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Abstract

Purpose – The aim of this paper is to characterize the rheological properties of the flux media exposed to different levels of solicitation and to determine its influence on the rheology of the solder paste. The data obtained experimentally are fundamental for the development of numerical models that allow the simulation of the printing process of printed circuit boards (PCB).

Design/methodology/approach – Rheological tests were performed using the Malvern rheometer Bohlin CVO. These experiments consist of the analysis of the viscosity, yield stress, thixotropy, elastic and viscous properties through oscillatory tests and the capacity to recover using a creep-recovery experiment. The results obtained from this rheological analysis are compared with the rheological properties of the solder paste F620.

Findings – The results have shown that the flux is viscoelastic in nature and shear thinning. The viscosity does not decrease with increasing solicitations, except in the case where the flow is withdrawn directly from the bottle. Even if the solder paste shows a thixotropic behavior, this is not the case of the flux, meaning that this property is given by the metal particles. Furthermore, the oscillatory tests proved that the flux presents a dominant solid-like behavior, higher than the solder paste, meaning that the cohesive/tacky behavior of the solder paste is given by the flux.

Research limitations/implications – To complement this work, printing tests are required.

Originality/value – This work demonstrates the importance of the rheological characterization of the flux in order to understand its influence in the solder paste performance during the stencil printing process.

Keywords Rheology, Flux, Solder paste, Viscosity, Thixotropy

Paper type Research paper

Nomenclature

- G' = elastic modulus (Pa);
- G'' = viscous modulus (Pa);
- J = compliance (1/Pa);
- J_c = creep compliance (1/Pa);
- J_r = recovery compliance (1/Pa);
- δ = phase angle (° "degree");
- LVR = linear viscoelastic region; and
- SMT = surface mount technology.

1. Introduction

Since 2006, lead-based solders have been replaced in the market because of environmental and legislation factors (Ren *et al.*, 2016) (Collins *et al.*, 2012). In this way, owing to its characteristics and mainly because it is a Pb-free material, the near-eutectic Sn-Ag-Cu (SAC) have been the most implemented solder alloy in electronic assemblies (Collins *et al.*, 2016). Because of the advances in the production of printed circuit boards (PCB), the gap between the solder paste melting temperature and the heat resistance of the electronic components has been narrowing. Recently, SnZn and SnBi solder alloys have demonstrated to be good candidates because of their lower melting points compared to SAC (Collins *et al.*, 2016). Fluxes play an important role to increase the performance of the solder pastes. Owing to these new requirements of the low melting point temperature, advances in solder flux design and formulation have been made to obtain more reliable Pb-free solder pastes and more active pastes (Ren *et al.*, 2016).

Solder paste is a mixture of a metal alloy powder (around 90 per cent by weight) and a resin-based flux, such as rosins (gum, tall, wood and derivatives such as polymerized, acrylated, hydrogenated, disproportionated, formylated and ester rosins) and

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resines (styrenemaleic acid, epoxy, urethane, polyester, phenoxy and terpene) (Inoue *et al.*, 2015). Other materials are added to improve the soldering process (Hwang, 1989): activators (content 0.1-20 per cent), such as halides (chlorides, bromides), that wet and clean surfaces. However high flux activity (>20 per cent) can cause reliability problems in the assembly because of the generation of more corrosive flux residues (Mallik *et al.*, 2013). Solvents, such as diethylene glycol monobutyl ether or diethylene glycol mono-*n*-hexyl ether (Ichikawa *et al.*, 2010)), must ensure not only a good solubility of the flux but also a stable viscosity at the continuous printing and a good electric insulation resistance during the melting of the solder paste. Thixotropic agents (0.5-25 per cent), such as hardened castor oil, beeswax, carnauba wax, stearic acid amide and hydroxystearic acid ethylenebisamide (Ichikawa *et al.*, 2010), are also part of the flux composition. Other additives such as antioxidants, rust-preventing agents and chelating agents can also be identified. Upon heating, the solvent evaporates and the activators attack the metal surface, promoting its cleaning. On further heating, the solder powder particles melt and form a liquid mass that makes the joint between the PCB and the electronic component.

