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TESTING OF LEAD-FREE SOLDER PASTES FOR COMPONENT SOLDERING ON PRINTED AND HYBRID CIRCUITS

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Key words: electronics industry, printed circuits, hybrid circuits, electronic components, soldering, lead-free solder pastes, wettability, viscosity, solder balls, soldering flux residues, soldering technologies, testings, functional reliability

Abstract: The basic properties of the investigated lead-free solder pastes such, as viscosity, solder ball, wetting, were tested in laboratories for printed-circuit and hybrid-circuit technology development.The influence of flux residues on the reliable functioning of electronic circuits was tested by measuring the ionic contents in the solder pastes and by copper corrosion tests after humidity conditioning.

On the basis of the results from incoming tests on new, lead-free solder pastes the most suitable solder paste for component soldering on printed and hybrid circuits was selected for reliability testing in a humidity chamber.

Preizkusi pastoznih spajk brez svinca za spajkanje elektronskih komponent na tiskana in hibridna vezja

Ključne besede: industrija elektronike, vezja tiskana, vezja hibridna, deli sestavni elektronski, spajkanje, spajke pastozne brez svinca, omočljivost, viskoznost, kroglice spajk, ostanki fluksov spajkalnih, tehnologije spajkanja, preskušanja, zanesljivost delovanja

Izvleček: V laboratoriju za razvoj tiskanih in debeloplastnih hibridnih vezij smo izmerili osnovne lastnosti pastoznih spajk brez svinca. Opazovali smo pojav kroglic po pretaljevanju, omočljivost in izmerili viskoznost pastoznih spajk. Vpliv ostankov fluksa na zanesljivost delovanja elektronskih vezij smo ugotavljali z meritvijo vsebine ionskih ostankov v pastozni spajki in s pregledom pojave korozije na bakreni ploščici po staranju v vlagi. Na temelju rezultatov preizkusov, smo izbrali primerne pastozne spajke brez svinca za nadaljnje meritve zanesljivosti elektronskih vezij s pritrjenimi komponentami po staranju v vlažni komori.

1. Introduction

The worldwide electronics industry is faced with a ban on the use of lead, which is planned to come into force in January 2007. A number of lead-free alloys are currently being offered as replacements for use in hand soldering (solder wires), wave soldering (bars) and reflow soldering (solder pastes). Most of these alloys are based on a high tin content with the addition of various other elements (silver, copper, indium, bismuth antimony), which lead to higher melting temperatures than standard solder materials that contain lead. /1/, /2/

Some of these alloys offer advantages over conventional tin-lead solders, such as higher joint strengths, better fatigue resistance, improved high-temperature life times and harder solder joints. However, not all of these benefits are found with all the various lead-free alloys. Although many of the lead-free alloys have demonstrated more-than-adequate reliability, it is still necessary to carefully evaluate new solder pastes. /3/ In this paper we present the results of incoming tests on new, lead-free solder pastes for soldering miniature electronic components on hybrid and printed circuits. The solder pastes of various producers were compared after solder-ball, wetting, and copper corrosion tests and ionic content measurements in flux residues after reflow soldering. Also, viscosity measurements on fresh samples and on stored samples were compared for all the solder pastes tested. On the basis of the test results the best solder pastes were selected for a reliability test: a surface insulation resistance (SIR) test; and a test of the shear strength of the soldered joint between the component and the substrate before and after temperature cycling.

2. Experimental and results

2.1 Soldering paste tested

The main characteristics of the lead-free soldering pastes produced by Ecorel, Alpha Metals, Heraeus , Microbond, Multicore and Interflux, are presented in Table 1.

All the tested solder pastes are prepared for fine-pitch printing (small grain sizes) with no-clean fluxes.

The viscosities of the as-received solder pastes were measured, and the test samples for the ionic content measurement were printed with a 150 μm-thick stainless-steel stencil. During printing the samples were handled with gloves.

For the solder-ball testing, the wetting test and the copper corrosion test the samples were printed manually.

