J. Am. Ceram. Soc., 93 [8] 2274–2280 (2010)
DOI: 10.1111/j.1551-2916.2010.03740.x
© 2010 The American Ceramic Society

journal

Microwave Sintering of Multilayer Integrated Passive Devices

Balasubramaniam Vaidhyanathan,*,† Ketharam Annapoorani, and Jon Binner*

Department of Materials, Loughborough University, Leicestershire LE11 3TU, U.K.

Ramesh Raghavendra‡

Littelfuse Ireland Limited, Ecco Road, Dundalk, Republic of Ireland

Microwave sintering of multilayer capacitor/varistor-based integrated passive devices (IPDs) has been investigated for the first time. The sintered samples were characterized for density, microstructure, composition, and electrical performance. It was found that IPDs with varistor/capacitor formulations could be microwave sintered to fully dense device components within 3 h of total cycle time, which is $<1/10$th of the time required by conventional methods. Microwave sintering resulted in products with a finer grain structure and without delamination or significant interdiffusion between the ceramic/electrode and varistor/capacitor interfaces. The microwave method also completely eliminated the need for a separate binder burnt-out step. The electrical properties of the microwave-sintered samples were found to better or match those obtained by conventional, industrial processing. In general, the simplicity, rapidity, and superior product performance make the microwave technique an attractive sintering methodology for the processing of IPDs.

I. Introduction

Modern electronic circuits are much more vulnerable to transient overstresses than earlier circuits, which used relays and vacuum tubes[1], and hence progress in the development of faster and denser integrated circuits (such as MOSFETs) has been accompanied by an increase in system vulnerability. The variety and severity of voltage transients have also increased over the decades; therefore, the development of multilayer varistor[§] (MLV) technology has evolved to provide adequate and reliable transient protection.[2] The various kinds of transient suppressors used in electronic devices include zener diode, spark gap, SiC, and ZnO varistors. Among these, ZnO-based varistors are the most preferred due to their versatility and superior properties; they are found to be very effective in suppressing high-amplitude and low-frequency transients.[3–6] However, they are less effective in suppressing high-amplitude, high-frequency transients[7] such as pulse interference generated by electric motors,[8] or malfunctions from electromagnetic interference (EMI) in the peripheral I/O ports of handheld wireless devices. To guard against EMI and high-amplitude, high-frequency noise, multilayer capacitors (MLCs) are typically used.[1] Owing to their huge demand, passive components such as MLVs and MLCs constitute a billion-dollar market.[9]

Given the drive for component count reduction and miniaturization in the electronic industry worldwide, the use of integrated passive components is becoming a necessity. Integration of multiple passive components into a single-integrated passive device (IPD) offers significant benefits in terms of multifunctionality, assembly cost, and space reduction. A combination of a cofired MLV and MLC is one example of an IPD[1], others being capacitor/inductor, varistor/ferrite, and capacitor/resistor combinations, and the cellular/mobile phone market is a key target for these devices. However, the integration of passive components has brought many material, electrical, mechanical, and production challenges.[7,10] Cofiring of multiple ceramic phases and with metal interlayers is fraught with difficulty in that differentials in sinter shrinkages, shrinkage rates, and thermal expansion coefficients must be minimized in order to reduce the delamination between materials and cracking in one or more layers. Possible phase changes, for example in $BaTiO_3$-based capacitors, can also contribute to an inability to fabricate IPDs with full integrity. The conventional cofiring procedures of these multilayer devices involve separate multistep binder removal stages and long sintering schedules, and hence are often of lengthy duration.[1] The conventional processing of IPDs also suffers from electrode delamination and unwanted inter diffusion of the matrix components at the high processing temperatures required, a key concern when dealing with electrical properties.[11] The high sintering temperatures and the associated high energy consumption necessitate strategies based on alternative processing methodologies.

Because IPDs have a considerable commercial market, any improvements made in their processing methodology could have a significant economic impact. The present study aims to use a microwave sintering technique for the efficient processing of these devices. Although microwaves have been used for the processing of a number of inorganic and organic materials,[12–18] they have not been investigated in detail previously for the sintering of IPDs to the knowledge of the authors.

II. Experimental Procedure

An IPD formulation consisting of an MLC on one side and an MLV on the other with no interlayer or barrier material at the interface was provided by Littelfuse Ireland Limited (Dundalk, Ireland). The average size of a typical IPD sample was 2.3 mm $\times$ 1.5 mm $\times$ 1.8 mm in length, breadth, and thickness, respectively. The capacitor formulation was bismuth niobate doped with magnesium oxide; this avoided the complications arising from the volatile nature of PbO-based capacitor formulations,[7,10] while the varistor formulation consisted of 98% zinc oxide and 2% additions of a range of additives including bismuth oxide, antimony oxide, and cobalt oxide.

The microwave sintering experiments were performed in a multimode cavity, Fig. 1, operating at 2.45 GHz and capable of providing an adjustable output power of up to 6 kW.[19] The microwaves from the magnetron were transmitted through a waveguide system and launched into the cavity after reflecting onto a

L. Levinson—contributing editor

Manuscript No. 27172. Received November 28, 2009; approved February 14, 2010.
Part of the results were presented at the 33rd ICACC meeting in January 2009, Daytona Beach, FL, USA, organized by the American Ceramics Society.
This work was financially supported by Littelfuse Ireland Limited.
*Member, The American Ceramic Society.
†Author to whom correspondence should be addressed. e-mail: b.vaidhyanathan@lboro.ac.uk
‡Present address: South Eastern Applied Materials Research Center (SEAM), Waterford Institute of Technology, Waterford, Ireland.
§Varistor is a voltage-dependent resistor whose primary function is to sense and limit voltage surges and hence protect sensitive circuit components.

