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Rheology Testing of Solder Pastes and Conductive Adhesives – A Guide

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Abstract

This guide discusses rheology practice for measuring solder paste and conductive adhesives. The main parameter, viscosity, can be measured to assess the flow properties of printing media. Additional tests can be conducted to assess the visco-elastic properties of printing media as a response to an oscillating shear stress at various frequencies. The rheology of printing materials is a complex science and as illustrated in this report cannot be simplified to a single static viscosity reading.

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CONTENT

1. INTRODUCTION

Rheology tests are measurements of material flow and as such can reflect parts of the printing process. During printing the media is rolled under pressure from a squeegee blade travelling at between 10-200 mm/s. Apertures are filled when the squeegee blade is in close proximity, and the media particles are forced into the aperture and then stop moving. The final process step is when the stencil and a substrate separate as the board drops away, the stencil apertures empty, and the board now has the printed deposits. During the stencil separation adhesive force between deposit and stencil walls, have to be smaller that adhesion force between print deposit and a substrate and the cohesive force within the deposit. During the printing process the printing media is exposed to variable stress levels, and responds in a time dependent manner. The complex flow properties of printing media can be characterised by the Rheological Test Protocol illustrated in the Figure 1.

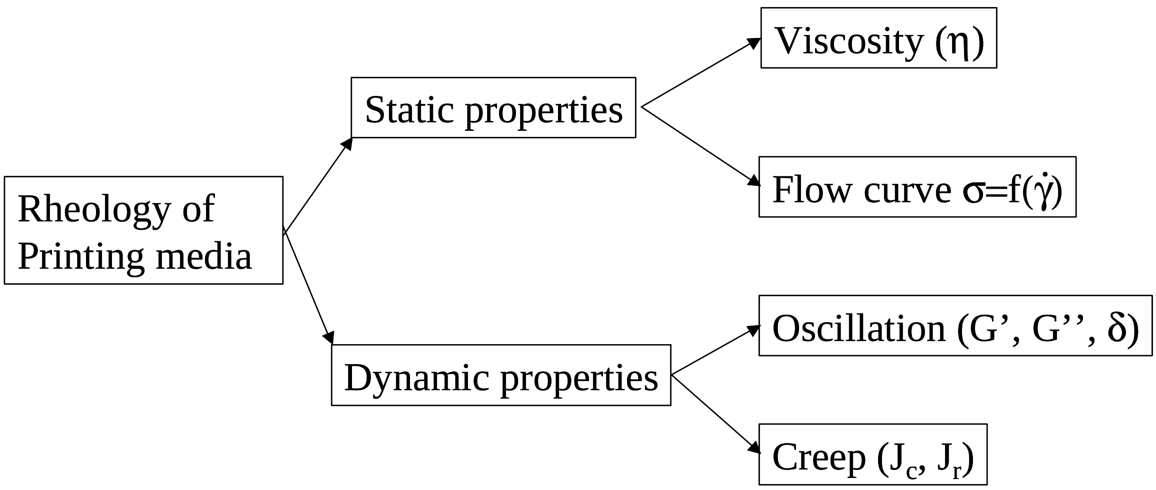

Figure 1: Differentiation of rheology properties by the Rheological Test Protocol

Before describing in detail the rheology tests for the printing media, solder paste and conductive adhesive, their structure has to be understood and analysed.

Solder Paste

Solder paste (cream) is a suspension of solid particles in a liquid vehicle. The whole system is composed of the following parts:

- ***Solder particles**:* These are SnPb or lead-free alloy particles of approximate spherical shape, and range in diameter from 5 to 70 µm. To assess the solder particles size and shape a conventional SEM can be used. Particles can be inspected directly after dissolving from solder paste. Metal content can be checked by weighing printed deposits before and after reflow, providing flux residues are removed after reflow (usually with iso-propyl-alcohol). The metal content, size and shape significantly affect the rheology.