The flux plays an important role in the solder paste as its main function is to achieve optimal solderability. In this way, several features need to be ensured by the flux during the soldering process to (Baluch and Minogue, 2007):

- make the metal surfaces active and wettable by the solder alloy through the removal of the passivation layers;
- protect cleaned surfaces, preventing contact with air prior to the application of the melted solder, using a substance layer of a specific material, usually the resin rosin;
- promote wetting of the surfaces to be joined by controlling the surface forces; and
- provide good rheology features for accurate paste printability, tack and slump.

To be commercially acceptable for SMT manufacturing, a flux must have all the characteristics mentioned above. The properties that control these requirements include chemical activity, activation temperature, thermal stability, surface tension, wetting power, rheology, print performance, toxicity and nature/amount of residue (Baluch and Minogue, 2007).

To analyze the performance of a solder paste, the rheology is fundamental as the flow and deformation affect the print quality and post-print behavior of the solder paste (Bao *et al.*, 1998). Several works were performed to correlate the flux rheology and the solder paste's performance. Bao *et al.* (1998) suggested that the yield stress and recovery are the only two properties dicted by the flux (Bao *et al.*, 1998). Dušek *et al.* (2002) mentioned that it is more appropriate to study the flux directly to determine the solder paste performance, as it is responsible for the time-dependent properties of the paste (Dušek *et al.*, 2002). Jackson *et al.* (2002) observed that the flux medium plays an important role in achieving a stable suspension, proving that the flux predominantly controls the solder paste rheology (Jackson *et al.*, 2002). Billotte *et al.* (2006) characterized the rheological properties of a specific solder paste and its flux, and they identified not only that the solder paste is shear thinning and thixotropic but also that the yield stress value of the flux is lower than the solder paste (Billotte *et al.*, 2006). Durairaj *et al.* (2008) determined that the solder paste exhibits poor recovery compared with flux media; however, the results obtained in the thixotropy and viscosity tests were not able to determine the differences regarding the rheological behavior between them (Durairaj *et al.*, 2008). Marks *et al.*(2011) observed that the changing of the flux selection during the paste manufacture leads to a decrease of the paste recovery by about five per cent, which results in an increase of the slumping behavior (Marks *et al.*, 2011). Recently, Bušek *et al.* (2016) showed that the use of increased amount of proper flux (with higher activity) in the solder paste minimizes the void occurrence, increasing the performance of the reflow soldering process (Bušek *et al.*, 2016), whereas Tu *et al.* (2016) verified that the viscosity of the solder paste is determined by the flux (Tu *et al.*, 2016).

From all these studies, the importance of the flux in solder paste performance is clear. One way to gain insight into the flux performance is by studying the rheological properties of this component. However, a study performed by Durairaj *et al.* shows that it is difficult to analyze clearly the difference between the rheological properties of the solder paste and the flux (Durairaj *et al.*, 2008). Thus, to better understand the rheological properties of the flux and their influence on the behavior of the corresponding solder paste, this study presents a rheological analysis of the flux and the results were compared with the rheological behavior of the solder paste F620.

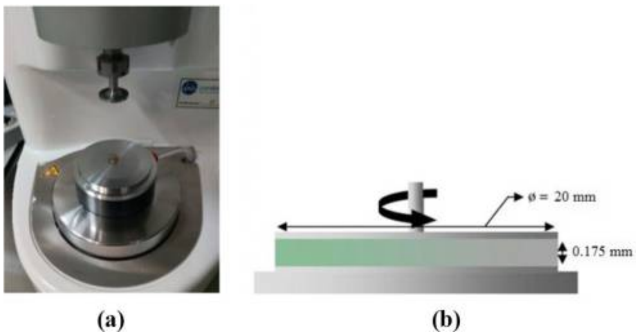
2. Experimental procedure

The sample used to perform the rheological studies is the flux media of the solder paste F620, which is lead-free and composed by SAC405 alloy, Sn95.5/Ag4/Cu0.5. This solder paste has a metal content of 88 per cent ± 0.5 per cent, a density of 4.0 g/cc ± 0.3 g/cc and a grain size ranging between 25 and 45 μm in diameter. The flux, presented in Figure 1, is a combination of solvents, thickeners, fluxing agents and blinders (Hwang, 1989). It is known that the formulation of the flux directly influences the deformation, flow characteristic and printing performance of the solder paste (Mallik, 2009). However, the exact chemical composition of the flux is unknown as the producers of flux media do not disclose this information, which makes it difficult to interpret the results obtained from the rheological tests.