Solder paste	E808.3	OM 310	F817	NCD 8010-9	CR39	IF 9002 JAC
Alloy	Sn96 Ag4	Sn95.5 Ag 4 Cu0.5	Sn95.5 Ag4 Cu0.5	Sn95.2 Ag2.5Cu 0.8Sb0.5	Sn96.5 Ag3.5 Cu0.5	Sn95.5 Ag3.8 Cu0.7
Metal content [%]	-	89	89	-	-	88
Powder type	-	3	3	3	3	3
Powder size [μm]	-	25-45	325/500 mesh	325/500 mesh	325/500 mesh	25-45
Density [g/cm³]	-	4.4	4.7	-	-	4.3
Melting temp. [°C]	221	217	217-219	219	219	217
Peak of reflow temp. [°C]	240-250	235-240	232-245	232-240	232-245	232-245

Table 1: The main characteristics of lead-free soldering pastes

2.2 Viscosity measurement

The viscosities of the pastes were determined with a cone-and-plate Haake VT700 viscometer at the J.Stefan Insitute (IJS), Ljubljana. The following parameters were determined:

-Viscosity versus shear speed (Fig.1, Fig.2, Fig.3 and Fig.4), measured on solder pastes from various producers on both a fresh sample and a sample that had been stored in a refrigerator for 4 months.

Fig. 1 shows the viscosity of the F817 solder paste, Fig.2 is for CR39, Fig.3 is for NCD 8010-9 and Fig.4 is for IF9002 JAC

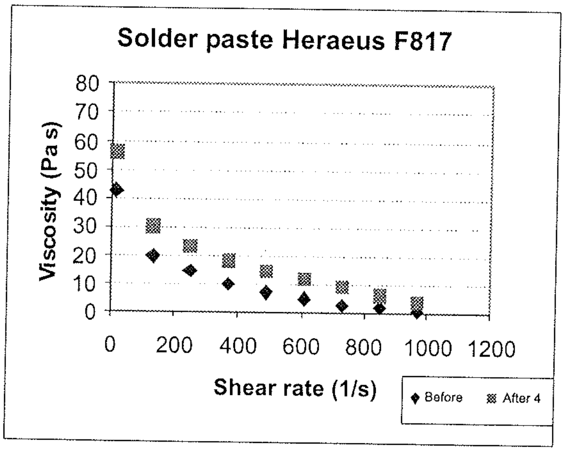

Fig. 1

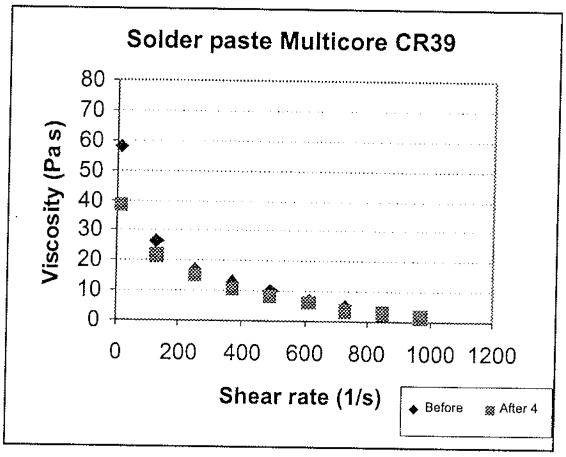

Fig. 2

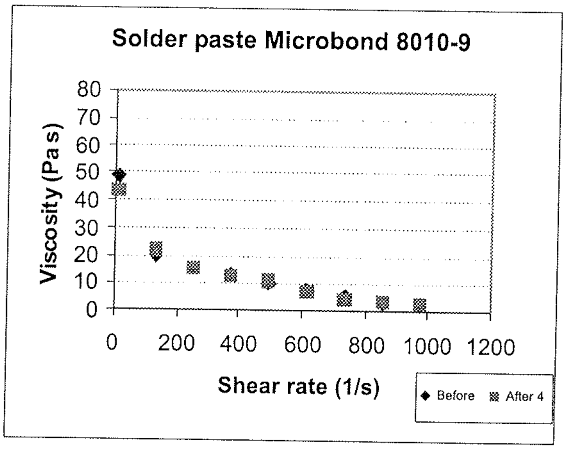

Fig. 3

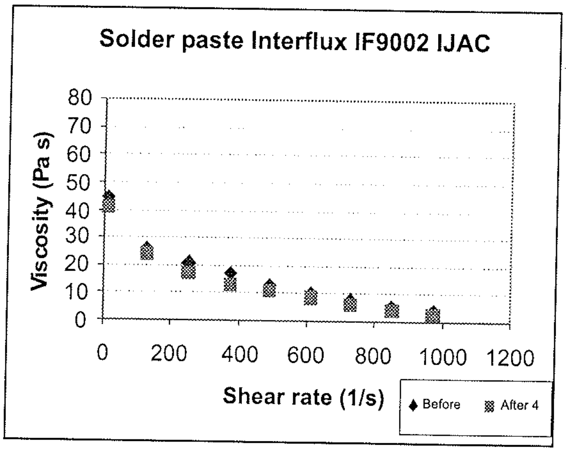

Fig. 4

2.3 Ionic contamination measurement

The ionic contamination measurement of the solder paste after soldering was made with a conductometer at Iskraemeco according to the MIL-P-28805A Standard.

