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Electrical properties of multicomponent CMM in correlation with their microstructure

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Abstract

In this work, electrical properties (resistance, resistance temperature coefficient, thermoelectric voltage, current–voltage curve) of multi-component ceramic matrix materials have been investigated. The influence of the structure of discrete particles on the microstructure and properties relations of CMM composites has been studied. Three groups of resistive materials were analyzed: (a) high resistance matrix and percolation clusters; (b) ionic or (c) covalent ceramic matrix and metal-like percolation clusters.

The percolation threshold in composite materials is defined as the composition at which the conducting phase forms a continuous three-dimensional network through the material. However, in typical CMC we have observed a multi-threshold situation: at some content of conducting particles the first percolating clusters are formed; when the number of particles increases, clusters are destroyed to be created again at some higher content of conducting particles. These processes are strongly dependent on the starting materials properties, as well as on the thermal treatment of composites.

Based on this approach a new product has been designed. Much attention was paid to the particulate structure and processing. A CMM with the resistance ratio $R_{800}/R_{20} = 1.4$ has been manufactured. Intensive resistance growth begins at 700 °C and the maximum is achieved at 1100 °C.

Keywords: Ceramic matrix materials; Silicon nitride; Zirconium carbide; Multicomponent composite; Electrical resistivity; Percolation; positive temperature coefficient of resistance; Energy savings

1. Introduction

Studies of the electrical behavior of multi-component CMM are not numerous and their results are not yet sufficient to design and engineer technical components with property levels higher than those of the individual constituents. Most of the work on CMM composites was done to improve materials mechanical properties, but few to improve their electrical behavior [1,2] by studying the electronic conductivity as a function of composition. Even less attention was given to the influence of dispersed particles microstructure shape and evolution on the electro-physical processes of multi-component systems [3]. The main question concerning the development of advanced resistive CMMs is the prediction of their effective electrical properties, starting from composition and size to focus on their microstructure evolution during the technological cycle and service. This is particularly necessary for the development of advanced, highly reliable and power saving devices in applications such as infra-red radiators, gas igniters, high-temperature super high frequency absorbers, air and fluid heaters in the intensive heat exchange technology.

All the varieties of resistive materials can be sorted into three different groups:

(a) Electro-ceramics with perovskite structure and positive temperature coefficient of resistance (PTCR). The development of these materials contributed to improve the low-temperature heating technology. Unfortunately, the area of application of these materials is limited to temperature levels up to 180 °C, although sometimes the working temperature for the materials with high Curie point can reach 250–300 °C.
(b) Resistive materials based on ionic ceramic matrix (ZrO_2, Al_2O_3, SiO_2) with the addition of metallic (Ti, Ni, Mo) or metal-like ($MoSi_2$) conductive particles [1,2]. These composites are also of interest because they may exhibit simultaneously high conductivity and high fracture toughness.

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(c) Resistive materials based on a covalent ceramic matrix (Si_3N_4, SiC) with additives made of nitrides, silicides and borides of transition metals (TiN, TiC, $MoSi_2$, TiB_2, ZrB_2) [4]. These materials are suitable to withstand working conditions at high temperature and aggressive mediums, and also to endure the large thermal surface loads often higher than what resistive materials based on ionic matrix could bear. A special place within these materials is given to the gradient-ceramic heaters, obtained by embedding thick-film resistive layers between isolating ceramic substrates [5].

Significant improvement of this last group of resistive materials was achieved with the application of the gradient-concept to all-ceramic heaters Si_3N_4–TaN [6] made with the resistive layer, 0.5–2.0 mm thick, placed between two other layers of thermo shock resistant Si_3N_4–Si_2ON_2 isolating composite. The new resistive materials exhibited a PTRC effect in the 800–1000 °C temperature range; to explain this effect it was supposed it to be the result of a micro-structural evolution of the CMMs during heating when the packing of conductive particles undergo a rearrangement with reduction of the coordination number (“geometrical phase transfer”) [7]. Another distinguishing feature of gradient Si_3N_4–TaN heaters is the high surface temperature obtained with a relatively low power consumption. The physical reason of this phenomenon is not yet clearly understood.

