Fort Wayne Metals Solder Project Design of Experiment

By: Jenica Griffiths

Nitinol Intern

Problem:

Many customers have asked about joining Nitinol to itself and Nitinol to other alloys. However, Fort Wayne Metals has not researched or developed this process. Using a WES51 soldering iron, soldered samples were tested for strength in tension.

Objective:

The intent of this experiment was to determine which flux and solder combination will maximize the amount of force it takes to break the soldered bond when joining .016" etched straight annealed Nitinol wire.

Data Collection:

Indium Corporation and Memory Metalle have successfully soldered Nitinol so two solder kits were ordered from these companies. However, these fluxes and solders do not represent the entire scope of materials available for soldering Nitinol. A full factorial experiment was used to test three varieties of fluxes on each of the four solder types. Each combination of solder and flux was replicated five times. After each bond was soldered, the breakload was measured using the Instron machine in Inspection.

Data Set:

Variable	Description	Values
Flux	Type of flux applied to the overlapped wires	Flux #2, Flux #3, Flux 400, Flux 5RMA, Flux 5R, Flux 5RA, Flux 4R
Solder	Type of solder applied to overlapped wires	96 Sn/ 4 Ag, 80 Au/20 Sn, 95.5 Sn/3.8 Ag/.7 Cu, 96.5 Sn/ 3.5 Ag
Breakload	Amount of force required to break solder bond	Breakload Values (lbf)

Material Suppliers

Supplier	Material
Indium Corporation of America 1676 Lincoln Avenue Utica, NY 13502 (315) 853-4900	Fluxes: Flux #2 Flux #3 Flux 5RMA Flux 5R Flux 5RA Flux 4R Solders: 96 Sn/4 Ag 96.5 Sn/3.5 Ag 80 Au/20 Sn
Memory Metalle Am Kesselhaus 5 D-79576 Weil am Rhein +49. (0)7621 799 121	Fluxes: Flux 400 Solders: 95.5 Sn/3.8 Ag/ .7 Cu

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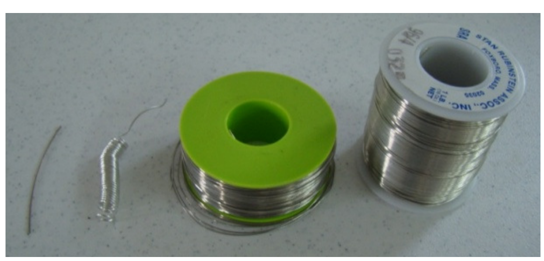
General Full Factorial Design Example with Actual Values:

Std Order	Run Order	Blocks	Flux	Solder	Breakload (lbf)
59	1	5	Flux #3	80 Au/20 Sn	18.389
51	2	5	Flux 400	80 Au/20 Sn	8.682
54	3	5	Flux #2	96.5 Sn/3.5 Ag	42.808
53	4	5	Flux #2	96 Sn/4 Ag	27.432
50	5	5	Flux 400	96.5 Sn/3.5 Ag	30.163
57	6	5	Flux #3	96 Sn/4 Ag	24.515
56	7	5	Flux #2	95.5 Sn/3.8 Ag/.7 Cu	19.547
52	8	5	Flux 400	95.5 Sn/3.8 Ag/.7 Cu	37.707
49	9	5	Flux 400	96 Sn/4 Ag	33.643
55	10	5	Flux #2	80 Au/20 Sn	10.283
58	11	5	Flux #3	96.5 Sn/3.5 Ag	45.274
60	12	5	Flux #3	95.5 Sn/3.8 Ag/.7 Cu	22.206

**All combinations were run 5 times each totaling 60 trials.*

Fluxes: Flux #2, Flux #3, Flux 400, 5RMA, 5R, 5RA, 4R. However, 5RMA, 5R, 5RA, 4R did not work on Nitinol because the flux prevented the solder from wetting to the metal.

