

# Preparation and electrical properties of multilayer ZnO varistors with water-based tape casting

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Received 16 October 2007; received in revised form 5 November 2007; accepted 3 January 2008

Available online 8 April 2008

## Abstract

Multilayer ZnO varistors were prepared by water-based tape casting with water-soluble acrylic as binders. Zeta potentials of the doped ZnO suspensions as a function of pH with and without dispersant were measured. Viscosity measurements were used to find the optimum dispersant concentration needed to prepare a stable slurry. Viscosity properties of the tape casting slurry were investigated. The results showed that aqueous acrylic binders have shear thinning properties suitable for tape casting of ceramic powders. Scanning electron microscopy (SEM) studies revealed that the green sheets have a smooth defect-free surface and that the multilayer varistor (MLV) ceramics prepared by water-based tape casting have a fine grain microstructure with a uniform grain size and dopant distribution. The multilayer ZnO varistors prepared by water-based tape casting display comparable good electrical properties to those prepared by solvent-based tape casting. This is believed to be attributed to the well dispersed water-based slurry, which makes more uniform dopant distribution throughout the multilayer ZnO varistors. Therefore, water-based tape casting is suitable for the manufacture of high performance multilayer ZnO varistors.

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**Keywords:** ZnO; Varistors; Tape casting; Electrical properties

## 1. Introduction

In 1947 Howatt et al. [1] described tape casting for the fabrication of thin ceramic sheets for capacitor applications. Tape casting has been widely used for the production of multilayer ceramic capacitors (MLCCs), varistors, inductors, resistors, and ceramic substrates since then [2]. Tape casting involves the dispersion of a ceramic powder in a liquid vehicle (slip), which typically contains binders, dispersants, plasticizer, defoamer and surfactant to achieve certain rheological characteristics [2,3]. In general, the slip (slurry) system can be divided into water- or solvent-based system. A water-based (aqueous) tape casting has advantages such as non-toxicity and low cost, but has disadvantages such as difficult to control drying condition, casting composition or film thickness (smaller tolerance to minor change) [2–8]. On the other hand,

a solvent-based (non-aqueous) tape casting has advantages such as faster drying rate, lower crack sensitivity, but has disadvantages such as health and environmental hazards and high cost. The water-based systems have steadily increased in industry at the expense of their solvent-based counterparts, due mainly to safety and economic considerations [4]. Polyvinyl alcohol (PVA) and polyacrylates are usually used for the water-based system, while polyvinyl butyral (PVB) is only used for the solvent-based system.

ZnO varistors have been used widely to protect electrical circuits due to their excellent non-linearity and their very large surge-withstanding capabilities [9,10]. In 1981 Shohata et al. [11] developed a ZnO-based multilayer chip varistor by applying the same manufacturing process as a multilayer ceramic chip capacitor. A multilayer chip ZnO varistor is a device designed for low breakdown voltage and high surge absorption. It can be applicable as low-voltage surge protection devices in surface mount hybrid integrated circuit technology. MLV's monolithic multilayer construction provides protection from voltage transients caused by electrostatic discharge (ESD), lightning, etc. With the rapid development of surface

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mount technology, the miniaturization of circuits and protection against ESD are increasingly becoming important. High performance multilayer ZnO varistors are necessary to meet these demands [12].

It is well known that the properties of varistors result from grain boundary phenomena and the uniformity of dopant distribution is an important factor that influences the figures of merit of ZnO varistors. The non-linear current–voltage behavior of ZnO varistors is determined by the physical and chemical characteristics of the ceramic microstructure of varistors, which in turn is strongly affected by the nature of starting powder. An ideal varistor has a crystalline microstructure of uniform grain size, shape and composition as well as minimal presence of mechanical defects such as voids, porosity and cracks [13].

