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RESEARCH ARTICLE

Methodologies to Improve Experimental Research Processes in Soldering Technology

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Abstract

This article introduces the possibility of using optimization methods Failure Mode and Effect Analysis (FMEA) and Design of Experiments (DOE) to improve experimental research processes. The FMEA methodology was implemented in the pre - experimental phase of the experiment. Potential risks has been established by FMEA methodology. Risks that should affected the overall result of the experiment have been determined in brainstorming process. These risks were tested during the experiment and data were evaluated by DOE and Student's t-test method. The results of this research confirmed that proposed methodology helped to verify the major risks which have significant impact to results of an experiment.

Keywords

DOE, FMEA, soldering, plasma cleaning, factor analysis

1 Introduction

This article is aimed at verifying the use of optimization methods commonly used to optimize manufacturing processes to improve experimental research processes. The experiment is an essential part of scientific research. Each experiment has three phases: pre-experimental planning, execution and finally the analysis of collected data. The aim of the experiment is to verify certain predetermined hypothesis. For this reason, scientific experiments should be repeatable and therefore verifiable. The experiment is actually a pre-planned process in which the researcher can change the input conditions and parameters. It is therefore a process of factors combination testing that will affect the final result of the experiment. It would be appropriate to reduce the duration of the experiment and to prevent defects and testing of all factors. That means only test factors with the greatest influence on the experiment are tested. [1]

The impact of cleaning methods on mechanical shear strength of the solder joints on the rigid substrate flame retardant (FR- 4) was selected to verify the suitability of optimization methods using to experiment evaluating. After the phase of the experiment results were evaluated according to the Design of Experiments (DOE) methodology, which is used to determine the important factors and Student's t-test, method of mathematical statistics. J.C.F. de Winter presented in his article, e.g [2], that the t-test can be applied if the researcher conducts research with an extremely small sample size ($N \leq 5$) and also in case of unequal variances or unequal sample sizes. The evaluated factors were compared with selected factors by Failure Mode and Effect Analysis (FMEA) methodology. At the end of the experiment there was evaluation whether it is useful and appropriate to use FMEA methodology for determining factors that should be tested.

FMEA methodology is currently used to optimize not only manufacturing processes, but also in new product concepts. Currently, there are three basic types of FMEA methodology. These types include: System FMEA, Process FMEA and Design FMEA. These types differ on the basis of what is to be assessed. System FMEA is focused on entire system. Conversely Design FMEA assesses the risks associated only with

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product design. The last type is the Process FMEA, which is used for assembly or manufacturing processes. [3]

FMEA methodology is based on identifying potential risks of specific product or process. It is therefore an analytical tool used to evaluate the various failure modes and their potential consequences, which may be at a given statement or process occurrence. [4] The various failure modes and their potential consequences are determined using a brainstorming by team of experts, because it is necessary that they have enough experience and knowledge of the tested product or process for the proper determination of potential defects. [5] A team of experts then classifies individual risks in terms of three criteria - the severity, occurrence and detection. Rating criteria are numeric on a scale from 1 to 10. Risk priority number (RPN) is then determined on the basis of the evaluated criteria.

$$\text{RPN} = \text{Severity} \times \text{Occurence} \times \text{Detection} \tag{1}$$

RPN determines the degree of risk and must be assigned to each of the potential failures. The higher number RPN represents a higher risk of defects. [6]

For the analysis of the various failure modes and their potential consequences could be applied Ishikawa diagram also called as Fishbone diagram. This diagram is simple graphic method for root cause analysis of any problems. The possible causes of the solved problem are divided into pre-defined categories. This arrangement creates a diagram which resembles the skeleton of a fish. Typical pre-defined categories include Method, Material, Machines, People, Environment and Measurement, etc. [7]

