Characteristics of ionic liquid-based electrolytes for chip type aluminum electrolytic capacitors

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Abstract

Since ionic liquids (ILs) possess several attractive properties, including chemical and thermal stability, nonflammability, high ionic conductivity, and negligible vapor pressure, a new electrolyte system based on ILs has been proposed for chip type aluminum electrolytic capacitors. Four ILs based on imidazolium/pyrrolidinium cations and maleate/phthalate anions have been synthesized and their thermal stabilities have been examined. The 25 wt.% solutions of the four ILs in gamma-butyrolactone (GBL) solvent were prepared as electrolytes of chip type aluminum electrolytic capacitors. The conductivity, sparking voltage and thermal stability of these electrolytes have been systematically investigated. The results revealed that the four IL-based electrolytes exhibited high conductivity. Furthermore, the conductivity of maleate anion-based electrolytes is higher than that of phthalate anion-based electrolytes, whereas the high-temperature durability of phthalate anion-based electrolytes in conductivity is superior to that of maleate anion-based electrolytes irrespective of pyrrolidinium or imidazolium cation. However, this type of electrolytes could only be utilized in low-voltage type capacitors due to their comparatively low sparking voltage. In addition, the capacitors utilizing the four IL-based electrolytes show excellent thermal stabilities during a reflow soldering process.
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1. Introduction

Recently, the use of chip type electronic components has been rapidly increasing as electronic equipment requires reduction in size and weight and the surface mounting technology progresses forward. In general, the surface mounting of chip type electronic components on a printed circuit board is implemented by a reflow soldering process. This soldering technology requires electronic components to withstand a very high soldering temperature, for example, a peak temperature of 230 °C for a short time. Further, the soldering temperature will be expected to become higher while lead-free solder is utilized to meet the demand of environmental protection. This is a great challenge for chip type aluminum electrolytic capacitors that contain the electrolyte solutions. The electrolyte solutions for conventional aluminum electrolytic capacitors are generally composed of ammonium salt and ethylene glycol (EG)/H$_2$O mixed solvent,

which have a relatively low boiling point (commonly less than 160 °C). If this type of electrolyte solutions were employed in chip type aluminum electrolytic capacitors, the performance of the capacitors would be markedly deteriorated due to the decomposition of ammonium salt in the electrolyte solutions at the high soldering temperatures. Moreover, the high vapor pressure of the electrolyte would create a high internal pressure in the capacitors in this high temperature environment. In this case, the capacitors would result in a failure due to expansion of the capacitor can or even explosion after a reflow soldering process. Therefore, it is necessary to search for a new electrolyte solution system consisting of an electrolyte salt of high thermal stability and a solvent of higher boiling point to resist the high reflow soldering temperature.

Ionic liquids (ILs), which consist entirely of ionic species, are finding wide use as electrolytes for electrochemical applications [1–4]. ILs possess several attractive properties, including chemical and thermal stability, nonflammability, high ionic conductivity, and negligible vapor pressure [5,6]. These unique properties of ILs correspond exactly with the requirements of

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chip type aluminum electrolytic capacitors. Accordingly, it is expected that ILs could be promising candidates for electrolyte materials serving as the chip type capacitors.