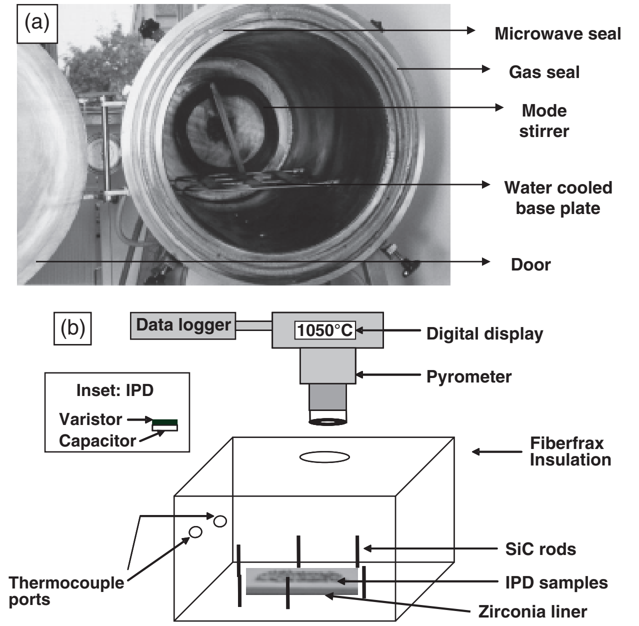

Fig. 1. (a) The microwave cavity used and (b) schematic of the experimental arrangement for the sintering of the integrated passive device (IPD) samples. The inset shows the different parts of the IPD.

mode stirrer rotating at 70 rpm. The cavity was provided with a water-cooled base plate to hold the samples and an outer cooling jacket for safety. A reflected power monitoring unit and a three-stub tuner helped to tune the microwave system with minimum reflected power. In conel sheathed, shielded K-type thermocouples and an infrared pyrometer (LAND Instruments, International Ltd., Dronfield, Derbyshire U.K.) were used for temperature measurement.[20,21] The thermocouple was placed horizontally very close (<1 mm) to the pile of IPD samples. The samples were placed on a ceramic liner in the same arrangement used commercially with conventional furnaces, as indicated in Fig. 1(b). Temperature calibration was achieved using the boiling point of water and the melting point of vanadium oxide in the microwave cavity. The field of view of the pyrometer was <2 mm in diameter, using a close-up lens.

Fiberfrax high-purity insulation was used as a casket for heat containment with SiC rods being used as secondary susceptors to ensure thermal homogeneity.[18,22] Around 300 IPD samples were used for each microwave sintering trial, which were

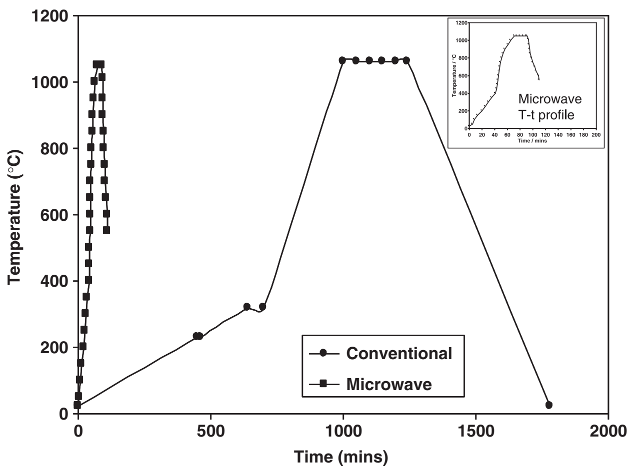

Fig. 2. Comparison of microwave (current work) and conventional (industrially used) time–temperature profiles for the processing of integrated passive devices. The time-and energy-efficient nature of the microwave method is evident. The inset shows an expanded view of the microwave heating profile.

Table I. Densification of Integrated Passive Devices (IPDs) in Conventional and Microwave Processing

	Density (g/cm^3) Error $\pm 1\%$
Green IPD	3.71
Sintering conditions	Conventional sintering
(Control) 1060°C/4 h	5.85
	Microwave sintering
1025°C/20 min	5.54
1050°C/20 min	5.90
1100°C/20 min	5.95
1050°C/30 min	5.92
1050°C/45 min	5.95

performed at 950°, 1000°, 1025°, 1050°, and 1100°C with the soaking time being varied between 20 and 60 min. Cooling was achieved by switching off the microwaves and allowing the system to cool naturally. Conventionally processed IPD samples sintered at 1060°C for 4 h using an optimum industrial processing schedule involving 30 h of total cycle time, Fig. 2, were used as a control. For comparing the densification kinetics of the two heating methods (microwave and conventional), further conventional sintering experiments were also performed using 10°C/min heating and cooling rates with a 3-h soaking time, involving a total cycle time of nearly 480 min in an electric furnace.

