

Statistical Evaluation of Joint Performance and The study of Corresponding Heat Affected Zone (HAZ) Status as the Composition of Zn50 Brazing Alloy Varies in a Mild Steel Application

Nwigbo S. C., Mbam S. O., Nwoye F.C., Omenyi S. N.

Abstract— This paper presents the development of regression models to predict the liquidus temperature of Zn50 brazing alloy and important mechanical properties of its brazed joint in mild steel application. It also examined the characteristics of heat affected zone (HAZ) of the base metal. The Scheffe Mixture Model method was employed to study the correlations of the Zn50 alloying components. The adequacy of the developed models was tested by Analysis of Variance (ANOVA). Validation experiments were conducted to verify the models. The ANOVA and scatter diagrams results indicate adequate models. Maximum tensile strength was obtained at 718.36 oC liquidus temperature with Vicker hardness of 180 Hv. These were obtained at the ratio of 16.5:1:7.5 brazing alloy additives of manganese, silver and modifier respectively. The maximum tensile strength is 512.36 Mpa. Within the predicted parameters, a maximum percentage error of 3.34 was obtained and the characteristics of some of the studied parameters obtained in the heat affected zone of the base metal indicate reliability in the developed models.

Index Terms— Brazing alloys, mild steel, mathematical modeling, mechanical properties and zinc.

I. INTRODUCTION

The usage of metals is not limited to one industry or area of application. In fact they are in use in a variety of areas including domestic and industrial. In review of abundance and material use, zinc is the 23rd most abundant element on earth

It was mined in more than fifty countries and 5th most used metal after iron, aluminium, copper and titanium (Sullivan, 2013).

In brazing alloy development, zinc is used mainly as temperature depressant and sometimes to aid wetting of the brazing alloy (Jacobson *et al*, 2002, Massalski *et al*, 1986, Villars *et al*, 1991, Watanabe *et al*, 2005 and Cao *et al*, 2011). It was also proved that with high chemical composition of zinc ($\geq 46\%$ wt), the brazing alloys became very volatile and that limited the amount of zinc that could be added and temperature reduction attainable (Jacobson *et al*, 2002, Sisamouth *et al*, 2010).

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Nwigbo *et al*, (2014) successfully developed zinc-based brazing alloy for joining mild steel to mild steel within a temperature of 690.90 to 735.10 °C. The liquidus requirement and the obtained mechanical properties were dependent on the brazing alloy components, control of brazing parameters and the brazing atmospheric conditions. The joints produced from the zinc-based brazing alloys had acceptable strengths with improved stress-strain behaviour when compared with muntz brass in mild steel application. There was no numerical relationship between the brazing alloy components and the obtained mechanical properties.

Mathematical models for predicting ultimate tensile strength, yield strength and percentage elongation of brazing alloys has been successfully developed by regression analysis. The response surface methodology was used and the adequacy of the models was tested by analysis of variance (ANOVA) and Scatter diagrams. Each result was considered to be satisfactory if the ANOVA gave adequate coefficient of determination (R^2) above 60% and the plotted scatter diagrams gave positive relationship (Elangovan *et al*, 2008, Palanivel *et al*, 2011).

Heat application during brazing is not uniform. As a result of this, the expansion at different point in the plate relative to the brazed joint varies. There is usually a resistance to the expansion of metal in the hottest region by metals at lower temperature causing the plate to bend in an arc at each end away from the heat source. At the end of brazing however there is a reversal in direction of the deforming forces due to change in heat flow direction. The heat now flows away from the molten joint (causing it to cool and shrink) to the cold area (causing it to expand). The consequence is dimensional alteration of the plate causing the plate to be warped when cooled to room temperature; this stress is referred to as residual stress.

It is expected that a brazed joint should be able to withstand static and dynamic loads over time in a given condition of service. Hence a wide range of mechanical test are usually carried out in other to establish some design rules based on failure criteria established by the test.

Zn₅₀ brazing alloy which is newly developed filler in mild steel application still lack some basic analysis to completely prove the reliability of the new product (Nwigbo *et al*, 2014). This paper presents the development of regression models that can reliably predict the liquidus, tensile strength and hardness of Zn₅₀ brazed joint employing experiment data and

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Scheffe mixture model method. The study also analyzed the heat affected zone (HAZ) of the base metal to investigate the reliability of the newly developed brazing alloys.

