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FABRICATION OF CERMET FILM RESISTORS TO CLOSE TOLERANCES

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FIG. 1

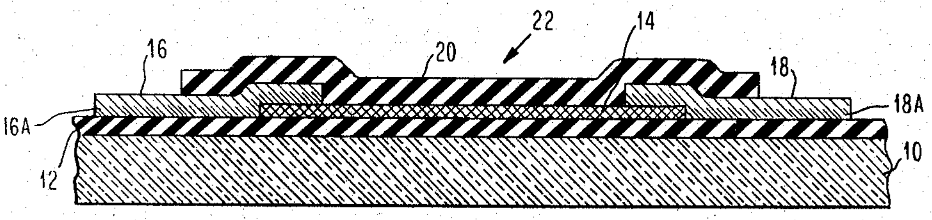


FIG. 2

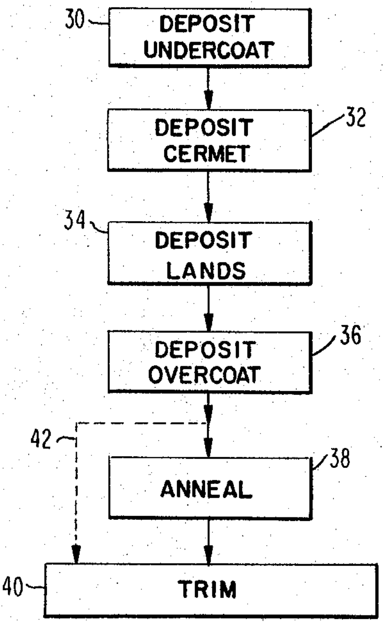
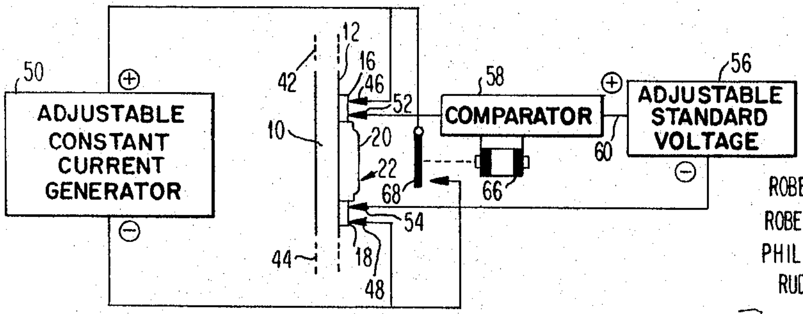


FIG. 3



INVENTORS
ROBERT L. BULLARD
ROBERT A. HASBROUCK
PHILIP S. SCHLEMMER
RUDOLF E. THUN

BY Frederick D. Poag
ATTORNEY

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FABRICATION OF CERMET FILM RESISTORS TO CLOSE TOLERANCES

Robert L. Bullard, Wappingers Falls, Robert A. Hasbrouck, Saugerties, Philip S. Schlemmer, Hyde Park, and Rudolf E. Thun, Poughkeepsie, N.Y., assignors to International Business Machines Corporation, New York, N.Y., a corporation of New York

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This invention relates to film electronics manufacture, and more particularly to the fabrication of film electronics panels having a multiplicity of cermet resistors wherein the ohmic value of the resistors is to be held to close tolerances. A prior, co-pending U.S. application, Ser. No. 182,818 filed Mar. 27, 1962 (now U.S. Patent No. 3,261,082 issued July 19, 1966) having a common assignee with this application, is directed to the general proposition of tailoring a film resistor by subjecting it to an electrical impulse. This application is directed to the peculiar problem of trimming cermet film resistors.

Cermet resistors, and more particularly chromium-silicon monoxide thin film resistors have found considerable acceptance in the manufacture of film electronic panels because such materials can be vacuum-deposited by methods and equipment which are compatible with other steps in the fabrication of such panels, and may be made to yield a resistivity of the high ohms per square desired for compact circuitry panels.

