Contact Angle and Surface Tension Measurements of Solders

October 15, 1997

This application note will illustrate surface tension and contact angle measurements of molten solder using the FTÅ200 system.

The FTÅ200 has an optional Environmental Chamber which can maintain a temperature and, if desired, an atmosphere. The temperature was set to 200°C, hot enough to melt the 63% tin, 37% lead Kester "44" brand rosin core solder used in this work. The atmosphere was air.

Both pendant drop and sessile drop surface tension measurements were made on the solder. Pendant drop measurements are inherently more accurate because the re-quirement of axial symmetry (the drop must have the same shape when viewed from any side) is more easily met when the fluid hangs from a stainless steel needle. However, the sessile drop technique more closely mimics how solder is used and shows the effects of flux more realistically.

The needles used in the pendant drop work were loaded in the fashion described in FTÅ's application note ***Surface Tension Measurements of Polymers, Waxes, and Solders***. Basically, the sample material is melted in a crucible over a hot plate and a small amount drawn into a syringe with a stainless steel dispensing needle by applying a vacuum with the syringe plunger. The lower, say, 15mm of the needle is heated by letting the tip stay immersed in the molten sample for a few seconds before withdrawing the syringe plunger to pick up sample. This pulls material up into the heated portion but no further. By using 2 inch (50mm) long needles, the upper portion never gets heated, either during the filling operation or during subsequent testing, so inexpensive disposable plastic-hubbed needles may be used.

The first figure shows a pendant drop of solder which was not yet oxidized. The flux-cored solder was melted in the crucible and the 20 gauge (0.635mm inside diameter) dispense needle loaded immediately. A film of flux remained over the molten drop in the crucible.

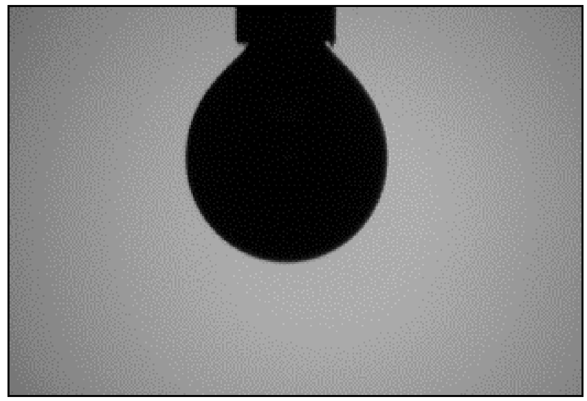

Figure 1. Unoxidized solder, γ=511dy/cm.

Compare the shape of the drop in the next figure of the same heated solder after exposure to air.

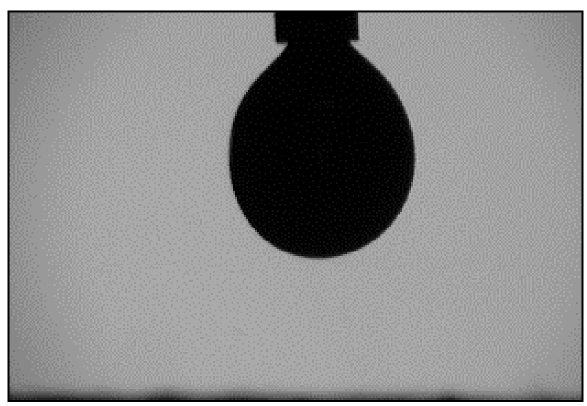

Figure 2. Oxidized solder, γ=380 dy/cm.

The droplet is noticeably narrower, and the surface tension has decreased significantly. This is due to the oxidation of the surface layer. The oxidation could be seen visibly also, as the droplet surface was a dull gray rather than a bright silver color.

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Metal oxides have lower surface tensions than the pure metal and, more importantly in this application, a higher melting point. The purpose of flux is to prevent solder oxidation and help prevent any oxidation of the work.

