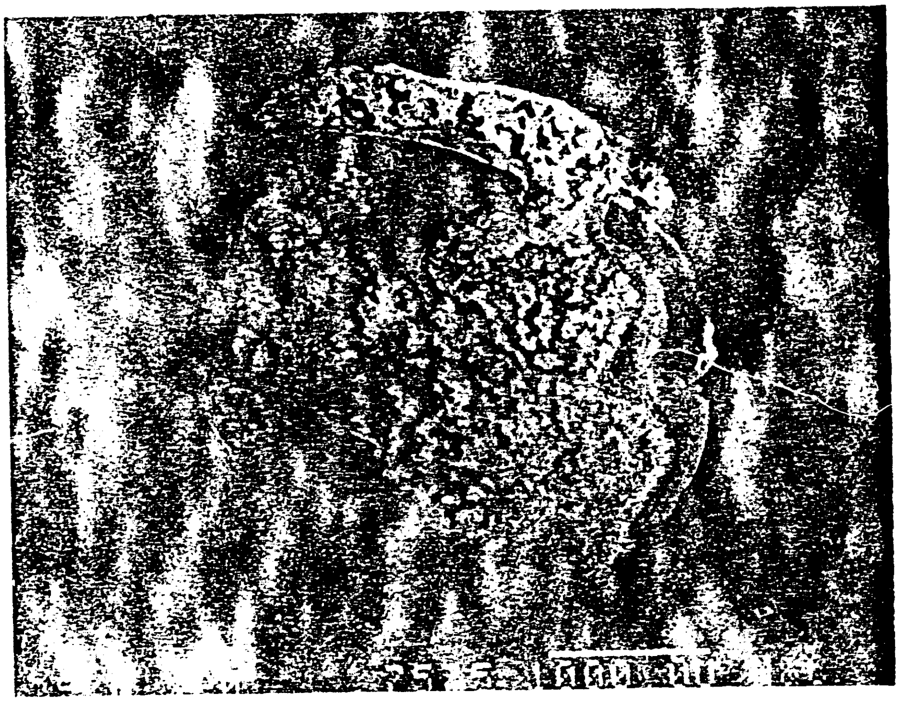

Fig. 10. Micrograph of defective self-calibrating thermocouple.

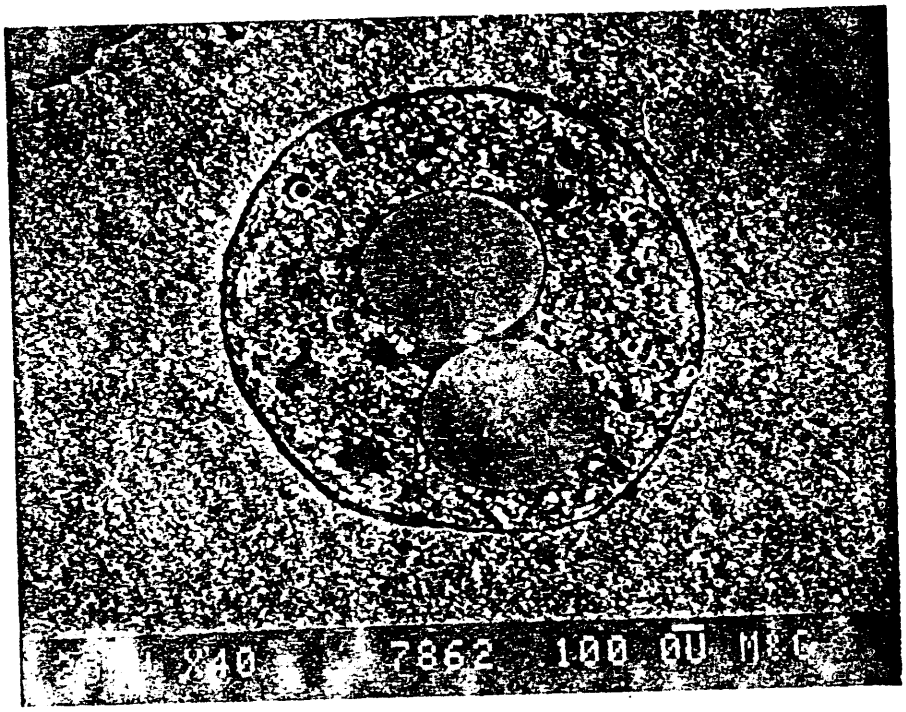

Fig. 11. Micrograph of tested type K thermocouple with tin reference cell.