The manufacture of optimum or even successful cermet resistors is not without problems, however. It is difficult or impossible to deposit a myriad of such resistors on a circuit panel in such manner that each has precisely its planned geometry, thickness and composition. Moreover, the opportunities for selection or mechanical trimming in order to bring resistors into tolerance are limited since, on the one hand, it is often desirable that as many circuits as possible be deposited in an integral unit on a single substrate, and, on the other hand, the individual resistors are likely to be so small and so closely located adjacent to, over, or under other elements as to be difficult to work upon by any mechanical or other grossly obtrusive means. Also, cermet resistors have the peculiarity of being highly susceptible to absorption of elements from the air when at an elevated temperature. Accordingly, it has become a practice to encapsulate cermet film resistors completely in a protective material such as silicon monoxide at the earliest practicable step in the manufacturing process, even before the resistors are annealed.

The annealing process is provided to bring about changes in the internal structure of the cermets whereby subsequent exposure to operating or other anticipated environmental temperatures will not cause significant alteration in the ohmic value of the resistors. During the annealing the ohmic value of the resistors decreases toward their design value, but there is little opportunity to treat separate resistors of a multiplicity on a common substrate individually. Thus, the non-uniformity among the several resistors of a plurality on a common substrate occurring at the time of deposition is perpetuated through the conventional annealing step.

One might expect that since each resistor is encapsulated in silicon monoxide, it would be impracticable to attempt to apply heat to resistors on an individual basis. Since silicon monoxide is a good heat insulator, it might be expected that by the time externally applied heat had very much effect on one resistor it would also have an undesirable effect on its neighbors. On the other hand, one might expect the same insulating environment to result in confining the heat in a particular resistor in such manner that if heat were applied internally to such a resistor rapidly enough to avoid affecting its neighbors, hot spots within the resistor would result in its destruction and defeat for the method.

However, another characteristic of cermet resistors overcomes this difficulty. Since cermet resistors decrease in ohmic value upon application of heat, internally generated joule heat serves to effect the greatest alteration on the most resistive spots in the resistor so that hot spots tend to be eliminated and the application of such joule heat equalized everywhere within the resistor. In accordance with preferred embodiments of the present invention such joule heat is applied to the resistors on an individual basis, by an apparatus which makes contact with lands connected to opposite ends of a resistor for passing current through the same and includes automatic means for measuring the ohmic value of the resistor and terminating the joule current promptly as the design value is reached. In this way, adjustment of individual resistors can be undertaken with rapidity, both to expedite the procedure and to guard against unwanted propagation of the heat to adjacent elements, and yet without danger of overshooting the mark. The silicon monoxide encapsulation of each resistor provides an individual oven or enclosure for the cermet element thereof which not only provides a degree of heat isolation, but, more importantly affords an air-free environment for the cermet during the joule heat treatment.

Accordingly, it is a primary object of this invention to provide an accurate and facile means of producing cermet film resistors to very closely toleranced ohmic value.

It is another object of the invention to produce resistors as aforesaid in a manner which does not expose the resistors or associated or adjacent parts to seriously destructive or damaging influences.

It is yet another object of the invention to provide in a process as aforedescribed, an improved means of adjusting the values of individual cermet resistors in a multiplicity of such resistors on a single, integral, film electronics panel.

The foregoing and other objects, features and advantages of the invention will be apparent from the following, more particular description of the preferred embodiment of the invention, as illustrated in the accompanying drawing.

FIG. 1 is a cross-sectional view of a cermet film resistor which may be brought into closely toleranced ohmic value in accordance with the present invention;

FIG. 2 is a schematic diagram of a typical manufacturing process for preparing the resistor of FIG. 1, including the final step of adjusting or trimming its resistance in accordance with the present invention; and

FIG. 3 is a schematic diagram of an apparatus which may be employed to treat and monitor the resistor of FIG. 1 in accordance with the process step of FIG. 2.