Both geometrical and bulk density measurements were performed on 15 representative sintered IPD samples for every set of conditions; the bulk density measurements were obtained

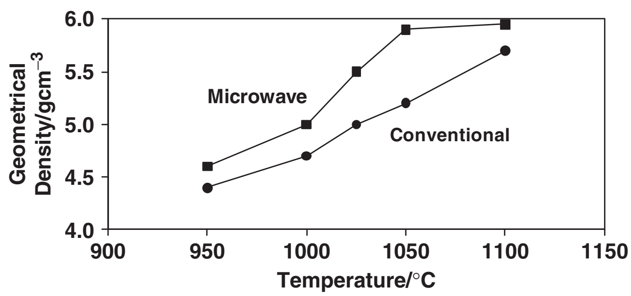

Fig. 3. Densification of integrated passive devices in microwave (20-min soaking at temperature) and conventional (3 h soaking) sintering experiments.

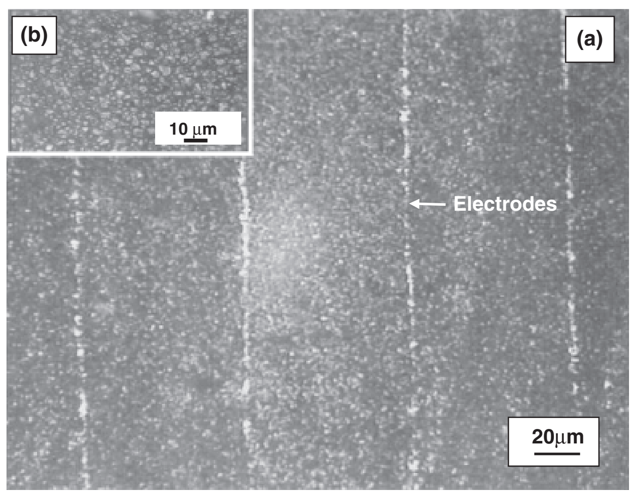

Fig. 4. (a) Optical micrograph of the (as-prepared—side view) microwave-sintered integrated passive device sample—the varistor side, indicating that the electrodes are intact without delamination and (b) the inset shows a high-magnification image of the varistor indicating rounded grains with good grain–grain boundary contrast.

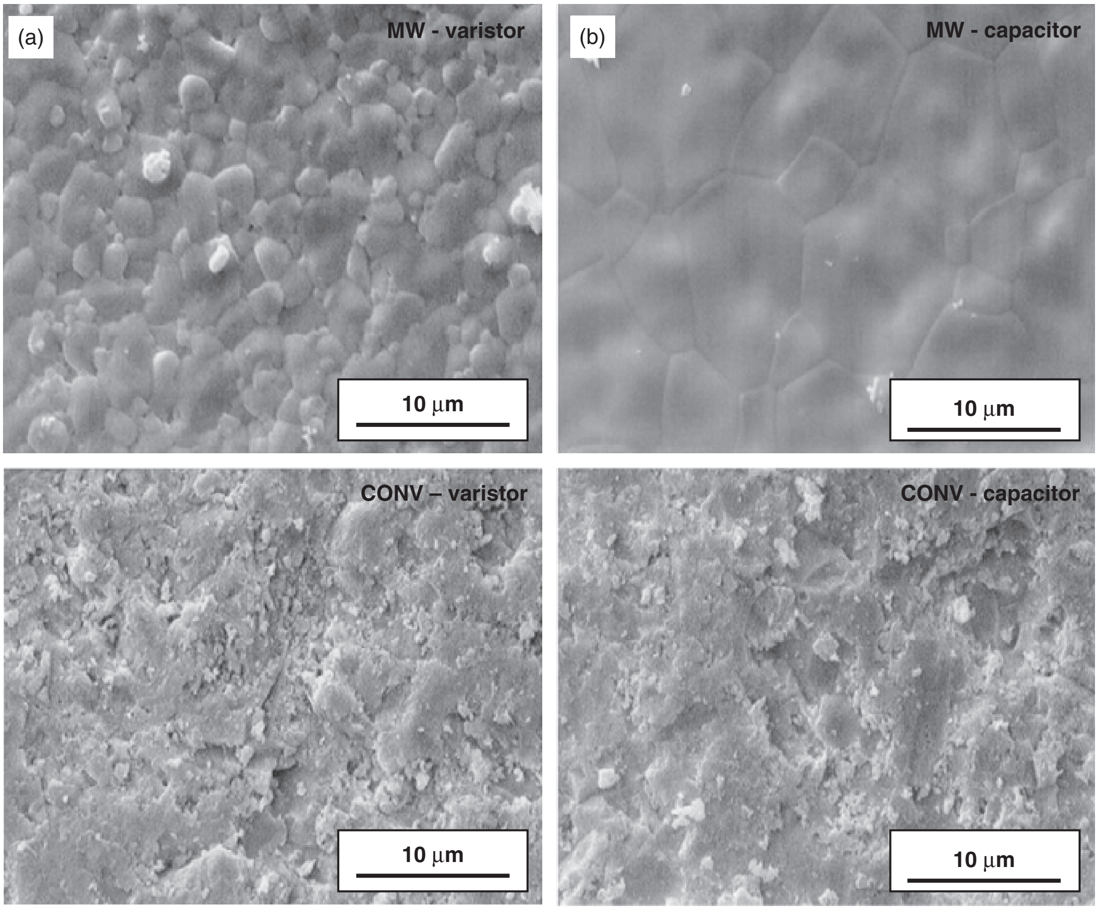

Fig. 5. Scanning electron micrographs of the (as-prepared) microwave and conventionally sintered integrated passive devices, (a) varistor and (b) capacitor regions. MW, microwave processing (1050°C/20 min); CON, conventional processing-control (1060°C/4 h).

