Solderability of Thick Film Substrate

Nickel plating, used as a conductor on thick film substrate, exhibits deteriorating solderability in high humidity and high temperature atmospheres

BY T. YOSHIYA AND A. TAMURA

ABSTRACT. Since the density of components on a thick film substrate has been increased greatly as a recent trend in electronic circuit module manufacturing, even a single connection defect can cause serious damage to the module. Checking the solderability of the thick film substrate before soldering is an important step. Observing the fritting voltage is an effective means of determining the solderability of thick film substrates.

This paper describes the results of an experiment on the effect of the thick film surface condition on solderability. These results can be applied as the standard in connecting components on the thick film substrate by soldering.

The following conclusions were drawn from fritting voltage measurement and solderability testing:

1. Measurement of the surface film breakdown voltage (i.e., the fritting voltage) is an effective means of evaluating the surface condition prior to soldering.

2. Nickel plating, employed as a conductor on the thick film substrate, is fairly stable in a dry atmosphere, but it corrodes markedly in a damp atmosphere and deteriorates in its solderability.

3. Oxidation of nickel plating in air proceeds rapidly when the temperature exceeds 200 C (392 F), and solderability is lost completely at temperatures above 300 C (572 F).

Introduction

Reliability of soldering depends on the stress in the soldered joint, vibration, and environmental conditions under which the soldered component is placed. The most important requirement for attaining high soldering reliability is that the solder should wet well in the initial stage of the soldering operation. This wetness, or solderability, depends upon such factors as the soldering conditions, chemical composition of the solder, type of flux, and surface condition of the metal to be soldered. These factors, in many instances, have a collective effect on the solderability. An electronic component that has excellent solderability after plating may deteriorate in solderability as time elapses under unfavorable storage conditions. Thus, the authors feel it is necessary to establish a method for evaluating the surface condition of components to be soldered.

This paper discusses evaluation of the surface condition and solderability of thick film substrates for high-density component packaging.

Table 1—Test Piece of Thick Film Substrate

Items	Specification
Materials of ceramics	90 ~ 92% aluminum oxide (Al_2O_3)
Conductor lines	Tungsten (W), 15 ~ 20 μm^t
Plating	Chemical nickel (Ni) plating, 2 ~ 5 μm^t
Surface roughness	R max. = 5 ~ 6 μm

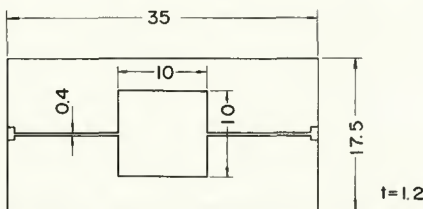

Fig. 1—Thick film substrate test piece (measurements shown in mm)

Test Pieces and Tests

Test Pieces—Thick Film Substrate

The thick film substrate subjected to the tests was such that the specified conductor line was thick film printed with tungsten (W) paste on a green sheet ceramic made from 90 to 92% aluminum oxide (Al_2O_3). The green sheet ceramic and tungsten paste were fired at high temperature in the range of 1600-1700 C (2912-3152 F), and then the conductor line was nickel-plated. The specification for the test piece is shown in Table 1, and its plane view in Fig. 1.

Soldering of a thick film substrate is basically the same as that of a printed circuit board. Since aluminum oxide ceramics have a higher thermal conductivity of 26.3 kcal/hour as compared with the 0.3 to 0.4 kcal/hour of ordinary printed circuit boards, they permit direct connections of IC's and LSI's with smaller conductor line width and pitches than those of printed wiring boards. Usually thick film substrates having a minimum line width of 150 to 200 μm are widely used.

The surface roughness of the thick film substrate is as large as 5 to 6 μm because the materials of both the green sheet ceramics and conductor were originally powders, and the conductor line was screen-printed.

Test Pieces—Solder

Pieces of solder preformed to 2 mm (0.079 in.) dia. and 1 mm (0.039 in.)

