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Influence of Nitrogen Environment on the Surface of LM25 using GTA

R. Saravanan*, R. Sellamuthu, Arjun Nair, Sarat Dev Mangattu, A. Susaritha, S. Shruthilayaa and Vineeth Vasudevan

Department of Mechanical Engineering, Amrita School of Engineering – Coimbatore, Amrita Vishwa Vidyapeetham University, Coimbatore - 641112, Tamil Nadu, India; r_saravanan@cb.amrita.edu, r_sellamuthu@cb.amrita.edu, arjun.nair108@gmail.com, sarat.dev95@gmail.com, susarithaanbarasu@gmail.com, shruthnair.95@gmail.com, furoneton@gmail.com

Abstract

Objective: The main aim of this project is to modify the surface of LM 25 ingot in order to have a refined microstructure, improvement in hardness and in the wear rate of the modified region. **Methods:** Surface modification of LM 25 is carried out using GTA method. Argon flow was minimised and simultaneously pure nitrogen (99.999%) was introduced into the environment. Microstructural observation was carried out using Zeiss Axiovert 25CA metallurgical microscope and the hardness was measured using Mitutoyo Vicker's hardness testing machine. Wear test was carried out for the substrate and the modified region using pin-on-disc wear tester. EDAX analysis was carried out to find the presence of intermetalic compounds. **Findings:** From the microstructure, it was observed that there is grain refinement in the modified region. The hardness for the substrate was found to be 80.6 HV and 764.4 HV for the modified region. The wear rate in the substrate was 49.34x10^{-4} mm^3/m and 7.9x10^{-4} mm^3/m for the modified region. As the hardness increases, wear rate decreases. EDAX report confirms the presence of intermetalic compound in the form of silicon nitride (Si_3N_4). The presence of the silicon nitride is not reported previously using GTA as heat source on surface modified LM25. The increase in the hardness and decrease in the wear rate is attributed to the presence of silicon nitride in the surface. **Application/Improvements:** Due to the presence of silicon nitride it can be used as an insulator and chemical barrier in electrical circuits etc. It can also have wider automobile and aeronautics applications as its hardness has increased.

Keywords: GTA, Hardness, Microstructure, Surface Modification, Wear Rate

1. Introduction

Aluminium is the second most abundantly used metal. Aluminium and its alloys have a large number of applications due to its advantageous properties. The alloy shows poor mechanical properties. This can be overcome by modifying properties in bulk or in surface. Modifying the bulk properties consumes large quantity of material. Researchers have suggested a technique known as surface refining/modification to improve the surface property for the required application. Many researchers have worked to improve the surface properties using various techniques like Physical Vapour Deposition (PVD), Chemical Vapour Deposition (CVD), Laser, E-beam and coatings. The researchers have analysed and proved that the properties increase. Coatings are effective on the top layer of the base metal whereas modification using a heat source effect the surface and near surface as layers thereby improving the properties to a desired depth which is known as Surface Modification Process (SMP). The surface material is heated using a suitable heat source to form the molten pool. The molten pool is allowed to solidify quickly as the heat source moves on the base material[1]. In their study of optimising laser transformation hardening process have reported that the controlled formation of microstructural refinement will have great impact and improve the properties of the materials like hardness and wear[2]. Have observed in their study that nitride layers get formed when treating aluminium

