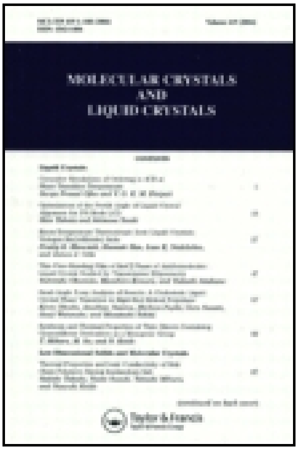

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ORMOCER®S - Inorganic-Organic Hybrid Materials for e/o-Interconnection-Technology

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ORMOCER®S – Inorganic-Organic Hybrid Materials for e/o-Interconnection-Technology

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Hybrid inorganic-organic polymers (ORMOCER®s) have been developed and tested for evaluation in optical and electrical interconnection technology. The photopatternable materials with negative resist behaviour are composed of inorganic oxidic structures cross-linked or substituted by organic groups. They are prepared from organosilane precursors by sol-gel-processing in combination with organic crosslinking of polymerizable organic functions. As a result of these functionalities the properties of the ORMOCER®s can be adjusted to particular applications. Systematic variation of composition combined with adaptation to micro system technology allows great flexibility in processing. The main features of these materials are:

- Combined use as dielectric and passivation layers in electrical systems and devices as well as core and cladding for optical applications enables e/o applications with high integration levels.
- Postbaking at moderate temperatures (120 °C – 170 °C) enables processing on low-cost substrates such as FR-4 and BT.
- Easily adaptable to thin film technology: spin-on with planarisation >90% and via diameters down to 20 μm have been achieved.

Keywords: ORMOCER®; inorganic-organic polymer; dielectric; optical waveguide; e/o-applications; SBU-layers on advanced PCB

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INTRODUCTION

This paper is written to offer sol-gel researchers an introduction into the field of applied research on electrical and optical applications based on our inorganic-organic hybrid materials. Beside chemistry some new results on metallisation will be given. According to the low cost approach towards high density electrical [1,2] and optical packaging [3,4] based on ORMOCER®s finally a summary on several demonstrators for later application is given. Most of the material properties of the ORMOCER®s to be discussed have been published previously [1-4] and will therefore only be summarised in this introduction:

Optical properties:

- Optical loss $\alpha = 0.23$ dB/cm at 1310 nm (not fluorinated!)
- Refractive index (at 588 nm) = 1.52-1.56 (primary index tuning range)
- The ORMOCER®s show an isotropic nature, which is important for polarisation-independent operation of waveguides.

Electrical properties:

- Permittivity ε_R at 10 kHz = 3.2
- Dielectric loss at 1 MHz $\tan(\delta) = 0.004$
- Volume resistivity $R_D \geq 10^{16}$ Ωcm
- TDR measurements have also been performed on microstrip lines to verify the 50 Ohm design rules. These lines supported the step pulse in time domain without significant distortion across a length of 10 cm (due to a measured cut-off frequency of 2.7 GHz).

Thermal stability:

- Despite moderate curing temperatures, the material withstands standard metallization and soldering processes.

ORMOCER®s

During the last decade a fast growing interest in inorganic-organic hybrid materials for use in electrical and optical applications could be observed. The main reason for that trend is that the incorporation of an inorganic oxidic network into a polymer will raise its thermal stability. In addition, if there is a chemical crosslinking between the inorganic and organic parts, the material properties are much easier influenced than within pure organic polymers. Finally, the availability of a large number of functionalised alkoxysilanes on the market accompanied with much increased fundamental knowledge on sol-gel processing enable the reproducible synthesis of inorganic-organic hybrid materials.

For more than 16 years the Fraunhofer-Institute for Silicate Research has been working with ORMOCER®s, inorganic-organic hybrid materials on a molecular scale, for all kinds of applications. Abrasion resistant coatings, barrier systems, corrosion protection and dental materials, all based on ORMOCER®s, are on the market [5]. Different to other investigations in that field, leading to inter-penetrating networks, the inorganic-organic matrix of an ORMOCER® results from chemical crosslinking. Of coarse, in addition the established matrix further could also be interpenetrated by additional polymers. In general ORMOCER®s are prepared from functionalised organosilane precursors by polycondensation reactions (sol-gel-processing [6]) in combination with organic crosslinking of the resulting inorganic-oxidic oligomers. The properties of these amorphous optically transparent materials can be modified easily by molecular design of the chosen organosilanes.