II. MATERIALS AND METHODS

The design of experiment, brazing experiment and performance tests are based on the development of Zn₅₀ brazing alloy (Nwigbo *et al.*, 2014; Arkan *et al.*, 2007). The findings are as in Tables 1 and 2

Table 1: Design of experiment

Experiment runs order	Zn(%wt)	Mn(%wt)	Ag(%wt)	Modifier (%wt)
1	50.00	35.00	0.00	15.00
2	50.00	33.00	3.33	13.67
3	50.00	33.00	2.00	15.00
4	50.00	35.00	2.00	13.00
5	50.00	34.00	4.00	12.00
6	50.00	35.00	4.00	11.00

Table 2: Design of experiments and determined mechanical properties.

Sr.No	Alloys	Liquidus (°C)	Tensile (MPa)	Hardness (0.1Hv)
1	Zn ₅₀ Mn ₃₅ Ag ₄ Mod ₁₁	708.22	340	182
2	Zn ₅₀ Mn ₃₃ Ag _{3.33} Mod _{13.67}	690.18	405	187
3	Zn ₅₀ Mn ₃₃ Mod ₁₅	682.85	335	190
4	Zn ₅₀ Mn ₃₄ Ag ₄ Mod ₁₂	724.11	488	189
5	Zn ₅₀ Mn ₃₅ Ag ₂ Mod ₁₃	716.81	510	180
6	Zn ₅₀ Mn ₃₃ Ag ₂ Mod ₁₅	718.36	512	180

Microstructure analysis was carried out on the brazed specimen after it was cooled to ambient temperature using optical microscope. To this end the specimen was prepared by polishing with silicon carbide abrasive and etched with nitric acid and ethanol in 10% and 90% concentrations respectively.

The analysis of the residual stress developed in a lap brazed joint using the Zn₅₀ brazing alloy as it cools from brazing temperature to the atmospheric temperature was carried out using Finite Element Model (FEM). The model was developed using Plane stress iso-parametric linear quadratic element (IPLQ) as defined in GTStrudle version 13 Software. The input data used for the modeling are as shown in Table 3 and the boundary condition is such that the model is fixed at both ends (Nwigbo *et al.*, 2015).

2.1 Development of Regression Equations

The mechanical properties were thought to be a function of the components composition of the brazing alloys.

$$\Psi = f(\alpha, \beta, \gamma, \zeta) \quad [1]$$

Where;

Ψ = mechanical properties (liquidus, tensile strength and Vickers hardness).

α = percentage weight of zinc.

β = percentage weight of manganese.

γ = percentage weight of silver.

ζ = percentage weight of the modifying element.

The coefficients of the suggested models were calculated by Design expert from the design of experiments and obtained

mechanical properties results values. These coefficients were substituted into the expanded form of equation [1] to get the mathematical model for predicting any of the mechanical properties (liquidus, tensile strength and hardness).

2.1

Final Developed Mathematical Models

The final developed mathematical equations in actual components form are given as follows;

$$\Psi_L = 189.19\alpha + 339.46\beta + 4288.35\gamma + 841.32\zeta - 10.51\alpha\beta - 77.02\alpha\gamma - 20.17\alpha\zeta - 138.64\beta\gamma - 28.53\beta\zeta - 39.26\gamma\zeta + 2.46\alpha\beta\gamma + 0.58\alpha\beta\zeta - 0.48\alpha\gamma\zeta + 1.74\beta\gamma\zeta$$

[2]

$$\Psi_{TS} = -353.42\alpha - 1059.4\beta + 41472.81\gamma - 5385.55\zeta + 31.49\alpha\beta - 695.94\alpha\gamma + 134.80\alpha\zeta - 811.66\beta\gamma + 229.82\beta\zeta - 1310.98\gamma\zeta + 12.57\alpha\beta\gamma - 5.37\alpha\beta\zeta + 17.11\alpha\gamma\zeta + 12.45\beta\gamma\zeta$$

