

DuPont™ Green Tape™ 9K7 Low Temperature Co-fired Ceramic (LTCC) Low Loss Dielectric System for High Frequency Applications

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Abstract

The DuPont™ Green Tape™ 9K7 Low Temperature Co-fired Ceramic (LTCC) system is a major technological advancement for use in high frequency applications in consumer, telecommunications, automotive and military electronic devices. The system provides low electrical resistance at high frequencies; compatibility with low-cost silver conductors; feasibility for miniaturization; the ease to incorporate buried resistors & capacitors; and long term reliability. In this work, the new low loss dielectric LTCC systems consisting of tapes, gold and silver conductors, resistors, and capacitors are examined to evaluate the effects of chemistry, processing conditions, processing latitude, microstructure and dielectric properties.

The 9K7 tape chemistry is based on a proprietary, partially crystallizable high refractory glass and ceramic fillers which form a fired “glass ceramic-ceramic-glass” LTCC composite structure. The high frequency properties include a dielectric constant of ~ 7.1 and dielectric loss of 0.0010 at ~ 9.5 GHz with little or no variation as a function of refires. Also demonstrated is the stability of the system and its long term reliability in terms of the functional properties such as wire bonding, soldered adhesion, surface smoothness and bulk microstructures.

Key words: LTCC, Low loss, Green Tape™ Dielectric

Introduction

DuPont™ has been inventing, developing, marketing, and supporting a complete portfolio of industry leading LTCC systems for several years. LTCC combines the benefits of pure ceramic HTCC and thick film hybrid technologies to provide the ideal material system for high frequency circuit designers to further miniaturize high density interconnect designs. LTCC can use highly conductive metal systems – Gold, Silver, etc. – and provides very high levels of dielectric insulation between signal lines very high routing density, capability to add or embed passive functions (resistors, capacitors, inductors), stable dielectric properties over both frequency and temperature, uniform layer thickness CTE match to typical MMIC semiconductor materials, higher thermal conductivity than that of organic materials, very low dielectric losses, relatively smoother surface finish, etc. Moreover, being a ceramic composite LTCC is highly hermetic by nature. LTCC tape systems and metallizations are compatible with wire-bonding, soldering and low temperature brazing.

The purpose of this paper is to report the development of the newest addition to DuPont’s LTCC material portfolio, the 9K7 low-loss, microwave grade LTCC tape system and associated support materials. The 9K7 ceramic tape dielectric represents a major technological advancement for use in high frequency applications for consumer, military, telecommunications and automotive electronic devices. The 9K7 system provides circuit designers and LTCC fabricators with the ability to design and build high frequency circuits using electronic materials that offer improvements over other commercial tape systems in terms of superior dielectric properties, as well as improved process capability. In this work, the new low loss dielectric LTCC system consisting of a glass ceramic tape with compatible gold and silver conductors is examined to evaluate the effects of chemistry, processing conditions and latitude, microstructure and transmission properties. Results of microwave high frequency characterizations to evaluate transmission losses on typical interconnect structures such as Microstrip, Coplanar Waveguide (CPW), and Stripline designs resulting from lower surface

roughness were reported in an earlier paper [1]. This paper covers the chemistry, processing and materials properties of the 9K7 system in detail.

Chemistry

The 9K7 tape system is based on a new proprietary crystallizable glass with added alumina fillers for mechanical strength. On heat treatment, the glass will partially crystallize and react with fillers to give the LTCC a random composite of glass ceramic-ceramic-“remanent” glass with several crystal species within the microstructures. The presence of several crystal structures within the fired LTCC reduces preferential crystal growth. Additional crystal growth after the initial firing is kept to a minimum due to the reduced amount of “network” modifying ions in the “remanent” glass. The so-called glass “network-forming” and “network modifying” ions and the selection of the physical characteristics of the frit and filler powders are designed in such way as to reduce the surface roughness of the fired LTCC. This lack of additional crystal growth keeps the surface roughness to a minimum through six 850°C refires.

The dielectric tape is designed such that the viscosity of the “remanent” glass remains high when fired at the 850°C processing temperatures. This controls any unwanted interactions between the tape ingredients and the conductor/resistor binder systems.

Figure 1 illustrates a summary of the X-ray diffraction analysis of 9K7 fired tape surface after the initial firing at the 26.5 hour box furnace profile. Also included in the graph are the chemical analysis results for parts refired an additional six times at an 850°C/30 minute belt furnace profile. Data for three different tape lots are included.