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Table 2—Chemical Composition and Weight of Solder Preforms

<table>
<tr><th rowspan="2">Solder preform</th><th colspan="12">Chemical composition, %</th><th rowspan="2">Weight, g</th></tr>
<tr><th>Sn</th><th>Pb</th><th>Sb</th><th>Cu</th><th>Bi</th><th>Zn</th><th>Fe</th><th>Al</th><th>As</th><th>Ag</th><th>Cd</th><th>Others</th></tr>
<tr><td>No. 1</td><td>62 ~ 64</td><td rowspan="2">Bal.</td><td rowspan="2">< 0.3</td><td rowspan="2">< 0.05</td><td rowspan="2">< 0.05</td><td rowspan="2">< 0.05</td><td rowspan="2">< 0.03</td><td rowspan="2">< 0.005</td><td rowspan="2"><0.03</td><td rowspan="2">—</td><td rowspan="2">—</td><td rowspan="2">—</td><td>0.022</td></tr>
<tr><td>No. 2</td><td>9 ~ 11</td><td>0.032</td></tr>
<tr><td>No. 3</td><td>9 ~ 11</td><td>Bal.</td><td>< 0.2</td><td>< 0.08</td><td>< 0.03</td><td>< 0.005</td><td>< 0.02</td><td>< 0.02</td><td>< 0.02</td><td>1.7 ~ 2.4</td><td>< 0.005</td><td>< 0.10</td><td>0.032</td></tr>
</table>

Table 3—Test Conditions

Items	Conditions
High temperature storage	125 C (257 F), 1000 h
High temperature, high humidity storage	45 C (113 F), 95% R.H., 1000 h
Oxidation in air	100, 200, 300, 400, 600 C (212, 392, 572, 752, 1112 F) for 30 min
Storage in air	Normal temperature and humidity at design room

thick were subjected to the solderability test. Three types of solder were used. Table 2 shows their chemical compositions and weights.

Solder No. 1, a eutectic solder with a melting point of 183 C (361 F), is most commonly used for soldering of printed circuit boards. Solders No. 2 and No. 3, with a comparatively high melting point (300 C/572 F), have been used for thick film substrates. Solder No. 3 contains approximately 2% silver (Ag). Generally, solder with a high melting point is used for thick film substrates which are subjected to high temperatures in the subsequent airtight sealing and packaging processes.

Accelerated Test

The accelerated test mentioned in Table 3 was conducted for the nickel (Ni) surface of the thick film substrate.

Fritting Voltage Measurement

Fritting voltage measurement was made on the test pieces that had undergone the accelerated test, using the device shown in Fig. 2. Then, the film formed on each test piece was evaluated.

The probe of the measuring apparatus was a 0.2 mm (0.008 in.) gold (Au) wire formed into a loop. The measurement was made by employing the same principle as the cross-rod method for contact resistance measurement. The voltage applied to the film formed on the test piece surface was increased from 0 to 20 volts, and the voltage at which the film was broken and sudden voltage drop was observed was measured as the fritting voltage. Figure 3 shows a representative V vs. I curve. The load applied to the probe was detected by a load cell.

To prevent the surface film from being mechanically broken, a load as light as 0.1 gram was applied to the test piece under measurement. The radius (α) of the contact area when a pressure of 0.1 gram is applied can be calculated approximately from the equation:

$$\alpha = 0.0761 \times 3\sqrt{\frac{P \times D}{E}} \qquad (1)$$

where: P = load; D = diameter of wire; and E = Young's modulus.

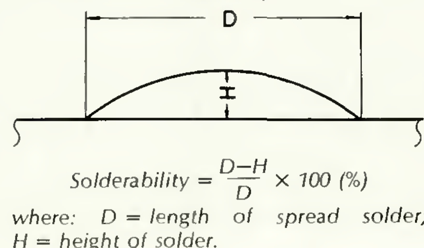
In case D = 400 μm, $E_{Au} = 7.8 \times 10^{10}$ N/m^2, then $\alpha = 6.1 \times 10^{-5}$ (mm). The contact load of 0.1 gram converted into the stress will be 1.64×10^6 kg/m^2. The fritting voltage was measured on 50 test pieces and expressed in the graphs by using cumulative frequency curves.

Evaluation of Solderability

In parallel with the fritting voltage evaluation, the solderability of test pieces stored for different lengths of time was evaluated. The solder preforms listed in Table 2 were placed in the center of the test piece, melted by heating in a conveyor furnace, and the solderability was evaluated by the solder spreading area. The melting temperature was set higher than the melting point of solder by 40 to 60 C (104-140 F).

The solder preform No. 1 was melted at 240 C (464 F) for 3 minutes, and No. 2 and No. 3 were melted at 330 C (626 F) for 3 minutes. Benzyl alcohol solution of 25% rosin was used as the flux, since the regular flux would be scorched at a temperature approximately 100 C (180 F) higher than that applied to the commonly used eutectic solder.

