Hybrid Microcircuitry for 300°C Operation

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Abstract—Microelectronic instrumentation for geothermal well logging must operate in ambient temperatures up to 300°C for several hundred hours. This study involved an extensive survey of 25 to 300°C operation of resistors, capacitors, conductors, interconnections, and active devices. Three major selection criteria were: 1) part lifetime of at least 1000 h at 300°C; 2) minimum change in electrical parameters from 25 to 300°C; 3) availability to the common circuit builder (no one of a kind). Certain thick film resistors, capacitors, and conductors were found compatible with such high-temperature operation. In addition, reconstituted mica and aluminum solid electrolytic capacitors were found useful up to 300°C.

Simple circuits for a geothermal temperature logging tool have been fabricated using these hybrid materials, components, and Si MOS and JFET devices. Oven tests show satisfactory stability from 25 to 300°C and at least 100-h circuit operation at 300°C.

I. INTRODUCTION

MOST existing commercial and military microelectronics is qualified for temperatures up to 125°C. However, for instrumentation in deep geothermal and oil wells the ambient temperature can easily reach 300°C. In this hostile environment, continuous 100–1000 h measurements are desired, thus simple insulation of the microelectronics package is not the engineering solution. The next most straightforward approach is the development of a microelectronics technology that works from 25 to 300°C, and has a circuit lifetime of at least 100 h at 300°C. For a high-temperature microelectronics technology to be of immediate and lasting value it must utilize only materials, components, and devices that are commercially available. Thus motivated and spirited, this investigation developed a limited but not excessively restrictive technology and set of parts which allow for 300°C microelectronics. Continuing development is creating more versatile electronics which will lend itself to other high temperature uses such as monitoring instrumentation on turbine blades, jet engines, nuclear reactors, *in situ* coal burns, and fossil fuel power plants. The work performed so far also helps chart directions for development of microelectronics up to 500°C [1].

Manuscript received February 25, 1977; revised May 16, 1977. was supported by the U.S. Energy Research and Development Administration.

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II. EXPERIMENTAL PROCEDURE

All commercial components were tested in a forced air convection oven with temperature uniformity and 24-h drift of ±0.5°C. Electrical leads were run from the components through openings in the oven wall to the measurement instruments which were: General Radio resistance bridge 1644-A, General Radio Capacitance Bridges 1615-A and 1611-B, and a Dana digital multimeter 5900. Typically, measurements were taken at temperature increments of 25°C starting at +25°C and going to +300°C. Aging was done both at a constant 300°C and in a cycling oven that held 300°C for 6 h and then slowly dipped to 100°C and returned to 300°C over a 2-h interval. Thick film circuitry and components were

 Annals No. 709PH010

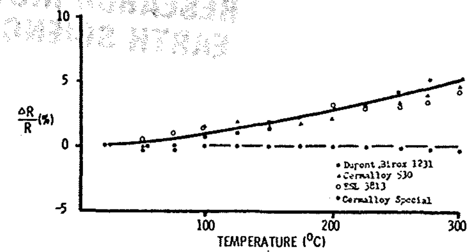

Fig. 1. Thick film resistance change as a function of temperature.

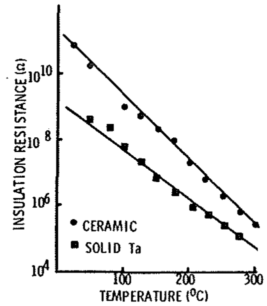

Fig. 2. Plot of the sharp decrease in IR as the ambient temperature is increased. Complex capacitor types like Semtech ceramic and Union Carbide tantalum capacitors did not display a logarithmic dependence on $1/T$ but rather on T.

fabricated with a standard screen printer (Affiliated Manufacturers Inc., CP-88) and belt furnace (BTU Engineering Corp., GP-11B). Ink manufacturers' printing, drying, and firing procedures were closely followed. Gold ribbon connections were made with a Hughes VTA-66 parallel gap welder and aluminum ultrasonic bonds with a Sola Basic Tempress EMB1100 bonder.