The resistor shown in FIG. 1 is an example of the film electronic structures the resistive element of which can be brought into closely toleranced stable values in accordance with the invention. Typically, the structure may include a glass or other suitable substrate **10** which forms a mechanical base upon which has been deposited a layer of silicon monoxide **12** over which the cermet resistance material **14** is laid down. Copper or other suitably conductive terminals **16, 18** provide electrical connection to the opposite ends of the resistance film **14**, and an overcoat of silicon monoxide is provided which cooperates with the undercoat **12** to complete encapsulation of the resistance element **14**. Thus, the cermet of the resistance element **14** is protected from the action of the atmosphere.

The techniques for preparation of film electronics elements such as shown in FIG. 1 are well known and it is also well known that the exercise of care in the deposition of the various layers can result in reasonably repeatable results, that is, resistors which have, after annealing, an ohmic value within five or ten percent of their planned or nominal value. The substrate **10** utilized may be glass polished to a surface roughness of fifty angstrom units and an over-all flatness of 1/1000 of an inch across the working area of a substrate which will receive a multiplicity of such resistors, typically four by five inches in dimension. All of the layers are deposited by evaporation in vacuo, and process parameters such as vacuum chamber pressure, substrate temperature, evaporator geometry, evaporation rate, film thickness and film composition are closely controlled. Thus, the chamber pressure may be monitored with ionization gauges, and substrate temperatures measured with thermo-couples and controlled to within 10° C. by appropriate heater control circuitry. A chromium underflash **16***a*, **18***a* may be used prior to deposition of the copper terminal or land elements **16**, **18** to assure good adhesion, and evaporation rates for these metallic parts and the silicon monoxide encapsulating layers **12**, **20** may be controlled by an ion gauge rate monitor of the type having a heated collector for continued accuracy during its operation. The thickness of the resistance film **14** itself can be estimated by utilization of a test slide within the evaporation chamber which is connected to a resistance meter.

Typically, the silicon monoxide is deposited at a substarte temperature of 350° C., a pressure of 2×10^{-6} torr, and a deposition of 25 angrstrom units per second. Chromium and copper for the elements **16**, **18** may be deposited at a substrate temperature of 160° C. and a pressure of 5×10^{-6} torr, the chromium underflash being sublimed from an open tungsten boat at 3 angstrom units per second and the copper deposited thereover from a radiantly heated baffled carbon crucible at 10 angstrom units per second. The most critical element, the cermet film **14** itself, may be deposited by flash evaporation of mixed chromium-silicon monoxide powders at a substrate temperature of 160° C. and a pressure of $5-10\times10^{-5}$ torr. As one example, the resistive film **14** may have a resistivity of 300 to 400 ohms per square as deposited, which resistivity is reduced to about 250 ohms per square by annealing. Such a cemet film may be formed from 70 atomic percent chromium and 30 percent silicon monoxide and may be in the order of 90 to 100 angstrom units thick. However, by varying the atomic percentages of chromium and silicon monoxide and thickness of the deposit, resistivities ranging from 10 ohms per square to megohms per square may be fabricated.

Referring to FIG. 2, the foregoing typical deposition steps may be summarized as a first step **30** of depositing a 5,000 angstrom unit underlayer **12** on a clean substrate **10**, followed by a second step **32** comprising the flash evaporation of premixed chromium-silicon monoxide powders, 70 atomic percent chromium-30 percent silicon monoxide, to form the cemet film **14** having an unannealed resistivity of 300 to 400 ohms per square. In the next step **34**, 5,000 angstrom units of copper with a 300 angstrom unit underflash of chromium are deposited to form the land or terminal elements **16**, **18** and then in step **36**, a 10,000 angstrom overlay **20** of silicon monoxide is deposited over the resistor material **14**. All of the steps to this point may be undertaken in a continuous process vacuum apparatus with masks brought into place at the various stages of the operation to define the geometries of the several layers. In any case, it is preferable that the cermet element **14** be covered and encapsulated by the silicon monoxide layers **12**, **20** before ever being exposed to the air at an elevated temperature. It is a feature and advantage of the present invention that no modification or interruption of this encapsulation is necessary to achieve closely toleranced results.