The test samples were prepared at the IJS on 2x2-inch ceramic substrates using thick-film conductors soldered with following lead-free solder pastes:E 808.3, OM310, F817, NCD 8010-9, CR39 and IF 9002 JAC. The samples were not cleaned before the measurements and during preparation they were handled with gloves.

The results of ionic content measurements after reflow soldering of the samples on a hotplate at 240°C are given in Table 2.

Paste name	Ionic Content (μgNaCl/cm^2) Measured at Iskraemeco
E 808.3	0.2
OM 310	0.1
F817	0.25
NCD 8010-9	0.1
CR39	0.2
IF9002JAC	0.1

Table 2: Ionic contamination test results on a ceramic substrate for solder pastes printed using a fine-pitch stencil.

2.4 Solder-ball testing

The solder-ball test was used to determine the reflow properties of the solder pastes. These tests were carried out using ceramic substrates. The paste was deposited through a 0.2-mm-thick stainless-steel stencil with a 6-mm hole diameter. After 1 h of conditioning in the laboratory atmosphere the samples were soldered with a reflow peak temperature at 240 °C for 20 s. The samples were reflow soldered in three different furnaces: at the IJS, at Iskratel Electronics and at HIPOT-HYB.

The degree of coalescence was determined by comparing with the criteria given in the ANSI Standard for tin-lead solder paste /4/. According to the test criteria, when a paste forms a single sphere without small balls after soldering (criteria 1) or if in addition to one big sphere up to five small spheres are formed (criteria 2). Our test results are presented in Table 3 (according to test criteria /4/) and in Fig.6.

Fig.5 shows a reflow-soldering temperature profile in the production furnace at Iskratel Electronics.

The time-temperature reflow-soldering profile was selected according to the suggestions of the different producers of the tested solder pastes. The peak temperature was set at a minimum value of 235°C for the reflow soldering of both solder pastes with composition SnAg and SnAgCu. The selected profile was identical for all the tested solder pastes, however, for some solder pastes will have to be optimized later. The aim of the solder pastes testing on solder balls and wetting, after reflow soldering in different furnaces was, to observe the influence of the time-temperature profile on the test result.

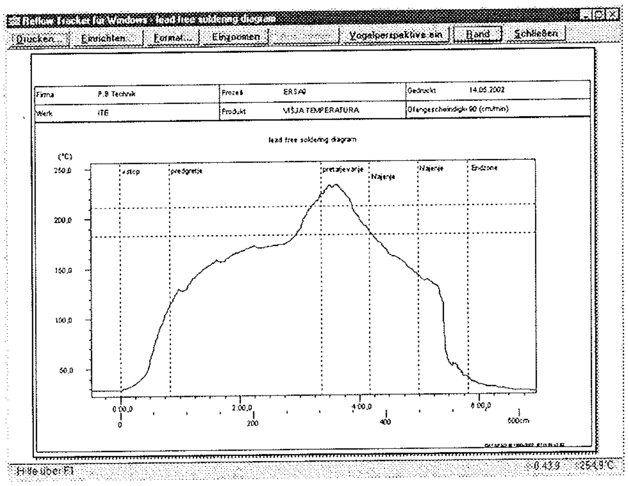

Fig.5: Reflow-soldering profile of the furnace at Iskratel Electronics for lead-free solder pastes (peak temperature higher than for SnPb solder pastes)

The similar reflow-soldering temperature profile was obtained in the production furnace at HIPOT-HYB, only the peak temperature was higher, 245°C in comparison with 235°C obtained in the furnace at Iskratel Electronics. The temperature profile for the small reflow-soldering furnace at the IJS is not shown, because this furnace has only a two-zone profile, and it was not possible to have a profile like in Fig.5, however, the peak temperature was 240°C for 20 s.

