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Studies on Flux Action of Soldering (Report-1)†
— Stearic Acid—

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Abstract

This research was carried out to clarify the relation between flux action of organic acid and spreading phenomenon of solder, and the reactions of stearic acid with base metal (Cu) or with solder (Sn-Pb eutectic solder) were studied.
The results obtained are summarized as follows:
1) The spread on oxidized copper using stearic acid flux was greater than that on electropolished specimens.
2) Stearic acid reacted with copper oxide on heating to give copper stearate, with cleaning the surface to copper plate.
3) With stearic acid flux containing a little of copper stearate, the spread area increased markedly.
4) Copper stearate reacts with molten solder to give stearic acid and metal copper.
From these results, stearic acid reacts with copper oxide to give copper stearate which reacts with solder, and the Cu-rich layer at outer surface of molten solder is formed. The spreading of solder on copper specimen is improved by the reaction and the dissolution of Cu metal in solder.

1. Introduction

The use of soldering has afforded a ready means of joining common metals such as steel, brass and copper at a comparatively low temperature. In making good joints, the liquid metal of solder is required to flow and spread over base metal. It has been found that some combination of solder, flux and base metal plays an important role in spreadability of solder. But the flux action of controlling flow and spread has been little investigated systematically for its difficulties in the quantitative measurement of the spreadability. Recently studies on soldering and its flux action have been reported by some workers,[1),2),3),4)] however, the basic factors controlling flow and spread are not well understood.

In this report, we wish to report the chemical phenomenon of soldering and the relation between flux action of stearic acid and spreadability of solder. So, the reaction between stearic acid and base metal (Cu) or between the flux and solder (Sn-Pb solder), was studied as well as the spreading of the solder.

2. Experimental procedures

The flow or wet characteristics of solder on base metal are shown by the spread area to which a pellet of solder spread when melted under flux on a heated copper plate. In carrying out these spread tests, copper plates were polished with 06# emery paper and then electropolished. Grease and dust were removed by washing with acetone just before the testing. Both solder (100 mg) and flux (100 mg) were placed in the centere of the copper plate on a hot plate held at a controlled temperature as shown in **Fig. 1.** The specimens were heated to required temperature and maintained for 30 sec, and then removed and cooled. The spread area of the solder was measured. The all spread tests were carried out under an atmosphere of air.

The reaction of flux with solder or base metal (Cu) was done in a glass flask under similar conditions as described in the test. The reaction products were identified by IR spectra and measurement of *mp,* and chemical analyses. The cross section of specimens of soldering was analyzed by EMX analyzer.

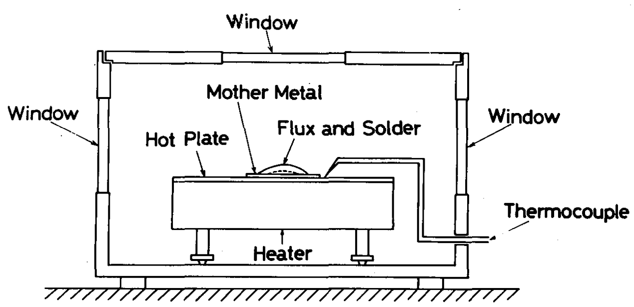

Fig. 1. Spreading test apparatus.

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3. Results and Discussions

3. 1 Reaction of molten stearic acid with Cu plate

Clean copper plate (3.5041g) was immersed in molten stearic acid as shown in **Fig. 2,** at 240°C for 2 hours. At the place of (1) in **Fig. 2,** stearic acid reacted with Cu plate to give green compound, which dissoled into molten stearic acid. The green compound (0.78g, *mp* 225°C) was isolated from the stearic acid part, reccrystallized from alcohol, and identified as Cu-stearate by comparison with authentic sample, (synthesized according to eq-1), in terms of its infrared spectra as shown in **Fig. 3.**

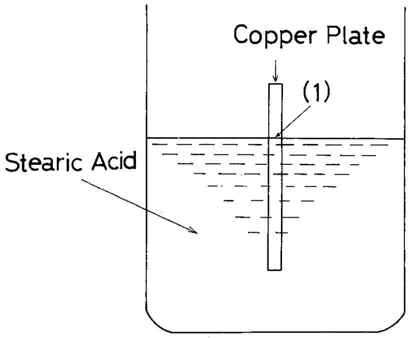

Fig. 2. Schematic explanation for dissolution part on copper plate dipped in stearic acid (at 240°C).

