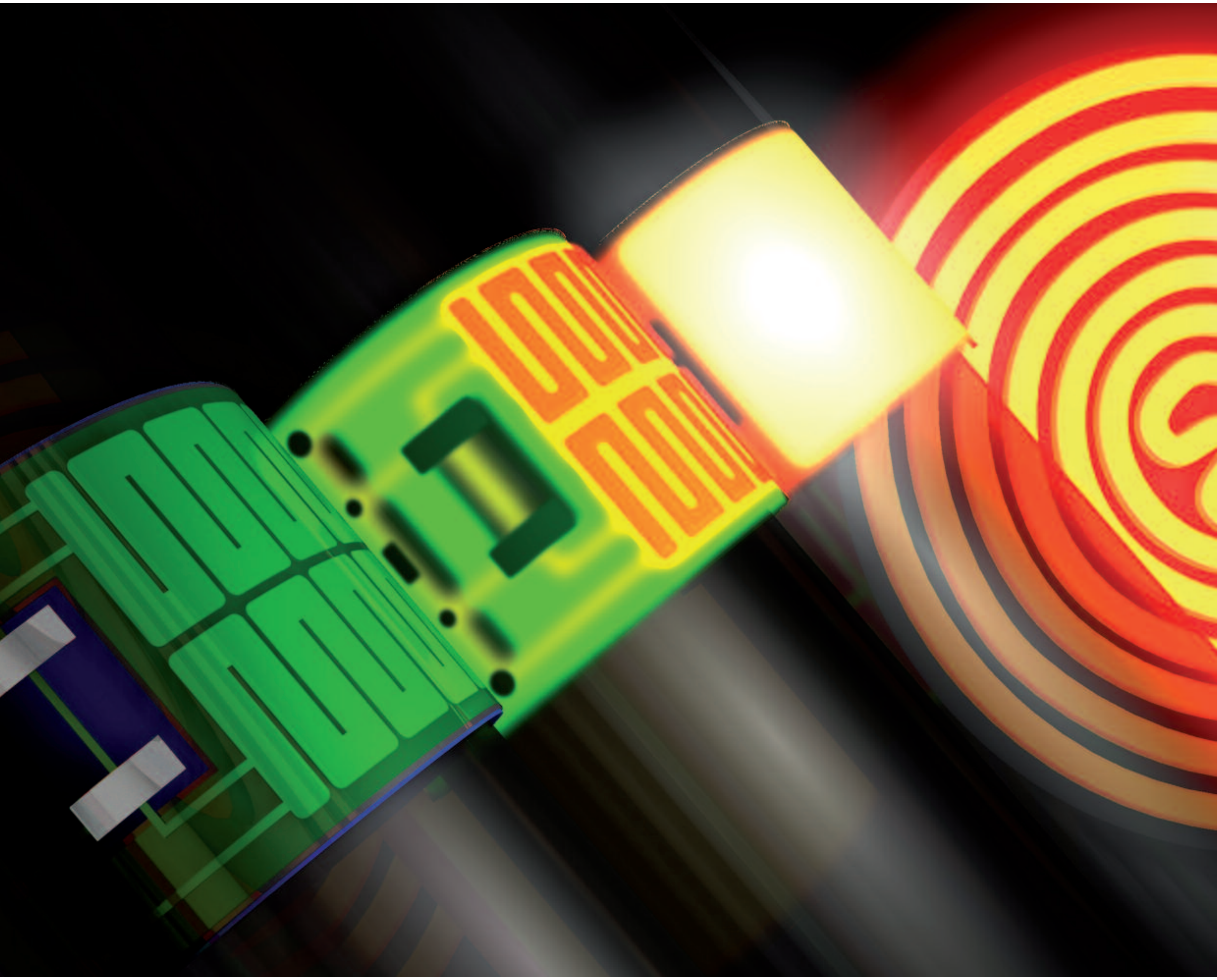
Heraeus



Product Spotlight HeraMic

Pb free system for Heating Applications on Steel Substrates

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Background

Thick Film Materials Division has developed two Pb free and REACH / RoHS compliant thick film material systems of compatible dielectrics, resistors, conductors and overglazes to manufacture high performance insulated stainless steel heater circuits and assemblies suitable for demanding concepts. In addition to our new printable paste system for established applications we developed an alternative LTCC tape / paste system to realize even complex geometries and reduce firing steps. The dielectric material fires to an extremely dense and hermetic film allowing excellent electrical performance at fired thickness of ≥ 75 µm. Excellent adhesion of heater and termination pastes leads to a reliable system. A new pure silver based resistor system enables for reduction of costs. All materials can be fired at 850 °C and are compatible with a variety of steel substrates.

