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Factorial design and design of experiments for developing novel lead free solder alloy with Sn, Cu and Ni

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Abstract. Inherent toxicity makes lead a banned material in solder alloy making process. Lead-tin alloy was a favorable alloy used for soldering in electronic packaging manufacturers. As a result of the ban on lead, electronics package industries were looking for novel lead free alloys which can substitute the conventional Sn-Pb alloy. Many alloys were discovered by the scientists. None of them were able to substitute the Sn-Pb alloy and become the market leader. In this paper a new composition with Sn, Cu and Ni is made to analyze which can potentially replace the lead containing solder alloy. Using the design of experiments method, the optimized composition of Cu and Ni is predicted. The full factorial design of experiments with two replications is used to find the optimized composition. Melting temperature, contact angle and hardness were taken as the critical output parameters. Results obtained shows that the optimum composition of Cu and Ni are 1 and 1% by wt.

Keywords: Design of experiments / factorial regression / lead free / solder alloy

1 Introduction

Soldering is a metallurgical joining method without melting the parent metals [1]. Soldering has varied application in the electronics package manufacturing industry. The solder gives electrical and mechanical continuity in the electronic packages [2]. Sn-Pb was the most widely used solder material due to its good solderability. Soldering process has a long history. Only few realized the fact that soldering has been used by human beings thousands of years before. The recovered jewelry from the preserved burial sites found by the archeologists throws light about the soldering process used during olden times. Au-Cu, Ag-Cu and Pb-Cu were the alloys used in those times. The Romans introduced Sn-Pb alloy long back. Sn-Pb (63Sn-37Pb) was extensively used in the modern electronics industry. Due to the inherent toxicity of the lead, it is banned from alloy making [1–3]. Various environmental legislations came to existence to ban lead in alloy making. Waste of Electrical and Electronic Equipment (WEEE), Restrictions on Hazardous Substances (RoHS) by European Union (EU) are examples [4]. Many countries also followed the same path. Therefore electronic industries shifted to lead free solder alloys. Need for new lead free alloys which can replace the Sn-Pb alloy aroused. Researchers around the world started searching for new lead free solder alloy. The desirable qualities of new alloys are low melting point, good hardness, low contact angle, low cost, availability etc. Many compositions were discovered as a replacement for Sn-Pb alloy. Some major compositions include Sn-Bi-Ag [5], Sn-Bi [6], Sn-Zn [7], Sn-Ag-Cu [8,9], Sn-Cu [7], Sn-Zn-Bi [5], Sn-Cu-Ni [10,11], Sn-Cu-Bi [12].

SAC (Sn-Cu-Ag) alloys were having good acceptance (e.g. SAC305 and SAC405. But the amount of silver adds the cost of the alloy. Some of the SAC alloys incur huge patent costs also. In this paper new composition of lead free solder alloy is developed with Sn, Cu and Ni. The full factorial design of experiments with two replications is used to find the proper combination. Assuming a location dispersion model, dispersion effects tests were conducted and presented with two level factorial experiments [13]. Here the melting temperature, hardness and the contact angle are taken as critical output parameters. The criteria for the optimized composition are, the melting point should be low, hardness should be high and the contact angle should be minimum.

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Table 1. Output from the 8 runs conducted.

Experiment Runs	Cu (% by wt.)	Ni (% by wt.)	Melting Temp(°C)	Contact Angle(°)	Hardness (Hv)
1	1	0.5	239.12	38.66	8.1
2	1	0.5	238.76	39.1	8.22
3	1	1	232.2	36.75	16.1
4	0.5	1	240.12	38.12	10.2
5	0.5	0.5	238.12	43.22	7.9
6	0.5	0.5	238.34	43.58	7.6
7	0.5	1	240.34	38.48	10.39
8	1	1	232.45	36.82	16.2

Table 2. Analysis of variance.

