Effect of Alloy and Flux System on High Reliability Automotive Applications

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Abstract
The July 2006 implementation of ROHS exempted automotive applications from converting to lead free technology. Nine years later, all major OEM and Tier 1 automotive manufacturers have converted or are in the process of converting to lead free circuit assembly processing.

Starting with SAC (SnAgCu) alloys as a baseline for lead free soldering, in the last years several specific alloys were developed in order to improve resistance to high temperature creep, vibration survival and the ability to withstand thermal cycling and thermal shock.

The paper compares three different solder alloys and two flux chemistries in terms of void formation and mechanical / thermal fatigue properties. Void content and reliability data of the alloys will be presented and discussed in relation to the acceptance criteria of a Tier 1 /OEM automotive supplier. As a result, a ranking list will be presented considering the combined performance of the alloys. In order to analyze the void formation and mechanical behavior of different solder alloys and flux chemistry combinations, statistical methods are used.

Introduction
Lead-free solder has been used intensively for more than 10 years now, even in high reliability applications such as Automotive under the hood electronics. Starting with SAC (SnAgCu) alloys as a baseline for lead free soldering, in the last years several specific alloys were developed in order to improve resistance to high temperature creep, vibration survival and the ability to withstand thermal cycling and thermal shock [1, 2]. Besides the mentioned mechanical properties of the alloys and the solder joints produced with these alloys, the resistance to voiding is another important aspect of modern lead free solder paste and highly reliable solder joints. As reported in several publications, one alternative solder alloy Sn3.8Ag0.7Cu3Bi1.4Sb0.15Ni (Alloy A) seems to be subject to more voiding than the standard SAC alloy [3].

Because of the high level of reliability requested in automotive applications, OEM and tier 1 suppliers have developed reliability test protocols that exceed industry standards developed by the IPC (Institute of Printed Circuits), JIS (Japanese Industrial Standards) and DIN (Deutsches Institut für Normung). This paper will focus on a subset of a unique test method used by a Tier 1/OEM automotive supplier. The testing involved voiding and solder joint shear strength after thermal shock. Voids can be a source of electrical failure if a catastrophic failure occurs due to a crack propagating through the solder joint [4]. Voids insulate the transmission of heat away from high current density regions in an electronic device, often reducing the useful life of devices [4]. Shear strength is an issue when components are subjected to forces associated with opening and closing doors, trunks and the hood of motor vehicles. Jolts from high speed travel over uneven road surfaces can also create shear forces that challenge the reliability of electronic systems.

This study looks at the effect of three different solder alloys and two different solder paste flux systems on the level of voiding and the shear strength of passive components using a standard automotive OEM/Tier 1 test vehicle. All results were compared with the customer requirements for voiding and shear force reduction after thermal cycling, so the most favorable material could be easily identified. In order to avoid misinterpretation of the results, all investigations were analyzed using statistical methods such as ANOVA (analysis of variances). With this approach, the fulfillment of customer requirements can be proven on a specific confidence level.

Methodology Description
Automotive customers request low void content in solder joints. Based on the specification of an international customers, which is the baseline for the evaluation done in this paper, the average ratio of void (in area %) has to be measured in the soldering area by means of X-ray testing. In paste deposits with no devices placed, an average void level below 10% of the soldered area must be reached in order to fulfill the specification requirements. Several papers have been published dealing with void formation in solder joints. In most cases only a few data points per test condition are examined (small sample size); therefore a direct and statistically safe conclusion for a large number of solder joints (basic population) cannot be drawn. So far, mainly mean values have been analyzed without considering variances indicating variable process influences. As a

conclusion, statistical methods are necessary to make the results commonly applicable and commonly valid. Regarding solder joint strength, the situation is quite comparable. Most evaluations of initial solder joint strength and solder joint reliability (e.g. strength after thermal cycling) do not consider the confidence level of the results. Mean values gained from small samples sizes and with large variances do not give a realistic picture of the situation. Therefore, the fulfillment of the customers' requirement of less than 35 % drop of shear force (average value) for passive components is verified for different alloy and flux chemistry combinations using an identical statistical approach.

In this paper, the void formation as well as the reliability of different solder alloys and flux chemistry combinations is analyzed using statistical methods. The tools mainly used to evaluate significant differences between these combinations and to verify the fulfillment of customers' requirements on a statistically proven basis is the analysis of variances (ANOVA) and the 1-sample t-test, implemented in the statistical software package.

Using the ANOVA requires the fulfillment of a specific approach, which is listed below:

1. Analyze the distribution of the raw data
2. Test for equal variances
3. Conduct ANOVA (confidence level 99 %)
4. Analyze the ANOVA results and interpret the result
5. Test for fulfillment of customer requirement (1-sample t-test; confidence level 99 %)

Experimental Procedure

In this paper, a study of the voiding and mechanical strength (shear force) of three lead-free alloys will be presented. In total, four no clean solder pastes with two different flux chemistries were examined, as shown in Table 1. Alloy 3 is an alloy originally based on Alloy A, designed for high resistance to thermal cycling, vibration and creep.

Table 1. Matrix of solder pastes used in this study

	Powder – alloy	Flux chemistry	Powder type
Paste 1	SAC 305	Flux 1	4
Paste 2	Alloy 3	Flux 1	4
Paste 3	Alloy 3	Flux 2	4
Paste 4	Alloy A	Flux 2	4

The test vehicle used for the experiments was based on a standard FR-4 material suitable for lead free reflow soldering. To ensure solderability, the PCB surface was covered with OSP. Solder paste was printed automatically using a 0.2 mm thick laser cut stencil for voiding investigations and a 0.15 mm thick stencil for shear force examination according to the customer specification. For void testing, the test used 5 test vehicles with 8 opportunities per vehicle for a total of 40 data points per test condition. The voiding portion of the test vehicle used is shown in Figure 1. According to the customer specification, there were no devices placed into the paste deposits that could entrap gasses which would lead to voids. All test vehicles were reflowed under air atmosphere. A soak to ramp reflow profile, generally known to minimize voiding was used on all samples (see Figure 2).

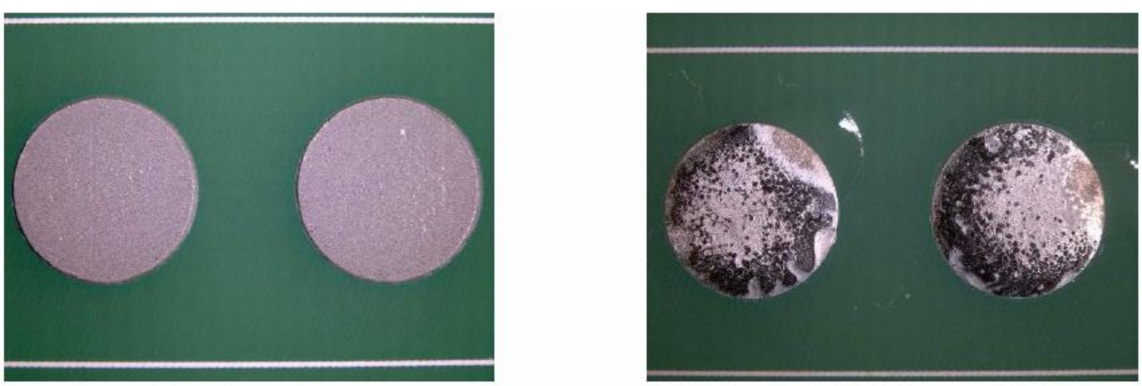

Figure 1. Voiding test structures -print and reflow results on circular pads (diameter 6.5mm)

For shear force testing, after components placement using a production placement system, all test vehicles were reflowed under air atmosphere. The same soak to ramp reflow profile was used on all samples with peak temperature from 235°C to 245°C and time over liquidous of 50 to 60 seconds(see Figure 2).