The nominal melting point of this solder is 361°F ≈ 183°C. Therefore the 200° chamber temperature was only about 17° above the melting point in this work. Once fully oxidized, the surface was no longer molten and would hold a shape if disturbed. This is an important point when doing measurements of this type: if the sample is no longer a fluid, the shape may not represent its surface tension and remarkably wrong results can be obtained.

Another important point in working with the FTÅ200 Environmental Chamber is that it offers the convenience of bottom loading for changing the solid sample. Fluids, on the other hand, are introduced with syringe needles through a small hole in the top. The bottom loading lets the chamber remain at an elevated temperature while the specimen is replaced and this shortens the cycle time. However, sufficient "soak" time must be allowed for the solid sample and bottom plate to come back up to temperature after loading. Something like 5 minutes is required at 200°C after the main readout says 200 again.

Surface tension is only mildly temperature sensitive, but viscosity and oxidation time are very sensitive to temperature. Viscosity falls with temperature and affects the flow characteristics of solder, while oxidation will raise the melting temperature.

Next a sessile drop surface tension experi-ment was run. In this case, unmelted solder was placed in a 19 gauge (0.787mm inside diameter) needle tip. Normally about 10mm of solder was used, so as to make an appreciable drop. The inside diameter of the needle matched the solder's outside diameter closely, so the solder would stay in place when the needle was inserted through the top of the chamber. As soon as the solder heated up, its flux melted and came out. At this point the solder could be held in place by pulling back with the syringe plunger and forming a slight vacuum, or the syringe could be depressed and the solder would be ejected downward onto the sample. If the syringe was left undisturbed, the solder would fall down when it was molten or almost so. Using this last approach, a movie was made of the sessile drop of solder, with its normal amount of flux, on a non-solderable (non-wettable) metal surface.

The first frame of the stabilized drop after it formed on the surface is shown in Figure 3. The solder drop, almost a ball, is sitting in a small pool of flux; the flux forms the "fillets" at the surface on the left and right sides of the drop profile in the image. The point of this is that an "excess" of flux was present (but this is normal for flux-cored solder). The surface tension of the drop at this time was 542dy/cm. The drop's "age" at this point was about 1 second.

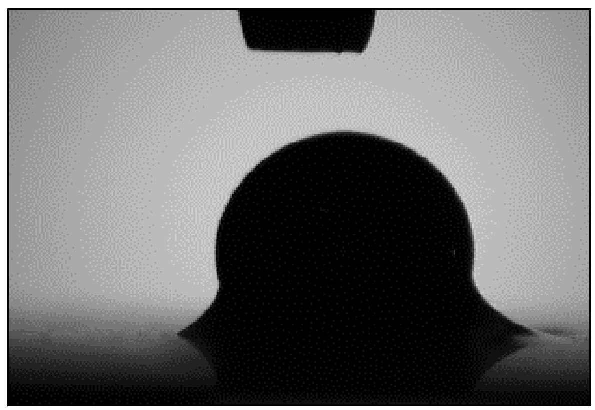


Figure 3. New drop of solder in flux pool.

The shape of the drop was recorded over the next 150 seconds and the surface tension analyzed. The results are shown in the graph of Figure 4.

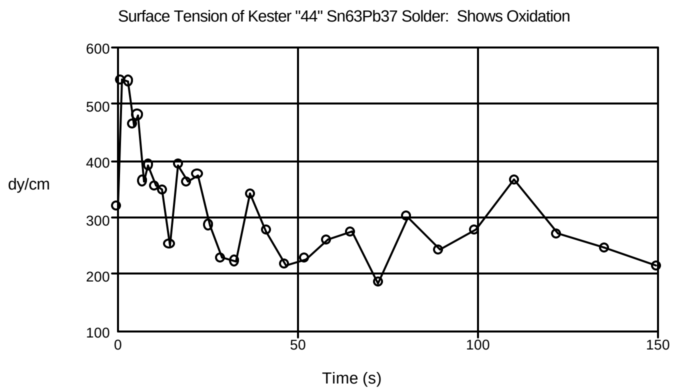


Figure 4. Surface tension of oxidizing solder.