Evaluation of solderability:



Solderability $= \frac{D-H}{D} \times 100$ *(%)*

where: D = length of spread solder; H = height of solder.

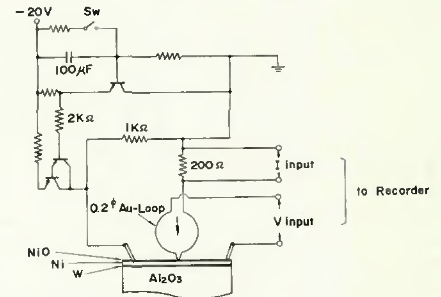


Fig. 2—Schematic diagram of fritting voltage measurement

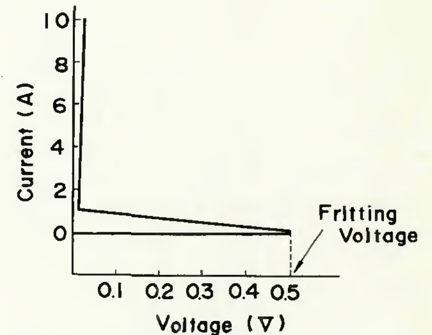


Fig. 3—Fritting voltage diagram

The solderability was measured by means of a pick-up type shape measuring device. It was calculated from equation (2) after measuring the length (D) of spread solder and the height (H) of solder.*

$$\text{Solderability} = \frac{D-H}{D} \times 100\,(\%) \qquad (2)$$

Test Results and Discussion

Results of Fritting Voltage Measurement

Figure 4 shows the cumulative frequency of fritting voltage measured on the test pieces which were oxidized in the air. Fritting voltage variation is scarcely observed with the test pieces oxidized at 100 C (212 F), but sudden rise of voltage is seen with those oxidized at 200 C (392 F) and higher temperatures. No surface film breakdown was observed with the test pieces oxidized at 600 C (1112 F) through voltage application of up to 20 V.

Visual check of the test piece surface revealed that no significant

change was observed with test pieces oxidized at temperatures up to 200 C (392 F). The surface of test pieces oxidized at 300 C (572 F) was discolored into very pale brown, while that of 400 C (752 F) oxidized pieces was deeper brown, and that of 600 C (1112 F) oxidized pieces was the deep green of nickel oxide (NiO).

Cumulative frequency of the fritting voltage measured on the test pieces kept in the ambient atmosphere, in test chambers at 125 C (257 F), and at 45 C (113 F), 95% R.H., with the storage time being used as a parameter, is shown in Fig. 5 (a), (b) and (c).

As seen from the graphs, the fritting voltage scarcely varied on the test pieces exposed to the ambient atmosphere for up to 1000 hours. This means that the nickel-plated surface is comparatively stable in the air.

In the high temperature (125 C/257 F) storage test, the fritting voltage gradually increased as the time elapsed, indicating gradual surface oxidization. No conspicuous difference was observed visually between the nickel plated surfaces of test pieces in the air exposure test and the high temperature (125 C/257 F) storage test for a period of up to 1000 hours.

The fritting voltage increased rapidly with the lapse of time on the test pieces that were stored at a high temperature (45 C/113 F) and high humidity (95% R.H.), indicating the progress of nickel plated surface corrosion. Visual check of the test pieces revealed that stain-like speckles were produced locally on the nickel plated surface when the test period exceeded 500 hours. This corrosion mechanism was different from the oxidation of the test pieces exposed to the atmosphere or stored at the high temperature of 125 C (257 F). It is believed to have been caused by the local cell phenomenon resulting from uneven internal strain distribution and existence of impurities in the plated layer. Structural analysis of the surface film is not discussed in this paper.

Let us return to the test pieces oxidized in the ambient atmosphere. In general, the rule of parabolic curve applies to the thickness (d) of oxidized film versus time (t); thus, $d^2 = kt$. The constant k, together with the temperature, follows the Arrhenius equation.

Utilizing equation (3) established by Dr. R. Holm for determining the relationship between the oxidation temperature, time and oxidized film thickness, the authors calculated the nickel oxide (NiO) film thickness and examined its relationship with the fritting voltage.

$$d^2_{NiO} = 5^2 + t \times 10^{13.3 - \frac{6000}{P}} (\text{Å})^2 \quad (3)$$

where, T = absolute temperature (K).