*Author for correspondence

in nitrogen atmosphere. The alloy's major compositions are Al -99.3%, Fe-0.4%, Si-0.25% and Cu-0.05% (in wt%). The authors found the hardness of the base metal to be 80 HV and had increased to 690 HV in the modified region[3]. Performed their study on AlN coatings using Laser Surface Modification. They came to a conclusion stating that the formation of nitrides in the base metal will help to improve the hardness and wear resistance of the metal[4]. On his study on 4 and 5 wt% Cu-Al alloy using lasers as heating source observed that the materials treated via rapid solidification tends to show better refined microstructure. The formation of cellular dendritic structure was observed on the surface and near surface region (upto 5 µm) which is theoretically believed to improve the surface hardness[5]. On remelting with Al-Cu(32.7wt%) eutectic alloy the microstructure becomes is regular and lamellar at a solidification rate of 20cm/s , this causes a potential effect in practical application[6]. Reported that laser surface melting of Al-Cu (Cu 15wt%) improves the microstructure and yields a higher surface hardness. The maximum hardness observed in their investigation was 210 HV[7]. Experimented on investment cast D357 aluminium alloy and found out that the fatigue strength of GTAW repaired aluminium alloys have decreased. The alloy composition is Al -91.5%, Si-6.5%, Mg-0.55% and Fe-0.15% (in wt%). The decrease in fatigue strength was due to the formation of defects during the process and not due to the process chosen. This can be avoided by choosing proper parameters for the GTA process[8]. Conducted an experiment on Aluminium 2219 alloy for grain refinement through arc manipulation process using GTA welds and reported that severity of weld defects if the solidification structure is refined[9]. Conducted experiment on Al -11wt%Si to investigate the wear and corrosion resistance it was observed that the hardness has been increased from 55VHN to 87VHN[10]. In conducted an experiment on Al - 12wt%Si to study the microstructure and corrosion properties and he observed that the structure changes from eutectic to dendritic structure. Investigated the effect of surface refining on Al - 7.3%Si alloy and observed a hardness of 120 HV compared to the 80 HV of the base metal[11]. High power CO_2 and YAG lasers have been used to produce laser conducting welds on AA5083 with gauge size 2mm and 3mm with the help of defocused beams[12]. A ceramic mixture of Al_2O_3-SiC-C is fabricated by carbothermal reduction fly ash[13]. Compared to the other experiments GTA produce much cleaner weld in presence of nitrogen gas and is comparatively cheap. In this study an attempt was made in LM25 to increase the hardness and wear rate by Surface Modification Process (SMP) using GTA as heat source under nitrogen environment.

It's been observed from the earlier studies that the different surface modification processes such as CVD, PVD, e-beam and laser surface melting refine the microstructure which improves the surface properties. A few researchers have suggested that employing GTA as heat source was an inexpensive method for surface modification process. The motivation for this study was to identify the presence of nitrides when LM25 was surface modified under nitrogen environment using GTA.

2. Experimental Procedure

LM25 ingot was acquired. The composition found out using arc spectrometry test is mentioned in Table 1.

The ingot was sliced and melted in a graphite crucible at 750 °C in a furnace, in inert atmosphere. The molted alloy was sand cast and the cast bar was machined to a size of 150x30x30 mm using face milling machine. The surface of the bar was modified using GTA as heat source. The parameters considered for the GTA process are tabulated in Table 2.

The arc was initiated using argon as the shielding gas and nitrogen was introduced into the argon environment by slowly minimizing the argon flow rate.

Figure 1 shows the experimental setup. The surface modified specimen was wire cut along the cross sectional area, polished metallurgically and etched using Keller's reagent solution. The microstructure was observed under Zeiss Axiovert 25CA metallurgical microscope was used to observe the dissimilarity between the substrate and the modified region.

Table 1. Composition of substrate

COMPONENT	Al	Si	Fe	Cu	Mg
Wt%	89.5	8.2	0.9	0.5	0.4

Table 2. GTA parameters

PARAMETERS	VALUE
Current	150 A
Travel speed	2 mm/s
Arc length	3 mm
Electrode tip angle	180°
Argon flow rate	12 L/min
Nitrogen flow rate	18 L/min

Figure 1. Experimental equipment.

2.1 Hardness Testing

The hardness on the surface, the modified layer and the substrate was measured using MitutoyoVicker's hardness testing machine. A indenter made out of industrial diamond was used to indent the surface of the modified region and the substrate with an applied constant load of 100 gm for a dwell time of 15 s were used for hardness measurements.

2.2 Wear test

The wear rate was measured using pin-on-disc wear testing apparatus and the parameters were fixed in accordance with the ASTM G99 specification. Parameters for the testing were track diameter-110 mm, load - 20 N, speed - 424 rpm and time - 600 s. the material used for the counterpart is EN 31 hardened steel disk of 60 HRC and the surface roughness 0.15 Ra. The wear rate for the substrate and the modified layer was calculated from the wear vs. time graph.

3. Result and Discussion

3.1 Microstructure

Figure 2 shows the microstructure of the substrate where the silicon was found as globular structure which were dispersed in the α-Al matrix. Figure 3 shows the microstructure of the modified region. From Figure 3 it can be confirmed that the formation of finely dispersed globular eutectic silicon by GTA process under nitrogen environment was observed. On observing the microstructure of the modified region, it was observed that the grain size has been refined results in improvement in hardness in the modified region.

Figure 4 shows the Scanning Electron Microscope (SEM) image. From Figure 4, it is observed that the formation of nitride compounds in the modified region as the experiments were carried out in nitrogen environment. The presence of nitrides in the modified region has influence in the enhancement of the surface hardness and reduction in the wear rate.