A number of time and money limitations were externally imposed on this survey. Although most types of microcapacitors and thick film inks were investigated, each type was purchased from only a few vendors. In addition, parts were tested in small batches of 10 due to oven space and time limits. All materials and parts used in this study were tested beyond manufacturer environmental specifications, and therefore no part of this report states or implies that one vendor's product is superior to another within their specifications.

III. RESISTORS

Three commercial resistor inks (DuPont 1231, Cermalloy 530, and Electro Science Lab 3813) were tested for their temperature coefficient of resistance (TCR) from 25 to 300°C and for resistor drift after aging at 300°C for 1000 h. These three ink samples performed roughly the same. Resistor ink 3813 was Pt Group metal based, the rest were ruthenium based. In addition, one nonstandard resistor ink with a special TCR adjustment was tested, Cermalloy 530 lot 2430. It is important to state that all other ink manufacturers contacted thought they could also mix special ink lots which would have a total change $\Delta R/R < 1$ percent from 25 to 300°C.

The change in resistance as a fuction of temperature is plotted in Fig. 1. The change in resistance of 5 percent from 25 to 300°C was typical; however, resistor characteristics are extremely sensitive to the exact ink lot and firing profile. In fact, during these tests several ink lots and furnace profiles showed only a 1.5 percent change over the temperature range. The three commercial inks extensively tested all had resistivities of 1000 Ω per square. Inks in the same commercial series but with 10 Ω per square showed a three times greater TCR.

Aging for 1000 h at 300°C produced no pattern of change in resistivity after an initial 24-h "stabilizing" period. All aging resistance drifts were less than 1 percent. During these life tests, dc voltage was applied to the resistors producing 1-10 W/cm² power dissipation. Using both an analytic heat flow model and thermosensitive paints, it was estimated that 0.2 W/cm² dissipation caused a temperature increase over ambient of 1°C in the thick film resistor.

Since most thick film inks are manufactured for use from approximately –50 to +125°C, the TCR is commonly optimized in this range. However, ink manufacturers can choose another temperature range for which to minimize TCR without much difficulty [2]. A special version of Cermalloy 530 was made with a minimum TCR of –9 ppm/°C at 150°C as opposed to +25°C. As a consequence, a total change in resistance of less than 0.2 percent from +25 to +300°C was measured. This Cermalloy thick film resistor material required a 100-h cure at 300°C during which 1 percent change in resistance occurred. After this cure a minimum TCR over the temperature range existed and less than a 0.2 percent change in resistance at 300°C occurred for the next 1000 h.

Several tests were performed above 300°C for thick film resistors. Stability in the range between 300 and 400°C was not well determined, although some tolerance to this temperature was observed. However, noticeable irreversible degradation was shown in all inks subjected for a 10-h period above 400°C. Quick catastrophic failure was observed above 500°C.

Another circuit component closely related to the resistor is the thick film thermistor. Firon thermistor inks TM-102 and TM-104 from Electro Materials Corporation of America were tested from 25 to 300°C, held at 300°C for 200 h, and rechecked from 300 to 25°C. At most, 1 percent resistance changes were measured at each temperature due to aging.

IV. CAPACITORS

An increase in the operating temperature can have a profound effect on capacitors. For example, in many simple dielectrics (those with one dominating activation energy) the insulation resistance, breakdown voltage, dissipation factor, and capacitor lifetime depend exponentially on inverse temperature [3]. In addition, ferroelectric materials ($BaTiO_3$) exhibit large changes in dielectric constant near their Curie temperatures. The more complex commercial capacitors tested in this survey also displayed precipitous changes in electrical characteristics with increased temperatures (see Fig. 2). Despite these discouraging tendencies, several capacitor types were found which function from 25 to 300°C. In this capacitor survey all insulation resistances were measured with 10 VDC and all dissipation factors at 60 Hz.