[3]

$$\Psi_{HV} = -540.8\alpha - 1039.02\beta - 19528.4\gamma - 320.82\zeta + 30.05\alpha\beta + 358.94\alpha\gamma + 8.77\alpha\zeta + 522.19\beta\gamma + 8.77\beta\zeta + 335.55\gamma\zeta - 9.08\alpha\beta\gamma + 0.18\alpha\beta\zeta - 3.33\alpha\gamma\zeta - 5.05\beta\gamma\zeta$$

[4]

Where ; Ψ_L =liquidus temperature, Ψ_{TS} =tensile strength and Ψ_{HV} =Vickers hardness.

III. RESULT AND DISCUSSION

3.1 Design of Experiment Error

Errors were generated during blending of the mixtures. The errors were estimated using Fraction of design space (FDS) graph. Generally, it is desirable for the graph to have relatively low values (1.0 or less) across the region of interest. Tolerable values must be less than or equal to four. It could be seen that the errors is directly proportional to the percentage weight composition of zinc and modifying element as shown in Figure 1. Also, error contours indicated tolerable values at 50 % weight composition of zinc. This shows a good design of experiment in the brazing alloy blends or mixture.

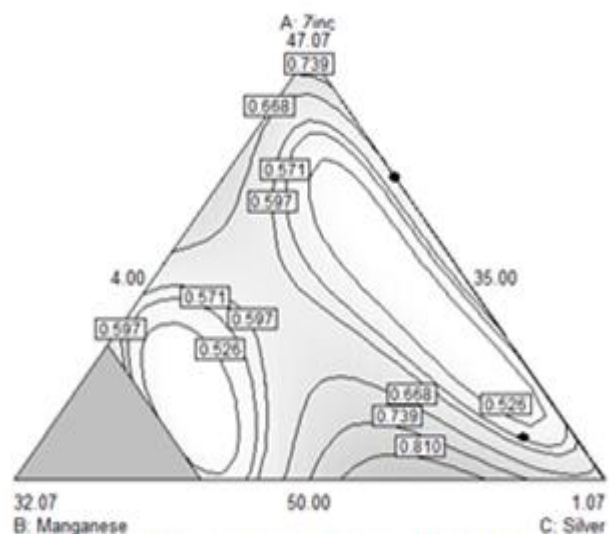


Figure 1: Standard error of experiment design blend of Zn₅₀ brazing alloy.

3.2 Checking Adequacy of the Developed Models

The adequacy of the developed mathematical equations was tested by analysis of variance (ANOVA).

The F-value should correspond to P-value of less than 0.050. P-Value between 0.05 and 0.10 is marginally significant and indicate relatively poor model. ANOVA results of the parameters in this investigation are as shown in Table 3 and 4.

The models F-values of 408.67, 113.35 and 315.37 implies these models are significant. There is only a 0.01% chance that models of F-value these large could occur due to noise. These P-values of less than 0.0001 indicates very acceptable models. Also, these signal ratios of 86.263, 31.850 and 81.832 indicated adequate signals. These models can be used to navigate the design space. Further, R²-values and predicted R²-values of greater than 90% as indicated are further confirmation of adequacy of the developed mathematical relationships. These are in tandem with literatures (Elangovan *et al*, 2008, Palanivel *et al*, 2011).

Hence, the developed models are surely adequate for the intended applications.

Table 3: Base metal and brazing alloy properties used in the FE simulation

Mechanical properties	Base metal (Mild steel)	Brazing alloy (Zn ₅₀)
Young's modulus	210x10 ³ Mpa	15.4x10 ³ Mpa
Poisson ratio	0.3	0.34
Yield stress	320Mpa	305Mpa
Density	7.69x10 ³ kg/m ³	6.8x10 ³ kg/m ³
Coefficient of Thermal expansivity (CTE)	1.2x10 ¹ /°C	1.7x10 ¹ /°C