3.2 Hardness

The hardness profile along the different layers of depth is shown in Figure 5. It can be seen that the hardness decreases in depth direction. The increase in hardness is

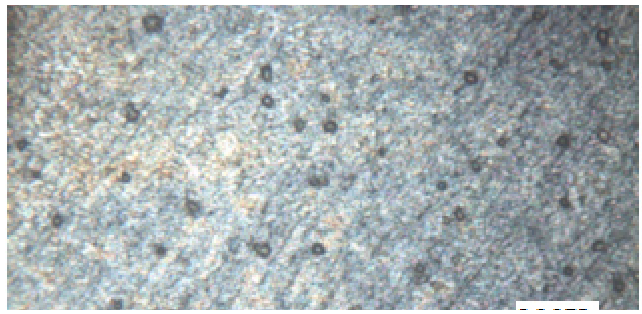

Figure 2. Microstructure of the substrate.

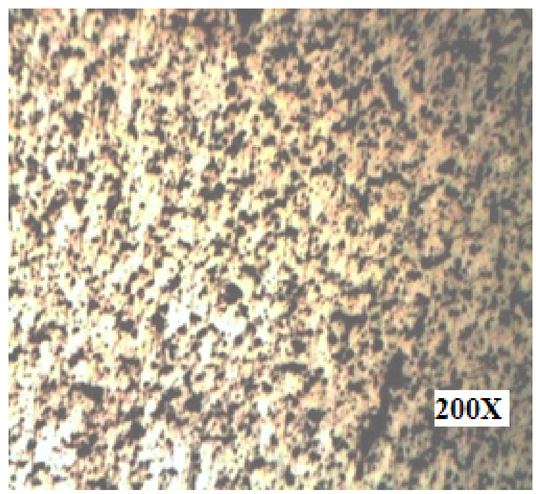

Figure 3. Microstructure of the Modified region.

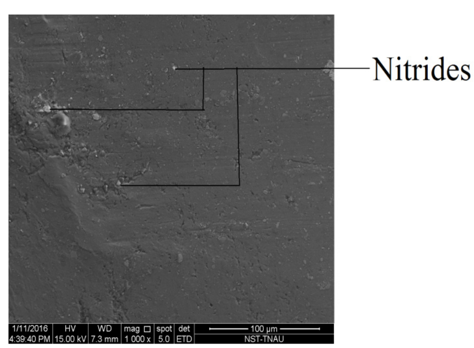

Figure 4. SEM image of the modified region.

due to the change in the microstructure which was due to the formation of nitride layers. The highest hardness of the nitride layer was about 764.4 HV on surface of the modified region which is found to be higher than that of the substrate which was measured to be 80.65 HV.

Noted as the depth increases, the amount of nitrogen that gets diffused to form nitrides reduces. This result shows the gradient in the hardness value[14].

3.3 Phase Identification

Figure 6 shows the EDAX setup. The composition in the modified region was analysed using EDAX testing facility. The focus was to find the availability of nitrides in the modified region. The analysis was carried out at specific points in the modified region. Figure 7 shows the EDAX analysis which confirms the presence of different phases of silicon and nitride. 40.28 wt% of N and 59.72 wt% of Si composition was found near the top surface in the modified region. This composition was identified to be Silicon Nitride(Si_3N_4). Figure 8 shows the EDAX report[15]. reported that silicon could undergo nitriding to form β-Silicon Nitride(Si_3N_4) when heated to about 1450°C in nitrogen atmosphere. It could also be formed by decomposing α-silicon nitride at temperatures greater than 1850°C[15]. From their study concluded that silicon nitride(Si_3N_4) is a stable compund. They performed XRD analysis on N-Si alloys eqilibrated at 1100 to 1700°C and confirmed the formation of Si_3N_4[16,17]. It was also mentioned that the formation of other silicon nitrides like silicon sequinitide(Si_2N_3) and silicon mononitride(SiN) is very less as they are unstable at low temperatures.

