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Flowing and pressurizing a solid-liquid two phase monodispersed fluid with high solid content in a transparent microfluidic high-pressure chip

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Abstract. Handling highly concentrated solid-liquid two-phase fluids in microfluidics is challenging. In this paper, we present the first studies of flowing solder paste with a high solid content in a transparent high-pressure tolerant glass chip, thereby increasing the understanding of how multiphase liquids with high density difference between the phases behave in small channels (840 µm in diameter). The system, including a custom made high-pressure, low resistance, interface, was continuously operated at pressures up to of 6 MPa and devices where shown to have pressure tolerance up to 17 MPa. During flow through the chip, the packing density of the solder balls displayed inhomogeneity over the channel where chains of solder balls in contact with each other were formed together with voids. These in-homogeneities persisted along the channel during flow. The flow rate of the paste through the chip oscillated between 63 to 350 µm/s when pumping at constant volume rate of 30 µl/min. When a pressure of 2 MPa was applied, the volume of the solder paste particle segment decreased 1.6%, and 0.1% was elastically recovered when the pressure was released. It is concluded that this transparent microfluidic high-pressure glass chip with the special developed interface is suitable for flow studies of solder paste with a high solid content.

1. Introduction

One of the remaining major challenges in microfluidics is the handling of solids [1]. Both in technological and biological systems, concentrated suspensions are of vast importance. Blood, samples from organic matter, inorganic slurries, some drugs and paints, are a few examples of two-phase systems containing solid particles in a liquid [2]. Diluted solid-liquid microfluidic systems have been studied extensively [3], where particle focusing [4], trapping [5] and aligning the particles [6] are ways to manipulate the fluids. However, very few studies have been made using high solid content fluids in microfluidics. Our aim with this paper is to show how pumping of a concentrated, high viscosity solder paste suspension can be done in a microsystem and to highlight some of the difficulties that are necessary to deal with.

Solder paste is used to connect surface mounted components to printed circuit boards. The fluid is very tacky since one purpose is to temporarily keep the component in place before the actual soldering. The paste is a mixture of micrometer-sized metallic solder balls suspended in liquid flux. The non-Newtonian pastes are formulated in order to be highly thixotropic, meaning that the viscosity is lowered when the paste is sheared [7, 8]. This is a property seen in many fluids of industrial value, including paint, allowing for the liquid to flow readily while keeping its shape after being dispensed. The viscosity can also be very temperature dependent. Viscoplastic materials, like solder paste, mud

and concentrated slurries are also Bingham plastics, *i.e.* the stress needs to be above a yield stress before the fluid starts to flow [2]. This is a behavior found in concentrated solid-liquid two-phase systems, when there are large interactions between the particles in the liquid, forming a weak continuous solid structure.

To dispense the paste to the solder pads on printed circuit boards, stencil printing, pneumatic dispensing or jet printing can be used [9]. Jetting, being both flexible and fast, has become a standard process in surface mount technology production [10]. The size of the solder particles in jettable pastes are, depending on grades, in the range 2 to 35 µm, and the solid loading is typically 84-86% by weight. During jetting through orifices that can be down to 100 µm in diameter, the paste is subjected to extreme stresses with pressures up to 15 MPa and shear rates up to 10^{-6} s^{-1}. Since solder paste in many aspects is a very difficult fluid to handle, many details of the behavior of the movements in the microchannels inside the jet head is still unknown, making it hard to model and optimize the jetting.

In this paper, we present a high pressure tolerant transparent microfluidic glass chip designed for studies of the rheological behavior and compressibility of solder paste in order to obtain a deeper understanding on how multiphase liquids with a high solid content and high density difference between the phases behave in a microfluidic chip. Since the flow resistances and viscosities are much higher than from what normally is seen in microfluidics, we utilize large cross-sections channels and interfaces for our study.