Another methodology used to determine the most important factors was the methodology DOE. This methodology is the next tool commonly used for process improvement. The main aim of the methodology is not only detection of more and less significant factors that influence response but also to better understand their impact on the product or process. The first and most important phase of DOE is pre-experimental planning. During this phase the problem is defined and factors are selected, with their levels and ranges. Incorrect definition of the problem leads to useless data. The next steps are execution of the experiment and finally data evaluation by statistical analysis. [8]

The Student’s t-test was used for more accurate comparison if the selected factors were the most significant for the experiment. Student´s t-test is the most common type of t-test used for statistical testing hypotheses. Student’s t-test is two-sample t-test based on assess of statistical divergence between two groups. The standard deviation represents statistical divergency between two groups. [9] The result of Student’s t - test is called p-value. This value determines whether the difference between the groups are significant or not. P-value associated with the significance level is also denoted as alpha, its nominal value is usually 5%. If the calculated p-value is less than 5% means that the null hypothesis is rejected and the difference between the compared groups is statistical significance. Conversely, if the p-value is greater 5% then there is no statistical significance between the compared groups. [10]

2 Experimental

Process where connection of components with substrates is performed is called soldering. Lead-free solder alloys are usually used in soldering. The main factor that influences the mechanical and electrical properties is solderability. That is the reason why the experiment was aimed at improving the solderability. Solderability is primarily affected by solder alloys, thickness of oxide layer, material of solder pads, surface roughness and soldering atmosphere. [11] To reduction of oxides from solder pads are used chemical cleaners. The most common chemical cleaners are fluxes, but disadvantage of these type of cleaners is that fluxes could contaminate the PCB and this contamination reduce reliability of PCB. [12]

The hypothesis of the experiment was that one of the possible solutions to improve solderability is cleaning soldering pads before soldering process. During the first part of the planning phase of the experiment necessary materials as well as procedures were determined. In the second part of this planning phase FMEA methodology was used. The methodology should help researchers to identify the potential risks that may occur during the experiment but also to determine which factors should be tested on the basis of the calculated RPN. Procedure for applying the methodology had three parts - determining the potential risks, evaluation by FMEA methodology and discussion about the results.

2.1 Pre-planning phase of performed experiment

At the first step the substrate flame retardant (FR–4) was cleaned by one of the selected methods. At the next step one of solder pastes was applied to the printed circuit board (PCB). After application of the solder paste, 10 chip resistors with size 0805 were mounted to each sample. The samples were reflowed by continuous reflow oven Mistral 260. The last step of the experiment was measurement of mechanical properties of solder joint. The mechanical shear strength test was performed by the device LabTest 3.030.

2.2 FMEA methodology

In the second part the FMEA protocol was made by experimenters. Individual risks that may occur during the experiment were chosen by Ishikawa diagram and brainstorming. Brainstorming is another analytical technique. During a brainstorming the team of experts discusses possible solutions for the problem. The possible causes of the solved problem were divided into three categories - Material, Experiment Process Parameters and Human Factor. The Ishikawa diagram for the selected experiment is shown in Fig. 1. The FMEA worksheet was compiled based on this diagram. The Table 1 shows the FMEA worksheet prepared for the experiment evaluating the

