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Thermal degradation of rosin during high temperature solder reflow

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Abstract

Rosin chemical changes induced by heat and air during solder reflow were studied by thermal analysis, liquid chromatography and FT-IR. It was shown that in addition to isomerization, the rosin undergoes significant dimerization in an inert atmosphere. The dimerization mechanism does not require the presence of oxygen. During air reflow, additional auto-oxidative polymerization reactions occur through hydroperoxide intermediates. The auto-oxidative polymerization is largely a surface phenomenon and is strongly dependent on the thickness of the applied rosin film.

INTRODUCTION

Lead-rich solders require considerably higher reflow temperatures (about 300°C) than eutectic solder alloys (63% Sn–37% Pb). The reflow process is generally performed in the presence of a soldering flux whose function is to promote solder wetting and enable the formation of proper soldered joints. Rosin is the principal ingredient in many of the commonly used soldering fluxes. Rosin is known to decompose in air at elevated temperatures and so high temperature solders, if used in conjunction with rosin, need to be reflowed under a nitrogen atmosphere. Unfortunately, even under inert atmospheric conditions, the rosin may undergo thermally induced chemical reactions which may be further complicated by the presence of low levels of oxygen impurity in the nitrogen atmosphere.

Isomerization of rosin is currently believed to be the predominant reaction taking place during conventional solder reflow [1–3]. Secondary reactions are perhaps polymerization, in addition to interactions with solder to form tin and lead abietate salts [2–7].

The goal of this study was to elucidate the rosin chemical changes induced by heat and air. Thermally induced chemical reactions of rosin were investigated and monitored as a function of rosin concentration,

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reflow temperature and reflow atmosphere (air vs. nitrogen). A series of analytical techniques were used, such as thermal analysis (TGA), liquid chromatography (HPLC and SEC) and FT-IR. To isolate thermal effects from potential chemical interactions with solder, substrates were reflowed both in the presence and absence of solder.

EXPERIMENTAL

The sample preparation consisted of immersing either gold patterned ceramic substrates or bare silicon wafers into 10% and 40% by weight rosin solutions in alcohol. Tall oil in addition to water white gum rosin (supplied by Alpha Metals) were used in most studies. The differences between the two fluxes were in most cases negligible, (especially in the context of our objective), so the discussion here is limited to the tall oil rosin, mainly because of its claimed higher temperature stability. After rosin coating, the substrates were suspended vertically for five minutes to remove any excess rosin. They were then reflowed using two temperature profiles: peak temperature of 220°C to simulate eutectic solder reflow, and peak temperature of 310°C to reflow 95/5 lead-rich solder. 220°C and 310°C were the temperatures, as measured directly on a calibration substrate with a thermocouple. The inert atmosphere reflow was performed on a BTU Int. T.R.S. turbo reflow solder furnace, at a conveyor speed of 29 in min^{-1} at 200°C and 18 in min^{-1} at 310°C, with the capability of bleeding in air to increase the oxygen content from 15 ppm (minimum achievable) to $\approx$ 100 000 ppm. The conventional air reflow was performed on a Vitronics 700 series IR oven, at a conveyor speed of 18 in min^{-1}. Special care was taken to insure that the temperature profiles of the two reflow ovens matched as closely as possible.

HPLC and SEC analysis studies were performed on a Waters 840 system with a Waters 490E programmable multiwavelength detector. The column used for HPLC was a 4.6 mm $\times$ 25 cm reverse phase octadecylsilane with a mobile phase of 90% methanol and 10% water at a flow rate of 1.5 ml min^{-1}. A set of three microstyragel columns of pore sizes ranging from 100 to 1000 Å were used for the SEC studies. The columns were always maintained at room temperature and the detection wavelengths ranged from 210 nm to 285 nm, as described in the text. The extraction procedure consisted of placing the substrates in isopropyl alcohol in an ultrasonic for 30 minutes, followed by evaporation down on a hot plate to a preset volume and filtering. For the SEC studies, the isopropyl alcohol was evaporated from the extract solutions and replaced with THF (THF is the optimum solvent for SEC). In all cases, surface analysis was performed on the extracted substrates to insure complete extraction by isopropyl alcohol. Typically, injection volumes of 125–250 μl were used.

