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# PROPERTIES OF THICK FILM INKS FOR MICROCHANNELS DESIGN

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## RESUMEN

*Presentamos en este trabajo un estudio del comportamiento de las características físicas de pastas de película gruesa dieléctricas y conductoras, en relación al proceso de sinterizado. El objetivo es definir los parámetros relevantes para el diseño de microcanales de película gruesa en una estructura multicapa. Se realizó un análisis dimensional de los caminos impresos para obtener una función paramétrica que describa el comportamiento de las paredes laterales antes y después del sinterizado. Los resultados experimentales demostraron que el ancho y la razón alto/ancho, antes y después del sinterizado, tienen una relación lineal.*

## ABSTRACT

*We present in this work a study of the physical characteristic behavior of dielectric and conductive thick film inks related with their firing process. The aim is to define the relevant parameters for the design of thick film microchannels in a multilayer structure. A dimensional analysis of the printed tracks was performed to get a parametric function that describes the lateral walls behavior before and after firing. The experimental results showed that the width and height/width ratio, before and after firing, have a linear relationship.*

## 1. INTRODUCTION

The development of microchannels manufactured using thick film technology has received little attention in spite of their potential applications, e.g. in the field of chemical and biotechnological processing. These channels are an efficient alternative to their conventional counterparts owing to their capability to tolerate high temperatures and aggressive environments or severe operating conditions. Furthermore microchannels and electronics can be integrated in a sensor/actuator housing like in micromachined channels onto silicon [1] [2], thus obtaining a one-piece, safe, easily manufactured and economic component.

With the aim to explore this new niche for thick film technology a study focused on defining the relevant parameters for the design and construction of thick film microchannels arranged as a sandwich structure is presented in this work. Three aspects were studied:
- geometrical and mass-volume behavior of channel lateral walls (tracks),
- adhesion of the floor and ceiling of the channel, and
- design of a microchannel with specific dimensions.

## 2. MICROCHANNEL STRUCTURE

The thick film microchannel structure consists of a couple of alumina substrates in a sandwich configuration, forming the floor and ceiling of the channel. The tracks, which are the channel lateral walls, are screen printed and fired over the lower substrate and set up the channel height. A glass via, which seals the channel and adheres the lower to the upper substrate is screen printed and sintered outside the track.

It is a well-known fact that solvent evaporation from the inks and the viscosity reduction at peak firing temperatures during the firing process produce dimensional changes in screen printed shapes. Changes in height, width and cross-section shapes occurred during the firing process were measured in order to obtain a proper geometrical description of the microchannel behavior. Consequently, for the purpose of defining microchannel initial dimensions in terms of the desired final geometry, the variation pattern of geometrical parameters has to be known. The study of conductor and dielectric track behaviors was carried out using alumina and alumina coated with dielectric and overglaze inks as substrate.

## 3. CHANNEL LATERAL WALLS

### 3.1 Samples preparation

Samples were constructed on Kyocera 96% alumina (50 x 12.5 x 0.6 mm). Heraeus C8710 and DuPont PdAg6120 conductor inks and Heraeus IP9117 dielectric ink were used as tracks. Each sample contained 10 straight tracks, 10 mm long with widths varying from 100 to 1000 μm and 4 mm apart from each other. The study of track behavior onto alumina has been extended to alumina coated with Heraeus IP137 overglaze and IP211 dielectric ink. Stainless steel screens of 300 mesh and a 45° angle of attack between the squeegee and the screen were used for printing the tracks. All samples were dried during 10 min. in an IR oven at 120°C and fired in a Lindberg 810 continuous belt furnace. A 45 min. firing cycle was used, which included 10 min. at a peak firing temperature of 850 °C.

