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Experimental Study and Prediction the Mechanical Properties of Nano-Joining Composite Polymers

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Abstract: This research study an experimental and optimization work of the effect of temperature, curing time and copper concentration on the shear strength of adhesive joints by mixing Polyvinyl Pyrrolidone PVP K30 and polyvinyle alcohol with copper. In this research, shear strength of the adhesive made from dissolving 16 g of Polyvinyl Pyrrolidone PVP (K30) and 8 g of polyvinyle alchol in 50 mL of water was studied. This study condensed on the effect of variable under pressure of a fixed rate of 5 MPa. Therefore, study five temperatures 200, 215, 225, 240 and 265°C with times 10 min under the same pressure in all cases was studied by taking pictures. It was noticed that the best resultedfound at a temperature of 265°C and time of 10 min and 15% from copper, reaching overhead hanging to 1760 N, that there is an increase in the resistance of the adhesive with the increase in temperature and up to 265°C, also using neural network for optimization result.

Key words: Adhesive, temperature, polyvinyl pyrrolidone, copper, curing time, adhesive

INTRODUCTION

The Low Temperature Joining Technique (LTJT) has been proposed as a possible alternative to lead-free die attach materials and high temperature application mentioned above. LTJT is a technique pioneered by Masson *et al*. (2013) and others to produce die attach joints from micron-scale Ag paste for power electronics packaging in the late 1980s (AL-Shammari *et al*., 2011). Since, then, LTJT technology has garnered a great deal of interests amongst the academia as well as industry.

There are numerous benefits associated with the use of Cu particles in LTJT. Copper has good thermal and electrical conductivities than the commonly used Sn-Pb or Pb-free joints. Nano-Cu joints are sintered at temperatures significantly lower than the melting temperature of Ag, typically 0.2-0.4 Tm. Once sintered the Ag joint will have a melting temperature similar to bulk Cu (1021°C). Based on these properties, nano-Cu has been listed in recent literature as a leading candidate for lead free die attach as well as die-attach for silicon carbide technologies (Siow, 2012). The test of mechanical properties of adhesives was conducted at variable temperatures from 200-275°C. The aim of the research was to improve the shear strength of adhesive joints using Cu NPs as additive materials in the polymers solution mixed of PVP/PVA nano composites and examine its effect on the shear strength of adhesive nano-joining of Cu-Cu metals joining.

MATERIALS AND METHODS

Experimental work: Polyvinyle Alcholole (PVA) M.W 1750 and Polyvinyl Pyrrolidone (PVP. K30) which a molecular weight M.W 40,000 with nano silver (50 nano) was used in this research as basematerial. 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 PVP K30 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 g and 0.02 mol. L^{-1} (PVA) by solving 8gm in 50 mL water placed on stirrer device for 30 min at room temperature for PVP and 15 min at 60°C according to Eq. 1 and 2:

$$C_x = n_x V \quad (1)$$

$$n_x = gWM \quad (2)$$

Where:

C_x = The concentration mol.L^{-1}
n_x = The number of moles
V = 50 mL volume of solvent
g = 8 g is the mass of PVP

Preparation of adhesive joint: The single shear lap joints of adherent were copper sample with dimension (25.4 *1.6 *90 mm) having a contact area (21.7*25.4 mm) 330 mm^2

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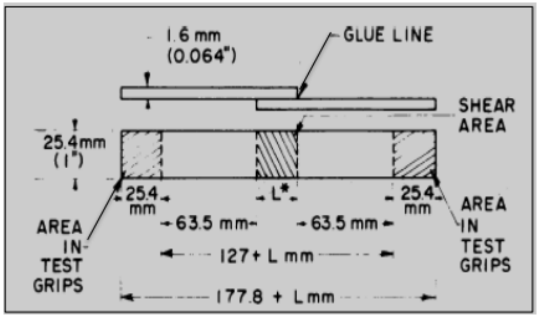

Fig. 1: Sample according ASTM D1002 -01

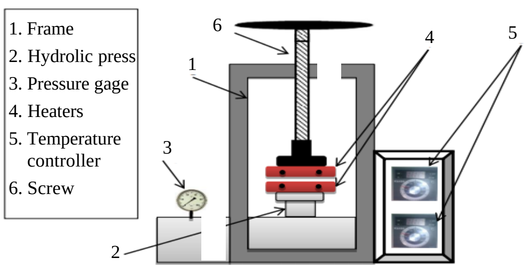

Fig. 2: Heating and pressure system

of the adhasive joint that was prepared according to ASTM D1002 (Anonymous, 2001) as shown in the Fig. 1.