According to the above, we have focused our attention to the systematic research of the electrical behavior of multi-component resistive CMMs, based on silicon nitride matrix, with different conducting additives in correlation with their microstructure and evolution during hot-pressing under different environmental condition.

2. Experimental procedure

2.1. Materials and sintering method

Commercial powders[1] of β-Si_3N_4 obtained by direct nitridation of silicon in N_2 atmosphere (DNS) or via the self-propagating high-temperature synthesis (SHS) and some carbides (ZrC, HfC, B4C) were used. Alumina[2] in the amount of 5–7 vol.% was used as an activator. The ZrC powders were subjected to air sizing. To prepare mixtures, both fine (<3 μm) or coarse (28–42 μm) fractions and also poly-dispersed (3–42 μm) powders were used. The conventional processing method to prepare the insulator matrix mixtures was followed. The resistive CMM composite was realized mixing powders for 6 h to prevent aggregation of conductive particles. The green ceramic sheets were prepared by the rolling method, described earlier in detail [7,8]. Sintering was obtained by reactive hot-pressing in CO or CO–NH_3 atmospheres. With this processing method we had the opportunity to control the evolution of the CMM microstructure during sintering. Samples were sintered in a hot-pressing furnace with graphite moulds at 1680–1720 °C for 25–45 min under about 20 MPa pressure [7,8].

2.2. Materials characterization

The electrical resistance of the composites was measured by the two-electrode method using direct (dc) and alternating current (50 Hz ac) according to the volt–ampere method. The samples were sized as pieces $6\ \text{mm} \times 8\ \text{mm} \times 100\ \text{mm}$ (layered composites) and $12 \times 12 \times 2$ size (bulk samples). The resistance measurements were performed both along and across the hot-pressing direction. The surface temperature of layered composites was regulated by the current density through the resistive layer. Temperatures were measured by pyrometers with a spot diameter of about 3 mm. The temperature dependence on the bulk-samples resistance was determined while heated in the oven under the rate of 0.5 °/min. Electrodes were fixed on the contact areas by firing-on the silver paste at 180 °C. The quantitative estimation of the microstructures was accomplished by image analyses obtained with metallographic methods at 375–800 magnification factors, whereas XRD, SEM and XRF spectral analyses were used to recognize the phases present.

The parameters of the percolation equations were defined according to the principles previously presented [7]. Starting from a particular value of the exponent index t and percolation threshold value X_c, the characteristic micro-structural parameters, namely the h/d ratios were defined as h and d are the two principal dimension of the ellipsoids that represent the dispersed particles. It was accepted, that if the quantity $h/d = 1$, the inclusions shape is spherical; if $h/d < 1$ it is of a plate-like shape, and if $h/d > 1$ then it is closer to a cylinder shape.

3. Result and discussion

In previous papers [7,8] we have found that both layered composites and 3D bulk composites made of Si_3N_4–TaN exhibit specific electrical resistance that is not a monotonous function of the dispersed particles concentration Similar results were obtained with composites made of ZrO_2–WC and AlN–SiC mixtures [11]. We have shown that there is a wave-like change of resistance at particular concentration of conductive additives that revert the resistance trend direction. This phenomenon is associated with the aggregation and self-organization of particles during the evolution of their microstructure. The extension and amount of this process depends both on particles size and sintering method in the presence of a more or less viscous liquid phase that may allow or prevent particles movement and aggregation.

Similar results have been replicated with other 3D composites, as presented in Fig. 1. Specific resistance exhibits an overall decreasing trend as the concentration of the dispersed conductive phase increases, but there are fluctuations in the opposite direction (resistance increases) at about 20 and 40 vol.% of coarse dispersed B_4C and ZrC particles. It is worth

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[1] Donietsk Chemical Plant, Ukraine.

[2] Nikolayev Chemical Plant, Ukraine.