Solders: 96 Sn/4 Ag, 80 Au/20 Sn, 95.5 Sn/3.8 Ag/.7 Cu, 96.5 Sn/3.5 Ag

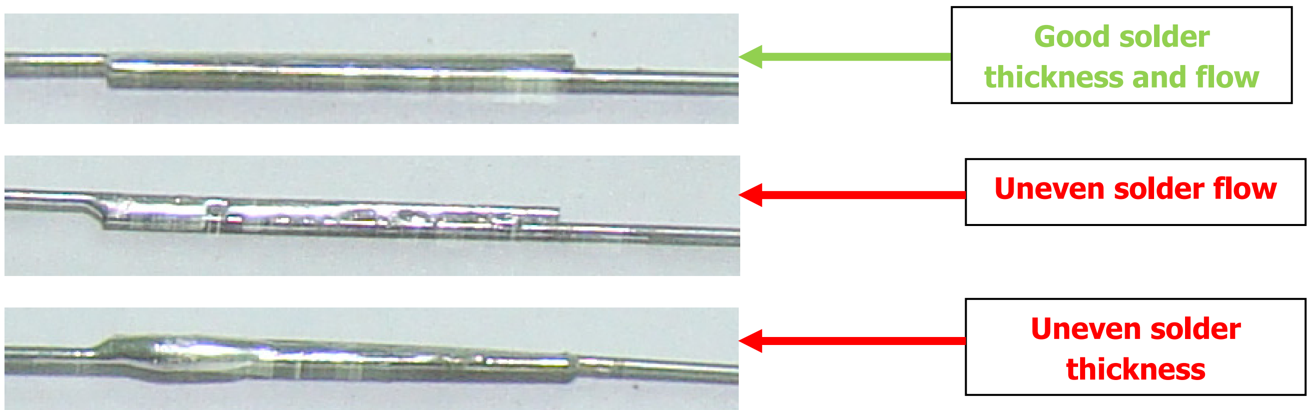


Design:

In this full factorial design, fluxes with three levels (Flux #2, Flux #3, Flux 400) and solders with four levels (96 Sn/4 Ag, 80 Au/20 Sn, 95.5 Sn/3.8 Ag/.7 Cu, 96.5 Sn/3.5 Ag) were tested. Replicating each combination five times resulted in 60 runs.

Limitations:

The runs were not followed in a random order to allow for more efficient test times. Another limitation was the soldering iron temperature. Once the iron was past 550°C, the tip oxidized, making solder flow very difficult. There were also many inconsistencies in breakload data. These inconsistencies were due to the amount of solder on each bond, the thickness of solder applied to the bond, the oxidation in the solder and on the iron tip, the amount of wire overlap, the amount of flux used, and the solder iron temperature. Below are some examples of *acceptable* soldered bonds and *unacceptable* soldered bonds.



Analysis and Interpretation:

Multilevel Factorial Design

General Linear Model: Breakload versus Blocks, Flux, Solder

Factor	Type	Levels	Values
Blocks	Fixed	5	1,2,3,4,5
Flux	Fixed	3	Flux 400, Flux #2, Flux #3
Solder	Fixed	4	96 Sn/4 Ag, 96.5 Sn/3.5 Ag, 80 Au/20 Sn, 95.5 Sn/3.8 Ag/.7 Cu

Factors:	2
Base Runs:	12
Base Blocks:	1

Replicates:	5
Total Runs:	60
Total Blocks:	5

Analysis of Variance for Breakload, using Adjusted Sum of Squares (SS) for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	4	38.07	38.07	9.52	0.26	0.903
Flux	2	29.25	29.25	14.63	0.40	0.675
Solder	3	2530.05	2530.05	843.35	22.86	0.000
Flux*Solder	6	274.19	274.19	45.70	1.24	0.305
Error	44	1623.15	1623.15	36.89		
Total	59	4494.72				

**S = 6.07370   R-Sq = 63.89%   R-Sq(adj) = 51.58%*

=Significant

Unusual Observations for Breakload

Obs	Breakload	Fit	SE Fit	Residual	St Resid
11	45.2740	34.3882	3.1364	10.8858	2.09 R
36	46.2280	34.7411	3.1364	11.4869	2.21 R
39	43.5930	29.0969	3.1364	14.4961	2.79 R
44	17.6740	31.3319	3.1364	-13.6579	-2.63 R

**R denotes an observation with a large standardized residual.*

General Linear Model: Breakload versus Blocks, Solder

Factor	Type	Levels	Values
Blocks	Fixed	5	1,2,3,4,5
Solder	Fixed	4	96 Sn/4 Ag, 96.5 Sn/3.5 Ag, 80 Au/20 Sn, 95.5 Sn/3.8 Ag/.7 Cu