### 3.2 Experimental results and discussion

Track geometrical dimensions were measured with a Carl Zeiss D-7082 light intersection microscope. The photographic images so obtained were scanned and digitized to process them and predict channel shape after firing. The height ($b_n$) and width ($a_n$) of each track were measured before and after firing ($b'_n$, $a'_n$). Experimental results show that from a set of measured parameters only two relationships having a linear characteristic were founded suitable for the prediction of track dimensions. The ratios are:

$$b'_n / a'_n = f\,(b_n / a_n) \qquad \text{(I)}$$

$$a'_n = f\,(a_n) \qquad \text{(II)}$$

Fig.1 illustrates geometrical changes and the best curve fitting for IP9117 tracks onto alumina. Fig.1a) plots the height-width ratio after versus before firing; while Fig.1b) represents the width after versus before firing. The slope behavior of the fitted curve of the height-width ratio before and after firing (*slope* 1) and the slope of widths before and after firing (*slope* 2) were compared for tested pastes and substrates (Table 1). Slope>1 means that after firing, width or height/width ratio are bigger than before firing and viceversa.

Of all studied pastes, substrates covered with IP137 demonstrated highest width variation after firing. This is in agreement with height-width ratios before and after firing, in which the maximum is

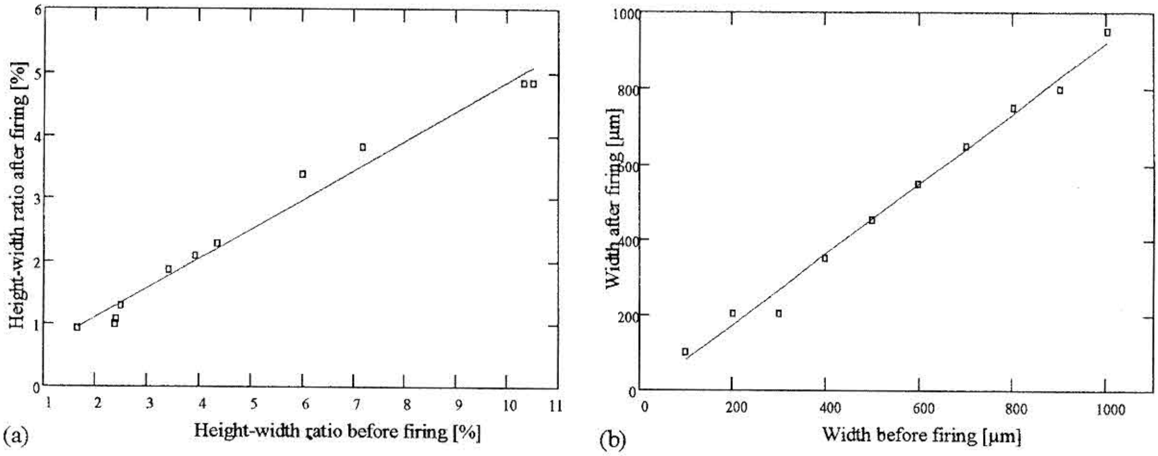

**FIGURE 1.** Characteristics of dimensional changes of IP9117 tracks onto alumina a) height-width ratio after vs before firing, b) width after vs width before firing of the tracks.

**TABLE 1.** Slope behavior of fitted curve of the height-with ratio before and after firing.

| | *Slope 1* | | |
|---|---|---|---|
| | *Alumina Uncoated* | *Alumina Coated* | |
| *Pastes* | | *IP 137* | *IP 211* |
| Pd/ Ag 6120 | 0.468 | 1.045 | 0.626 |
| IP 9117 | 0.562 | 2.589 | 0.468 |
| C 8710 | 0.667 | 0.973 | 0.854 |
| | *Slope 2* | | |
| Pd/ Ag6120 | 1.078 | 0.773 | 1.017 |
| IP 9117 | 0.935 | 0.749 | 1 |
| C 8710 | 0.987 | 0.795 | 1.068 |

also observed in substrates coated with this material. Height-width ratio maintains or increases its value after firing using IP137 coated alumina, with them being more pronounced in the case of IP9117 track. We can explain this mechanism as follow: firing temperature of IP137 is between 750 and 800 °C, so at 850 °C it is soft and fluent. IP9117 is more viscous at the same temperature and upon cooling the ink shrinks, owing to its expansion coefficient, while IP137 has not hardened yet. This situation produce a reduction in the coating layer thickness near the track, changing the reference level used to measure the IP9117 thickness (Fig. 2).