- ***Binder (resin)***: This part is responsible for **tackiness.** This is sensitive to solvent loss, which can reduce tackiness. During reflow resin reacts with metal oxide layer in a reduction reaction and helps maintain clean surfaces for wetting.
- ***Solvent:*** Used to hold binder in solution. They can absorb water, which makes the binder less efficient. Loss of solvent (evaporation) is responsible for shortening solder paste stencil life (drying).
- ***Thickener:*** Thixotropic materials responsible for gel structure (elastic rather than viscous) of solder paste. Paste needs some recovery time from stressed state (time-dependent, non-Newtonian), and this recovery can be characterized as a time period related to ratio of G' and G" (elastic and viscous modulus). Dispersing solder particles in a gel is a way to eliminate sedimentation. This is attributed to yield stress (famous as shaking ketchup onto a shirt). Adding a gelling agent (thixotropic part) into the flux causes an increase in viscosity at very low strain rate. A controlled strain rate rheometer (rather than stress controlled) is required to measure this property.
- ***Activators***:– Improve activity of binders in reduction of surface metal oxide layer of a pad/termination. Their contribution in rheology of solder paste system is minimal.

Isotropic Conductive Adhesive

An isotropic conductive adhesive (ICA) is a solid particle suspension in a polymer resin. The main difference between the solid particles in solder paste and solid particles in a conductive adhesive is in size and shape of particles. The conductive adhesive particles are several times smaller and are rod and flake shaped. These particles do not reflow and have to be electrically conductive even when oxidised on the surface. The metal loading of these materials is a compromise between volume conductivity and strength in the cured state. Curing of temperatures of these materials range between 120-150°C.

Rheological Parameter Range

One of the main tasks in characterising the rheology of these materials is to identify the relevant **shear strain rate range** of the process. For printing this can range from 1 to 20 rad/s. The exact shear strain rate can be obtained from a visual examination of the flux bleed on the underside of the stencil, as described in [1]. Having estimated the working range of shear strain rate the appropriate parameters for the rheology tests can be decided.

2. RHEOLOGICAL TESTS

The various test methods as identified in Figure 1 are considered and their attributes to describing print media discussed.

2.1 Viscosity & Flow Curves

Flow curves are basic rheology characteristics showing static properties of flowing materials. They express a relation (function) between shear strain rate and shear stress applied to a material, which flows. The property called the apparent (synonym of instantaneous or dynamic) viscosity is defined as a ratio between shear stress and resulting shear strain rate of flowing material. A typical flow curve is shown in Figure 2. Historically, solder paste manufacturers have performed the viscosity measurement of solder paste as a statistical process control (SPC) tool for evaluating product quality (and typically measured on a Brookfield or Malcolm viscometer). Unfortunately a single value of apparent viscosity of solder paste does not characterize the material sufficiently, and hence a parallel plate rheometer is recommended, as discussed later. The value of viscosity is very dependent on metal loading and hence comparing solder pastes with different metal loading can be problematic.