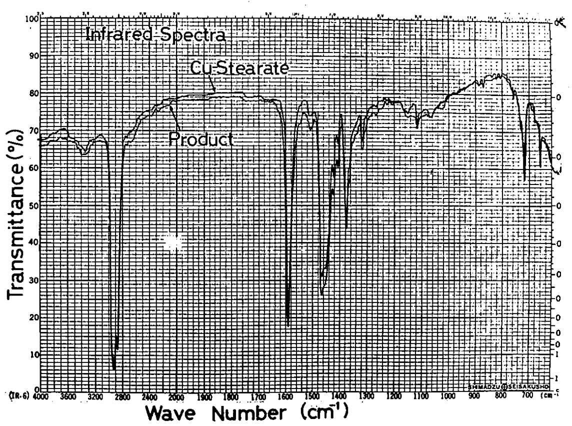

Fig. 3. Infrared spectra of Cu-stearate and reaction product.

$$C_{17}H_{35}COOH + NaOH \rightarrow C_{17}H_{35}COONa + H_2O$$

$$2C_{17}H_{35}COONa + CuSO_4 \rightarrow Cu\,(O\overset{\underset{\|}{O}}{C}\,C_{17}H_{35})_2 + Na_2SO_4 \quad (1)$$

3. 2 Spreadability of Sn-Pb eutectic solder with stearic acid on electropolished copper plate and on oxidized copper plate

As described in section 3.1, Cu-stearate was generated at (1), in **Fig. 2** in contact with O_2 in air. In order to clarify the source of copper stearate, weight loss of two kinds of Cu plates, electropolished and oxidized, was measured in molten stearic acid at 240°C. The results are shown in **Fig. 4.** From these results, it was recognized that stearic acid reacted with only copper oxide to give Cu-stearate (eq-2), but does not do so at all with pure Cu metal.

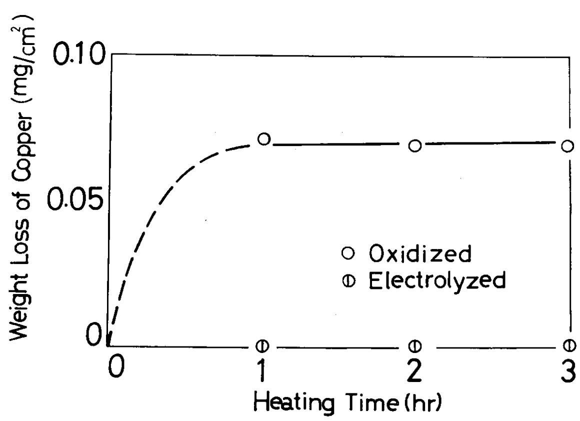

Fig. 4. Weight loss of copper plate immersed into molten stearic acid at 240°C.

$$CuO + 2C_{17}H_{35}COOH \xrightarrow[\text{heat}]{240^\circ C} Cu\,(O\overset{\underset{\|}{O}}{C}\,C_{17}H_{35})_2 + H_2O\uparrow \quad (2)$$

To examine the effect of copper stearate on spreadability, spreading tests were carried out on clean electropolished copper and on oxidized specimens using stearic acid flux. As shown in **Fig. 5,** the spreads on oxidized specimens were greater than those on clean specimens. This fact was coincide with that recognized by Bailey et al.[1] on $ZnCl_2$ - NH_4Cl system flux, however the reasons were not apparent. In our experiments, the reasons of improving spreading on oxidized copper in comparison with the results on

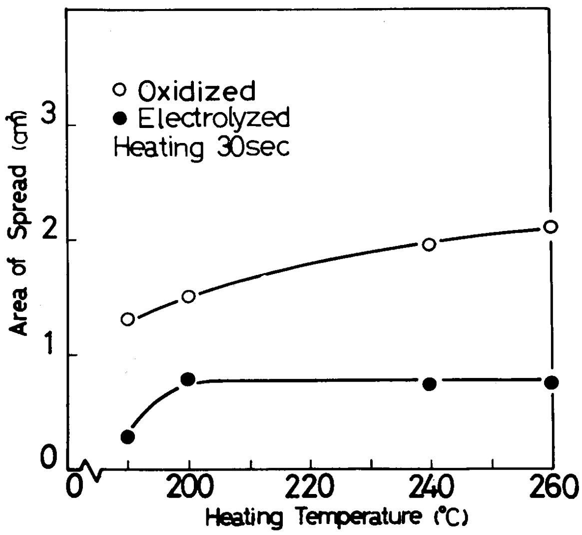

Fig. 5. Spread area of solder on electropolished and oxidized copper plate with stearic acid.

clean copper, may depend on copper stearate formed by reaction of stearic acid with copper oxide. So, we wish to report in next section the spreadability of solder when flux added Cu-stearate to stearic acid was used.