Usually, a decrease of mass of about 10-12% is normal when the ink is dried because of evaporation. We measured about 25-35% main component loss during firing, which is responsible of thickness decrease in the height track. IP9117 has one of the lowest coefficients of mass loss, so when comparing IP9117 and C8710 pastes for the same width reductions, IP9117 is found to be about 2.5 times higher in height/width ratio. This means that in the same screen printing and firing conditions IP9117 is the material that best maintains its initial geometry.

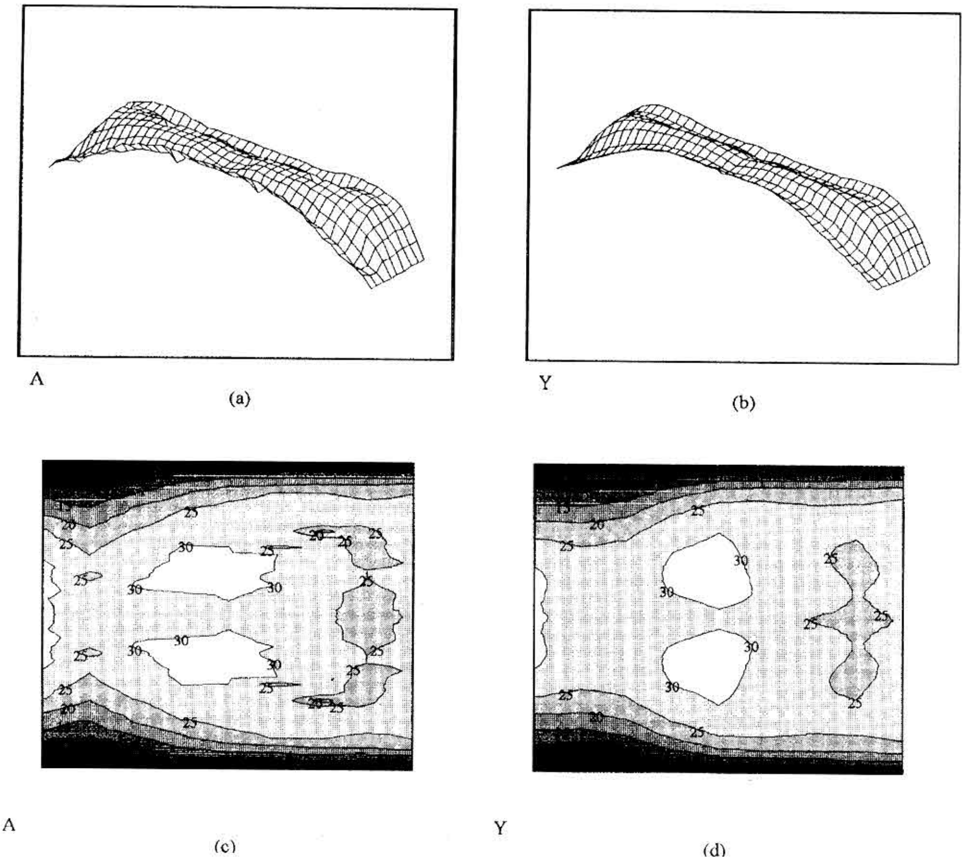

**FIGURE 2.** Adjusted surfaces of IP9117 track profiles onto alumina before firing. a) data matrix surface outline; b) adjusted data matrix surface outline; c) data matrix graphic contour; d) adjusted data matrix graphic contour.