Traditionally, multilayer ZnO varistors are made with solvent-based tape casting [11,12,14–19]. To the best of our knowledge, there are no publications on the preparation of multilayer ZnO varistors with water-based tape casting using water-soluble acrylic as binders, though only a few papers on the aqueous ZnO suspensions for tape casting with aqueous emulsion as binders have been published [20–23]. According to the literatures [3,8,24], the water-soluble binder system (WB4101, Polymer Innovations, Vista, CA) can offer advantages over other aqueous systems, and also outperforms typical solvent-based systems like PVB and acrylics. The system is a high molecular weight acrylic polymer with copolymerized active groups. The molecular weight, acrylic monomer composition and number of active groups have been optimized for tape casting, and can be used in both pure aqueous and aqueous/solvent hybrid systems.

An ideal slurry for water-based tape casting has well dispersed, stable and high solids loading (concentrated suspension) and shear thinning rheological behavior. In general, suspensions can be dispersed by electrostatic, steric, or electrosteric stabilization mechanisms. Electrostatic stabilization is accomplished by generating a common surface charge on the particles. Steric stabilization, on the other hand, is achieved by adsorption of polymeric additives that serve to form protective colloids. Electrosteric stabilization requires the presence of adsorbed polymer or polyelectrolyte [25]. The aqueous suspensions of zinc oxide could not be stabilized electrostatically because of the high solubility of zinc oxide at high and low values of pH, but they could be stabilized electrosterically with polyacrylic acid [21].

In this study, the doped zinc oxide powder was chemically synthesized by the oxalate precursor method [12,13]. The purpose of this study was to evaluate the feasibility of water-based tape casting using water-soluble acrylic as binders for the preparation of high performance multilayer ZnO varistors.

## 2. Experimental procedure

### 2.1. Materials

The doped ZnO powder (96 mol% ZnO and 4 mol% dopants including Bi<sub>2</sub>O<sub>3</sub>, Sb<sub>2</sub>O<sub>3</sub>, Co<sub>3</sub>O<sub>4</sub>, Mn<sub>3</sub>O<sub>4</sub> and Cr<sub>2</sub>O<sub>3</sub>)

used in this study was chemically synthesized by an oxalate precursor method (Pharmacie Centrale de France (PCF), France). The average particle size of the powder is about 1.2  $\mu\text{m}$  (Mastersizer 2000, Malvern Instruments Ltd., UK). Deionized water and ammonium polyacrylate (Ceramo D-134, Dai-ichi Kogyo Seiyaku Co., Japan) were used as a solvent and dispersant, respectively. Ceramo D-134 dissociates in water to produce negatively charged polyions and ammonium counter ions. The binder (WB4101) used in tape casting slurries was a proprietary acrylic formulation binder solution. According to the supplier's data [8,24], it contains acrylic resin (28 wt%), plasticizer (7.0 wt%), and defoamer (0.25 wt%) in water. The polyether polyol (PL005, Polymer Innovations, Vista, CA) was used as a plasticizer. To improve the wetting behavior of the slurry during tape casting, an S465 (Air Products and Chemicals, Allentown, PA) wetting agent was also added to the doped ZnO slurry. Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O was added to the doped ZnO slurry to achieve a better non-linear property.

### 2.2. Zeta potential and viscosity measurements

0.01 vol% doped ZnO aqueous suspensions with and without 0.5 wt% Ceramo D-134 were prepared for the analysis. 0.01 M HCl and 0.01 M NaOH were used as the titrants to adjust the suspension pH accordingly. The zeta potential of slurries was measured by a zeta potential analyzer (ZetaPALS, Brookhaven Instruments Co., USA).

The doped ZnO suspensions were prepared with solids loadings at 60 wt% with different amounts of dispersant for a dispersant optimization study. The viscosity was then measured at a single shear rate of 100 s<sup>−1</sup> using a concentric cylinder rheometer (Physica MCR300 Modular Rheometer, Anton Paar GmbH, Austria).