In the preferred embodiment of the process of the invention, as in the prior art, the foregoing steps are followed by an oven annealing step **38** which is carried out at about 350 to 475° C. in a reducing atmosphere of "forming gas" (10% hydrogen, 90% argon) to protect the exposed copper land parts **16**, **18** until a reduced, operationally stable resistance value is reached. This so-called annealing step brings about changes in the cermet as a function of the time and tempearture of the annealing. The resulting gradual reduction of the resistivity of the cermet is monitored during the annealing, and the product pieces are taken out of the oven when a certain resistivity is reached. This heat treatment has been found to result in resistors whose value is stable at operating temperatures below the annealing temperature.

In the prior art, the annealing step aims to achieve the design or nominal resistivity of, for example, 250 ohms per square ±5%. In accordance with the present invention, the annealing is stopped somewhat short of the goal, such as at the nominal resistivity +10/—0%, and the final adjustment to the design or nominal resistivity is made by the trimming step **40** of the invention. Alternatively, the annealing step **38** can be omitted altogether, and, as indicated by the dotted line **42** in FIG. 2 the entire heat stabilizing and adjusting process can be carried out by the joule heat procedure of the invention.

The trimming step **40** is carried out by passing current through the resistor so as to produce joule or resistive heating therein in such concentration and for such a time as to bring the stabilized resistance of the cermet element to, or closer to, the design or nominal objective for this value. This is accomplished by connecting the resistor into suitable circuitry by use of its own terminals, without in any way disrupting the silicon monoxide overcoat **20** which protects the cermet film **14** of the resistor device **22**. Thus, the silicon monoxide undercoat-overcoat encapsulation **12**, **20** continues to protect the cermet film **14** during joule heating of the same pursuant to the trimming step.

This procedure may be carried out with facility by use of an apparatus of the kind shown in FIG. 3. It will be understood that the resistor **22** to be trimmed may be, and usually is, one of a multiplicity on a single substrate **10**, the substrate **10** being for this purpose extensive compared to the area covered by a single resistor **22**, as indicated by the broken line portions **42**, **44** thereof. Thus, the apparatus of FIG. 3 may be duplicated a plurality of times in a jig adapted to treat the number of resistors **22** on a single substrate at the same time. It is nevertheless desirable that the treatment of each resistor be an individual matter, for enabling close, individual trimming of each resistor. Thus, the circuit of FIG. 3 is typical of that which may be employed for treatment of a single resistor or with others like it in a jig, for simultaneous treatment of a plurality of resistors.

In the apparatus of FIG. 3, the power for joule heating is applied to the resistor through contacts **46**, **48** which bear against the exposed surfaces of the resistor terminals **16**, **18** for connecting them to an electrical power source preferably in the form of a constant current generator **50**. As the cermet film **14** within the resistor **22** is subjected to the resulting joule heat, during the trimming operation, its resistivity will decrease. Therefore, a constant current generator is a preferred form of power supply since the rate of resistance heating will decrease somewhat as the nominal or final resistance value is approached, rather than the converse which would occur in the case of a constant voltage supply. For minimizing inaccuracies due to contact resistance, it is preferred that the resistance value of the element **22** being trimmed be monitored through a separate pair or containers **52**, **54** which also contact the resistor terminal lands **16**, **18**. The several contactors **46**, **48**, **50**, **52** may be in the form of stainless steel rods the ends of which have been lapped to a smooth, transverse, planar shape, and are spring-mounted so as to press firmly against the terminal lands **16**, **18** of the resistor.