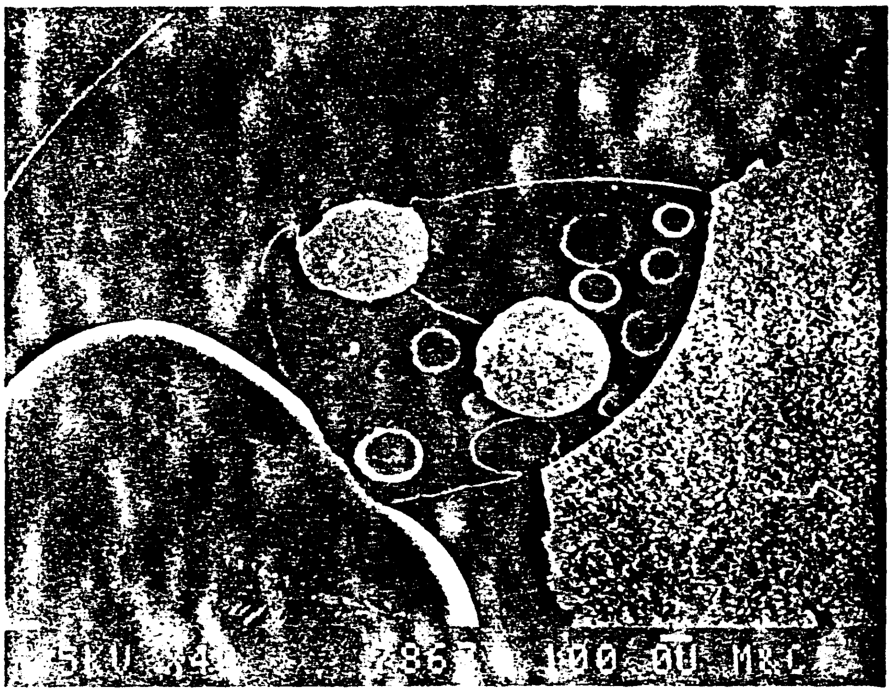

Fig. 12. Micrograph of tested type S with gold thermocouple.