The copper specimens were pressed at pressure 5 MPa and heating temperatures 200, 215, 225, 240 and 275°C for times 10 min by Khazaka *et al.* (2014) using press under heating system, pressure 5 MPa was chooses from on some references (Siow, 2014) as shown in the Fig. 2.

The temperature range 200-275°C were selected to be in consistent with the adhesive material properties. Where, the melting point of PVP is 172°C and PVA is 192°C according to the melting temperature test while the decomposition temperature reach to 483°C according to TGA test, so, the required temperature is with in this range with mixing 50% from PVP and 50% PVA and nano copper (50 nano diameter) are used. Curing times 10 min were chooses for reproached between and since, increasing the curing time in a test for more than 10 min produced weaking and burning of the adhesive material, formation of a black adhered region, the existence of cracks in the adhesive material structure and weakness in shear strength as shown in Fig. 3 (Diwan *et al*., 2017).

Shear test: The shear strength of the joint was found by using a universal tensile testing machine by drawing apart the two plates with a rate of 1 mm/min^{-1} as shown in Fig. 4.

An Artificial Neural Network (ANN) can be utilized for studies of the mechanical properties of different materials including the adhesive materials. In this

Fig. 3: Copper adherents after pressing

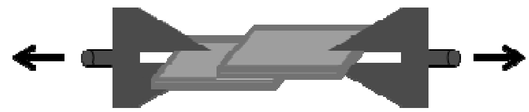

Fig. 4: Shear test

research, the ANN approach will be used for predicting certain mechanical properties of PVP/PVA with copper as adhesive material. These properties include shear effect. theseresults are tested under combined independent situations such as curing time, sintering temperature, concentration of copper-nanoparticles.

RESULTS AND DISCUSSION

Figure 5 and 6 show the Thermo Gram Analysis (TGA) and (DSC) of PVP and PVA, respectively which has been plotted with weight loss as a function of the temperature. These measurements were conducted for PVP and PVA precursor with a heating rate of 10°C/min in the temperature range of 40-800°C for PVP polymer and 50-350°C for PVA polymer.

It is clear that for PVP polymer the TGA initial weight loss 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 and 678.08°C, respectively. 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 closed to that obtained by Sivaiah *et al.* (2010).

Table 1 summary of results of curing temperature, load and ratio (PVP/PVA) at pressure 5 MPa and curing time 10 min.

Obtainnd results show that the highest values of shear load was reached at (50 and 50% PVA) which has better distribution in composite structure and has a dynamic viscocity is 37cP before and after this ratio (50% PVP/PVA) a nonhomogeneous structure in the adhesion area, causes a decrease in shear strength as shown in Fig. 7 (Ebnesajjad and Landrock, 2008).