Key benefits

- Extremely dense, hermetic film, allowing excellent electrical performance at fired film thickness of ≥ 75 µm
- Pure silver resistors system to reduce costs
- Dielectrics also available as LTCC to enable for special shapes and reduced firings
- Excellent adhesion of heater and termination pastes
- Free of Pb, Ni and Cd, free of phthalates
- REACH and RoHS compliant
- Acid proof overglaze to withstand harsh environments with pH values of < 1
- Highly resistant to alkaline solutions with pH values of ≤ 9

The following chart shows the available materials for heating element build-up.
All products in the chart are Pb free and RoHS / REACH* compliant.

Materials for Heating Elements

HeraMic	Paste System	Tape System
Dielectric	**SD 1010** (blue colour)	**CT 810** (blue colour)
Ag Resistor ▪ (post firing)	**SR 21 350- 25** (25 mΩ/□, HTCR 3500 ±200 ppm/K) **SR 21 350-100** (100 mΩ/□, HTCR 3500 ±200 ppm/K)	
AgPd Resistor ▪▪ (post firing)	**SR 20 150- 60** (25 mΩ/□, HTCR 1500 ±100 ppm/K) **SR 20 150- 90** (90 mΩ/□, HTCR 1500 ±100 ppm/K) **SR 20 150-180** (180 mΩ/□, HTCR 1500 ±100 ppm/K)	
Contact Pads	**SC 1001** (AgPt 99:1)	**TC 7303** (Ag)
Overglaze	**SD 1019** (green colour)	**CT 819** (clear colour)

Chart 1: Materials for heating element build-up on steel.
▪ R values at 15 µm, post fired on Dielectric IP 9117 ▪▪ R values at 13 µm, post fired on Dielectric IP 9117 (AgPd Resistor contains Pb)

Resistor pastes of each TCR group are blendable to achieve desired resistivity. The resistivity is adjusted for post-firing on dielectric (or alumina). Co-fired with dielectric tape, the resistivity is about 60 % lower than dielectric paste (or alumina). Several steel types like 1.4016 / AISI430 and 1.4521 / AISI444 (ferritic stainless steels) have been tested successfully.

During the firing process the tape shrinks only in the Z dimension, when properly laminated to the substrate. It does not shrink in the other dimensions. The total shrinkage in thickness (from tape to fired layer) is approx. 50 %.

There are also materials for alumina substrates available but these materials have not yet been fully evaluated for this application.

** REACH: EC Regulation No. 1907/2006 Registration, Evaluation, Authorization and Restriction of Chemicals by European Chemicals Agency. RoHS Directives 2002/95/EC and its subsequent amendments.*

HeraMic
Application and Customer Benefits

Process

Paste and tape materials have been tested on steel and alumina substrates. The build-up is subsequently described in figure 1. Ground layer on steel has to be an insulating layer, e.g. dielectric. Typically this layer needs to withstand a voltage of 1500 V AC for 60 s, which requires a fired film thickness of min. 75 µm. This can be achieved by three sequent print-dry-fire cycles (pdf cycles). Heater and conductor are applied in one pdf cycle for each.