The Malvern rheometer Bohlin CVO was used to perform the rheological analysis of the flux. The sample was introduced between a stainless steel plate with a 20 mm diameter and a stationary bottom plate, considering a gap of 0.175 mm, as illustrated in Figure 2. Parallel plates are propitious for sample analysis with different characteristics as the gap can vary over a

Figure 1 Flux media of the solder paste F620

Figure 2 (a) Malvern rheometer Bohlin CVO and (b) parallel plate



wide range of values. For this study, the gap was selected according to the values used in the printing process of PCB. As it is important to perform the experiments at room temperature, the flux was removed from the refrigerator 8 h before the rheological tests.

The rheological experiments were conducted considering three levels of solicitation and one without solicitation. The solicitation parameter represents the number of times that the material was exposed to external forces, allowing us to analyze the behavior of the material when it is not stationary. This property must be analyzed as in the printing process, the solder paste is not exposed to the same forces over time. At the beginning, the solder paste is removed from the container and placed inside the printer deposit to feed the squeegee. Because the squeegee is continuously in motion during the printing process, the solder paste will also be exposed to these external forces, suffering different levels of solicitation throughout the process. Level 1 corresponds to 1-2 solicitations, Level 2 represents 3-5 solicitations and Level 3 involves 5-10 solicitations. Before each rheological test, the sample was pre-sheared for a period of 30 s at 300 Pa. All the experiments were performed at room temperature (22°C) and each test was spaced by 30 s. Note that the applied conditions were selected according to the results obtained from preliminary tests.

3. Rheological behavior of flux

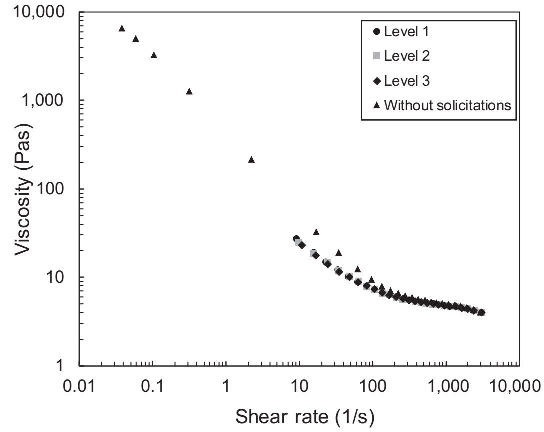
3.1 Viscosity

Viscosity is considered one of the most important rheological properties as it influences the solder paste behavior ahead of the squeegee, the filling of the apertures and its subsequent release from the apertures (Amalu *et al.*, 2011).

To study the viscosity of the flux, a shear stress ramp test was performed according to the following conditions: a pre-shear at 300 Pa was applied during 30 s, followed by a shear stress ramp from 250 to 12,000 Pa during 150 s.

The results presented in Figure 3 show that the viscosity decreases with increasing shear rate, proving that the flux is shear thinning. This is a desirable property as it ensures an effective flow of the solder paste through the stencil's apertures during the printing process (Mallik *et al.*, 2013). On the other hand, the results also show that without any solicitation, the flux presents a higher viscosity, showing the strong cohesive forces inside the flux system prior to the application of shear stress or stain. However, after the application of external forces, the internal bonds of the flux break, resulting in viscosity

Figure 3 Flux viscosity in function of the shear rate

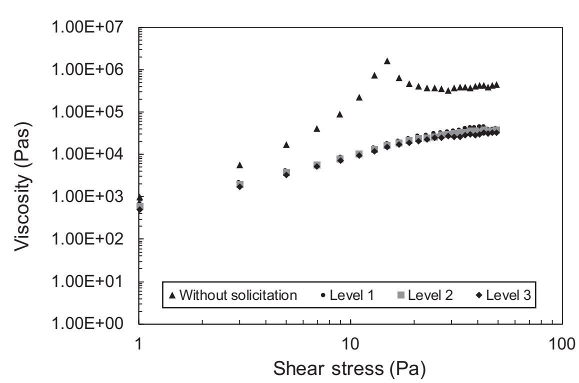


reduction. These results show that the flux cannot recover its initial properties immediately, as it can be observed through the viscosity comparison between the Level 1 and the flux without solicitation. Regarding the second and third levels of solicitations, it appears that the viscosity behavior is approximately the same compared with Level 1. In this way, the solicitations seem to have little influence on the flux's viscosity.