The resistivity monitoring contactors **52**, **54** are connected in a circuit which may include a standard voltage source **56** and a comparator circuit **58**. The polarity relation of the output of the standard voltage source **56** is made the same as that of the current generator **50**, as indicated on the drawing, and the function of the comparator circuit **58** is to yield an output whenever the potential at contactor **52** drops in magnitude below that at output line **60** of the standard voltage source. Any convenient circuit can be used for the comparator **58**; as a simple example a high gain, saturating amplifier could be utilized, as will be obvious to those skilled in the electrical arts. The output of the comparator **58** controls a shunt **62**, **64** across the current generator **50**. Thus the output device of the comparator may be in the form of the solenoid **66** of a relay having a normally open contactor **68** in the shunt circuit **62**, **64**. The same apparatus may be utilized to trim resistors of various design or nominal resistivity, and accordingly the standard or reference voltage supply **56** is preferably adjustable. So also, since resistors may vary not only in ohmic value but in other parameters such as area, it is desirable that the constant current generator **50** be adjustable for enabling selection and/or approximate standardization of the power dissipation rate to be applied to different resistors.

Thus, an apparatus is provided whereby a desired rate of joule heating may be applied to the resistor **22** to be trimmed, and which terminates promptly the application of that power when the potential dfference across the resistor falls to that across the standard voltage of the monitoring apparatus, in other words, when the resistance of the device **22** on being trimmed falls to the desired value.

A number of parameters enter into selection of the optimum rate of joule heating to be applied to a particular resistor. The general objective is to raise the temperature of the cermet film of the resistor well above its annealing temperature so as to bring its resistivity to the desired value in a short period of time, both for speeding the operation and to minimize unwanted conduction of heat to adjacent elements. At the same time, it is desired that the application of heat not be so great as to seriously burn the terminals **16**, **18** or damage surrounding elements by heat, shock or otherwise. As a general rule, it has been found that a heating rate of about one killowatt per square inch of cermet being treated is usually satisfactory for producing marked and rapid adjustment of the resistivity of most cermets without serious damage. The substrate glass is usually of the heat shock resistant kind, and any slight discoloration of the terminal lands **16**, **18** can be cleaned off afterward with a suitable etch. In a few cases, as shown in some of the examples in Table I below, it is necessary to go to higher heat application rates in order to "move" the value of the resistance. These conditions are encountered in the cermet mixes having a very high proportion of chromium such as the 90 percent chromium cermet or in cases where the resistivity as deposited has required long, high temperature annealing.

Table I is a correlation of data, showing the interrelation of certain parameters in the carrying out of the process in accordance with the invention. In the cases of sample #3, 4 and 7, the resistors were left in the hot vacuum chamber after deposition of the SiO overcoat until they had reached a reduced, substantially stabilized value. Thus, the "as deposited" resistivity was not measured, and there is no oven anneal data, per se.

TABLE I

No.	Deposition					Anneal			Trim		Final R./Sq.
	Cr/SiO Mix, at. percent	Area, in.²	Length/ Width Ratio	Nom. R./Sq., ohms	Act. R./Sq., ohms	Time, Hrs., Min.	Temp., ° C.[1]	R./Sq., ohms	I, ma.	Nom. Power, W./in.²	
1	90/10	0.002	0.5	40	54.8	2:40	400	45.6	173	3,000	43.8
2	90/10	0.0012	.83	40	53.2	2:40	400	46.2	100	2,750	40.3
3	90/10	0.0024	1.67	40	----------	----------	----------	43.9	100	2,750	40.1
4	90/10	0.0036	2.5	40	----------	----------	----------	43.8	85	2,000	41.1
5	60/40	0.0176	0.227	150	(2)	7:00	450	152	224	1,000	150.1
6	65/35	0.0143	3.57	150	(2)	1:45	450	151.4	51.7	1,000	150
7	50/50	0.008	2	500	----------	----------	----------	551	28	1,000	506
8	50/50	0.008	2	500	861	2:00	450	523	40	2,000	502
9	50/50	0.0418	1.67	600	1,342	0:30	350	770	65	1,000	609
10	50/50	0.025	1	1,000	1,866	1:10	400	1,092	50	1,000	1,023
11	50/50	0.025	4	1,000	1,988	1 hr.±	400	1,046	25	1,000	1,039
12	50/50	0.094	.953	1,000	(2)	22:13	450	1,100	311	1,000	1,058
13	50/50	0.133	0.19	1,000	(2)	22:13	450	1,110	254	1,000	1,068
14	50/50	0.15	0.375	1,000	(2)	22:13	450	1,129	200	675	1,120