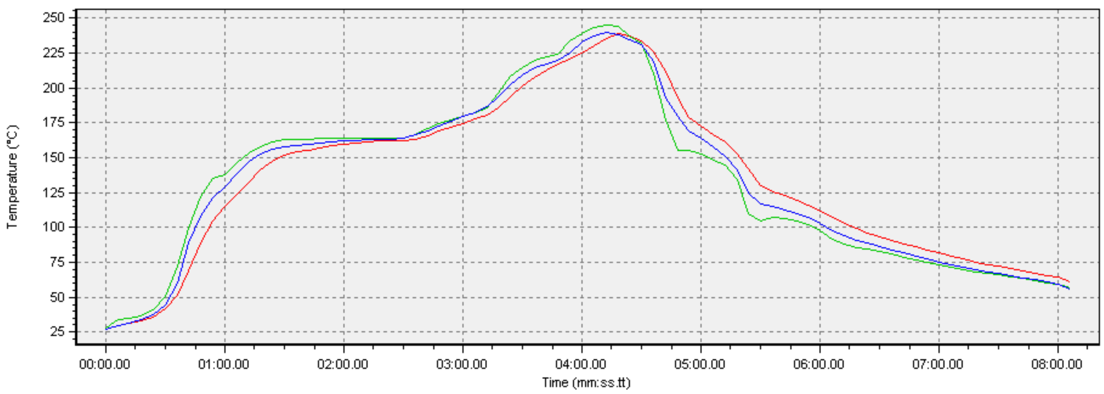

Figure 2. Soak to Ramp profile used in the investigation

After reflow soldering the voiding test vehicle was examined by X-Ray inspection using a production X-ray system. 40 X-Ray images were analyzed per paste to measure the rate of voiding. In order to evaluate the mechanical behavior of the solder joints, shear force testing on soldered chip resistors (size CR 1206, termination Sn) was done using a production shear force tester. Shear force testing was conducted initially and after thermal shock (-40 °C/+125 °C, dwell time 10 min; 1500 cycles). 48 individual force values per paste and test condition were measured and analyzed.

Results and Discussion

Voiding

As described above, a total of 40 datapoints per solder paste was evaluated in order to determine the effect of alloy and flux vehicle on void formation. In figure 3 the boxplot diagram summarizes the data of the X-ray testing for easier analysis.

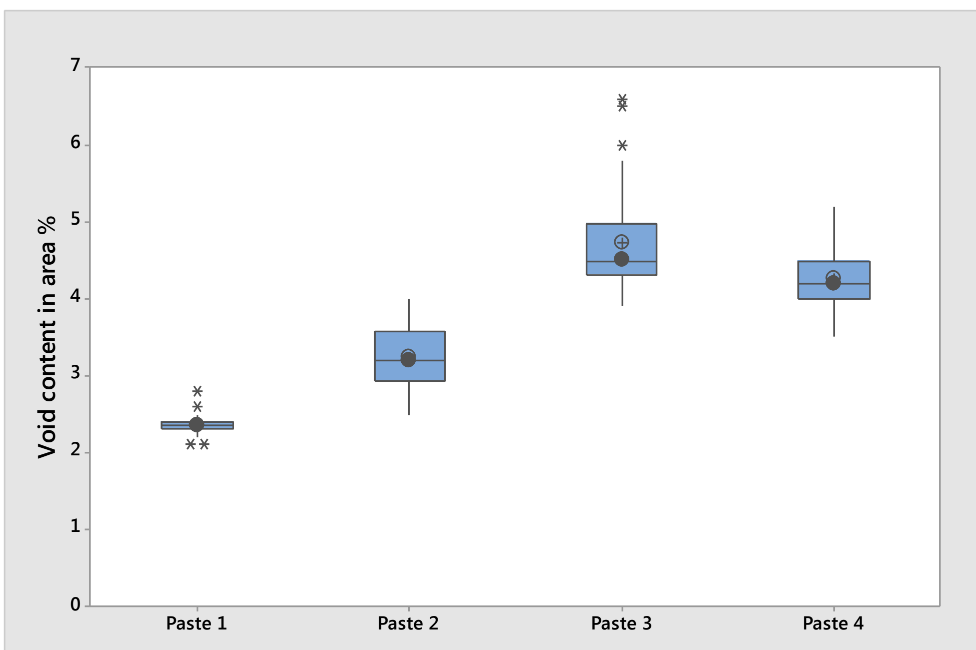

Figure 3. Boxplot of the void content (* indicates outliers)

Exemplary X-Ray images for all four combinations investigated during this analysis are shown in figure 4. As can be seen, paste 1 (SAC 305 with flux 1) shows the lowest voiding content. As an average, slightly more than 2 % voiding can be observed with a very small range. The same flux chemistry results in significantly higher voiding when used in combination with the Alloy 3 under the experimental conditions used during the investigation program. Mean value and span width as an indicator for variance are more than doubled. Combining flux 2 with the Alloy 3 as well as Alloy A leads to voiding results between both extremes.

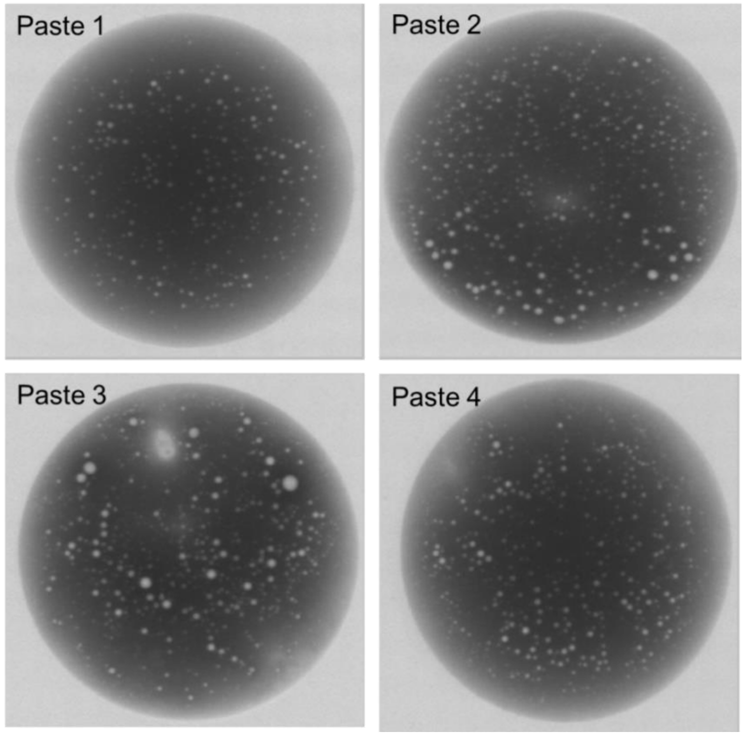

Figure 4. X-Ray images of the four results

From the boxplot diagram, a direct conclusion whether the four pastes behave significantly different cannot be drawn. Using the analysis of variances (ANOVA), a statistical method, this question can be answered. In the present case, one-way ANOVA provides a statistical test of whether or not the observed void means of the four pastes are equal. Preconditions for using this approach are as follows:

- Normally distributed raw data or residuals
- Equal variances for each sample group to be compared
- Random samples drawn from the population

The fulfillment of these assumptions was tested prior to applying ANOVA. As a result it can be stated, that specifically the requirement of equal variances is not fulfilled in the example. Therefore, an adapted ANOVA, the so called Welch's ANOVA, had to be conducted. The result indicates a significant impact of the solder paste type on the void averages. Further analysis using pairwise comparison between the four pastes shows that all combinations are significantly different from each other. Based on these results, a statistically proven ranking for the investigated pastes regarding voiding can be made (fig. 5).

Games-Howell Pairwise Comparisons

```
Grouping Information Using the Games-Howell Method and 99% Confidence

Factor    N    Mean  Grouping
Paste 3  40   4,735  A
Paste 4  40  4,2700    B
Paste 2  40  3,2350      C
Paste 1  40  2,3625        D

Means that do not share a letter are significantly different.
```

Figure 5. Screenshot of statistical software session window indicating significant differences between the pastes related to voiding: worst paste 3, best paste 1

The following graph shows the 99 % confidence intervals for the void averages for each paste, emphasizing the void ranking already listed in figure 5.