Source	DF	Adj SS	Adj MS	*F*-Value	*p*-Value
Model	3	73.6422	24.5474	679.75	0
Linear	2	22.5906	11.2953	312.78	0
Cu	1	17.3911	17.3911	481.58	0
Ni	1	22.536	22.536	624.04	0
2-way interactions	1	37.1091	37.1091	1027.6	0
Cu × Ni	1	37.1091	37.1091	1027.6	0
Error	4	0.1445			
Total	7	73.7867			

2 Methods

2.1 Design of experiments (DoE) and full factorial design

Design of experiments is a methodology proposed by Robert Fischer in 1935. This methodology is used to design any job that plans to explain the variations under conditions. There are hypothesis to reflect this variations. The change in one or more independent variables is generally hypothesized to result in a change in one or more dependent variables. The method also identifies control variables that must be kept constant, to avoid the external factors to affect the results. The main concerns in the DoE are the reliability, replicability and validity. In this paper, with the aid of design of experiments, the optimum composition of new lead free solder alloy is predicted. This new composition contains Sn, Cu and Ni. Full factorial and fractional factorial designs at 2 and 3 levels are the most commonly used experiments designs in the manufacturing industry field. Factorial designs help the researcher to analyze the joint effect of the design or process parameters on an output or response. A factorial design can be full factorial or fractional factorial. This paper discusses the full factorial design at level 2. When the number of process parameters is less than or equal to four, full factorial design is a good choice. This is done at early stage of experiment work. For studying k factors at 2 levels, number of experiments required is 2^k. The response is almost linear over the range of the factor settings selected. This is one of the assumptions in this analysis [14].

2.2 New lead free solder alloy

The aim of this project is to find a new lead free solder alloy with Tin (Sn), Copper (Cu) and Nickel (Ni). The Sn is considered to be the base metal. Small amount of Cu and Ni is added to make the required alloy. With the addition of Cu and Ni, the melting temperature and contact angle is expected to be decreased. The hardness of the alloy is expected to be increased. This analysis has to be done prior to our experiments. An optimum composition of Cu and Ni is studied and tried to predict so that the melting point and contact angle is lower. This alloy should exhibit good hardness properties.

3 Results and discussions

3.1 Design of experiments

The experiment was run with two factors (F), which is Cu & Ni composition and two levels (L), which is 0.5% and 1% by wt. for Cu, 0.5% and 1% by wt. for Ni. 8 runs were conducted with 2 replications, as per the formula:$(L)^F$, $(2)^2 = 4 \times 2$ replications = 8. Three responses were recorded as output out of each run, Melting temperature, Contact Angle & Hardness. It is shown in the Table 1.

3.2 Factorial regression: melting temp(C) versus Cu, Ni

Factorial regression of melting temperature versus Cu and Ni were conducted. The analysis of variance is shown in the Table 2. The model summary is tabulated in the Table 3. The coded coefficients details are shown in the Table 4.

Table 3. Model summary.

S	R^2	R^2 (adj)	R^2 (pred)
0.190022	99.80%	99.66%	99.22%

Table 4. Coded coefficients.

Term	Effect	Coef	SE Coef	T-Value	p-Value	VIF
Constant		226.905	0.672	337.72	0	
Cu	37.3	18.65	0.85	21.94	0	10
Ni	42.46	21.23	0.85	24.98	0	10
Cu × Ni	−68.92	−34.46	1.07	−32.06	0	19

Table 5. Analysis of variance.

Source	DF	Adj SS	Adj MS	F-Value	p-Value
Model	3	48.6096	16.2032	283.21	0
Linear	2	13.1941	6.5971	115.31	0
Cu	1	11.3251	11.3251	197.95	0
Ni	1	13.1382	13.1382	229.64	0
2-way interactions	1	4.515	4.515	78.92	0.001
Cu × Ni	1	4.515	4.515	78.92	0.001
Error	4	0.2289			
Total	7	48.8385			

Table 6. Model summary.

S	R^2	R^2 (adj)	R^2 (pred)
0.239191	99.53%	99.18%	98.13%

Regression Equation in Uncoded Units is given by

$$\text{Melting Temp (C)} = 226.905 + 18.650\,\text{Cu} + 21.230\,\text{Ni} - 34.46\,\text{Cu} \times \text{Ni}. \quad (1)$$

3.3 Factorial regression: contact angle versus Cu, Ni

Factorial regression of contact angle versus Cu and Ni were conducted. The analysis of variance is shown in the Table 5. The model summary is tabulated in the Table 6. The coded coefficients details are shown in the Table 7.