[1] The anneal temperatures were equal to or up to about 25° C. higher than the figure given.
[2] No reading.

Since the cut-off of the trimming current is automatic, the duration of the trimming current was not measured. It is ordinarily less than two seconds. In Example #1, however, the high-chromium cermet did not trim into a closer approach to its nominal value of 40 ohms per square with application of three kilowatts per square inch for several minutes, and therefore trimming was discontinued manually to avoid destruction of the resistor. This example is included to show the remarkable heat dissipating capability of these resistors.

An experiment was conducted for the purpose of estimating the time-temperature relation which exists during the trimming of cermet resistors in accordance with the invention. The results were consistant with the conclusion that the mechanism of the trimming step is that of a further, higher temperature annealing—what one might call "joule annealing" as opposed to oven annealing as above described with respect to step 38, FIG. 2, or the variant of that oven annealing which occurs when the workpiece is kept hot in the deposition vacuum chamber as in the case of Examples 3, 4 and 7 of Table I.

This experiment is reported in Table II below. All of the sample resistors were on a single substrate and were of 50/50 at. percent Cr—SiO cermet of nominal resistivity of 1000 ohms per square. They were undercoated with 10,000 A. of SiO and overcoated with 10,000 A. of SiO. They had been oven-annealed for a total of 31½ hours at about 450 to 475° C. A bit of the indicator was placed on the SiO overcoat, about midway over the cermet element, and trimming current was applied to the resistor until the indicator was observed to melt. The trimming current was calculated to produce joule heating of one kilowatt per square inch at the nominal resistivity; therefore the actual power applied was somewhat above that amount.

TABLE II

Sample	Nominal Resistance	Act. Resistance, ohms		Trim. Time to melt indicator	Indicator	Melt Temp., °C.
		Before Trim	After Trim			
A	4,000	4,635	4,213	14 sec	Al	660
B	4,000	4,722	4,519	20.5 sec	Al	660
C	4,000	4,622	4,611	0.25 sec	$CdCl_2 \cdot 2\frac{1}{2}H_2O$	568
D	1,000	1,173	1,159	0.25 sec	$CdCl_2 \cdot 2\frac{1}{2}H_2O$	568
E	4,000	4,730	4,727	Instantaneous	Pb	327
F	1,000	1,167	1,167	do	Pb	327
G	1,000	1,164	1,130	1.3 sec	Zn	419
H	4,000	4,681	4,610	3.8 sec	Zn	419
I	4,000	4,717	4,713	1.2 sec	CuCl	422
J	1,000	1,059	1,057	1.1 sec	CuCl	422

Discounting Sample #H as experimental error, it will be seen that the outer surface of the SiO overcoat where the indicator was placed rose to the vicinity of the oven annealing temperature in well under two seconds. In the case of Samples #C and D, a rise to about 100° C. over the oven annealing temperature was indicated in only about a quarter second, perhaps because the cadmium chloride indicator was in the form of flat crystals making better thermal contact than the aluminum or lead, which were in chip form, or the zinc or cuprous chloride which were in powdered form. Of course, the cermet itself would in every case be hotter than the indicator, since the SiO therebetween is an insulator. Thus, it is believed clear that the trimming is brought about by an annealing action in which the cermet is subjected to a temperature higher than its previous annealing experience.