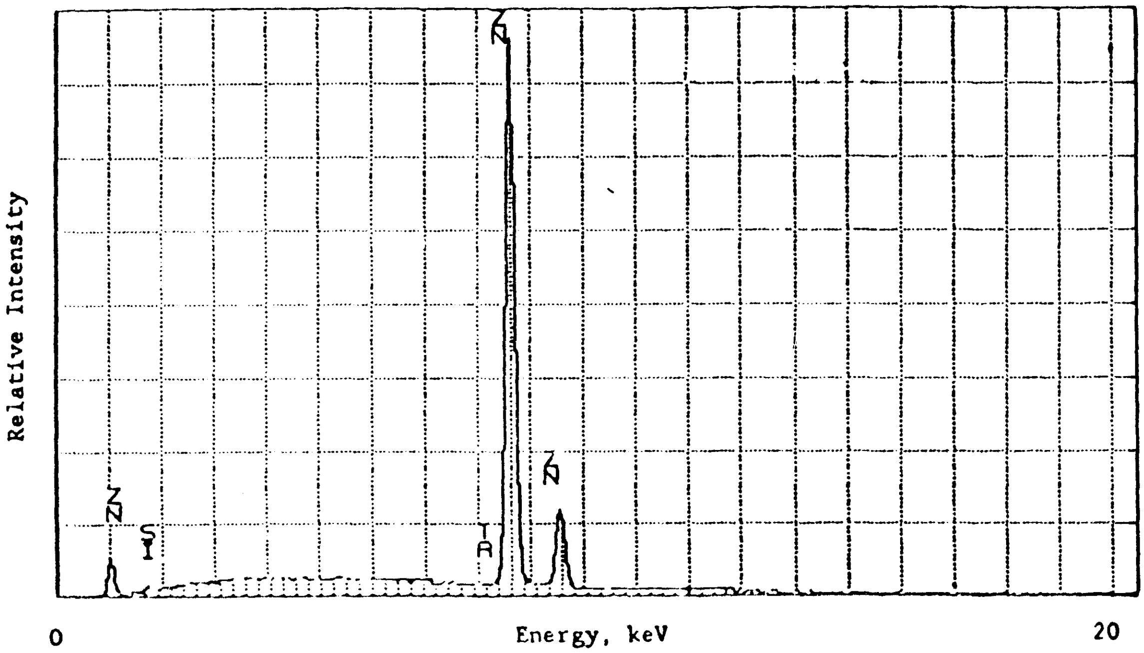

Fig. 13. Elemental spectrum of defective type K thermocouple with zinc reference cell.

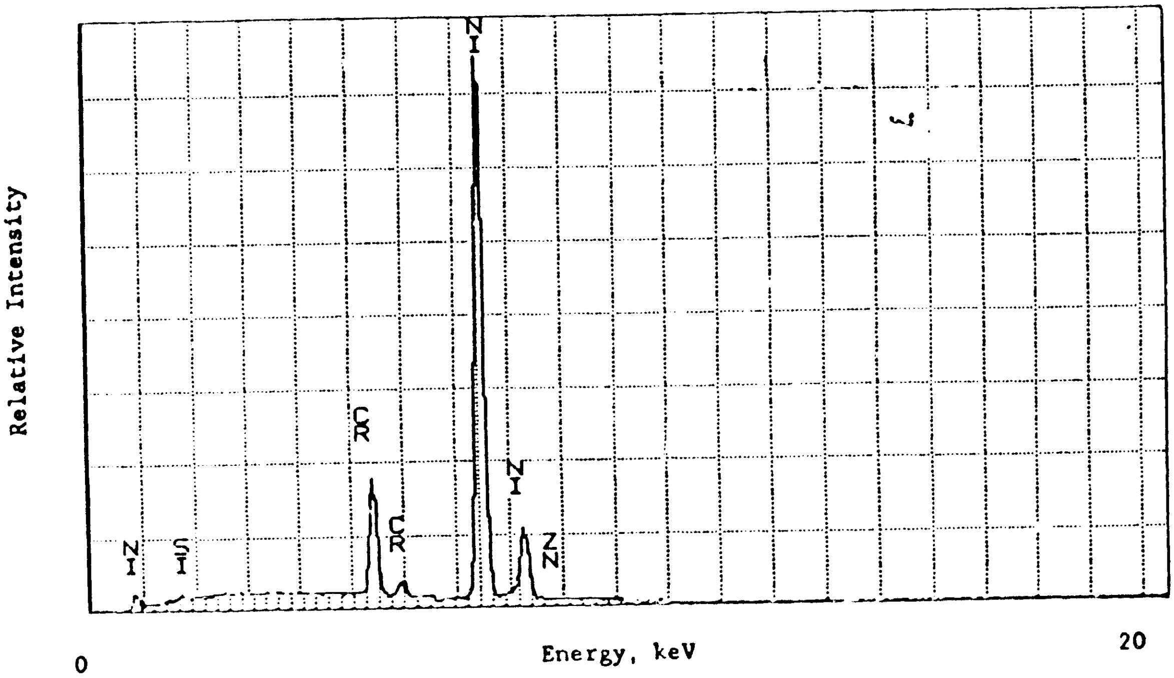

Fig. 14. Elemental spectrum of tested type K thermocouple with tin reference cell, Chromel wire.