ILs usually consist of inorganic or organic anions and nitrogen-containing organic cations such as alkylammonium [7,8], alkylphosphonium [9], *N,N′*-dialkylimidazolium [1,10], *N,N*-dialkylpyrrolidinium [11] and *N*-alkylpyridinium [12], etc. However, it has been reported that the use of alkylammonium or alkylphosphonium electrolyte in electrolytic capacitors could cause some issues of liquid leakage, i.e., the leakage of the electrolyte solution from the location of the capacitor cathode lead wire [13,14]. Moreover, these electrolytes have some disadvantages (e.g., poor conductivity [5], thermal stability, and solubility) compared with the *N,N′*-dialkylimidazolium and *N,N*-dialkylpyrrolidinium salts of a heterocyclic ring. So, in this paper, we chose *N,N′*-dialkylimidazolium and *N,N*-dialkylpyrrolidinium as organic cations of ILs developed for chip type capacitors. On the other hand, an electrolyte for aluminum electrolytic capacitors cannot contain a number of "aggressive" anions such as halide ions because of their damage to anodic oxide films [15]. Since the most commonly used electrolyte salts for aluminum electrolytic capacitors are ammonium salts containing carboxylate anions, we chose maleate and phthalate anions to constitute target ILs for chip type capacitors. Thus, in this paper, we have synthesized four typical ILs which are *N,N′*-dimethylimidazolium maleate ($DMIm^+M^-$), *N,N′*-dimethylimidazolium phthalate ($DMIm^+P^-$), *N,N*-dimethylpyrrolidinium maleate ($DMPr^+M^-$) and *N,N*-dimethylpyrrolidinium phthalate ($DMPr^+P^-$), respectively.

For a conventional aluminum electrolytic capacitor with high performance, the electrolyte should possess such properties as (i) high ionic conductivity, (ii) a wide operation temperature range (including high thermal stability), and (iii) high ability towards self-restoration (healing) of the aluminum oxide film. Besides the resistance to the high reflow soldering temperature mentioned above, the electrolyte for chip type capacitors should also possess these properties. Because the addition of organic solvents into the ILs can enhance the ionic conductivity and operation temperature range of the electrolyte system, the ILs are commonly used in the form of solutions. Here, we report a new electrolyte system based on these ILs as electrolyte for chip type aluminum electrolytic capacitors. The new electrolyte system is solutions of these ILs in gamma-butyrolactone (GBL). Since GBL, a very common solvent in the field of aluminum electrolytic capacitors [16,17], is an aprotic polar solvent, which has a sufficiently high dielectric constant (39.1 at 25 °C), a relatively low viscosity (1.75 mPa s at 25 °C), a low fusion point (−45 °C) and a high boiling point (204 °C), these IL-based electrolytes consisting of ILs and GBL can exhibit a relatively high conductivity and a wide operation temperature range. Owing to a very high boiling point of GBL and negligible vapor pressure of the ILs, the IL-based electrolyte solutions would have a high boiling point or a low vapor pressure compared with the conventional ammonium salt/EG electrolytes. Thus, it is possible for the IL-based electrolyte solutions to inhibit the deformation of the capacitor can or failure of the capacitor at high reflow soldering temperatures.

The concentrated GBL solutions of these ILs can be prepared easily due to their good miscibility with solvents, but maximum conductivities in these solutions have been found to be at about 30 wt.% concentration. Therefore, on account of the electrolyte cost, we attempted to prepare 25 wt.% solutions of the four ILs in GBL as electrolytes for chip type aluminum electrolytic capacitors. In this paper, we report results from our investigation into physicochemical properties of the ILs and the IL-based electrolyte solutions. As mentioned above, ionic conductivity is one of the most important electrolyte characteristics determining electrolytic capacitor performance, in this work we have focused on this topic. We report the results of changes in conductivity of the electrolytes at an elevated temperature and reflow soldering test for the capacitors impregnated with these electrolytes, which were used to evaluate the long-term and short-term thermal stabilities of the IL-based electrolytes, respectively [17].

2. Experimental

2.1. Synthesis of ionic liquids

The imidazolium and pyrrolidinium halides were synthesized according to standard procedures described in literatures [10,18]. For example, *N,N′*-dimethylimidazolium halide was synthesized from the reaction of 1-methylimidazole (Fluka) with methyl iodide (Sigma) in trichloroethane. Then silver oxide (silver hydroxide) freshly prepared from the reaction of ammonia water with silver nitrate was added to an aqueous solution of imidazolium/pyrrolidinium halides. The mixture was stirred continuously for a half hour or so. Then the precipitated silver halides was filtered off and the filtrate of imidazolium/pyrrolidinium hydroxides were obtained. The target imidazolium/pyrrolidinium salts were prepared by neutralization of imidazolium/pyrrolidinium hydroxides and maleic/phthalic acid. Finally, the aqueous solutions obtained were concentrated at 70 °C under 100 mbar of pressure and the residual liquids were dried in vacuo at 150 °C to constant weight. All chemicals used in the synthesis, not otherwise specified, were analytical purity and used without further purification, which were purchased from Nanjing Chemical Reagent Company (China). Solutions were prepared using deionized water. The structure of the ILs synthesized ((a) imidazolium, (b) pyrrolidinium) is shown below, where X^- is either maleate or phthalate anion.