FT-IR spectra were measured in the transmission mode using a Mattson POLARIS spectrometer in the mid-IR range (4000–650 cm^{-1}) with a liquid N_2 cooled Hg–Cd–Te (MCT) detector. Typically 100 scans at 4 cm^{-1} resolution were required for the analysis of liquid extracts, which required 3–5 minutes of measurement time. In order to look directly at surface phenomena, bare Si wafers were coated with rosin and reflowed, and the changes were studied by IR transmission. Ceramic substrates are not suitable for IR transmission because they are IR opaque. Transmission IR is attractive because other IR analysis techniques (such as ATR, diffuse reflectance, etc.) all have significant limitations, primarily arising from poor signal to noise ratio.

The UV analysis was performed on a Varian DMS 100S UV/Vis spectrometer. The samples for UV analysis were diluted using isopropyl alcohol, which was also used as a reference. TGA studies were performed on a Du Pont system.

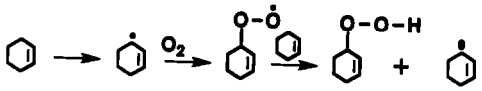
ROSIN FLUX

Pine tree stump and wood pulp extracts are steam distilled to separate the rosin from a steam volatile fraction consisting of fatty acids (C-18) or turpentine oils. Because of incomplete separation, the rosin fraction may contain 1–3% of the fatty acid component. The resulting rosin is in turn composed of 90% diterpene acids (C-20) and 10% of a neutral component consisting of one third volatile species in the form of diterpenes and monoether alcohols, and two thirds non-volatile species such as resin acid esters and various terpene oligomers. The neutral 10% component is believed to contain up to 100 different chemical species. However, all efforts to isolate them have met with little success [8,9].

The diterpene acids consist of a series of structural isomers which can be classified as conjugated acids (abietic, neoabietic, levopimaric), and non-conjugated acids (pimaric, isopimaric). Dehydroabietic acid is strictly not an isomer of abietic acid. However, the conversion of abietic acid to dehydroabietic acid occurs as easily as the isomerization reaction, so we include dehydroabietic acid "loosely" as an isomer of abietic acid in the remainder of the discussion.

Determination of the relative proportion of each of these isomers in a rosin mixture is difficult because of the ease with which isomerization occurs. Most of the acid isomers are sensitive not only to heat, but also to air and light [8,9]. For instance, abietic acid is extremely sensitive to air oxidation and becomes discolored (yellow) on air exposure. At higher temperatures, it suffers disproportionation to give mixtures of dehydroabietic acid and di- and tetra-hydroabietic acids. In contrast, dehydroabietic acid exhibits the highest oxidative stability of all the isomers [8]. This is not surprising if one considers the oxidation reaction mechanism. The oxida-

tion reaction (reaction 1) is known to proceed through a three stage free radical chain reaction involving formation of hydroperoxides [10].



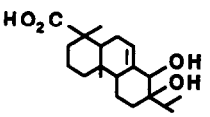
(1)

The reaction rate is governed by the C–H bond strength and thus allylic hydrogen atoms (H–C–C=C) are more susceptible to oxidation owing to the resonance stabilization of the resulting radical [11,12]. Comparison of the structures of the various abietic acid isomers clearly shows that dehydroabietic acid, followed by isopimaric acid, is most stable to oxidation, and abietic and levopimaric acids, followed by neoabietic acid, are least stable to oxidation. The increased stability of dehydroabietic acid is also due to its aromaticity.

The chain termination reaction produces [10]

$$2\ RO_2^* \rightarrow O_2 + RO_2R \text{ (or alcohols or carbonyl compounds)}$$

The hydroperoxides are known to be unstable and decompose readily in the presence of water and acids to produce a variety of products. The first relatively stable oxidation product of abietic acid is most probably a glycol [9,10], shown in Structure 1. At elevated temperatures, further oxidative polymerization reactions occur, which will be discussed in greater detail in the following section.



Structure 1.