3.2 Yield stress

Yield stress is defined by Billotte *et al.* (2006) as the transition point between the solid-like and liquid-like behaviors of a flow (Billotte *et al.*, 2006). According to Mallik *et al.* (2010), yield stress is induced by the formation of a particle structure, which results from an agglomeration and interlocking of particles that need to achieve a specific applied stress to start to deform the material (Mallik *et al.*, 2010). To determine the yield stress, a stress ramp test was conducted according to a shear stress between 0 and 50 Pa during 150 s, considering the same pre-shearing of the viscosity test. The results in Figure 4 show that the yield stress point is approximately 15 Pa. This transition point is only observed when the flux does not suffer any solicitation. These results indicate that after the transition into the liquid-like behavior, the particle structure of the flux was completely broken. According to Bao *et al.* (1998) higher yield stress values decrease the printing defects as the material will

Figure 4 Determination of the flux yield stress



have less tendency to be smeared underneath the stencil (Bao *et al.*, 1998).

3.3 Oscillatory test

The oscillatory test aims to determine the elastic and viscous behavior of the flux through the application of a sine oscillation of shear stress at a constant frequency (that can take a value between 0.1 and 10 Hz), being subsequently measured with the shear strain rate response (Nguty *et al.*, 1999). This experiment allows the measurement of the elastic (G') and viscous (G'') moduli of the sample, which represent the mechanical energy stored and recovered and the mechanical energy dissipated as heat, respectively (Bao *et al.*, 1998). The phase angle (δ), which indicates the delay of the response of the sample, is another viscoelasticity parameter that can be measured. Through this experiment, it is also intended to identify the LVR, i.e. the region where the internal structure of the sample remains undamaged (Durairaj *et al.*, 2009), characterized by the flat region at the beginning of each test.

The experimental conditions applied in the oscillatory test were the following: a pre-shear equal to the one applied in the viscosity test, a frequency of 1 Hz and a shear stress varying between 0 Pa and 400 Pa during 5 min. These conditions were repeated for the three solicitation levels and the results are shown in Figures 5, 6 and 7. As it can be observed in the curves presented in each plot, the elastic modulus presents a higher value than the viscous modulus, showing that the flux has a dominant solid-like behavior. In solder paste printing process, a