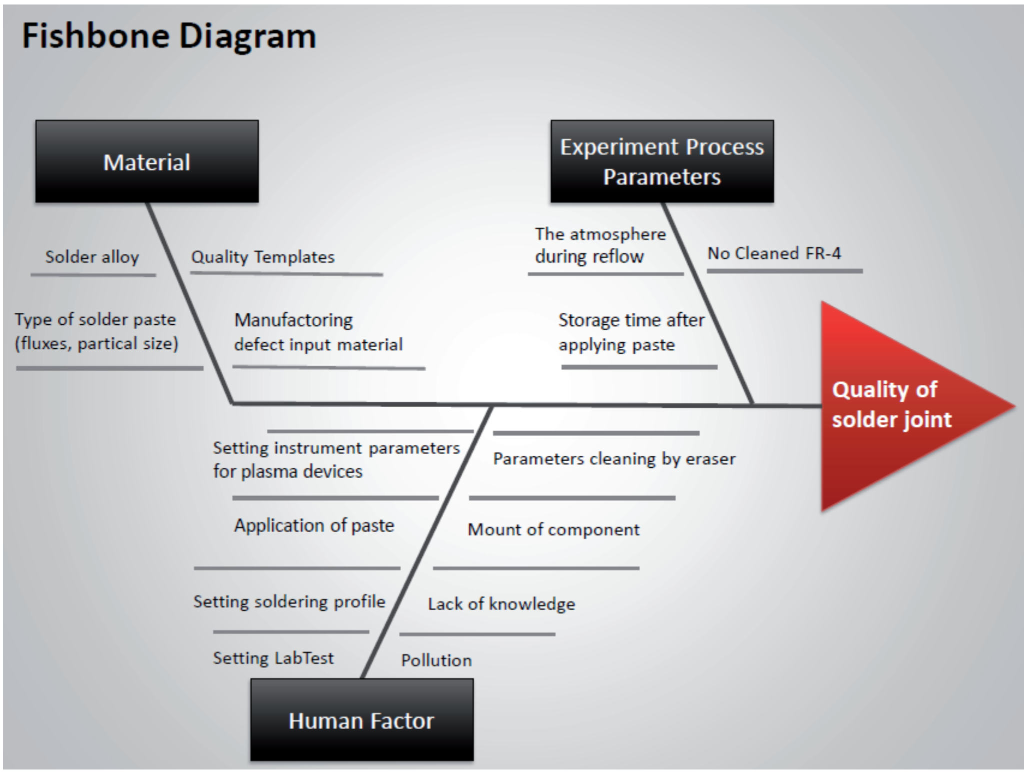

Fig. 1 The Ishikawa diagram of the experiment

impact of cleaning methods on mechanical shear strength. After the team determined the potential failure modes that may occur during the experiment, the risks were divided into three same basic categories as Ishikawa diagram. These categories were Material, Human factor, Process parameters of the experiment. After dividing the potential failure modes potential effects and causes of failure were identified.

At the conclusion the individual risks were numerically classified according to three criteria and risk priority number was evaluated. The highest RPN had the following factors – quality templates, solder alloy, setting soldering profile, lack of knowledge, no cleaned FR-4. For testing these factors were selected – four different type of solder paste and three methods for cleaning surface of substrate. Other factors with high RPN weren't selected because the testing of these factors would be difficult.

2.3 Performed experiment

Rigid printed circuit board FR-4 with copper pattern was used for the experiment evaluating the impact of cleaning methods on the mechanical shear strength of soldered joints (see Fig. 2).

For soldering four different solder alloys were used SnBi, SnPbAg, SAC305 and SCANGe. For each alloy two different volumes were used, 100% and 47%. Because the purity of the substrate surface has an effect on solderability, three methods were used for cleaning surface of the substrate. Three different cleaning methods were used on the copper pattern - without cleaning, eraser and plasma.

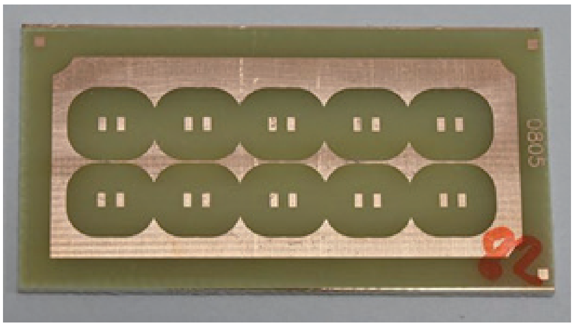

Fig. 2 Sample with copper conductive pattern for mounting of 0805 chip components