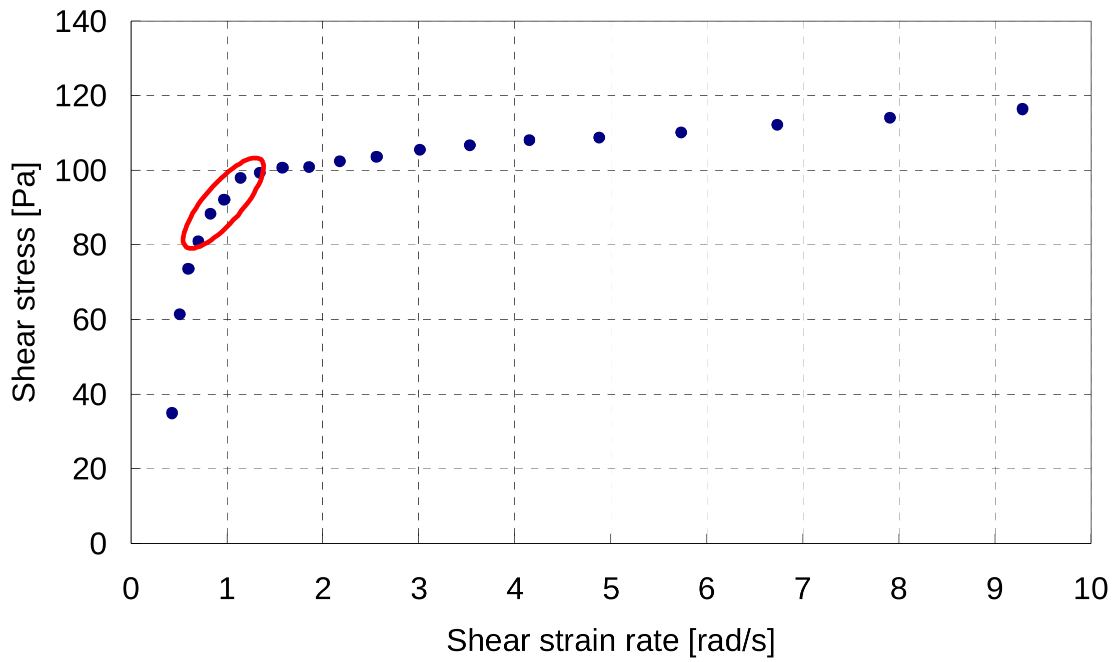

Figure 2: Flow curve, red ellipse indicates working region of shear strain rate and related stress for a solder paste extract

An alternative approach is to look at the solder paste extract separated from solder particles. An advantage of this approach is that the measurement is independent of metal loading and particle size. To achieve this metal particles have to be separated by centrifuge from solder pastes, which typically can be achieved at 6000 rev/min for 1 minute. The measurement gap should reflect the printing process studied and can be varied from 50–500 µm. Typically apparent viscosity of a solder paste extract with a low metal content is higher than of paste with high metal content.

Figure 3: Parallel plate geometry with a liquid solder paste extract as a measurement sample

The viscosity of any printing media strongly depends on the solid particle content, size and shape and on the compounds that are added to make the paste thixotropic (time – dependent behaviour). Viscosity is temperature and humidity sensitive, and at temperatures above 30°C rapidly decreases. The viscosity can increase substantially during storage hence printing media should be stored in a temperature controlled store, i.e. in a fridge or freezer. In case of conductive adhesive the material should be stored at -40°C. For rheometry tests a controlled stress/strain rate rheometer (e.g. Bohlin, Haake) should be used with ∅20 mm parallel plate geometry at 25 °C as seen in Figure 3, such an approach permits rheology to be studied over a wide range of conditons.

To evaluate viscosity of a conductive adhesive the size of solid particles must be considered. As the particle size can be below 1 µm, particles do not separate from the liquid vehicle and whole system is more compact than solder paste during printing operation. Additionally it is very difficult to separate resin from metal particles and filler by centrifuging. Although the viscosity characteristic of a conductive adhesive is similar to that of solder paste extract, the system behaves differently in the dynamic range. The rheology of materials in the dynamic range can be evaluated by the oscillation tests.

2.2 Oscillation tests

The oscillation test is a non-destructive test for determining a sample behaviour in the visco-elastic range. This is done through applying sine oscillations of shear stress at constant frequency to the parallel plate (spindle) and measuring a shear strain rate response. The delay of the response, called phase lag, is a measure of the visco-elasticity. The range where the phase lag shows a linear dependency on shear stress amplitude is known as the linear visco-elastic region, an example is presented in Figure 4.

Once the visco-elastic region is determined the material can be tested in a frequency domain, while maintaining constant amplitude oscillations. Typically the frequency is swept over a range of 0.1-10Hz.