THERMALLY INDUCED REACTIONS OF SOLDER

Weight loss and TGA studies

Possibly the most obvious thermally induced change taking place during reflow is the volatilization of rosin, together with the evaporation of the alcohol based solvent. The rate of rosin volatilization under the various conditions tested was estimated by weight loss measurements. The weight of a previously cleaned and thoroughly dried ceramic substrate was recorded prior to rosin application and immediately following the reflow operation. The weight gain (after equilibration) was assumed to be entirely due to the rosin residue. The original weight of the applied rosin cannot be accurately determined owing to continuous solvent evaporation, so this procedure can only provide a relative measure of volatilization with respect to the reflow conditions used. For consistency, the weight of the original non-reflowed

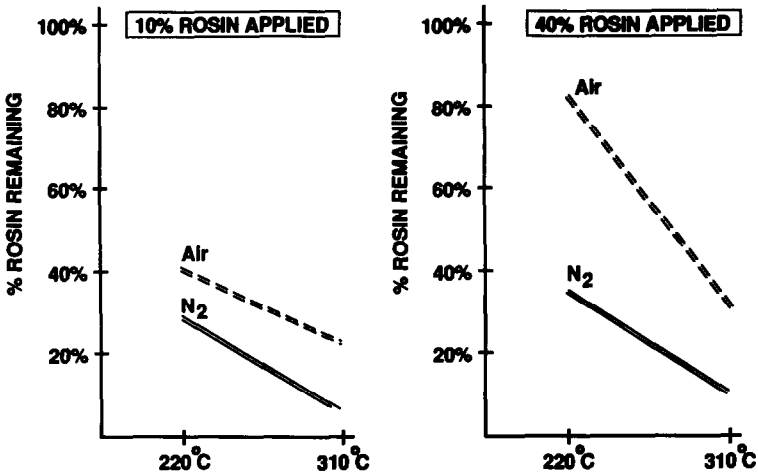


Fig. 1. Rosin volatilization by gravimetric analysis.

rosin was always recorded 1 hour after application. Figure 1 shows the percent of rosin remaining on the substrate after the reflow operation. The reflow was performed under nitrogen and air, at two temperatures (peak temperatures of 220 and 310°C). Each point represents an average of five independent measurements.

The most significant observation is that substantially more rosin volatilizes upon heating under an inert atmosphere. The implication may be that oxygen facilitates the formation of non-volatile chemical species which then inhibit the rate of volatilization. It is interesting to note that the overall extent of volatilization is quite extensive: during the high temperature nitrogen reflow less than 10% of the original rosin applied remains on the substrate.

The weight loss measurements are further substantiated by the TGA shown in Fig. 2 and also in agreement with other published studies [13,14]. Under nitrogen purge, the TGA trace is basically a smooth curve showing a pronounced decay around 250°C, then levelling off around 450°C to yield a residue of 4% of the original weight. When heated under air, the curve suffers the same sharp decay around 250°C. However, around 340°C, after 65% of the rosin is volatilized, a plateau is formed extending to almost 500°C. The final residue comprises 14% of the original material, in sharp contrast with the measurements under nitrogen.

The TGA scans, in agreement with the weight loss measurements, suggest the formation of a non-volatile component upon exposure to air. The basic question arising now is whether this component is a by-product of an oxidative reaction involving the rosin acid isomers.

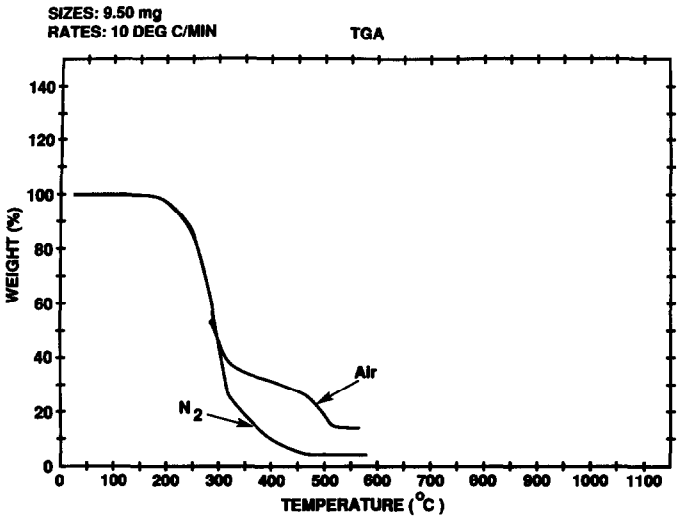


Fig. 2. Thermogravimetric analysis of rosin under air and nitrogen atmospheres.