using the Archimedes method and deionized water as the immersion medium; the mass was measured using a Sartorius balance (Sartorius, Goettingen, Germany) having an accuracy of 10^{-4} g. Because the samples were a combination of two different components plus the metallic interlayers, the exact theoretical density of the IPD was not available. The samples were also investigated using optical microscopy (Olympus Microscopes, Tokyo, Japan) for electrode delamination and interface cracking after being mounted and finely polished using diamond polishing (DAP-2, Struers Ltd., Rotherham, U.K.). Scanning electron microscopy (SEM) was performed (using Leica Cambridge Stereoscan S360, Cambridge, U.K.) for the analysis of the microstructure. The composition of the device components and the extent of diffusion of niobium and bismuth at the interface were studied using energy-dispersive X-ray analysis (EDX). High-resolution field-emission gun SEM (FEG-SEM; Leo 1530VP, Cambridge, U.K.) was also used for a more detailed microstructural analysis of some IPD samples.

The varistor-type electrical properties of the sintered IPD samples were measured at Littelfuse Ireland Ltd. (Dundalk, Republic of Ireland). *I–V* characteristics from 0.1 to 0.01 A was measured using a Keithley instrument (Model 2410, Cleveland, OH) and from 0.1 A to 100 A using a Keytek (Model 711, Lowell, MA) surge generator. Around 100 representative samples for each experimental condition were examined for electrical performance. The electrical results were used as a pointer for optimizing the experimental parameters of the subsequent microwave sintering trials.

III. Results and Discussion

From the initial microwave sintering experiments, it was observed that a maximum of 1.5 kW of microwave power was found sufficient to fully sinter the IPD samples at the optimum temperature, 1050°C. A typical time–temperature profile for microwave sintering is shown in Fig. 2 (inset). The entire cycle took only 110 min, $<1/10$th of that required for the processing of IPDs by the current industrial practice. The microwave procedure did not require a separate binder burn-out stage, which was a great advantage (probably due to the inside-out heating ability of the microwave method) because this saved both time and energy.[14,21]

Table I provides the green and sintered densities of the IPD samples processed using both conventional and microwave methodologies. The microwave-sintered IPDs achieved the required level of densification after 20 min of soaking at 1050°C, although increasing the time at temperature resulted in further slight increase in densification. At temperatures >1050°C, the capacitor region sintered more than the varistor part, leading to nonuniform shrinkage and delamination.

Figure 3 compares the densification of the IPDs over a range of temperatures using both conventional and microwave sintering. Note that the microwave sintering involved just 20 min at the sintering temperature while the conventional densification was performed using a 3-h soaking time. It is clear that at any given temperature the microwave process provides a much higher densification than conventional sintering, a result that has been observed before in many other systems.[14,23,24] Although the exact mechanism for the enhanced densification is not yet clearly understood, the electric field component of the EM radiation could be playing a crucial role in enhancing the densification kinetics during the microwave sintering of electroceramics.[21,25,26]

Optical microscopy of the samples microwave sintered at 1050°C for 20 min revealed that the metal electrodes (average electrode spacing = 69 μm) in these devices were fully intact, Fig. 4, without delamination and no electrode "buckling" was