3.3 Spreadability of Sn-Pb eutectic solder with stearic acid/Cu-stearate system flux

As shown in **Fig. 6,** addition of a small portion of Cu-stearate to stearic acid has a great effect on improvement of spreadability of Sn-Pb eutectic solder on clean specimens.

The relation between spread area and heating temperature in the case of spread tests using stearic acid and Cu-stearate flux is shown in **Fig. 7.** As shown in **Fig. 7,** the spreadability of Sn-Pb eutectic solder under stearic acid is much inferior to that of solder under Cu-stearate flux and almost independent of heating temperature. In the case of the spread tests using 6 mol % copper stearate system flux, the spread phenomena were observed as follows; At the beginning of spreading test, the color of molten flux was pale green, however flux contacted with solder became transparent in color with the lapse of heating time. And it was recognized that the solder spreaded over the base metal.

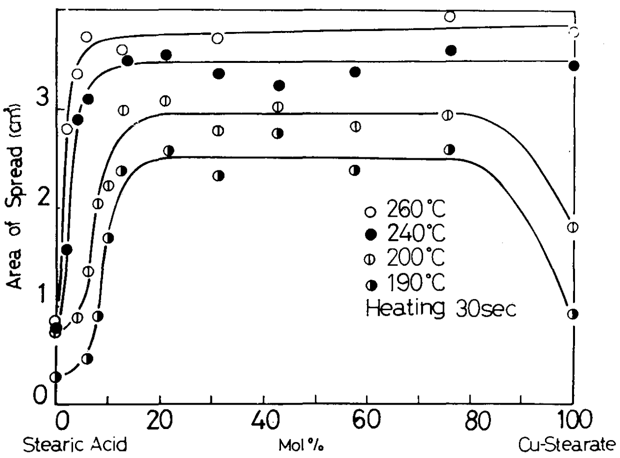

Fig. 6. Effect of flux composition on spread area of solder.

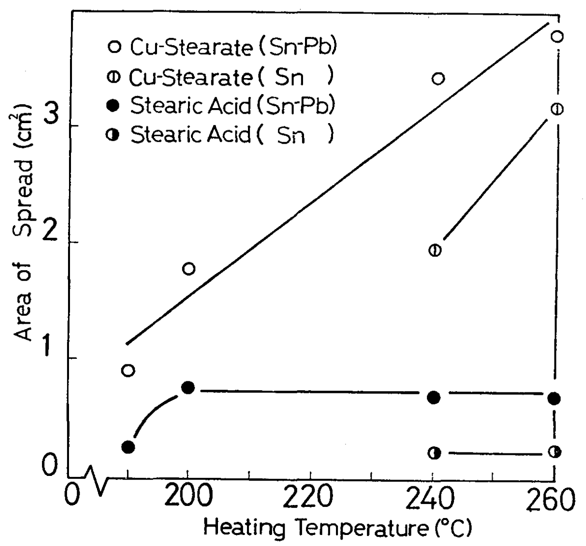

Fig. 7. Effect of temperature on solder spreading with various fluxes.

From above results, it is considered that spreadability of solder is mainly related to reaction of copper stearate with molten Sn-Pb eutectic solder.

3. 4 Reaction of Cu-stearate with molten Sn-Pb eutectic solder

From the phenomenon shown in section 3. 3, reaction of copper stearate with solder may play an important role in spreadability of solder. So, in this section, pyrolysis of copper stearate was studied in the presence of solder.

3. 4. 1. Pyrolysis of Cu-stearate

By the pyrolysis of Cu-stearate (3.20g) at 230°C for 3 hours, stearic acid (1.6g, yield 96%) was identified by IR spectra and metal Cu (80.8%) were given. (eq-3) 1.3g of Cu-stearate was recovered.

$$Cu\,(O\overset{\overset{O}{\|}}{C}\,C_{17}H_{35})_2 \xrightarrow[3\ hr]{230^\circ C} 2C_{17}H_{35}COOH + Metal\ Cu \quad (3)$$

Hydrogen of carbonyl group in stearic acid may be derived from the pyrolysis of copper stearate to form polymeric compound.