The X-ray diffraction results indicate the presence of five different crystal species, besides the amorphous “remanent” glass, on the surface of the dielectric following the initial fire; two of the crystals are from the partial crystallization of the glass; two are a result of the interaction of filler with glass, and a ceramic crystalline structure. Examination of each set of bars shows there is no significant difference in the quantity of each crystal species: (1) between three tape lots (each manufactured with glass frit from a different glass melt) and (2) between initial fire and 6x refired parts. These results show the crystallization is at an equilibrium state after the initial firing and no further increase in the amount of crystal formation occurs with refires. This also indicates that the amorphous “remanent” glass is in a stable glass structure and further interaction with conductor/resistor binders is unlikely to occur with refires.

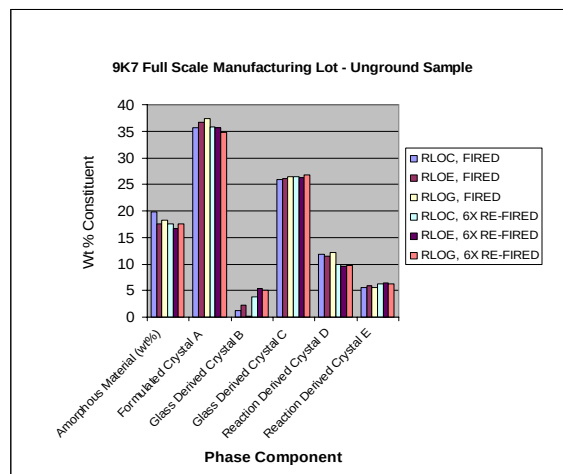


Figure 1: X-Ray diffraction analysis of the tape surface after the initial fire and 6x (850°C) refires

DuPont™ Green Tape™ 9K7 Dielectric Properties

9K7 Green Tape’s as-fired typical physical and electrical properties are listed in Tables 1 and 2.

Table 1: Typical Physical Properties

Physical Properties	Value
Unfired thickness, (um)	127 +/- 9; (9K7)
	254 +/- 14; (9K7X)
X, Y shrinkage, (%)*	9.8, +/- 0.3
Z shrinkage, (%)*	10.1, +/- 0.3
TCE, (ppm, 23° - 300° C)	4.4
Density, (g/cm ³)	3.1
Camber (inch/inch)	0.001
Surface roughness, (um)	0.52
Thermal conductivity, (W/m-K)	4.6
Flexural strength, (MPa)	230
Young’s modulus, (GPa)	145
Poisson’s ratio	0.25

Table 2: Typical Electrical Properties

Electrical Properties	Value
Dielectric constant, (10 GHz)**	7.1
Loss tangent, (10 GHz)**	0.0010
Insulation resistance, (Ohms)	> 10 ¹²
Breakdown voltage, (kV/25 um)	>= 1100

* 3.5 hr. belt furnace profile

** Measurements using split cylinder cavity method

With 9K7, the system integrators/OEM customers will see benefits versus not-in-kind organic laminates (LCP, Duroid® and variants) in the form of ~30% reduction in footprint, embedded resistor capability, better hermeticity and ~80% better TCE match with GaAs and Si die.

The system offers processing advantages to the transactional customer (fabricator) versus other merchant tape systems. These advantages include ~25% lower loss, a 10 – 15% increase in mechanical strength and significantly improved reflow (6x) stability of dielectric constant (~60%) and surface roughness (~90%).

Dielectric Tape Stability

To establish the dielectric's stability, three 9K7 tape lots were fired under identical conditions at an 850°C/26.5 hour box furnace profile. The dielectric properties of each lot were measured using a split cylinder cavity method at 9.5 GHz after the initial fire and following 3x and 6x reflows at a standard 850°C/30 minute profile. The observed variations in dielectric constant and loss tangent are shown in Figures 2 & 3. Figures 4 & 5 show the reflow stability of surface roughness and shrinkage. The results demonstrate 9K7 has significantly stable dielectric properties and shrinkage performance when subjected to multiple reflows – a significant improvement versus other low loss tape systems. As stated earlier, the selection of constituents and the processing conditions employed result in the 9K7 base material achieving an almost complete crystallization and crystal growth during the initial firing. The chemistry of the material composition is such that little or no network modifying agents are present after the initial firing and no further micro-structural changes occur with reflows. This is the reason for the exceptional reflow stability of the material system.