Table 4: ANOVA results of mathematical models

		Liquidus (°C)	Tensile strength (Mpa)	Vickers hardness(Hv)	Remarks
Sum of squares	regressions	7482.10	1.038E+005	2186.55	Adequate
	residuals	8.45	423.29	3.20	
Mean squares	regressions	575.53	7993.21	168.20	Adequate
	residuals	1.41	70.53	0.53	
F-values(=10)		408.67	113.35	315.37	Adequate
P-values		<0.0001	<0.0001	<0.0001	Adequate
Adequate precision(=4)		86.263	31.850	81.832	Adequate
R ² values		0.9989	0.9959	0.9983	Adequate
Predicted R ² values		0.9964	0.9872	0.9954	Adequate
Mean values		707.65	393.45	188.25	Adequate

At constant values of any of the elements, increase in zinc with corresponding decrease in any of the elements continuously decreases the liquidus of Zn₅₀ brazing alloys. The approximate liquidus attained at 50 % weight zinc addition is 684°C as shown in Figure 2.

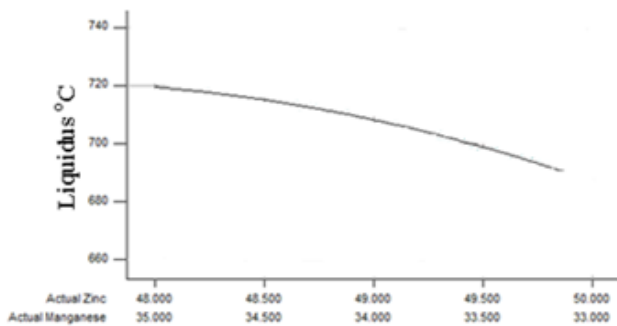


Figure 2: Variations of Zinc and Manganese

Therefore, it could be concluded that all of the additives increases the energy requirement. But, addition of zinc reduced the energy requirement of Zn₅₀ brazing alloy. This effect of liquidus reduction by zinc addition is in correlation to brazing alloy developments where, zinc were mainly used as temperature depressant (Massalskiet *al*, 1986,

Villars *et al*, 1991, Jacobson *et al*, 2002, Watanabe *et al*, 2005 and Cao *et al*, 2011).

3.3.2 Effect of Alloy Components on Tensile Strength

When any of the elements was varied with zinc and keeping others constant, it was noticed that the element which mostly enhanced the tensile strength of Zn₅₀ brazing alloy is silver as indicated in Figure 3.

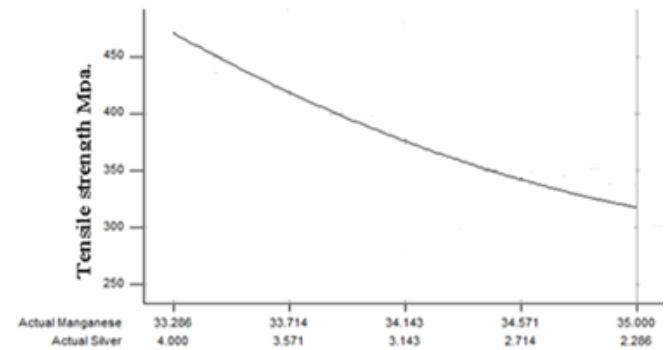


Figure 3: Variations of Manganese and Silver

Decrease in silver sharply decreases the tensile strength of the alloy from approximately 475 MPa to 325 MPa. This effect of tensile strength enhancement of zinc alloy by addition of silver is as reported in literature (Purwanto *et al*, 2012).

3.3.3 Effect of Alloy Components on Vickers Hardness

It was discovered that effect of variation of any of the three components keeping any one of them constant was distributive. There is none of the components that were observed to have good control over the hardness of Zn₅₀ brazing alloys. This can be seen in Figure 4. However, manganese seemed to have predominant effect on the hardness of the alloy as indicated by the contours.