Modification of ORMOCER® properties

In Fig. 1 a general formula is given for typical commercially available functionalised alkoxysilanes, novel specially synthesised perfluoroaryl silanes [7] and the influence of chemical functionality on the polymer network. Beside the functionality, materials for the optical and electrical interconnection technology in general require educts of high purity in terms of ions and chemical by-products. If necessary, distillation of the liquid educts are standard procedures to increase purity.

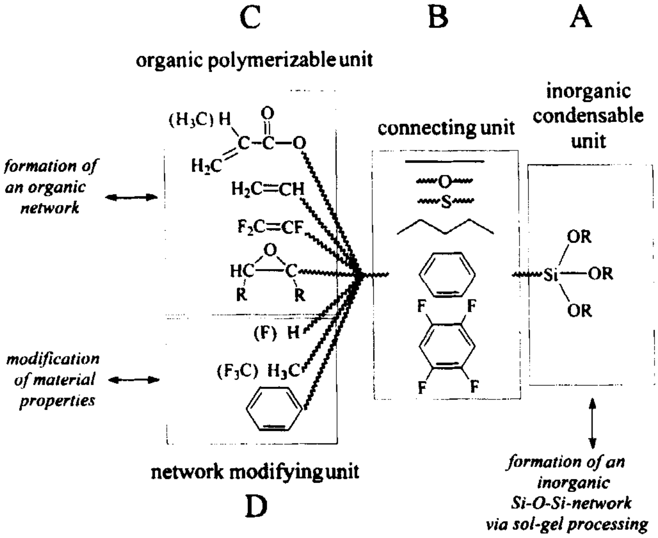


FIGURE 1 Formula of silane precursors for ORMOCER® synthesis and their influence on the network and materials properties.

A: The alkoxysilyl-unit establishes a Si-O-Si-network via hydrolysis/condensation reactions, forming organically modified inorganic nanoscaled oligomers. Their size and shape can be influenced easily by modification of the polycondensation conditions (catalyst, concentration, solvent, temperature). Using of bifunctional silanes will result in chains and/or rings, whereas trialkoxysilanes give three-dimensional oxidic networks. The resulting nano-scaled particles - depending on the used alkoxysilanes and the polycondensation conditions - are typically 2 - 5 nm in size according to SAXS-measurements [8] and size exclusion chromatography (SEC). The higher the percentage of the Si-O-Si-network in the ORMOCER®-resin/lacquer the higher will be the E-modulus, the thermal and mechanical stability and the lower will be the thermal expansion coefficient (CTE) and the optical loss in the NIR. For special applications remaining SiOH-groups can be reduced by silylating agents. For dielectric applications dense oxidic networks have to be avoided otherwise a rise of the permittivity will occur due to the higher ε_R of SiO_2.

B: The longer the chain length of the connecting unit the lower will be the E-modulus and the higher will be the thermal expansion coefficient of the ORMOCER® [9,10]. For very high transparency in the NIR the connecting unit should be partially fluorinated or perfluoroaryl-groups should be used [7,11-14].

C: The organic polymerisable unit and its functionality have to be chosen with regard to later application in thin film technology. Whereas for the photolithographic process of thin film technology there is a need for easy polymerisable groups like methacryl or epoxy. These functionalities will cause higher thermal expansion coefficients and optical loss in the NIR than vinyl or fluorinated vinyl. The latter can on the other hand not be polymerised easily to build up the organic network via the organic crosslinking of the oxidic oligomers . To avoid ions within the matrix and to establish a negative resist behaviour photoinitiators like phosphinoxides and α-hydroxy-ketones were chosen (initiating a radical polymerisation).