Lateral walls were finally studied on IP9117 track profiles for different an before and after firing. An adjustment of measured values was made in order to predict the behavior after firing. For this purpose profile measured values y were sorted in a matrix:

$$A= \begin{bmatrix} y_{0,0} & \cdot & y_{0,n} & \cdot & y_{0,v} \\ y_{r,0} & \cdot & y_{r,n} & \cdot & y_{r,v} \\ y_{m,0} & \cdot & y_{m,n} & \cdot & y_{m,v} \end{bmatrix} \qquad \alpha= \begin{bmatrix} a_0 \\ a_n \\ a_v \end{bmatrix} \tag{III}$$

where $n$ is the number of each track $a_n$ and $r$ is the index of each element widthwise in each track $a_n$. In order to obtain an expression to predict profile behavior widthwise and for different widths, a 5th order polynomy was adjusted. But as the polynomy coefficients so obtained vary with the different $a_n$ widths, a new adjustment was done for these. This polynomy was also of the 5th order. Then, polynomy coefficients for each $n$ may be calculated by means of [3]:

$$\beta^{<n>} = (\lambda^{T}.\lambda)^{-1}.\lambda^{T}.A^{<n>} \tag{IV}$$

where $A^{<n>}$ is each one of the $n$ vectors of matrix A and $\lambda$is a matrix calculated as:

$$\lambda^{<j>} = (x_r)^j \tag{V}$$

with j=0,1..5 and $x_r \in [0,1]$.

Then, adjustment of the coefficients obtained in matrix (IV) is made through:

$$\delta^{<j>} = (\rho^{T}.\rho)^{-1}.\rho^{T.}(\beta^{T})^{<j>} \qquad \text{(VI)}$$

being

$$\rho^{<i>} = (\alpha_n)^{i} \qquad \text{(VII)}$$

with i=0,1...5 and *n*=0, 1...n.

In order to achieve a prediction of vector components values in [0,1], with width tracks elements Mr and a vector of different track widths elements N*n* , the reconstructed function will be:

$$Y_{n,r} = \sum_{j=0}^{5}\left[\sum_{i=0}^{5}\delta_{i,j}(N_n)^{i}\right].(M_r)^{j} \qquad \text{(VIII)}$$

Fig. 3a) and 3b) show the surface of the different channel profiles for 100 to 1000 μm widths of IP9117 tracks onto alumina. The maximum of the curve moves from the center to the track ends in the direction of increasing widths. Fig. 3c) and 3d) show the increasing contours from right to left of the A data matrix and of the Y adjusted matrix. These figures are an example of the good adjustment due to the topologic similarity of sectors with the same level. The same procedure is applied to values measured after firing. Adjustments (I), (II) and data (A matrix) were used and compared to the measured values after firing; the differences were less than 3.2%.