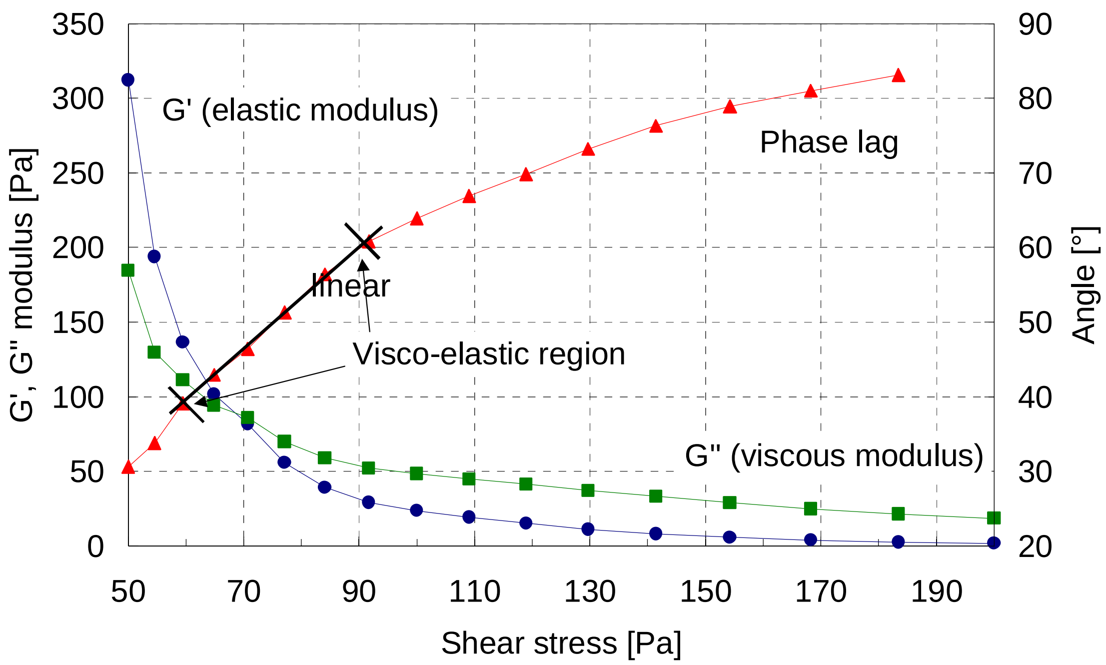

Figure 4 Oscillation test run at 1 Hz for determination of visco-elastic region

An example of frequency response of a solder paste extract is shown in Figure 5. From this response the visco-elastic behaviour of a material can be predicted. From rheological point this liquid extract demonstrates more elasticity as the shear stress frequency is increased. The increase in elasticity as opposed to flow is equivalent to **solidification** of a material and ability to return input energy in a short time.

Conductive adhesive measured by oscillation technique can be similarly performed as in the case of solder paste liquid extract. The only difference is that the sample of conductive adhesive is not centrifuged and enters the test in unchanged state.

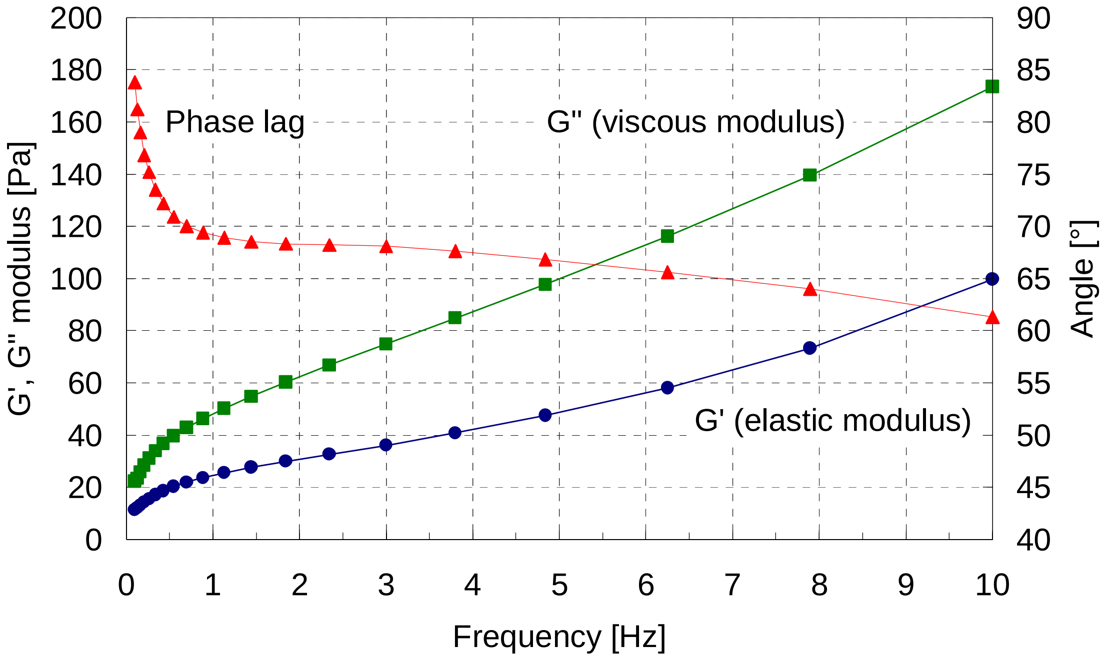

Figure 5: Oscillation test run at 90 Pa shear stress of harmonic amplitude for determination of frequency response of solder paste extract