Solder paste	Reflow temperature profile IJS	Reflow temperature profile HIPOT	Reflow temperature Profile Iskra
	Criteria	Criteria	Criteria
E 808.3	A	A	A-B
OM 310	B	C	C
F 817	A-B	A	B
NCD 8010-9	A	B	B
CR39	A	B	B
IF9002 JAC	B	C	B-C

Table 3: The results of the solder-ball testing according to ANSI Standard (criteria A,B,C or D), tested at the IJS , HIPOT-HYB and Iskratel Electronics

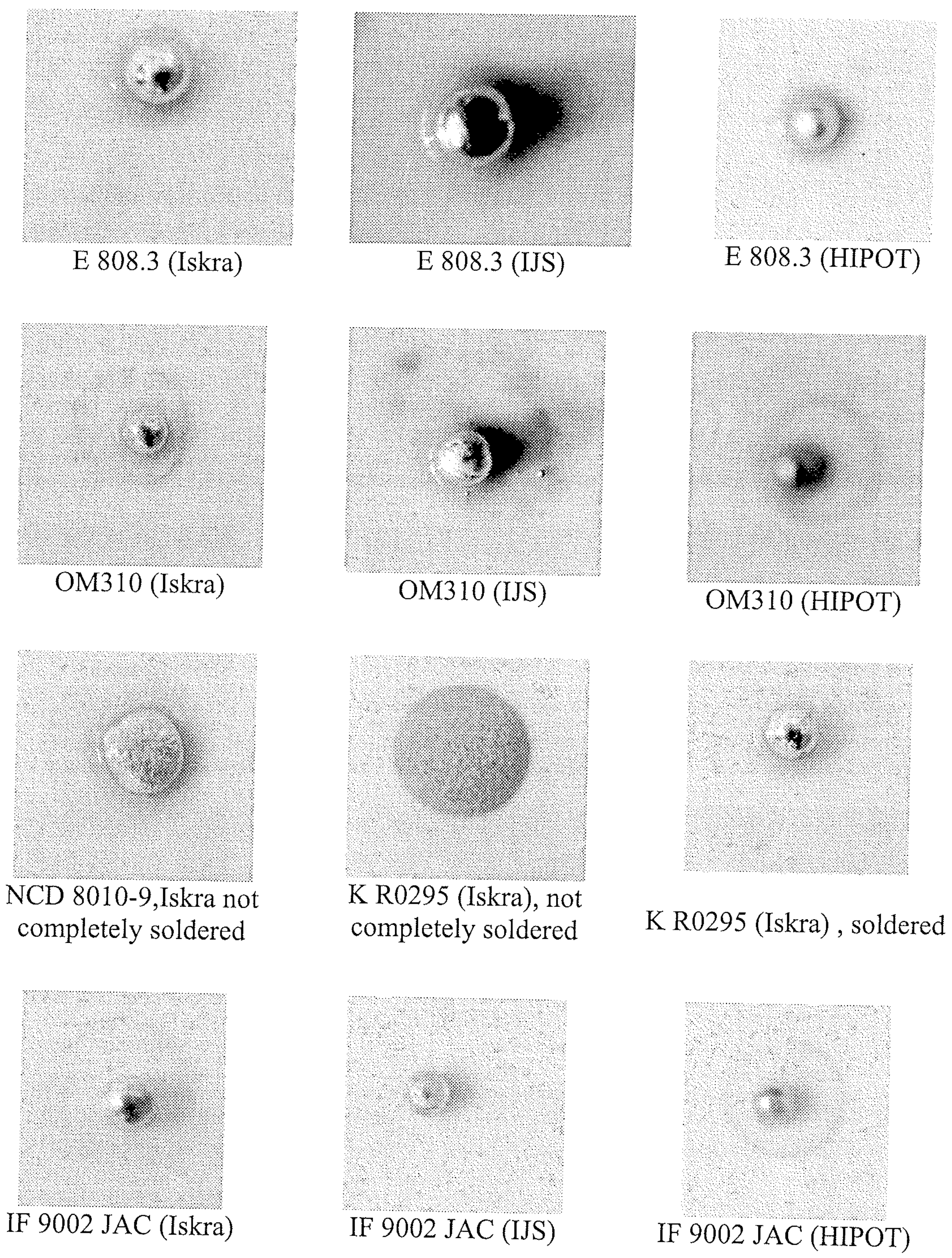

Fig.6: The results after solder-ball testing

Fig 6 shows the results of the solder-ball testing after reflow soldering of solder pastes E 808.3, OM310, NCD 8010-9 and IF 9002JAC. Also, the results of the solder-ball test for the solder paste K R0295, reflow soldered in the furnace at Iskratel Electronics are presented for various test samples.