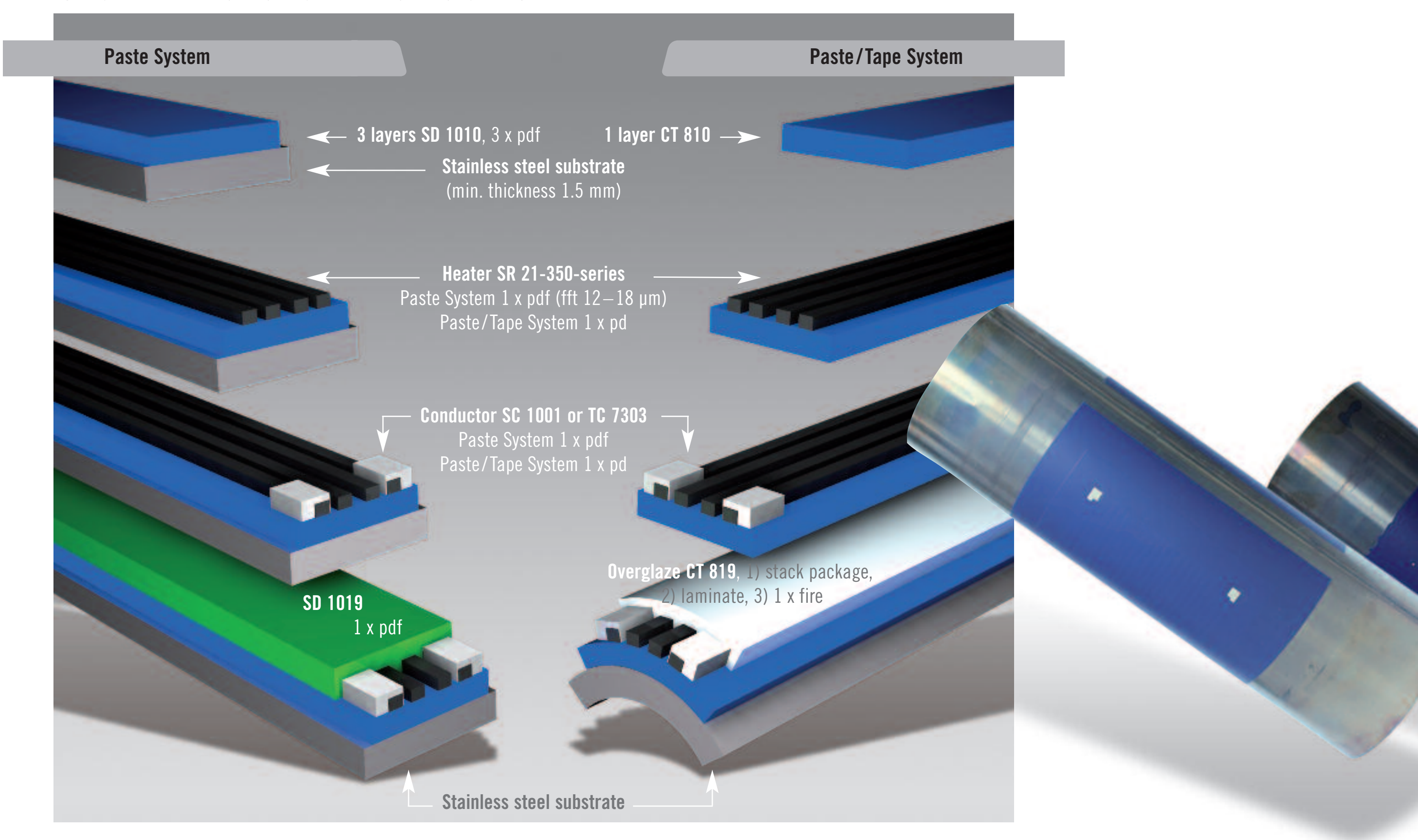
The sequence of heater and conductor can be changed. For protection of the circuit it can be covered with further insulating layers. The covering, depending on the requirements, can also be performed with other dielectric layers or overglazes. At least one pdf cycle is necessary for the coverage.

LTCC tape process

Special LTCC tape materials (dielectric and overglaze) were developed to save up to five firing steps. Heater and conductor tracks are printed and dried on the tape prior to lamination to the steel substrate. The Thick Film Printing process is performed with a standard printing machine for Thick Film applications equipped with a porous stone as nest. Using 1 layer of tape CT 810 or CT 819 on top (as overglaze) showed increased BDV values. The lamination of the tape package to the substrate can be performed by uniaxial or isostatic pressing with a pressure of 20 – 30 N/mm², a temperature of 70 – 80 °C and 1 – 15 min dwell time (depending on process and tape thickness). Firing of the laminated package can take place either in a box or a belt furnace. A standard firing profile can be used: 850 °C peak temperature with 10 min dwell time, 60 min total time. For very thick build-ups a slower profile might be necessary.