Standard microcircuits contain many different capacitor types: porcelain, multilayer ceramic brick, solid tantalum, and thin and thick film. Different types are needed for the many different voltage and capacitance ranges, and the different drift and temperature characteristics that are acceptable. In addition to the above-mentioned microcapacitors, this study considered two macrocapacitors: 1) reconstituted mica; and 2) solid aluminum electrolytic capacitors.

Initial high-temperature tests eliminated the porcelain and solid Ta capacitors. Several porcelain capacitor brands had dissipation factors of 30 to 60 percent and insulation resistance of only 0.004 MΩ-μF at 10 WVDC at 300°C. Solid tantalum capacitors underwent irreversible changes because of solid-state chemical reactions above 250°C. This increase in dissipation and capacitance occurred at a rate unacceptable for 1000-h operation.

Thin film and multilayer ceramic brick capacitors performed somewhat better at 300°C. Unfortunately, thin film capacitors (SiO_2 and Si_3N_4) were only commercially available with capacitance values less than 1000 pF, a range where thick film capacitors had proven more convenient. Multilayer ceramic brick capacitors appeared useful at least to 220°C. By choosing NPO dielectrics with high voltage ratings, limited circuit performance was achieved up to 275°C. The two basic problems found with multilayer ceramics were 1) low insulation resistance at 300°C, and 2) random drift in electrical characteristics above 250°C. Three ceramic capacitor brands which proved most dependable were: Semtech high voltage; Erie Red Cap series; and KD Components HV series.

For small value capacitances (up to 5000 pF) thick film capacitors had the best high temperature characteristics and were the easiest to incorporate into the hybrid. Using low dielectric constant inks ($K \sim 10$), thick film capacitors of 2 × 2 cm area were fabricated having a capacitance of around 1000 pf. The electrode metallization consisted of DuPont 8653, and the dielectrics tried were DuPont 8299, 8315, and 9428 and Electro Science Lab 4110 and 4301 (see Figs. 3 and 4). The highest yield was obtained when the dielectric was double printed with sample rotation between the printings. After each metal and dielectric printing the sample was fired. Yield with this geometry was more than 90 percent; in the case where dielectric pinholes were present a voltage discharge could be used to eliminate any short.

The ink with the highest yield and best high temperature characteristics was DuPont 8299. Figs. 5 and 6 illustrate the electrical properties as a function of temperature. At 300°C the dissipation factor was only 7 percent, the total capacitance change only 5 percent, and the insulation resistance 2 MΩ-μF. Aging for 1000 h at 300°C produced only 1 percent drifts in electrical characteristics. Tests were also run at 500°C. Although the thick film capacitors physically survived the 500°C environment for 2000 h and even showed little electrical degradation as measured at room temperature, at 500°C their insulation resistance was too low to be functional in a circuit. Voltage standoff was 900 VDC at 25°C and 600 VDC at 300 °C for a nominal dielectric thickness of 1.4 mils.

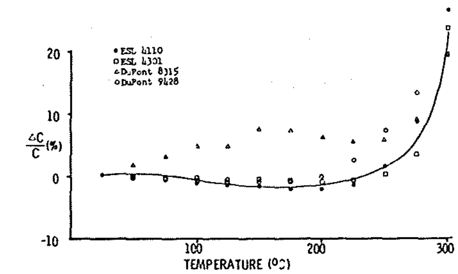

Fig. 3. Temperature changes of capacitance of thick film capacitors.

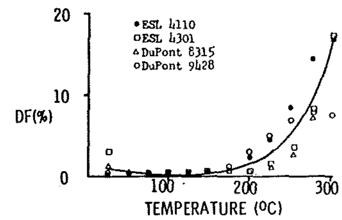

Fig. 4. Dissipation factor plotted as a function of temperature for various dielectric inks. Measurements made at 60 Hz.