Regression Equation in Uncoded Units is given by

$$\text{Contact Angle} = 56.025 - 15.05\,\text{Cu} - 16.21\,\text{Ni} + 12.02\,\text{Cu} \times \text{Ni}. \quad (2)$$

Table 7. Coded coefficients.

Term	Effect	Coef	SE Coef	T-Value	p-Value	VIF
Constant		56.025	0.846	66.25	0	
Cu	−30.1	−15.05	1.07	−14.07	0	10
Ni	−32.42	−16.21	1.07	−15.15	0	10
Cu × Ni	24.04	12.02	1.35	8.88	0.001	19

Table 8. Analysis of variance.

Source	DF	Adj SS	Adj MS	F-Value	p-Value
Model	3	89.9422	29.9807	1593.66	0
Linear	2	7.8721	3.9361	209.23	0
Cu	1	5.0702	5.0702	269.51	0
Ni	1	1.682	1.682	89.41	0.001
2-way interactions	1	14.824	14.824	787.99	0
Cu × Ni	1	14.824	14.824	787.99	0
Error	4	0.0753			
Total	7	90.0175			

Table 9. Model summary.

S	R^2	R^2 (adj)	R^2 (pred)
0.137159	99.92%	99.85%	99.67%

Table 10. Coded coefficients.

Term	Effect	Coef	SE Coef	T-Value	p-Value	VIF
Constant		10.24	0.485	21.12	0	
Cu	−20.14	−10.07	0.613	−16.42	0	10
Ni	−11.6	−5.8	0.613	−9.46	0.001	10
Cu × Ni	43.56	21.78	0.776	28.07	0	19

3.4 Factorial regression: hardness versus Cu, Ni

Factorial regression of hardness versus Cu and Ni were conducted. The analysis of variance is shown in the Table 8. The model summary is tabulated in the Table 9. The coded coefficients details are shown in the Table 10.

Regression Equation in Uncoded Units is given by

$$\text{Hardness} = 10.240 - 10.070\,\text{Cu} - 5.800\,\text{Ni} + 21.780\,\text{Cu} \times \text{Ni}. \quad (3)$$

Table 11. Parameters.

Term	Effect	Coef	SE Coef	*T*-Value	*p*-Value	VIF
Constant		10.24	0.485	21.12	0	
Cu	−20.14	−10.07	0.613	−16.42	0	10
Ni	−11.6	−5.8	0.613	−9.46	0.001	10
Cu × Ni	43.56	21.78	0.776	28.07	0	19

Table 12. Solutions.

Solution	Cu	Ni	Hardness fit	Contact angle fit	Melting temperature fit	Composite desirability
1	1	1	16.15	36.785	232.325	0.985358

Table 13. Multiple response prediction.

Variable	Setting
Cu	1
Ni	1

3.5 Response optimization: hardness, contact angle, melting temp(C)

The optimized response from the analysis is shown in the Tables 11 and 12. The prediction of the optimized composition is shown in the Table 13 and Table 14. It can be found that the composition of Cu is 1% and of Ni is 1% by wt.

3.6 Main effect plots

The mean response of the level factors can be represented in the main effect plots. This is done by connections made by the lines. It can be interpreted that there is no main effect if horizontal line is present in the diagram. Little deflection from the horizontal direction means that it will significantly affect the response. Lines with higher slope give the information that the magnitude of the main effect is higher. The effect can be defined as the variations in melting temperature, hardness and the contact angle when the factor changes from one level to another level. Calculations are done at minimum and maximum values of each factor [15,16]. The information about the direction of the effect (i.e. Increase or decrease in the average response value) is obtained from the sign of the main effect plot. The strength of the effect is obtained from the magnitude [17]. The main effect diagrams give vital information about the information about the factor influence in the properties like melting temperature, hardness and the contact angle of SCN110. The main effect plots for melting temperature, hardness and contact angle is given in the Figures 1, 2 and 3.

