

CHARACTERISTICS OF HOOK AND INTERMIXING LAYER OF FRICTION STIR SPOT WELDING AA5052/C10100 JOINTS REINFORCED BY ZNO NANO-PARTICLES

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ABSTRACT

FSSW is a solid state joining method gained its significance as an alternate welding technique in the in electrical, electronic and automobile industries that used FSW welding. FSSW can be used to welding dissimilar joints of material with variation in metallurgical and mechanical characteristics. In this paper, dissimilar welding joints of aluminum alloys (5052) and pure copper (10100) with a thickness of 1.67 mm were welded by FSSW. The variation of rotational speed, dwell time and filler content have been used. The welding joints has been examined by tensile shear test. The scanning electron microscope SEM and X-ray diffraction are used to investigate the hook and intermixing layer characteristics. It was found the best welding condition is (1400 rpm) and (30 sec) which the joint is possess the tensile shear force (3340 N). Adding ZnO filler led to increase the thickness of the copper hook, and expanding the stir zone (weld width bond). Joints with V2-ZnO have highest thickness of intermixing layer (IM), length of weld width bond and best tensile shear force (4300 N).

KEYWORDS

Friction Stir Spot Welding, 5052 Alloy, Dissimilar Metal Welding, Filler Content.

1. INTRODUCTION

Joints of aluminum and copper are required in electrical and electronic industries to utilize the individual properties of both the materials such as good thermal conductivity and high resistance to corrosion in volatile environments (Siddharth & Senthilkumar 2017). As a solid-state joining technique with low and control-lable heat input, friction stir welding (FSW) shows a great potential in producing joints between dissimilar alloys. FSW can join Al alloys at a plastic state and therefore avoid some fusion welding defects (Liang et al. 2013; Dong et al. 2019). Friction stir spot welding (FSSW) is a spot welding process and an alternative joining method of friction stir welding (FSW) technique (Mubiayi, Akinlabi & Makhatha 2019). A non-consumable rotating tool is plunged into the similar or dissimilar metal joints to be welded. The rotating tool is held in this situation for a predetermined time after the selected plunge depth is achieved. this time is called the dwell time. After that, the rotating tool is removed from the weld joint departing a keyhole spot weld behind (Siddharth & Senthilkumar 2018), as shown in Fig. 1. By FSSW can be join dissimilar metals without addition filler material (ASM Metals Handbook 1993). But reinforcing the welding zone can be done by incorporating nanoparticles, to improve the properties such as strength, stiffness and producing structures with enhanced properties (metal matrix composites joints (MMC)). Therefore, there is a great interest to use different of a nano-powders to strengthen the dissimilar joints (Sun & Fujii 2011; Jasiūnienė et al. 2017; Kianezhad & Honarbakhsh Raouf 2019).





A number of investigations were being carried out for joining materials by using FSSW process and adding fillers over the years as follows: (Mubiayi & Akinlabi 2017) investigate the characterization of the intermetallic compounds in aluminium and copper friction stir spot welds. They found that the most common intermetallic compounds formed in the spot weld samples were $Al_{\epsilon}Cu_{9}$, $Al_{c}Cu_{7}$, $Al_{r}Cu_{3}$ and $Al_{2}Cu$, which also showed low peaks intensity in

XRD results. Higher microhardness values were obtained in the stir zone for all the welds due to the smaller grains. The high hardness values correlated to the high peaks of the intermetallics formed at the interface. Regensburg et al. (2019) investigate the liquid interlayer that formation during friction stir spot welding of aluminum/copper. They found that the wetting layer effect at the interface shows a positive influence on the shear strength with ductile failure behavior even at high layer thickness. The formation of intermetallic compounds other than Al₂Cu was mostly inhibited by the short process times and high cooling rate. Asadollahi & Khalkhali (2018) investigate the enhanced welding features by incorporating SiC strengthening nanoparticles to AA 6061-T6 aluminum alloy welded by FSSW. They found that the nanoparticles gathering at grain borders and avoided the grain size from growth. In addition, the tensile shear strength and the average Microhardness of Stir Zone raised by up to 28% and 24% respectively when addition of SiC nanoparticles. The present work investigate the effect of adding ZnO Nano filler on the characteristics of hook and intermixing layer of Al/Cu joints.