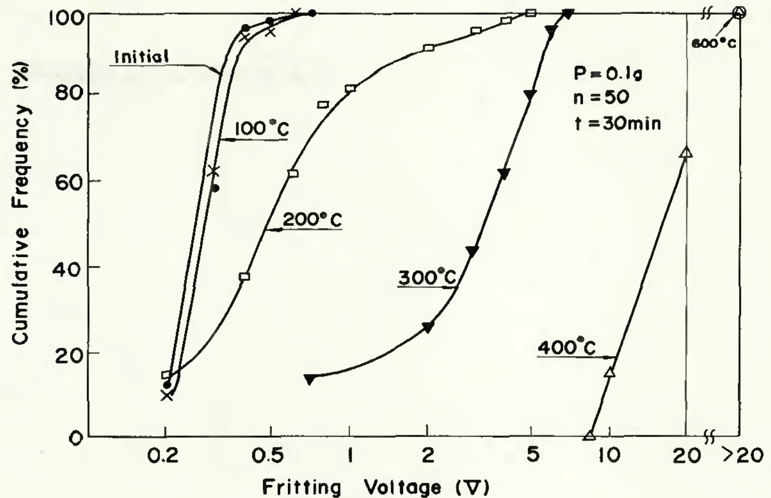


Fig. 4—Fritting voltage variations and oxidation in the air

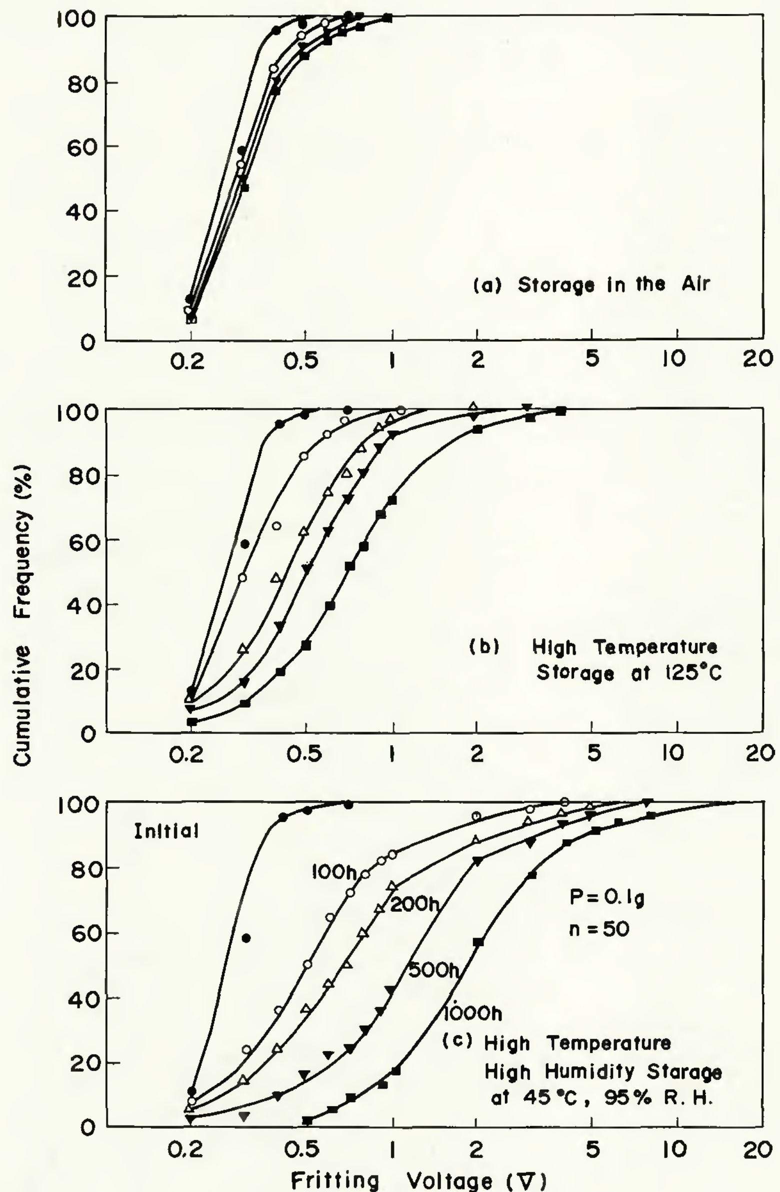


Fig. 5—Fritting voltage variations in various environmental tests

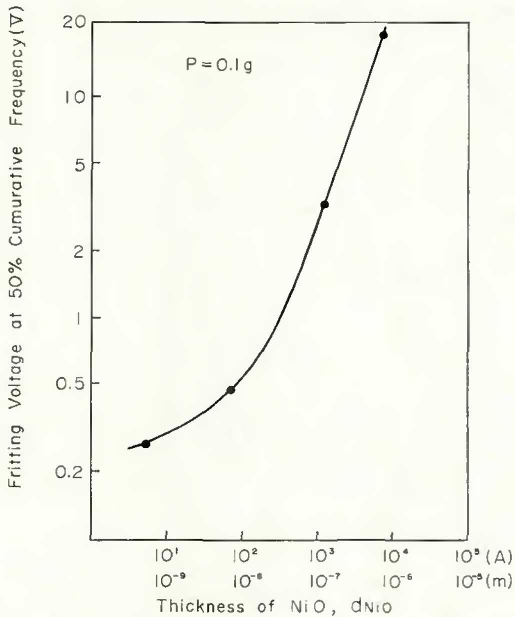


Fig. 6—Fritting voltage at 50% cumulative frequency vs. thickness of nickel oxide (NiO)