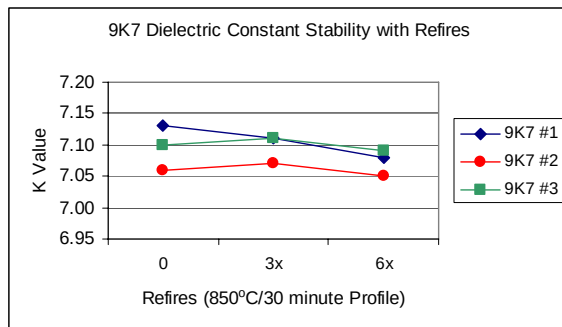


Figure 2: Dielectric constant variation with reflows

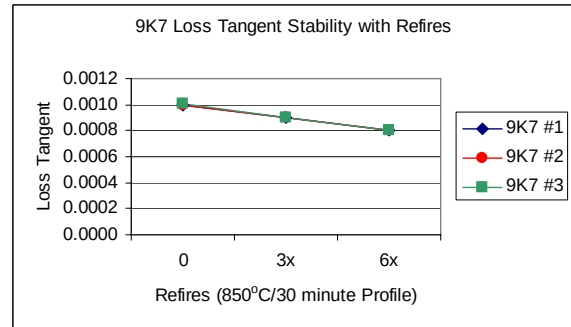


Figure 3: Change in loss tangent with reflows

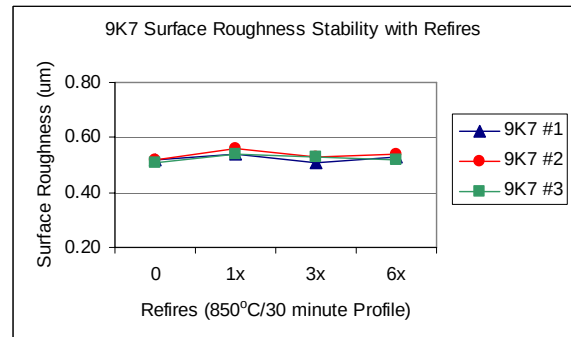


Figure 4: Surface roughness with reflows

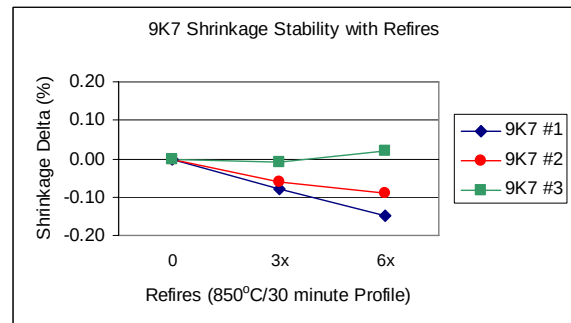


Figure 5: Shrinkage delta with reflows

The lot-to-lot variation of dielectric constant of twenty five production lots was characterized using the split cylinder cavity technique at 9.5 GHz. Six samples from each lot were measured and lot-to-lot variations of dielectric constant and loss tangent were analyzed. The results of this analysis are shown in Figures 6 and 7.

It is clear from the test data that the dielectric constant is very tightly distributed within the expected specification range of 7.1, +/- 0.2. The loss tangent of all lots was found to be less than 0.0012 at a test frequency of 9.5 GHz.

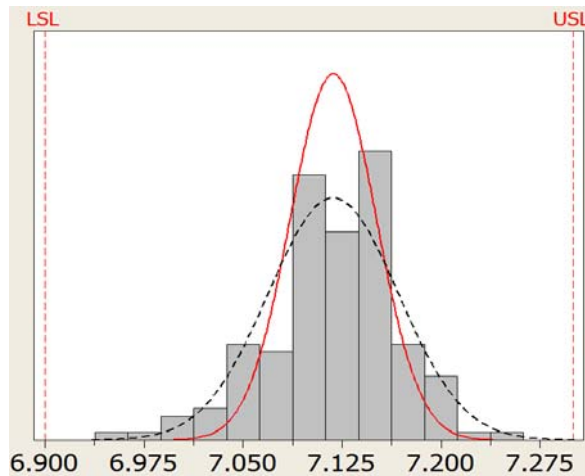


Figure 6: Distribution of Dielectric Constant – (25) tape lots

9K7 Green Tape™ System Components

The 9K7 tape system is comprised of two dielectric tape thickness offerings and a compatible set of cofired and post fired Au and Ag conductors. The dielectric tape is available in 0.005" (9K7V) and 0.010" (9K7X) green sheet thicknesses for use in various design-specific applications. A summary of the 9K7 Au and Ag conductor compositions is listed in Table 3.

