Experimental study of improvement shear strength and moisture effect PVP adhesive joints by addition PVA

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Abstract. This research study an experimental and optimization work of the effect of moisture on the shear strength of adhesive joints and improvement by adding variable concentration of PVA to PVP. In this work, shear strength samples from Cu-Cu and the adhesive made from dissolving 16 grams of polyvinyl pyrrolidone (k30) and 8 grams of polyvinyle Alcohol in 50 ml of water was studied. This study condensed on the effect of variable under pressure of a fixed rate of 5 MPa. For temperatures (225) °C with times (10) minutes under the same. It was noticed that the best resulted found at a temperature of 225 °C and time of 10 minutes and 50% from PVA and 50% PVP the best result of shear strength, reaching overhead hanging to 1240 N and 920 at 265 °C that there is decreasing in the resistance of the adhesive with the emerging time (0,2 ,4, 6 ,7, 9) at room temperature, and using a Neural network to prediction the results.

Keywords: Adhesive, Shear strength, Temperature, Poly vinyl pyrrolidone, moisture.

1. Introduction
The low temperature joining technique (LTJT) has been suggested as a possible alternative to lead-free die attach materials and high temperature application mentioned above. LTJT is a technique mainly by Schwarzbauer [1] and others to produce die attach joints from micron-scale Ag paste for power electronics packaging in the late 1980s [2]. Since then, LTJT technology has garnered a great transact of interests amongst the academia as well as industry. There are numerous benefits associated with the use many particles in LTJT. Many particles as Silver has best thermal and electrical conductivities than Sn-Pb or Pb-free joints Once sintered the Ag past will have a melting temperature like to bulk Ag (961 0C). depended on these properties, Ag has been remembered in recent literature as a leading candidate for lead free die attach as well as die-attach for silicon carbide technologies [3].The test of mechanical properties of adhesives was behaved at variable temperatures from( 200 to 275)0C. The aim of the work was to improve the shear strength and moisture resistance of adhesive joints using composite polymers of PVP/PVA and examine its effect on the shear strength of adhesive of Cu-Cu) metals joining.

2. Experimental Work

2.1 .Materials
Polyvinyl Alcohol (PVA) has M.W 1750 and Polyvinyl pyrrolidone (PVP, K30) which a molecular weight M.W 40,000 was used as base material. It have viscosity 32, 26 cps and was tested by using viscosity measurement device for 0.02 PVA and 0.008 mol/L PVP concentration. The melting point of PVPK30 is 172°C and 192 for PVA. The adhesive material was prepared of concentration of 0.008 mol. L-1 (PVP-K30) by solving 16 gm and 0.02 mol. L-1 (PVA) by solving 8gm in 50 ml water placed on stirrer device for 30 minutes at room temperature for PVP and 15 minutes at 60°C according to equations (1) & (2):

\[ cx = nx \times V \]  
(1)  
\[ nx = g \times M \times W \]  
(2)

Where: Cx is the concentration mol. L-1, and nx is the number of moles.

\[ V = 50 \text{ ml volume of solvent} , \quad g = 8 \text{ gm} \text{. is the mass of PVP} \]

2.2. Preparation of adhesive joint
The single shear lap joints of adherent were Copper sample with dimension (25.4 mm * 1.6 mm * 90 mm) having a contact area (21.7mm* 25.4mm) 330 mm2 of the adhesive joint that was prepared according to ASTM D1002 [4], as shown in the Figure 1.

![Figure 1. Sample according ASTM D1002 -01](image)

The copper specimens were pressed at pressure 5 MPa and heating temperatures 225°C for times (10) minutes by using press under heating system, as shown in the Figure 2.

![Figure 2. Copper adherents after pressing](image)

2.3. shear and moisture test
The shear strength of the joint was found by using a universal tensile testing machine [5], by drawing apart the two plates with a rate of 1 mm/min-1, as shown in Figure 3.
And moisture test was done on both shear specimens by immersing these specimens in water Glass basin for (2, 4, and 6) hours at room temperature according to (ASTM –D1151) and then tested by universal tensile test machine with speed 1.3mm/min for shear specimens.