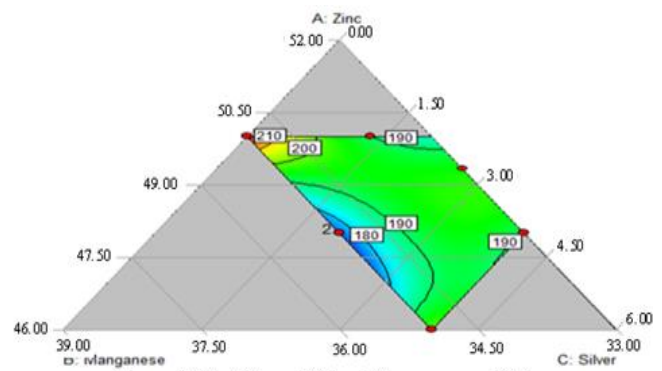


Figure 4: Variations of Zinc, Manganese and Silver

3.4 Mathematical Models Validation

Experiments were carried out to verify the developed mathematical models as given in equations 2, 3 and 4. Six Zn₅₀ brazing alloys were produced using different values of its components other than those contained in the design of experiment (Table 1). Samples were produced and tested using the same procedures described earlier in development of Zn₅₀ brazing alloys. Then, liquidus, tensile strengths and Vickers hardness were calculated using equations 2, 3 and 4. The two results were compared. The details of results obtained are presented in Table 5. A maximum percentage error of 3.34 obtained in the validation experiments is quite satisfactory.

Statistical Evaluation of Joint Performance and The study of Corresponding Heat Affected Zone (HAZ) Status as the Composition of Zn50 Brazing Alloy Varies in a Mild Steel Application

Table 5: Results of models validation experiments

Components (%wt)			Liquidus (°C)		Tensile strength (MPa)		Tensile strength Error (%)		Vickers hardness (Hv)		Vickers hardness Error (%)	
Zn	Mn	Ag	Mod. fir	experiment	model	Experiment	model	Experiment	model	Experiment	model	
50.0	33.5	1.5	15.0	725.11	728.23	-0.43	41282	407.20	1.38	224.71	230.61	-2.55
50.0	33.0	3.5	13.5	753.21	759.80	-0.87	51233	519.60	-1.40	218.21	216.00	1.02
50.0	34.0	1.0	15.0	769.30	764.01	0.69	41628	414.30	0.46	210.18	213.17	-1.36
50.0	35.0	1.0	14.0	690.31	694.39	-0.59	35614	355.24	0.25	270.38	274.02	-1.33
50.0	35.0	3.0	12.0	723.64	724.23	-0.08	451.72	450.30	0.32	200.12	195.26	2.49
50.0	35.0	3.5	11.5	730.77	732.14	-0.19	50098	505.97	-0.99	186.15	180.17	3.34

3.5 Analysis of Heat Affected Zone of the Base Metal

Figure 5 is the temperature decay curve plotted by FEHT version 7.308 as it cools in ambient after brazing. The cooling process followed an exponential path and stabilized at a point in which the temperature of the body is at equilibrium. At equilibrium the temperature at every point in the model is the same and the body cools uniformly in a straight line. The nodes closer to the brazed joint have higher exponent value and converge faster to the equilibrium temperature compared to those further away; this implies that the path of the body with higher temperature give up greater amount of heat energy per time on cooling compared to those with lower temperature.