The doped ZnO powder, deionized water, Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O and ammonium polyacrylate dispersant were first mixed in a polyethylene jar with ZrO<sub>2</sub> balls for 12 h. The optimum contents of binder, plasticizer and defoamer were then added and the slurry was mixed for another 6 h. An S465 wetting agent was finally added to the slurry and ball milled for 1 h. Viscosity measurement was conducted at room temperature at shear rates between 0 and 100 s<sup>−1</sup> using a concentric cylinder rheometer (Physica MCR300 Modular Rheometer, Anton Paar GmbH, Austria). Doped ZnO suspensions were prepared at solids loadings of 60 wt%.

### 2.3. Tape casting

According to the basic requirements for tape casting of ceramic powders [25,26], the optimum content of organic additives, including dispersant, binder and plasticizer, were determined, which is listed in Table 1.

Before casting, the slurries were slowly rolled at a rotational speed of 10 rpm for 24 h to remove entrapped air in the milling step. Then the slurries were filtered through a 200 mesh nylon sieve to remove agglomerates [2]. Tape casting was carried out using a tape caster (CAM-C1, KEKO Equipment, Slovenia)

Table 1  
Composition of doped ZnO aqueous tape casting slurry

Material	Function	Content (wt%)
Doped ZnO	Ceramic powder	60.00
Deionized water	Solvent	23.30
D-134	Dispersant	0.50
WB4101	Binder & plasticizer	15.00
DF001	Defoamer	0.20
S465	Surfactant	0.50
PL005	Plasticizer	0.50

with a stationary casting head at a casting rate of  $2.3 \text{ cm s}^{-1}$ , a gap height of  $120 \text{ }\mu\text{m}$  and a silicone-coated Mylar carrier film. The tapes were dried in the drying zone of the tape caster at an air flow temperature of  $70 \text{ }^\circ\text{C}$ . The final green sheets were  $25 \text{ }\mu\text{m}$  in thickness.

#### 2.4. MLV fabrication

On the top surface of a green sheet, Ag–Pd inner electrodes were printed by screen printing. The green sheets and electrodes were stacked alternately. The stacked green bar was laminated and pressed into a bar of  $1.1 \text{ mm}$  thick and cut into pieces with a dimension of  $2.2 \text{ mm} \times 1.1 \text{ mm}$ . Finally, the fabricated multilayer varistors (MLVs) have a same MLCC geometric structure. The organic binders were burnt out at  $350 \text{ }^\circ\text{C}$  with a slow heating rate in air. The green chips were sintered at  $980 \text{ }^\circ\text{C}$  for  $1.5 \text{ h}$  in air. MLVs were composed of five active layers and other dummy layers sandwiching them. Ag outer electrodes were attached to the end of the sintered chips. Chip style 1608 ( $1.6 \text{ mm} \times 0.8 \text{ mm}$ ) MLVs were fabricated.

#### 2.5. Microstructure of green tapes and sintered MLV ceramics

The microstructure of green tapes and MLV fracture surface and element distribution were investigated using a scanning electron microscopy (JSM-6301F, JEOL, Japan) with an energy dispersive analysis of X-rays (EDAX).

#### 2.6. MLV electrical testing

Current–voltage and  $8/20 \text{ }\mu\text{s}$  surge retention characteristics of MLVs were examined with an  $I$ – $V$  meter and a pulse generator incorporating an oscilloscope. Breakdown voltage ( $V_B$ ) was determined on the  $I$ – $V$  curves as the voltage at a current of  $1 \text{ mA}$ . Leakage current ( $I_L$ ) was read at a working voltage of  $0.8 V_B$ , and  $\alpha$  was determined by taking the ratio of  $\log[I_2/I_1]$  to  $\log[V_2/V_1]$ , where  $V_1$  and  $V_2$  are the voltages at  $I_1$  ( $0.1 \text{ mA}$ ) and  $I_2$  ( $1.0 \text{ mA}$ ) on the  $I$ – $V$  curve, respectively [12]. Clamping voltage ( $V_C$ ) was measured with a  $1 \text{ A}$  peak  $8/20 \text{ }\mu\text{s}$  current impulse. Peak current ( $I_P$ ) was defined as the maximum peak  $8/20 \text{ }\mu\text{s}$  current impulse applicable to MLV without causing device failure, where the change of  $V_B$  was less than  $10\%$  after the current impulse test.