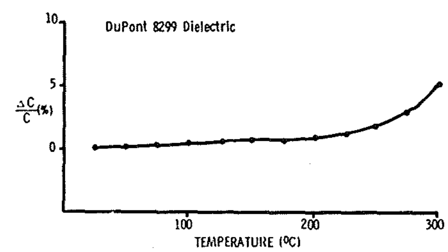

Fig. 5. In our hands DuPont 8299 dielectric ink gave the most consistent useful dielectric parameters. The capacitance changed only about 5 percent from 25 to 300°C. Because of a dielectric constant of 8–10, this ink is only useful for small capacitances.

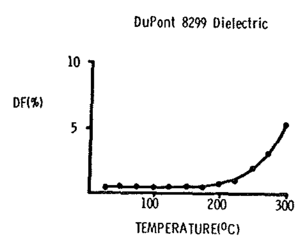

Fig. 6. Dissipation factors as a function of temperature for DuPont 8299 dielectric ink. (Frequency = 60 Hz.)

For capacitance values between 0.1 and 100 μF, no suitable microcircuit capacitors have been found. Solid tantalum capacitors irreversibly degraded a bit too fast and ceramic multilayers were a bit too leaky. Fortunately, two discrete capacitors provide the necessary function: reconstituted paper mica with silicone impregnation for the range 0.1–1.0 μF, and solid Al electrolytic for the range 1.0 to 100 μF. These capacitor formats carry the disadvantages of large size, wire leads, and larger expense. For example, a 0.1 μF paper mica 500 VDC capacitor has a volume of 4 cc and costs approximately $35.00. Figs. 7 to 12 show the temperature dependence of

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[4] B. Selikson and T. A. Longo, "Study of purple plague and its role in integrated circuits," *Proc. IEEE,* vol. 52, p. 1638, 1964. D. W. Bushmire, "Gold aluminum interconnect stability on thin film hybrid microcircuit substrates," SAND76-0306.

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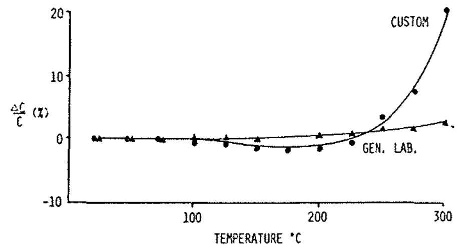

Fig. 7. Temperature change of capacitance of reconstituted paper mica capacitors from two companies. Capacitor from Custom was only rated to 260°C so this test is an over test. Custom P/N CHR3A1545SP, General Laboratory Associate 500 V/0.1 μF P/N 27932.

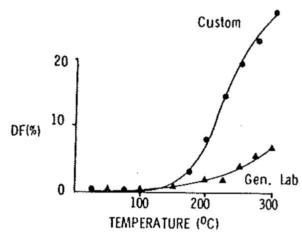

Fig. 8. Dissipation factor versus temperature for reconstituted paper mica capacitors. (Frequency = 60 Hz.)

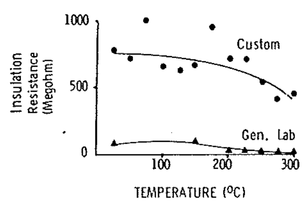

Fig. 9. Insulation resistance as function of temperature for reconstituted paper mica capacitors.

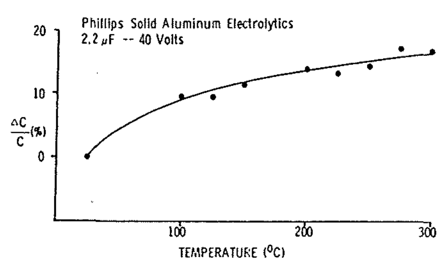

Fig. 10. Capacitance change plotted as a function of temperature for an Al solid electrolyte capacitor.

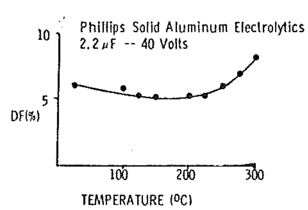

Fig. 11. Dissipation factor as a function of temperature of an Al solid electrolyte capacitor. (Frequency = 60 Hz.)