At last pulse atmospheric plasma arc technology (PAA®) with a whirlpool in the nozzle was used for cleaning and activation pad. The use of atmospheric plasma for industrial applications has become widespread in many different sectors. The technology is based on pulsed high voltage arc rotating in a combustion chamber. The arc's corona is pushed out of the nozzle by vortex gas flow (see Fig. 3). The vortex gas flow and pulsed high voltage prevents overheating and erosion on the electrodes. Three different nozzles can be used with plasma system – high, medium and low temperature ones. The typical plasma application is cleaning, improving wetting and surface tension of the substrates as well as the applications in the hygiene sector and in medical technology. The lab is equipped with Relyon plasma PB3 + PS2000 system. The setting of 54 kHz discharge frequency, 30L/min nitrogen flow, low temperature nozzle and motion speed above the substrate 150 mm/s was used for the experiment. The nozzle – substrate distance was set to 8 mm.

Table 1 The FMEA worksheet for experiment

Category	Potential Failure Mode(s)	Potential Effect(s) of Failure	Potential Cause(s)	Controls	Severity	Occurrence	Detection	Risk priority number (RPN)
Material	Manufacturing defect input material	Reduced mechanical shear strength	Poor Contractor	Acceptance inspection	8	1	9	72
	Type of solder paste (fluxes, particle size)	Reduced mechanical shear strength	Selected paste	Experiences	3	7	7	147
	Quality Templates	The wrong quantity of solder alloy, Reduced mechanical shear strength	Poor Contractor	Acceptance inspection	6	5	6	180
	Solder alloy	Reduced mechanical shear strength	Selected alloy	Experiences	6	6	5	180
Human Factor	Setting instrument parameters for plasma devices	Exceeding the critical temperature	Experimenter without experience	Experiences	10	3	3	90
	Parameters cleaning by eraser	Change of surface roughness	Experimenter without experience	Experiences	8	2	7	112
	Pollution	Reduced mechanical shear strength	Experimenter without experience	Experiences	5	5	7	175
	Application of paste	Reduced mechanical shear strength	Experimenter without experience	Experiences	8	4	5	160
	Mount of component	Change of mechanical shear strength	Experimenter without experience	Experiences	8	7	2	112
	Setting soldering profile	Change of mechanical shear strength	Experimenter without experience	Experiences	6	10	3	180
	Setting LabTest	Destruction of the test sample	Experimenter without experience	Experiences	8	2	4	64
	Lack of knowledge	Erroneous results	Experimenter without experience	Experiences	10	6	4	240
Process parameters of the experiment	No Cleaned FR-4	Change of solderability	Experimenter without experience	Experiences	10	3	6	180
	Storage time after applying paste	Oxidation of surface	Experimenter without experience	Experiences	6	1	8	48
	The atmosphere during reflow	Oxidation of surface	Experimenter without experience	Experiences	8	4	4	128

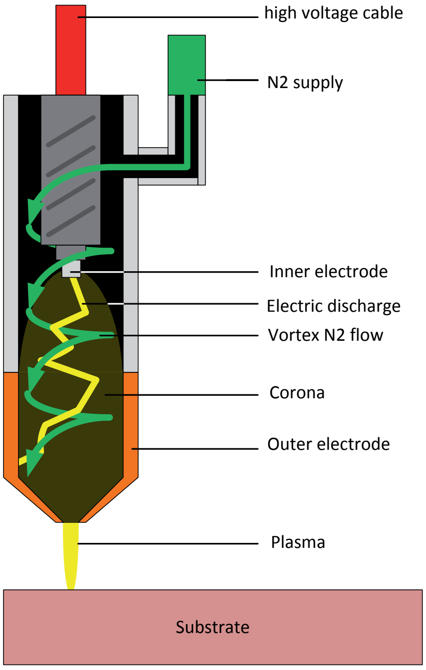

Fig. 3 Atmospheric plasma schematics

Stencil printing was used for application of solder alloys onto the substrates. The thickness of the stencil was 120µm and the apertures were reduced by 5% in length and width due to solder pads for the first volume. That was marked as a 100% of paste. The thickness of the stencil was 80µm and the apertures were reduced by 20% of the length and width due to solder pads for the second volume. This volume is 47% of paste due to the first volume. Then 10 chip resistors with size 0805 components were mounted to the PCB and the samples were reflowed by continuous reflow oven. At the last step the samples were tested by the mechanical shear strength test. The component is pushed with force by thorn of device. This process continues until the destruction of the join and component shear-off from the PCB.