D: Non-reactive groups like aryl or alkyl can also modify the material properties. Raising their percentage will decrease the organic crosslinking by steric means and the sol-gel-process according to their hydrophobic behaviour. Both cause a modification of the network of the inorganic-organic material for example by modifying the density of the material. Material properties such as the refractive index can be adjusted in this way. Arylic functions will raise the refractive index (up to 1.6) and establish good dielectric behaviour. Alkyl- or, even better, fluorinated alkyl-groups will reduce the refractive index (down to

1.42, depending on the degree of fluorination) and result in lower optical loss in the NIR.

Example for the synthesis of ORMOCER®s

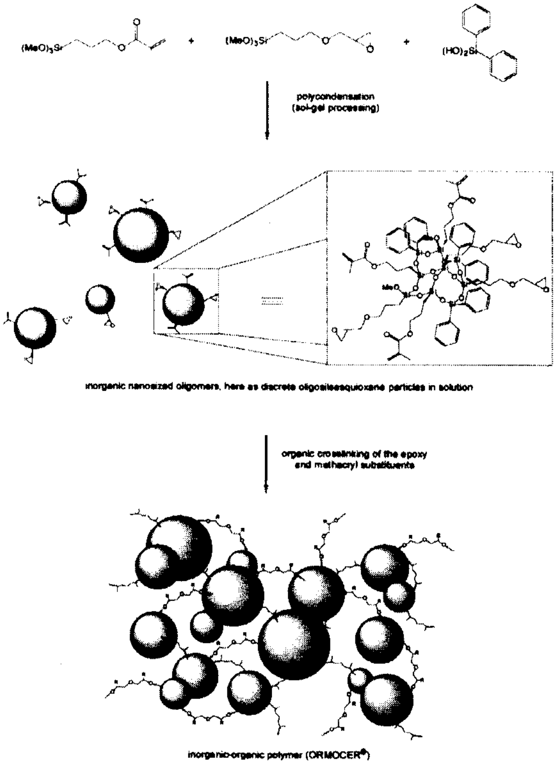


FIGURE 2 Scheme for ORMOCER®- synthesis

Fig. 2 represents a scheme for ORMOCER® synthesis. Polycondensation reactions (sol-gel processing) of commercially available alkoxysilanes results in functionalised oligomers.

Depending on polycondensation conditions of functionalised trialkoxysila-nes according to our recent studies by SAXS, SEC and ^{29}Si-NMR-spectroscopy with *ab initio* simulations mostly oligosilsesquioxanes will be formed. After application as coating by spin-on, spraying, knife-coating etc. the oxidic oligomers will be crosslinked via organic bridges of polymerised methacryl and epoxy-substituents.

A large variety of ORMOCER®s was synthesised [2,13-15]. The system for passivation and dielectric applications (system 2) [15] shown in Fig. 3 is composed of the following four co-condensed alkoxysilanes: 3-(glycidoxy-propyl)-trimethoxysilane (G), 3-(methacryloxypropyl)trimethoxy-silane (M), diphenylsilane-diole (P2) and tetraethoxysilane (T).

The polycondensation reaction results in oligomeric particles dissolved in alcohols (sols), where the solvent can be easily replaced [16].

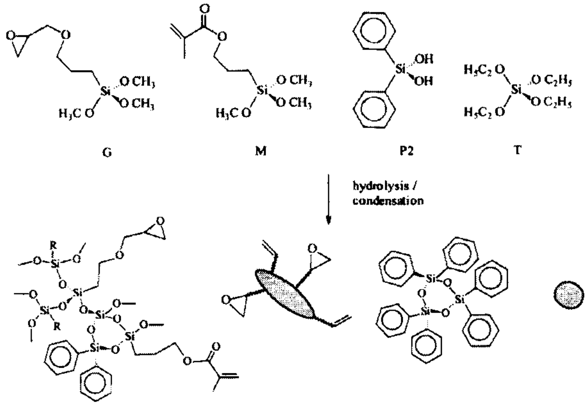


FIGURE 3 Scheme for synthesis of ORMOCER® - resin 2. The resulting functionalised oligomers, simplified in the figure as shaded ellipses and rings, form the base resin for thin film processing.

For further information on ORMOCER® 1 - here the phenyl group was exchanged by a vinyl group - and ORMOCER® 2, respectively, see [15].