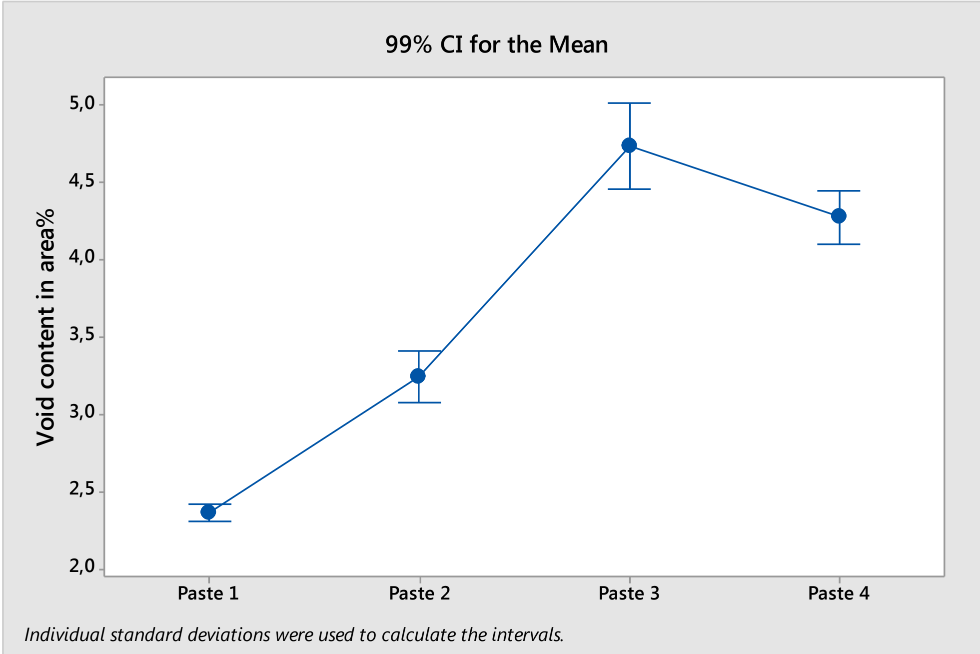

Figure 6. Confidence interval for the average void content

From a customers' perspective more interesting than the direct comparison of the voiding results for each solder paste is the information whether all pastes investigated can fulfill the 10 % void level criterion. Thus, in a subsequent analysis, a basic hypothesis test was conducted to verify this.

The 1-sample t-test computes a confidence interval or performs a hypothesis test of the mean when the population standard deviation σ is unknown. This procedure is based upon the t-distribution, which is derived from a normal distribution with unknown standard deviation. This procedure works best if the raw data are drawn from a distribution that is normal or close to normal. In the present case, the 1 sample t-test compares the mean of the single sample with the void area limit of 10 %, as specified by the customer. This is repeated for all four pastes investigated. The Null hypothesis assumes that there are no significant differences between the void area limit and the sample void mean, whereas the Alternative hypothesis assumes a significant difference, in the specific case a value below 10 %. The results of the 1-sample t-test are shown in the figure 7(confidence level 99 %).

One-Sample T: Paste 1; Paste 2; Paste 3; Paste 4

Test of μ = 10 vs < 10

Variable	N	Mean	StDev	SE Mean	99% Upper Bound	T	P
Paste 1	40	2,3625	0,1280	0,0202	2,4116	-377,44	0,000
Paste 2	40	3,2350	0,3887	0,0615	3,3841	-110,09	0,000
Paste 3	40	4,735	0,652	0,103	4,985	-51,05	0,000
Paste 4	40	4,2700	0,4109	0,0650	4,4276	-88,20	0,000

Figure 7. Screenshot of statistical software session window indicating significant differences between the pastes related to voiding: worst paste 3, best paste 1

In all cases, as a result a p-value of 0,000 is obtained. This indicates that the Null hypothesis has to be rejected and that the Alternative hypothesis void area < 10 % is accepted. All average void measurements are significantly below the specified value of 10 %.

Based on the results of the investigation program, the customer requirement of an average area void content of below 10% will be fulfilled in a statistically significant way.

Shear force

Mechanical stability is one fundamental prerequisite for reliable electronic applications and products. According to the requirement specification of the OEM, the shear strength of solder joints needs to be evaluated initially (as soldered) and after exposure to thermal shock treatment (-40°C/+125°, dwell time 10 min; 1500 cycles). 48 individual force values per paste and test condition were measured and analyzed afterwards. Test boards were produced by means of solder paste printing (stencil thickness 150 µm) and automated placement of chip resistors CR1206, as requested by the customer. The fulfillment of the customers' requirement of less than 35% reduction of shear force for the selected 1206 passive components is verified for the different alloy and flux chemistry combinations using an identical statistical approach.

As shown in figure 8, the measured shear force for all pastes (as soldered) seem to be quite comparable. The shear values are normally distributed, all initial shear force values show similar variances. For analyzing differences between the values, thus the standard ANOVA can be used.

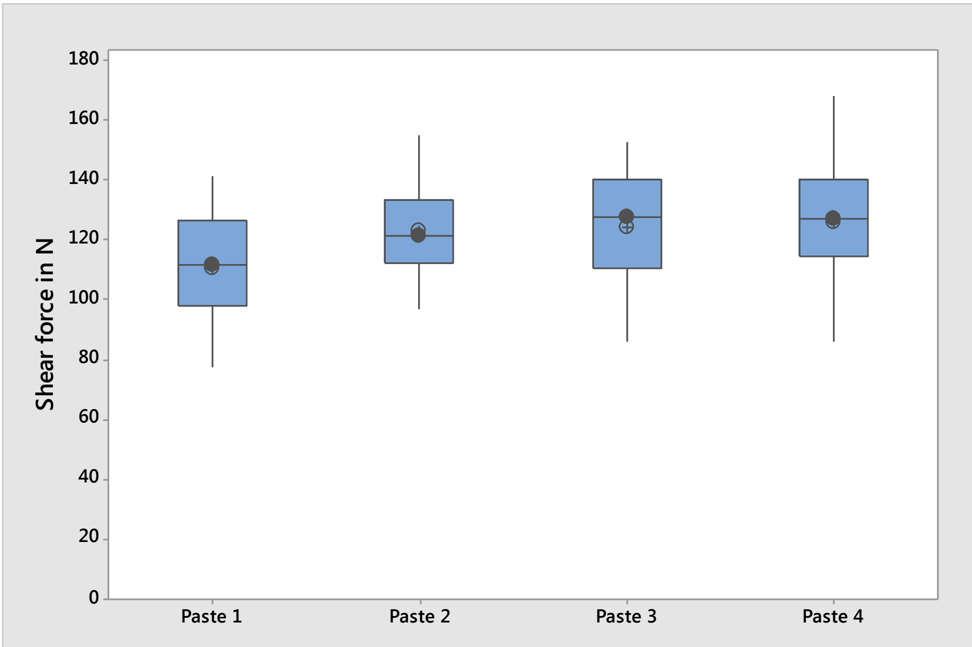

Figure 8. Boxplot of shear forces for each combination (initial values)

Statistical analysis of the measured results shows a clearer picture. Applying ANOVA's pairwise comparison between the four groups clearly demonstrates paste 1 (SAC 305) having a significantly lower initial shear force than the other alloys. Between the remaining alloy and flux combinations, no differences were found.