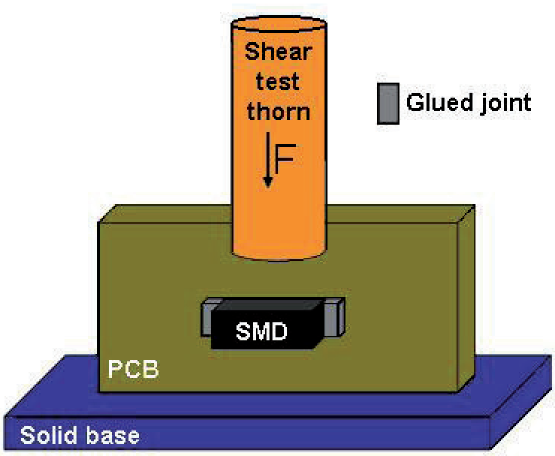

Fig. 4 The principle of the shear strength test

3 Results

After the experiment, the determined data were analytically evaluated and used for creation of the boxplot diagrams. In the Tables 2 to 5 there are calculated average values of mechanical shear strength. The average values were calculated from measurements of the mechanical shear strength of 10 chip resistors with size 0805 components that were mounted to the PCB. In Fig. 5 there is the first boxplot diagram that shows maximal force when shearing-off 0805 chip resistors soldered on FR-4 for different types of cleaning. The tin-bismuth solder paste was chosen as the first paste. This alloy has 42% Sn-58% Bi composition and is produced by Shenmao Technology Inc. This alloy was used in two different volumes 47% and 100%.

The diagram indicates that maximal force needed to shear-off the component soldered by SnBi with 100% and 47% of paste volume.

Table 2 The maximal force - solder paste SnBi

Paste volume	No cleaning	Plasma	Eraser
100%	50,526	52,568	53,462
47%	44,641	45,289	45,238

The best mechanical shear strength has the sample with 100% of quantity cleaned by eraser. The best result for the reduction of solder paste quantity to 47% was cleaned by plasma.

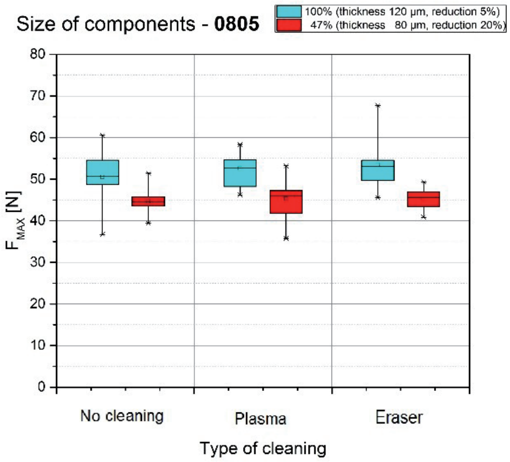

Fig. 5 Maximal force for disruption of the joint with solder paste SnBi

The second boxplot diagram that shows another paste SnPbAg used for the experiment is shown in Fig. 6. The composition of the paste was 62.5% Sn-32.5% Pb-1% Ag and the paste was produced by BalverZinn (Cobar).

Table 3 The maximal force - solder paste SnPbAg

Paste volume	No cleaning	Plasma	Eraser
100%	49,519	51,480	51,094
47%	42,194	44,368	45,067

Again this alloy was used for experiment in two different volumes 47% and 100%. The best result for the reduction of solder paste quantity to 100% was cleaned by plasma. The best mechanical shear strength has the sample with 47% of quantity cleaned by eraser.