Systematic chemical modification and optimisation resulted in ORMOCER® 3 (for dielectric applications) and ORMOCER® 4 (for e/o-applications) [17]. Focusing on low-cost materials and application technologies, non-flourinated, commercially available educts were used.

The Si-O-Si-network of the oligomers is responsible for a thermal stability up to 300 °C (5% weight loss), according to DSC and TG/MS [8, 15]. Also the Si-O-Si-network lowers the optical loss at 1310 nm by diluting the CH-absorptions of the organics.

The shelf life of resins/lacquers is approximately 4 months at room temperature (RT). At RT was observed a strong influence on the stability of the lacquer shelf according to the polarity of the solvent. This becomes less important at lower temperatures. The shelf life is much longer if the material is stored in a freezer at -18°C.

CHEMICAL BACKGROUND OF ORMOCER® THIN FILM PROCESSING

The methacrylic silane was chosen for later photo-induced polymerisation in combination with photo-initiators (less than 3 wt.%).

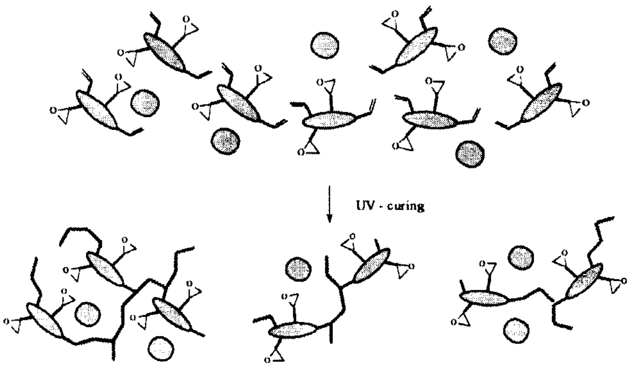


FIGURE 4 Scheme for photo-curing of ORMOCER® resin 2

The epoxy silane was introduced into the oligomer to allow the precursor to be cured thermally at moderate temperatures. The phenyl-groups guarantee good dielectric behaviour in the MHz-range and lower optical loss at 1310 nm. In addition by polycondensation of P2, network modifying rings of different sizes were formed, as confirmed by size exclusion chromatography (SEC) and FT-IR-spectroscopy [16]. In the simplified sketch for thin film processing, the oxidic oligomers have been reduced to shaded ellipses and rings (see Fig. 3).

After application of ORMOCER® lacquers by spin coating an organic cross-linking is formed by UV-exposure (radical polymerisation of the C=C-bond of the methacrylic group) see Fig. 4. Before post-baking, within the development step all not UV-exposed parts will be removed using solvents.

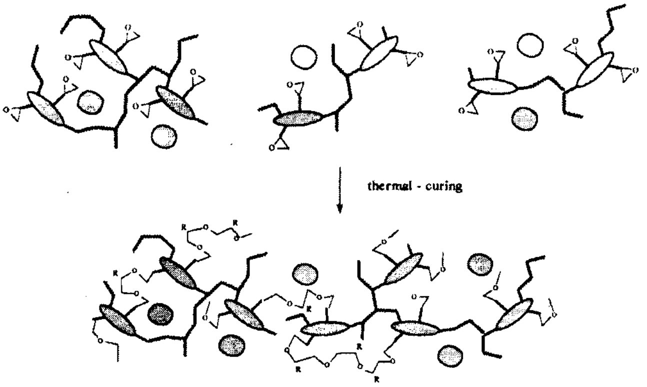


FIGURE 5 Scheme for the post-baking of the UV-cured patterned ORMOCER® system 2 after development

Environmentally friendly less polar organic solvents like esters (e.g. propyl-acetate and γ-butyrolactone) and ketones (e.g. butanone) were used both as solvent and as development agent for wet etching. The ORMOCER®s behave as negative resists which easily achieved photo-polymerisation and development. Epoxy polymerisation (post-baking) starts at temperatures well below 170 °C (see Fig. 5), resulting in a hydrophobic film (contact angle with water >76°) that withstands a variety of chemicals.