Figure 5 Oscillatory test results for Level 1 of solicitation

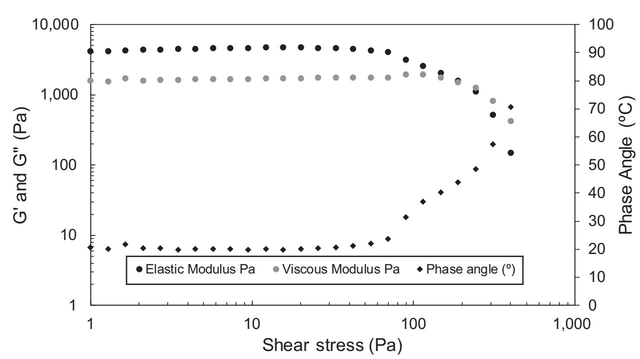


Figure 6 Oscillatory test results for Level 2 of solicitation

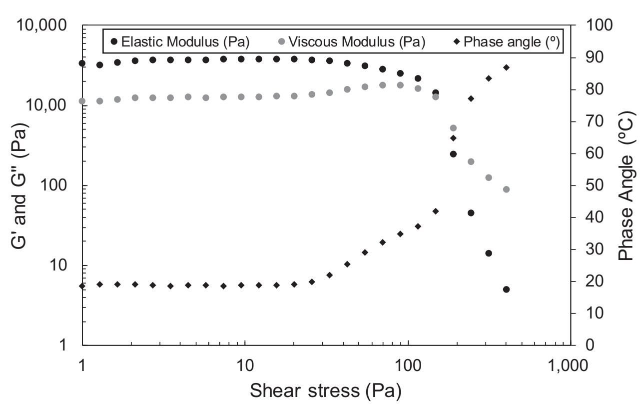
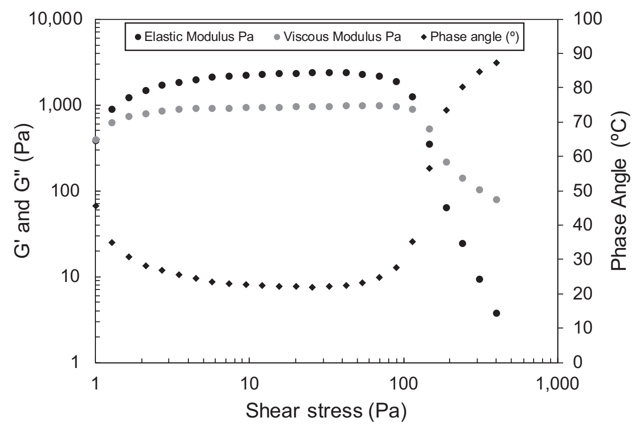


Figure 7 Oscillatory test results for Level 3 of solicitation



high G' value indicates a high resistance of the paste during the stencil separation process and slumping. On the other hand, this high value can also lead to paste hang-up on the squeegee, which in turn will require a higher squeegee speed during the printing process and a higher resistance during the stirring process (Durairaj *et al.*, 2006). Considering the LVR region, it appears that the lower-end value decreases with an increasing number of solicitations. This latter result indicates that the structural resistance of the flux tends to be deteriorated by increasing the forces applied on the sample (Mallik *et al.*, 2010). Regarding the crossover stress, $G' = G''$, which represents the transition from elastic to viscous behavior, this value is the same regardless of the level of solicitation. According to Bao *et al.* (1998), a high stress value at this point is an indication of a material with a solid-like behavior. The phase angle is another important parameter that provides information about the liquid- and solid-like behaviors of the sample. A viscoelastic material presents a phase angle between 0° and 90°, which is the case with the flux. Furthermore, a phase angle close to 0° indicates an elastic behavior, while a phase angle closer to 90° shows a viscous behavior (Mallik *et al.*, 2013). In this context, the results show that at higher shear stress values, the flux presents a viscous behavior, being intensified with the increase in the number of solicitations.

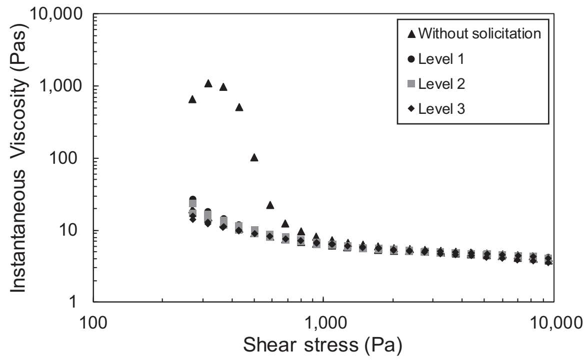
3.4 Thixotropy

According to Barnes (1997), thixotropy is defined as the decrease in a material's viscosity over time, when exposed to a constant shear rate or shear stress value, followed by a gradual recovery of the structure when the applied shear stress or rate is removed (Barnes, 1997).

Thixotropy tests were performed by shearing the sample from zero to a maximum value and down to zero, which results in a loop. The area identified inside the loop will be an indication of thixotropy. In this experiment, the loop varies between 10 and 2,000 Pa and from 2,000 to 10 Pa during 300 s (150 s for each side).

As can be seen in Figure 8, there is some recovery in the case of the flux without solicitation. Regarding the other levels of solicitations, it seems that the flux does not present a thixotropic behavior as the forward and backward curves are overlapped. This property of the flux can be confirmed by the

Figure 8 Thixotropic behavior of the flux



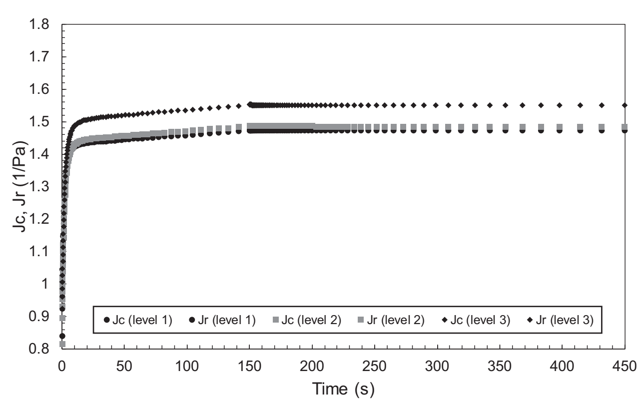
creep-recovery test. If no recovery is observed after the removal of the constant shear stress applied to the sample, this will be an evidence of a non-thixotropic behavior.