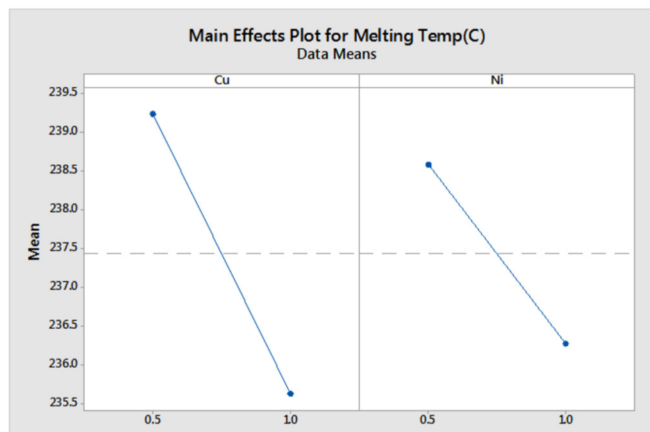

Fig. 1. Mean effects plot for melting temperature.

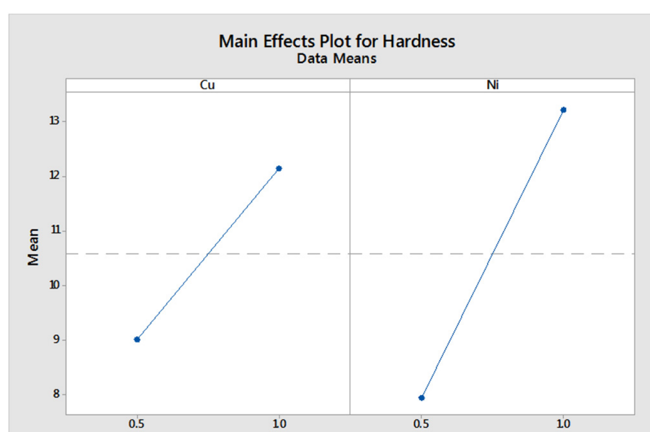

Fig. 2. Mean effects plot for hardness.

3.7 Interaction plots

The behaviors of one variable which is depending on the value of another variable is better understood by interaction plots. An interaction plot displays the levels

Table 14. Best fit solution.

Response	Fit	SE Fit	95% CI	95% PI
Hardness	16.15	0.097	(15.8807, 16.4193)	(15.6836, 16.6164)
Contact angle	36.785	0.169	(36.315, 37.255)	(35.972, 37.598)
Melting temperature	232.325	0.134	(231.952, 232.698)	(231.679, 232.971)

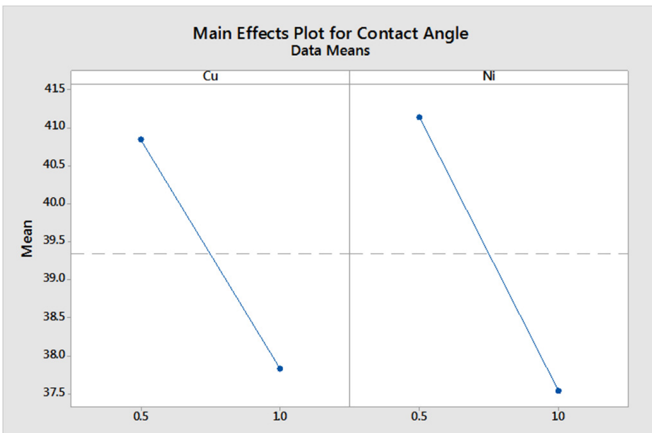

Fig. 3. Mean effects plot for contact angle.

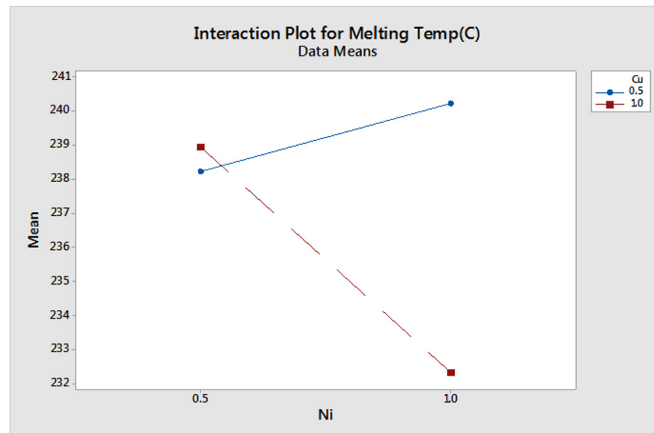

Fig. 5. Interaction plot for melting temperature.