PROCESSING OF ORMOCER®s

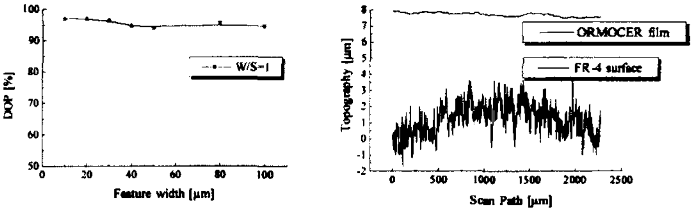
ORMOCER® lacquers have been developed to come as *ready-to-use photosensitive* mixtures that can be stored in a freezer at -18 °C for up to six months. They appear transparent to slightly reddish and have been filtered through membranes whose pore sizes were 0.2 μm in mean diameter. At room temperature, a series of experiments indicates a pot life of the ready to use systems of about one month.

The general fabrication sequence starts with thin film deposition on clean and dry surfaces. Then, in a pre-bake step at 75 °C, that can be done in an oven or on a hotplate, surplus solvent is evaporated. Pattern transfer is done with a standard Karl Süss mask aligner in proximity mode. Most of the absorption of this negative-tone material is at the i-line in the UV-C spectrum. A post-exposure bake is done followed by development in different ketones.

The final curing of the material (below 170 °C in ambient atmosphere) completes the fabrication of the first thin film layer.

Planarisation

Periodic copper bars of t_{Cu}= 4 μm thickness at a spacing S (equal to their widths W) have been coated with ORMOCER® 2 whose thickness was $T = 2t_{Cu}$. After flood exposure and curing at 150 °C planarisation was scanned with a Tencor profilometer at low scan speed. Planarisation was determined to be in the 90+ percent region, see Fig. 6a. The degree of planarisation is defined as $DOP = 100\ \% * [1-\Delta h/t_{Cu}]$. There are two explanations for the good planarisation of ORMOCER® system 2: (a) it is a low molecular weight material that has the ability to smooth out easily and (b) shrinkage is not a major concern here, because film densification due to polymerisation, which is "only" an organic crosslinking of oxidic oligomers, during curing is negligible for practical purposes. The excellent planarisation properties are also shown in Fig. 6b, where a FR-4 substrate was coated.



A b

FIGURE 6 Planarisation of: **a**, 4 µm thick Cu lines on Al_2O_3 substrate and **b**, an FR-4 surface using ORMOCER® 2

UV-curing

Figure 7 presents the contrast of the material which was tuned to $\gamma = 0.87$. The steepness of the normalized thickness curve is sufficient to produce a solubility modulation that can be used to develop vias at various aspect ratios.

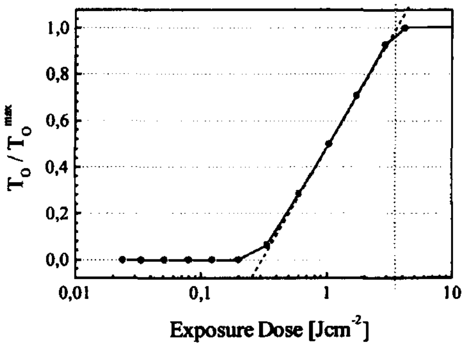


FIGURE 7 Plot of the normalized thickness of ORMOCER® 2. The contrast, $\gamma = 0.87$ [18]

To measure the mechanical integrity of sequentially built-up ORMOCER® layers, bulk samples were prepared from resin 3 and then analyzed by TMA and

DSC measurements. Test samples for the TMA experiments were 5 mm in diameter and 5 mm deep. All samples were polished before the tests started to ensure reproducibility. Sample preparation had to include a prolonged vacuum drying step at 75 °C to remove the remaining solvent. Temperature was increased slowly. Exposure dose was adjusted as needed.

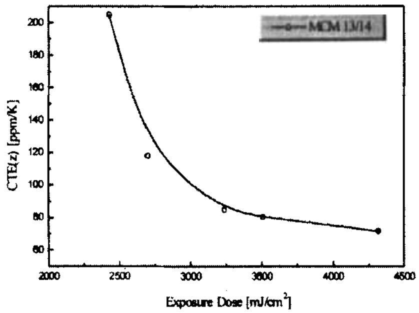


FIGURE 8 CTE versus exposure dose of ORMOCER® 3

The linear coefficient of thermal expansion (CTE) in z-direction (in the range of RT to 150 °C) decreases substantially with exposure dose, indicating stronger cross-linking as expected (figure 8). For further information see [1,2].