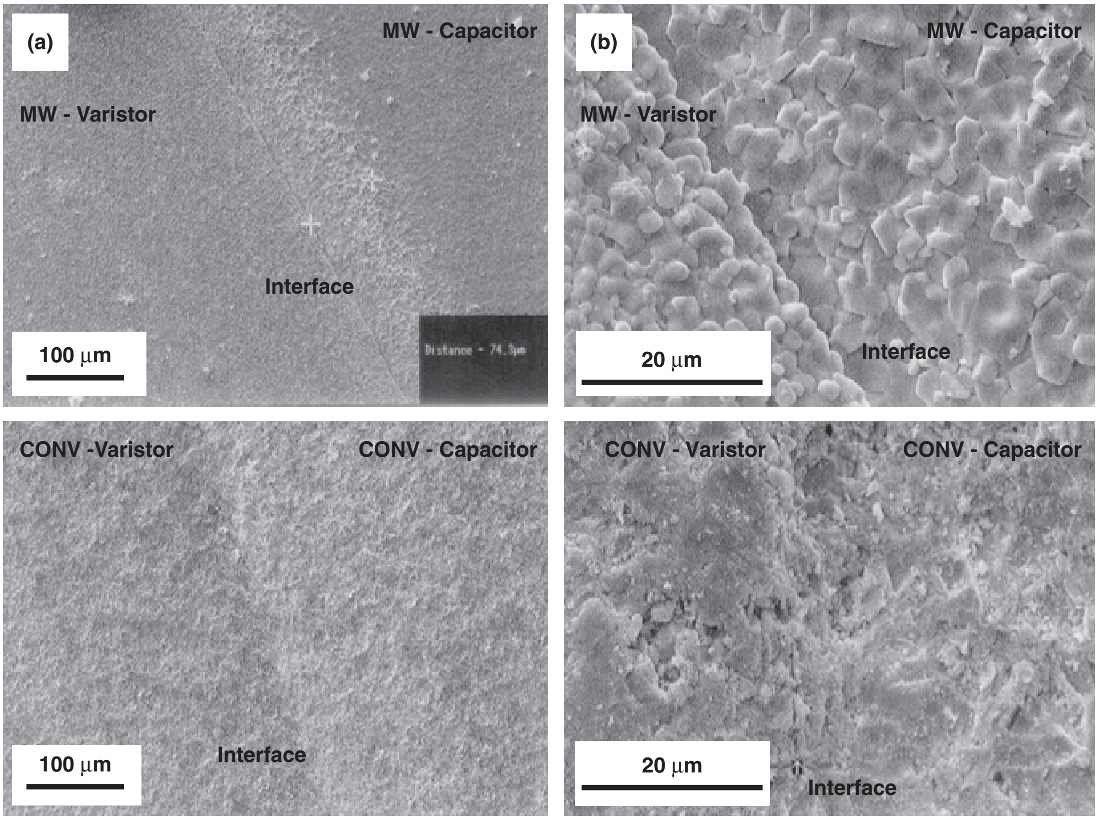

Fig. 6. Comparison of the interfaces in the microwave (1050°C/20 min) and conventionally (1060°C/4 h) sintered integrated passive device samples at (a) low and (b) high magnification. The interface is well defined in the microwave-sintered sample.

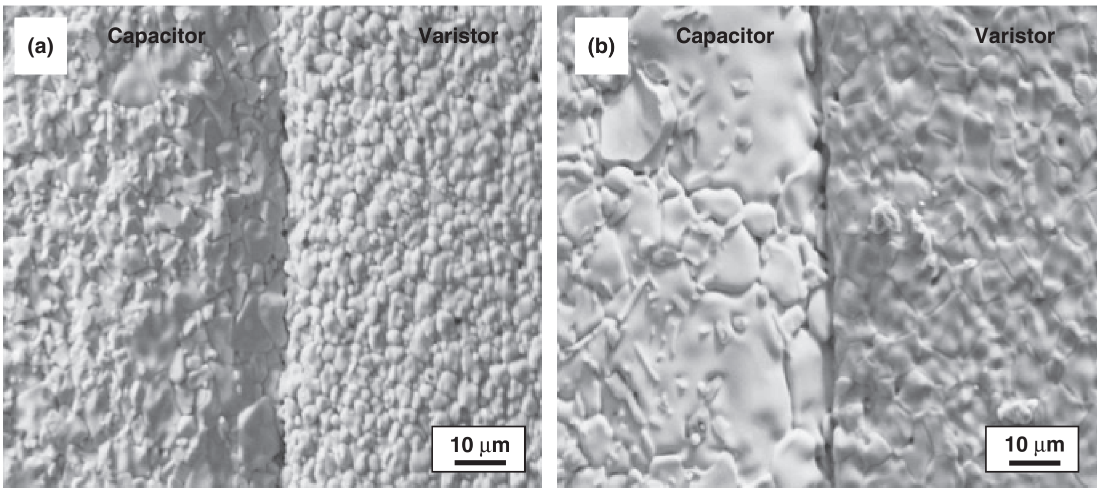

Fig. 7. Field-emission gun scanning electron microscopic images of the interface of integrated passive device (IPD) samples: (a) Microwave-sintered and (b) conventionally sintered IPD samples; left-capacitor and right-varistor. The finer grain structure of the microwave-processed sample in both regions is evident.

Table II. Composition Analysis of Microwave and Conventionally Sintered Integrated Passive Devices (IPD) Samples

	Microwave-sintered IPD (at.%)		Conventionally sintered IPD (at.%)	
Elements	Varistor	Capacitor	Varistor	Capacitor
Zn	98.8	0.6	99.2	9.8
Bi	1.2	59.6	0.4	49.6
Nb	0.0	31.6	0.3	31.4
Mg	0.0	8.1	0.0	9.1

noticed. The latter is a common problem observed in multilayer ceramic devices due to nonuniform sintering. The inset provides the high-magnification image of the varistor material and exhibits clear grain–grain boundary contrast. Figure 5 shows SEM micrographs of the as-prepared, microwave-sintered (1050°C/20 min), and control IPD sample (industrially sintered) surfaces for comparison. The average grain size, estimated using the linear intercept method, was found to be 4.0 μm on the varistor side and 7.4 μm on the capacitor side for the microwave-processed IPDs with no residual porosity observed in either region. In contrast, the microstructure of the conventionally processed IPD sample was not well defined and a clear resolution between