Table 3: 9K7 Au and Ag Conductor Systems

Application	Gold	Silver
External signal line, 1 mil Au wire bond	LL507	
External signal line, 2 mil Au wire bond	LL507	
External ground plane	LL507	LL617
External solderable	LL509	LL617
Internal signal line, 1 mil Au wire bond	LL505	
Internal signal line		LL612
Internal ground plane	LL505	LL602
Via fill, standard printing	LL500	LL600
Via fill, high speed printing		LL601
Buried resistor (blendable) - low end		HFB12
Buried resistor (blendable) - high end		HFB22
Post fired, solderable	4597	7484
Post fired, braze	5062D 5063D	5081 5082

Gold System: HiRel applications targeting aerospace, military and other industries require the use of gold conductor materials due to their wire bonding, corrosion resistance and metallization migration resistance properties. DuPont™ has developed as set 9K7 conductors to meet this need

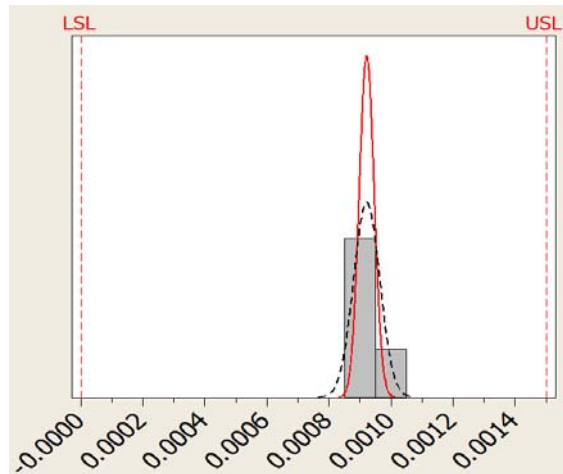


Figure 7: Distribution of Loss Tangent – (25) tape lots

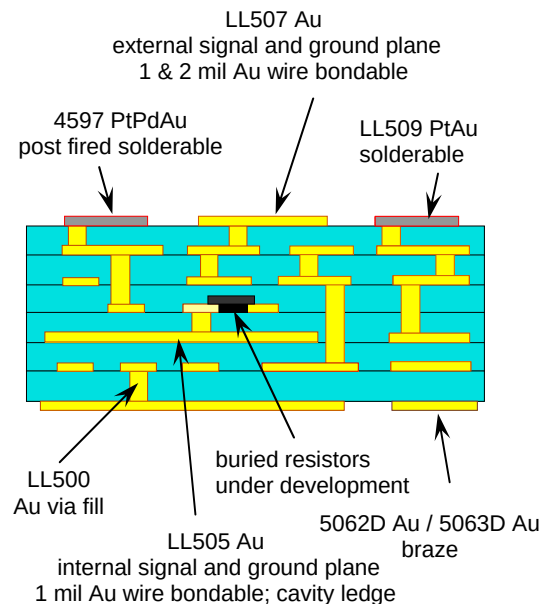


Figure 8: Gold Conductor System Components

and they are identified in Figure 8. Evaluations of compatible buried resistor blend members for the Au system are currently undergoing R&D trials.

Silver System: A silver-based conductor system is available to address high volume, low cost structure applications such as the automotive and telecommunication fields. 9K7's silver conductor system members are comprised of the conductors shown in Figure 9.

The two Ag via fill compositions, LL600 and LL601, were designed for use under different types of processing conditions. LL600 is used where traditional, slower speed printing operations are

employed. In contrast, LL601 was specifically designed for high speed via fill applications where the LTCC punchable backing serves as a carrier film throughout the build process. The application differences dictate the rheology of the two pastes be significantly different from one another.