2. EXPERIMENTAL WORK

2.1. Materials used

A sheets of AA5052 ($100 \times 30 \times 1.68$) mm, they have a chemical composition, as shown in Table 1 and 2 respectively. The samples are joined in a configuration where the AA5052 sheet was placed over the C10100 sheet with 30mm overlap, as shown in Fig. 2.

Element Wt%	Mg	Fe	Cu	Cr	Mn	Si	Al
Standard	2.2-2.8	0.4 max	0.1 max	0.1 max	0.15 max	0.25 max	95.7-97.7
Obtained	2.66	0.288	0.022	0.0329	0.179	0.0424	Bal.

Table 1. The chemical composition analysis of AA5052 alloy.

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Element Wt%	Cu	Ag	Fe	Pb	S	Ca	Р	Others
Standard	99.9	0.0025	0.001	0.0005	0.0015	0.0001	0.0003	Bal.
Obtained	99.9	0.0016	0.0012	0.0008	0.001	0.0001	0.0005	Bal.

Table 2. The chemical composition analysis of pure copper C10100 sheet.



Fig. 2. The dimensions of dissimilar welding Specimen.

2.2. FSSW procedure

Friction stir spot welding process is performed by the universal milling machine (model F1-250 with spindle rotational speed between 40 to 2000 rpm). The used tool was made from high speed steel (HSS) that has a flat shoulder with a pyramided cylindrical shape pin, which has the dimensions that shown in the Fig. 3. Welding process is carried out by fixing the aluminum alloy with the copper sheet on the machine anvil as a lab-joint type by using rigid fixtures to prevent the movement of sheets during the welding process. The different welding parameters of rotation speed and dwell time that used in welding process. The plunge depth (2.3) mm and plunge rate (14) mm/min that used throughout welding process. The ZnO filler material added manually and immediately prior to welding through a pre-hole at the top sheet for a three contents separately, as shown in Fig. 4. Table 3 shows the dimensions of ZnO filler content. The ZnO powder has the diameter 50 nm and purity 98%.

Ellen ernen Ander	Dimensions of Filler content					
Filler content No.	Diameter "D" (mm)	High "H" (mm)	Vol. (mm ³)			
V1	1	1.68	1.32			
V2	2	1.68	5.28			
V3	3	1.68	11.88			

Table 3. The dimensions of filler content.



Fig. 3. The dimensions and geometry of welding tool.



Fig. 4. Schematic of illustration of adding ZnO filler material between the plates before FSSW.

2.3. Tensile shear test

A universal tensile testing machine (WEW-100, China) has been used to evaluate the lapshear joint. Specimens are pulled at speed rate of 2 mm/min.

2.4. Scanning electron microscope (SEM)

The thickness and morphology of the hook with the intermixing layer in spot weld joints were investigated by using scanning electron microscopy (SEM, VEGA3 LM TESCAN Company, Czech).

2.5. Microstructure examination

The best conditions of cross sectioned weld joints with and without ZnO filler addition were tested by using optical microscope type NMM-800RF. The metallographic preparation was done by using different grades of emery papers and polishing cloth with MasterPrep suspension of $0.05 \,\mu$ m.

2.6. X-ray diffraction (XRD)

X-ray diffraction (XRD) was performed to define the presence of intermetallic compounds (IMC) phases in the cross section joints by using (LabX 6000, Shimadzu, Japan origin). The resulted peaks are analyzed by HighScorePlus 3.9 software with COD database 2016.

2.7. Microhardness test

Micro-hardness test was performed on cross section joints by using Digital Micro Vickers Hardness Tester (TH715 Time, China). Vickers microhardness measurements were taken in a contour line for different one side regions of the spot weld cross-section by utilizing 4.5 N load for 15 sec.