METALLISATION OF ORMOCER®s

Metallisation on ORMOCER®s can be performed with different methods and metals. Sputtered aluminium (Al) has been chosen in some cases, e.g. for the opto-electrical MCM-L/D-demonstrator. There, all the three metal-layers are 3 µm thick. The pad areas have additional 3µm Al, 0.1 µm titanium (Ti), 0.4 µm copper (Cu), 3 µm nickel (Ni) and on top 0.2 µm gold (Au) layers.

However, Cu with its superior conductivity is often a preferred candidate for metallisa-tion. Copper could also be sputtered to full thickness although electroplating of Cu often is more cost effective. Prior to electroplating of Cu a conductive layer on top of the ORMOCER® is needed.

As an initial metallisation process electroless copper plating [19] or sputtering of thin Cu-layers can be used. When the substrate has been metallised by the electroless plating process or sputtering and patterned with traditional photo-resist technology - electroplating is used to build up the desired structure. The electroplating process has the advantage over the electroless process of being fast, less expensive and deposit a copper coating having better properties.

Pretreatment and electroless copper plating

Electroless metallisation is an auto-catalytic process by which metal ions are reduced at the surface of the substrate. In order to improve a good adhesion between the electroless deposited copper and deposited dielectric materials some surface treatment or preparation is required. Prior to the electroless Cu deposition the ORMOCER® was etched by the "alkaline permanganate etchback system" [20-22].

The permanganate etch-system treatment consists of three steps:

The first step - *the alkaline treatment* – is swelling and somewhat etching the surface of the ORMOCER® polymeric network causing a replacement of some polymer-polymer bonds with polymer-solvent bonds and opening some Si-O-Si – bonds by nucleophilic attack.

As a result, the permanganate in the second step - *the permanganate etch-step* - can more easily access and break down the polymer surface layer into soluble components. In this way the topography of the surface will be changed. The surface of an un-etched ORMOCER® has an average surface roughness of R_a = 0.374 nm (see figure 9a) whereas the etched ORMOCER® has a surface roughness of R_a = 21.715 nm (see figure 9b).

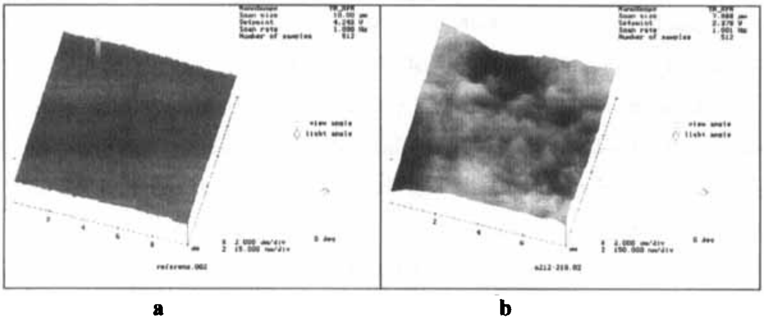


FIGURE 9 Spin-coated ORMOCER® 3 on silicon: **a**, without any treatment; **b**, etched with potassium permanganate etch system.

In the third step - the etched surface is reacted by a Pd/Sn colloid offering adhered particles to the surface. After removal of the catalyst solution the substrate is rinsed with deionized water and immersed into the electroless Cu bath initially resulting in small islands of Cu (Cu-thickness is about 200 nm) on the surface (fig. 10a). Fig. 10b shows a SEM image of the final electroless deposited copper on an ORMOCER® - layer (Cu-thickness is about 500 nm).