2. Experimental details

To enable the flow of solder paste, a microfluidic chip and special interface must be designed, allowing for low flow resistance. First, an initial flow study using a syringe pump (Harvard, PHD 2000) was conducted using centimeter long tubing with an inner diameter ranging from 75 to 810 µm to determine what minimal dimensions that are needed for the solder paste (experimental sample, Mycronic) to flow through. The volume fraction of solid particles in the solder paste was around 0.4 and the particles had an averaged diameter of 5 µm. Based on the results from this study, a microfluidic chip having a T-junction and meander was constructed, Fig. 1. The chip was used to study the flow velocity, as well as investigating the response to the paste upon pressurization.

Glass chips were fabricated by the methods described in [11]. In short, borosilicate glass wafers were first sputtered with an etch mask made from a 2 µm thick Mo layer. This mask was patterned by lithography to create 60 µm wide openings followed by etching of the Mo with a solution of H_3PO_4:HAc:HNO_3:H_2O at a ratio of 180:11:11:150, thereby defining the channels. Using concentrated HF, trenches in the glass are isotropically etched down to 420 µm. After bonding two structured wafers together, the semi-circular cross-sections of the trenches form an almost circular channel. Before bonding, the glass was activated using 10 wt% KOH at 65°C followed by 69 wt% HNO_3 at 80°C. Bonded wafers were thermally treated at 625°C.

A low flow resistance fluid interface was specifically designed to connect to the chip. The interface consisted of 5 cm of stainless steel tubing (OD 1/16", ID 810 µm, IDEX) that was joined closely to the chip by inserting 6 mm long PEEK capillaries (OD 1/32", 800 µm, ID 510 µm, IDEX) into both the tube and chip inlets. A two-component epoxy (Araldite Rapid, Huntsman Advanced Materials) was applied around the interfaces of the tube and chip, forming a sealed and rigid junction. The two outlets marked b and c, Fig. 1, were then connected to two high-pressure needle valves (15-11AF, HIP). The valves were oriented so that when they were in a closed position, the needle cuts the fluid path from the chip perpendicular, thus minimizing dead volume in the valve.

To investigate the durability of the chip, a fracture test was done by inserting a silica capillary (150 µm OD, 75 ID, IDEX) into a straight segment of a channel and plugging the outlets with epoxy. Ethanol with a flow rate of 200 µl/min was pumped (ISCO 100 DM, Teledyne) into the chip by the capillary, resulting in an average pressure increase of 2.1 MPa/minute, read by the internal pressure sensor of the high-pressure pump.

For the flow study, a syringe pump (Harvard, PHD 2000) was used, set to 30 µl/min. For the flow studies, the temperature of the chip was raised to 27-32°C to decrease the viscosity of the paste and allow for pumping. A low friction syringe (1005, Hamilton) was filled with the solder paste.
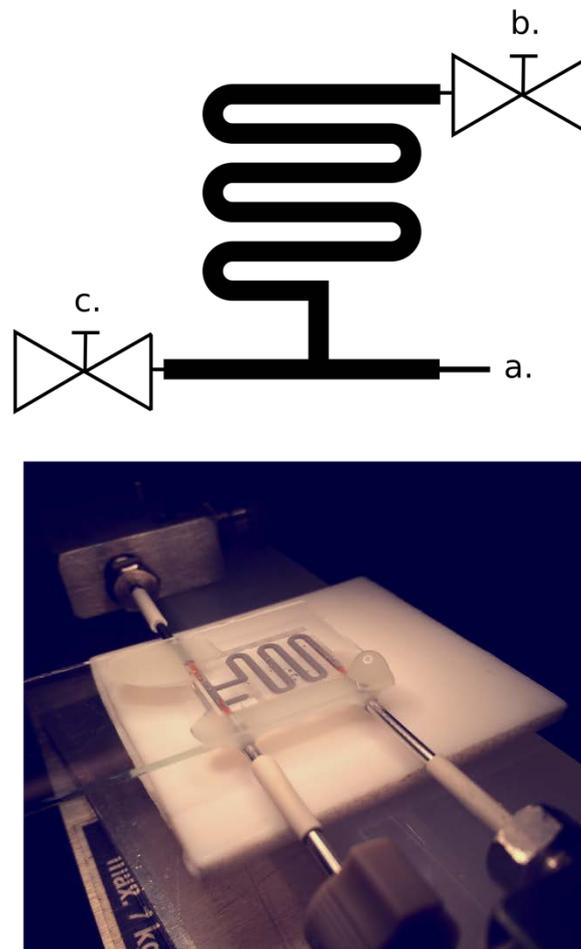
Figure 1. A sketch showing the channel geometry (top) and a mounted chip with fluid inlets and solder paste flowing in the channels (bottom). The inlet (a) is connected to the pumps, (b) and (c) are outlets connected to high-pressure needle valves. The high-pressure tolerant interfaces consisting of orange PEEK and stainless steel tubing are visible in the photo.