3. RESULTS AND DISCUSSION

3.1. Optimizing of welding parameters and filler content

Table 4 shows the results of the tensile shear test of joints at different rotation speed and dwell time. From this table, it can be seen that the best welding conditions are in specimen No.4 at (1400 rpm) rotation speed and (30 sec) dwell time. In these conditions, the obtained tensile shear failure load was the highest of (3340 N). Since the rotation speed is responsible for heat generation and the mechanical effect of stirring, the low rotation speed and dwell time result in low heat generation and mechanical effect of stirring. Consequently a thin and little copper ring extrude from the lower to upper sheets and insufficient temperature for recrystallization to begin in the stir zone. For this reason the weld would be weak . On the other hand, increasing the rotation speed (from 1000 rpm to 1400 rpm) will lead to increase the extruding of copper ring and consequently the weld strength would be improved comparing with the previous conditions. Although, the intermixing layer formed at this speed and the recrystallization temperature are dependent on the dwell time.

FSSW condition No.	Rotation speed (rpm)	Dwell time (sec)	Av. Tensile Shear load (N)
1	1000	15	1240
2	1000	30	1610
3	1400	15	2320
4	1400	30	3340
5	2000	15	2050
6	2000	30	1780

Table 4. Results of tensile shear test of Al-5052/Cu-C10100 joints.

Fig. 5 shows the effect of (ZnO) filler content on the behavior of tensile shear load and deflection of joints welded at (1400 rpm) rotation speed and (30 sec) dwell time. For V1 and V2 filler content, the tensile shear load increased by 21% and 29% respectively, while the ductility of spot welds has been decreased by 32% and 46% respectively compared to specimen No.4. However, for the V3 filler content, the tensile shear load and spot weld ductility is decreased by 7.8% and 65.5% respectively as compared to the specimen No.4.

Adding ZnO filler led to refine grain size due to the presence of filler particles on grain boundary that prevent the grain from growth (pinning strengthening) and this would Improve the hardness of SZ and tensile shear force .In addition, the filler particles will enhance the dispersion of intermetallic compounds in matrix such as $AlCu_4$ at matrix of Al/Cu interface (dispersion strengthening), which in turn will led to the formation of MMC joints. The high content of filler particles cause brittleness of joint due to the excessive hardness. No linear relationship between ZnO filler content with tensile shear load is observed. The best filler content is V2, that have the maximum tensile shear (4300 N).



Fig. 5. The effect of (ZnO) filler content on the behavior of tensile shear load and deflection of joints welded at (1400 rpm) rotation speed and (30 sec) dwell time.

3.2. Microstructure of welds

The characteristics of the hook and the intermixing (IM) layer such as size, thickness and penetration angle consider the most important features of this type of welding that play an important role in promoting the bonding between sheets. In addition, the failure load are highly influenced by weld bond width (Shiraly et al. 2014; Sadeghi et al. 2015; Regensburg et al. 2019). Fig. 6 show the SEM photograph of cross section joints were welded by (1400 rpm and 30 sec) with V1, V2 and V3 ZnO filler content. By using ImageJ software, the SEM photographs were analyzed and the obtained results were listed in Table 5.



Fig. 6. The SEM photograph of cross section joints were welded by (1400 rpm and 30 sec) (A) without filler (B) with V1-ZnO filler, (C) with V2-ZnO filler and (D) with V3-ZnO filler.

Table 5. The dimensions and features	of hook and IM layer	of FSSW joints.
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Joints No.	Highs of hook (µm)	Av. Thickness of hook (µm)	Size of bond width (µm)	Av. Thickness of IM layer (µm)	Layer type of IM	Penetratio n angle (deg.)
A-without	680	94	424	67	Discontinuous	65
B- withV1	524	247	251	120	Discontinuous	119
C- with V2	611	220	534	179	Continuous	99
D- with V3	572 (Cleavage)	266	456	73	Continuous	85