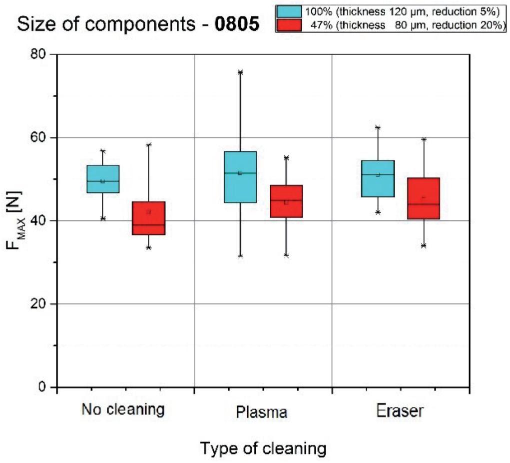

Fig. 6 Maximal force for disruption of the joint with solder paste SnPbAg.

Next solder paste used for the experiment was SAC305 with composition 96.5%Sn-3%Ag-0.5%Cu produced by Balver Zinn (Cobar). In Figure 7 there is the boxplot diagram for this alloy.

Table 4 The maximal force - solder paste SAC305

Paste volume	No cleaning	Plasma	Eraser
100%	39,481	37,687	42,951
47%	35,339	36,417	39,908

The graph indicates that the maximal force needed to shear-off the component was used for samples soldered by SAC305 with 100% of paste volume and cleaned by eraser. Conversely, if the quantity of solder paste was 47 %, then it was necessary to develop the biggest strength for the samples cleaned by eraser.

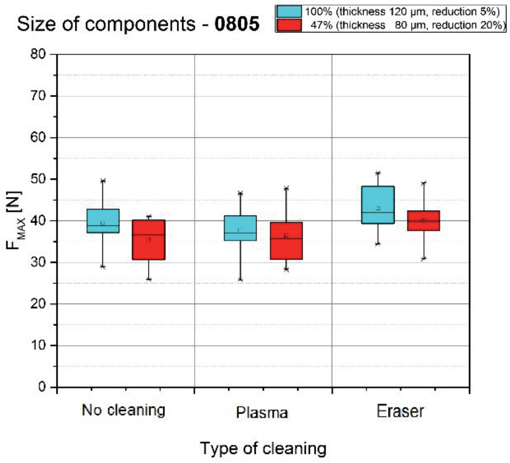

Fig. 7 Maximal force for disruption of the joint with solder paste SAC305

Last solder paste used for the experiment was SCANGe. This alloy has 98.3%Sn-0.7%Cu-1.0%Ag-0.05%Ni-0.005%Ge and is produced by Balver Zinn (Cobar). The diagram indicates that maximal force needed to shear-off the component soldered by SCANGe with 100% and 47% of paste volume. The best mechanical shear strength has the sample with 100% of quantity cleaned by eraser. The best result for the reduction of solder paste quantity to 47% was cleaned by plasma.

Table 5 The maximal force - solder paste SCANGe

Paste volume	No cleaning	Plasma	Eraser
100%	46,078	45,369	46,861
47%	38,310	41,572	38,227

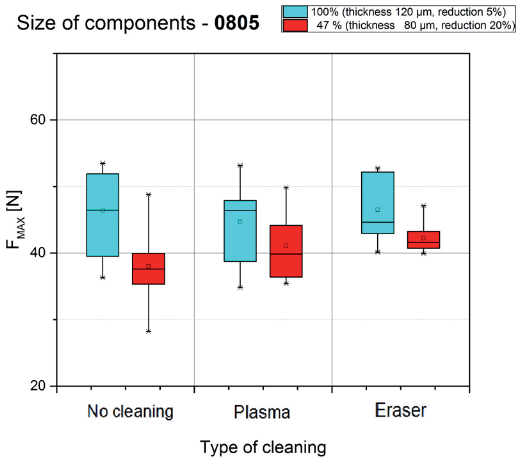

Fig. 8 Maximal force for disruption of the joint with solder paste SCANGe

Finally, for verification of the suitability of the methodology FMEA the DOE and Student's t-test have been used for evaluation of data. DOE methodology results are shown in the Fig. 9. The graph shows that the solderability is actually affected by factors such as amount and type of alloy, but also the type of cleaning methods. The important factors that influence the mechanical shear strength are cleaning of samples, type of solder paste and solder paste quantity.