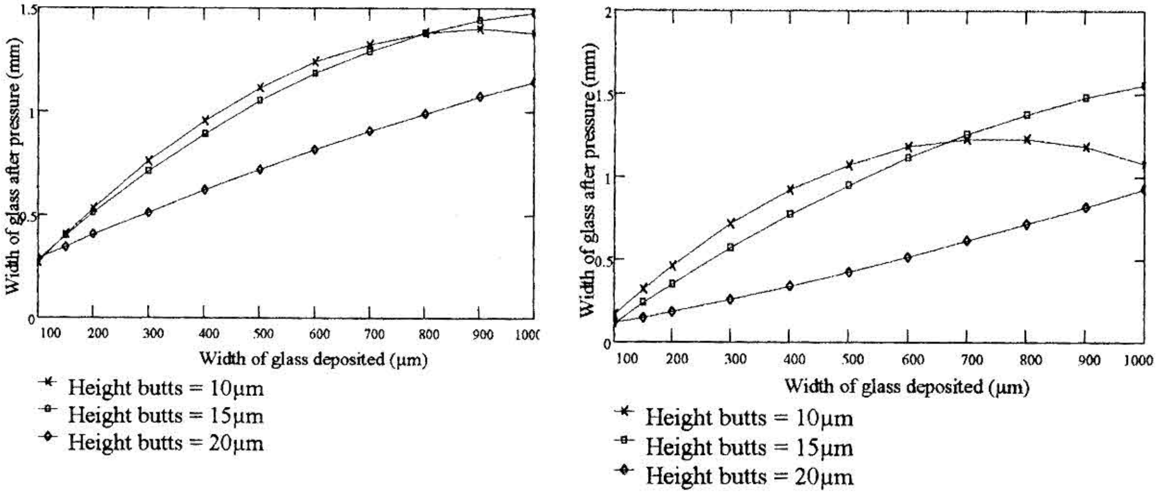

**FIGURE 3.** Behavior characteristics of adhesive width for different butts: a) floor substrate, b) roof substrate.

A second firing at 550 °C for 10 min., that is an operation required for sandwich type channels fabrication, did not bring about significant changes in the geometrical behavior. Therefore, the result obtained after the first firing will be taken as the final channel geometry.

## 4. UPPER SUBSTRATE ADHESION

As mentioned above, an adhesive is needed to build up microchannels by gluing both alumina substrates, since tracks only determine the channel height. Channel assembly requires that the lower and upper alumina substrates are firmly pressed together until contact with the adhesive lines distort the wetted glass ink. This deformation depends on the glass type, initial line width, printing and firing process and track heights (working as butts). The study of magnitude of this distortion was based on the measurement of the deposit width before and after placing the upper substrate applying the required pressure. This was analyzed in terms of the adhesive lines width for different butt heights.

IP9025W ink was studied for its application as adhesive, because of its low viscosity, high wettabilty and low firing temperature. IP5137, IP9117 and C8710 pastes were selected as butts obtaining heights of 10, 15 and 20 μm and widths of 300 μm, which were inserted between the adhesive lines from 100 to 1000 μm widths in 100 μm increments.

Before lines dry, the upper alumina was placed on the lower one and pressure was applied until butts were reached; the assembly was dried at 120 °C during 10 min. Then, the sandwich was opened to measure the adhesive lines width.

Measurements were made of the maximum widths obtained both in the lower alumina used as channel floor and in the ceiling alumina. It is worth mentioning that requirements for the characterization of the adhesive behavior are not so severe, unless an increase in channel density was needed. Fig. 4a) shows that higher butts produces the lower glass expansion. The behavior has a parabolic pattern whose maximum depends on butts height and adhesive width. A similar behavior was observed in the adhesive of the top alumina acting as microchannels ceiling (Fig. 4b).

## 5. CONCLUSIONS

The structural elements necessary for channel design were studied and their characteristics properly established. A design method that allows selecting tracks width as a function of the geometric profile was obtained and the procedure may be extended to any other paste not considered in this work. The knowledge obtained from the study of track and adhesive behavior allowed the design of sandwich microchannels with specific dimensions. Channels obtained were measured with SEM (Philips 505). Sealing of the channel was tested by filling it with zinc rich and observed with X-rays. Obtained images in both cases allowed checking the final dimensions and verifying that no fluid was lost along the channel.

## 6. ACKNOWLEDGMENT

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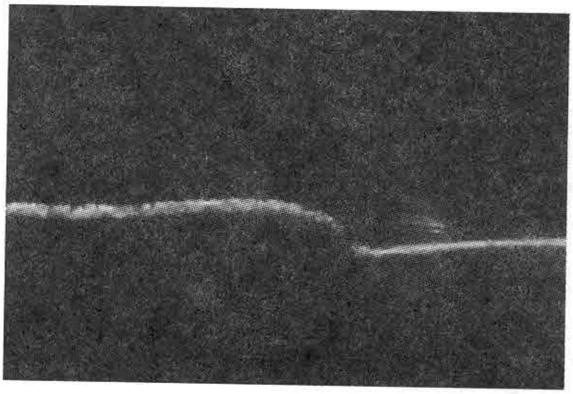

**PHOTO 1.** Cross section profile image of IP9117 track onto alumina coated by sintered IP137.