To investigate the effect of pressurizing the solder paste, a paste segment with a defined volume was first made. Paste was pumped in the chip from point a to b (Fig. 1) at 5 µl/min for 10 min, then the valve at point b was closed thereby cutting the paste segment off. By flowing hydraulic oil (HDZ 68, Texaco) from point a to point c, a defined segment of solder paste was formed having oil-paste interface at the T-junction. Using this procedure to fill the chip with paste, it was ensured that there were no air pockets present in the channel. While filling the chip with solder paste, the flow was visually monitored to ensure that no air segments were present in the fluid.

The volume, including tubing and channels in the chip up to the T-junction, was 5.6 µl. By closing the valve at point c and pressuring the chip using a high-pressure piston pump (ISCO 100 DM, Teledyne), the compression and the following the displacement of the paste segment could be studied. The temperature during the compression test was 21 ± 1 °C. The pressure was recorded using the internal pressure sensor of the high-pressure piston pump. Imaging and video recording were done with a stereoscope (Nikon SMZ800) with ring light coupled to a CCD camera (DFKMN33UXE174, Imaging Source). Image analysis of the videos was used to calculate the flow rates using the solder balls to track the flow velocity.

3. Results and Discussion

The durability test showed that the chip fractured at a pressure of 17.3 MPa and inspection of the cracked chip revealed that the crack propagated close to the bond plane inside the glass and in close proximity to the glued interface of the inlet. During the flow and compressibility tests, a maximum pressure of 6 MPa was applied.
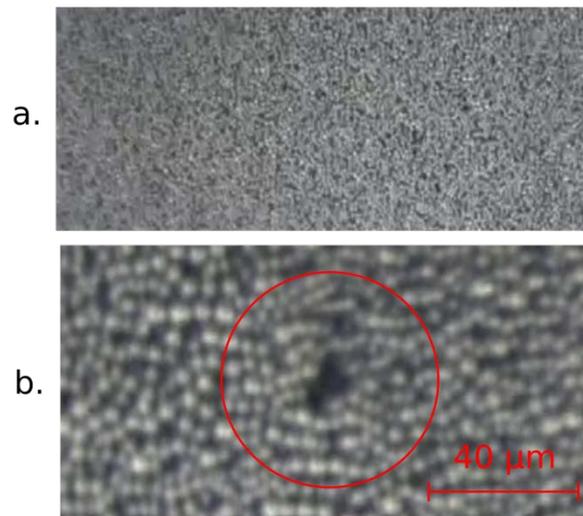

Figure 2. The solder paste flowing in the channel at 30 µl/min. In (a) an overview of the channel width is seen. (b) shows a close up, showing the solder balls. The solder balls are generally in an unordered glassy state, but local ordering in the form of chain structures can be seen. A void having no solder balls is marked with a red circle.

The initial trials lead to the conclusion that solder paste at room temperature could not be reliably pumped through tubes having an inner diameter smaller than 160 µm, and for the chip, the smallest diameter of the system was that of the interfacing tubing. This diameter was chosen as 510 µm, in order to not restrict the flow too much.

For the flow study, the resulting flow speed was not constant, but showed pulsing with faster flow periods, having a frequency of 0.07 +/- 0.007 Hz, lasting on average 15 s for each pulse. The flow velocities for fast and slow periods were 350 and 63 µm/s, respectively.

