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Manufacturing of Nano-Surface Composites on AA7075 Using Friction Stir Processing for Military Vehicle and Transportation Applications

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Abstract: Friction stir processing (FSP) is solid state process that has been used recently for microstructural modification and surface composite development. In this study FSP has been used to incorporate Al_2O_3 nanoparticles on the surface of AA7075 with the aim of manufacturing low density high hardness surface nanocomposites for military vehicles and transportation applications. The Al_2O_3 nanoparticles were packed into grooves of 1mm width and 7.5mm depth that were machined in 15mm thick plates of AA7075. The FSP was done using FSP tool of cylindrical probe of 6mm diameter and 20mm diameter shoulder. The tool rotation rate and tool travel speed were selected based on available literature to produce uniform dispersion of Al_2O_3 nanoparticles. The effect of post processing heat treatment after incorporating Al_2O_3 nanoparticles into different temper conditions of AA7075-T6 and AA7075-O was investigated. The developed nanocomposites were investigated and characterized using optical microscopy, scanning electron microscopy, and hardness testing.

Keywords: Friction stir processing, nanocomposites, AA7075, optical microscope, SEM, hardness testing.

1. Introduction

Aluminum alloys are usually used in many lightweight applications such as aircraft structures, marine frames, transportation industries and military vehicles due to their high specific strength [1], particularly aluminum based metal matrix composites of improved resistance to wear and fatigue and exhibiting high strength [1,2]. However, Metal Matrix Composite (MMC) are known to suffer from poor toughness and low ductility since adding hard ceramic reinforcements causes embrittlement and leads to imposed restrictions in their applications. The idea of replacing bulk metal matrix composites by surface composites is inspired from the well-known metallurgical processes of surface hardening, which yields a hard surface of the material which is reinforced by ceramic particles while the rest is still keeping the original properties and structure with higher toughness. By using different techniques (e.g. high energy electron beam irradiation and plasma spraying) surface composites have been developed [1,5,6].

Friction stir processing (FSP) is a new solid state processing technique which was established by Mishra et al. [7-9]. FSP is a promising technique for modification of the microstructure depending on the main concept of friction stir welding (FSW). The main idea of the FSP is

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very simple where the rotating tool with pin and shoulder is integrated into a single piece of material. In addition, it has been applied to the manufacture of a surface composite on an aluminum substrate [9,10] and is identical to powder metallurgy (PM) aluminum alloys, cast aluminum alloys and metal matrix composites (MMC) [11-13] in the sense of producing a new composite with enhanced properties to the main matrix alloy. FSP has special advantages compared to other techniques of metalworking due to many reasons; First, FSP is a multipurpose technique with a full function for the synthesis, manufacturing and processing of materials [9], second, FSP is a short route with one step processing that accomplishes densification, homogeneity and refinement in microstructure [9], third, by adjusting FSP parameters, (tool design and active heating), the mechanical properties and microstructure can be precisely controlled [9], and fourth, by changing the length of the tool pin with the depth being between several hundred micrometers and ten of millimeters, the processed zone depth can be changed and it is so difficult to be obtained using other techniques of metal working [9].

FSP has been used recently for the development of MMCs in a number of metallic alloys [14-16] for example Bozorg et al. [14] used FSP to incorporate nano alumina in AA6082. Mahmoud et al [15] used FSP has examined the effects of tool probe shape and size on the formation of surface composite by uniformly distributing SiC particles into a surface layer of an A1050-H24. Also Azizieh et al. [16] have incorporated nano alumina particles in magnesium alloy AZ31 using FSP. The aim of this work was to incorporate nano alumina particles in AA7075 for military vehicles and automotive applications, where the enhancement in surface hardness and strength properties while maintaining the bulk toughness and ductility will allow the expansion of these novel materials in applications where light weight combined with high specific strength and toughness are required.