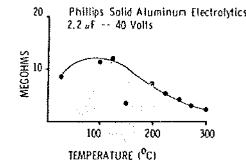

Fig. 12. Insulation resistance versus temperature for an Al solid electrolyte capacitor.

these capacitor electrical characteristics. Aging studies at 300°C showed a tendency of solid aluminum to drift ±5 percent over a 100-h period, whereas the paper mica drifted only ±2 percent. Paper mica are inherently built for high voltage, 500–2000 VDC; however, the solid aluminum is limited by the thickness of anodic oxide to about 40 VDC.

V. BONDING

Au-Au interconnections were used wherever possible to prevent harmful intermetallic formation at interfaces. Such bonds were tested for 1000 h at 500°C with no electrical or mechanical degradation. Unfortunately, many semiconductor devices do not come with Au metallization so it is necessary to bond Al wires to the thick film system. From thin film hybrid technology the Al-Au intermetallic problem is well known [4]. Three acceptable Al wire systems are currently being used for comparison. The first system employs Al wire bonded directly to the Pt-Au thick film conductor. A fritless Pt-Au conductor is preferred for ease of Al wire bonding although both the fritless 4121 Cermalloy and the fritted DuPont 8653 have been successfully tested. The hardness of the Pt-Au material caused excessive deformation of the Al bond pad, hence weak pull strengths. However, with this system no electrical degradation has been noticed on bonds after 300°C aging for 1000 h. The second system involves DuPont 9910 Au conductor ink which is made with special additives to reduce the Al-Au intermetallic problem. It is advertised that 1000-h tests at 150°C have produced little mechanical degradation [5]. The third approach involves a buffer material between the Au thick film and the Al wire. Ni discs 30 mils in diameter and 2 mils thick are used for this material matching purpose. The disc is Au plated on the side that is thermocompression bonded to the conductor thick film. Then the Al wire is ultrasonically bonded to the exposed Ni side. No bond strength degradation was noticed during 300°C aging, but again, the wire deformation was excessive. All three methods appear satisfactory in the small number of test circuits produced to date, but none of the systems give the strengths and endurance expected for standard hybrids.

Add-on component bonding and crossovers were made with Au ribbon (3 × 15 mils) parallel gap welded to the Pt-Au conducting layer. This interconnection has survived 1000 h at 500°C with no electrical or mechanical degradation.

VI. ACTIVE DEVICES

Although this study concerned passive components and interconnections, a complete high-temperature microcircuit

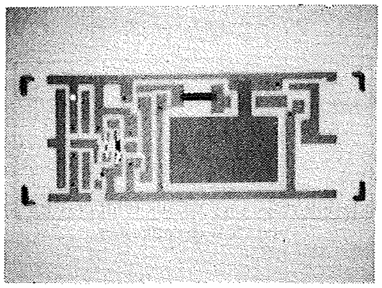

Fig. 13. Example of high-temperature thick film hybrid (×2). This comparator circuit consists of a Pt-Au conductor pattern (light gray), ruthenium resistor (black), crystallizable glass dielectric (white), Si MOS chips eutectically attached (small squares), and Au ribbon crossovers (left center).

must also have active devices. Unfortunately, the most common micro device, the Si bipolar transitor, could not be used because of excessive leakage above 200°C. However, four device types functioned well from 25 to 300°C and showed the necessary 1000-h high-temperature life. They were Si MOS, Si JFET, GaAs FET, and miniature ceramic vacuum tubes. The last two are manufactured for high-power microwave applications and are quite expensive, hence their incorporation has been avoided to date. All geothermal logging circuits have used the n- and p-channel MOS as switching devices and the n- and p-channel JFET's for linear functions. JFET's show little aging at 300°C and are therefore used whenever possible.