The interlocking between the two plates is caused by Cu ring (or the hook) excreted from the lower sheet into an upper aluminum sheet, helping to maintain the plates during tensile testing and achieve a strong pre-failure load. The strong joints tends to have a thick hook without cleavage, orients outwards from the tool axis, and terminates away from the keyhole for (Rao, Yuan & Badarinarayan 2015). It can be seen from results in Table 4 the addition of ZnO nano-filler led to increase hook thickness because of increasing the plunge depth, as a result to filler particles presence that will led to push plasticized copper, therefore increasing the extruding of copper ring. The cleavage defect clearly appears in joint No. D with V3 filler content, due to rise heat, as a result to presence large amount of filler particles that leads to increase the heat of friction. The lamellar structure layer formed in the Al/Cu interface that contain a mechanical mixing of intermetallic compounds in aluminum matrix, is called intermixing layer (Sadeghi et al. 2015; Regensburg et al. 2019). The IM layer has a beneficial effect on the load of failure, which increase by increasing the thickness of the layer (Shiraly et al. 2014; Regensburg et al. 2019). There was a clear increase in the thickness of the intermixing layer when adding the filler material, as in joints No. B and C. However, for joint No. D the thickness of the intermixing layer has been decreased due to presence high amount of filler particles that formed an obstacle to the spread of the intermixing layer. The distance from the tip of hook to keyhole interface is called as the weld bond width. The strength of spot welds increased whenever the weld bond width increased (Shiraly et al. 2014; Rao et al. 2015). Joints with V2-ZnO have highest thickness of intermixing layer and weld bond width. Therefore, it is likely to have the best welding strength. Fig. 7 show the microstructure of layer formed on the hook and the EDS in joint No. C. The IM layer at interface solidify to an eutectic with a lamellar and anomaly structure, when temperature approaching to the eutectic temperature and cooling rapidly due to presence large amount of melt is undergo solidify to eutectic structure at a brief interval of time (Shiraly et al. 2014; Regensburg et al. 2019). The laminar layer was appeared, with fine grain size, consist of two types of intermetallic compounds were embedded in the aluminum matrix, these compounds are (Al₂Cu that appear in light brown) and (Al₂Cu-Mg, which appears in dark brown due to the presence of magnesium in them) (Liu et al. 2014). The presence of (Al₂Cu-Mg) grains gives the bonding region desirable combination of strength, fracture toughness, and better isotropy in shear and stiffness. The (Al₂Cu) grains gives the bonding region excellent hardness (Zhang et al. 2012; Shi et al. 2014).



Fig. 7. The lamellar and anomalous microstructure form of IM layer (left) and the EDS (right) for joint No. C.

3.3. X-ray diffraction (XRD)

Fig. 8 show the XRD results of the cross section of weld joints that welded at (1400 rpm and 30 sec), with V2 ZnO filler content and without addition filler. It can be seen the copper and aluminum appear as the main elements, with very high of intensity peak. The intermetallic compounds Al₂Cu, Al₄Cu₉ and Al₂Cu-Mg, that were formed during welding between different amounts of scratched copper particles with aluminum (and sometimes magnesium) were appear in the stir zone due to the availability of proper heat. Also were noted that the peaks of these compounds within a very low of the intensity range (Mubiayi & Akinlabi 2017). In addition, the intermetallic compound Mg₂Si is appeared, which is found mainly as a strengthening compound in aluminum. The ZnO nano particles peaks were appeared which also within a low of the intensity range in Fig. 6 with V2-ZnO (Khorsand Zak et al. 2011; Pratap Goutam, Kumar Yadav & Jyoti Das 2017).



Fig. 8. The XRD results of the cross section of weld joints that welded at (1400 rpm and 30 sec), with V2 ZnO filler content and without addition filler.

3.4. Microhardness test

The microhardness values of the parent materials before welding are (98-103) Hv for copper (C10100) and (70-75) Hv for aluminum (AA5052). Fig. 9 shows the microhardness values of SZ with ZnO filler content for FSSW joints. Generally, the grains in SZ becomes dynamically fine, equiaxed and predominantly finer than the base metal grains. The microhardness values in SZ are higher than the TMAZ, HAZ and BM zones because of SZ has a finer grain structure and work hardening (by intensive stirring) during FSSW process (Özdemir, Sayer & Yeni 2012; Siddharth & Senthilkumar 2016; Sanusi & Akinlabi 2017). Adding filler led to refine grain size due to the presence of filler particles on grain boundary that prevents the grain from growth (Zener effect) and this would improve the hardness of SZ and tensile shear force (Barmouz et al. 2011; Bodaghi & Dehghani 2017). In addition, the filler particle will enhance the mixing and flowing of marital compounds, which in turn will lead to the formation of MMC joints. The high content of filler particles causes brittleness of joint due to the excessive hardness as in V3 filler content. Fig. 10 shows the results of the Vickers microhardness value through the cross section of specimen with V2-ZnO filler. The behavior of microhardness values varies depend on the distance from the welding spot. Four different regions were observed: (SZ), (TMAZ), (HAZ) and base metal zone. The microhardness values of TMAZ and HAZ decreased and become lower than the base metals due to their coarse grains size and dissolution of strengthening precipitates as a result of exposure of this zones to thermal cycles (Yuana et al. 2011; Asadollahi & Khalkhali 2018). The hardness values begin recovering to original values, whenever moving away from the shoulder effect.