3. 4. 2. Pyrolysis of Cu-stearate in the presence of Sn-Pb solder

By the pyrolysis of Cu-stearate (3.80g) in the presence of Sn-Pb eutectic solder (6.00g), stearic acid (2.95g, yield 86.5 %), brown solid (0.7g) were isolated. (eq-4) The solid was analyzed chemically to contain Cu (2.58 %), Sn (47.28 %) and Pb (0.38 %).

$$Cu\,(O\overset{\overset{O}{\|}}{C}\,C_{17}H_{35})_2 + Sn\text{-}Pb\ alloy \xrightarrow[3\ hr]{215^\circ C} 2C_{17}H_{35}COOH$$

$$+ Cu\text{-}Sn\text{-}Pb \quad (4)$$

Cu-stearate was not recovered in difference with pyrolysis of in 3. 4. 1. From these results, it was clear that copper stearate reacted with solder and pyrolysis of copper stearate was accelerated by Sn-Pb solder. And metal copper deposited from Cu-stearate was recognized to dissolve in solder.

3. 4. 3 The effect of heating temperature on reaction of Cu-stearate with solder

After Cu-stearate (lg) and Sn-Pb solder (lg) reacted in test tube at required temperature for 30 sec, weight loss of Cu-stearate was measured. The results were shown in **Fig. 8.**

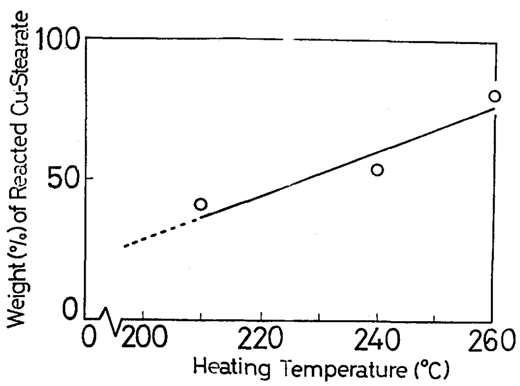

Fig. 8. Effect of temperature on decomposition of Cu-stearate.

The relation between spread area and heating temperature in **Fig. 7,** when copper stearate flux was used, reconciles almost with the results from decomposition of Cu-stearate in **Fig. 8.**

3. 5 EMX analysis of cross section of specimens after speading test

To understand well the reasons of differences in spreadability in the case of stearic acid flux and Cu-stearate flux, the cross section of two speci.nens after spreading, using two kinds of flux, was analyzed by EMX analyzer. **Figure 9** shows the concentration distribution curve obtained by the EMX analyzer of the cross section of specimens in the case of spreading using Cu-stearate flux. **Figure 10** illustrates the concentration distribution curve in the case using stearic acid. The left side of concentration curves in **Fig. 9** and **Fig. 10,** shows surface layers of solder and the right side shows boundary layer between base metal and solder. From **Fig. 9,** it was recognized that metal Cu deposited by reaction of copper stearate with solder dissolved into Sn-Pb eutectic solder when copper stearate flux was used. However, such a phenomenon was not recognized in the case of stearic acid flux as shown in **Fig. 10.** From these results, it is considered that Cu deposited by reaction of copper stearate with Sn-Pb solder has a effect on improvement of spreadability.

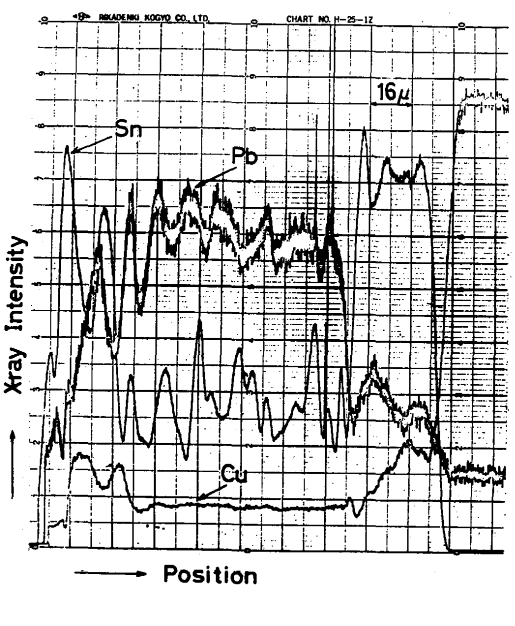

Fig. 9. Typical microprobe X-ray fluorescent scans through base plate and solder (Cu-stearate as flux).