Fisher Pairwise Comparisons

```
Grouping Information Using the Fisher LSD Method and 99% Confidence

Legierung/Flussmittel   N    Mean  Grouping
Paste 4                49  125,94  A
Paste 3                48  124,41  A
Paste 2                48  122,93  A
Paste 1                48  110,64    B

Means that do not share a letter are significantly different.
```

Figure 9. Screenshot of statistical software session window indicating significant differences between paste 1 and the rest

After thermal shock treatment, the situation is somewhat different. As can be seen in figure 10, all tested combination show more or less remarkable drops of the measured shear force values. In parallel, the variances for all tested combinations increased. The most significant changes seem to be present for paste 1. After initial shear forces with an average value of 110 N, after 1500 thermal shock cycles a drop below 70 N can be observed. Related to the initial value, this result is already a shear force drop of more than the tolerable 35 %. As a consequence it can be stated, that paste 1 (SAC 305) is not acceptable for this OEM.

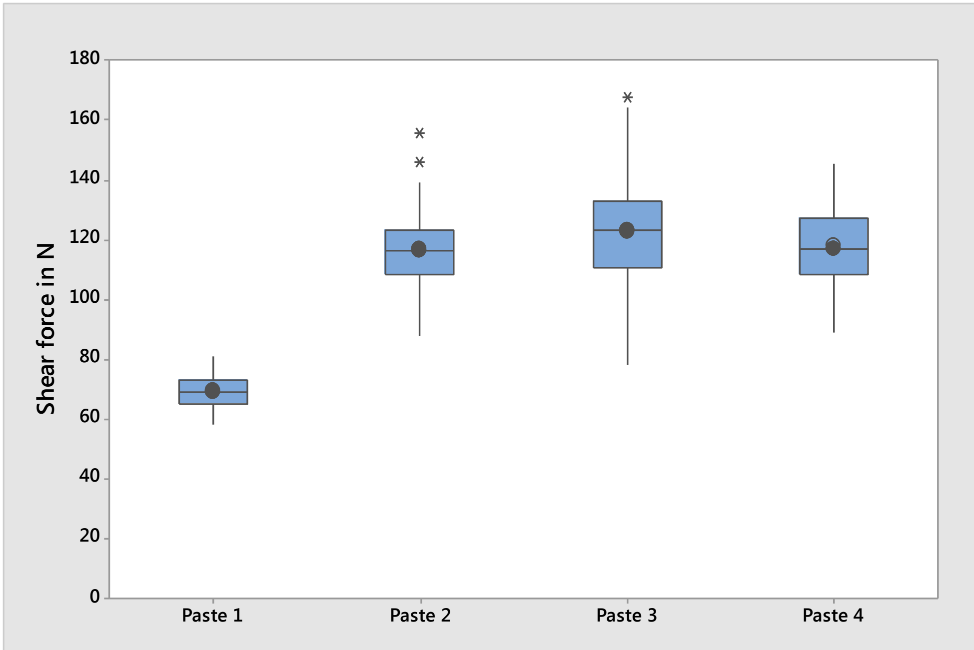

Figure 10. Boxplot of shear forces for each combination (after 1500 shocks)

In order to verify the individual fulfillment of the 35 % drop criterion for the remaining pastes, again the 1-sample t-test was used. As the 35 % shear force drop is related to the initial shear force value, three different absolute limits had to be calculated and applied in the test. In this case the Null hypothesis assumes that there are no significant differences between the paste-specific shear force limit and the sample shear force mean, whereas the Alternative hypothesis assumes a significant difference, in the specific case a shear force drop of less than 35 %. The results of the 1-sample t-test are shown in figure 11 (confidence level 99 %).

One-Sample T: Paste 2

```
Test of μ = 79,9 vs > 79,9


Variable   N    Mean  StDev  SE Mean  99% Lower Bound      T      P
Paste 2   46  117,29  12,76     1,88           112,76  19,88  0,000
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One-Sample T: Paste 3

```
Test of μ = 80,8 vs > 80,8


Variable   N    Mean  StDev  SE Mean  99% Lower Bound      T      P
Paste 3   46  122,94  18,19     2,68           116,47  15,71  0,000
```

One-Sample T: Paste 4