Fig. 9. The Microhardness values of SZ with ZnO filler content for FSSW joints that welded at (1400 rpm and 30 sec).



Fig. 10. The Vickers microhardness values through the cross section of specimen with V2-ZnO filler that welded at (1400 rpm and 30 sec).

4. CONCLUSIONS

From the current study, it can conclude the following:

1. The best welding conditions are (1400 rpm) rotation speed, (30 sec) dwell time that produce a maximum tensile shear force (3340 N).

2. The adding of ZnO nano-filler led to increase hook thickness and the intermixing (IM) layer.

3. The best filler content is V2, which have been the maximum tensile shear (4300 N), the highest thickness of intermixing (IM) layer (179 μ m) and the weld bond width (534 μ m).

4. Presence of the intermetallic compounds Al_2Cu , Al_4Cu_9 and Al_2Cu -Mg in intermixing layer, it would help to enhance the mechanical properties of the joints.

5. No linear relationship between ZnO filler content and tensile shear load.

6. Linear relationship between microhardness values of SZ and ZnO filler content up to (11.88 mm³).

5. REFERENCES

Asadollahi, M. & Khalkhali, A. (2018) 'Optimization of mechanical and microstructural properties of friction stir spot welded AA 6061-T6 reinforced with SiC nanoparticles', Materials Research Express, 5(11).

Barmouz, M., Asadi, P., Besharati Givi, M.K. & Taherishargh, M. (2011) 'Investigation of mechanical properties of Cu/SiC composite fabricated by FSP: Effect of SiC particles' size and volume fraction', Materials Science and Engineering A, 528(3), 1740–1749.

Bodaghi, M. & Dehghani, K. (2017) 'Friction stir welding of AA5052: the effects of SiC nano-particles addition', International Journal of Advanced Manufacturing Technology, 88(9–12), 2651–2660.

Dong, Z., Song, Q., Ai, X. & Lv, Z. (2019) 'Effect of joining time on intermetallic compound thickness and mechanical properties of refill friction stir spot welded dissimilar Al/Mg alloys', Journal of Manufacturing Processes, 42, 106–112.

Jasiūnienė, E., Žukauskas, E., Dragatogiannis, D.A., Koumoulos, E.P. & Charitidis, C.A. (2017) 'Investigation of dissimilar metal joints with nanoparticle fillers', NDT and E International, 92, 122–129.

Khorsand Zak, A., Razali, R., Abd Majid, W.H. & Darroudi, M. (2011) 'Synthesis and characterization of a narrow size distribution of zinc oxide nanoparticles', International Journal of Nanomedicine, 6(1), 1399–1403.

Kianezhad, M. & Honarbakhsh Raouf, A. (2019) 'Effect of nano-Al2O3 particles and friction stir processing on 5083 TIG welding properties', Journal of Materials Processing Technology, 263, 356–365.

Liang, Z., Chen, K., Wang, X., Yao, J., Yang, Q., Zhang, L. & Shan, A. (2013) 'Effect of tool offset and tool rotational speed on enhancing mechanical property of Al/Mg dissimilar FSW

joints', Metallurgical and Materials Transactions A: Physical Metallurgy and Materials Science, 44(8), 3721–3731.

Liu, Y., Liu, M., Luo, L., Wang, J. & Liu, C. (2014) 'The solidification behavior of AA2618 aluminum alloy and the influence of cooling rate', Materials, 7(12), 7875–7890.

Mubiayi, M.P. & Akinlabi, E.T., 2015, 'Friction stir spot welding between copper and aluminium: Microstructural evolution', Lecture Notes in Engineering and Computer Science, 2, 819–823.