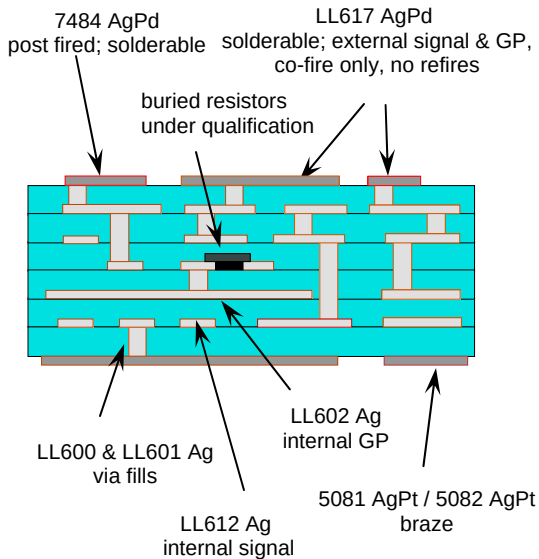


Figure 9: Silver Conductor System Components

The LL617 AgPd conductor is recommended for use in cofire-only, solderable Ag part builds. The post fired, solderable 7484 AgPd should be used when one or more post firings is required. R&D efforts are currently underway to develop a cofirable, solderable Ag-based conductor material capable of withstanding multiple 850°C refires.

The post fired 5081/5082 AgPt braze and 7484 AgPd solderable conductors compliment the Ag conductor materials set. Compatible buried resistor blend member compositions, HFB12 (20 Ohms) and HFB22 (200 Ohms), are currently undergoing further qualification testing.

9K7 Module Fabrication Process

The build techniques, process flow and equipment required to fabricate 9K7 LTCC modules are very similar to those used for other DuPont™ Green Tape™ LTCC systems. The initial cofire requires a 26.5 hour firing profile. The use of dedicated alumina-clad setters (~100 to 200 um surface Ra) prevent parts from sticking during the co-fire and re-fire processes.

The techniques and process parameters used for the green sheet conditioning, via punching, conductor printing and lamination portions of the

fabrication process are the same as those employed with other DuPont™ LTCC systems.

Daisy Chain / Stacked Via Fill Integrity

To determine the compatibility and integrity of the 9K7 tape, via fill and conductor materials with each other, the compositions were evaluated in “daisy chain” test patterns and stacked via arrays.

The daisy chain pattern consists of an array of 308 vias, each 0.010” in diameter, and a 0.006” wide serpentine daisy chain trace that is 5133 squares in length. Following the via fill step, the conductor trace patterns are aligned and printed such that the vias are alternately interconnected on the top and bottom surface of a single tape layer. Each printed sheet is then laminated to several blank tape layers and co-fired at the 26.5 hour profile.

Following the initial co-fire and each subsequent 850°C/30 minute firing through 10x refires, circuit continuity and resistance was measured using a 4-point probe method. Sample parts are removed for cross-sectioning following the initial co-fire and predetermined re-fire intervals for SEM micro-structural analysis purposes.

Figures 10 and 11 display the daisy chain re-fire characteristics of the LL505/LL507 Au and LL612 Ag conductor compositions when tested in the daisy chain pattern. The resistivity of the LL505 Au / LL507 Au test circuits increase approximately 6% after 6x refires. The initial trace resistivity of silver system was approximately 10 ohms (~2.0 milliohms per square excluding vias; the 6x refires approximately 12.5 ohms (~ 2.5 milliohms per sq. excluding vias).

Separation of a single via from the conductor line or a single line break within the 5133 squares of signal lines will result in an infinite resistivity. The gradual increase in resistivity after each re-fire is due to the combination of sintering differences, if any, and the increase in interactions of the conductor and tape ingredients.