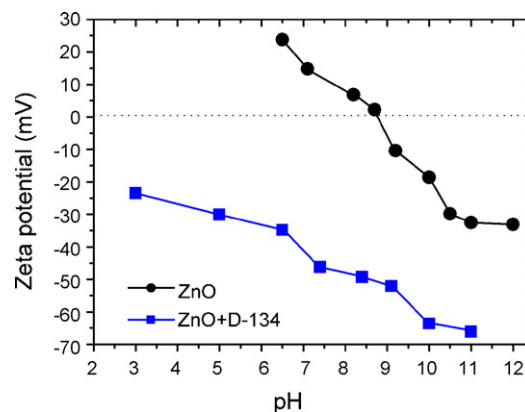


Fig. 1. Zeta potential of  $0.01 \text{ vol\%}$  doped ZnO suspensions as a function of pH with and without D-134.

### 3. Results and discussion

#### 3.1. Colloidal processing of doped ZnO suspensions

The value of  $\zeta$ -potential is a valuable guide to the stability of colloidal suspensions. High zeta potentials usually mean more stable suspensions, although this is only a rough correlation [7]. As a working rule, a zeta potential of at least  $30 \text{ mV}$  is necessary for long-term dispersion stability [27]. Fig. 1 shows the zeta potential of a  $0.01 \text{ vol\%}$  doped ZnO suspension as a function of pH, where the isoelectric point (IEP) of doped ZnO without D-134 is  $9.0$ . The isoelectric point of zinc oxide found in the literature ranges from  $8.7$  to  $10.3$  [7,20–22]. Fig. 1 also indicates that D-134 can change the IEP of doped ZnO particles. The absorption of a small amount of D-134 always makes the charge on the doped ZnO surfaces negative between pH  $3$  and  $11$ . Since D-134 dissociates in water to produce negatively charged polyions, such as  $\text{RCOO}^-$  and positively charged counter ions, in this case  $\text{NH}_4^+$  ions. Electrosteric stabilization by charged polymer molecules adsorbed on to the particle surfaces forms an important method of stabilizing particles in a suspension. For pH values up to IEP, the doped ZnO particle surfaces are positively charged, leading to attraction between the particles and the negatively charged polymers and hence to adsorption of the charged polymers onto the particle surfaces. Repulsion between the adsorbed charged polymers can then provide effective electrosteric stabilization of the suspension. Electrosteric stabilization of doped ZnO particles in water was achieved by using D-134.

Without titrants to adjust the suspension pH, the zeta potentials of the  $0.01 \text{ vol\%}$  doped ZnO suspension with and without  $0.5 \text{ wt\%}$  dispersant are  $-49.3 \text{ mV}$  (pH  $8.4$ ) and  $14.8 \text{ mV}$  (pH  $7.1$ ), respectively. A zeta potential of  $-49.3 \text{ mV}$  is high enough to assure the stability of an aqueous suspension.

#### 3.2. Rheology of tape casting slurries

Fig. 2 shows the relationship between the viscosity of doped ZnO suspension at  $100 \text{ s}^{-1}$  and additive concentration of the D-134 dispersant. At relatively low additive content below  $0.5 \text{ wt\%}$ , the viscosity decreased with increasing additive

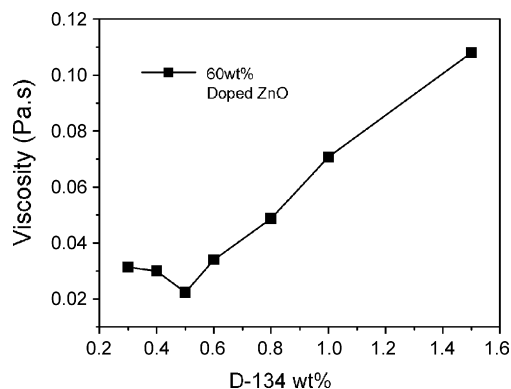


Fig. 2. Viscosity of 60 wt% doped ZnO suspension as a function of increasing D-134 amount.