3.5 Creep-recovery test

The creep-recovery test is used to analyze the behavior of a material when exposed to constant stress and its capacity to recover after the removal of the stress, with the strain monitored as a function of time. The results obtained from this test are expressed by the compliance parameter, $\mathcal{J}$, i.e. the strain divided by the corresponding stress (Durairaj *et al.*, 2008). According to Bao *et al.* (1998), the shear stress applied corresponds to the lower stress of the viscoelastic region (Bao *et al.*, 1998). Through the oscillatory test, it was observed that the lower end of the viscoelastic region of the flux is approximately 50 Pa. In this way, the creep-recovery test was run at this shear stress value during 150 s, followed by a recovery time of 300 s. The results obtained (Figure 9) are expressed in terms of creep and recovery compliance, $\mathcal{J}_c$ and $\mathcal{J}_r$, respectively.

From the data, it is observed that $\mathcal{J}_c$ increases with the solicitation level. According to Chen *et al.* (2014) an increase of $\mathcal{J}_c$ means that the sample can be easily deformed. Regarding the analysis of the recovery capacity of the flux, Durairaj *et al.* (2008) introduced the ratio of recovery (per cent) given by $(\mathcal{J}_c\text{-}\mathcal{J}_r)/\mathcal{J}_c$. This parameter was calculated for the three levels of solicitation of the flux, showing that no recovery is observed in none of the cases. These results are in accordance with the thixotropy test as

Figure 9 Creep-recovery of the flux with a shear stress of 50 Pa



no recovery of the structure is observed after the removal of the shear stress applied to the sample, proving the non-thixotropic behavior of the flux. This test demonstrates the high viscous behavior of the flux, also identified in the oscillatory test, as the sample presents extremely low recovery and a high creep compliance.

4. Comparative analysis with solder paste

The results obtained from the rheological analysis of the flux were compared with the rheological properties of the solder paste F620 (Barbosa *et al.*, 2017). These experiments were conducted with the same conditions applied for the flux.

4.1 Viscosity

Overall, the comparison between the viscosity of the solder paste and the flux (see Figure 10) indicates that the viscosity of the flux is lower than that of the solder paste. For the same shear rate, e.g. 10 s^{-1}, the flux viscosity is approximately five times lower than the solder paste. The results are in accordance with Evans and Beddow (1987) and Durairaj *et al.* (2006), who mentioned that the viscosity tends to increase with increasing metal content and tends to decrease with an increase of the particle size distribution and temperature (Evans and Beddow, 1987). Moreover, it seems that the effect of solicitations is more pronounced in the solder paste because of the interactions between the metal particles that are more susceptible to break with the successive shear stress applied to the sample. Finally, as it can be observed in Figure 10, both samples present a shear thinning behavior, which is a desired property during the stencil printing process.

4.2 Yield stress

Regarding the results obtained in the yield stress test (Figure 11), it is observed that the transition from the solid- to liquid-like behavior occurs at 15 Pa in the case of the flux and approximately at 7 Pa for the solder paste. These results demonstrate that the flux requires higher forces to change from an elastic to a viscous behavior. In this context, it is suggested that the metal particles induce locally higher deformation of the solder paste when exposed to low shear stress. This is a disadvantage during the printing process as higher elastic properties helps not only the pastes to pull together during stencil release, reducing the probability of clogging, but also the tendency to be smeared under the stencil (Bao *et al.*, 1998). These results are in