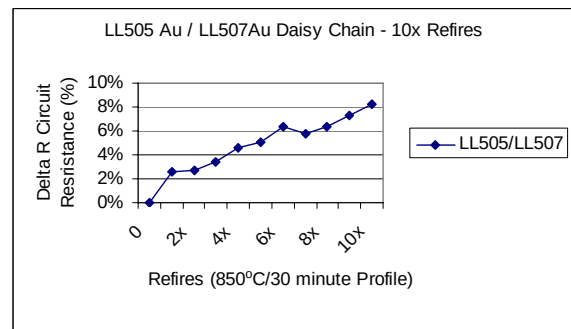


Figure 10: LL505/LL507 Daisy chain with refires

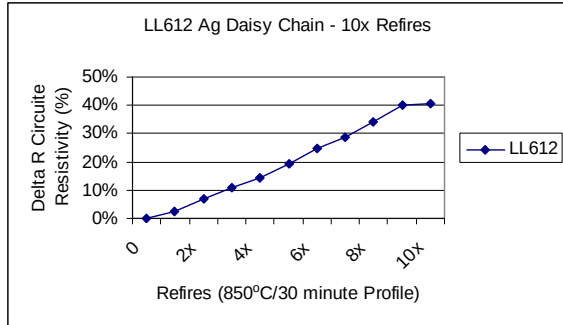


Figure 11: LL612 Daisy chain with refires

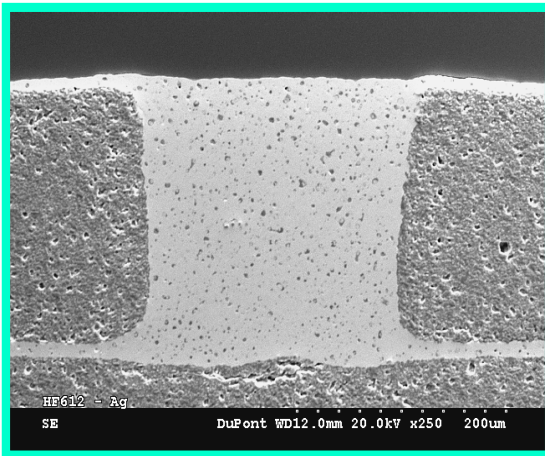


Figure 12: Cross-section of LL601 Ag via fill and LL612 Ag conductor in daisy chain test pattern

The microstructure in Figure 12 shows that even after ten refires, the via fill and dielectric remain well bonded to each other. Also, the via fill conductor interface connections at the top and bottom signal line conductors are excellent.

Stacked via fill arrays are incorporated into LTCC structures as a method of shielding high frequency circuits from outside electrical interference and providing an avenue for the heat dissipation and thermal management of high power integrated circuits and other devices. Since LTCC designs often incorporate ten or more tape layers, it is important to have a via fill composition's Z-shrinkage closely match that of the surrounding tape layer. In doing so, the overall fired posting of a stacked via array can be minimized. Having a minimal amount of via posting or variations in the module's surface topography is desirable when applying post fired prints, attaching discrete components, seal rings or other devices. Figure 13 is an example of an LL601 Ag stacked via fill array in 10 layers of 9K7X tape.

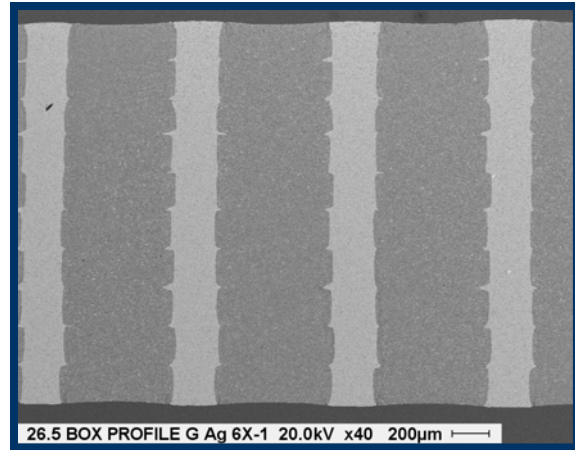


Figure 13: Stacked LL601 Ag via fill in 10 layers of 9K7X (10 mil) tape

Wire Bond Adhesion

Hermetic die attach packages require the use of wire bondable conductors to serve as attachment pads for wire interconnects between the LTCC module's circuitry and the silicon die. In high reliability designs, such as those targeting the aerospace and defense industries, Au conductors are used as the wire bond attachment pads on the LTCC module. The 9K7 Au conductor system offers two co-firable, wire bondable conductors, LL505 and LL507 - each of which is designed for a specific purpose.

LL505: internal signal line and ground plane conductor. It is wire bondable with 1 mil Au wire and can be used in applications where an internal signal trace transitions to the wire bond shelf of an external cavity. When incorporated as a ground plane configuration, a cross-hatch pattern is recommended in areas where solid metallization prints are not required.

LL507: co-fired, external, wire bondable Au composition. Its purpose is to serve as an external, wire bondable signal line as well as ground plane conductor. The material is wire bondable with both 1 and 2 mil Au wire. LL507 should not be used for internal conductor signal line or ground plane applications.