(a)　　　　(b)
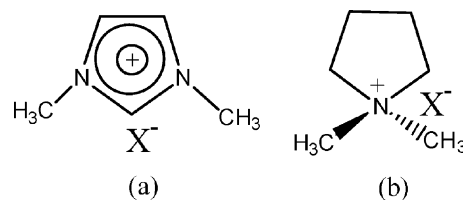
2.2. Preparation of IL-based electrolytes

The IL-based electrolytes were prepared by simply dissolving the ILs synthesized above in GBL (Aldrich). The contents of ILs in GBL were all 25 wt.% in this work. All electrolyte

preparation and work was performed in a glove box under a nitrogen atmosphere.

2.3. Measurements

The thermal stabilities of ILs in air and under nitrogen were determined on a Shimadzu TGA-50 at a heating rate of $20\,°C\,min^{-1}$. Conductivity of electrolytes was measured with a conductivity meter (CM-20S, TOA electronics Ltd.) by using a pair of Pt-black electrodes. Variable temperature data were obtained in a MINI-SUBZERO environmental chamber (MC-810, TABAI ESPEC Co.)

To test the stability in conductivities of the IL-based electrolytes, an appropriate amount of electrolyte was sealed in a glass tube and then was stored in an oven at $125\,°C$. After regular intervals, parts of the electrolytes were taken out from the tube and the conductivity of the samples measured at $30\,°C$.

In order to measure the sparking voltage of various IL-based electrolytes, the anodic oxidation behavior of aluminum was examined in these electrolytes. Two aluminum foils (>99.99% purity) were placed into the suitable volume of electrolytes. A constant current density of $10\,mA\,cm^{-2}$ was applied and the cell voltage between anode and cathode was recorded until sparking occurred at the anode surface. The voltage–time curves were recorded automatically by a computer system described in our previous paper [19]. Sparking voltage values of various electrolytes were obtained using the appearance of voltage oscillation in the voltage–time curves as criterion for identifying the sparking voltage.

Reflow soldering test on laboratory capacitors made from the IL-based electrolytes were carried out under the following conditions: preheat temperature is about $150\,°C$ within $180\,s$, peak temperature is less than $240\,°C$ within $5\,s$ and the time for more than $200\,°C$ is $30\,s$. The values of capacitance and dissipation factor of the capacitors were measured at a frequency of $100\,Hz$, and the value of leakage current was obtained at the loading of $16\,V$ for $60\,s$. The capacitance, dissipation factor and leakage current of the capacitors were measured by means of a LCR meter (hp-4284A), and a leakage current tester (TH2685B), respectively.