This shows a trend towards lower surface tension as oxidation took place. The solder also visibly changed appearance. The very first data point, at t=0, has a lower value because the droplet was covered with flux and was still forming as the solder completed its melting, so this is a little premature. Much of the fluctuation of measured surface tension in the later times is due to the flux boiling and disturbing the sessile drop shape. The droplet shows considerable motion during these times, so these variations are understandable. The important point is the trend of surface tension from a starting point in the 500's to a steady state value of about 275dy/cm. Because of the motion of the drop from the flux, it is desirable to repeat these types of experiments and average the time functions to obtain a "true" picture of surface tension versus drop age. Finally, sessile drop surface tension measurements often have greater variance than pendant drop measurements because the sessile drop will not be as axial symmetric ("round" when viewed from above). However, the sessile drop experiment is sometimes the only practical approach.

What should the surface tension of solder be? Common rules of thumb are in the mid to upper 400's. Typical values are 550 for Sn and for 460dy/cm for Pb. Carrying these across on a percentage basis, one gets 517dy/cm. Metals have a wider range of reported surface tensions than, say, fluids like water, alcohol, or oils. This reflects the varied experimental conditions for molten metals. Reported values for Sn range from 526 to 590 and Pb values range from 444 to 480dy/cm.

The final set of experiments concerned contact angle and drop spreading on wettable substrates. The solder was Kester "44" and the chamber temperature 200°C. The solid substrate was brought to temperature before the solder was applied, but the temperature was only a few degrees above the melting point of the solder. This had the effect of slowing down the wetting process. The first substrate was solid bare copper and the second was a 10µm thick film of PtAg on alumina. The solder was applied from a 19 gauge needle onto the surface. Figure 5 shows the initial drop of solder on the Cu specimen and Figure 6 shows the PtAg specimen.

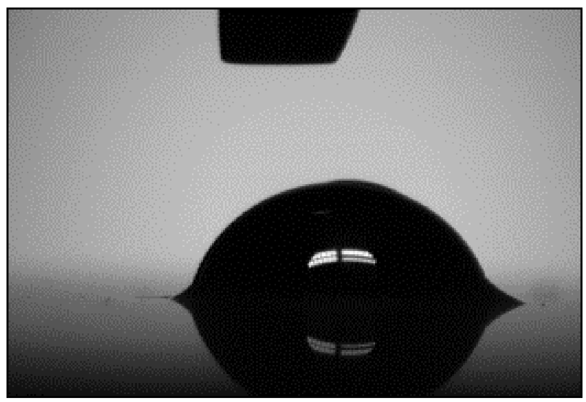


Figure 5. Cu sample with solder ball and flux.

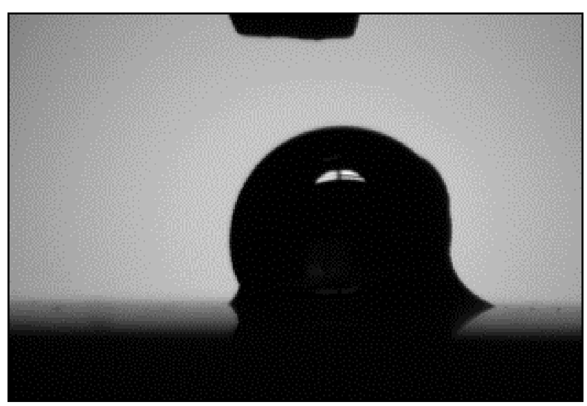


Figure 6. PtAg film with solder ball and flux.