Lamination is an important process step. It is key to achieve a uniform initial adhesion of tape to the substrate. Tape thickness refers to an unfired, dried („green“) state.

Fig.1: Layers for heater build-up with paste system (left) and paste/tape system (right)



Heating element build-up with LTCC tape on steel

The aim of manufacturing heating element build-ups with tape on steel is to reduce the number of printing and firing cycles. Customer requirements are:

- Only one firing step
- Low cost
- Process capable for high volumes

Methods for providing adhesion

Different methods for providing adhesion were tested:

- Lamination, uniaxial, 80 °C, 10 min
- Glueing with a very thin adhesive tape*
- Sticking with solvent (ethanol)

Internal testing with 135 µm (unfired) CT 810 showed that lamination leads most successfully to an uniform adhesion after firing. Glueing/Sticking showed partial chip off effects on the dielectric.

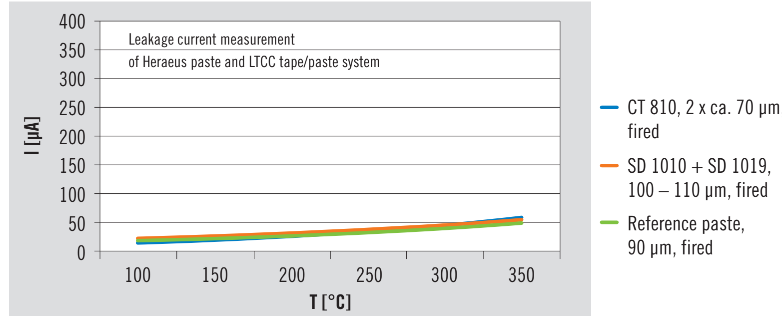
Break down voltage (BDV) tests

To understand influence of tape thickness vs. break down voltage CT 810 and CT 819 thickness and number of layers were varied. A complete heater build-up with meander heater layout (2" x 2" substrates) was manufactured and tested.

To achieve a BDV of > 1500 V for 60 seconds an unfired tape thickness of at least 140 µm is recommended for a meander heater layout. 140 µm unfired tape thickness leads to a final (fired) thickness of approx. 70 µm. This corresponds to thickness applied by paste printing. Usage of an overglaze layer leads to additional increase of BDV. An overglaze thickness of 55 µm (unfired) seems to be sufficient. For highly insulating applications, thickness should be higher.

* technology is patent-registered

Heater build-up on stainless steel tubes with fired tape CT 810, covered and uncovered with CT 810.



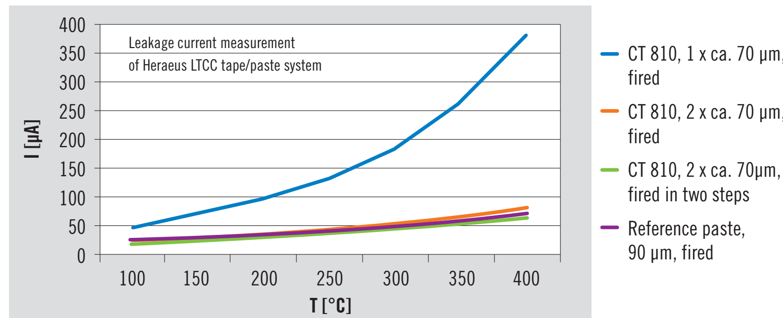


Fig. 3 + 4: Leakage measurement of Heraeus paste / tape system.