3. Results and discussion

3.1. Thermal properties of ILs

The thermal stabilities of $DMIm^+M^-$, $DMIm^+P^-$, $DMPr^+M^-$ and $DMPr^+P^-$ in air and under nitrogen were examined. The thermal gravimetric analysis (TGA) indicated the four ILs all exhibit great thermal stability both in air and under nitrogen. Figs. 1 and 2 show the TGA thermograph of $DMIm^+M^-$ and $DMPr^+M^-$, respectively. The initial mass loss below $100\,°C$ is mainly due to the release of moisture of the ILs having a strong hydrophilic nature. The obvious weight loss between $100\,°C$ and about $200\,°C$ of $DMIm^+M^-$ in air shown in Fig. 1 may be ascribed to the reaction of oxygen with $DMIm^+M^-$. However, the true thermal decomposition of $DMIm^+M^-$ occurs at above $200\,°C$ corresponding to the
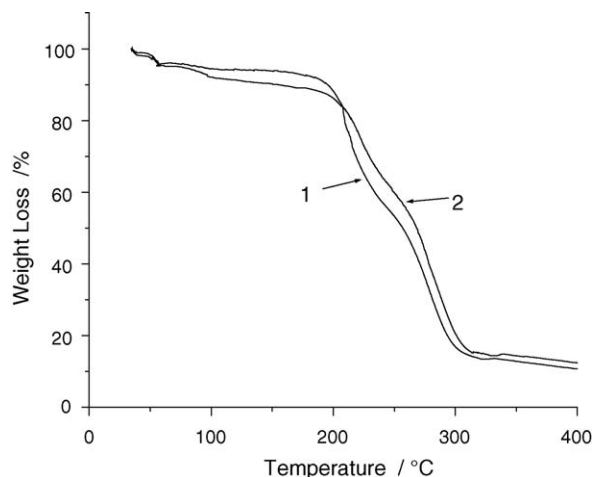

Fig. 1. TGA curves for $DMIm^+M^-$ (1, under nitrogen; 2, in air).

maximum rate of weight loss both in air and under nitrogen. On the other hand, $DMPr^+M^-$ exhibits a single decomposition step but a higher thermal stability than that of the $DMIm^+M^-$ (Fig. 2). Figs. 1 and 2 show their thermal decomposition rates are unlike. The former is slower than the latter, which illustrates their different decomposition mode. In addition, they exhibit similar thermal stability both in air and under nitrogen. It is worth noting that they seem to have a higher thermal decomposition temperature in air, which is highly advantageous in view of practical condition of capacitor manufacturing. The thermal behaviors of $DMIm^+P^-$ and $DMPr^+P^-$ in air and under nitrogen are very similar to that of $DMIm^+M^-$ and $DMPr^+M^-$, respectively. The thermal properties of the four ILs are summarized in Table 1. In a word, the thermal stabilities of the four ILs shown in TGA are adequate for their application in chip type capacitor.

3.2. Properties of IL-based electrolytes

3.2.1. Conductivity

The specific conductivity for the four IL-based electrolytes at $30\,°C$ and $-40\,°C$ are listed in Table 2. The conductiv-

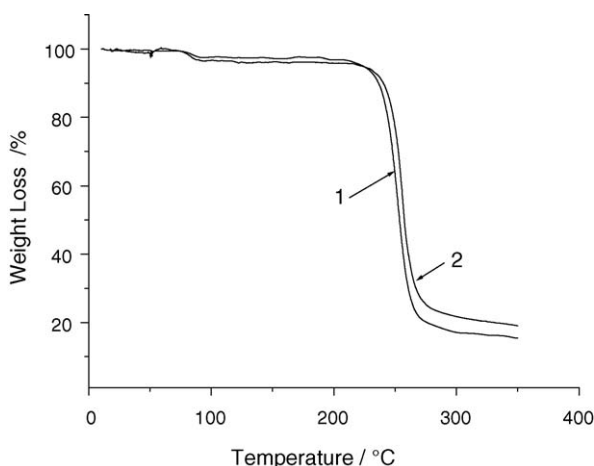

Fig. 2. TGA curves for $DMPr^+M^-$ (1, under nitrogen; 2, in air).

Table 1
TGA data for the four ILs

ILs	T_{ini}[a] (°C)		T_{max}[b] (°C)	
	In air	Under N_2	In air	Under N_2
DMIm+M−	198	199	272	267
DMIm+P−	202	201	270	265
DMPr+M−	225	226	255	251
DMPr+P−	227	225	254	251

[a] Initial temperature of thermal decomposition.
[b] Temperature where the thermal decomposition rate is maximum.

ity of pyrrolidinium cation-based electrolytes is slightly higher than that of imidazolium cation-based electrolytes. This difference in conductivity between the two types of electrolytes is likely related to different molecular configuration between pyrrolidinium and imidazolium cations. The imidazolium ring exhibits a planar configuration, while the pyrrolidinium cation being fully saturated and the alkyl substituents on the tetrahedral nitrogen project above and below the plane of the ring [5,20].