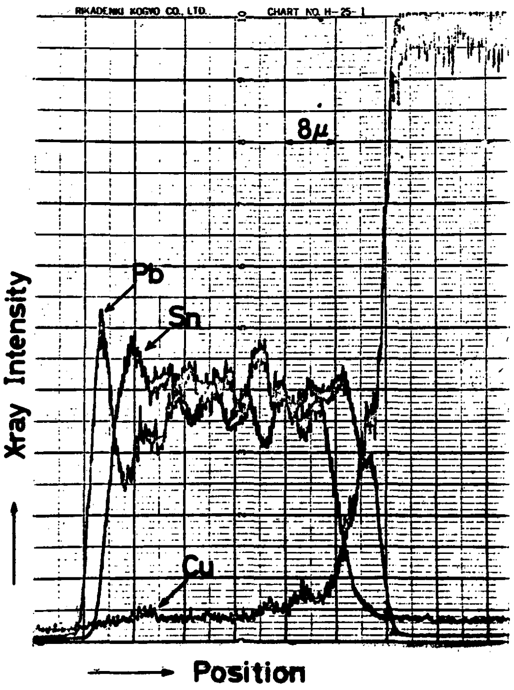

Fig. 10. Same as that indicated in Fig. 9 (Stearic acid as flux).

3. 6 The effect of heating time on spread area

The spreadability of Sn-Pb solder on copper plate may mainly depend on reaction time of Cu-stearate with Sn-Pb solder. When 94 mol % stearic acid-6 mol % Cu-stearate system flux was used at 190°C, the spread area was comparatively small, as shown in **Fig. 6.** The relation between spreads and heating time in the spread test at 190°C with the above flux is shown in **Fig. 11,** which contains other results of spread test with stearic acid flux or in stearic acid melt. In the case of 6 mol % Cu-stearate system flux, spread area is remarkably increased with time. But the case of spread test in stearic acid melt, spread area shows steady and small value. In the other case,

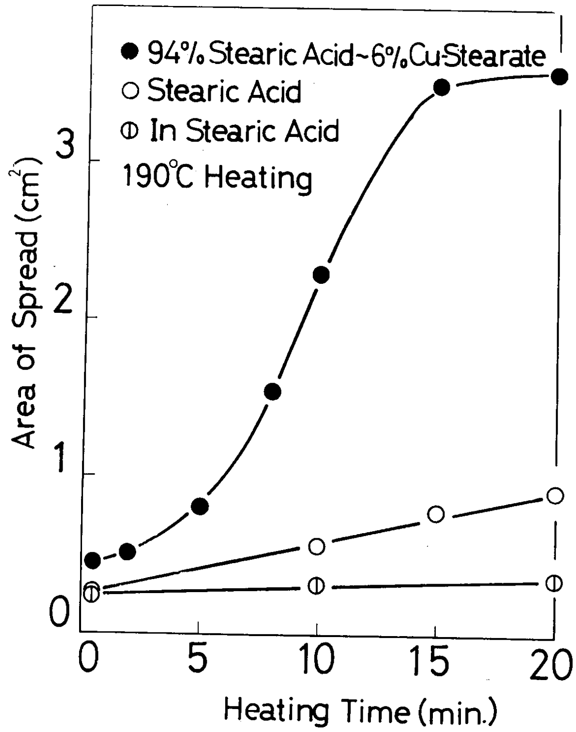

Fig. 11. Effect of time on solder spreading with various fluxes.

spread area is increased linearly. This increase of spread area may be due to preparation of Cu-stearate at the spread edge of molten stearic acid. The production was recognized by the fact which the color at the spread edge of molten stearic acid became pale green.

From these results, reaction time of Cu-stearate with solder may play an important role in spreadability of solder. Quantitative study on the relation between the reaction of Cu-stearate with Sn-Pb solder and the spread area using Cu-stearate flux is in progress.

Conclusion

In the case using stearic acid flux, spreadability of Sn-Pb eutectic alloy on oxidized copper is superior to that on electropolished specimens. Addition of a small portion of Cu-stearate to stearic acid has a great effect on improvement of spreadability of solder. Copper stearate reacted with solder to give stearic acid and metal Cu, which dissolved in solder. From these results, the flux action of stearic acid may be considered as follows; Stearic acid reacts with copper oxide to give copper stearate, which then reacts with solder and copper rich layer at outer surface of solder is formed. So, the spreading of solder on copper plate is improved.

Ackowledgment

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