UV / Vis and HPLC studies

To examine the impact of air and heat on the integrity and distribution of the rosin acid isomers, HPLC and UV spectroscopy were performed on extracted solutions. Figure 3(a–f) compares the UV absorption spectrum of a 30 ppm neat rosin solution to approximately 30 ppm solutions of extracts obtained from ceramic substrates reflowed under the conditions indicated (220 vs. 310°C, air vs. nitrogen).

In the absorption spectrum of pure rosin Fig. 3(a), the strong absorption at 203 nm is attributable to the dehydroabietic acid component, whereas the broader envelope of absorptions at 234, 241 and 250 nm is due to abietic acid, with a small contribution from neoabietic acid at 250 nm. The weak shoulder barely visible at 272 nm is due to levopimaric acid. The absorbance wavelengths for each of the isomers can easily be rationalized, using the empirical rules proposed by Fieser and Fieser for homoannular and heteroannular conjugated diene systems, in addition to those applicable to substituted benzene systems [8,15].

The absorption spectra of the extracted rosin after various heat treatments show dramatic differences. Under nitrogen reflow (Figs. 3(d,f)) the abietic acid concentration decreases considerably with respect to dehydroabietic acid, especially at the higher temperature reflow. A gradual broadening of the peaks is also observed in the higher wavelength region above 250 nm and around 210 nm.

Under air heating (Figs. 3(c,e)), these same changes are significantly more pronounced, to the extent that the spectrum of the rosin reflowed at 310°C shows none of the distinguishing features present in pure rosin.