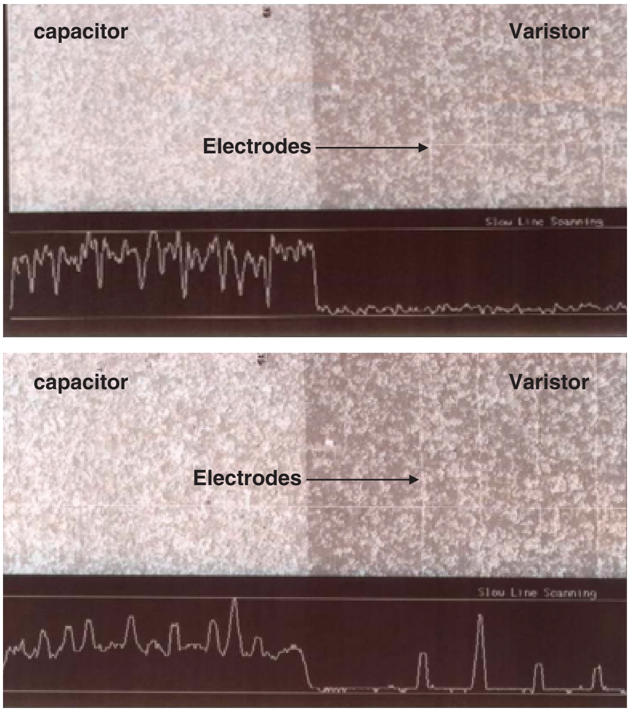

Fig. 8. Compositional line analysis of Bi (top) and Nb (bottom) on an IPD sample microwave sintered at 1050°C for 45 min.

Fig. 9. Density distribution of the microwave-processed integrated passive device samples (1050°C/30 min) as a function of position inside the cavity, when using better casket design+airflow (see text).

the grains and grain boundaries could not be achieved even after extensive polishing. Because of the presence of internal electrodes, the fractured surfaces were not looked at. The clearer structure observed in the microwave-sintered samples might be due to the rapid heating and cooling cycles associated with this approach, which can lead to a situation somewhat similar to "thermal etching."[27] This is further evidenced by the fact that the rounded grain boundary edges of the microwave-processed samples are clearly visible even in the optical micrographs at high magnification, Fig. 4 inset.

A comparison of the interfaces in the as-prepared microwave (1050°C/20 min) and conventionally sintered (1060°C/4 h) IPDs is illustrated in Fig. 6, at low and high magnifications. It is evident that the varistor/capacitor interface is very well defined in the microwave-processed sample while it is more difficult to detect in the conventionally processed sample, suggesting greater interdiffusion. In order to determine the grain sizes of the capacitor and varistor regions of the conventionally sintered sample (which was difficult to elucidate in the as-prepared devices), the polished surfaces of the sintered IPDs were chemically etched using 0.5% hydrogen fluride solution for 10 s. The resultant microstructures examined using a high-resolution FEG-SEM are shown in Figs. 7(a) and (b). The average grain sizes of the varistor and capacitor regions for the conventionally sintered samples were estimated as 7.4 and 10.5 μm, respectively. The finer grain structure in both regions of the microwave-sintered product is evident.

As IPDs are combination devices, one of the most important concerns is the undesirable interdiffusion of the matrix components at the interface during high-temperature, long-duration conventional processing procedures.[11] The average elemental compositions of the different regions of the sintered IPD samples is provided in Table II. It can be observed that no detectable diffusion of Nb into the varistor region was observed in the microwave-processed samples while the level of diffusion of Zn into the capacitor region was significantly reduced. One of the important constituents of both the varistor and capacitor ceramics used was Bi_2O_3, which is very volatile at high temperatures. It may be observed that the rapidity of the microwave process also enabled more of the Bi-based phases to be retained in both regions of the device compared with conventional processing.[28] Similar results have been reported during the microwave processing of volatile PbO- and phosphate-based compositions.[16,22] It is interesting to observe that when the microwave sintering was extended to 30 and 45 min, no significant increase in Nb interdiffusion was observed. Figure 8 shows the results for Bi and Nb compositional line analysis, respectively performed on IPDs microwave sintered at 1050°C for 45 min. The interface between the varistor and capacitor regions is very clearly defined, as are the location of the electrodes. Note that the Nb energy lines overlap with that of the electrode material, and hence sharp peaks are seen at the electrode positions, confirming the well-preserved nature of the electrodes. In contrast, it has been observed that in the case of conventionally cofired composite multilayer ceramic capacitors (MLCCs), there was an appreciable amount of electrode material migration into the ceramic layers.[7,13,29,30] In this context, microwave sintering ap-

Table III. Electrical Characteristics of Microwave-Sintered Integrated Passive Devices (IPDs)

Sintering conditions	Average V_{nom} (V)	Clamp ratio	Leakage current (μA)	Variance (V)
1050°C/20 min	39.6	1.39	0.923	8.26
1050°C/30 min	45.2	1.29	0.927	6.60
1050°C/45 min	38.5	1.34	0.925	6.93
1050°C/30 min with better insulation and air flow (see text)	43.7	1.35	0.930	3.93
Values required for current commercial product	>22	<1.5	>0.8	~ 3.5

pears to be very attractive because it not only minimizes the interdiffusion of ceramic components but also can be used to reduce electrode migration.[18,30]