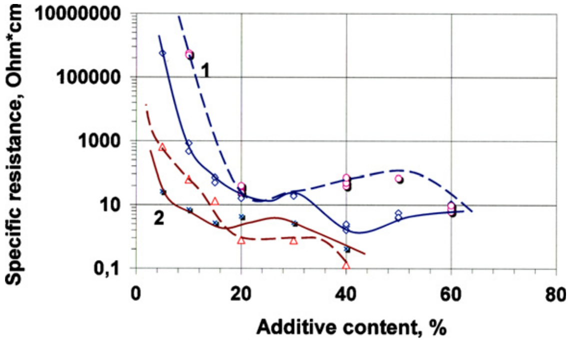

Fig. 1. Specific resistance vs. additive content (vol.%) of 3D compositions, (matrix Si_3N_4 "DNS", "CO" HP environment): 1, B4C; 2, ZrC; line solid, coarse additive; line dashed, fine additive.

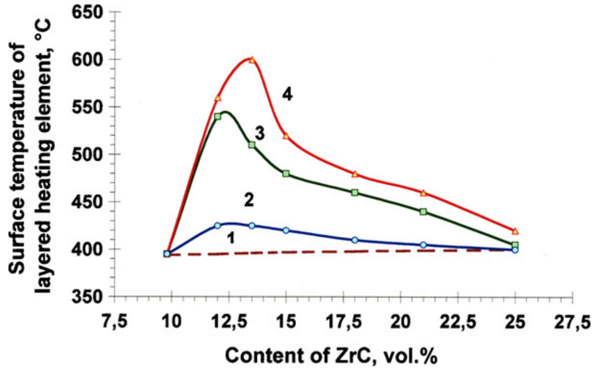

Fig. 3. Surface temperature of layered compositions (at warm loading 3 W/cm^2) vs. content of ZrC (Si_3N_4 type/ZrC type, Hot pressing in CO environment): 1, SHS/poly-dispersed; 2, DNS/fine; 3, DNS/poly-dispersed; 4, DNS/coarse.

pointing out that fine powders, at the same concentration, in general produce higher values of resistance up to about 22 vol.% (B4C) and 18 vol.% (ZrC) as in Fig. 1. After this point values each system behaves in it is own way (Fig. 2).

It is worth pointing out that, besides particles concentration, resistance depends on mixing and processing time. Mixing of powders in the ball mill for less than 6 h was not considered. Longer mixing up to 12 h lowered porosity and increased resistance by values rather scattered in a range that thins down as the mixing time moves up to 14 h. In the 14–18 h range, resistance values and densities are reliably reproduced, whereas, by increasing the mixing time from 20 to 36 h, results spread again. Density is less affected by the prolonged milling than resistance that shows an increasing uncertainty at prolonged milling times. This behavior may be assumed as the evidence of phenomena that may influence resistance that should be searched within the processes of agglomeration and segregation of conductive particles during sintering.

The heating elements work in accordance with the Joule–Lenz law, in fact their surface temperature increases as current flows through the resistive material. Temperature is defined by power consumption and the conditions of heat exchange equilibrium. Samples of the same size consuming the same power and working under the same conditions of heat exchange should have the same surface temperature. But we have observed something different: surface temperature of layered CMM, made with different contents of conductive additives, (Fig. 3) exhibited surface temperature 1.5 times higher than their minimum ($600/400 \approx 1.5$, curves 1 and 4, Fig. 3) depending both on the grain size of ZrC (fine, coarse or poly-dispersed) and the type of insulating phase (DNS or SHS silicon nitride). Resistive composites with maximum surface temperature can be obtained when inclusions are kept in the 12–18 vol.% ranges. The power value required by layered composite (like the materials presented by curve 4, Fig. 3) to achieve the same surface temperature (550 °C) is strongly depended on the combination of two factors: resistance and temperature coefficient of resistance. The required power was lower if the resistance is lower and the temperature coefficient of resistance is positive. Hereinafter we will name this CMM as the PTCR materials. On the contrary, the power required was larger if the composition had a large resistance value and a negative temperature coefficient of resistance (Fig. 4). The composites, which exhibit lower surface temperature (like materials presented by curves 1 and 2, Fig. 3), will be referred to as the NTCR materials.