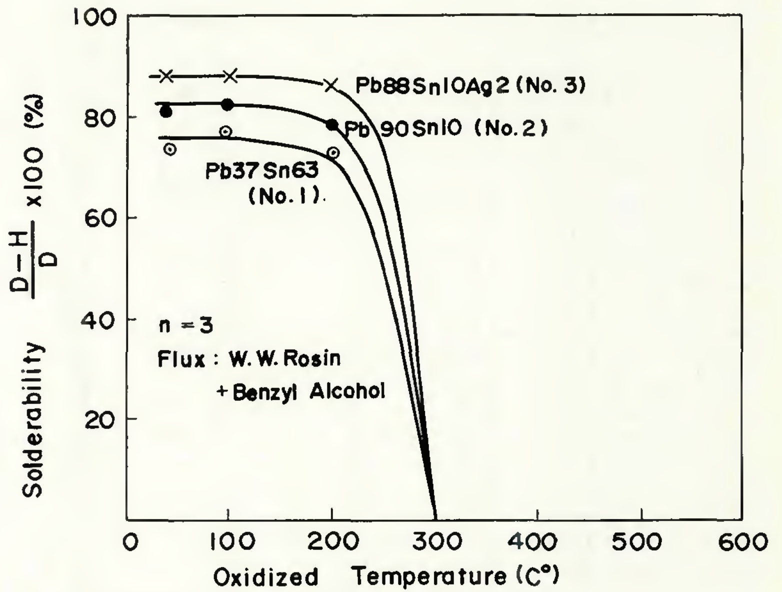


Fig. 7—Effects of oxidation temperature on solderability

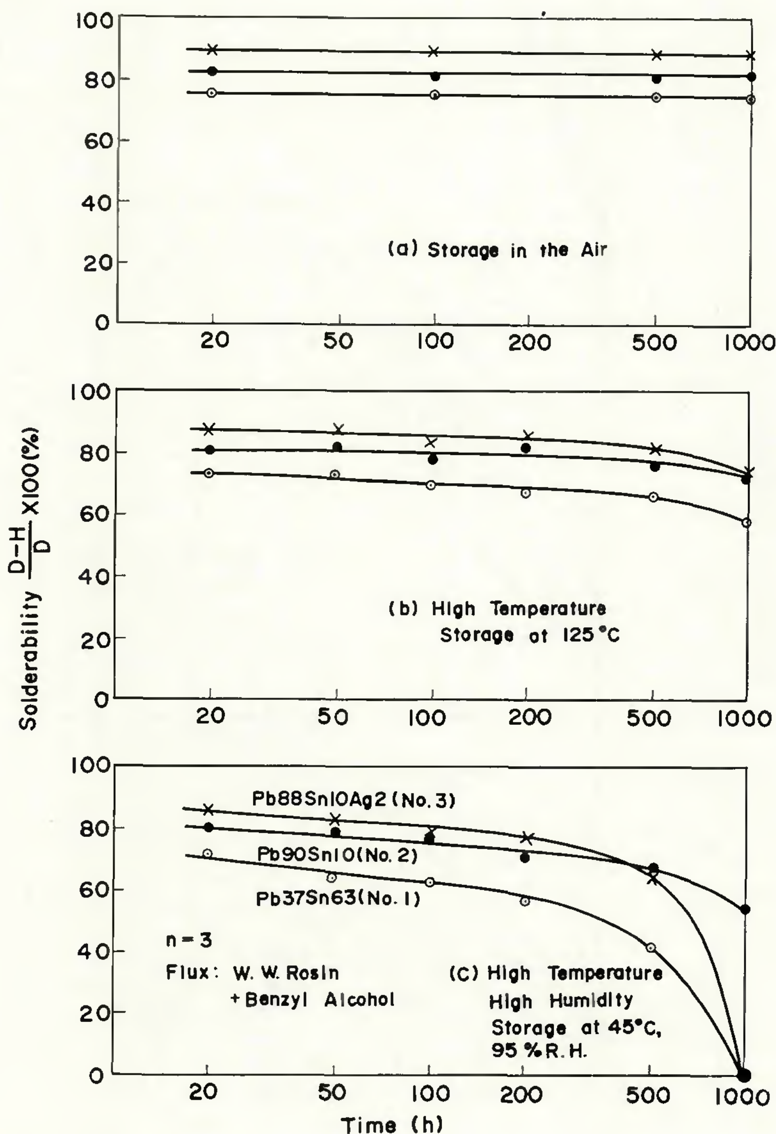


Fig. 8—Solderability variations in various environmental tests: a (top)—storage in the air; b (center)—high temperature storage at 125 C (257 F); c (bottom)—high temperature, high humidity storage at 45 C (113 F), 95% R. H.

Figure 6 illustrates the results. The nickel oxide (NiO) film thickness is shown along the horizontal axis, and the fritting voltage values are shown at 50% cumulative frequency of Fig. 4 along the vertical axis. It can be seen from Fig. 6 that the fritting voltage has a nearly linear relationship with the nickel oxide film thickness in the range where the thickness exceeds a few hundred angstroms (Å). The electric field strength at the time-oxidized film breakdown is calculated as 6×10^8 Vm^{-1} and 3×10^8 Vm^{-1} for the film thicknesses of 10^2 and 10^3 Å, respectively.

Results of Solderability Measurement

There are various factors that affect solderability, and a change in any of them will produce a different result. The nickel plated surface condition and solderability of the thick film substrate are discussed here, referring only to the factors considered in the preliminary test.

Figure 7 shows the results of solderability testing on the test pieces oxidized in the ambient atmosphere. All solders showed gradual deterioration in solderability for oxidation temperatures up to 200 C (392 F). When oxidized at a temperature above 300 C (572 F), the solder was formed in a spherical shape and showed no solderability at all.