Figure 10 Viscosity comparison between the solder paste and the flux

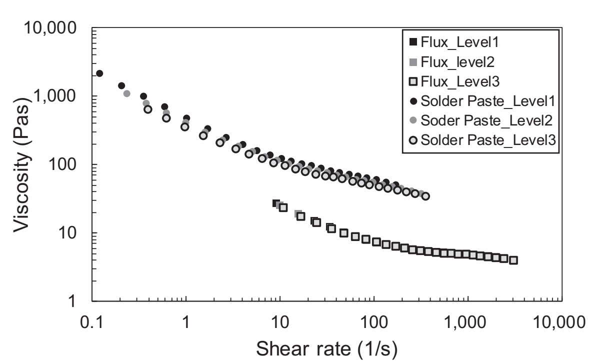
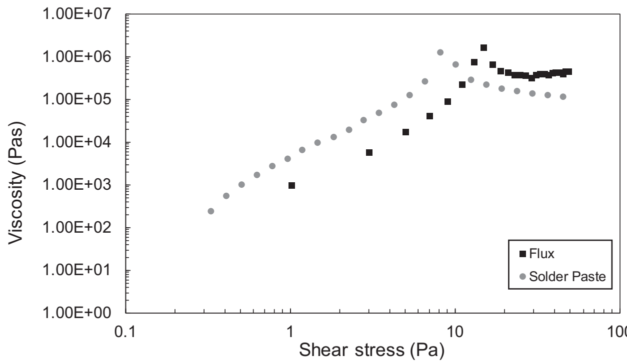


Figure 11 Yield stress comparison between the solder paste and the flux

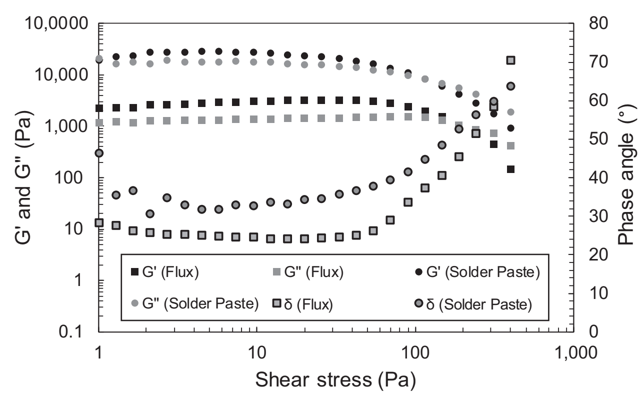


accordance with Bao *et al.* (1998) that have shown that the yield stress property is dictated by the flux.

4.3 Oscillatory test

The results presented in Figure 12 show that the elastic and viscous moduli of the flux are lower than the values obtained in the solder paste because of the absence of strength interaction that occurs between the metallic particles. On the other hand, the crossover stress $G' = G''$ occurs at a higher shear stress value in the case of the flux sample, which means that the cohesive/tacky behavior of the solder paste is given by the flux as it requires a higher shearing to change from a solid- to liquid-like behavior. According to Bao *et al.* (1998), a higher crossover stress implies a higher solid characteristic and that will help to reduce slump. On the other hand, a low crossover stress is desired to reach a high tack value that is both linked to the cohesion and adhesion of the solder paste, reducing failures and enhancing the wetting between the paste and the PCB surface. Regarding the phase angle, as expected, this parameter increases with the increase in shear stress and lies between 0° and 90° in both samples. These results confirm that the flux and solder paste are viscoelastic in nature. However, it is observed that by increasing shear stress, the phase angle of the flux reaches a value close to 70°, whereas the solder paste reaches a value close to 65°. Hence, these results indicate that by increasing shear stress, the flux presents a more pronounced

Figure 12 Viscoelastic properties of the solder paste and its flux at Level 1 of solicitation



viscous behavior than the solder paste. Moreover, since a low phase angle characterizes a tacky behaviour and a high phase angle defines a paste that could slump easily (Durairaj *et al.*, 2009), the results show that the property of a solder paste to slump easily during the printing process is given by the flux since it presents a higher phase angle.

4.4 Thixotropy

As it can be observed in Figure 13, while the flux does not present a thixotropic behavior, the solder paste is thixotropic as the forward and backward curves do not overlap. The thixotropic effect is more pronounced at low stresses, as can be seen through the plot. However, this behavior is less pronounced with the increase of the solder paste solicitation; the area inside the loops decreases over the second and third levels of solicitation.

Through the analysis of this experiment, the results indicate that the thixotropic behavior of the solder paste is given by the metal content. The interactions between the particles hinder the ability of the solder paste to recover after the application of shear stress.