Our calculations of the real heat removal of PTCR materials (curve 4 Fig. 3) gave values of about 3.4 W/cm^2 (Table 1) whereas for the NTCR materials (curve 1, Fig. 3) the heat removed was limited to 1.2 W/cm^2. To highlight the physical reasons of the phenomena we examined the voltage–ampere characteristics of PTCR and NTCR materials and their power

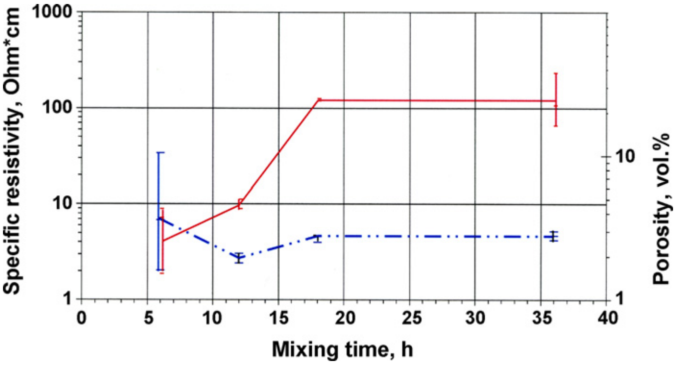

Fig. 2. Specific resistance (solid) and porosity (dashed) vs. mixing time of 3D compositions (matrix Si_3N_4 "SHS", "CO" HP environment).

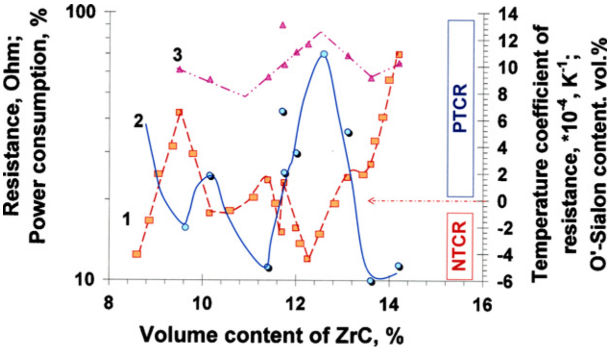

Fig. 4. Temperature coefficient of resistance (1), dc resistance (2) and power consumption (3) for achieving a surface temperature of 550 °C for layered composition (curve 4, Fig. 3) vs. content of additive.

Table 1
Calculation of output warm loss from surface of layered CMM (Fig. 3) at input warm loading 3 W/cm^2

The curves number (Fig. 3)	Maximum of surface temperature of CMM at warm loading 3 W/cm^2 (°C)	Output warm loss by convection, W/cm^2 $Q_\alpha = \alpha\ S\ (T_S - T_0)$[a]	Output warm loss by IR radiation, W/cm^2 $Q_\lambda = \varepsilon_\lambda \sigma ((T_s/100)^4 - (T_0/100)^4)$[b]	Total output warm loss: input warm loading ratio
1	620	0.6	2.81	1.13
2	550	0.53	2.0	0.85
3	430	0.41	1.1	0.49
4	400	0.38	0.81	0.42

[a] $\alpha = 10$ W/m^2 K; $S = 1$ cm^2; $T_0 = 20$ °C.

[b] $\varepsilon_\lambda = 0.9$; $\sigma = 4.96$; T (K).

consumption by means of their surface equilibrium temperatures (Fig. 5) and determined their thermo-emf in the temperature range up to 900 °C (Fig. 6).

We also examined the power consumed by PTCR composite at constant-voltage regime (Fig. 7). We observed that power consumption decreases as surface temperature increases; the volt–ampere characteristic in this case looks like the one of a tunnel-diode. The NTCR samples show the opposite trend of power versus time and their volt–ampere characteristic looks like as ohmic only. It is worth pointing out that this behavior coincides with that of the metal-insulator-semiconductor structures (MIS), which are called ‘‘tunnel-diodes’’ [9]. The NTCR composites require 1.3–1.5 times more power than PTRC as it is reported in Fig. 5. The process of additional heat generation in the PTRC materials can slightly decrease at temperatures higher that 1100 °C.