From the foregoing, it will be seen that the invention provides a means of treating cermet resistors in a most flexible manner. Not only can each resistor of a multiplicity on a substrate be treated separately from its neighbors, but also the scope and precision of the treatment is such as to make up for wide variation in the process steps preceding it and to enable relaxation of some of the controls of those steps. Thus, instead of attempting to carry out the oven annealing steps of the prior art to maximum accuracy, it is feasible to stop the oven annealing considerably before the nominal resistivity is reached, leaving all of the final accuracy to the trimming or joule annealing step of the invention.

Table I above was assembled from data which show application of the invention under a wide variety of conditions. Table III, below, shows more typical results which illustrate the uniformity and reproducibility of the process.

Table III relates to a single panel having 48 resistors of 50/50 atomic percent chromium-silicon monoxide cermet which had been oven annealed at 350–390° C. for one-half hour. The cermet film had a nominal resistivity of about 300 ohms per square and the resistors were of 2:1 length-width ratio, or 600 ohm nominal resistance. The variation of the 48 untrimmed resistors was from 616 ohms to 702 ohms, or 86 ohms, while the values for the same resistors after trimming were found to be 600, +1/−2 ohms or a total variation of 0.5%.

TABLE III

Example No.	Resistance as deposited	Resistance as annealed	Resistance as trimmed
a	769	628	600
b	799	638	600
c	791	631	600
d	849	699	600
e	841	702	601
f	839	670	599
g	849	682	598
h	839	674	601

It is possible where desired to omit the oven annealing as a separate procedure and substitute the relatively crude but sometimes more convenient technique of utilizing so called "vacuum annealing," for example as in the cases of Samples #3, 4 and 7 in Table I. It will be recalled that the substrate temperature during the final deposition of silicon monoxide is about 375° C., so that retention of the workpiece in the hot vacuum chamber after the deposition of that layer for thirty minutes or so produces a degree of annealing which can be finished by the further annealing of the trimming step of the invention. As still another option, the entire annealing process can be performed by the joule heating of the invention without any substantial anneal step therebefore, as indicated by line 42, FIG. 2.

While the invention has been particularly shown and described with reference to preferred embodiments thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of the invention.

What is claimed is:

1. A method of fabricating a cermet thin film resistor to a predetermined design resistivity comprising

providing a substrate and forming thereon conductive terminal elements and a chromium-silicon monoxide cermet film in lapped relation to form an electrical circuit path between said elements through said film, said film having a resistivity greater than said design resistivity and being encapsulated completely in silicon monoxide,

then annealing said film by subjecting the same to a temperature of about 400° C. in an oven until the resistivity of said film approaches said design resistivity,

and finally heating the cermet film circuit element to a further annealing temperature by passing electrical current through said element until the resistivity of said film reaches said design resistivity.

2. A method of fabricating a cermet thin film resistor to a predetermined design resistivity comprising

providing a substrate and forming thereon conductive terminal elements and a chromium-silicon monoxide cermet film in lapped relation to form an electrical circuit path between said elements through said film, said film being in the order of one hundred angstrom units thick and having a resistivity greater than said design resistivity and being encapsulated completely in silicon monoxide,

then annealing said film by subjecting the same to a temperature of about 400° C. in an oven until the resistivity of said film approaches said design resistivity, and finally heating the cermet film element to a further annealing temperature, while monitoring the resistance of said element, by passing electrical current through said element sufficient to produce joule heating of about one kilowatt per square inch in said element until the resistivity of said film reaches said design resistivity.

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JOHN F. CAMPBELL, *Primary Examiner.*

WHITMORE A. WILTZ, *Examiner.*

W. I. BROOKS, *Assistant Examiner.*