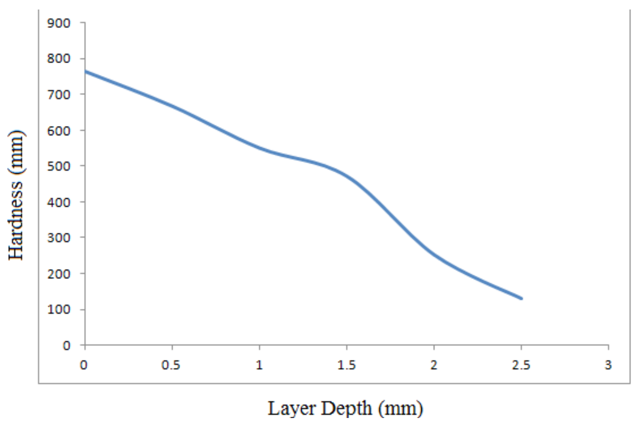

Figure 5. Hardness VS Depth (mm).

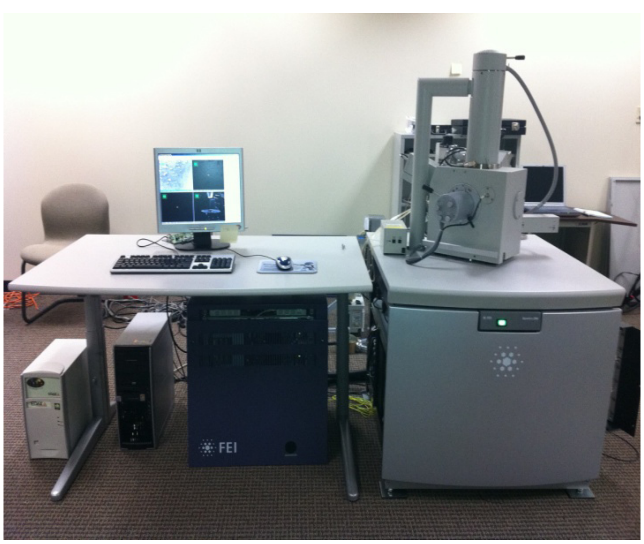

Figure 6. SEM and EDAX equipment.

3.4 Wear Rate

Figure 8 shows the wear rate graph. From the graph the wear rate of the modified region was calculated to be $7.9x10^{-4}$ mm^3/m which is significantly less when compared to that of the substrate $49x10^{-4} mm^3/m$. This is due to the

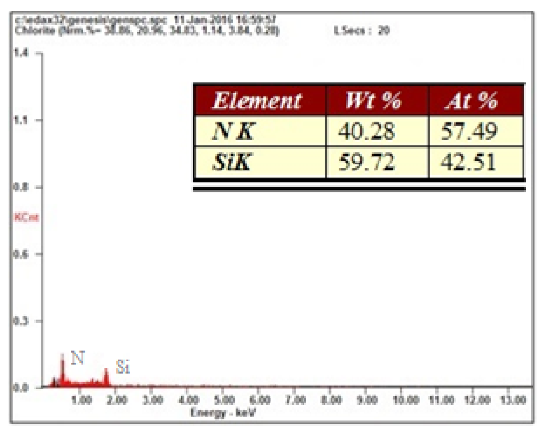

Figure 7. EDAX report.

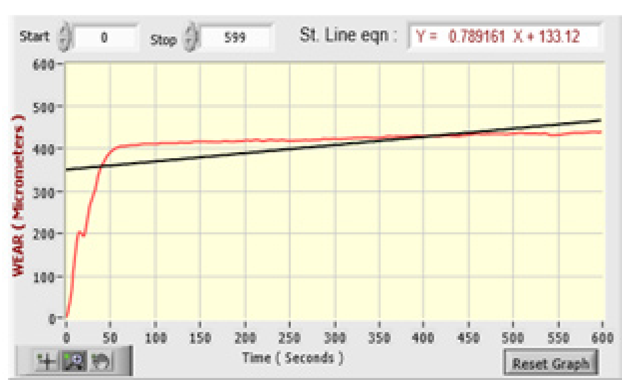

Figure 8. Wear (micrometer) v/s time (s).

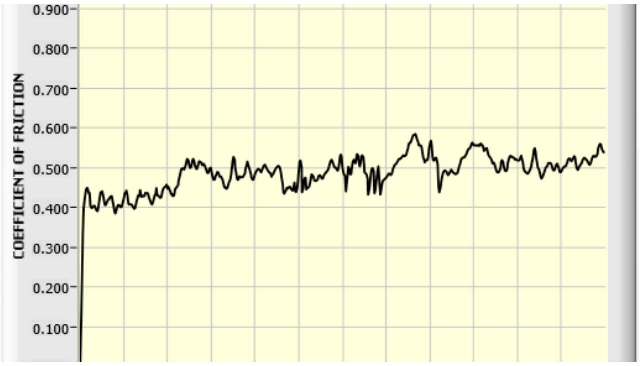

Figure 9. Coefficient of Friction v/s time (s).

formation of the nitride layers. As the hardness increases the wear rate decreases. This statement is in agreement with the Archard's theory (1953).