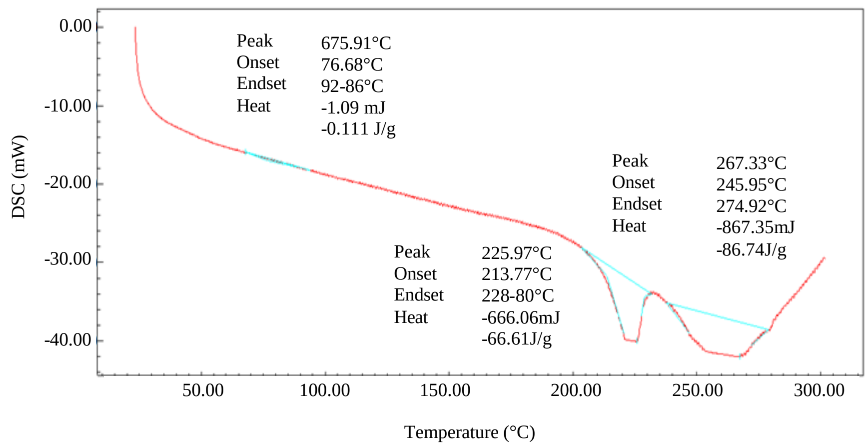

Fig. 5: TGA for PVP

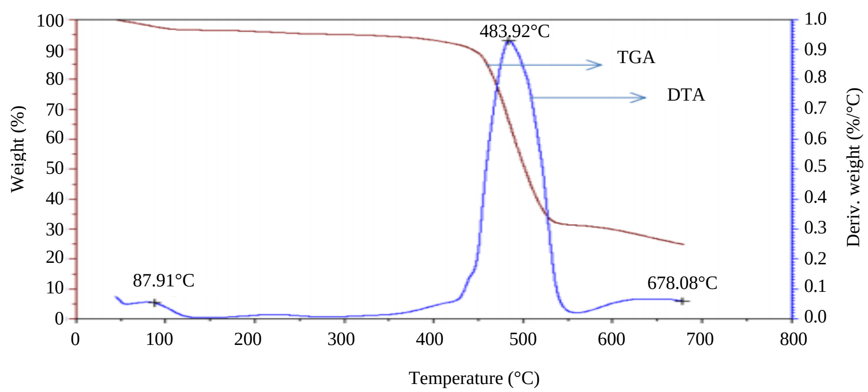

Fig. 6: DSC for PVA

Table. 1: Shear force with temperature and PVA/PVP

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	1210	720	498
240	445	1120	1250	680	620
265	0	0	920	715	515

Table 2: Shear force with temperature and concentration of Cu (%)

T(°C)	5%	10%	15%	20%
200	640	615	610	625
215	653	600	660	640
225	690	650	670	660
240	700	675	727	680
265	1360	1550	1760	920

Table 2 shows the summary of results of curing temperature, load and concentrate of nano copper at pressure 5 MPa.

This table manifests that the best result for shear load at (15% copper) as shown in Fig. 8, the pure and addition (5, 10%) of copper have non homogenous area and porosity, so that, a shear force at this concentration was low. The addition 15% Cu nano with a viscosity 42 cp particles to base adhesive was enough for homogenization of the paste area and well distortion and improve the shear load as shown in Fig. 9.

These models discussed the shear for 50% PVP/PVA mixed with copper nano particles. One and two hidden layer were utilized to estimate the properties of the PVP/PVA as the adhesive material. The effect of variation of the sintering temperature and the concentration of copper nano particles on the output results of the shear load for single lap joints were observed. These parameters can be defined as input variables of the model and the shear load may be the output of the ANN. Many attempts that indicated found that the best structure of the neural networks for predicting of shear loads are the two hidden layer structure which can be coded as 2-6-7-1. For shear load. It is clear that the correlation coefficients are 1 and 0.9982 for training and 0.99344 for testing samples, respectively. Where the best fit lines for predicting properties for training and testing samples of this model are shown in Fig. 10.