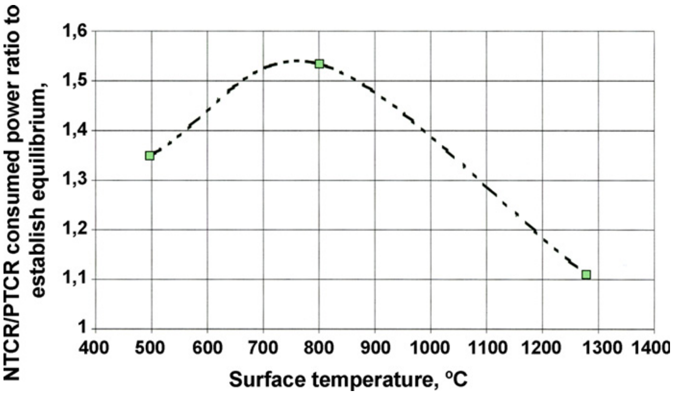

Fig. 5. Ratio of required power for NTCR/PTCR compositions (curves 1 and 4, Fig. 3) to reach a state of thermal equilibrium at a preset surface temperature level.

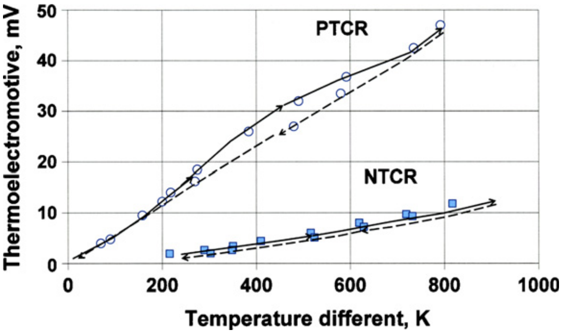

Fig. 6. The absolute value of thermo electromotive vs. temperature difference $\Delta T = T_{\text{Surf}} - T_{\text{Envir}}$ of layered compositions at 15 vol.% ZrC additions for NTCR and PTCR cases reported by curves 1 and 4 of Fig. 3.

The thermo-electromotive force (T-emf) of PTCR materials significantly exceeds that of NTRC (Fig. 6). Thus it may be assumed that by applying an electric potential to the resistive material, a thermo-emf effect is being generated in the volume, with a concurrent rise of surface temperature. Part of the T-emf effect can be used to generate more heat by the direct flow of charge carriers.

Taking into account the combination of the reported phenomena it may be assumed, that the directed movement of carriers in resistive PTCR composites is the tunnel current provided by the discharge of the filled trap level through the tunnel-transparent inter-crystalline phase boundary.

Then both power-saving effect and the effect of positive temperature coefficient of resistance can be regulated not only by the properties of the inclusions, but much more by the peculiar structural conditions of the silicon nitride matrix. The samples distribution versus specific resistance in pilot batch of CMM with ZrC additions at 15 vol.% present on figures (Fig. 8). It was found that NTRC samples exhibit resistance values spread from exceedingly low to rather high values being more frequent at the 0.1 Ω/cm level (25% of the samples). On the other hand PTCR spread is lower and the distribution is more concentrated at the 0.06 ohm/cm level (32%).

These samples have been investigated to reveal the microstructure of both the matrix and of the dispersed conducting phase. The height/diameter ratio was calculated and reported in the same Fig. 8. It is worth recalling that if the

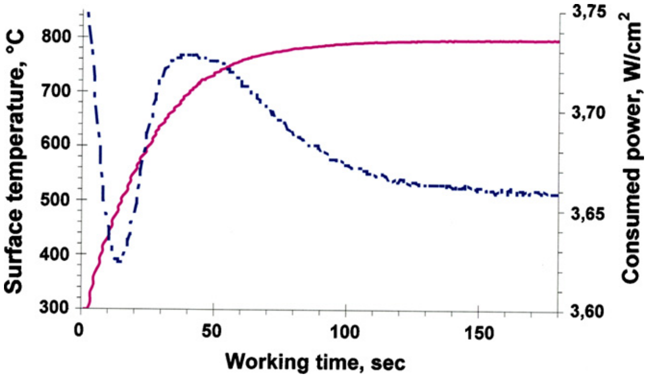

Fig. 7. Trends as a function of time of both surface temperature and consumed power at constant-voltage regime (materials PTCR, curve 4, Fig. 3).