content, and the optimum additive content to obtain the minimum viscosity appeared at 0.5 wt%. The excess addition of D-134 dispersant remarkably increased the viscosity of the suspension with increasing additive content. At 0.5 wt% content of the D134 dispersant, the viscosity of slurry is minimum, so the optimum dispersant concentration is 0.5 wt%. According to the literature [28], with the addition less than the optimum dispersant concentration, a poor coverage of the solid surfaces was achieved which increased the possibility of adhesion between the solid surfaces, this resulted in a high suspension viscosity. The optimum addition of polymer dispersant of 0.5 wt% generated the maximum value of electrosteric repulsion force, almost zero adhesive force and the suspension viscosity was minimized. While the excess addition of D-134 dispersant at 1.0 wt% increased the free polymers in the suspension, it reduced the steric repulsion force and generated bridging, causing the suspension viscosity to increase.

The rheological behavior of the doped ZnO slurry listed in Table 1 exhibited a shear thinning type flow behavior (Fig. 3). Shear thinning flow behavior of a suspension usually indicates a weakly flocculated system [5,29]. However, this type of flow behavior is desirable for tape casting since low shear rates render a high viscosity of the slurry at rest, which helps to prevent flocculation and sedimentation of particles during drying. On the other hand, at high shear rates the slurry possesses a low viscosity to flow smoothly during tape casting.

Fig. 3 shows the shear stress versus shear rate curves for the full doped ZnO slurry (Table 1). The shear stress of the slurry

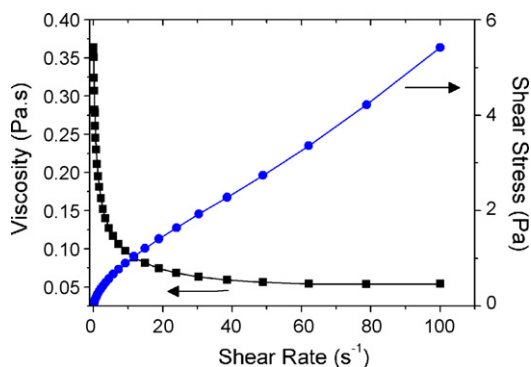


Fig. 3. Shear thinning behavior of the final tape casting slurry.

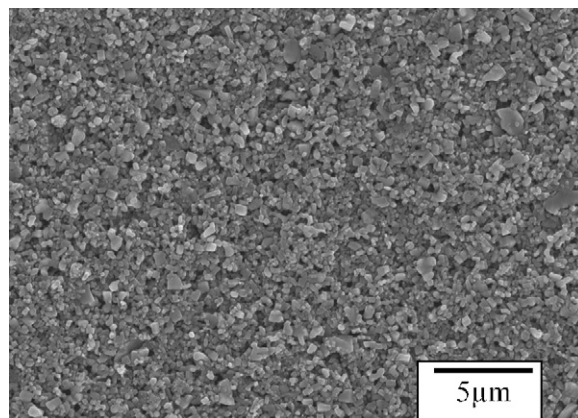


Fig. 4. SEM image of the top surface of green tapes made by water-based tape casting.

shows an almost identical increase and decrease with shear rate with little or no hysteresis. This indicates that the flow behavior of the slurry may not be time dependent or thixotropic [5].

### 3.3. Microstructure analysis

Fig. 4 shows the typical microstructure of water-based tape casting green tapes. The tapes have a smooth defect-free surface, which is believed to be attributed to the well dispersed and stable slurry with a shear thinning rheological behavior.

The microstructure of aqueous tape casting prepared MLV ceramics is shown in Fig. 5 and the ceramics have a uniform fine grain microstructure, which is a result of good quality green sheets with a smooth defect-free surface. Ag–Pd inner electrodes can be seen clearly in Figs. 5 and 6(a, b). Elemental mapping shown in Fig. 6 c and d indicates that Zn and Sb are uniformly distributed throughout the MLV ceramics prepared by water-based tape casting.