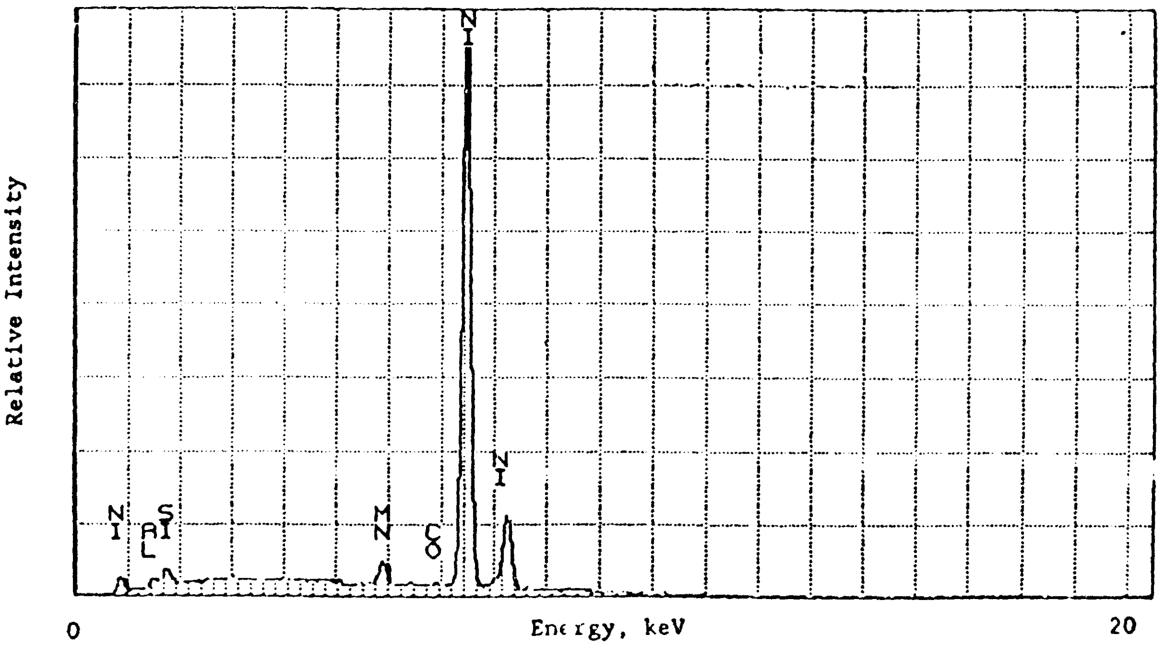

Fig. 15. Elemental spectrum of tested type K thermocouple with tin reference cell, Alumel wire.