4.5 Creep test

Regarding the capacity of the solder paste to recover after the removal of the constant shear stress applied, these results show some recovery in contrast to the flux results. By performing the calculation of the ratio of recovery, the values obtained for the three levels of solicitation, and presented in Figure 14, are the following: 87 per cent at Level 1, 40 per cent at Level 2 and 14 per cent at Level 3. These results present two interesting conclusions. On the one hand, the creep-recovery test is in accordance with the thixotropic test, proving that the solder paste is thixotropic. On the other hand, it was observed that the solder paste with the lowest level of solicitation presents the highest capacity to recover its structure, compared to the most requested one.

Concerning the deformation, the result indicates that the capacity of the solder paste to deform is ensured by the flux as it presents a higher extent of deformation. On the other hand, regarding the recovery, it appears that this property is essentially ensured by the metal particles. A high percentage of recovery is a desired feature as it minimizes the tendency of the solder paste to slump (Durairaj *et al.*, 2008), to ooze out underneath the stencil and to reduce clogging (Chen *et al.*, 2014).

Figure 13 Thixotropic behavior of the solder paste and its flux

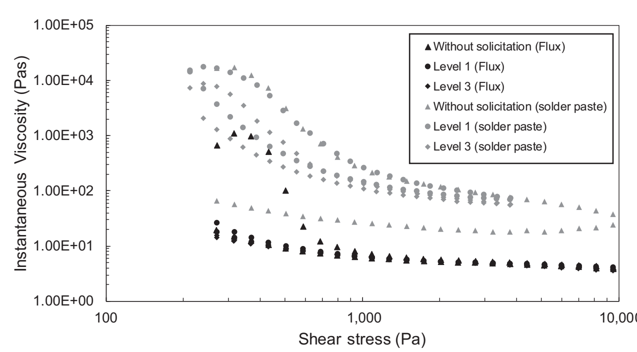
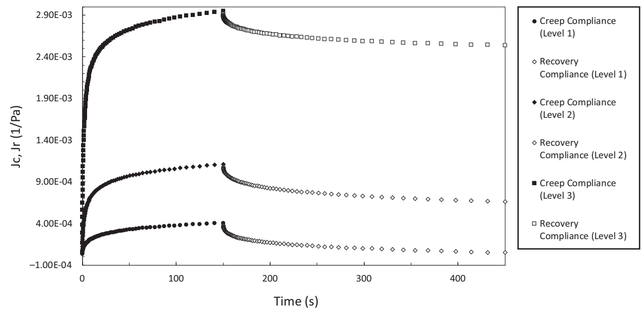


Figure 14 Solder paste creep-recovery test results



Source: Adapted from Barbosa *et al*. (2017)

5. Conclusions

This paper presents a rheological analysis of the flux media of the lead-free solder paste F620, to understand the influence of the flux in the rheological properties of the solder paste. According to the rheological experiments, it was observed that the flux's viscosity decreases with the increase of the shear rate. Moreover, it was observed that the metal content is majorly responsible for the increase of the solder paste viscosity. Another parameter that plays a vital role in the rheological behavior of the solder paste is thixotropy. The results presented in this paper show that the flux is not thixotropic, as opposed to the solder paste. In this way, the results indicate that the solder particles are majorly responsible for this property. Regarding the oscillatory tests, the results confirmed that the cohesive/tacky behavior of the solder paste is given by the flux as it requires a higher shear stress to change from a solid- to liquid-like behavior. In addition, the results show that the LVR region decreases with an increasing number of solicitations, indicating that the structural resistance of the flux tends to deteriorate as the forces applied on the sample increase. Regarding the creep-recovery tests, the results demonstrated that the flux does not recover after the application of a constant shear stress, which is not the case with the solder paste. The solder paste has shown a higher percentage of recovery, which has the tendency to decrease with an increasing level of solicitation. Through these tests, the results indicate that the capacity of the solder paste to deform is attributed to the flux, while the capacity to recover is essentially due to the metal content. Furthermore, in the case of the solder paste sample, it was observed that the lowest level of solicitation presents the highest capacity to recover the structure when compared to the most requested one.

The rheological analysis of the flux presents promising results that help to understand the influence of the flux in the rheological properties of the solder paste during the stencil printing process. However, further analyses are required to test the printing performance of the solder paste.

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