For a more accurate evaluation of the measured data the statistical tool called as Student's t – test have been used. This method assess statistical different between two groups. This comparison is done on the basis of standard deviations of the two selected groups and the result is called p-value. The p-value of less than 5% increases the statistical significance of the test groups. Statistical significance is divided into three categories $P<0.05$ (*), $P<0.01$ (**) and $P<0.001$ (***). If $P<0.001$ (***) then there is the most statistical significance. The comparison between two applied cleaning methods is shown in Table 6. The results in Table 6 were calculated in a spreadsheet.

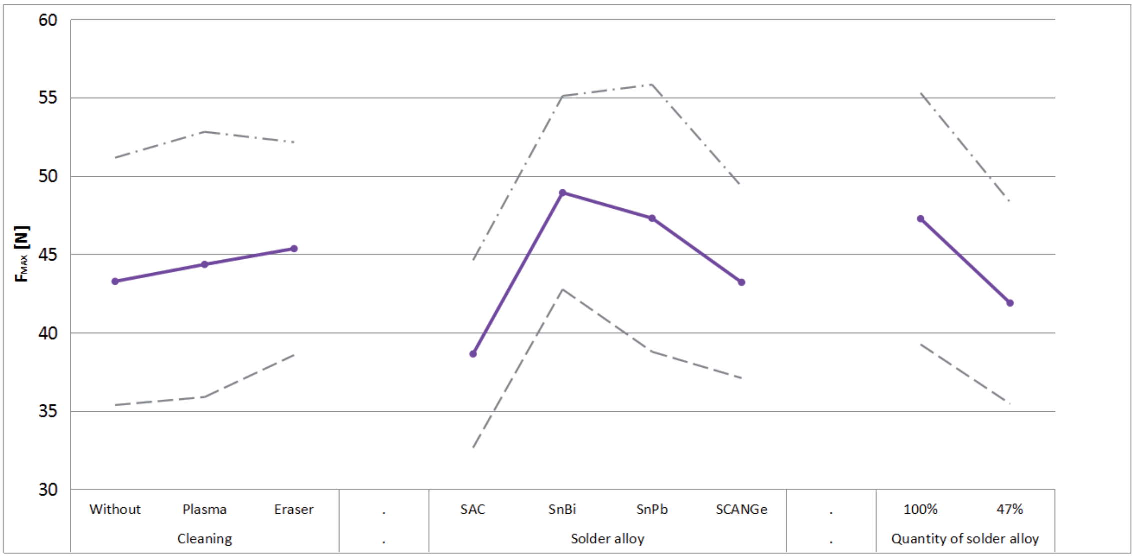

Fig. 9 Influence of factors on the mechanical shear strength from DOE method

Table 6 T-test of cleaning methods

Selected cleaning methods	p- value	Statistical Significance
Without cleaning/Plasma	52.93%	Insignificant
Without cleaning/Eraser	4.63%	Significant (*)
Plasma/Eraser	19.12%	Insignificant

Table 6 shows that the statistical t-test confirmed significance at the comparison methods without cleaning and eraser. This result is consistent with the result of the methodology DOE, which identified cleaning substrate FR-4 by eraser as the most important factor. In terms of the methods used for cleaning substrate FR-4 further statistical test was not confirmed any other significance between the compared groups. For this reason in Table 7 the confidence intervals stated at the 95% confidence level were determined for individual cleaning methods.

Table 7 Confidence interval

Selected cleaning methods	CI
Without cleaning	43.26±1.71
Plasma	44.34±1.71
Eraser	45.35±1.52

Confidence intervals indicate that the intervals of the individual types of cleaning method in some cases may overlap. Comparing of remaining two groups were insignificance for this reason. Additionally t-test for solder paste was evaluated. This evaluation is shown in Table 8.