2.5 Wetting test

The wetting properties of the solder paste were tested according to the ANSI Standard /5/. The paste was deposited by means of a stencil, on a cleaned, double-copper-clad laminate FR 4, with a thickness of 1.5 mm. Immediately after printing the paste was reflowed in the production furnace at Iskratel Electronics, in the production furnace at HIPOT-HYB and in the laboratory furnace at the IJS. The samples for the wetting test at the IJS were prepared on both copper-clad laminate and on the ceramic substrate with a AgPd thick-film conductor. The results are presented in Table 4 and in Fig.7.

Solder paste	Reflow-soldering profile Iskra	Reflow soldering profile HIPOT	Reflow-soldering profile IJS 240°C, 20s	
			Cu substrate	AgPd conductor
E 808.3	A-B	B-C	A	B
OM 310	C	C	B-C	B
F 817	B-C	C	B	A
NCD 8010-9	B-C	B-C	B	A
CR 39	B-C	C	B	A-B
IF9002 JAC	C	C	B	B

Table 4: The results of the wetting test according to ANSI Standard (criteria A, B,C or D) for samples prepared at Iskratel Electronics , HIPOT-HYB and the IJS

Fig.7 shows the results of the wetting tests when the solder pastes E 808.3, OM310 , NCD 8010-9 and IF 9002 JAC were reflow-soldered in the laboratory furnace at the IJS or in the production furnaces at Iskratel Electronics and HIPOT-HYB.

2.6 Copper corrosion test

The samples for the copper corrosion test were prepared on 0.05-mm-thick Cu foil according to ANSI Standard IPC-TM-650, 2.6.15. /6/ The foil was carefully cleaned before the solder paste deposition. A circular depression was formed in the foil, into the middle of which 1 g of solder for test was placed. The solder paste was reflowed on the hotplate at 240°C and placed vertically in a humidity chamber at 40°C, 93%RH for 10 days. After the exposure period the samples were examined at 20X magnification with