```
Test of μ = 81,8 vs > 81,8


Variable   N    Mean  StDev  SE Mean  99% Lower Bound      T      P
Paste 4   46  118,44  14,18     2,09           113,40  17,52  0,000
```

Figure 11. Statistical software session window indicating rejection of the Null hypothesis, therefore shear force drop significantly lower than 35%

In all cases, as a result a p-value of 0,000 is obtained. This indicates that the Null hypothesis has to be rejected and that the Alternative hypothesis -average shear force drop < 35 % - is accepted. All observed average shear force drops are significantly less than the specified value of 35 %.

Conclusions

Based on the results obtained during this study it can be summed up that the investigated combination of SAC-alloy with flux chemistry 1 results in the lowest void formation. All other alloy and flux chemistry combinations resulted in significantly higher void means. The results of this study therefore are in-line with previously published studies. Nevertheless, especially the Alloy 3 combined with a proper paste flux showed promising void results, which were only slightly higher than for SAC with the same flux. In contrast, flux 2 always showed higher voiding, regardless of the alloy. In total, all investigated combinations fulfill the requested average area void level of below 10 area %. This was proven using statistical tests.

Initial shear strength of CR1206 components is comparable for the Alloy A and Alloy 3, independent of the flux chemistry. SAC standard solder shows significantly lower initial values of around 110 N. After 1500 shock cycles, all three Alloy A and Alloy 3 containing solder pastes show no significant difference with shear force drops of about 5 % maximum. In contrast, measured shear forces for SAC are significantly lower, with absolute values below 70 N. The 35 % shear force drop criterion cannot be fulfilled by solder paste 1 (SAC), whereas the Alloy A and Alloy 3 containing pastes pass the reliability evaluation without problems.

Based on the existing results, paste 2 (alloy 3 combined with flux chemistry 1) is slightly favorable due to best combined performance (considering void formation as well as mechanical stability). This paste exhibits low voiding (significantly lower than paste 3 and paste 4), high initial shear forces (equal to paste 3 and 4) and relatively stable shear forces after thermal cycling (drop only 5 %). Even after thermal shock, the observed shear forces do not show significant differences to other Alloy A/Alloy 3 combinations. The observed variations are equal to or lower than for paste 3 and paste 4.

References

[1] Innolot Consortium, Final Report 2004