2. Experimental

The material used in this study is AA7075 sheet plate of 15mm thickness and composition limit given in Table 1. AA7075 was received in T6 temper condition with a measured Vickers hardness number of 177Hv. Some parts of AA7075-T6 were annealed to have it in the O temper condition of measured Vickers hardness number 66Hv. Both AA7075-T6 and AA7075-O were used as a metal matrix and Al_2O_3 nanoparticles with average size of 40 nm as the reinforcements. Work pieces were prepared with a length of 200 mm, width of 150 mm. A groove was machined through the surface of the AA7075 with a depth of 7 mm and 1mm width. Al_2O_3 nanoparticles were packed into the grooves after closing the sides of each groove to prevent escaping of nanoparticles during FSP. Then a probeless tool of 20mm shoulder diameter was used for top closing of the grooves after packing of the nanoparticles at the same FSP parameters used.

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	others	Al
Wt.%	0.40	0.50	1.2-2	0.3	2.1-2.9	0.18-0.28	5.1-5.6	0.20	0.15	Bal.

 Table 1 Chemical composition limits of AA7075

FSP using the conventional FSP tool made of H13 tool steel that heat treated to obtain 55HRC hardness was carried out. The FSP tool dimensions were of 6mm probe diameter, 10mm probe length and 20mm diameter shoulder. FSP was carried out at constant rotation rate of 840 rpm and constant traverse speed of 40mm/min and tool tilt angle was set at 3°. The material was processed in O and T6 temper conditions for one, two, three and four passes all

in the same direction. After FSP the material of O temper condition was age hardened to T6 temper condition by solution treatment at 515° C and water quenching followed by age hardening at 120° C for 24hrs.For the sake of this work the designations R4 will be used to describe the O temper condition and the designation R7 will be used to describe the T6 condition.

After the manufacturing of the new nano-surface dispersed material, samples from the different FSP processed materials (O, T6 and T6 after FSP) were cut perpendicular to the processing direction. The samples were initially mounted using hot mounting press and prepared according to the standard preparation technique starting with grinding of different grades up to 1200 size paper and then mechanically polished using 6 μ m, 1 μ m diamond suspension and using 0,05 μ m alumina suspension. A number of material characterization tests were carried out; namely: optical microstructure, microhardness, and scanning electron microscopy (SEMZeiss Leo Supra 55) investigation. The samples for metallographic examination and after final polishing were etched using diluted keller's reagent of chemical composition (100ml distilled water, 10 ml HNO3, 10 ml HCL and 2ml HF) for 20 sec. The microstructure investigation was carried out for all samples using optical microscopy and for only one sample from each condition using SEM.

The microhardness measurements were made on a Vickers Microhardness Tester machine where a number of at least 20 readings were taken at 0.5 mm intervals, using load of 4.9 N through the whole processed zone.

3. Results and Discussion

3.1 Macrostructure Investigations

A friction stir processed surface usually includes 4 distinct zones: stirred zone SZ, thermally mechanical affected zone TMAZ, heat affected zone HAZ and the base metal BM.

The friction stir processed surface for all samples showed semi-circular streaks at contact surface between the shoulder and the plate. The following Figure 1 shows the typical macrographs of the FSPed AA7075-O before and after T6 heat treatment. It is worth noticing that the currently used processing parameters (linear and rotational speeds) caused the appearance of tunnel type defects in the form of cavities that have been reported [17] to result from low tool rotational and high welding speeds. The cavities usually appear in the advancing side of the direction of the pin and are attributed to insufficient heat input during the process [17]. However, these defects are usually a result of poor casting quality [17].

3.2 Optical Microstructure Examination

Figure 2 illustrates the advancing side (AS) and the retreating side (RS) at the transverse cross section of FSPed AA7075. IT can be observed that the AS is sharp with clear interface between the base material grain structure and the new fine grain structure in the NG region.

Figure 3 illustrates the onion rings in the NG region of FSPed AA7075-O before and after T6 heat treatment.



Figure 1 Optical macrograph of FSPed AA7075-O (left); and after heat treatment to T6 condition (right).



Figure 2 Optical microstructure at the advancing side (AS) (right), and retreating side (RS) of FSPed AA7075-O (left).

Figure 4 shows the grain structure before and after T6 heat treatment of FSPed AA7075-O. Before heat treatment the grain structure is extremely fine however after heat treatment some grain growth can be observed. It should be noted here that the grain growth is just of a normal type with no abnormal grain growth occurring. It has been reported in many studies [18] that the post weld heat treatment of FSPed or FSWed aluminumis accompanied with abnormal grain growth. The non-existence of AGG in this case can be attributed to the dispersion of the material with Nano alumina particles.