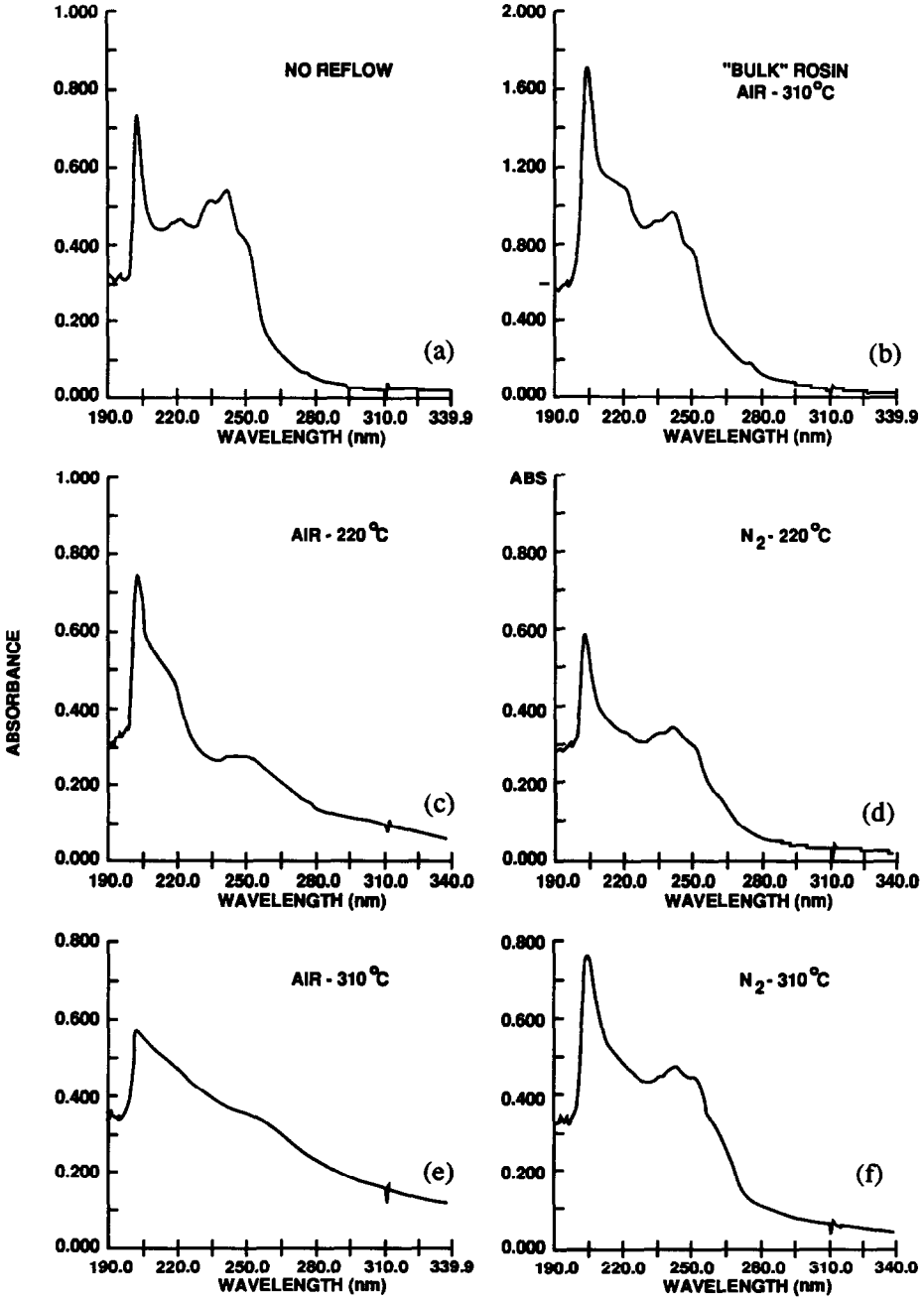


Fig. 3. UV absorption spectra of rosin extracts after solder reflow, in air and nitrogen.

Coupled with the disappearance of abietic acid peaks, there is considerable broadening of the entire spectrum with loss of practically all spectral features. In fact, these effects are even clearly visible at the lower temperature associated with conventional reflow, indicating that, contrary to cur-

TABLE 1

Rosin acid concentration by HPLC vs. gravimetric analysis

Reflow condition	By weight gain ($\mu g\ in^{-2}$)	By HPLC ($\mu g\ in^{-2}$)	Fraction by HPLC (%)
10% rosin applied			
N_2-220°C	250	35	14
N_2-310°C	50	3	6
Air-220°C	350	25	7
Air-310°C	200	0	0
40% rosin applied			
N_2-220°C	1400	308	22
N_2-310°C	400	24	6
Air-220°C	3200	576	18
Air-310°C	1100	55	5
40% rosin–bulk			
Air-310°C	10^6	0.72×10^6	72

rent belief, isomerization is not the only heat induced chemical reaction taking place. The severe spectral broadening can be explained by the formation of a large number of chemical species with different levels of saturation or conjugation. The absorbances of the individual functional groups overlap to produce the featureless spectrum shown. In the visible region (340–600 nm) none of the specimens exhibited any absorption.

Interestingly, if the rosin is reflowed in bulk form (≈ 5 gm in a small aluminum pan), instead of being applied as a thin layer, a rather different absorption spectrum is obtained (Fig. 3(b)). This spectrum, when contrasted to that for pure rosin (Fig. 3(a)), reveals that the bulk rosin has suffered very little change upon reflow. The abietic acid peaks are clearly visible. However, their intensity relative to dehydroabietic acid has diminished, indicating isomerization, aromatization and/or thermal rearrangement of the acid components. This result then suggests that the thermally induced chemical reactions of rosin are strongly dependent on the thickness of the applied film. This observation will be explained at the end of this section.

Each of the rosin extracts was studied by HPLC. The concentration of the rosin acid isomers was calculated using standard procedures [16] and was then compared to the rosin concentration as obtained by gravimetric measurements. The results are shown in Table 1. The first data column shows the amount of rosin present on the substrate after reflow by gravimetric analysis in $\mu g\ in^{-2}$; the second column gives the rosin acid isomer concentration calculated from the HPLC chromatogram and the third column computes the fraction of the rosin detected by HPLC (simply by dividing column 2 by column 1).

The most important observation is that the acid isomers identified by HPLC are only a small fraction of the total amount of rosin residue known to be present on the substrate by weight gain measurements.

These results, if interpreted in conjunction with the UV absorption spectra, suggest the formation of reaction products which, although UV absorbing, do not elute from the C18 column in an appropriate manner for LC detection. For instance, higher molecular weight species could elute at longer retention times, resulting in considerable peak broadening, to the point of becoming indistinguishable from the baseline. In fact, even though the rosin acid isomers elute after about 11 minutes, the baseline returns to zero absorbance only after 35–40 minutes.