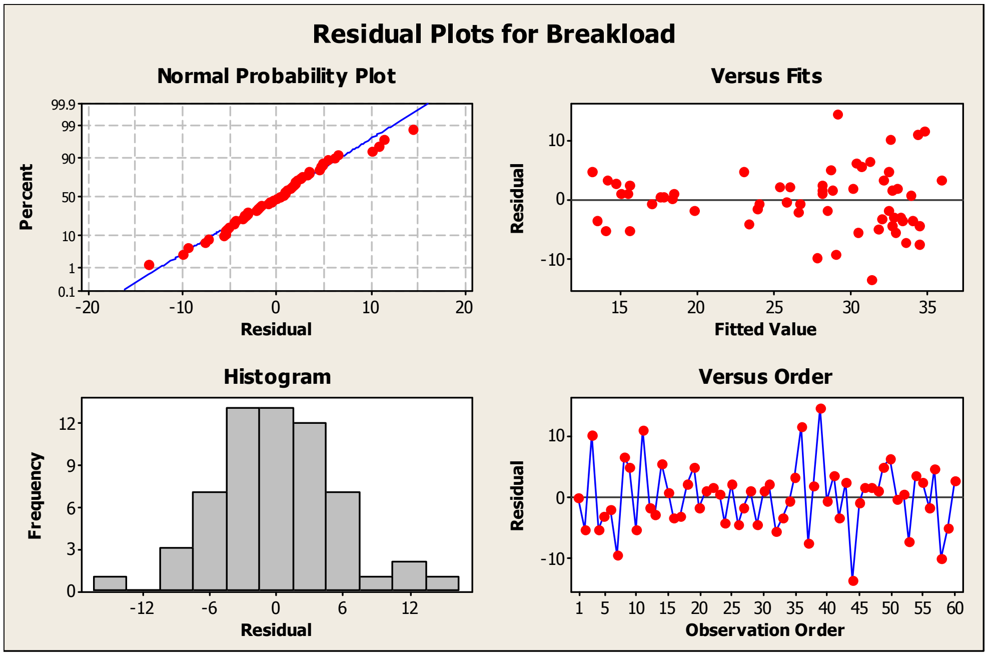
Analysis of Variance for Breakload, using Adjusted SS for Tests

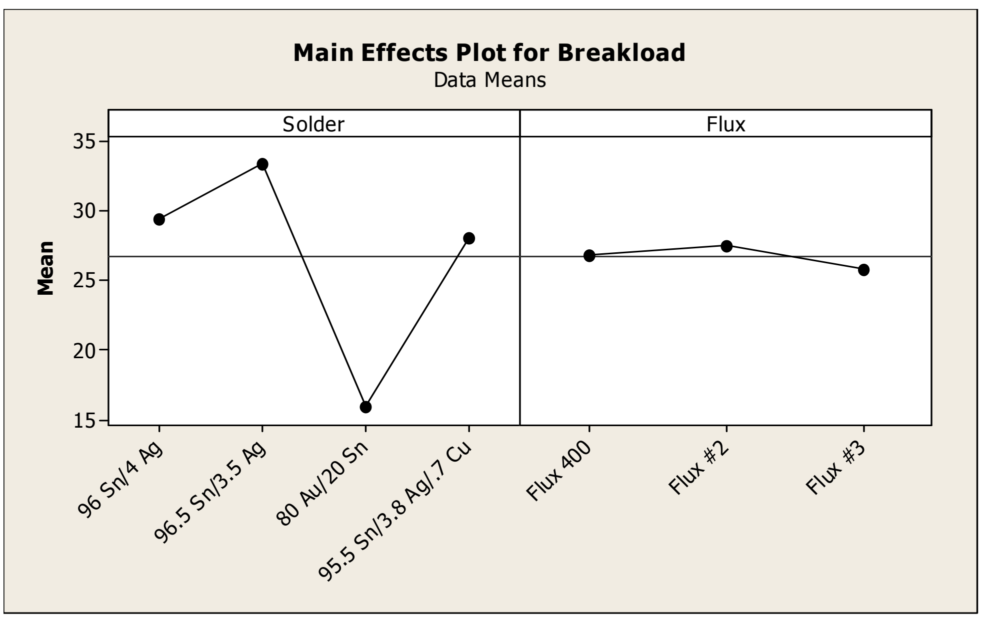
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Blocks	4	38.07	38.07	9.52	0.26	0.904
Solder	3	2530.05	2530.05	843.35	22.76	0.000
Error	52	1926.60	1926.60	37.05		
Total	59	4494.72				