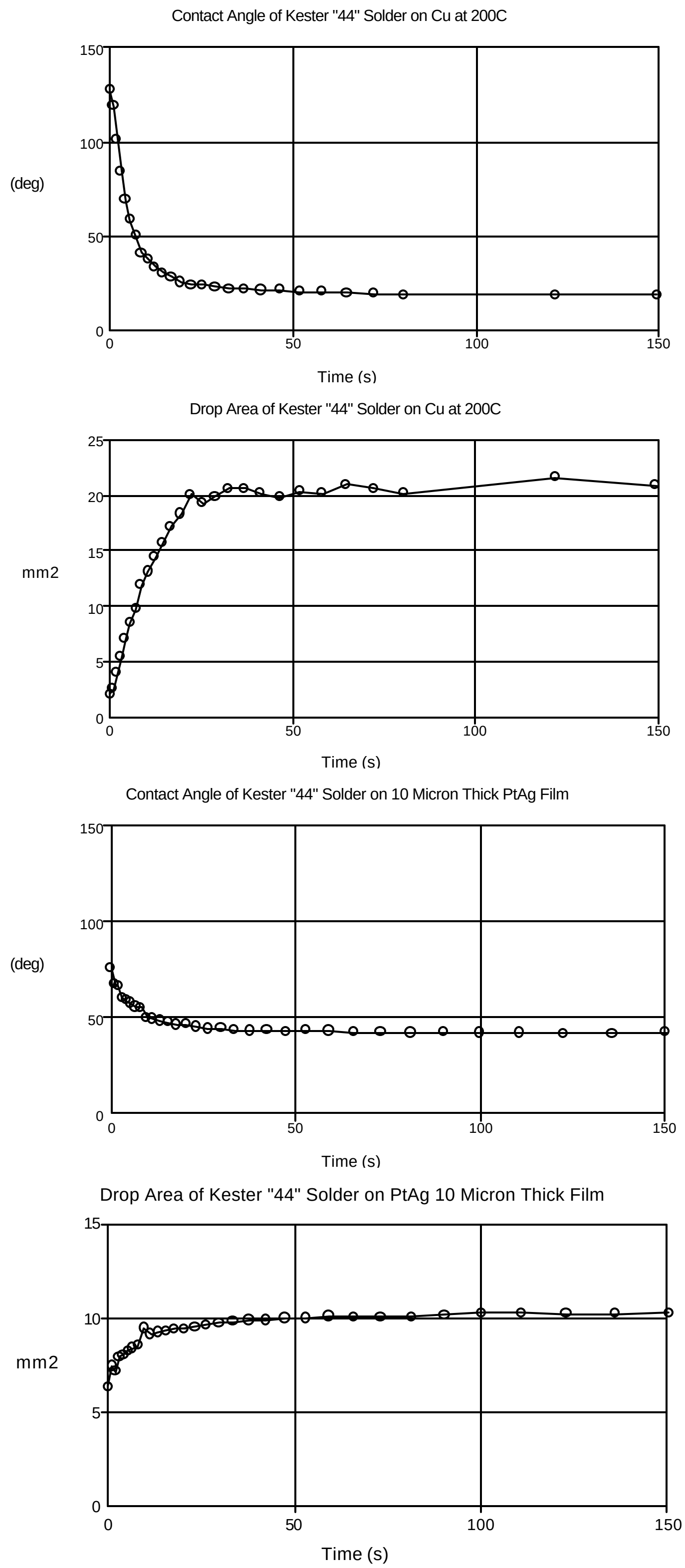
Except for the fact that the temperature is below what one would normally use for soldering, these experiments represent realistic solder applications.

The initial contact angle on Cu is higher, but overall it wets better and the ultimate contact angle is much lower, about 20° compared to 40°. The solder on Cu spreads to a much greater degree also.

One can measure the spreading rate with these experiments, but this is strongly affected by temperature since viscosity drops very rapidly with increasing temperature.

All data used in this report were obtained from sources listed in the bibliography in the software's Help file. The density used for the molten solder was 8.4g/cc.

This note was prepared entirely electronically. Images and graphs from the FTÅ software were transferred directly into the manuscript, which was then sent to printer via e-mail where it was output.



Figures 7-10. Contact angle and drop spreading