The finer grain structure and more defined interfaces obtained in the microwave-sintered products were expected to be advantageous for the electrical performance. The dielectric constant ($\varepsilon_r = 182$) and the dielectric loss ($\tan \delta = 0.0005$) of the capacitor region of the microwave-sintered (at 1050°C/30 min) IPDs were found to be matching well with that of the conventionally sintered MgO-doped bismuth-niobate-based capacitors.[1] Table III shows the average varistor-related electrical characteristics of representative IPD samples microwave sintered under various experimental conditions. The important varistor-based electrical parameters viz., average values of V_{nom}, clamp ratio (C_r), insulation resistance (leakage), and variance are tabulated[¶] based on the performance of 100 samples for each set of conditions. As can be seen, with the exception of Variance, high V_{nom} (almost double) and acceptable values of C_r, leakage current, were obtained for the microwave-sintered IPDs due to the finer grain size and high densities achieved. This correlates well with the literature reports where almost twice the breakdown voltage was also measured for microwave-processed single-component and multilayer varistors compared with conventionally processed samples.[21,23,25] Extending the soaking time at temperature gradually improved the variance due to the slightly higher densities achieved; however, at longer durations the V_{nom} slightly dropped owing to larger grain sizes in the resulted device components. Nevertheless, the variance still remained high. This value, which is the standard deviation of the nominal voltage, is affected by parameters such as the temperature distribution within and across the samples and the thermal stresses stored inside the sample, which in turn affect the density distribution when a large number of samples are considered. Taking into account the very small size of the individual IPDs, the uniformity (or the lack of it) of the temperature over the whole liner was considered to be an important contributor to the large variance values. Hence, a higher quality and thicker insulation (casket) arrangement were developed and a gentle flow of air was blown across the samples intermittently to ensure thermal homogenization. Use of secondary susceptors would also have been beneficial in this respect. As a result, the density distribution of the microwave-sintered IPDs as a function of their position inside the cavity, Fig. 9, was found to be remarkably uniform and the electrical properties showed a clear improvement in their variance values, bringing it closer to what was considered to be acceptable, Table III. Additional work based on the use of a genuine hybrid microwave/conventional sintering cavity[14,21,25] has improved the variance during the processing of multilayer electroceramic devices further (this method also eliminated the need for "casketing") and showed that there is a clear potential for scaling up the process.

IV. Conclusions

Microwave sintering of multiplayer IPDs has been performed. It was found that the IPDs could be sintered to the required theoretical density within just 3 h of total cycle time. This is <1/10th of that required by existing conventional approaches. The microwave technique also completely eliminates the need for a separate binder burnt-out stage. Microstructural investigations revealed that microwave sintering resulted in a finer grain structure, preservation of electrodes, minimization of component interdiffusion and a reduction in the loss of volatile Bi-based phases. With the initial exception of variance, the electrical properties of the microwave-sintered samples were found to be better than the conventionally sintered commercial parts; a modification to the experimental system allowed the variance to become almost acceptable. In general, the simplicity, rapidity, and better product performance associated with the microwave technique make it an attractive alternative sintering methodology for IPDs.

[¶]Nominal voltage (V_{nom}): This is the voltage measured corresponding to 1 mA current ($V_{1\,mA}$) through the varistor.
Clamp ratio (C_r): This parameter gives the clamping ability of the varistor and is expressed by $C_r = V_c/V_{1\,mA}$ where V_c is the voltage measured for a given peak current I_c.
Leakage: It is the maximum voltage (or corresponding current) at which the varistor can be operated without getting significantly heated. Beyond this limiting current the joule heating (I^2R) of the ceramic becomes dominant and leads to degradation.
Variance: It is the square root of the standard deviation of the V_{nom} values.

Acknowledgments

The authors thank Littelfuse Ireland Limited for research support.

References

[1]R. Raghavendra, P. Bellew, and N. Mcloughlin, "Coprocessing of Electroceramics Technology: A Simple IPD for Combined Transient and EMI Suppression Applications," *Passive Comp. Indus.*, **1**, 13–34 (2002).

[2]M. Matsuoka, "Nonohmic Properties of Zinc Oxide Ceramics," *Jpn. J. Appl. Phys.*, **10** [6] 736–46 (1971).

[3]D. R. Clarke, "Varistor Ceramics," *J. Am. Ceram. Soc.*, **82** [3] 485–502 (1999).

[4]T. P. Gupta, "Application of Zinc Oxide Varistors," *J. Am. Ceram. Soc.*, **73** [7] 1817–40 (1990).

[5]P. R. Emtage, "The Physics of Zinc Oxide Varistors," *J. Appl. Phys.*, **48** [10] 4372–84 (1977).

[6]L. Levinson and H. Phillip, "Zinc Oxide Varistors—A Review," *Am. Ceram. Soc. Bull.*, **65** [4] 639–47 (1986).

[7]F. J. Toal, J. P. Dougherty, and C. A. Randall, "Processing and Electrical Characterisation of a Varistor-Capacitor Cofired Multilayer Device," *J. Am. Ceram. Soc.*, **81** [9] 2372–80 (1998).

[8]H. Okinaka and T. Hata, "Varistor, Thermistor Manufacturing in Japan," *Am. Ceram. Soc. Bull.*, **75** [2] 62–6 (1995).

[9]P. Ward, "Multilayer Ceramic Capacitors Materials, Processes and Reliability"; pp. 49–66 in *Electronic Ceramics*, Edited by B. C. H. Steele. Elsevier Science Publishers Ltd., Essex, U.K., 1991.

[10]H. C. Ling, M. F. Yan, and W. W. Rhodes, "Monolithic Device with Dual Capacitor and Varistor Functions," *J. Am. Ceram. Soc.*, **72** [7] 1274–6 (1989).

[11]C. W. Nies, "Compatibility Studies of Zinc Oxide Varistor Materials with Integrated Ceramic Packaging Technology"; Ph.D. Thesis in Ceramic Science, The Pennsylvania State University, PA, 1989.