Table 8 T-test of solder paste

Solder paste	p-value	Statistical Significance
SAC/SnBi	0.00%	Significant (***)
SAC/SnPb	0.00%	Significant (***)
SnBi/SnPb	24.09%	Insignificant
SnBi/SCANGe	0.00%	Significant (***)
SAC/SCANGe	0.00%	Significant (***)
SnPb/SCANGe	0.80%	Significant (***)

Table 8 shows that the statistical t-test confirmed significance of the comparison of solder paste SAC/SnBi, SAC/SnPb SAC/SCANGe. P-value in these cases was less than 0.001. This result is consistent with the result of the methodology DOE. Their comparison in terms of the statistical t-test of significance is shown in Table 9.

Table 9 T-test of the quantity of solder paste

Quantity of solder paste	p-value	Statistical Significance
100%/47%	0.00%	Significant (***)

The result of t-test of the quantity of solder paste demonstrates that the significance of the comparison was confirmed by the statistical t-test. P-value was less than 0.001. Based on these findings, it was proved that the FMEA methodology can be used for improving experimental research processes.

4 Discussion

The aim of the article is to assess whether it is suitable for pre-experimental phase to use the FMEA methodology for identifying parameters that would have a high impact on the entire outcome of the experiment and therefore should be tested. These parameters were chosen based on the calculated

RPN. After identification of parameters with the largest RPN brainstorming was performed and factors with potential influence on the mechanical shear strength were chosen and tested.

These factors were additionally evaluated by the DOE methodology and by the Student's t-test. DOE determined whether the selected factors actually have significant influence and the purpose of the article was to evaluate whether they actually have significant influence on the mechanical shear strength. Subsequently, the results of the experiment were evaluated by the method of statistical testing hypotheses also called t-test. Final results of DOE methodology and statistical t-test were compared. The results show that FMEA methodology could be used. The processes are dependent on human factor as knowledge and experience. To eliminate these factors, the researcher should established own knowledge database. Figure 10 presents recommended process for research and experiment.

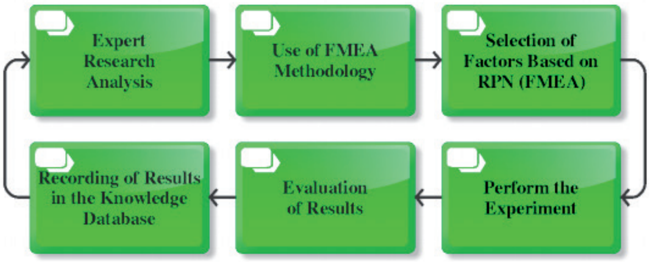

Fig. 10 Recommended process

First, the researcher should make a preliminary expert research analysis. This analysis should include a study of the theory of the issue from the available literature. Furthermore, the researcher should focus on already performed similar experiments including their procedures and results in this phase. In the next step, the researcher should use FMEA methodology and create FMEA worksheet for the experiment or team of experts can fill Ishikawa diagram by the brainstorming and use the diagram to filling of FMEA worksheet. Based on RNP of FMEA methodology should be selected factors with the greatest influence on the results of the experiment. Consequently, the experiment should be performed and then its results should be evaluated. Finally, all results and findings should be recorded in the researcher knowledge database. Repeating the experiment contributes to more precise results and improves the skills and abilities of the researcher.

5 Conclusion

This article is focused on the possibility of using optimization methods Failure Mode and Effect Analysis (FMEA) and Design of Experiments (DOE) to improve pre-experimental phase of research processes in soldering technology. The possibility of using FMEA and DOE methodology was confirmed by the method of statistical testing hypotheses also called t-test. The main benefits of using this process is to reduce error and time-consuming. Another advantage is the extension of the knowledge database researcher. In the future it could be this process of improvement extended to other areas of research and put into practice as standard. But it necessary to perform additional experiments into practice.

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