Another observation worth noting is the fact that the rosin fraction detected by HPLC is slightly higher when 40% vs. 10% rosin is applied (increased film thickness), and significantly higher when "bulk" rosin is used. This reinforces the conclusion drawn from the UV studies that the chemical changes which occur during reflow are strongly dependent upon the thickness of the applied rosin film.

During the last few years, HPLC has been widely accepted as a quantitative analytical tool for the detection of rosin residues on electronic assemblies [16]. The results presented above demonstrate that HPLC, if performed according to the procedure outlined in the electronics literature, only detects a small fraction of the total rosin present, i.e. the fraction involving the acid isomers. It appears then that the majority of the extracted rosin is composed of chemical species that have not yet been identified.

Size exclusion chromatography

SEC was performed with the goal of determining whether any rosin polymerization takes place during reflow to account for the rosin fraction undetected by HPLC. Figure 4(a) shows the chromatogram of a 40% rosin solution diluted to 1000 ppm. The peak molecular weight is 298, compared to the theoretical MW of abietic acid of 302. To the left of the main peak, there is a weak shoulder, corresponding to a molecular weight of 650.

Figures 4(b,c) and 4(d,e) represent the chromatograms of the rosin extracts after reflow in nitrogen and air, respectively. For clarity, the prominent peaks have been labelled as 1 (MW $\approx$ 300), 2 (MW $\approx$ 650), and 3 (MW $\approx$ 1000 and higher). The peak labelled 0 can be attributed to impurities in THF.

Comparing the chromatograms in Figs. 4(a–e), it is evident that the signal due to the rosin acid isomers diminishes to the point of disappearance at the higher temperature reflow. This effect is compensated for by the gradual increase of higher molecular weight peaks, which ultimately dominate the chromatograms at high temperatures, regardless of whether

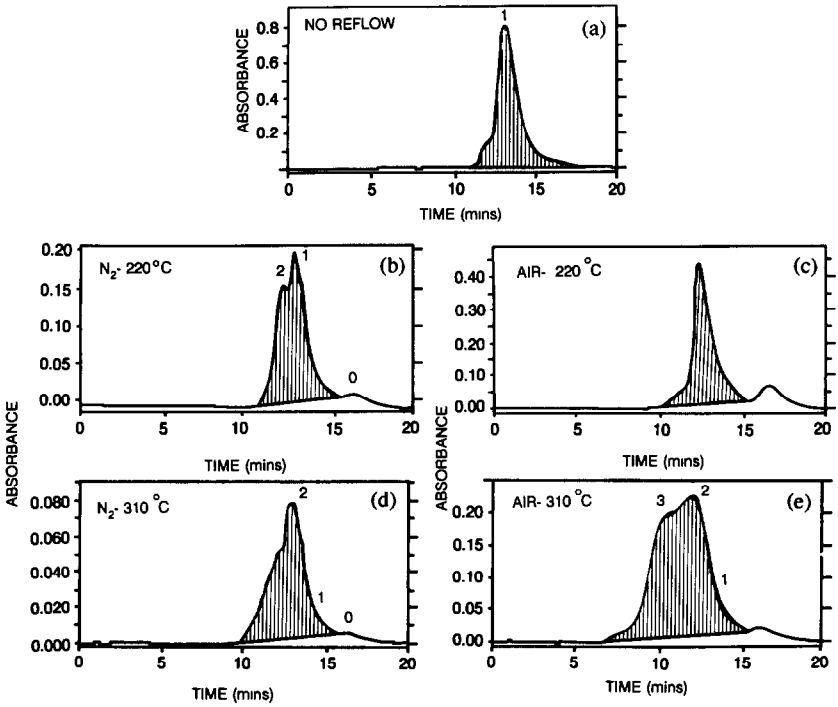


Fig. 4. Size exclusion chromatography of rosin extracts before and after solder reflow, in air and nitrogen.

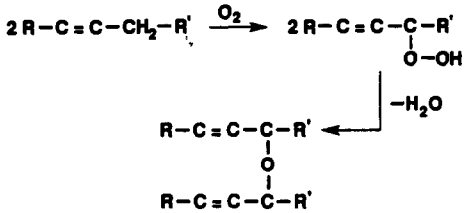
the reflow was performed under nitrogen or air. However, the chromatograms of the air reflowed specimens are considerably broader. Based on the relative molecular weight values calculated, it is suspected that these higher molecular weight components are due to dimers and low order oligomeric species (trimers). In fact, a low dimer fraction is even visible in the non-reflowed rosin sample (Fig. 4(a)).