Testing parameters for leakage measurement:

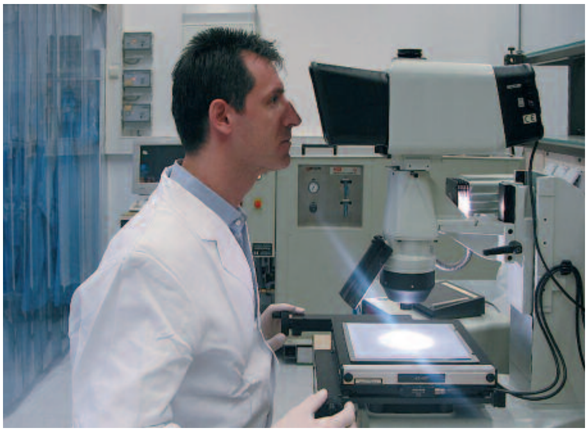
- Screens: heater: 250 mesh/45°, 40 µm film; conductor: 165 mesh/45°, 50 µm film;
- Printing parameters: 100 mm/s squeegee speed, 63 N squeegee pressure, 0.8 mm snap-off
- Drying at 150 °C for 10 minutes
- Firing at 850 °C with 10 min at peak-temperature and 60 min total pass time
- Measurement of BDV: AC, increasing in steps by 50 V, hold each step for 60 s, ambient temperature

Leakage current measurement

For some parts the leakage current vs. temperature was measured. For the measurement the heating elements were plugged and powered. The figures 3 + 4 show the leakage current for the build-ups of dielectric paste (figure 3) and tape (figure 4). 2 x 135 µm unfired tape thickness leads to a final (fired) thickness of approx. 140 µm. Our understanding is that CT 810 will work as efficient with lower final (fired) thicknesses in the range of 75 – 100 µm. Therefore the material consumption will not be higher than the traditional way of heater build-up applied by paste printing.

Heater layout

Ideally, the heater track should be circular and should not have 90° bends which may result in hot spots. Trim tracks can be included in the design to make adjustments on the fired resistor values. Spring-loaded contacts are normally used to connect these types of heaters. As a rule of thumb calculate power output of 1 W/mm² with resistor paste of 100 mOhm/sq.



Surface treatment of the substrates

LTCC tape with a green thickness of 135 µm (CT 810) was laminated on 2" x 2" steel substrates (uniaxial, 100 kN, 80 °C, 10 min) and fired. Adhesion was measured with impact test (impact force when dielectric starts to chip-off). Usually the substrate surface is polished. The tests were performed with polished, brushed and sandblasted surfaces (the surface was also cleaned with ethanol in all cases). As a result the adhesion was high for all surface-treatments, but slightly higher for brushed and sandblasted surface.

Chemical resistance of overglaze SD 1019/CT 819

Because of the high firing temperature the newly developed overglazes are highly passivated to withstand abrasion and aggressive media e.g. salts solutions and acids. SD 1019 and CT 819 withstand pH values < 1. SD 1019 and CT 819 is also resistant to alkaline solutions with pH values of ≤ 9.

Processing

- Spatulate well prior to processing
  When stored in a refrigerator allow paste to come to room temperature prior to opening, to avoid condensation.

- **Printing dielectric and overglaze**
  Insulating dielectrics are screen printed onto appropriate steel, using 165 mesh stainless steel screens. We recommend 3 separate layers by print-dry-fire sequences with 30 – 35 µm dried thickness of each layer to realize an extremely dense and hermetic film, allowing excellent electrical performance at a total fired thickness of ≥75 µm.

- **Printing resistors and conductors**
  Print through a 325 mesh screen.

- Level at room temperature for 5 – 10 minutes.

- Dry at 150 °C for 10 minutes.

- Fire at 850 °C (peak) for 10 – 12 minutes and with a total firing cycle time of 30 – 60 minutes.

Application Center – Support in Practice and Know-how

Understanding the specific application is key to exploit the advantages of the thick film and LTCC materials. 40 years of thick film experience and state-of-the-art equipment enabling for small scale prototyping will help you to find the perfect solution for your specific application.

Competent, technically experienced engineers carry out and monitor the tests either in the Application Center or on your site. Customers are welcome to provide their own materials for testing.

With our fully equipped thick film and LTCC laboratories, we have the technical capability to meet the widest range of requirements to carry out tests on customer materials.

Applications

- **Food Service**
  (Hot plates, kettles, electric pans)

- **White Ware**
  (Dish washer, washing machines)

- **Semiconductor**
  (Custom heater and test board solutions)

- **Industrial**
  (Shrink wrap tools, motor heaters, battery and oil heating)

- **Medical & Life Sciences**
  (Dialysis, blood diagnostics, DNA analysis, blood warming, fluid warming, sterilization, instrument warming)

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