On the other hand, the conductivity of maleate anion-based electrolytes is higher than that of phthalate anion-based electrolytes, which is mainly due to a difference in the anion size (M− < P−) [21]. Generally speaking, conductivity of electrolytes is increased by decreasing the ion size [1].

Fig. 3 gives the dependence of specific conductivity on temperature for the DMIm+P− and DMPr+M−-based electrolytes. Similar trends for the other two IL-based electrolytes also exist (not shown in Fig. 3). Linear dependences in Fig. 3 suggest that the ionic conductive behavior in the electrolytes is of the Arrhenius type:

$$\sigma = \sigma_0 \exp\left(-\frac{E_a}{RT}\right) \qquad (1)$$

where σ_0 is the pre-exponential factor, E_a is the activation energy for ionic conduction and R is the gas constant. The conductivity activation energies (E_a) estimated from the slope of the fitted lines in Fig. 3 are summarized in Table 2. Excellent fit of the conductivity data to the above Arrhenius equation is observed ($r > 0.999$). In conclusion, the temperature dependences of conductivity for the four IL-based electrolytes are analogous on account of their similar E_a values (Table 2). In

Table 2
Conductivity data and activation energy (E_a) from Arrhenius fits of various electrolytes

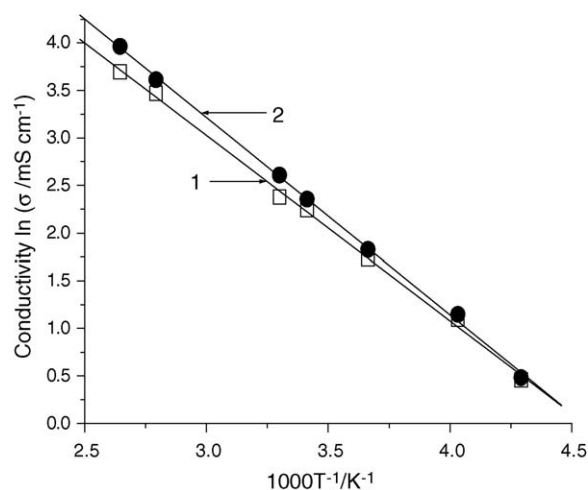| Electrolytes	Conductivity σ (mS cm^{-1})		Activation energy E_a (KJ mol^{-1})
	30 °C	−40 °C	
DMIm+M−/GBL	12.05	1.56	17.1
DMIm+P−/GBL	10.76	1.58	16.2
DMPr+M−/GBL	13.58	1.62	17.2
DMPr+P−/GBL	11.35	1.59	16.5
Ammonium adipate/EG/H$_2$O	10.85	0.152	–

Fig. 3. Arrhenius plot of specific conductivity (ln σ vs. 1/T) for the electrolyte: (1) DMIm+P−/GBL; (2) DMPr+M−/GBL.

addition, their conductivities could be measured down to −60 °C where solidification had not yet occurred. Comparatively high conductivity (>1.56 mS cm^{-1}) is found at a lower temperature (−40 °C) for them. For comparison, Table 2 also lists conductivity values at 30 and −40 °C for a solution of ammonium adipate (8 wt.%) in hybrid solvents of EG and water, which is a conventional electrolyte solution for low-voltage type aluminum capacitors.