### 3.4. Electrical properties

Table 2 shows  $V_B$ ,  $I_L$ ,  $\alpha$ ,  $V_C$ ,  $I_P$  of MLVs made by water-based tape casting and solvent-based tape casting. The water-based tape casting prepared MLVs give almost the same  $V_B$ ,  $I_L$

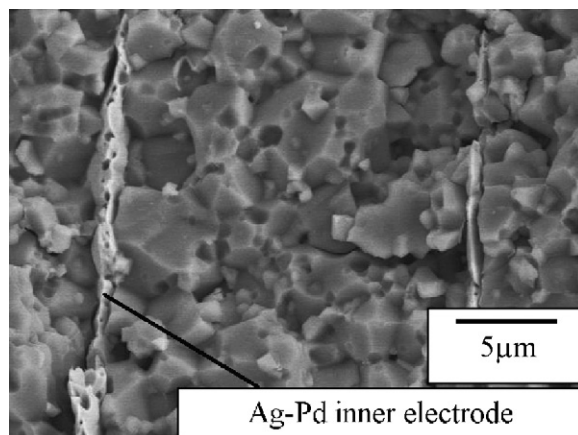


Fig. 5. SEM microstructure of the water-based tape casting prepared MLV fracture surface.



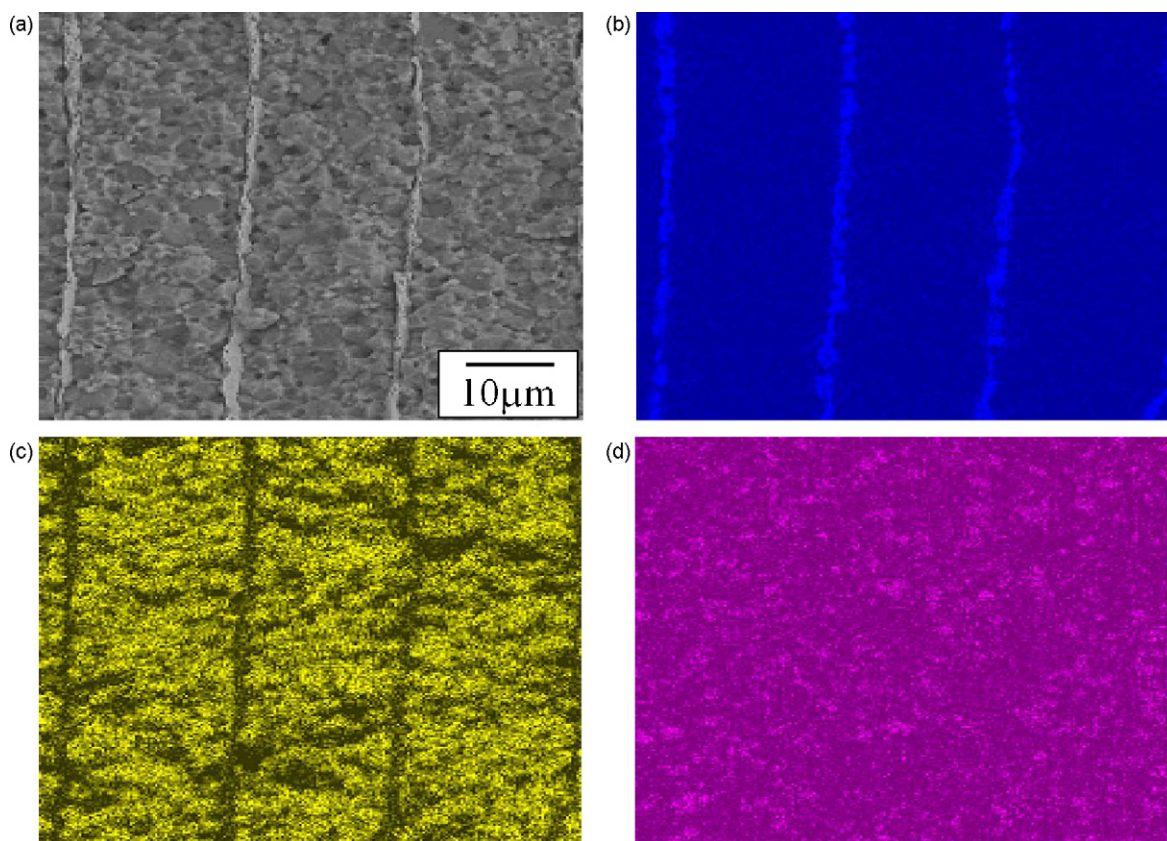


Fig. 6. (a) SEM microstructure and (b–d) Pd, Zn, Sb distribution map in the water-based tape casting prepared MLV ceramics by EDAX.

and  $I_P$  as the solvent-based tape casting. But the former gives better electrical properties, such as  $\alpha$ ,  $V_C$ , than the latter. It is well known that physical properties of ZnO varistor materials depend strongly on the microstructure, including grain size and boundary characteristics. The chemical homogeneity of specific dopants and ceramic microstructure play an important role in determining the electrical properties of varistors. The superior electrical properties of water-based tape casting prepared MLVs are attributed to the well dispersed water-based slurry, which results in a more uniform dopant distribution throughout MLVs. The leakage current strongly depends on the distribution of additives, and a more uniform distribution improves the electrical non-linearity. Scanning surface potential microscopy (SSPM) images revealed that the grain boundary resistance of a uniformly distributed sample is higher than that of a poorly distributed sample [30]. The better non-linear properties (higher  $\alpha$ ) and lower  $V_C$  have been attributed to the presence of uniform potential barriers in entire microstructure and  $Al^{3+}$  doping. SEM and EDAX analyses show the water-based tape casting prepared MLVs have a

uniform microstructure and good uniformity in the dopant distribution, so they have a high  $I_P$ . The low  $I_L$  may be related to the high grain boundary resistance caused by the presence of a high Schottky barrier at the grain boundary regions as the result of a more uniform dopant distribution throughout MLVs.