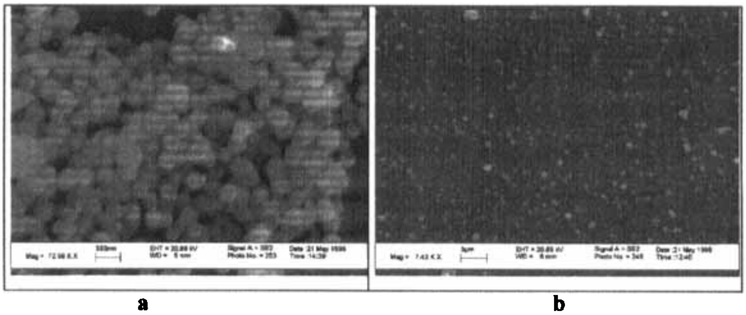


FIGURE 10 **a**: Nucleation of Cu on ORMOCER®-surface;
b: electroless Cu on ORMOCER®-surface.

Electrodeposition of copper

When the substrate has been metallised by the electroless plating process and patterned, electroplating is used to build up the desired structure. The electroplating process has the advantage over the electroless process of being fast, less expensive and deposit a copper coating with better properties. A commercial process developed for printed circuit boards was first employed, but the result was a coating containing pin-holes originating from gas bubbles. By lowering the current density it was possible to avoid the pin-holes, but then the coating became rough.

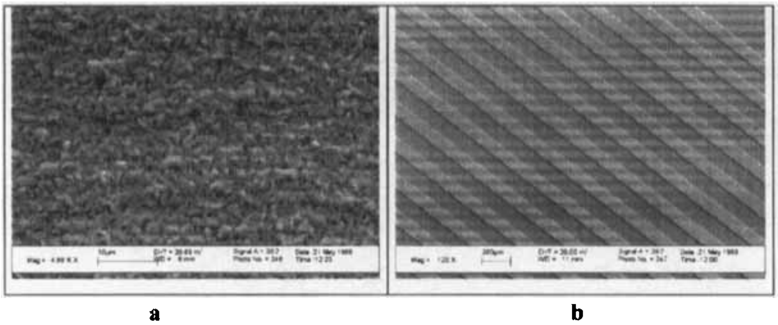
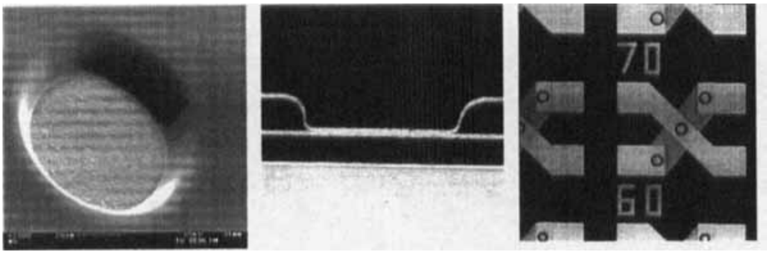


FIGURE 11 a: Surface of electroplated Cu: thickness about 10 µm;
b: electroplated 10µm · 80 µm Cu-conductors.

Only by decreasing the current density further and by diluting the solution with respect to copper content it was possible to obtain an acceptable coating. In order to decrease the long processing time the pulsed current technique was introduced [23]. This was done in a simple solution containing no additives. By the pulse plating technique it was possible to optimise both the roughness and the material distribution (figs. 11a,b). Nickel and gold have also been electroplated for wire bonding (WB) and tape automated bonding (TAB) to a thickness of 3-6 µm and 0.1 µm, respectively. For the demonstration of flip chip bonding (FC) on FR-4 laminate an electro-plated Cu-metallisation with a total Cu-thickness of 10 µm has been used.

DEMONSTRATORS

Based on the results described, several test objects and demonstrators were produced. Detailed information is given in [1-3,24]. For a short impression a summary on the different applications is given in the following figures.



a b c

FIGURE 12 Different views of a wafer with Kelvin test structures
a: SEM of a via on metal land (Ti:Cu:Ti);
b: SEM of a cross-section of the metallised via,
c: videoprint of the Kelvin test structures

Figure 12 shows different views of a silicon wafer with Kelvin test structures. In figure 12**a** a SEM picture of a via prior to metallisation is given. Figure 12**b** presents a cross-section of a metallised via, 30 μm in diameter, of the patterned vias in a Kelvin test array shown in figure 12**c**. The Kelvin test structure represents three sputtered metal layers (Al) and two dielectric (ORMOCER®) layers on SiO_2.