3. Result and Discussion

Figure 4 and Figure 5 noticed the analysis (TGA) and (DSC)

![Figure 4. TGA for PVP](image)

Figure 4. TGA for PVP

![Figure 5. DSC for PVA](image)

Figure 5. DSC for PVA

has been plotted with weight loss as a function of the temperature for the reference PVP and PVA precursor with a heating rate of 10 °C/min in the temperature range from 40 to 800 °C and from (50 to 350 for PVA) [6]. It is clear that the initial weight loss from the TGA curve is 12% in the temperature range 40-87.9 °C, this is due to loss of OH content. In the Differential Thermal Analysis (DTA) curve, two exothermic peaks were observed at 483.92 °C and 678.08 °C, respectively [7]. The sharp and
strong exothermic peak at 483.92°C is due to the combined effect of combustion of organic residuals and the decomposition of PVP and which is well above the heating temperature employed in the present work. These results approximate to which in [8].

**Table 1.** Shows the summary of results of curing temperature, load and ratio (PVP/PVA) at pressure 5 MPa.

| T(°C) | PVP | 25%PVA | 50%PVA | 75%PVA | PVA |
|-------|-----|--------|--------|--------|-----|
| 200   | 590 | 1002   | 995    | 450    | 510 |
| 215   | 575 | 995    | 1020   | 480    | 490 |
| 225   | 490 | 1320   | 1240   | 720    | 498 |
| 240   | 445 | 1120   | 1210   | 680    | 620 |
| 265   | 0   | 0      | 920    | 715    | 515 |

This table manifests that the best result for shear load at (50%PVP and 50%PVA) good distribution in composite structure, Figure 6 Obtained results show that the highest values of shear load was reached at (50%PVP and 50%PVA) which has better distribution in composite structure and has a dynamic viscosity is 37cP, before and after this ratio (50% PVP/PVA) a nonhomogeneous structure in the adhesion area, causes a decrease in shear strength [9].

![Image of shear force with temperature](image)

**Figure 6.** Shear force with Temp.

**Table 2.** Shows the summary of results of moisture time, shear load and ratio (PVP/PVA) at pressure 5 MPa

| Moisture time(h) | PVP | (50%PVP,50%PVA) | PVA |
|------------------|-----|-----------------|-----|
| 0                | 590 | 995             | 510 |
| 2                | 420 | 1020            | 440 |
| 4                | 215 | 525             | 325 |
| 7                | 0   | 315             | 211 |

Figure 7 show the effect of moisture time on the shear load between PVP, PVA and (50%PVP with 50%PVA), (50%PVP with 50%PVA) which has better resistance to water from PVP and PVA because a composite structure for PVP/PVA good distribution in structure.
Figure 7. Moisture effect on shear force

Many attempts indicated found that the best structure of the neural networks for predicting of shear loads, one and two hidden layers were used to predict the properties of the (PVP/PVA) as the adhesive material. The influence of variation of the curing time and sintering temperature on the output results of the shear load for single lap joints are investigated, after the necessary attempts that illustrated in tables 3. It was found that the best structure of the neural networks for predicting of the shear loads at two hidden layer structure with two neurons in the input layer, five neurons in the first hidden layer, four neurons in the second hidden layer and single output layer, which can be coded as 2-5-4-1.

Table 3. Mean square error (MSE) and correlation coefficients (R) for Resilience back propagation (train rp) for moisture effect on shear load experimental data of pure PVP/PVA