3.5 Coefficient of Friction

Figure 9 shows the marginal increase in coefficient of friction.

4. Conclusion

Surface modification of LM25 in the presence of nitrogen environment yielded a higher surface hardness and reduced wear rate. The rapid cooling associated with GTA process causes the formation of highly refined grain structure. Presence of nitrogen during surface modification process using GTA results in the formation of nitrides in the surface layers. The refined grain structure and the presence of hard nitrides are identified to be the reasons for enhancement in surface hardness and for the reduction in wear rate.

5. References

1. Cheung N, Ierardi MC, Garcia A, Vilar R. The use of artificial intelligence for the optimization of a laser transformation hardening process. Lasers in Engineering. 2000; 10(4):275–91.
2. Zheng X, Ren Z, Li X, Wang Y. Microstructural characterization and mechanical properties of nitrided layers on aluminum substrate prepared by nitrogen arc. Applied Surface Science. 2012 Oct; 259:508–14.
3. Meneau C, Andreazza P, Anderazz-Vignolle C, Goudeau P, Villain JP, Boulmer-Leborgne C. Laser surface modification: Structural and Tribological studies of AlN coatings, Surface and Coatings Technology. 1998 Mar; 100-101:12–6.
4. Munitz A. Microstructure of rapidly solidified laser molten Al-4, 5 WtPct Cu Surfaces. Metallurgical Transactions. 1985 Dec; 16(1):149–61.
5. Zimmermann M, Carrard M, Kurz W. Rapid solidification of Al-Cu eutectic alloy by laser remelting. ActaMetallurgica. 1989 Dec; 37(12):3305–13.
6. AparecidaPinto M, Cheung N, FilippiniIerardi MC, Garcia A. Microstructural and hardness investigation of an aluminium–copper alloy processed by laser surface melting. Materials Characterization. 2003 Mar; 50(2):249–53.
7. Li L, Liu Z, Snow M. Effect of defects on fatigue strength of GTAW repaired cast aluminium alloy. Welding Journal. 2006 Nov; 1–6.
8. Koteswara SR, Rao G, Madhusudhana R, Kamarj M, Prasad Rao K. Gain refinement through arc manipulation techniques in Al-Cu alloy GTA welds. Material science and Engineering. 2005 Sep; 404(1):227–34.
9. Biswas A, Mordike BL, Mannaand I, Dutta Majumdhar J. Studies on Laser Surface Melting of Al-11%Si Alloy. Laser in Engineering. 2009 Jan; 18(1):95–105.
10. Watskins KG, Mc Mahon MA, Steen WM. Microstructure and corrosion properties of laser surface processed Aluminium alloys: A review. Material Science Engineering A. 1997 Jul; 231(1):55–61.
11. Saravanan R, Sellamuthu R. An investigation of the effect of surface refining on the hardness and the wear properties of Al-Si alloy. Applied Mechanics and Material. 2014 Jun; 592-594:53–7.
12. Moarrefzadeh A. Finite-Element simulation of aluminum temperature field and thermal profile in laser welding process. Indian Journal of Science and Technology. 2012 Aug; 5(8):1–6.
13. Senapati AK, Mishra PC, Routray BC, Ganguly RI. Mechanical behavior of Aluminium Matrix Composite Reinorced with untreated treated Waste Fly Ash. Indian Journal of Science and Technology. 2015 May; 8(89):111–8.
14. Heydarzadeh M, Sohi M, Ansari M, Ghazizadeh, Zebardast H. Liquid phase surface nitriding of aluminium using TIG process. Surface Engineering. 2015; 31(8):598–604.
15. Forgeng WD, Decker BF. Nitrides of Silicon. Transactions on Metallurgical Society American Institute of Mechanical Engineering. 1958; 212:343–8.
16. Gupta M, Rathi VK, Thangaraj R. The preparation, properties and applications of silicon nitride thin films deposited by plasma-enhanced chemical vapor deposition. Thin Solid Films.1991 Sep; 24(1):77–106.
17. Carlson ON. The N-Si (Nitrogen-Silicon) System. Bulletin of alloy phase diagrams. 1990 Dec; 11(6):569–73.