[2] H.-J. Albrecht, P. Frühauf, K. Wilke: Pb-Free Alloy Alternatives: Reliability Investigations. SMTA International, 2009.

[3] S. Brown, M. Holtzer, G. Renaud: Process considerations in Reducing Voiding in High Reliability Lead Free Solder Joints. SMTA China East - NEPCON Shanghai, 2015

[4] K. Wilke : Einfluss von Voids auf die Zuverlässigkeit von Lötverbindungen. DVS Jahrbuch 2009/2010.

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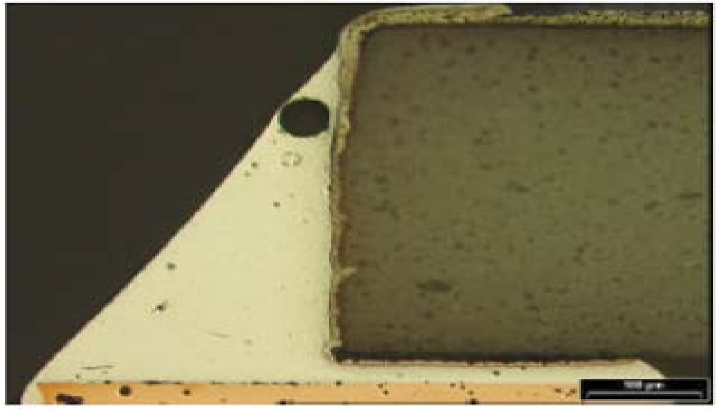
Outline/Agenda

- Introduction
- Methodology description
- Experimental procedure
- Results and Discussion
  - Voiding
  - Shear force
- Conclusions

Introduction

- Lead-free solder intensively used for more than 10 years now, standard solder alloy SAC
- Alloys with higher creep resistance such as Alloy A (Sn3.8Ag0.7Cu3Bi1.4Sb0.15Ni)



are available, but prone to voiding

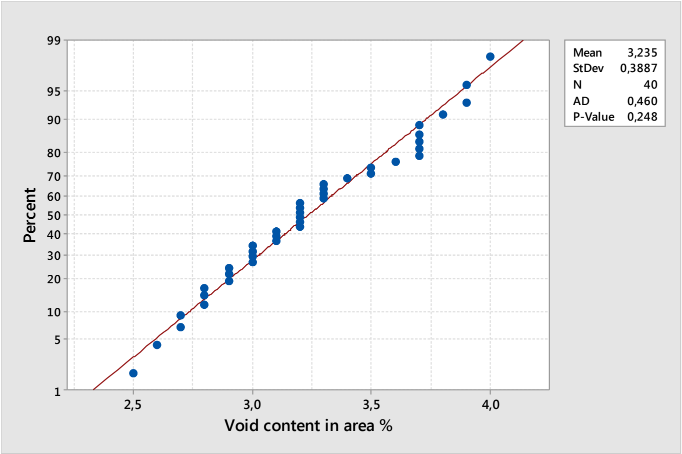
- Scope of presentation
  - Evaluation of four different solder pastes (3 alloys, 2 flux chemistries) in terms of voiding and solder joint shear strength
  - Investigation program based on the requirements of an Automotive OEM

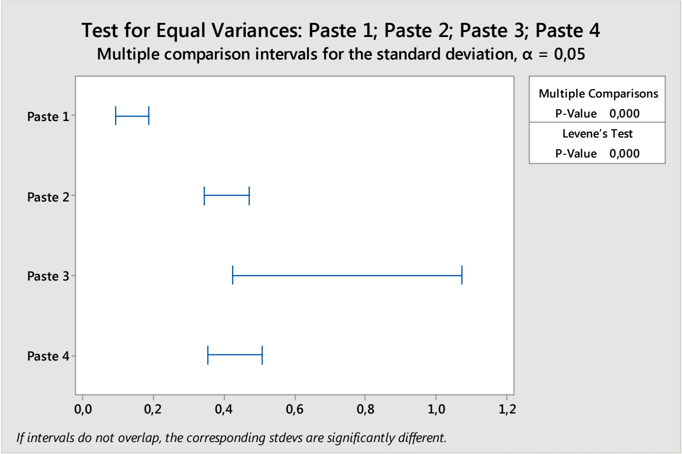
Methodology Description

- Test criteria based on customer specification
  - Average void content in area % below 10%
  - Shear force drop after 1500 thermal shocks less than 35% related to initial value (as soldered)
- Fulfillment of the customers’ requirement verified for alloy and flux chemistry combinations using an identical statistical approach
- Tools mainly used:
  - the analysis of variances (ANOVA)
  - the 1-sample t-test

Statistical Analysis - Approach

- Check preconditions
  - Analyze the distribution of the raw data (normal distribution)
  - Test for equal variances
- Conduct ANOVA (confidence level 99 %)
- Analyze the ANOVA results and interpret the result (“Are the results different?”)
- Test for fulfillment of customer requirement (1-sample t-test; confidence level 99 %)





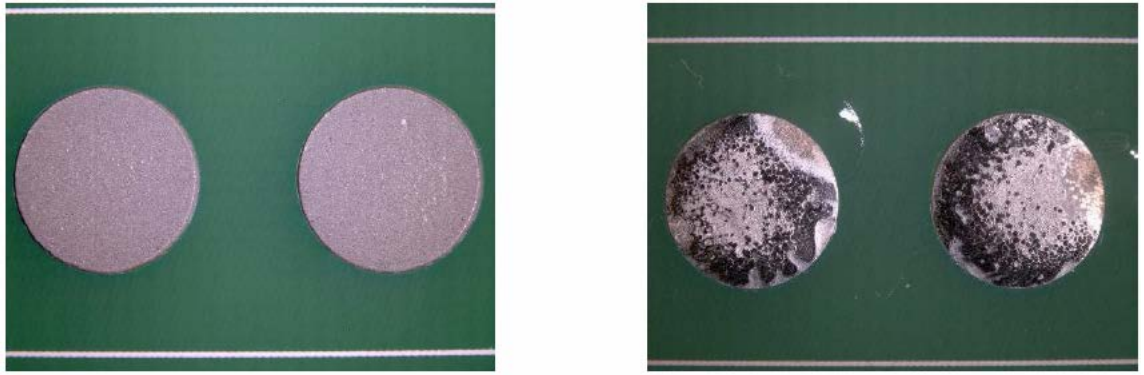
Experimental Procedure (I)

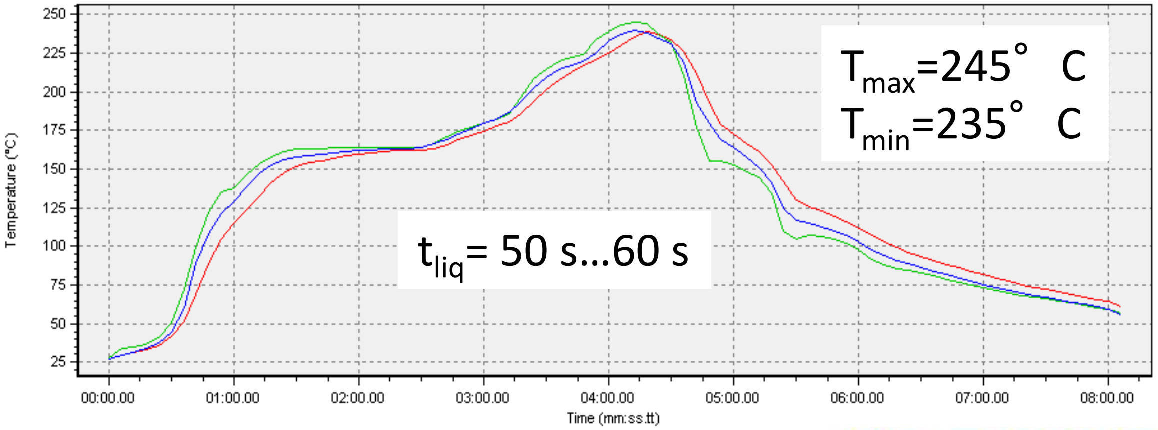
- Four solder pastes
  - Three alloys
  - Two flux chemistries
- FR-4 PCB with OSP finish
- Stencil printing with
  - 0,2mm thick stencil for void formation investigation (AXI with 40 samples for each combination)
  - 0,15mm thick stencil for shear force investigation (Component CR 1206; 48 samples for each combination)

	Powder – alloy	Flux chemistry	Powder type
Paste 1	SAC 305	Flux 1	4
Paste 2	Alloy 3	Flux 1	4
Paste 3	Alloy 3	Flux 2	4
Paste 4	Alloy A	Flux 2	4

Experimental Procedure (II)

- According to customer specification, there were no devices placed into the paste deposits that could entrap gasses which would lead to voids.