[12]T. R. Shrout, D. K. Agrawal, and B. Vaidhyanathan, "Microwave Assisted Sintering of Multi-Layer Capacitors with Base Metal Electrodes"; U.S. Patent No.: 6,610,241, 2003.

[13]Y. Fang, M. T. Lanagan, D. K. Agrawal, G. Y. Yang, C. A. Randall, R. R. Shrout, A. Henderson, M. Randall, and A. Tajuddin, "An Investigation Demonstrating the Feasibility of Microwave Sintering of Base-Metal Electrode Multilayer Capacitors," *J. Electroceram.*, **15**, 13–9 (2005).

[14]J. G. P. Binner and B. Vaidhyanathan, "Microwave Sintering of Ceramics: What Does it Offer," *Key Eng. Mater.*, **264–268**, 725–9 (2004).

[15]W. H. Sutton, "Microwave Processing of Ceramic Materials," *Am. Ceram. Soc. Bull.*, **68** [2] 376–8 (1989).

[16]B. Vaidhyanathan, D. K. Agrawal, and R. Roy, "Microwave Assisted Synthesis and Sintering of NZP Compounds," *J. Am. Ceram. Soc.*, **87** [5] 834–9 (2004).

[17]S. Gedevanishvili, B. Vaidhyanathan, D. K. Agrawal, and R. Roy, "Microwave Processing using Highly Absorbing Powdered Material Layers"; U.S. Patent No.: US6512216, 2003.

[18]D. Agrawal, R. Raghavendra, and B. Vaidhyanathan, "Production of Passive Devices"; U.S. Patent No: 6,399,012, 2002.

[19]N. A. Hassine, J. G. P. Binner, and T. E. Cross, "Synthesis of Refractory Metal Carbides Via Microwave Carbothermal Reduction," *Int. J. Refract. Metals Hard Mater.*, **13** [6] 353–8 (1995).

[20]B. Vaidhyanathan and J. G. P. Binner, "Microwave Assisted Synthesis of Nanocrystalline YAG"; pp. 273–6 in *Proc. of the 9th International conference on Microwave and High Frequency Heating*, Edited by J. G. P. Binner. Loughborough University, Loughborough, U.K., 2003.

[21]B. Vaidhyanathan, K. Annapoorani, J. G. P. Binner, and R. Raghavendra, "Microwave Assisted Large Scale Sintering of Multilayer Electroceramic Devices"; pp 11–8 in *Ceramics Engineering and Science Proceedings*, Vol. 30[8], Edited by D. Singh, and J. Salem. The American Ceramics Society, Westerville, OH, 2009.

[22]B. Vaidhyanathan, A. P. Singh, D. K. Agrawal, R. Roy, and C. Ganguly, "Microwave Effects During PZT Synthesis: Enhanced Kinetics and Changed Mechanisms," *J. Am. Ceram. Soc.*, **84** [6] 1197–203 (2001).

[23]G. McMahon, A. Pant, R. Sood, A. Ahmad, and R. T. Holt, "Microwave Sintering Technology for the Production of Metal Oxide Varistors"; pp 237–42 in *Materials Research Society Symposium Proceedings*, Vol. 189, Edited by W. B. Snyder Jr., W. H. Sutton, M. F. Iskander, and D. L. Johnson. Materials Research Society, Pittsburgh, PA, 1990.

[24]A. Birnboim, D. Gershon, J. Calame, A. Birman, Y. Carmel, J. Rodgers, B. Levush, Y. V. Bykov, A. G. Eremeev, V. V. Holoptsev, V. E. Semenov, D. Dadon, L. P. Martin, M. Rosen, R. Hutcheon, and D. Abe, "Comparative Study of Microwave Sintering of Zinc Oxide at 2.45, 30 and 83 GHz," *J. Am. Ceram. Soc.*, **81** [6] 1493–501 (1998).

[25]J. G. P. Binner and B. Vaidhyanathan, "When Should Microwaves be used to Process Technical Ceramics," *Mater. Sci. Forum*, **606**, 51–9 (2009).

[26]H. Saita, Y. Fang, A. Nakao, D. Agrawal, M. T. Lanagan, T. R. Shrout, and C. Randall, "Microwave Sintering of NiCuZn Ferrite Ceramics and Devices," *Jpn. J. Appl. Phys.*, **41** [1] 86–92 (2002).

[27]M. A. N. Nogueira, W. B. Ferraz, and A. C. S. Sabioni, "Diffusion of the ^{65}Zn Radiotracer in ZnO Polycrystalline Ceramics," *Mater. Res.*, **6** [2] 167–71 (2003).

[28]R. Subasri, M. Asha, K. Hembram, G. V. N. Rao, and T. N. Rao, "Microwave Sintering of Doped Nano Crystalline ZnO an Characterization for Varistor Applications," *Mater. Chem. Phys.*, **115** [2–3] 677–84 (2009).

[29]R. Zuo, L. Li, Y. Tang, and Z. Gui, "Mutual Diffusion at Heterogeneous Interfaces in Co-Fired Composite MLCCs," *Br. Ceram. Trans.*, **100** [1] 38–43 (2001).

[30]C. Y. Fang, C. A. Randall, M. T. Lanagan, and D. K. Agrawal, "Microwave Processing of Electroceramic Materials and Devices," *J. Electroceram.*, **22**, 125–30 (2009). □