The thermal dimerization of rosin has been observed and reported by Parkin et al. [17]. In their studies, different rosin types were heat treated under air and under an inert gas blanket, at temperatures ranging from 190–315°C. Product molecular weights were determined by vapor pressure osmometry. Heating gum rosin at 300°C for 20 minutes under nitrogen resulted in 22% dimer formation and significant loss of abietic-type acids. Heating in contact with air resulted in the formation of higher molecular weight species of unknown composition. Moreover, the non-heat treated rosin was found to contain 14% dimers. Our SEC results are in very good agreement with the results presented by these authors.

Parkin et al. postulated that these heat induced products are essentially a mixture of dimeric monobasic acids. Many of the products are believed to be esters, probably resulting from addition of the abietic acid carboxylic group across one of the double bonds of another abietic acid molecule.

Note that this reaction does not require the presence of oxygen. One of the reasons for this postulation is the presence of a strong IR absorption at 1730 cm^{-1}, characteristic of an ester group. The reaction in inert atmosphere does not produce alcohols, so esterification of rosin acids by alcohols can be ruled out.

Although the dimerization reaction is expected to proceed according to the same mechanism under air or nitrogen, it is suspected that under air, further auto-oxidation reactions occur [10,11,18], resulting in the formation of glycols, ketones and ethers of varying molecular weights, as indicated by the broader chromatograms. Auto-oxidative polymerization occurs when oxygen is absorbed by an unsaturated diterpene molecule to form a hydroperoxide on a carbon atom adjacent to the double bond. Hydroperoxides are not very stable and easily decompose, so an ether linkage has been postulated between two molecules, forming by decomposition of the peroxide and elimination of a molecule of water [10,18]. The auto-oxidative polymerization mechanism is schematically shown in reaction 2.



(2)

FTIR studies were therefore performed to better understand the nature of these chemical modifications.

FT-IR studies

Bare silicon wafers were coated with a thin film of rosin and reflowed. The surfaces were investigated before and after reflow by FT-IR in the transmission mode and the results are shown in Fig. 5. The spectrum obtained from the Si surface with a thin layer of 40% rosin and studied before reflow agrees reasonably well with published spectrum of abietic acid [19]. However, because the film contains some amount of trapped alcohol (the solvent in the commercially available rosin is a mixture of alcohols), the peaks are somewhat broader.

The strong peak in the carbonyl stretching region is centered at 1700 cm^{-1}, with a full width at half maximum (FWHM) of 70 cm^{-1}. This peak is characteristic of the carbonyl stretching vibration of the acid group (ν(CO)) of abietic acid.

The spectrum obtained after air reflow at 220°C shows significant changes from the scan before reflow. The spectrum is further broadened over the entire mid-IR range. The carbonyl stretching peak (at 1700 cm^{-1}) is almost twice as broad after air reflow (FWHM = 140 cm^{-1}, compared to

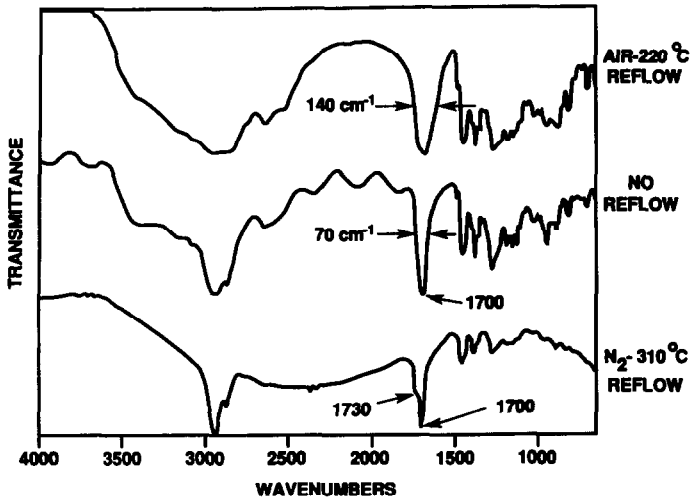


Fig. 5. FT-IR of 40% rosin before and after solder reflow, in air and nitrogen.