In figure 13 an o/e-MCM-L/D demonstrator (video print (from top) of a 5-channel optical transmitter) is given. The novel concept enabled by new ORMOCER®-technology aims at very low cost and comprises high density electrical interconnects and optical wave-guides integrated in three layers of ORMOCER®s. The thin film layers have been put on top of an FR-4 laminate with micro-vias [25]. The laminate is furnished with a ball grid array (BGA) underneath, eliminating the need of any extra package.

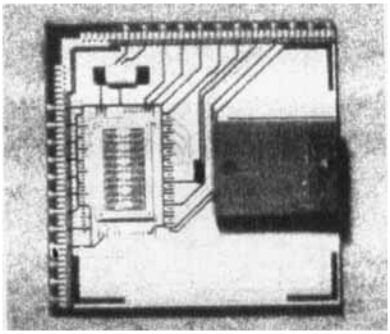


FIGURE 13 O/e-MCM-L/D with VCSEL [26] - array (*in the middle*), driver chip *(left)* and waveguide connector (*right*), before bonding, actual size: 19 x 19 mm.

The wave-guide-array with five multi-mode wave-guides (width = 50 μm and height = 20 μm) are facing the VCSEL-array (in the middle of the figure) via "45° mirrors" and the wave-guides are then going straight to the right edge (the wave-guides are mostly covered by the wave-guide connector). See reference [3] for more details.

Finally to demonstrate the feasibility of ORMOCER®s for use as an MCM L/D material a substrate for a Pentium™ MCM with a BGA interface was realised. Design and final layout were done within the EUROPRACTICE-MCM-program. The substrate of that demonstrator consists of a metal interconnection separated by ORMOCER® 3 dielectrics on top of an FR4 laminate which includes power and ground planes. Two sputtered aluminium layers (thickness 3 μm), were used for the signal interconnection. The signal layers were separated from each other and from the power plane by 6 μm thick spin-coated ORMOCER® layers in which via-holes were defined by direct photo-patterning. The FR4 laminate was provided with micro-vias and through hole vias connecting to a 1.27 mm pitch, ball grid array underneath [25]. The aim of that demonstrator was to show the possibility to realise a MCM-L/D substrate with ORMOCER®s. However, as can be seen in Fig. 14 the demon-strator was carried one step further and a Pentium™ chip set was also mounted on the substrate, but has so far not been wire-bonded.

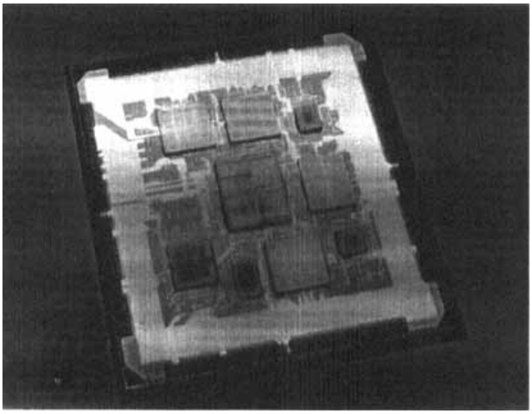


FIGURE 14 Pentium™ - MCM with BGA and bare dies

The Pentium™ MCM with BGA interface as well as the e/o-device demonstrate the possibility offered by low temperature curing materials like ORMOCER®s to achieve compact low cost devices by integration of the MCM substrate with the package.

CONCLUSIONS

ORMOCER®s, inorganic-organic hybrid materials with a unique combination of tailored properties and low-temperature (120 °C - 180 °C) curing behaviour enable the realisation of high density electrical interconnections with thin film sequentially built-up (SBU) processing on low cost polymer substrates (e.g. FR-4 or BT) as well as integrated, simultaneously processed electrical and optical interconnects due to good dielectric, optical and planarising properties.

Several advanced demonstrators with optical wave-guides and electrical multi-layer structures were realised. Chemical design of material properties based on knowledge on sol-gel and organic cross-linking reactions as well as the benefits according to the use of statistical methods for process development were demonstrated for thin film technology. The new material and process-technology is a promising candidate for low cost high density electrical and optical packaging.

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