VII. CIRCUITS

Fig. 13 is a photograph of a high-temperature thick film microcircuit. A 250 pF thick film capacitor, a 2 kΩ thick film resistor, and 12 n- and p-channel Si MOS chips are interconnected to form a comparator. Conductor patterns were oversized so that 30-mil diameter Ni discs could be attached if necessary for high reliability Al bonding. All circuit work was done on 96 percent alumina substrates. In this particular circuit the crystallizable glass dielectric was DuPont 8299, the Pt-Au conductor DuPont 8653, and the resistor material DuPont 1231. The 15 × 15 mil Si chips were Si-Au eutectically attached and wired with 1 mil Al wire. Tests of several prototypes of this circuit showed drift due only to the slow aging of the MOS devices after 100 h at 300°C.

Although this high-temperature technology is not a completely general microcircuitry due to the absence of bipolar transistors and temperature stable 0.1 μF capacitors, a variety of useful circuits have been made: comparators; line drivers; simple operational amplifiers; and differential amplifiers.

VIII. REMAINING DEVELOPMENT

Several aspects of thick film technology must be further developed before a truly versatile high-temperature microcircuitry will exist. First, since most high-temperature Si devices have Al metallization, it is necessary to find or develop a thick film conductor which will retain good Al wire bonds for thousands of hours at 300°C. Secondly, high K dielectric material must be further developed in order to form thick film capacitors of 0.1 μF capacitance that retain an insulation resistance of at least 1 MΩ-μF at 300°C. To date thick film high K dielectrics that were tested ($K > 100$) have at most 0.1 MΩ-μF insulation resistance at 300°C and show considerable aging drifts. Third, low K thick film dielectrics must be developed with reduced total capacitance change from 25 to 300°C (less than 1 percent).

The current circuit lifetime of 100 h at 300°C might be upgraded to 10 000 h by careful pretesting of parts, use of passivating films, and hermetically sealing the hybrids. All thick film components and Si devices have been individually tested while powered for 1000 h at 300°C with little degradation. By protecting the parts with passivating layers of glass or Si_3N_4 and hermetic packaging, only internal diffusion or solid chemical reaction rates would determine lifetimes.

Extending the temperature range of circuit operation above 300°C may prove a formidable task. Although all selected passive components and interconnections operate at least to 325°C, at this temperature common Si devices have intrinsic thermally induced electrical conductivity exceeding extrinsic doped conductivity. Above 325°C commercial thick film resistor compositions show aging, losses and leakage in thick film capacitors become excessive, and Si devices are useless. With some materials development, a 400°C thick film technology for passive components and interconnections should be possible. Si will have to be abandoned, probably for GaAs FET devices or ceramic vacuum tubes, for operation from 300 to 400°C. For operation above 400°C much materials and component study would be necessary.

IX. CONCLUSION

A hybrid technology functional from 25 to 300°C has been demonstrated using thick film conductors, resistors, and capacitors on alumina substrates. Thousand-hour aging tests at 300°C produced at most 1 percent drifts in thick film component electrical characteristics. Temperature variation from 25 to 300°C produced as little as 0.2 percent resistance change in thick film resistors. Thick film dielectric material had at best 5 percent dissipation factor, 2 MΩ-μF insulation resistance, and 7 percent total capitance change at 300°C.

Reconstituted paper mica and Al solid electrolytic capacitors qualified for capacitance values greater than 0.1 μF. Si JFET and MOSFET chips bonded with Al wires were found compatible with these high-temperature hybrids. Component lifetime at 300°C in excess of 1000 h and circuit lifetime over 100 h have been observed.

Refinement of this hybrid approach should lead to greater circuit versatility and lifetime. Although microcircuitry is possible above 300°C, the present lack of commercial materials, components, and devices makes development difficult.

REFERENCES

[1] For more high-temperature electronics history see, D. W. Palmer and G. L. Knauss, "275°C microcircuitry: resistors, capacitors, conductors, substrates, and bonding," SAND76-0611, Sandia Labs, pp. 4-5.

[2] Private communication from Howard Imhoff of Cermalloy.