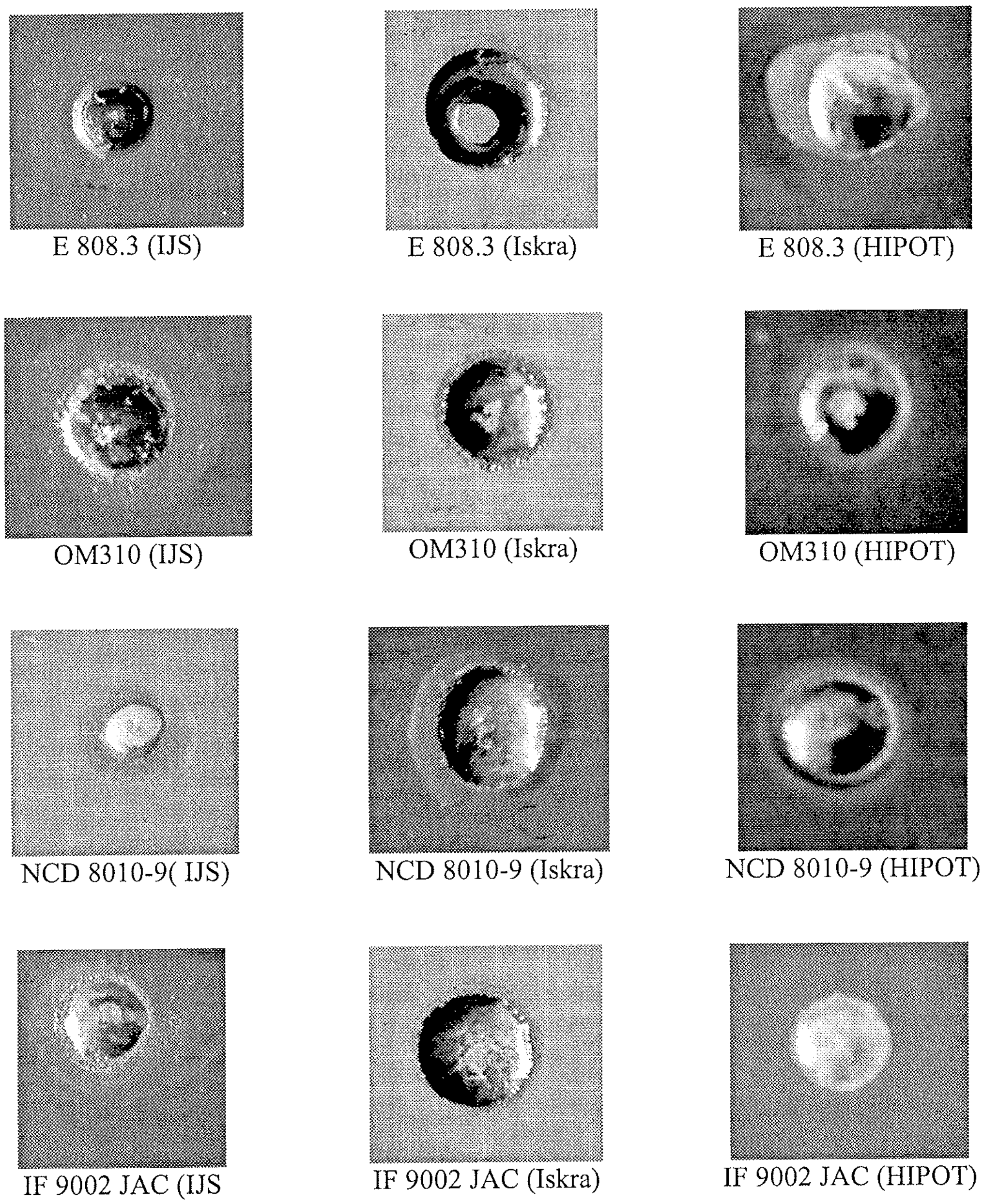

Fig.7: The results of wetting test

a microscope and the results for all the solder pastes are given in Table 5. Fig.9 are photographs taken after testing for the solder pastes E 808.3, IF9002 JAC, F817 and OM310.

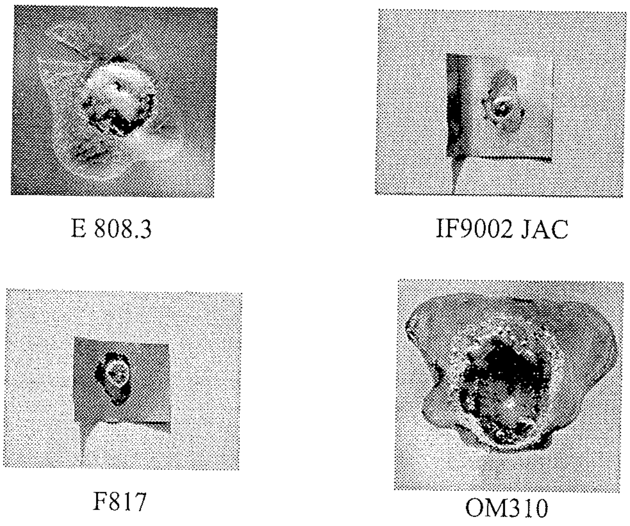

Fig.9: Results of copper corrosion test for solder pastes

Solder paste	Reflow-soldering profile IJS 240°C, 20s
	VISUAL ANALYSIS
E 808.3	No green residues
OM 310	Green residue
F 817	Olive green residue
NCD 8010-9	Olive green residue
CR 39	Olive green residue
IF9002 JAC	Green residues

Table 5: The results of copper corrosion test (10 days in humidity chamber at 40°C, 93%RH)

3. Discussion of results

The majority of the pastes measured with the Haake viscometer show a stable viscosity after 4 months of storage in the refrigerator. Only the solder pastes OM310, F369 and F817 show a slightly higher viscosity after storage. The solder paste IF 9002 JAC showed higher viscosity for the fresh sample than for the sample stored for 4 months in refrigerator for lower shear rates.

The results of the measurements of the ionic content in the flux residues after soldering (Table 1) show a very low ionic content from 0.1 to 0.25 μgNaCl/cm^2 for all the measured solder pastes, which is lower than allowed according to Standard MIL-P-28809A (1 μgNaCl/cm^2).

The results of the solder-ball testing presented in Fig.6 and Table 3 show, that all the investigated solder pastes passed this test (criteria A and B) except for the solder pastes IF 9002 JAC and OM310 (criteria C). The best result was obtained on samples with E 808.3 solder paste, with or without a very small number of small balls around the melted ball. In the samples with solder pastes IF 9002 JAC and OM310 around the melted ball were a large number of small balls, which is not acceptable.