**S = 6.08687   R-Sq = 57.14%   R-Sq(adj) = 51.37%*

=Significant

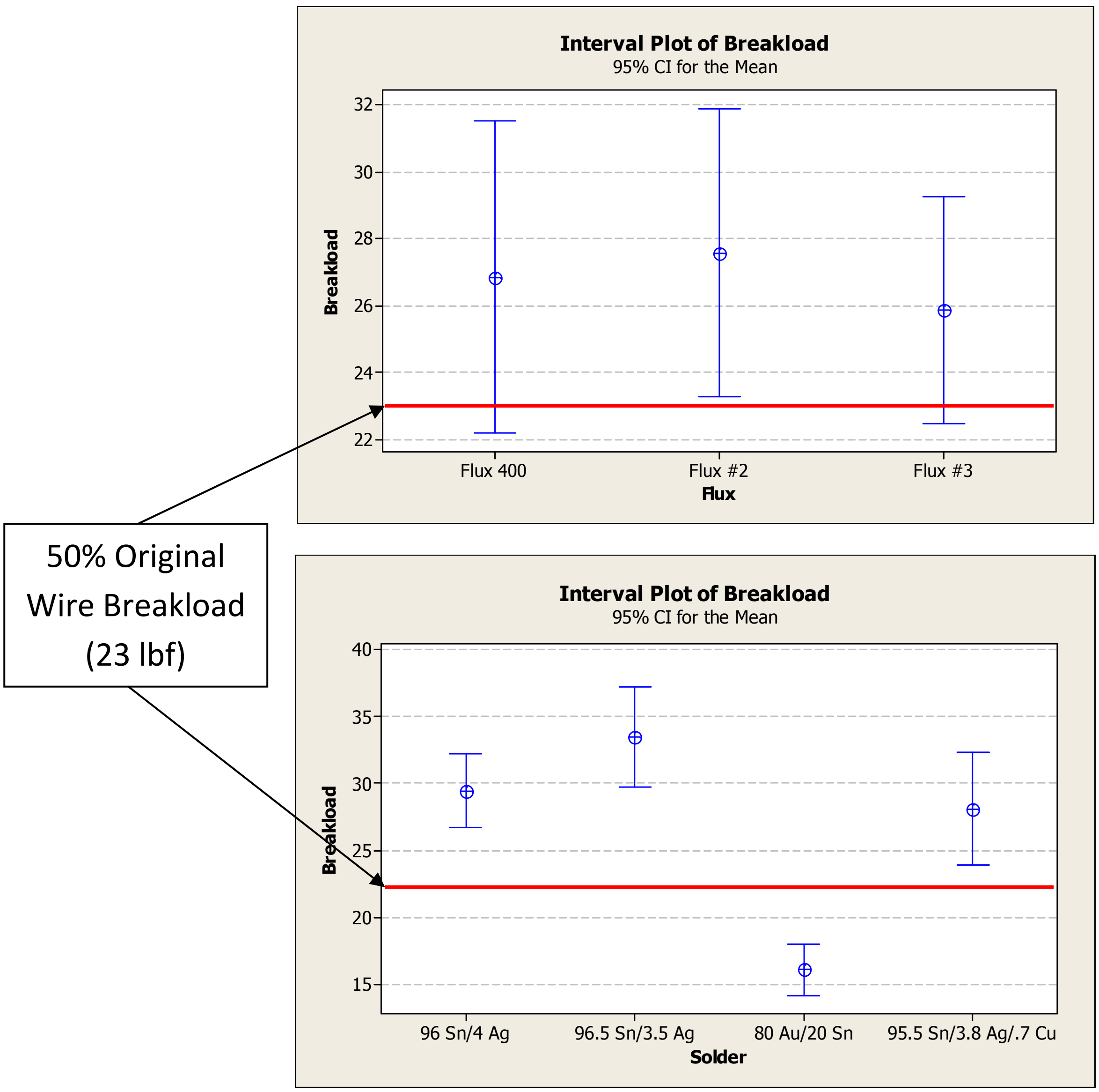
Plots for Breakload





The solder was the only factor that proved to be significant in this experiment while the blocks, flux, and the combination of flux and solder did not. The data followed a normal distribution as shown in the residual plots for breakload. The main effects plot indicates that the 96.5 Sn/3.5 Ag solder and the Flux #2 produced the highest breakload.

Statistical Analysis of Breakload



However, the interval plots of breakload show that the 96.5 Sn/3.5 Ag solder had a larger confidence interval than the 80 Au/ 20 Sn solder. The larger interval shows a larger standard deviation, meaning there is larger variability with the 96.5 Sn/ 3.5 Ag solder. While the 80 Au/ 20 Sn solder provided a lower breakload, it was a more consistent and reproducible solder than the 96.5 Sn/3.5 Ag solder. The fluxes were all comparable but the Flux #2 provided the highest mean and the Flux #3 gave the lowest variability in breakload strength.

Recommendations:

When soldering Nitinol, the customer prefers the breakload of the bond to be as large as possible (at least 50% the breakload of the wire). The ideal goal is for the wire to break before the bond breaks. Here is a list of solder and flux recommendations that will achieve these goals:

- **Flux –** This factor does not greatly influence the breakload of the bond. However, as determined by the main effects plot, Flux #2 did provide the highest breakload by a small amount while Flux #3 had the lowest variability in breakload strength. Also, the more flux applied to the seam, the easier the solder flow.
- **Solder –** This factor greatly influences the breakload of the bond. The 96.5 Sn/3.5 Ag solder provided the highest breakload average while the 80 Au/ 20 Sn solder provided the lowest breakload average as determined by the main effects plot. To achieve a higher breakload average and a lower variability in breakload strength, the 96.5 Sn/ 3.5 Ag solder should be used.
- **Solder Iron Temperature-** The soldering iron temperature should not exceed 550°C. At higher temperatures, the iron tip oxidizes, making solder flow very difficult.