2.3 Creep test

The creep test is a technique used to assess time-dependency behaviour of a printing media. A sample under the test is exposed to a constant value of stress in shear direction and the strain is monitored with time. After the strain stabilises the stress is removed while strain is still monitored. Typically time dependent behaviour called **thixotropy** is revealed and is shown schematically in Figure 6. This test can be useful for some pastes for characterising the tendency of a paste to slump, with the slump propensity correlated with the relaxation in strain after the stress is removed.

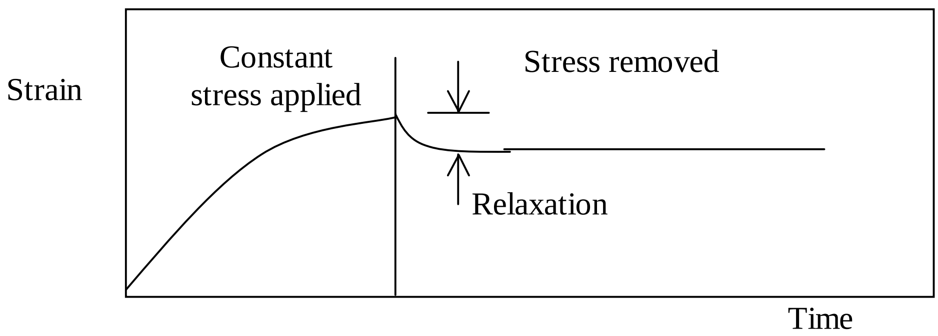

Figure 6: Schematic of creep response of print media

3. SELECTION OF THE APPROPRIATE RHEOLOGY TEST

Where rheological characterisation can be shown to be valuable the following approach can be adopted. The basic print defects are considered and the most relevant rheology test for characterising the defect is suggested.

Four basic print defects occur during stencil printing:

- Insufficients: low volume of solder paste
- Skips: random pads with virtually no paste at all
- Slump: print deposits lose height and spread at the base
- Bridging: particles and flux merge from adjacent prints to form a continuous deposit

Table 1: Suggested rheology tests for characterising stencil print defects

Print Behaviour	Rheology Test	
Insufficient	Viscometry:	The viscosity at a predefined shear rate will indicate the basic propensity for the paste to print through and may correlate with volume.
Skip		
Slump	Creep:	The degree of relaxation after an applied stress has been released can be correlated to slump propensity
Bridge	Oscillation:	The visco-elastic behaviour of the flux vehicle is important in how under stencil bleed takes place

4. CONCLUSIONS

This guide introduces measurement test methods that characterise rheology properties of solder paste extracts and conductive adhesives. Before these methods can be utilized the relevant shear strain rate to a printing process has to be identified. This can be undertaken for solder pastes using under screen videoing of the process [1].

Although the viscometry tests can be performed to rank performance of solder paste liquid vehicles by their apparent viscosity, this measure does not correlate well with printing performance. For solder pastes it is more appropriate to study the liquid vehicle directly, since this is responsible for the time dependent properties. Liquid vehicles can be separated from the paste by centrifuging. Static measurements of viscosity only provide limited information and do not characterise the complex behaviour of solder paste during printing, hence the need for dynamic measurements.

Dynamic properties should be tested with an oscillation test. Dynamic oscillation tests measure visco-elastic properties of a material and should done at the working shear stress. Of importance is the increase in elasticity on exposure to oscillations above 0.1 Hz. The values are important in terms of the aperture release process.

Creep measurements reveal the time dependent behaviour called thixotropy. This is a measure of the memory effect within a material with time and reveal something of the paste ability to prevent slump.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

[1] Rheology Testing of Solder Pastes and Conductive Adhesives Using in Stencil Printing, Miloš Dušek, Ling Zou and Christopher Hunt, NPL Report MATC (A) 102