Figures 14, 15 and 16 illustrate the aged wire bond adhesion performance of LL505 and LL507 as a function of 850°C refires and thermal aging at 150°C. Both compositions demonstrated very good performance across the entire 1000 hour testing interval.

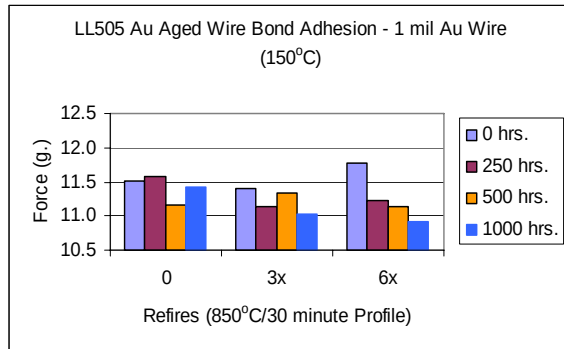


Figure 14: LL505 aged wire bond adhesion – 0.001” Au wire

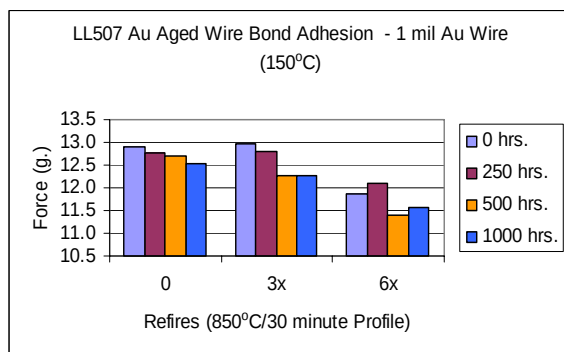


Figure 15: LL507 aged wire bond adhesion – 0.001” Au wire

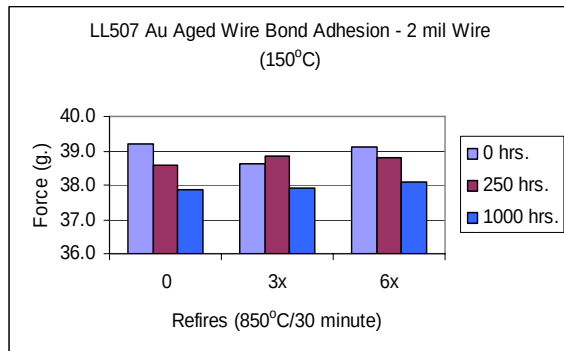


Figure 16: LL507 aged wire bond adhesion – 0.002” Au wire

Module Hermeticity

Hermeticity is vital for the successful and long term reliability of LTCC designs - particularly those addressing the aerospace and defense industries where performance is critical under harsh environmental or operating conditions. Non-hermetic packages provide a route by which moisture

or other contaminants can easily enter the cavity area and cause corrosion, premature failure or performance variability of integrated circuits and other critical components.

Brazed Adhesion

One of the benefits of LTCC technology is the ability to provide customers with a hermetic, integrated systems package solution. To achieve this, the material system must have the capability of providing the customer with a high temperature soldering (brazing) option for applications requiring the attachment of items such as pins, lead frames, seal rings and heat sinks. The following work evaluated the brazed adhesion performance of the standard DuPont™ 5062D/5063D Au & 5081/5082 AgPt braze conductors using 80%Au/20%Sn solder and a 340°C brazing profile with nitrogen firing atmosphere. The use of a 340°C brazing temperature range preserves the hierarchy in the processing of the low temperature fired systems, while retaining the same die attach and lid seal processes commonly used in the industry [2].

Testing was performed using the standard DuPont™ braze test pattern (0.072” pad diameters) and part preparation methods. For the respective metallurgies, the 5062D or 5081 adhesion layers were printed, dried, fired on a set of previously fired, unmetallized 9K7 blank substrates. This was followed by a subsequent print, dry, fire of the 5063D or 5082 barrier layer prints directly over the respective Au or AgPt adhesion layer prints. All conductor firings were at the standard 850°C/60 minute profile.

Half of the fired parts from each group were refired four additional times at the same 850°C/60 minute profile. The refires were performed to simulate the stress the initial barrier layer prints would experience if subjected to two additional adhesion/barrier layer firing cycles.