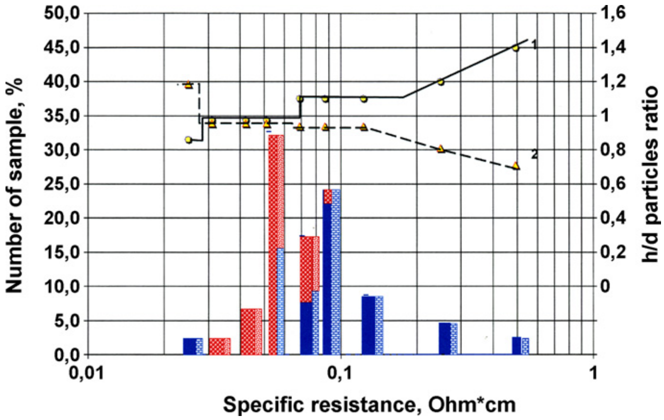

Fig. 8. Number of samples distribution vs. specific resistance in pilot batch of CMM (matrix DSN-Si_3N_4, additive–ZrC–15 vol.%): solid, NTCR, texture, PTCR; "*h*/*d*" of insulator—1, "*h*/*d*" of metallic particles—2.

h/*d* = 1, the inclusions shape is spherical; if $h/d < 1$ it is of a plate-like shape, and if $h/d > 1$ then it is closer to a cylinder shape. It has been observed that the resistive system acquires the PTCR state if both conducting inclusions and isolating inter particles are of the plate-like shape, $h/d < 1$, the long axis of which is directed transverse to the hot-pressing direction. If conducting particles remain plate-like but the isolating matrix becomes $h/d > 1$, the PTRC behavior does not survive while the other NTRC behavior is prevailing (Fig. 8).

Micrographs of two studied materials are presented on Fig. 9. The PTCR samples are characterized by relatively coarse-grained anisotropic microstructure: conductive grains were mainly of plain plates shape disposed transversely to the hot-pressing direction. The NTCR samples microstructure was characterized by combination of small and very large grains. During the hot pressing the phase content of conductive particles was subject to changes: zirconium carbide reacted with silicon nitride generating zirconium carbonitride. The X-ray analysis showed that there are thin layers close to bounds of conductive particles. These layers contain oxygen, nitrogen and silicon.

In order to better appreciate the phenomena some basics aspects will be highlighted as follows. The potential difference can be a source of thermo-emf in multi-component systems because of the simultaneous presence of conductive materials with different work functions. Another source of thermo-emf can also be the thermo-stimulated depolarization of the charge carriers which are localized in the shallow traps.

Layered structures such as metal–silicon nitride–silicon oxide–semiconductor MNOS and SAMOS are widely known in microelectronic. (the multilayered structures metal–nitride–oxinitride–semiconductor are abbreviated MNOS and the multilayered structure with self aligning gate are abbreviated SAMOS). They can localize the electrical charge on the shallow traps and preserve it for many years [9]. From our point of view the most probable reason for irregular, but important physical phenomena for technological applications, is the formation of nano-structured objects like MIS structures, in resistive materials with silicon nitride matrix. Charges can be preserved there for three years and the time of their preservation