- Reflow under air atmosphere
- Soak to ramp reflow profile, generally known to minimize voiding used on all samples



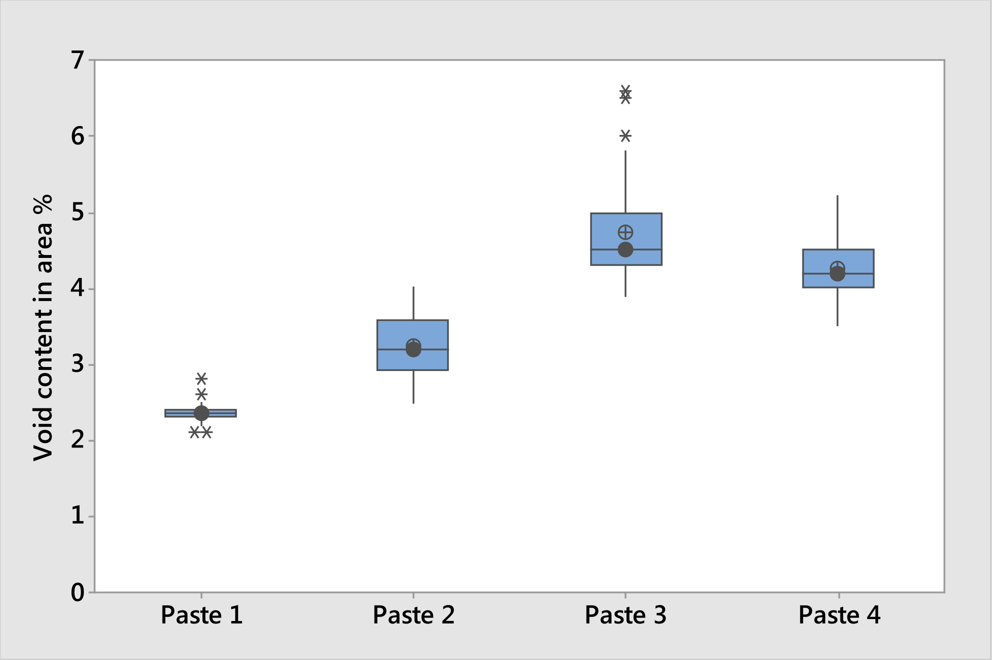


Experimental Procedure (III)

- Inspection for void content using automated X-ray inspection (40 X-ray photos taken per paste)
- Evaluation of mechanical integrity by means of shear testing (CR 1206 components)
  - Initial values (as soldered)
  - After thermal shock testing (1500 cycles, -40° C/+125° C, 10 minute dwell)
  - 48 individual values per solder paste and test condition

Results and Discussion: Voiding

- Lowest void content: Paste 1 (SAC)
- Same flux chemistry results in significantly higher voiding when used in combination with Alloy 3 (Paste 2)
- Mean value for void content and range as an indicator for variance are more than doubled
- Combining Flux 2 with the Alloy 3 as well as Alloy A alloy leads to voiding results between both extremes



Results and Discussion: Voiding

All pastes show statistically significant different voiding results, as shown below and illustrated in the graph

Games-Howell Pairwise Comparisons

Grouping Information Using the Games-Howell Method
and 99% Confidence

Factor	N	Mean	Grouping
Paste 3	40	4,735	A
Paste 4	40	4,2700	B
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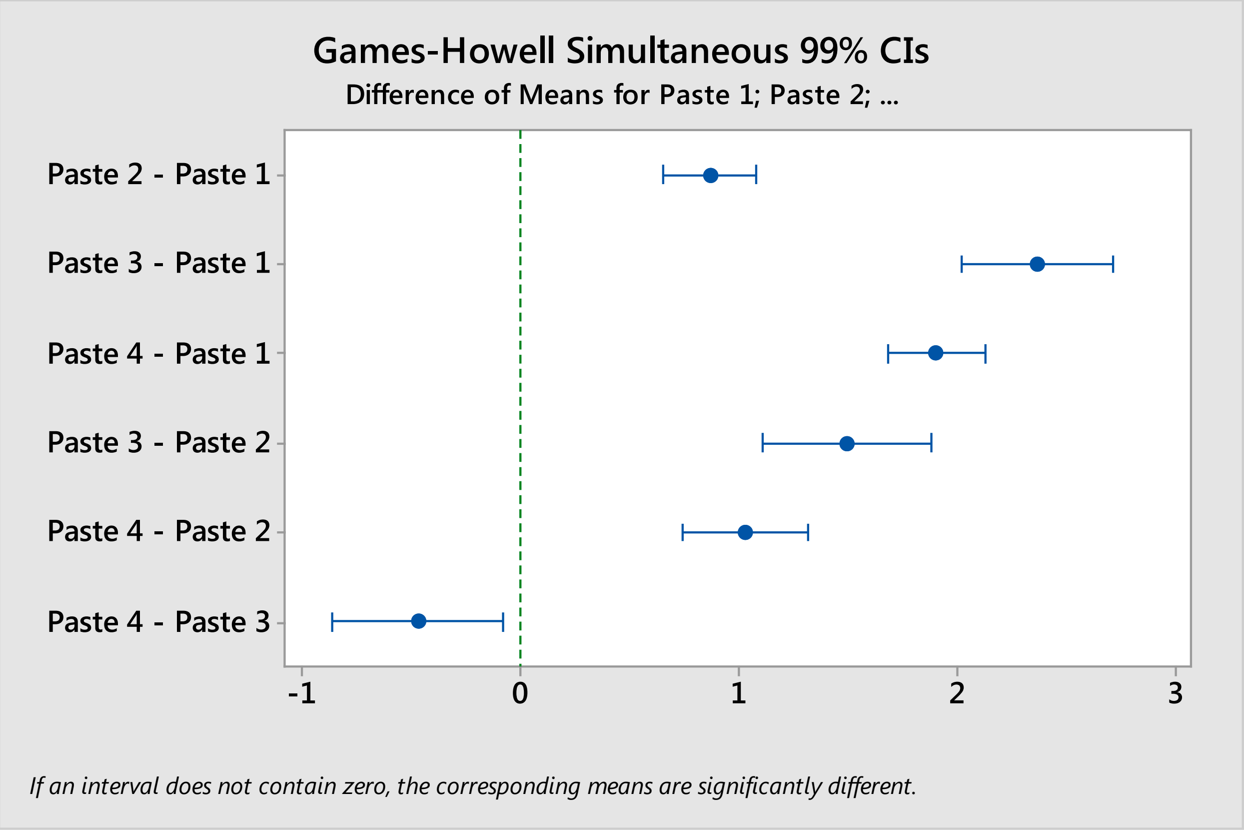
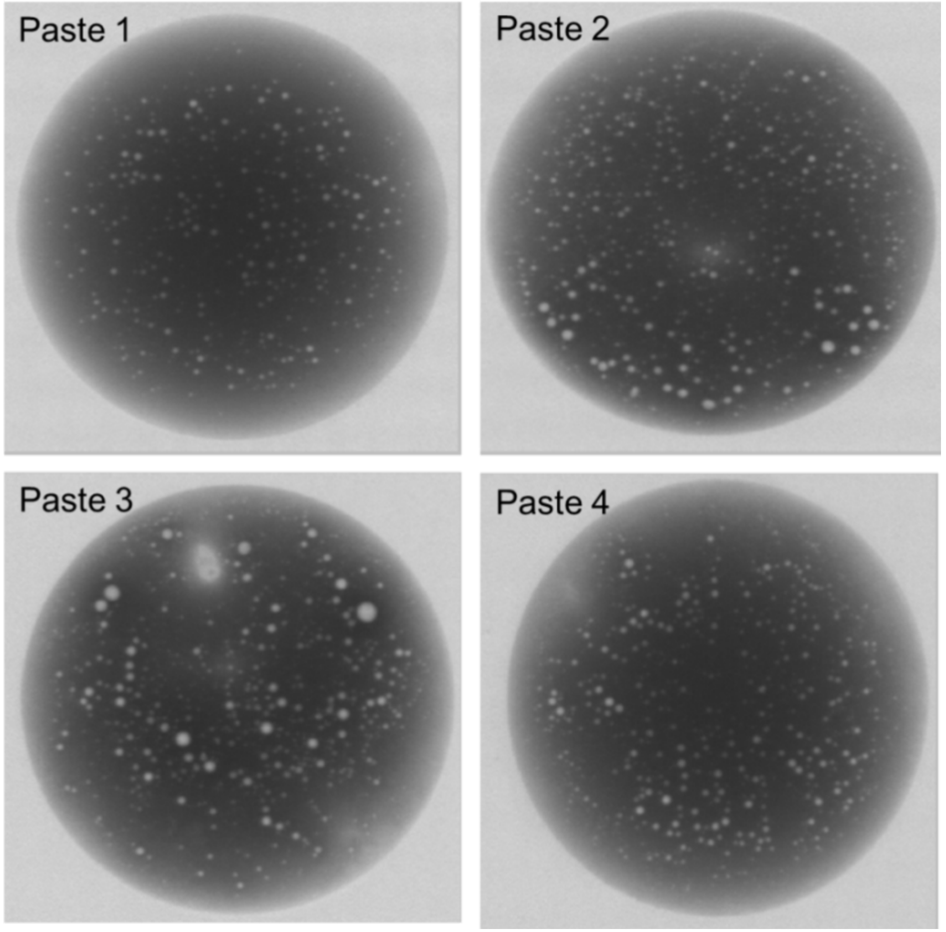
Means that do not share a letter are significantly different.

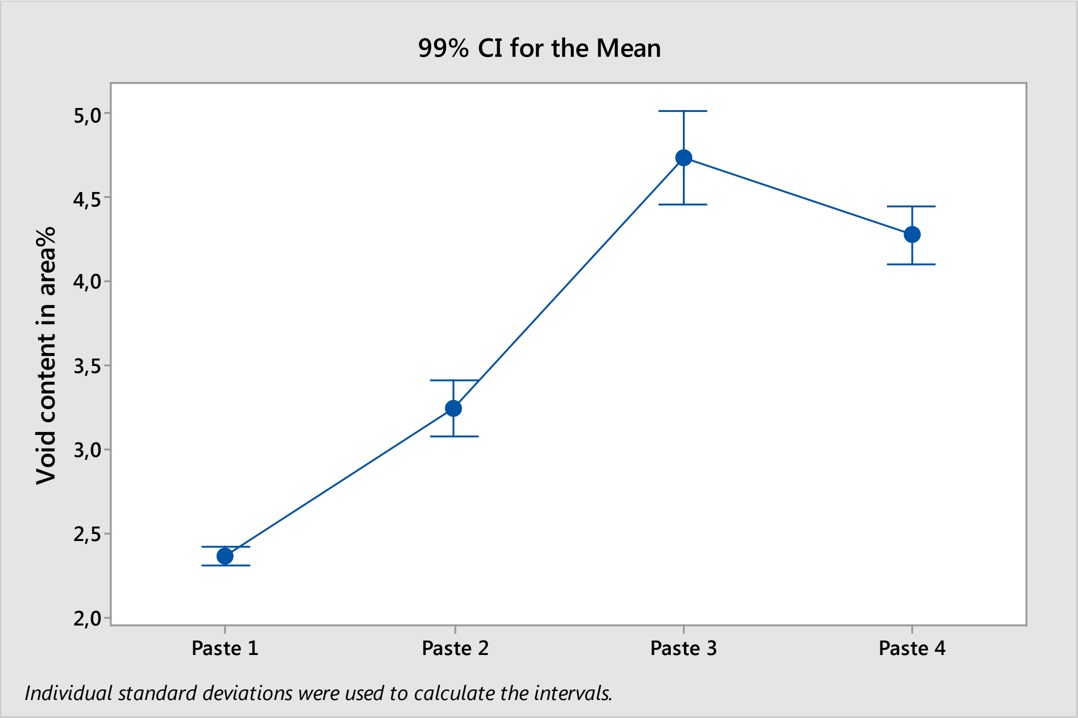


FORWARD THINKING
FOR TOMORROW'S TECHNOLOGY

Results and Discussion: Voiding

Exemplary X-ray pictures





99 % confidence intervals for the void averages of each paste

Average Void Area - 10 % criterion

- 1-sample t-test: Null hypothesis assumes that there are no significant differences between the void area limit and the sample void mean, whereas the Alternative hypothesis assumes a significant difference, in the specific case a value below 10 %

One-Sample T: Paste 1; Paste 2; Paste 3; Paste 4

Test of μ = 10 vs < 10

Variable	N	Mean	StDev	SE Mean	99% Upper Bound	T	P
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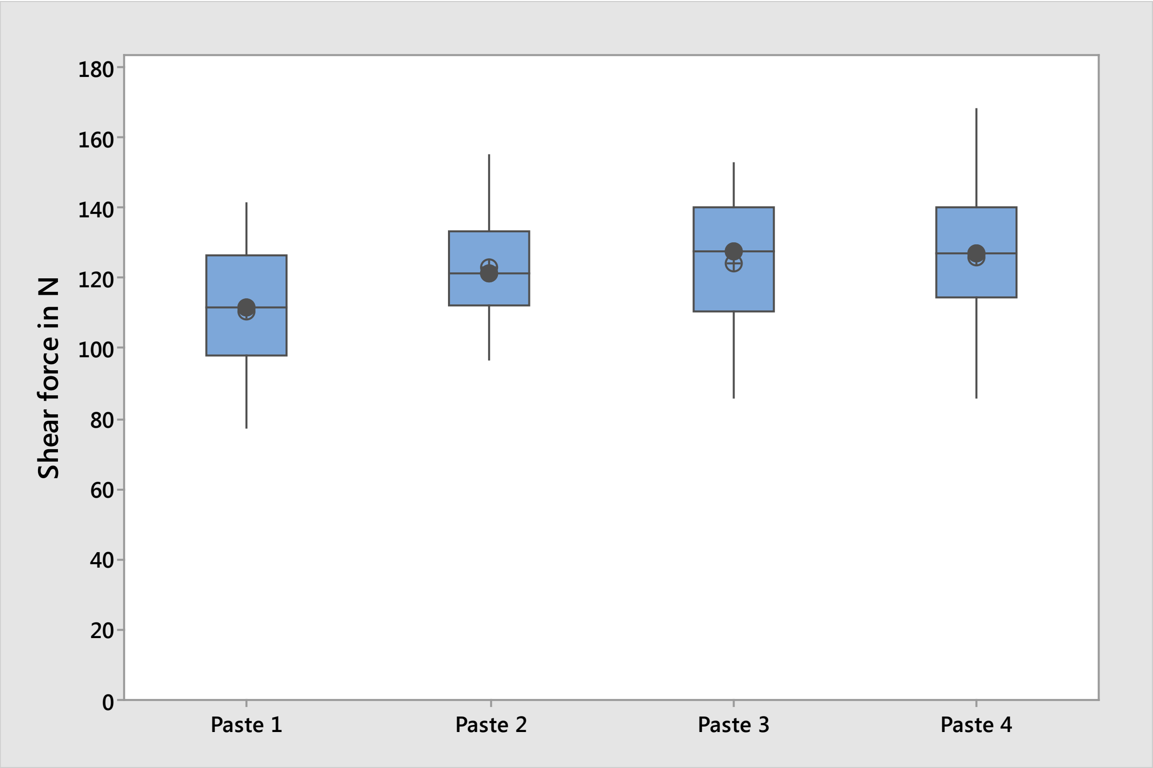
- **Result:** All pastes fulfill the customer requirement of a average area void content of below 10% (null hypothesis rejected)

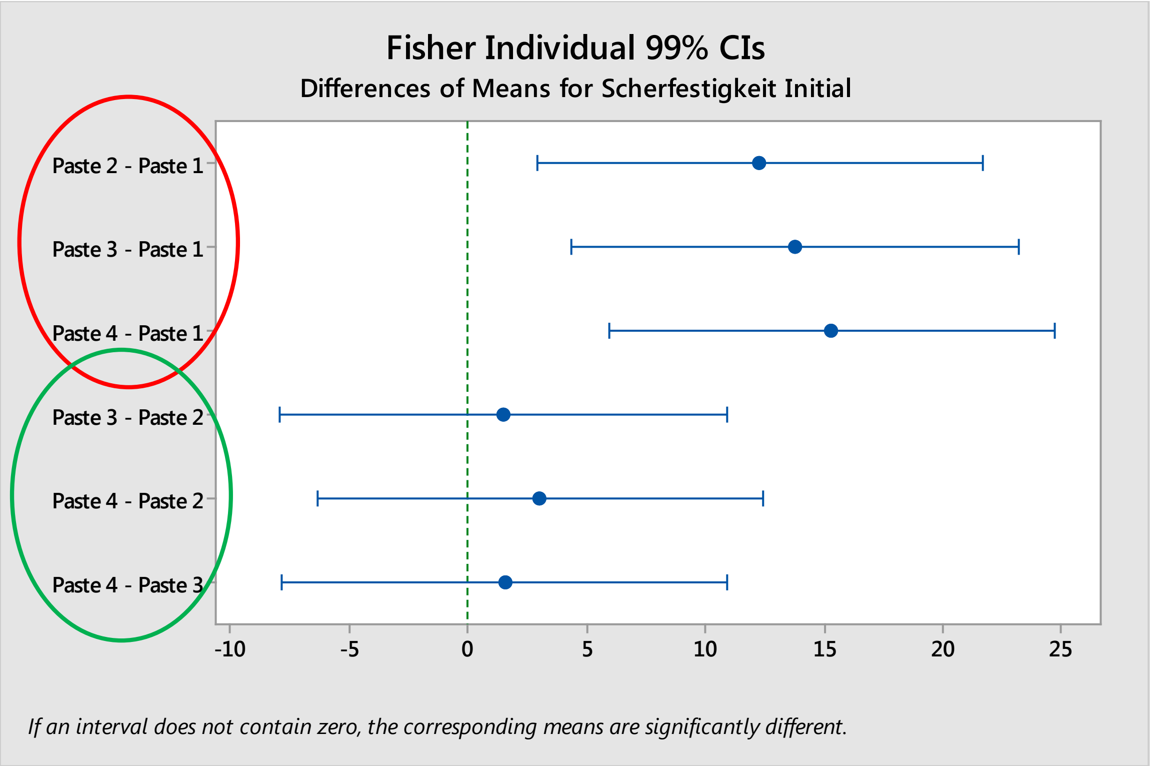
Results and Discussion: Shear Strength

- Measured shear force for all pastes (as soldered) <u>seem</u> to be quite comparable
- Shear values are normally dis-tributed, all initial shear force values show similar variances
- ANOVA method used for analyzing the differences between the materials



Shear Strength - ANOVA

- Initial shear forces significantly different for Paste 1