The results of solderability testing on the test pieces, which were exposed to the ambient atmosphere,

stored in a test chamber at a high temperature (125 C/257 F), and at a high temperature (45 C/113 F) high humidity (95% R.H.), are shown in Fig. 8 (a), (b) and (c), respectively. As in the case of the fritting voltage, deterioration in solderability was not observed with the test pieces exposed to the ambient atmosphere for 1000 hours. On the test pieces stored under the high temperature condition (125 C/ 257 F), the solderability started to decrease at 200 hours, but the amount of variation was within 20% of the initial value after 1000 hours.

All three types of solder showed rapid deterioration in their solderability on the test pieces stored under high-temperature and high-humidity conditions (45 C/113 F, 95% R.H.). In particular, the solder preforms No. 1 (Pb37, Sn63) and No. 3 (Pb88, Sn10, Ag2) presented sudden deterioration in their solderability when the exposure time exceeded 200 hours. These types of solder were formed in a spherical shape, showing no solderability, when applied to the test pieces stored for 1000 hours.

Figure 9 summarizes the results of fritting voltage and solderability obtained with the test pieces placed under various storage conditions. The fritting voltage values at 50% cumulative frequency are plotted along the horizontal axis. It is seen from the graph that the solderability of any type of solder decreases almost linearly with the increase of the fritting voltage up to 1 V, decreases rapidly when the voltage exceeds 1 V, and is lost completely when the voltage is about 2 V.