In the Figure 5 the optical microstructure of AA7075-T6 FSPed in T6 condition is illustrated. Initially the significant grain refining can be observed clearly from the top right micrograph. Also two different grain structure regions in the NG can be observed one is dark and the other is bright as can be seen from top left image. In the bottom micrographs, either the dark and bright regions are illustrated at higher magnification, from which it can be observed that the grain structure has clear and sharp grain boundaries in the dark region while it has a quite diffusive grain structure in the bright region.



Figure 3 Optical microstructure at the onion ring region at two different magnifications of the FSPed AA7075-O (left) and after T6 heat treatment (right).



Figure 4 Optical microstructure inside the NG region of the FSPed AA7075-O at the left and after T6 heat treatment at the right.



Figure 5 Optical microstructure at the onion ring region at two different magnifications of the FSPed AA7075-O to the left and after T6 heat treatment to the right.

3.3 Micro-Hardness Results

Figure 6 shows the microhardness profiles for the different conditions of the tested materials (FSPed AA70-75-O and after T6 heat treatment) in comparison with the base material hardness.

The microhardness results prove the increase in the microhardness gradually from the base metal to the SZ. Though the increase in microhardness is 7% for the T6, and 21% for the O condition.

3.4 SEM Examination

Figures (7-9) show the SEM images of sample R7 where the dispersion of the Al2O3 particles is evident both uniformly inside the matrix as well as agglomerating into larger particles in some areas as shown in Figs. (8 and 9). The uniform overall distribution of the nanoparticles in the FSPed matrix interprets the enhancement in the hardness after the FSPing (see Fig. (6)).



and after T6 heat treatment



Figure 7 SEM macrostructure of a friction stir processed sample R7

The size and analysis of the Al2O3 nanoparticles was confirmed by SEM analysis as shown in Figs. (9 and 10).



Fig. 8 SEM image of sample R7



Fig. 9 SEM image of sample R7 showing a 7 m agglomerate of Al2O3

The SEM proves the success of friction stir processing technique in incorporating the nanoparticles into the matrix during the friction stir processing which provides a continuous deformation and dynamic recrystallisation to the processed matrix.

Though the enhancement in hardness and other mechanical properties of the FSPed alloys has been attributed to many researchers by the significant refining of the grains resulting from the continuous dynamic recrystallization encountered during the process, the significant role of adding the nano particles cannot be overruled by comparing the hardness levels in the retreating and advancing sides. The denser concentrations of the Al2O3 particles is expected to occur in the advancing site since the particles will be carried by the tool and allowed to embed in the matrix.

Also, one significant observation in this work is the presence of the T6 precipitation phases of (AlZn compounds) in the base metal (BM) after T6 heat treatment, whereas the nugget heat affected zones (HAZ) seem to be free of these phases (gray phases in Fig. (11)). These observations suggest that the nanodispersions impede the well-known precipitation process during the aging process as has been suggested by one of the authors before [19].

Element	Weight%	Atomic%
O K Al K Cl K	64.50 30.15 5.35	76.07 21.09 2.85
Totals	100.00	



Fig. 10 Sample R7 showing an Al2O3 agglomerate (left) and nanoparticle (right, with analysis on top.



Element	Weight%	Atomic%
Al K Zn L	94.46 5.54	97.64 2.36
Totals	100.00	

Fig. 11 BM (left) and HAZ (right) of the FSped sample R7

4. Conclusions

Friction stir processing can be successfully utilized to develop surface- nanocomposites with Al2O3 nanodispersions homogeneously embedded in the matrix. The new surface nanocomposites showed enhancement in the hardness of the surface of A7075 in T6 condition compared to the monolithic alloy in similar T6 heat treatment state.