3.2.2. Sparking voltage

In general, the cell voltage of aluminum rises approximately linearly with time from the commencement of anodizing until dielectric breakdown under galvanostatic anodizing conditions [22]. Fig. 4 shows the typical anodic oxidation behavior of aluminum in the IL-based electrolytes at room temperature during constant-current anodization. According to experimental results of anodic oxidation of aluminum, it can be found that the values of sparking voltage for the four IL-based electrolytes are not high, because the voltage values are all less than 90 V when the
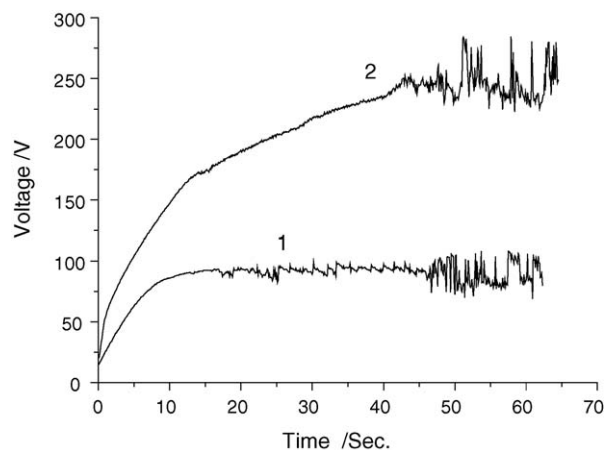

Fig. 4. Experimental records of galvanostatic aluminum anodization in various electrolytes: (1) DMIm+M−/GBL electrolyte; (2) ammonium adipate/EG/H$_2$O electrolyte.

Table 3
Data of sparking voltage for the four IL-based electrolytes and their corresponding resistivity at 30 °C

Electrolytes	DMIm$^+$M$^-$/GBL	DMIm$^+$P$^-$/GBL	DMPr$^+$M$^-$/GBL	DMPr$^+$P$^-$/GBL
Resistivity (S^{-1} cm)	83.0	92.9	73.6	88.1
Sparking voltage (V)	86	88	83	87

cell voltage appears oscillation. Consequently these IL-based electrolytes can only be used in low-voltage type aluminum electrolytic capacitors. Fortunately, the low-voltage type capacitors are of leading products for chip type aluminum electrolytic capacitors.

Ikonopisov [23] proposed a theoretical model of breakdown caused by an avalanche of electrons injections at the electrolyte/oxide interface. According to this model, the relationship of the sparking voltage with the electrolyte resistivity is represented by the following equation:

$$V_s = a_1 + b_1 \log \rho \qquad (2)$$

Albella et al. [24] have extended the Ikonopisov model to explain the details of the breakdown phenomenon. They reported evidence that electrolyte species incorporated into oxides act as a source of avalanching electrons. This assumption has yielded another equation about dependence of sparking voltage on electrolyte concentration:

$$V_s = a_2 - b_2 \log C \qquad (3)$$

In above equations, V_s, ρ and C are sparking voltage, electrolyte resistivity and electrolyte concentration, respectively, and a_1, b_1, a_2, and b_2 are constant values related to the electrolyte system. Table 3 presents data of sparking voltage for the four IL-based electrolytes and their corresponding resistivity (reciprocal of conductivity) at 30 °C. It should be noted that the constants a_1 and b_1 in Eq. (2) are different for the four IL-based electrolytes of various electrolyte species. From Table 3, it is found that the magnitude of sparking voltage for the four IL-based electrolytes is accordant to that of their resistivity. The larger the resistivity of the electrolyte, the higher is its sparking voltage. Thus, it can be supposed that the constants a_1 and b_1 in Eq. (2) are approximately identical for the four IL-based electrolytes, which indicate the influence of these electrolytes on the breakdown process of anodic alumina is similar. In addition, lower values of sparking voltage for these electrolytes are also ascribed to higher electrolyte concentration (25 wt.%), as seen in Eq. (3). As a comparison, the curve of sparking voltage measurement for ammonium adipate (8 wt.%) in EG/H$_2$O is also presented in Fig. 4, which shows a higher sparking voltage value (about 240 V). Understanding the reason of the lower sparking voltage values for the IL-based electrolytes will require future study.