As Fig. 2 shows, the packaging of the solder balls is not perfectly homogeneous and voids, typically 5 to 30 µm in diameter, where no balls can be found are present. These voids continue to travel with the balls along the channels for long distances and can be observed for several seconds over the recorded image area. It was also observed that these voids can disappear, possibly moving into the bulk of the fluid. No gas-liquid interfaces can be seen in the voids, suggesting that they consist of flux rather than air. It was possible to track the position of individual solder balls in the channel and in Fig. 2 b, areas where the local ordering of particles in chains structures can be seen. This type of random particle chains is known to cause jamming and the fluid has a microstructure resembling that of a glassy state [2]. While the pulsating flow cannot be correlated directly to the formation of these chain structures, the presences of them suggest that jamming as a result of periods with extensive ordering is possible. During periods where the paste is less ordered, flow is less restricted.

Pressurization of the paste using 2 MPa resulted in a large, permanent, rearrangement of the solder paste particles, Fig. 3. The interface of the solder paste balls moved about 1.6 mm into the channel, corresponding to a volume compression of 1.6%. Upon releasing the pressure, 0.1% of the compression was showed to be elastic and went back after reducing the pressure to 0.1 MPa.
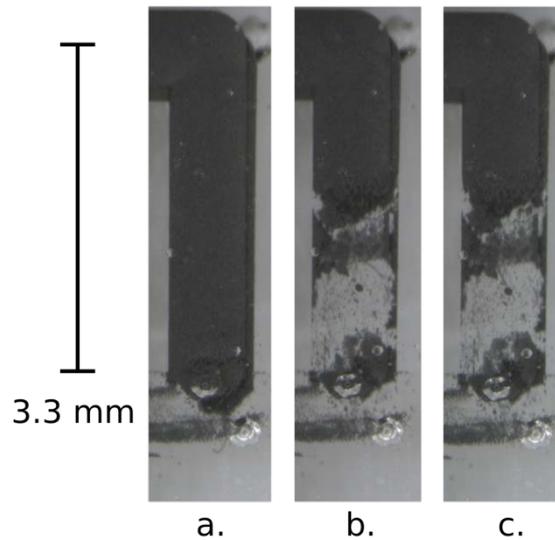
Figure 3. The T-junction of the chip with solder paste and hydraulic oil. In (a), hydraulic oil has been flowed from right to left in the lower channel, cutting the solder paste plug off and forming and interface. In (b), the channel is pressurized from 0.1 MPa to 2 MPa resulting of the solder paste interface moving about 1.6 mm in the channel. After the pressure is returned to 0.1 MPa (c), the interface retracts back to the T-junction by less than 0.1 mm.

The low optical contrast between the flux in the solder paste and the hydraulic oil used as the pressurizing medium made the meniscus between them not visible; making it difficult to judge to what degree the rearrangement of the solder particles was accompanied with a phase separation of the flux. To make more precise measurements of the compressibility, it is necessary to have better contrast between the flux and the media used for pressurization. This can either be done using a pressurizing fluid that has a different color, or by adding a dye that can give contrast at some wavelength.

For future studies, it would be advantageous if both the inlet and outlet part of the solder paste plug could be monitored at the same time by designing a chip where they are in close proximity to each other. Also, having a smaller channel diameter of the T-junction, where the length difference is detected, compared to the meander containing the bulk of the paste would make it possible to detect even smaller changes in compressibility. Further, a smaller diameter of the T-junction should also allow for a more focused cut off of the solder paste segment, thereby further increasing the resolution of the method.

As shown by the initial studies presented here, several complex behaviors are involved when high solid content fluids flow through microchannels. With the use of transparent, pressure tolerant glass chips, insight into the fluid handling can be given.

4. Conclusion

- The transparent microfluidic high-pressure glass chip with a special developed interface was shown to be suitable for flow studies of solder paste with a high solid content.
- The chip was pressure tolerant up to 17.3 MPa making it possible to study effects of applying pressure to the fluid. Operation with the low restriction fluid interface was demonstrated up to 6 MPa.
- The flow rate through the chip displayed a pulsing behavior that could be connected to local ordering effects and jamming of particles in the solder paste.

- Pressurization with 2 MPa resulted in a permanent re-organization of the solder paste balls, corresponding to a compaction of 1.6%. Only 0.1% of the compression was elastic and could be retrieved as the pressure was released.

Acknowledgment

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