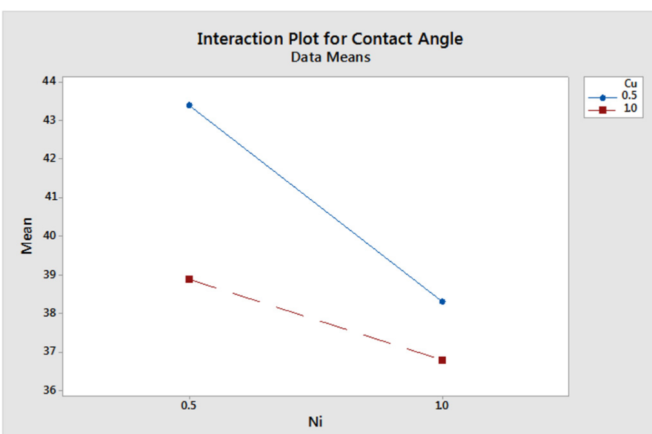

Fig. 4. Interaction plot for contact angle.

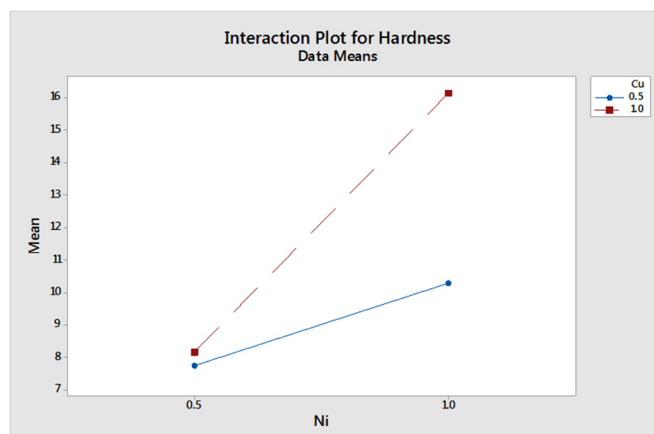

Fig. 6. Interaction plot for hardness.

of one variable on the X axis and has a separate line for the means of each level of the other variable. The Y axis is the dependent variable. The interaction effects are analysed in the regression analysis, ANOVA and DOE. The influence of one factor and a continuous response with the value of the second factor can be identified from the interaction plots [18]. The lines in the diagram can be used to interpret how the relation between the factor and the response are affected. If there are parallel lines, it means that there is no interaction. Non-parallel lines in the diagram interprets that there is interaction occurring. The strength of the interaction is proportional to number of non-parallel lines. Interaction plot for hardness, contact angle and melting temperature is shown in the Figures 4, 5 and 6. It can be interpreted that the influence of these factors are present.

4 Conclusion

Lead cannot be used in solder making process because of its inherent toxicity. New lead free solder alloys are made which can replace the Sn-Pb alloy. This paper, using design of experiments (DoE) checks the use of Sn, Cu and Ni to make a new lead free solder alloy. By design experiments it has been found that, the selected parameters are the most impacting factors for response. The ANOVA evidently shows with its p-values having less than 0.05 that Cu and Ni composition in the alloy is having significant impact in the Melting temperature, Contact Angle and Hardness. The R^2 values of the regression equation is calculated to be more than 95% which denotes that model is best and convey us that there are no other factors impacting the factors. The same can be seen graphically in the Mean effects plot. The Interaction plot shows the interaction between the Cu & Ni with the factors. From the results of Design of Experiments we can infer that Cu-1 and Ni − 1% by qt. is the best optimized composition for the new solder alloy. Therefore Sn-1Cu-1Ni will be an optimum composition of the new lead free solder alloy.

Nomenclature

Adj MS	Adjusted sum of squares
Adj SS	Adjusted mean squares
ANOVA	Analysis of variance
CI	Confident interval
DF	Degree of freedom
DOE	Design of experiments
PI	Prediction interval
p-value	Probability of significance
S	Standard deviation
S E	Standard error
VIF	Variance inflation factor

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