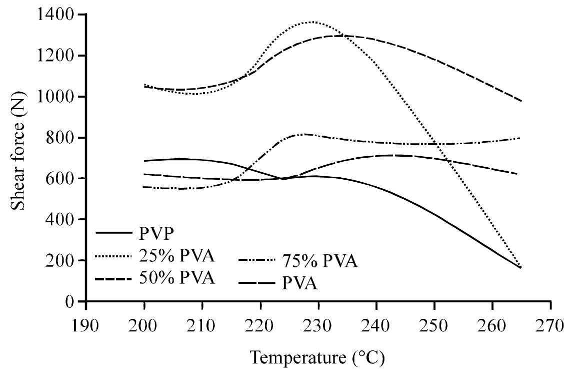

Fig. 7: Shear force with temperature

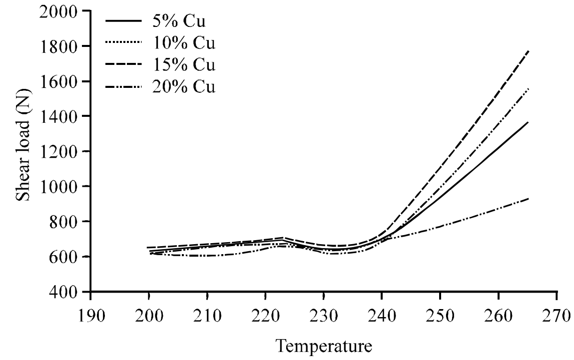

Fig. 8: Shear force with copper concentrations

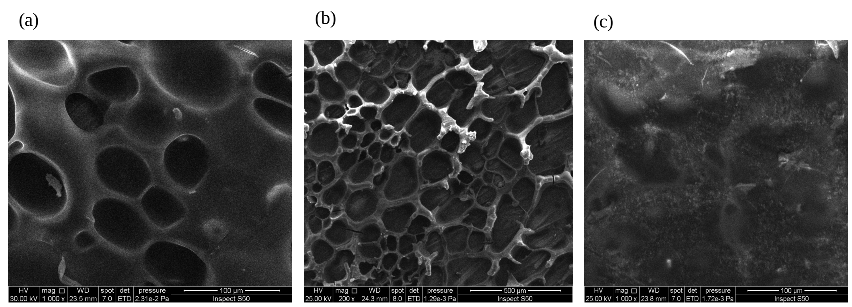

Fig. 9: SEM for: a) Pure; b) 5 and C) 15% copper concentrations

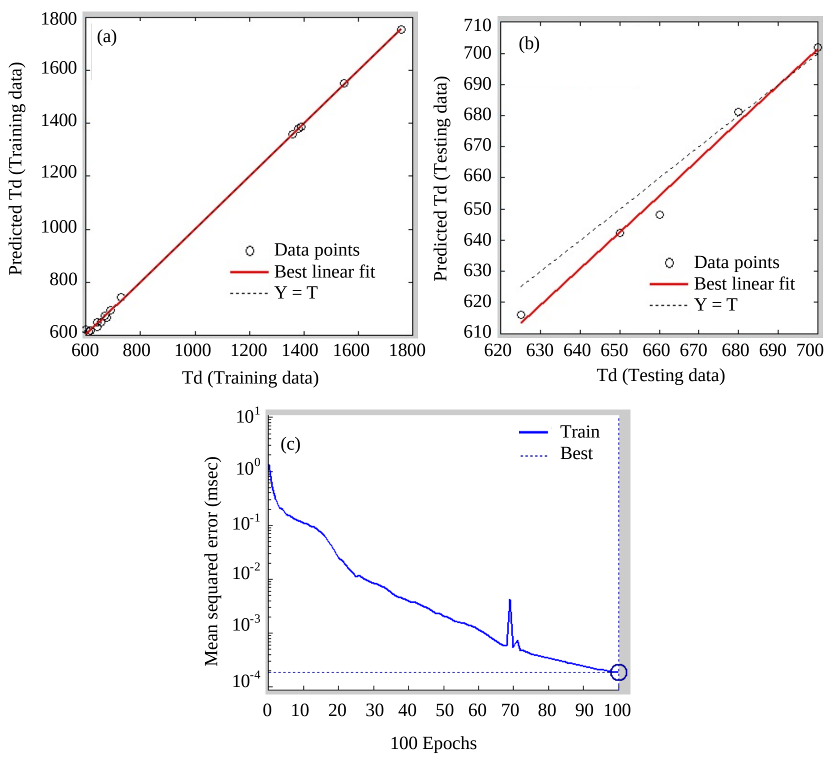

Fig. 10: Testing and training data of ANN: a) Outputs vs. Targets R = 0.99982; b) Outputs vs. Targets R = 0.99344 and c) Best training performence is 0.00018543 at epoch 100

Table 3: Experimental and neural shear force

Time (min)	Temp. (°C)	Exp.shear force	Nur.shear force	Error (%)
10	200	640	631.3526864	1.351142757
10	215	653	648.3694410	0.709120823
10	225	690	692.3619081	-0.342305515
10	240	700	701.9585573	-0.279793902
10	275	1360	1359.9899200	0.000741186
20	200	615	615.4234597	-0.068855233
20	215	600	621.0294482	-3.504908027
20	225	650	642.3889364	1.170932862
20	235	675	664.5963016	1.541288659

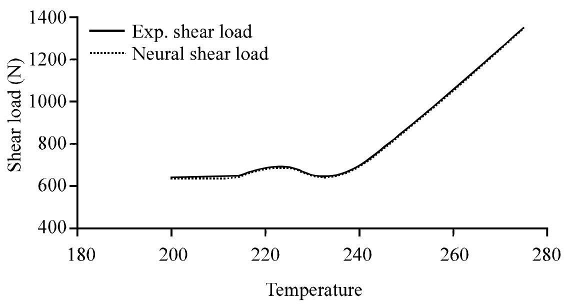

Fig. 11: Predication and experimental shear load

Table 3 shows the summary of results of curing temperature, time, experimental load and predication load in neural net work for PVP/PVA withNano copper at pressure 5 MPa as shown in Fig. 11, that good result between the experimental and prediction shear load.

CONCLUSION

From the above results, the following conclusions can be written: The temperature effect on the shear strength is a slight direct proportion from 200-225°C but it is inverse proportion from 225-275°C. Increasing in heating time caused weakness in shear strength of lap joints. The best value of adhesive joint loading is for 50 and 50% PVP, 10 min and 225°C because the viscosity of PVP k30/PVA increased slightly in direct proportionality from 200-225°C but decreased during the range 225-275°C.

There is non-homogeneity in the microstructure of adhesive joints forconcentration of copper for 10, 5, 20%. There is a degradation of shear strength of lap joints at temperature 275°C due to the crack formation in the microstructure of adhesive joints.

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