- Pastes 2, 3 and 4 show com-parable shear forces (as soldered)



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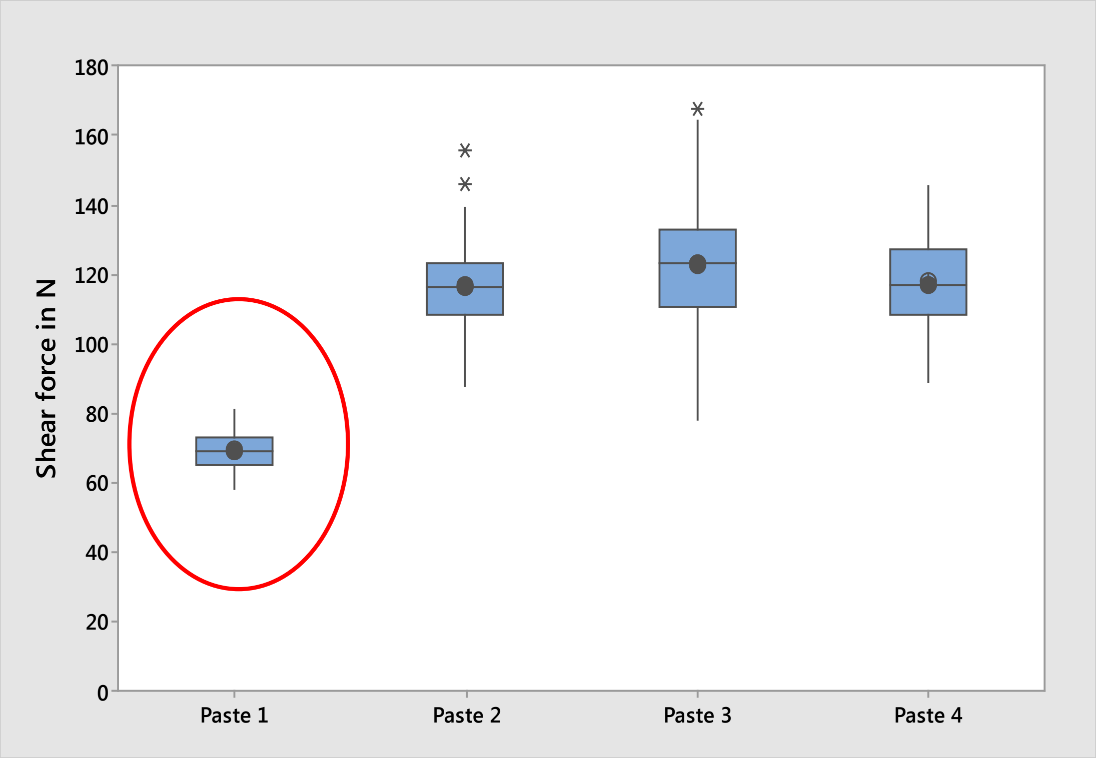
Paste 1	48	110,64	B

Means that do not share a letter are significantly different.

Thermal shock - Shear Strength

- Strength of all alloys decreases
  - Drops of the measured mean shear force values between 5 % and >35 %
  - Variances for all pastes increased
- Most significant changes for paste 1 (SAC)
  - Shear force drop of more than the tolerable 35 %
  - As a consequence it can be stated, that Paste 1 (SAC 305) is not acceptable for this OEM



Shear Strength - 35 % drop criterion

- Three different limits (35 % drop)
- 1-sample t-test
  - Null hypothesis: no significant differen-ces between the shear force limit and the sample shear force mean
  - Alternative hypothesis: shear force drop of less than 35 %

One-Sample T: Paste 2

Test of μ = 79,9 vs > 79,9

Variable	N	Mean	StDev	SE Mean	99% Lower Bound	T	P
Paste 2	46	117,29	12,76	1,88	112,76	19,88	0,000

One-Sample T: Paste 3

Test of μ = 80,8 vs > 80,8

Variable	N	Mean	StDev	SE Mean	99% Lower Bound	T	P
Paste 3	46	122,94	18,19	2,68	116,47	15,71	0,000

One-Sample T: Paste 4

Test of μ = 81,8 vs > 81,8

Variable	N	Mean	StDev	SE Mean	99% Lower Bound	T	P
Paste 4	46	118,44	14,18	2,09	113,40	17,52	0,000

- **Result:** Paste 2/3/4 fulfill the customer requirement of a shear force drop of less than 10 % (null hypothesis rejected)

Conclusions - Voiding

- Lowest voiding for paste 1 (SAC with Flux 1), followed by Paste 2 (Alloy 3 with Flux 1)
- Combinations of Flux 2 with Alloy A and Alloy 3 result in significantly higher voiding
- Void criterion (average void area below 10 %) fulfilled by <u>all</u> examined combinations (statistically significant)

Conclusions – Shear strength

- Initial shear strength of CR1206 components is comparable for Alloy A and Alloy 3, <u>independent</u> of the flux chemistry
- SAC standard solder shows significantly lower initial values
- After 1500 shock cycles, all three Alloy A and Alloy 3 containing solder pastes show shear force drops of about 5 % maximum
- The 35 % shear force drop criterion cannot be fulfilled by solder Paste 1 (SAC), whereas the Alloy A and Alloy 3 containing pastes pass the reliability evaluation without problems

Conclusions - Overall

Paste 2 (Alloy 3 combined with Flux chemistry 1) slightly favorable due to best combined performance (considering void formation as well as mechanical stability):

- Exhibits low voiding (significantly lower than Paste 3 and Paste 4)
- High initial shear forces (equal to Paste 3 and 4) and relatively stable shear forces after thermal cycling (drop only 5 %) with low variations