70 cm^{-1} before reflow). There is a significant increase in the intensity of the peaks between 3500–3000 cm^{-1}. Since the reflow operation involves heating the sample above 220°C, it is reasonable to expect all of the alcoholic solvents to evaporate. Hence, the spectrum must represent actual chemical changes of the rosin on reflow.

The IR spectrum after reflow in an N_2 atmosphere at about 310°C is also shown in Fig. 5. A cursory glance at the three spectra immediately shows that the spectrum after N_2 reflow is the simplest, with narrow, reasonably well-defined peaks compared to the other two. The most important feature is the narrowing of the 1700 cm^{-1} peak, so that a new peak centered around 1730 cm^{-1} is clearly resolved. The rest of the spectrum looks very similar to the published spectrum of abietic acid.

It is apparent from Fig. 5 that the spectra after air reflow and N_2 reflow are significantly different. This indicates that the products obtained under the two conditions are not the same. The major difference between the spectrum obtained after N_2 reflow and the published spectrum of abietic acid is the development of the new peak at 1730 cm^{-1}. As mentioned earlier, Parkin et al. [17] have also discussed the formation of new peaks at 1730 cm^{-1} in their heat treatment study of gum rosin, and explained it by proposing ester formation, probably resulting from addition of the rosin carboxylic acid group across the double bond of another abietic acid molecule. Pure abietic acid anhydride absorbs at 1790 cm^{-1}. This peak is not prominent, neither in the study of Parkin et al. nor in ours, indicating that thermally induced reactions of rosin in the absence of O_2 do not form the anhydride.

The broad spectrum obtained after air reflow would indicate a mixture of products strongly absorbing in the mid-IR region. As discussed earlier,

the products of thermally induced air-oxidation are a mixture of glycols, ketones, ethers, etc. The increase in the O–H stretching region at 3000 cm^{-1} and above indicates the formation of new alcoholic groups, due to the formation of glycols. Similarly, the carbonyl stretching region at 1800–1650 cm^{-1} and the C–O–C stretch (1200–1000 cm^{-1}) show increased intensity, which can be explained by formation of a variety of oxidized products, all containing carbonyl type linkages. The reactions in the presence of air might also involve the formation of the anhydride, which was clearly ruled out for N_2 reflow.

It should be remembered that solder was not present on any of the specimens examined here. Therefore, these reactions are specific to the rosin only and are not initiated by the presence of lead or tin.

CONCLUSIONS

This study was performed with the objective of elucidating the rosin chemical changes induced by heat and air during the reflow process. A variety of analytical techniques were employed, including TGA, HPLC, SEC, UV/Vis and FT-IR. Chemical degradation of rosin can have a profound effect on the possibility of cleaning electronic assemblies and, thus, the detection of rosin residues is essential for the assessment of cleaning quality.

HPLC is widely accepted in industry as analytical tool for the detection of rosin residues. The general belief is that during conventional reflow (200–250°C) the rosin undergoes predominantly isomerization reactions and decomposes at very high temperatures. Polymerization of rosin is often speculated about.

Our studies have shown that in addition to isomerization, the rosin undergoes significant dimerization in an inert atmosphere, to produce a mixture of dimeric diterpenes. The dimerization mechanism does not require the presence of oxygen. During air reflow, additional auto-oxidative polymerization reactions occur through hydroperoxide intermediates, resulting in the formation of glycols, ketones and ethers of varying molecular weights. These chemical changes occur independently of whether rosin reflow is accompanied by solder, indicating that they are not induced by solder components (Pb, Sn). The auto-oxidative polymerization is largely a surface phenomenon and is strongly dependent on the thickness of the applied rosin film. As film thickness decreases, the fraction of high molecular weight polymerized species becomes significant. For very high film thickness (bulk) the rosin exists to a large extent as a mixture of various diterpene acid isomers.

We have also demonstrated that HPLC is not the most suitable analytical technique for rosin residue detection because its capability is limited to the detection of rosin acid isomers, which provide only a small fraction of the total amount of rosin present. In contrast, SEC can sensitively detect

the higher molecular weight species and thus appears to be a superior analytical method for this application [20].

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