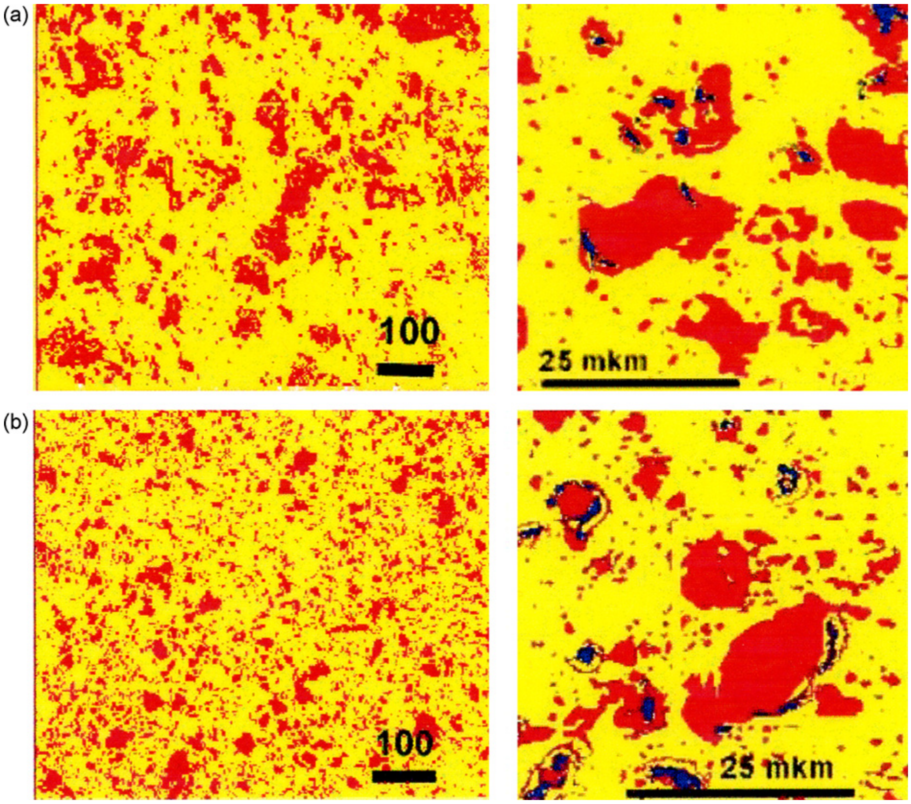

Fig. 9. Microstructures of PTCR (a) and NTCR (b) samples reported in curves 4 and 1 of Fig. 3).

can be defined by the following ratio:

$$t_S = \tau_0 \exp \frac{E_t - \sqrt{(e^3/\pi\varepsilon\varepsilon_0)\sqrt{\vartheta}}}{kT} \qquad (1)$$

where τ_0 is the drift time through the thin layer of silicon nitride, E_t the depth of the shallow trap calculated from the edge of conductivity zone, e the electron charge, k, the Boltzmann's constant, T temperature, ε the silicon nitride permittivity, and ϑ is an average electrical field inside the silicon nitride layer.

The depth of the shallow traps can be regulated during the synthesis process by temperature control and by the magnitude of nitrogen atoms loss or by their substitution with oxygen atoms. The field value inside the silicon nitride layer depends on the tension value and the thickness of the insulating layer. In resistive materials these values will be defined by the concentration and shape of the conductive inclusions particles.

Thus, it is likely that two types of MIS-structures are formed in the resistive materials under investigation: power-dependent permanent storage devices (MNOS) and storage devices with floating gate (SAMOS) as in Fig. 10. In both devices barrier layers are realized at the expense of secondary silicon nitride covering the surface of the metal-like particles dispersed in the matrix. In the SAMOS type structure, two barriers of insulating layers must be present made of silicon nitride and oxinitride. On one side of the metallic particles the layers must be not less than 8 nm thick, on the opposite side thickness must be a rather thick joint of silicon nitride–silicon oxinitride layer, up to 100 nm. The insulating layer from silicon nitride is the active element.

We have previously observed the formation of shallow traps with levels of 0.1–0.5 and 1.2–1.5 eV above the conduction band bottom, due to the chemical processes for the silicon nitride synthesis in a controlled environment [10]. These layers are separated by tunnel-transparent barriers and can be filled with carriers at relatively low tension (up to 10 V). And so we suppose the presence in CMM with silicon nitride matrix the state accompanied by the weak volume charge build-up in nanosized insulator layers, especially on the shallow traps in the band gap of silicon nitride. These traps are formed due to the loss or the substitution of some part of nitrogen atoms. The charge build up on the traps is accompanied by the reduction of charge carrier number participating in conductivity, what is accepted as a weak PTCR. Absolutely unique phenomenon is the displaying of this condition at high temperatures (700–1100 °C) [7]. If a resistive material is characterized by mentioned structural state, the prescribed surface temperature is achieved by lowering the power consumption.