For the brazing process, a machined graphite fixture was incorporated to hold the pre-coated Kovar test pins in place over the fired metallization prints as the parts went through the brazing profile. The diameter of the pin heads was 0.035 inches.

Samples from each of the test configurations were submitted for aged 150°C and -40°C/+125°C thermal shock pin pull adhesion evaluations through 1000 hours and 1000 cycles test intervals. Test parameters for the thermal cycle testing were a one hour cycle, air-to-air; 30 minutes at each dwell with less than a 5 second transfer rate between temperature extremes.

Both the 5062D/5063D Au and 5081/5082 PtAg braze metallization systems performed very

well when post fired on 9K7. Initial and aged adhesion values were ~23 pounds throughout the 1000 hour/1000 cycle life of the tests. Results for the sample groups subjected to the four additional post firings were in the ~23 pound range as well.

This series of evaluations identified the Au and Ag-based braze metallizations as having adhesion values well in excess of the standard 15 pound target value commonly used. Figures 17 and 18 exhibit the aged and thermal shock test data for the 5062D/5063D Au materials. Performance was very similar for the silver-based 5081/5082 AgPt set.

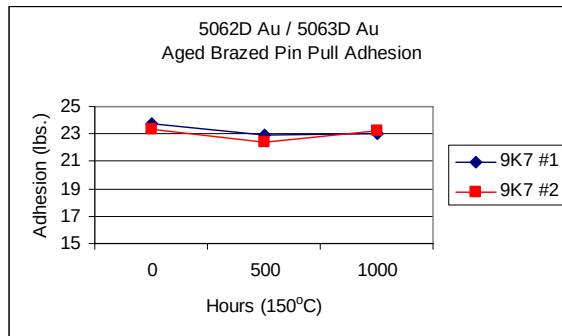


Figure 17: 5062D/5063D aged brazed adhesion

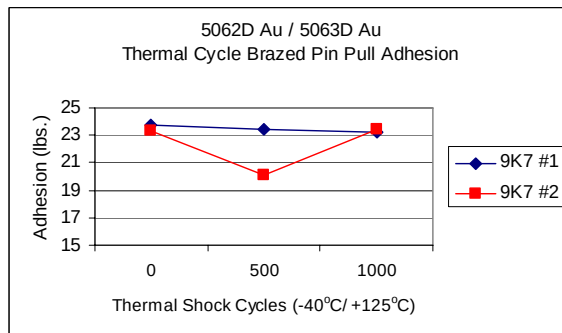


Figure 18: 5062D/5063D TC brazed adhesion

Solderability Properties

For low temperature soldered component or soldered lead attach applications, the 9K7 system offers a cofire-able and solderable conductor composition for each metallization system – the LL617AgPd for Ag and LL509 AuPt for Au designs. See Table 4 for the typical material properties of the solderable conductors

As noted earlier, the LL617 AgPd conductor is recommended for use in cofire-only, solderable conductor applications. Ag-based designs requiring soldering and 850°C refires should use 7484 AgPd in a post fired configuration.

Table 4: Cofired, solderable conductor properties

	LL617 AgPd	LL509 AuPt
Resistivity (mOhms/sq. @ 15 um)	< 55	<160
Solder acceptance (%)	> 95	> 95
Solder leach resistance (# dips)	5 - 7	4 - 5
Aged adhesion - cofire (N.)	> 15	>18
Aged adhesion - 3x refire (N.)	NA	> 18
Aged adhesion - 6x refire (N.)	NA	> 15

1. Resistivity: data normalized to 15 um dry thickness.
2. Solder: LL617, 62Sn/36Pb/2Ag; LL509, 63Sn/36Pb
3. Aged adhesion: 1000 hours at 150° C

Conclusions

The 9K7 system provides the fabricator and OEM with a low loss dielectric tape system having the stable, lot-to-lot physical and high frequency electrical performance properties necessary for the fabrication and operation of sophisticated, low loss LTCC module designs. The consistency and refire stability of the new tape system has been demonstrated on numerous material lots as a function of lot-to-lot stability with initial fires as well as multiple 850°C refires.

Acknowledgements

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References

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Trade Marks

Note: References within this paper to DuPont, 9K7, 9K7V, 9K7X, 9K7 tape system and 9K7 Green Tape™, apply to the DuPont™ Green Tape™ 9K7 Low Temperature Co-fired Ceramic (LTCC) System.