#### 4. Conclusions

Well dispersed and stable slurry with shear thinning rheological behavior could be obtained by D-134 dispersant and WB4101 binder. Doped ZnO suspensions were stabilized electrosterically by adsorption of dissociated dispersant molecules on ceramic particles. The isoelectric point of doped ZnO was found to be around pH 9.0 without D-134. Viscosity measurements were used to find the optimum dispersant concentration, which corresponded to 0.5 wt% D-134. The rheological behavior of the slurry proved to be shear thinning. SEM shows the water-based tape casting green sheets have a smooth defect-free surface and MLV ceramics have a fine grain microstructure with a homogeneous dopant distribution. The MLVs made by water-based tape casting exhibit the same good electrical properties as those made by the solvent-based tape casting but the former has a lower production cost than that made by the latter.

We believe that these superior electrical properties are a direct result of the well dispersed and stable slurry with a shear thinning rheological behavior, which makes dopants distribute more uniformly throughout MLVs. Although water-based tape

Table 2  
Electrical characteristics of MLVs (style 1608) made with different slurry system type

Slurry system type	$V_B$ (V/mm)	$V_{1\text{ mA}}$ (V)	$I_L$ ( $\mu\text{A}$ )	$\alpha$	$V_C$ (V)	$I_P$ (A)
Aqueous	520	9.1	0.1	28	16	35
Solvent	514	9.0	0.1	25	19	35

casting faces some challenges such as slow drying rate, dewetting and foaming compared to solvent-based tape casting, it provides good green and sintered ceramic properties for MLV applications. The water-based tape casting is suitable for the manufacture of high performance multilayer ZnO varistors.

## Acknowledgement

Financial support for this research work was gratefully received from the open foundation of the State Key Lab of New Ceramics and Fine Processing, Tsinghua University, China, project no. KF 0503.

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