| Serial No. | ANN Structure | MSE Training | Correlation coefficient (R^2) training | MSE Testing | Correlation coefficient (R^2) testing |
|------------|---------------|--------------|---------------------------------------|-------------|--------------------------------------|
| 1          | 2-1-1         | 0.0282       | 0.9956                                | 0.0229      | 0.9913                               |
| 2          | 2-2-1         | 0.0077       | 0.99994                               | 0.0055      | 0.9996                               |
| 3          | 2-3-1         | 0.002        | 0.99997                               | 0.0014      | 1                                    |
| 4          | 2-4-1         | 0.00028      | 1                                     | 0.00024     | 1                                    |
| 5          | 2-5-1         | 0.0573       | 0.9954                                | 0.04        | 0.977                                |
| 6          | 2-6-1         | 9.79e-07     | 1                                     | 1.08e-06    | 1                                    |
| 7          | 2-7-1         | 3.93e-06     | 1                                     | 6.29e-06    | 1                                    |
| 8          | 2-8-1         | 1.613e-04    | 0.99999                               | 1.25e-04    | 1                                    |
| 9          | 2-9-1         | 3.68e-05     | 1                                     | 3.87e-05    | 1                                    |
| 10         | 2-10-1        | 1.27e-6      | 1                                     | 9.681e-7    | 1                                    |
| 11         | 2-2-1-1       | 0.069        | 0.948                                 | 0.00573     | 0.9992                               |
| 12         | 2-2-2-1       | 1.79e-04     | 0.99998                               | 3.346e-04   | 0.99996                              |
| 13         | 2-2-3-1       | 0.0016       | 0.99972                               | 0.0018      | 0.99989                              |
| 14         | 2-2-4-1       | 0.0187       | 0.994                                 | 0.0219      | 0.9964                               |
Where, For shear load It is clear that the best training performance is $2.2369 \times 10^7$ and 1 for training and 1 for testing samples respectively as shown in Figure 8 and Figure 9.

|   |   |   |   |   |   |
|---|---|---|---|---|---|
| 15 | 2-3-1-1 | 1.97e-04 | 0.9997 | 1.862e-04 | 1 |
| 16 | 2-3-2-1 | 1.795e-05 | 0.99999 | 1.245e-04 | 1 |
| 17 | 2-3-3-1 | 1.39e-05 | 1 | 5.55e-04 | 1 |
| 18 | 2-3-4-1 | 2.14e-04 | 0.99998 | 1.32e-04 | 0.99998 |
| 19 | 2-4-4-1 | 6.55e-05 | 1 | 4.5e-05 | 0.99999 |
| 20 | 2-4-5-1 | 1.2306e-04 | 0.99999 | 1.26e-04 | 0.99998 |
| 21 | 2-4-6-1 | 1.206e-06 | 1 | 7.94e-07 | 1 |
| 22 | 2-4-7-1 | 5.49e-07 | 1 | 7.62e-07 | 1 |
| 23 | 2-5-3-1 | 1.0763e-04 | 1 | 1.039e-05 | 1 |
| 24 | 2-5-4-1 | 2.14e-07 | 1 | 2.98e-07 | 1 |
| 25 | 2-5-5-1 | 0.0012 | 0.99973 | 0.0012 | 0.99987 |
| 26 | 2-6-3-1 | 2.845e-05 | 1 | 3.434e-05 | 1 |
| 27 | 2-6-4-1 | 1.97e-05 | 1 | 1.75e-05 | 1 |
| 28 | 2-6-5-1 | 1.132e-05 | 1 | 1.08e-05 | 1 |
| 29 | 2-6-6-1 | 1.028e-05 | 1 | 8.62e-06 | 1 |
| 30 | 2-6-7-1 | 4.83e-07 | 1 | 5.549e-07 | 1 |

**Figure 8.** Training and testing prediction data PVP/PVA of two hidden layer
Figure 9. Best training performances

Table 4. The summary of curing time, Temperature, experimental shear force and predicted neural shear force.

| Time (h.) | Exp.shear force | Neu.shear force | Err%    |
|-----------|----------------|----------------|---------|
| 0         | 1220.0000      | 1221.0063      | -0.000318787 |
| 2         | 1055.0000      | 1054.8924      | 0.005799362  |
| 4         | 960.0000       | 962.0682       | -0.005013171 |
| 6         | 880            | 879.7470461    | 0.028744764  |
| 7         | 76             | 76.77186157    | -1.015607335 |
| 9         | 36             | 35.19366245    | 2.239826535  |

Good result between the experimental and neural network (ANN) because the convergence between the training and testing with the experimental result as shown in Figure 10.

Figure 10. The difference between the experimental and neural net work

4. Conclusions
From the above results, the following conclusions can be written:
1. The temperature effect on the shear strength is a slight direct proportion from (200 to 225) oC but it is inverse proportion from (225 to 275) C.
2. Increasing in moisture time caused weakness in shear strength.
3. The best value of adhesive joint loading is for(50%PVP and 50%PVP), at 225oC because the viscosity of PVP k30/PVA increased slightly in direct proportionality from (200-225) oC but decreased during the range (225-275) oC.

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