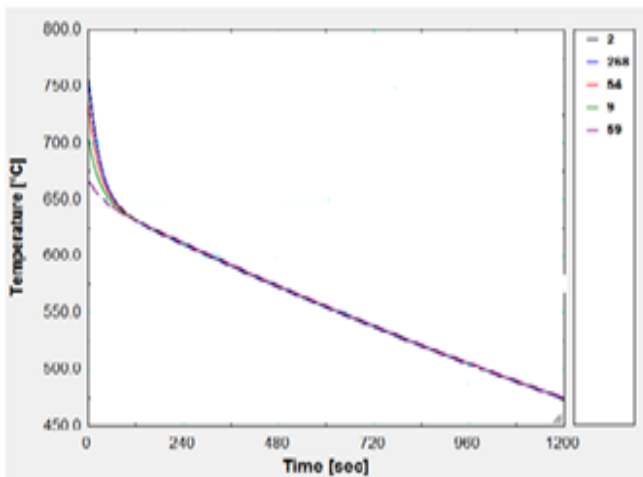


Figure 5: cooling curve in transverse direction

3.6 Microstructure Analysis

The images of the microstructure of the heat affected zone taken at different magnification using optical microscope is as shown in Figure 6. The white patches are the proeutectoid ferrite while the dark region is the pearlite as indicated by the arrow. In a typical binary phase diagram, at approximately 750°C (which is the brazing temperature) and at a carbon composition of 0.25%, the steel existed as a combination of Bcc and Fcc. These structures formed at this temperature are known as proeutectoid ferrite. On cooling below the eutectic temperature (727°C) under a stable condition, they transform to pearlite which is a combination of cementite (Fe₃C). Therefore, hypo eutectoid alloys on cooling to room temperature after brazing contains pro eutectoid ferrite and pearlite. This implies that the alloy will be ductile on cooling to room temperature having its property softer than pearlite whose properties are intermediate to soft ductile ferrite and hard brittle cementite.

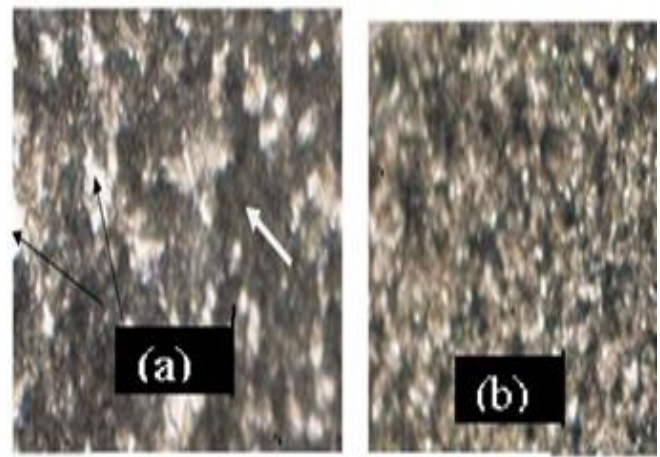


Figure 6: microstructure of heat affected zone

Applying the lever rule with tie line that extends from eutectoid composition to boundary for hypoeutectoid alloys in phase diagram calculations as shown in Figure 7, the fraction of pearlite and proeutectoid ferrite are 0.691 and 0.3089 percents respectively which are adequate in mild steel application.

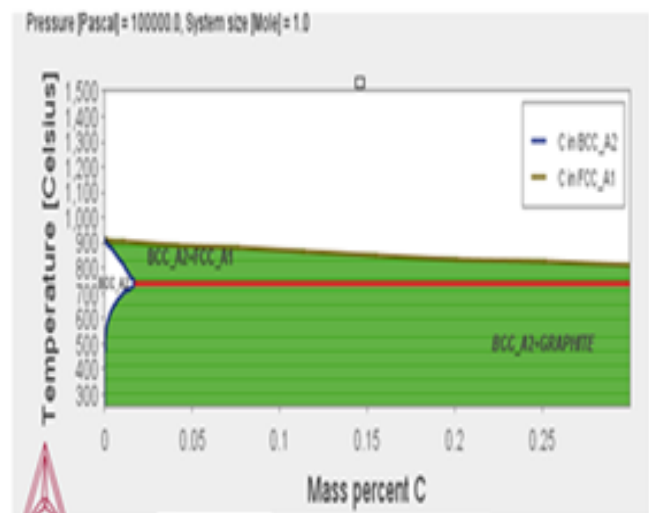


Figure 7: C-Fe phase diagram

IV. CONCLUSION

From the results and discussions, it is observed that mathematical models of Zn₅₀ brazing alloy components and some mechanical properties have been established in mild steel application. The adequacies of the models which were tested by ANOVA and scatter diagrams were found to be satisfactory.

All added elements except zinc increase the energy requirement of Zn₅₀ brazing alloy.

Silver contribute most in tensile strength enhancement of Zn₅₀ brazing alloy and there is none of the added components were observed to have good control over the hardness of Zn₅₀ brazing alloy.

The brazing heat input altered the microstructure of the heat affected zone result in the formation of pearlite and eutectoid ferrite at 69.1% and 30.89% respectively. Consequently on cooling to room temperature the heat affected zone will be ductile having its property softer than pearlite which is intermediate to soft ductile ferrite and hard brittle cementite.

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