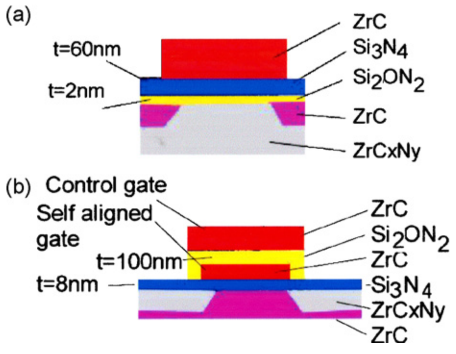

Fig. 10. Sketch of two possible nano-sized microstructure of MIS structures (metal–nitride–oxinitride–semiconductor (zirconium carbonitride), abbreviated MNOS—a and MNOS structure with self aligning gate: SAMOS—b) that might be formed in multicomponent CMM during sintering.

MNOS structures are those with the barrier composed of silicon nitride and metal-like added particles. The relatively large metallic particles surrounded by the insulating matrix particles are made by two-layered barriers made of joint silicon nitride, up to 60 nm thick, and 2 nm silicon oxinitride. In this case silicon nitride also is as active elements. But the infill of traps happens at higher voltage (nearly 30 V). In that case the two states will formed: (i) accompanied and (ii) not accompanied by the volume charge build-up in nanosized insulator layers. In cause (i) we have observed a large PTCR effect and the lowest energy saving effect. In other case, we observe in the CMM only the NTCR effect.

If during the process of obtaining resistive materials the layer of oxide or oxinitride is thin enough to be transparent for tunneling captured carriers, MNOS or SAMOS structures are discharged by tunneling electrons from traps through the thin layers of the oxinitride insulator. The flow of tunnel current causes extra local heating of the material, rising the temperature of CMM surface without the need of extra circuit-power. This non-ohmic process can be amplified if optimal charge density conditions are created at the boundary of ‘‘super-thin nitride-oxide’’. Thus, on the one hand there will not be much time to maintain reserved charges in the traps and, on the other hand, the conductive inclusions will not shorten the tunnel-transparent layer. This condition can be achieved by regulating the extent of chemical interactions in the resistive material during its thermal treatment.

The formation of a secondary silicon nitride can be controlled by reduction-dissociation during hot pressing in a CO medium. The density of traps centers formed and the thickness of the oxide-oxinitride layers in the silicon nitride matrix can be regulated by the hot pressing process in either ammonia or in a combined medium, containing both types of reagents, CO and NH_3 [10].

Starting from the combination of the phenomena under investigation we may assume that carriers flow in resistive PTCR composites is the tunnel current due to the discharge of filled trap levels through the tunnel-transparent grain-boundary. Then, both the power-saving effect and the effect of positive temperature coefficient of resistance are due, not so much to the properties of the metal-like inclusions, but to the peculiarities of the structure of the silicon nitride matrix [7,8].

4. Summary and conclusions

The percolation threshold in resistive materials is defined as the composition at which the conducting phase forms a complete three-dimensional path through the material. However, in typical CMCs we have observed a multi-threshold situation. At some content of conducting particles the first infinite cluster is formed, when the number of particles increases, the clusters is destroyed to be created again at a higher content of conducting particles. That is strongly determined by the initial powders properties, manufacturing method and microstructure evolution during sintering.

There are two types of structural state for CMM with silicon nitride matrix: (i) the state accompanied and (ii) the state not accompanied by the volume charge build-up in nanosized insulator layers. The volume charges in materials (i) were supposed to build up on the shallow traps in the band gap of silicon nitride. These traps are formed due to the loss or the substitution of some part of nitrogen atoms. The charge build up on the traps is accompanied by the reduction of charge carrier number participating in conductivity, what is accepted as a significant PTCR. Absolutely unique phenomenon is the displaying of this condition at high temperatures (700–1100 °C); we do not know other materials with similar effect.

If a resistive material is characterized by the structural state (i), the prescribed surface temperature is achieved by lowering the power consumption. It is assumed, that the basis for this phenomenon underlines nanostructure effects of MIS-structure: electrode tunneling from the traps through thin layers of nitride-oxinitride insulator. The use of this physical phenomenon seems to us particularly important for the design of advanced materials assigned for electrical devices, because it allows 20–50% decrease of power consumption without decreasing their effectiveness.

The data about the correlation between microstructure effects and electrical behavior of CMM can also be used for nondestructive quality control of structural ceramics or for designing of ceramic processing of composites with the improved properties especially such as fracture toughness, hardness and bending strength [11].

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