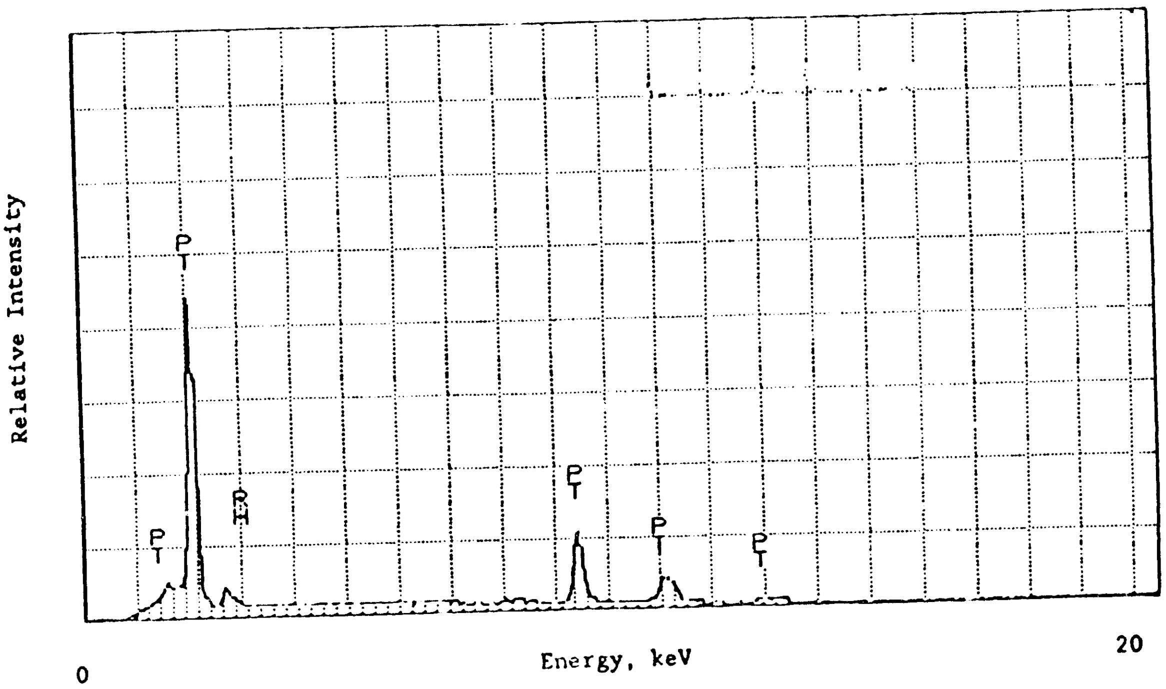

Fig. 16. Elemental spectrum of tested type S thermocouple with gold reference cell, Pt-10% Rh wire.

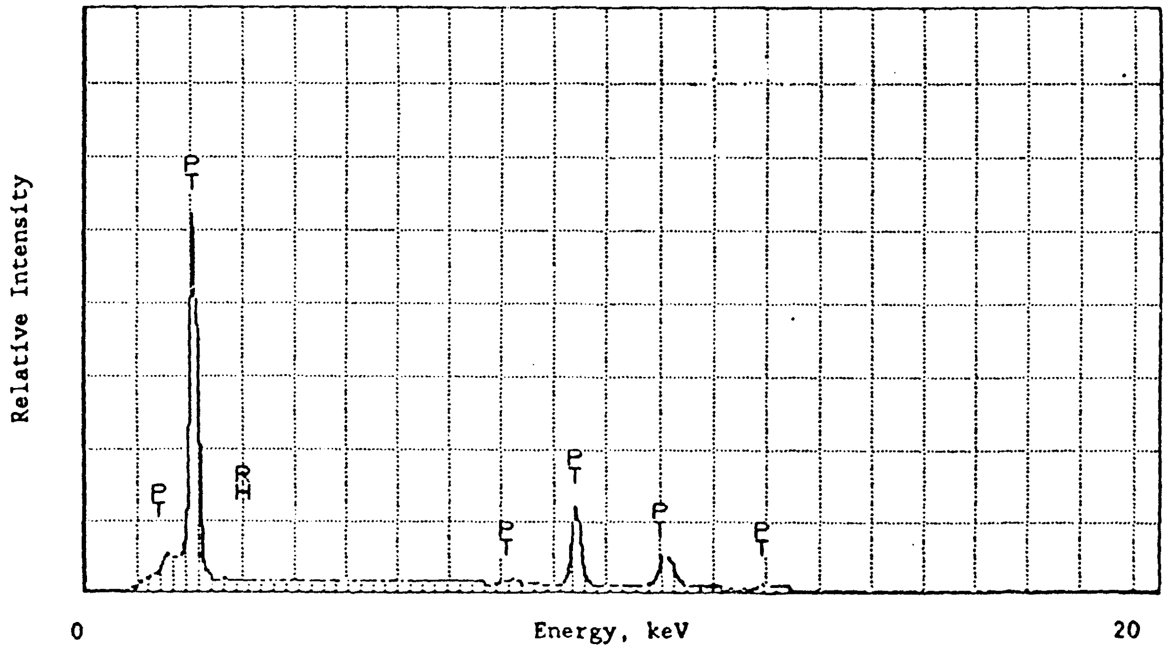

Fig. 17. Elemental spectrum of tested type S thermocouple with gold reference cell, Pt Wire.

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