3.2.3. Thermal stabilities

Fig. 5 shows the change in conductivities of the four IL-based electrolytes at an elevated temperature. It is clear that the deterioration behavior is principally dependent on the type of
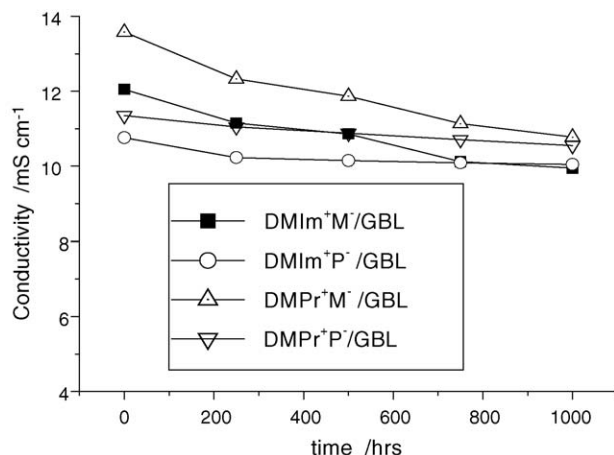

Fig. 5. Conductivity change of the four IL-based electrolytes at 125 °C.

carboxylate anion. Phthalate anion-based electrolytes are more stable in conductivity than maleate anion-based electrolytes irrespective of pyrrolidinium or imidazolium cation. This trend is consistent with the results reported by Ue et al. [17] in the case of quaternary ammonium carboxylate/GBL electrolytes. By means of gas and liquid chromatography, they had shown that the conductivity decrease in maleate anion-based electrolytes was caused by the decrease of ionogens.

In order to assess the thermal stability of the four IL-based electrolytes for chip type capacitors during a reflow soldering process, some capacitors impregnated with these electrolytes were prepared and reflow soldering tests were performed. Table 4 shows the results of reflow soldering test for the capacitors. The heat-resistant performance for the four IL-based electrolytes were all excellent during a reflow soldering process.

Table 4
Results of reflow soldering test for the four IL-base electrolytes

Electrolytes	Reflow soldering	C (μF)	tan δ	I (μA)
DMIm$^+$M$^-$/GBL	Before	451	0.113	4.8
	After	442	0.106	4.6
	Change percent (%)	−2.0	−6.2	−4.2
DMIm$^+$P$^-$/GBL	Before	448	0.124	4.9
	After	438	0.111	4.9
	Change percent (%)	−2.2	−10.5	0
DMPr$^+$M$^-$/GBL	Before	453	0.104	4.6
	After	442	0.092	4.7
	Change percent (%)	−2.4	−11.5	2.2
DMPr$^+$P$^-$/GBL	Before	452	0.116	4.8
	After	441	0.108	4.7
	Change percent (%)	−2.4	−6.9	−2.1

The capacitance of the capacitors are all decreased after reflow soldering test, but the capacitance change percent are all less than 2.5%. The change of dissipation factor and leakage current for the capacitors is not remarkable, and the dissipation factor values of the capacitors are even lower after reflow soldering test (Table 4). In addition, the deformation for the appearance of the capacitors tested was not observed after reflow soldering test. In fact, these results are in agreement with that of TGA for ILs mentioned above.

4. Conclusions

A new electrolyte system based on ILs has been proposed for chip type aluminum electrolytic capacitors. In the present work, four ILs based on imidazolium/pyrrolidinium cations and maleate/phthalate anions have been synthesized and the electrolytes based on the four ILs for chip type capacitors have been prepared. The temperature dependence of conductivity for the four IL-based electrolytes follows the Arrhenius equation. Compared with the conventional electrolytes for electrolytic capacitors, the ionic conductive properties for the four IL-based electrolytes at lower temperature ($-40\,^{\circ}$C) are excellent, but their sparking voltage was low (less than 90 V). So these IL-based electrolytes are only utilized in low-voltage type capacitors. In addition, the conductivity of maleate anion-based electrolytes is higher than that of phthalate anion-based electrolytes, whereas the long-term thermal stability of phthalate anion-based electrolytes in conductivity is superior to that of maleate anion-based electrolytes irrespective of pyrrolidinium or imidazolium cation. The results of reflow soldering test show that the capacitors impregnated with the four IL-based electrolytes exhibit satisfactory thermal stabilities.

Acknowledgement

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