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References

- [1] H. Bisadi, A. Abasi., "Fabrication of Al7075/TiB₂ Surface Composite Via Friction Stir Processing", American Journal of Materials Science, 2011, pp. 67-70.
- [2] A. Kurt, I. Uygur, E. Cete, "SurFace Modification of Aluminum by Friction Stir Processing", Journal of Materials Processing technology 211., 2011, pp. 313-317.
- [3] Palashpoddar, V.C. Srivastava, P.K. De, K.L. Sahoo, "Processing and Mechanical Properties of Reinforced Cast Magnesium Cast Matrix Composites by Stir Casting Process", Materials Science and engineering A 460-461, 2007, pp. 357-364.
- [4] T.P.D. Rajan, R.M. Pillai, B.C. Pai, K.G. Satyanarayana, P.K. Rohatgi, "Fabrication and Characterization of AL-7Si-0.35Mg/Fly Ash Metal Matrix Composites Processed by Different Stir Casting Routes" Composites Science and Technology 67, 2007, pp. 3369-3377.
- [5] K. Lee, K. Euh, D-H. Nam, S. Lee, N.J. Kim, "Wear Resistance and Thermal Conductivity of Zr-Base Amorphous Alloy/Metal Surface Composites Fabricated by High-Energy Electron Beam Irradiation" Material Science and engineering A 490-451, 2007, pp. 937-940.
- [6] W. Wang, Q.Y. Shi, P.Liu, H.K. Li, T. Li, "A Novel Way to produce Bulk SiCpReniforced Aluminum Metal Matrix Composites By Friction Stir Processing", Journal of Materials Processing technology 209, 2009, pp. 2099-2013.
- [7] Mishra, M. W. Mahoney, S. X. McFadden, N. A. Mara, and A. K. Mukherjee: *Scripta Mater.*, 2000, pp. 163-68.
- [8] Mishra, R. S., and M. W. Mahoney: *Mater. Sci. Forum*, 2001, vols. 357-359, pp. 507-12.
- [9] Ma, Z. Y. "Friction Stir Processing Technology: A Review ." *The Minerals, Metals & Materials Society and ASM International*, Vol 39A, 2008: p 642.
- [10] Mishra, R. S., Z. Y. Ma, and I. Charit: Mater. Sci. Eng., A, 2002, vol. A341, pp. 307-10.
- [11] Berbon, P. B., W. H. Bingel, R. S. Mishra, C. C. Bampton, and M. W. Mahoney: *Scripta Mater.*, 2001, vol. 44, pp. 61-66.
- [12] Spowart, J. E., Z. Y. Ma, and R. S. Mishra: *in Friction Stir Welding and Processing II*, K. V Jata, M. W. Mahoney, R.S. Mishra, S.L Semiatin, and T. Lienert, eds., TMS, Warrendale, PA, 2003, pp. 243-52.
- [13] Ma, Z. Y., S. R. Sharma, R. S. Mishra, and M. W. Mahoney: Mater. Sci. Forum, 2003, vols. 426-432, pp. 2891-96.
- [14] S.F.K. Bozorg, A.S. Zarghani, and A. Zarei-Hanzaki, Fabrication of Nano-Composite Surface Layers on Aluminium Employing Friction Stir Processing Technique, CP1217, Interntaional conference on advancement of materials and nanotechnology 2007(ICAMN 2007).

- [15] E. R. I. Mahmoud*, M. Takahashi, T. Shibayanagi and K. Ikeuchi, Effect of friction stir processing tool probe on fabrication of SiC particle reinforced composite on aluminium surface, Science and Technology of Welding and Joining (2009) VOL 14 NO 5, P. 713.
- [16] M. Azizieh, A.H. Kokabi, P. Abachi ,Effect of rotational speed and probe profile on microstructure and hardness, of AZ31/Al2O3 nanocomposites fabricated by friction stir processing, Materials and Design 32 (2011) 2034–2041.
- [17] T. S. Mahmoud, A. M. Gaafer, and T. A. Khalifa; "Effect of tool rotational and welding speeds on microstructural and mechanical characteristics of friction stir welded A319 cast Al alloy"; Materials Science and Technology, 24 (5), 2008 553-559.
- [18] I Charit and R. S. Mishra; "Abnormal grain growth in friction stir processed alloys"; Scripta Materialia 58 (2008) 367–371.
- [19] I. El-Mahallawi, H. Abdelkader, L. Yousef, A. Amer, J. Mayer, A. Schwedt.: "Influence of Al2O3 nano-dispersions on microstructure features and mechanical properties of cast and T6 heat-treated Al Si hypoeutectic Alloys", Materials Science & Engineering A 556 (2012) 76–87.