No big influence on the test results was observed when the samples were reflow soldered in the three furnaces with somewhat different temperature-time profiles.

The biggest difference was obtained on samples with OM310 and IF9002 JAC solder pastes, when soldered in the furnace at IJS, the result was positive (B criteria) and when soldered in production furnaces at Iskratel Electronics and HIPOT-HYB, the result was negative (C criteria).

When the test samples were reflow-soldered in the production furnace at Iskratel Electronics, on some samples the solder paste was not completely melted, which means that the peak temperature of 235°C is not sufficient for soldering. In Fig.6 some examples of non-melted solder pastes (M 8010-9 and K R2095) are presented.

When the samples were reflow soldered in the production furnace at HIPOT-HYB, with a peak temperature 245°C, on all the samples the solder paste was melted.

The results of the wetting test given in Fig.7 and Table 4 for all the solder pastes tested show non-wetting (criteria B) or dewetting (criteria C), when reflow soldered on a Cu plate. When the solder pastes were tested on AgPd thick-film conductors, all the solder pastes showed better or similar results as tested on Cu printed circuit.

The test result of non-wetting or dewetting after reflow soldering in the furnaces was obtained when the samples were soldered in production furnaces. In some cases (solder pastes F817, NCD 8010-9 and CR39), when the samples were soldered in the laboratory furnace at the IJS, the solder pastes printed on the thick-film conductor completely wetted the AgPd surface.

A small difference in the test results was observed when the solder pastes CR39 and IF9002 IJAC were soldered in the Iskratel Electronics and the HIPOT-HYB production furnaces, but in both case the copper surface of the printed circuit substrate was not completely wetted.

The results of the copper corrosion test after conditioning in a humid atmosphere, given in Fig.9 and Table 5, show that the solder pastes OM310 and IF9002 IJAC have green residues. This means the flux residue after soldering contains some corrosive components.

The best results after the corrosion test was obtained for samples with solder paste E 808.3, which had no sign of corrosion. All the other solder pastes, after humidity testing, showed olive green residues, which is not a sign of a corrosive process.

4. Conclusion

All of the investigated pastes, measured with the Haake viscometer, demonstrate relatively stable viscosities after 4 months of storage in a refrigerator. This means that during the printing of 4-month-old solder paste the difference in the precision of dimensions of small printed pads for component soldering cannot be noticed.

The results of ionic content in flux residues after soldering show, for all the measured solder pastes, a very low ionic content from 0.1 to 0.25 $\mu gNaCl/cm^2$.

The results of copper corrosion test after conditioning in humid atmosphere show that solder pastes OM310 and IF9002 IJAC have green residues, which means the flux residue after soldering contains some corrosive components.

We did not observe a big difference in the results of the solder-ball and wetting tests after the samples were soldered in two production furnaces and in a laboratory furnace with not exactly recommended temperature profiles, but with recommended peak temperature from the producers of the solder pastes.

The peak temperature of 235°C is not sufficient for reflow soldering of solder pastes without lead.

From our results of the preliminary tests on lead-free solder pastes, it is clear that solder pastes tested do not wet the Cu-printed circuit surface well after melting. A little better wetting after soldering was observed when the solder pastes were printed on the AgPd thick-film conductor.

For the reliability testing of measurements the surface insulation resistance (SIR) between the soldered lines after humidity conditioning, will be selected the solder pastes E808.3, F817, NCD8010-9 and CR39, where the incoming test results were rather good, and also solder paste OM 310, because of the interest of the producer of this solder paste.

5. References

/1/ K.Seeling, D.Suraski; Lead-free soldering: An overview and comparison of viable solution, 2000 International Symposium on Microelectronics, Paris, June 2000, 15-20

/2/ T.Suga; Trend of lead-free soldering in Japan-NEDO Project 1999-2000, Conference Electronics goes green, Berlin, September 2000,149-154

/3/ G.Grossman: Lead-free soldering, a state of the art, Conference Electronics goes green, Berlin, September 2000

/4/ American National Standard J-STD-005, IPC-TM-650, 2.4.43,

/5/ American National Standard J-STD-005, IPC-TM-650, 2.4.45,

/6/ American National Standard J-STD-005, IPC-TM-650, 2.6.15

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