Mubiayi, M.P. & Akinlabi, E.T. (2017) 'Characterization of the intermetallic compounds in aluminium and copper friction stir spot welds', Materials Today: Proceedings, 4(2), 533–540.

Mubiayi, M.P., Akinlabi, E.T. & Makhatha, M.E., 2019, Current Trends in Friction Stir Welding (FSW) and Friction Stir Spot Welding (FSSW), vol. 6, Springer, Switzerland.

Özdemir, U., Sayer, S. & Yeni, Ç. (2012) 'Effect of pin penetration depth on the mechanical properties of friction stir spot welded aluminum and copper', Materialpruefung/Materials Testing, 54(4), 233–239.

Pratap Goutam, S., Kumar Yadav, A. & Jyoti Das, A. (2017) 'Coriander Extract Mediated Green Synthesis of Zinc Oxide Nanoparticles and Their Structural, Optical and Antibacterial Properties', Journal of Nanoscience and Technology, 3, 249–252.

Rao, H.M., Yuan, W. & Badarinarayan, H. (2015) 'Effect of process parameters on mechanical properties of friction stir spot welded magnesium to aluminum alloys', Materials and Design, 66, 235–245.

Regensburg, A., Petzoldt, F., Benss, T. & Bergmann, J.P. (2019) 'Liquid interlayer formation during friction stir spot welding of aluminum/copper', Welding in the World, 63(1), 117–125. Sadeghi, E., S., K.J., Ahmad, M. & Habibi, M., 2015, Investigation on microstructure and mechanical properties of dissimilar lap Friction Stir Spot Welding of aluminum 1050 to pure copper, Management Study with Executives (MSE): Bogazigi University Engineering Club.

Sanusi, K.O. & Akinlabi, E.T. (2017) 'Material Characterization of Dissimilar Friction Stir Spot Welded Aluminium and Copper Alloy', IOP Conference Series: Materials Science and Engineering, 225(1).

Shi, H., Tian, Z., Hu, T., Liu, F., Han, E.H., Taryba, M. & Lamaka, S. V. (2014) 'Simulating corrosion of Al2CuMg phase by measuring ionic currents, chloride concentration and pH', Corrosion Science, 88, 178–186.

Shiraly, M., Shamanian, M., Toroghinejad, M.R. & Ahmadi Jazani, M. (2014) 'Effect of tool rotation rate on microstructure and mechanical behavior of friction stir spot-welded Al/Cu composite', Journal of Materials Engineering and Performance, 23(2), 413–420.

Siddharth, S. & Senthilkumar, T. (2016) 'Optimization of friction stir spot welding process parameters of dissimilar Al 5083 and C 10100 joints using response surface methodology', Russian Journal of Non-Ferrous Metals, 57(5), 456–466.

Siddharth, S. & Senthilkumar, T. (2017) 'Study of tool Penetration Behavior in Dissimilar Al5083 /C10100 Friction Stir Spot Welds', Procedia Engineering, 173, 1439–1446.

Siddharth, S. & Senthilkumar, T. (2018) 'Optimizing Process Parameters for Increasing Corrosion Resistance of Friction Stir Spot Welded Dissimilar Al-5086/C10100 Joints', Transactions of the Indian Institute of Metals, 71(4), 1011–1024.

Sun, Y.F. & Fujii, H. (2011) 'The effect of SiC particles on the microstructure and mechanical properties of friction stir welded pure copper joints', Materials Science and Engineering A, 528(16–17), 5470–5475.

Yuana, W., Mishra, R. S., Webb, S., Chen, Y. L., Carlson, B., Herling, D. R. & Grant, G. J. (2011) 'Effect of Tool Design and Process Parameters on Properties of Al Alloy 6016 Friction Stir Spot Welds', Journal of Materials Processing Technology, 211, 972-977.

Zhang, J., Huang, Y.N., Mao, C. & Peng, P. (2012) 'Structural, elastic and electronic properties of θ (Al 2Cu) and S (Al 2CuMg) strengthening precipitates in Al-Cu-Mg series alloys: First-principles calculations', Solid State Communications, 152(23), 2100–2104.