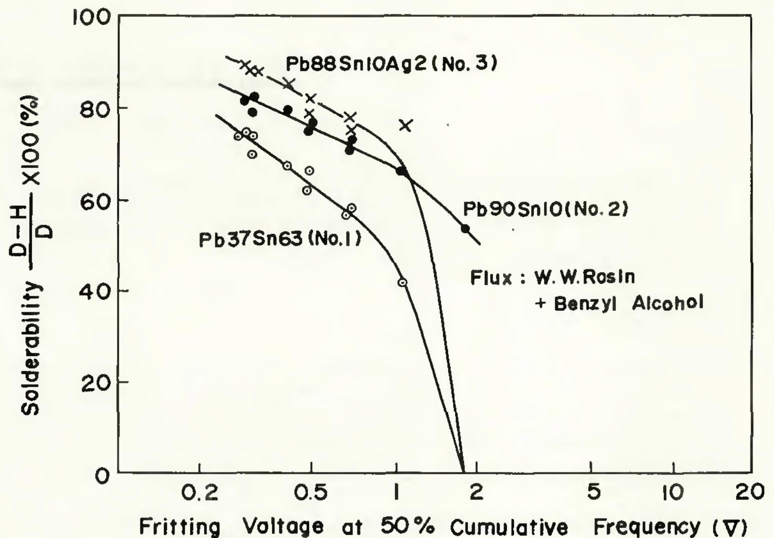


Fig. 9—Relationship between fritting voltage and solderability

Conclusions

The following conclusions can be drawn from the fritting voltage measurement and the solderability test:

1. Measurement of the surface film breakdown voltage (i.e., the fritting voltage) is an effective means of evaluating the surface condition prior to soldering.
2. Nickel plating, employed as conductor lines on thick-film substrates, is fairly stable in a dry atmosphere, but it corrodes markedly in a damp atmosphere and deteriorates in its solderability.
3. Oxidation of nickel plating in air proceeds rapidly when the temperature exceeds 200 C (392 F), and the solderability is lost completely at temperatures above 300 C (572 F).

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September 1977

1. An Experimental Study of Elasto-Plastic Response of Branch Pipe Tee Connections Subjected to Internal Pressure, External Couples and Combined Loadings

by Fernand Ellyin

Experimental results of nine branch-pipe tee connections and two plain pipes are reported. The models were exposed to individual loadings: internal pressure and external couples and their combination. The loads were applied well beyond the elastic limit, and the elasto-plastic response of the models is discussed. Experimental limit or yield-point load of each model is determined, and the interaction curves are plotted. All models showed considerable reserve capacity beyond the experimentally determined limit loads.

2. Collapse Test of a Thin-Walled Cylindrical Pressure Vessel with Radially Attached Nozzle

by R.L. Maxwell and R.W. Holland

This report deals with the collapse test of a thin-walled cylindrical pressure vessel with a nonreinforced circular radially-attached nozzle. The vessel had previously been tested under elastic conditions at the Illinois Institute of Technology Research Institute. The work reported here was performed as a part of the Oak Ridge National Laboratory program on experimental and analytical investigations of the structural behaviors of nozzle-to-shell attachments, which is a part of the overall U.S. Atomic Energy Commission nuclear safety effort.

Publication of this paper was sponsored by the Pressure Vessel Research Committee of the Welding Research Council.

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WRC Bulletin 221
November 1976

Analysis of Test Data on PVRC Specification No. 3, Ultrasonic Examination of Forgings, Revision I and II

by R. A. Buchanan

The purpose of this report is to review, analyze and draw conclusions from data developed during efforts to test the validity of the procedures allowed by PVRC Specification No. 3, Revisions I and II. The author states that the only way to reduce the variation in test data from one group to another would be to restrict the procedures of PVRC Specification No. 3 so that only certain equipment combinations or, if practical, only one combination could be used.

Analysis of the Nondestructive Examination of PVRC Plate-Weld Specimen 251J—Part A

by R. A. Buchanan

During the fabrication of PVRC plate-weld specimen 251J, 15 welding defects were deliberately introduced. After fabrication, the specimen was ultrasonically examined for welding defects by a number of different company teams. The teams conducted their tests independently and had no knowledge of the types or locations of the intentional defects. Two different PVRC procedures for ultrasonic examination were followed by the teams in their evaluation of the specimen.

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